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| <b>14. ABSTRACT</b><br>An experimental program to develop long life design criteria for 5-lbf thrust monopropellant hydrazine engines using Shell 405 catalyst is described. The test results of the effects of injector design variables, catalyst bed composition/retention, accumulated cold starts, reactor materials and thermal management on thruster life are discussed. The test matrices allowed for a variation of the superficial mass flux from 0.01 to 0.03 lbf/sec sq. in., injection velocity from one to 100 fps, orifice size from two to 20 mil and for a change in injection patterns and number of cold starts. Besides showerhead injectors, representative flight-type configurations were tested. Catalyst weight loss, rate of increase of bed pressure drop with number of pulses P sub c-roughness variation and the preand post-fired catalyst particle size spectrum were determined. |                                    |                                               |  |                                                     |                                                                         |
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INTERIM REPORT FOR PERIOD JANUARY 1973 - DECEMBER 1974

BELL AEROSPACE COMPANY  
P.O. BOX ONE  
BUFFALO, N.Y. 14240

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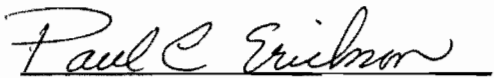
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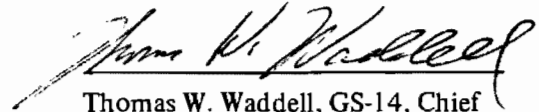
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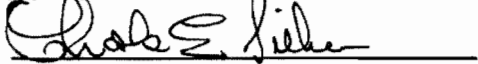


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The evaluation of 19 candidate materials and coatings, which were exposed to  $N_2H_4$  decomposition products for up to 10 hours, revealed that platinum-10% rhodium and tungsten are unaffected, and only minor effects were observed on Nichrome V.

Data on effective thermal conductivity and specific heat of Shell 405 catalyst were obtained and used in a multi-nodal catalyst bed thermal model. Predictions were correlated with test results from two instrumented reactors. A series of tests to examine effects of propellant detonation in manifolds indicated that this mechanism is not a significant contributor to catalyst attrition.

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## SECTION 1.0 INTRODUCTION AND SUMMARY

The breakup and physical loss of catalyst material, which can occur through a variety of mechanisms collectively termed "attrition," is the single most important cause of reactor failure. The intrinsic fragility of the ceramic catalyst, coupled with the fluid dynamic, thermomechanical, and mechanical forces necessarily applied to it, makes a certain amount of attrition inevitable, irrespective of reactor design. Indeed, it is this inescapable attrition which renders the catalytic reactor a life-limited device. However, the degree of attrition which can be tolerated before a particular reactor has "failed" is both design- and mission-sensitive. Thus, the only variables over which the designer has some control are:

- Catalyst configuration — choice of catalyst type, particle size, quantity, and distribution;
- Catalyst bed retention — compartmentation, rigidity and loading;
- Injector hydraulics — injection velocity and propellant distribution;
- Injector configuration — injection pattern
- Thermal management — bed-to-injector/manifold conduction and intra-injector heat transfer; and
- Selection of materials — high-temperature structural properties, environmental compatibility, and thermal properties.

Of course, none of these variables can be addressed independently as they are intimately and inextricably interrelated.

### 1.1 OVERALL OBJECTIVES OF THE PROGRAM

This program, then, was intended to provide an understanding of the effects of monopropellant hydrazine engine design parameters on catalyst attrition and thruster life. It was divided into four phases\*, the basic objectives of which were:

#### Phase I - Injector Design Effects

- Determine the influence of injection conditions on catalyst attrition.
- Assess the effects of thermal management on catalyst life.
- Derive basic thermal design criteria.
- Correlate fundamental parameters with attrition in different injector configurations.
- Correlate the degradation of steady-state and pulse performance with degree of attrition for a given duty cycle and injector configuration.

---

\*Originally, Phase IV was to demonstrate design criteria in a flight weight 5-lbf thruster. This was deleted and replaced by the more fundamental Phase IIIA effort.

## Phase II - Catalyst Bed Retention

- Evaluate catalyst containment/retention techniques.

## Phase III - Materials Selection

- Evaluate materials best suited for use in the hydrazine decomposition environment.

## Phase IIIA - Duty Cycle/Contamination Effects

- Determine effects of duty cycle and start temperature on catalyst attrition.
- Evaluate effects of sodium and iron contamination on catalyst life and thruster operation.

All of these phases are related, and in particular, Phases I, II and IIIA. This relationship together with an overview of the program approach is shown in Figure 1. A total of eighty-seven 5-lbf research thrusters and six special test units are involved. The data generated therefrom are correlated using appropriate techniques to identify primary factors, quantify relative effects and interactions, and provide other basic long-life design guidelines.

This program is fundamental in nature, and is intended to be relevant to all thruster geometries and designs within the constraints of the variables tested. However, the models and data provided should not be indiscriminately applied. Rather, they are tools for the engineer knowledgeable in this field to use in conjunction with his own observations and interpretations.

## 1.2 PROGRAM SUMMARY

The first three phases of the program were completed in November 1974 and are the subject of this report. Phase IIIA, which involves expansion of the data base to include effects of duty cycles, start temperature, and contaminants, began in November 1974 and will be reported at a later date.

Phase I, Injector Design Effects, included tests to determine effects of injection velocity, propellant distribution, catalyst selection, thermal management, and element geometry on attrition. In this phase, a standard two-piece 5-lbf thruster was used as the basic tool.

Injection velocity and propellant distribution effects were evaluated concurrently in a matrix of 39 tests designed to obtain both separate and combined effects. The test profile in each instance was a series of cold starts. The basic test matrix was done with 20-25 mesh Shell 405 ABSG catalyst. However, a cursory comparison of 14-18 mesh ABSG, 25-30 mesh ABSG, 20-25 mesh alpha-phase 405, and 20-25 mesh 90% attrited Shell 405 was included.

Two types of thermal management tests were conducted: Four pulse gun tests to assess the impact on catalyst attrition of pressure waves generated in the injector manifold, and a series of laboratory and firing tests to obtain and verify conductance inputs to thermal models. Thermal conductivities were determined as a function of catalyst particle size, packing pressure and temperature in a laboratory apparatus. These data were used in conjunction with the thermal model to instrument and predict thermal behavior of two reactors, a 1.1 inch diameter unit similar to the workhorse hardware and a larger unit designed to accommodate better instrumentation.

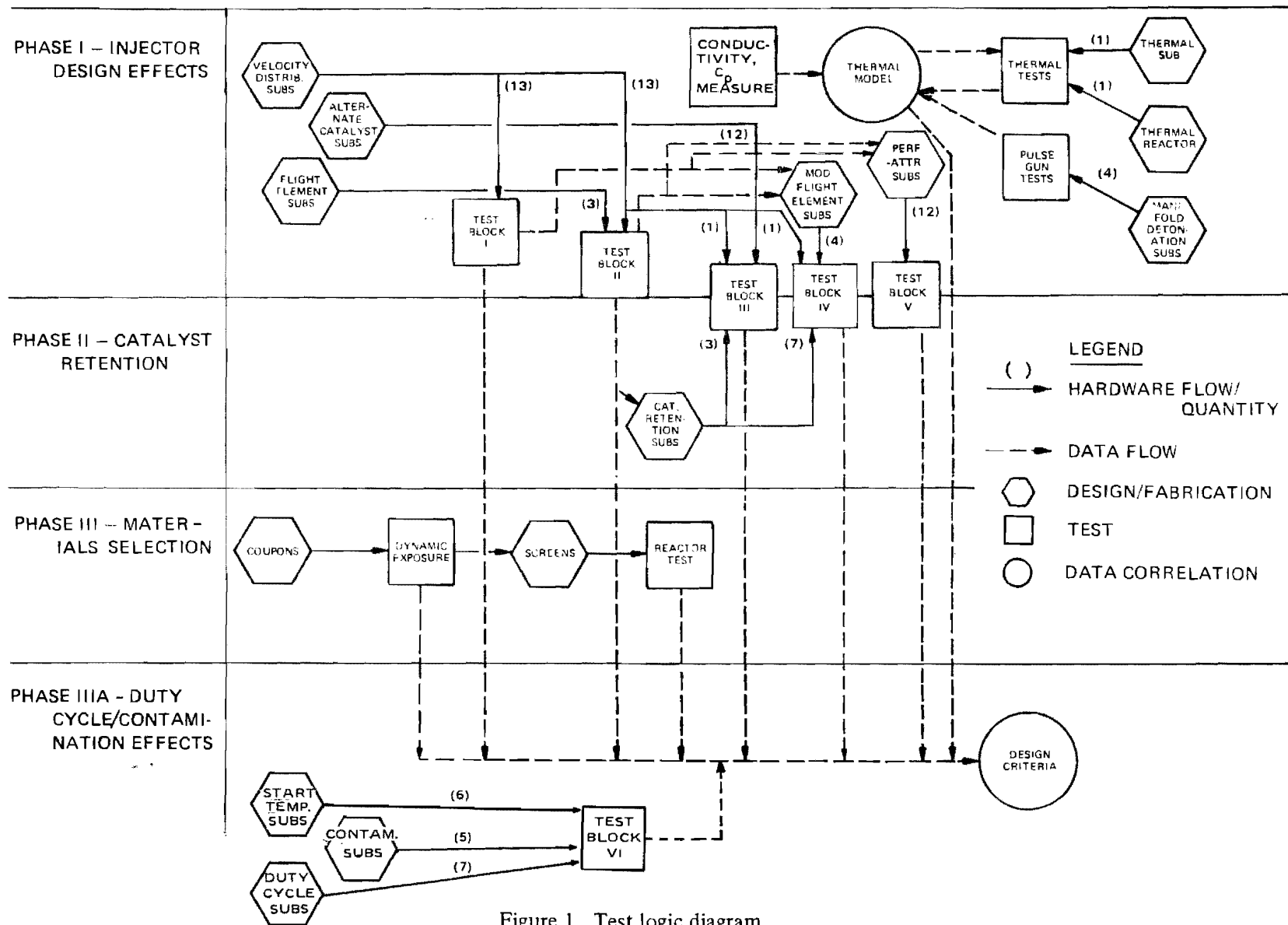


Figure 1. Test logic diagram.

A task to study injection element geometry was done to determine the effects of various injector designs on attrition. Three designs (a rigimesh spud type, a discrete stream spray/head-space type, and an imbedded capillary tube type) representative of current practice were tested. Modifications of these designs and a fourth "very low injection velocity" design were built and also tested.

The catalyst bed retention phase determined the relationship between catalyst bed retention and catalyst bed life using a "standard" injector. A total of ten modified workhorse units were tested. These included axial, radial, and circumferential compartmentation, and "spring" loading to maintain preload.

In Phase III, fourteen base materials were selected as representative of the best candidates from previous work. Also, five coatings/platings were tested in combinations with the base materials. Coupons of these materials were subjected to ten hours of exposure in a test vessel continuously replenished with hydrazine decomposition products.

From the analysis of the coupons sampled, the best representative materials attainable in screen form were selected. These were incorporated in a gas generator and subjected to 200,000 high-rate pulsing cycles. Metallographic examination of the exposed samples provided a quantitative assessment of their compatibility with this environment.

The culmination of the program to this point was the correlation of the data obtained and their incorporation into "models" relating design variables to attrition measurement parameters. This data correlation involved consolidation of relevant information from the various program phases. For this reason, it is not practical to report the results phase by phase. Therefore, the remainder of this report deals directly with the three technical areas addressed: Reactor Design Effects, Materials Evaluation, and Thermal Management - with only incidental reference to the program phase.

## SECTION 2.0 REACTOR DESIGN EFFECTS

The major components of a monopropellant reactor are the injector and the catalyst bed.

The catalyst is confined, within a surrounding shell, between the injector and a final screen or screens which are, in turn, supported by a retainer plate. Most operational reactors employ a layered catalyst bed with fine mesh catalyst near the injection element. This practice places the maximum specific surface area where it is most needed. Since fine mesh catalyst produces a higher pressure drop, the bulk of the bed is usually filled with coarser mesh catalyst. This gives some additional reactive area and "flame front" stability in the region where most of the decomposition has already taken place but results in a lower overall pressure drop. A few designs use a deeper bed of a single intermediate mesh size. If a layered bed is used, the separation between the different particle sizes is provided by a screen or screen-cum-retainer.

Perhaps the major difference between the various operational designs is in injection element configuration. Several different types have been used with some measure of success. The showerhead type, the penetrant, and the porous plate type are a few examples.

The intent of this part of the program was:

- To isolate, as far as possible, the key design/operating variables attributable to attriting mechanisms;
- To design and conduct a series of judicious hot-fire experiments intended to determine empirically the effect of those variables on the rate of attrition;
- To develop a correlative/predictive capability through analysis of the experimental data, and from there develop design criteria for the minimization of attrition rate.

### 2.1 APPROACH

Since the life of a Shell 405/ $N_2H_4$  engine is inherently limited because of the frangible nature of catalyst, understanding the catalyst attrition mechanisms and the relationship between these and the injector/upper catalyst bed assembly could be treated as an experimental unit (Refer Figure 3). Within these experimental bed assemblies, each of the suspect attrition-inducing variables could be carefully and velocity, distribution, mass flux, temperature, pressure, state (liquid or quality of vapor) and catalyst bed characteristics are influential.

Attention was, therefore, focused upon the upper catalyst bed of a 2-layer reactor typical of 5-lb thrust monopropellant hydrazine engines. The approach was to utilize a research tool in which the injector/upper catalyst bed assembly could be treated as an experimental unit (Ref Fig 3). Within these experimental bed assemblies, each of the suspect attrition-inducing variables could be carefully and independently controlled and varied in a parametric manner from test to test. The data therefrom could be used to develop correlations quantifying the influence of each attritogenic variable. The experimental reactor was a take-apart catalyst bed consisting of an upper (test specimen) and a lower bed section, the latter being a stabilizing zone to complete the  $N_2H_4$  decomposition. The reactor was designed to meet the following requirements:

- Lowest possible manufacturing cost.
- Ease of assembly/disassembly to prevent accidental displacement of the fired catalyst bed.
- Capability to re-use the lower bed hardware and catalyst.
- Compatibility with the techniques used to evaluate attrition of the fired catalyst in the zone of interest.
- Ease in varying test (design) conditions within the same basic reactor envelope.

Reactor design variables, such as propellant injection velocity, propellant distribution and catalyst, representative of current practice and expected to be most influential, were adopted. A cold start duty cycle, which accelerated attrition, was necessary to keep the test time and cost to a minimum. For the same reason the reactors were "gang-tested" by firing blocks of up to 16 units in a single test setup.

#### 2.1.1 Injector Design Effects

The specific objective of this task was to examine the effects of propellant injection velocity, superficial and local propellant mass flux, injection element geometry and number of cold starts on the attrition of 20-25 mesh ABSG Shell 405 catalyst.

Matrices shown in Figure 2a and b were formulated for the velocity/propellant distribution test series. These allowed for variation of the superficial mass flux ( $G$ ) from 0.01 to 0.03 lbm/sec-in<sup>2</sup> (flow rate ranging between 0.011 and 0.033 lbm/sec), local mass flux ( $G'$ ) from 0.02 to 0.2 lbm/sec-in<sup>2</sup>, and injection velocity ( $V_I$ ) from 1 to 100 ft/sec. In addition, they provided a cursory examination of the injection pattern (Figure 2b) and the orifice size influences.

Positioning of orifices in predetermined locations over the surface of a flat-faced showerhead injector was chosen as the means of controlling distribution for this series. As noted, either 10%, 25% or 100% injector coverage\* was selected. Two cluster arrangements of either three or six groups of orifices, distributed over 25% of the injector area, were investigated. In these cases the number and size of the orifices was held constant.

The catalyst for this series was the "standard" 20-25 mesh Shell 405 ABSG preloaded during assembly to 50 psi. Nozzle throat diameters were sized to maintain a constant nominal chamber pressure ( $P_c$ ) of 100 psia at the three operating propellant flow rates.

For the basic matrices (Figure 2a and b), a duty cycle of 100 cold starts ( $50 \pm 10^\circ\text{F}$ ) was used. This provided only end-point attrition data, and a specific test series was added to examine the variation of attrition magnitude and performance with increasing hot-fire exposure and injection velocity. Twelve engines were tested. Fifty, 100, 150 or 200 cold starts were made on each of four engines at an injection velocity of 10 fps; four more engines were similarly fired at 50 fps and four at 100 fps.

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\*% area of the total available injector face over which orifices were uniformly distributed.

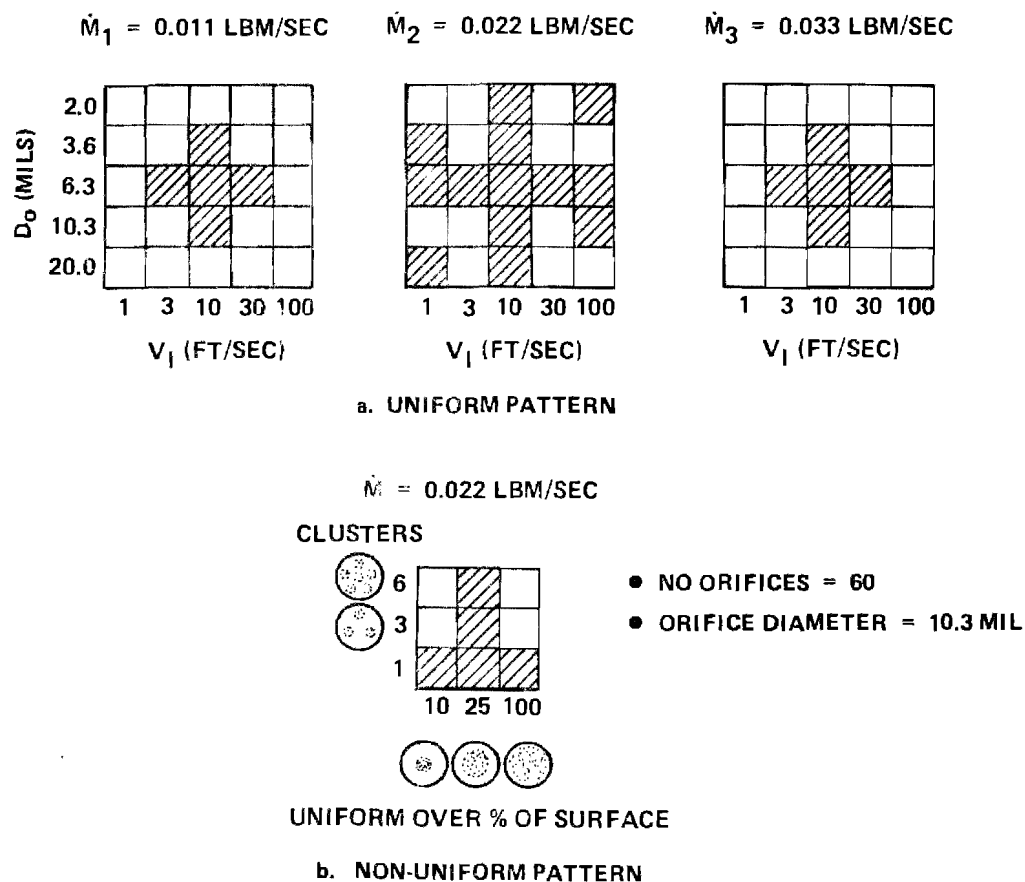


Figure 2. Velocity/distribution test matrix.

The geometric configuration of the injection elements themselves was studied as an attritogenic factor. This task consisted of two groups of experiments. In the first, three units were utilized, each containing an injector representative of one used in currently operational 5-lbf thrust hydrazine engines. The specific injectors tested were:

- Twelve-element embedded capillary penetrant
- Single-element rigimesh porous disc
- Discrete-stream-spray/head space

These designs (see Figures 4 through 6) are generally those used by Hamilton Standard, Rocket Research, and TRW, respectively. We acknowledge and appreciate the assistance of those companies in reviewing these designs for us.

These injectors were adapted to the standard reactor, loaded with 20-25 mesh Shell 405 ABSG catalyst and test-fired to the same 100 cold-start duty cycle and operating conditions imposed during the velocity/distribution test series. A constant flow rate of 0.022 lbm/sec was used for the element geometry tests.

Following the firing, these data were analyzed and based on those results, four "improved" flight-weight injector designs (group two) materialized:

- Six-element partial penetrant
- Six-element rigimesh porous disc
- Vortex
- Hemispherical

The first two are merely variations of those from group one; while the last two are different concepts. Their description is given later.

These injectors also conformed to the standard reactor envelope and were test-fired to the same duty cycle and operating conditions used for the first group, except that 14-18 mesh Shell 405 ABSG catalyst was loaded into the upper bed. The reason for the change in the catalyst size was the significantly smaller attrition observed in that size during the group one test series. This will be further discussed in Section 2.4.

We must emphasize that it was not an objective of this task to rate or rank these elements. Rather, these designs provided a means to make the basic experiment data more useful by extending the simple showerhead models to other operational configurations.

**Key Hardware** — The primary research tool in this test program was the Attritology Reactor Assembly (ARA). This unit, shown in Figure 3, is a take-apart reactor of the 5 lbf-thrust class. It consisted of the following components:

- Sacrificial Upper Bed (SUB) Subassembly
- Workhorse Lower Bed (WLB) Subassembly
- Clamp Ring
- Spacer
- Seal

and miscellaneous attach/mount hardware.

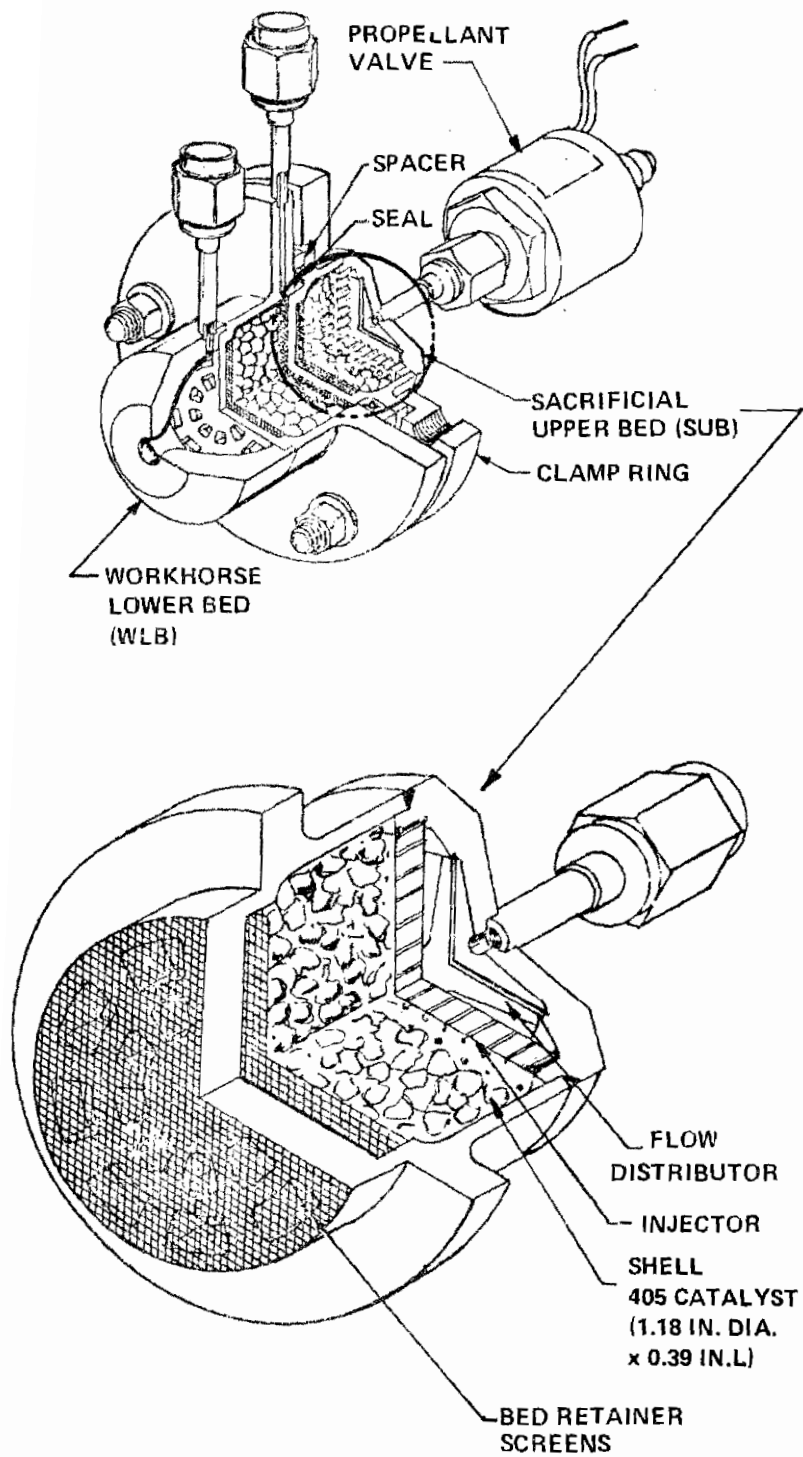


Figure 3. Attritology reactor assembly

The heart of the ARA was the SUB unit, an all-welded module illustrated in Figure 3. As the name implies, the SUB s were sacrificial modules, expended in each test on a one-for-one basis. Each SUB comprised: a flanged shell with integral retainer plate; two aft retention screens (40 x 40 mesh over 10 x 10 mesh) plus a 24 x 24 mesh screen downstream to retain lower bed; a bed of catalyst; and an injection head subassembly consisting of feed tube, cover, propellant distributor baffle, and injector plate. The design shown is typical. The SUBs tested to duty cycles requiring hot restarts had capillary tubes leading to an external propellant manifold thereby avoiding premature propellant heating before injection.

The WLB subassembly was a reusable unit designed to mate with the SUB and retain a stabilizing lower catalyst bed of 14-18 mesh Shell 405 ABSG. It consisted of a flanged shell with integral retainer plate, a nozzle, two aft-retention screens (24 x 24 mesh over 10 x 10 mesh), a catalyst bed, two pressure sensing lines, and a catalyst bed temperature probe. Three different WLB types were employed: the standard model was fitted with a nozzle appropriate to a propellant flow of 0.022 lbm/sec, while the low-flow and high-flow models embodied nozzles for the 0.011 and 0.033 lbm/sec flows, respectively. All of the nozzles had a sonic orifice with an expansion ratio of 1.0 and were designed for a nominal 100 psia chamber pressure.

The SUB and WLB shells were finish machined from 347 CRES investment castings. Most of the injectors for the flat-faced showerhead models were Brunswick Corporation "Collimated Hole Structure" units in 304 stainless steel: flat plates containing high aspect ratio orifices of the selected sizes and patterns. Coarse screening was Hastelloy B and the fine meshes Haynes 25. The remaining ARA components were machined from 304L stainless.

Catalyst was loaded in the SUB in accordance with an established procedure which prescribed vibration packing to volume and seating with a 50 psi preload. The SUB s were then EB-welded closed and assembled to the WLB. WLB catalyst loading was similar, but the closure was made with the flange rather than a weld.

Assembly drawings of the upper beds of the Penetrant Element, Rigimesh Porous Disc and Discrete-Stream-Spray Injector are shown in Figures 4 through 6. As previously noted, these are replicas of injectors utilized in currently-operational 5-lbf thrust hydrazine engines adapted to the standard SUB envelope. Their design is well known and therefore needs no further description. These SUBs were also mounted to the standard WLB. Figures 7 and 8 are photographs of variations of the Penetrant and Rigimesh Injector designs, respectively.

In addition to the number of elements, the basic difference between the original twelve-element penetrant and the six element unit was the shape of the penetrant. The latter was a hollow screen cylinder, about 5/32 inch in diameter x 1/4 inch long, protruding from the injector face. The screen was made from platinum-10% rhodium wire (45 mesh x 0.006 inch diameter wire). Each element was individually fed by a stainless steel tube (0.018 inch O.D. x 0.0095 inch I.D.), branching off a common external propellant manifold. The downstream end of the cylindrical penetrant was closed off to reduce the axial flow tendency. The anticipated improvement was a more uniformly distributed radial mass flux. This design also simplified fabrication and allowed a more direct comparison to other configurations.

The modified Rigimesh-element injector incorporated six 7/32 inch diameter spuds instead of one 19/64 inch diameter. As shown in Figure 8, one was positioned on centerline; while the remaining five were equally spaced on a 51/64 inch diameter circle. The manifolding was identical to that of the penetrant. The advantage of six elements versus one was the decreased local mass flux and propellant injection velocity which was expected to minimize catalyst break-up near the injection point.

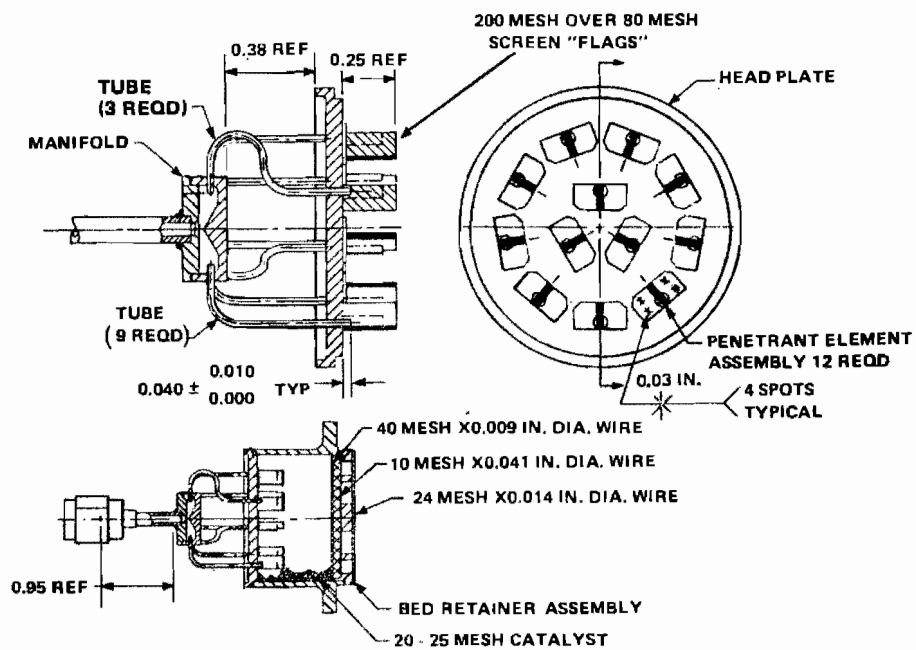


Figure 4. Upper bed-penetrant element.

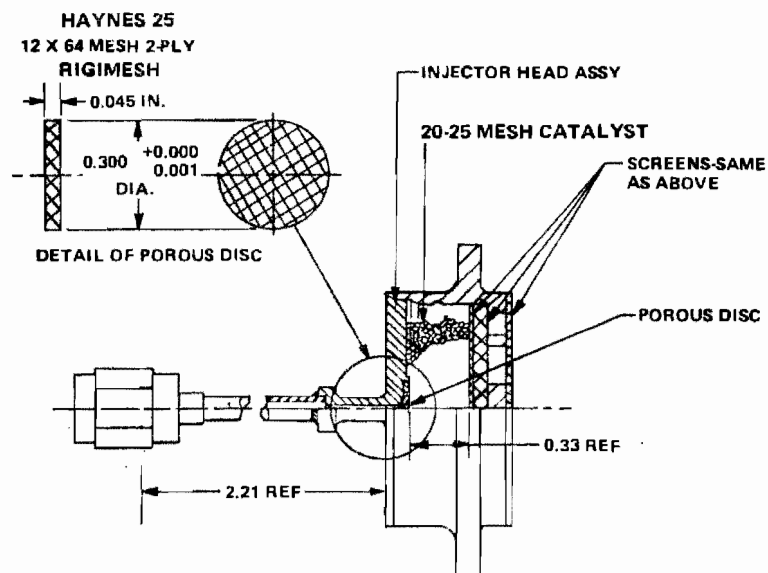


Figure 5. Upper bed-porous disc element.

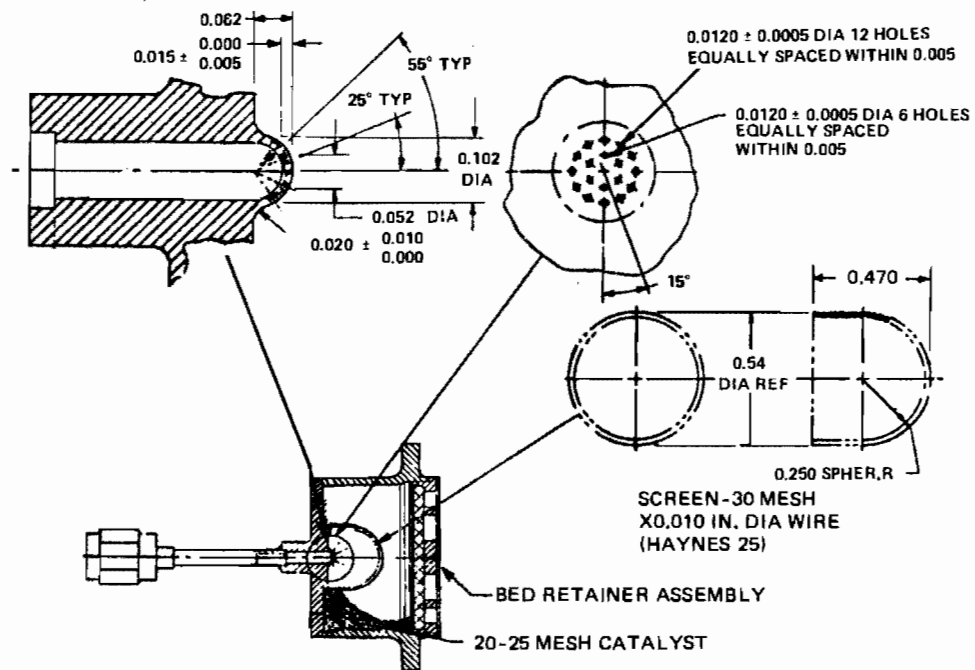


Figure 6. Upper bed-spray element.



Figure 7. Six element partial penetrant injector.



Figure 8. Six element Rigimesh injector.

In searching for injection schemes that produce hydrazine infiltration over the largest practical catalyst bed area (minimum mass flux), two concepts evolved: vortex and hemispherical injection. In the former, hydrazine enters a conical annulus tangentially via two feed tubes 180° apart, as seen in Figure 9. The diverging, conical flow passage is bounded by the solid chamber wall on the outside and by a two-layer screen on the inside through which the hydrazine diffuses into the catalyst bed. The hemispherical injector was similar to the vortex, except that the chamber wall and screens were hemispherically shaped, as illustrated in Figure 10. The hydrazine entered the spherical flow passage at the apex of the hemisphere through a radial diverter, located on centerline. This directed the propellant uniformly over the screen dome, allowing the fuel to diffuse into the catalyst bed. Both designs yielded very low injection velocities and local mass flux.

All four injector/chamber assemblies were mated with the standard WLB units.

#### 2.1.2 Catalyst Bed Composition/Retention Effects

To evaluate catalyst size and processing effects on attrition, Shell 405 catalyst of several different particle sizes and carrier conditionings was fired in conjunction with a 3-sample selection of research injector configurations from the matrix of types used in the velocity/distribution test series. This test-point matrix is given in Figure 11, which summarizes the various catalysts, propellant injection velocities and operating conditions used for this experiment.

Comparison with the results of the basic tests using 20-25 mesh catalyst thus permitted direct evaluation of the influence of catalyst selection under the selected test conditions.

To assess the feasibility of catalyst bed retention schemes to prevent or minimize attrition, compressive loading (mechanical and dynamic) and compartmentation of the bed were addressed. The underlying philosophy for bed compartmentation is that any voids and/or catalyst fines created during the firing may remain localized and not affect the balance of the bed. Similarly, the expected effect of compressive loading was to prevent void buildup and bridging in the catalyst column.

In the mechanical preload tests, the compressive load was controlled by the degree of interference between catalyst and injection head when the final closure weld was made. This preload, of course, changed significantly once the reactor was fired; only the initial value being determinable with any precision.

For the dynamic load tests, flexible support members were incorporated in the hardware to set and maintain the desired compressive load continuously on the bed. By comparison with the first group, these test results should indicate whether or not the benefits of a "constant" compressive load are worth the additional complexity. For the purpose of these tests, an external spring was used to reduce thermal effects and minimize spring rate changes.

In the compartmentation tests, appropriate separators were incorporated into the catalyst bed dividing it into discrete cells, either normal to or parallel to the direction of flow. The test point matrices are shown in Figure 12.

As noted, compressive loads of 50, 100 or 250 lbf (or psi since the bed cross section was approximately one in.<sup>2</sup>) were imposed on the upper catalyst bed for both the mechanical preload and the dynamic load reactors. The other operating conditions and duty cycle, listed in the figure, were identical to the ones used in the velocity/distribution test series.

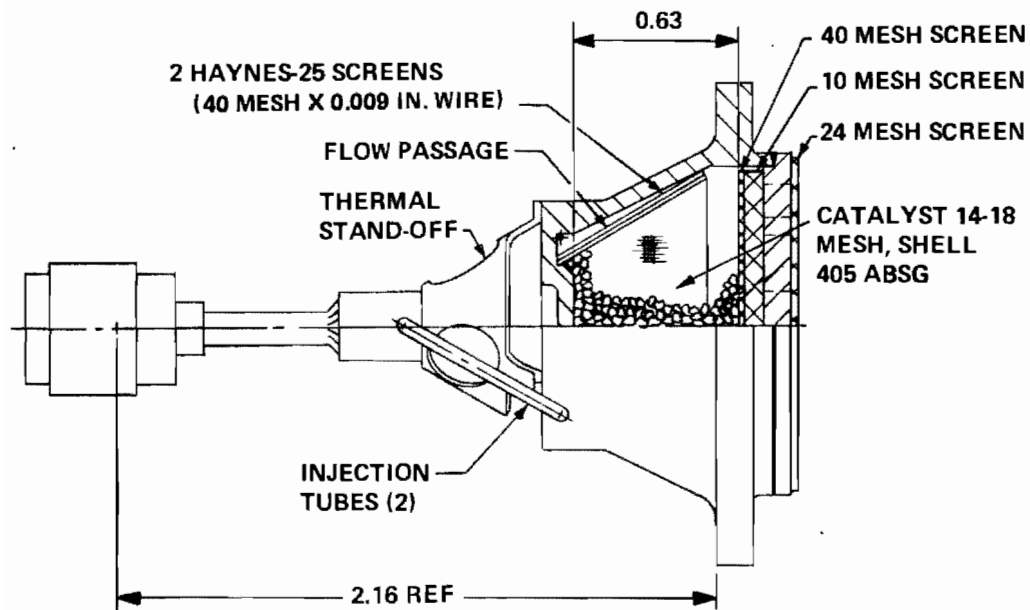


Figure 9. Vortex injector/upper bed.

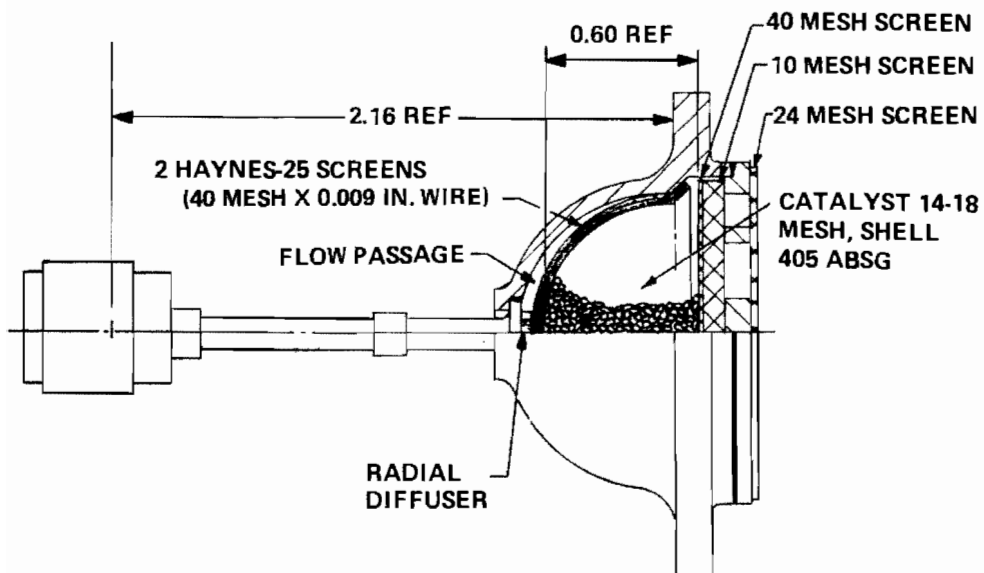


Figure 10. Hemispherical injector/upper bed.

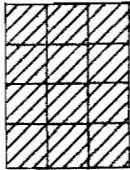
| CATALYST               |                                                                                   | OPERATING CONDITIONS           |
|------------------------|-----------------------------------------------------------------------------------|--------------------------------|
| 25 - 30 ABSG           |  | • $P_C = 100$ PSIA             |
| 14 - 18 ABSG           |                                                                                   | • $\dot{M} = 0.022$ LBM/SEC    |
| 20 - 25 90% ATT.       |                                                                                   | • UNIFORM PATTERN              |
| 20 - 25 $\alpha$ PHASE |                                                                                   | • 100 COLD START DUTY CYCLE    |
|                        | 3 10 30                                                                           | • 50 PSI CATALYST BED PRE-LOAD |
|                        | $V_1$ (FT/SEC)                                                                    |                                |

Figure 11. Alternate catalyst test matrix.

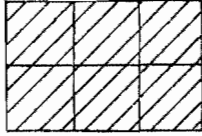
| COMPRESSIVE LOAD TESTS      |                                                                                     | COMPARTMENTATION TESTS |
|-----------------------------|-------------------------------------------------------------------------------------|------------------------|
| TYPE                        | COMP'N                                                                              |                        |
| MECH. PRELOAD               |  | AXIAL (SINGLE)         |
| DYN. LOAD                   |                                                                                     | AXIAL (DUAL)           |
|                             |                                                                                     | CIRCUM-FERENTIAL       |
|                             |                                                                                     | RADIAL                 |
|                             | 50 100 250                                                                          |                        |
|                             | COMP. STRESS, PSI                                                                   |                        |
| OPERATING CONDITIONS        |                                                                                     | 50 COMP. STRESS, PSI   |
| • $P_C = 100$ PSIA          |                                                                                     |                        |
| • $\dot{M} = 0.022$ LB/SEC  |                                                                                     |                        |
| • 100 COLD START DUTY CYCLE |                                                                                     |                        |
| • UNIFORM PATTERN           |                                                                                     |                        |
| • $V_1 = 30$ (FT/SEC)       |                                                                                     |                        |

Figure 12. Catalyst retention test matrix

Key Hardware — The research tool was again the ARA, as previously described. For the catalyst effect experiments, the hardware was identical to the one utilized for the velocity/distribution tests. Similarly, no hardware innovations were required for the varying mechanical preload tests since only the magnitude of the compressive load, applied during the injector/chamber closure, differed.

However, slight modifications were necessary for the catalyst retention investigations. For those, each SUB embodied essentially the same external envelope, injection assembly, screens, and catalyst load (20-25 mesh Shell 405 ABSG) as its sister units expended in the velocity/distribution test series. It contained a 30 fps injector having twenty-one 10.3 mil orifices, which were uniformly dispersed over the injector face. In the dynamic load ARAs, flexible support members were incorporated to set and maintain the desired compressive load on the bed. As seen in Figure 13, an external, co-axial coil spring, housed around the WLB, continuously forced a "floating" retainer plate (having a pintle sliding through the lower bed aft-retainer and nozzle throat) against the upper catalyst bed via the movable spring force fitting. The use of an external spring reduced thermal effects minimizing spring rate changes. Three such units were manufactured with spring forces set to either 50, 100 or 250 psi bed loading, accomplished by adjusting the spring load fitting with the load nut. All components were 304L stainless steel except for the springs, which were fabricated from type 17-7 PH stainless steel wire (spring temper).

The compartmented SUBs were fitted with separator baffles appropriate to the cellulization scheme, as depicted in Figure 13. The axially-compartmented SUBs contained hemilenticular screens resting on steps in the shell wall, one having one screen with the convex side toward the injector and the other having two such screens with their concave sides facing the injector. The radially-compartmented SUB contained three radial baffles, 120° apart, with a common central edge, forming a "Y" in plan view. This trifoliate baffle assembly slipped into grooves in the shell wall and the injector face.

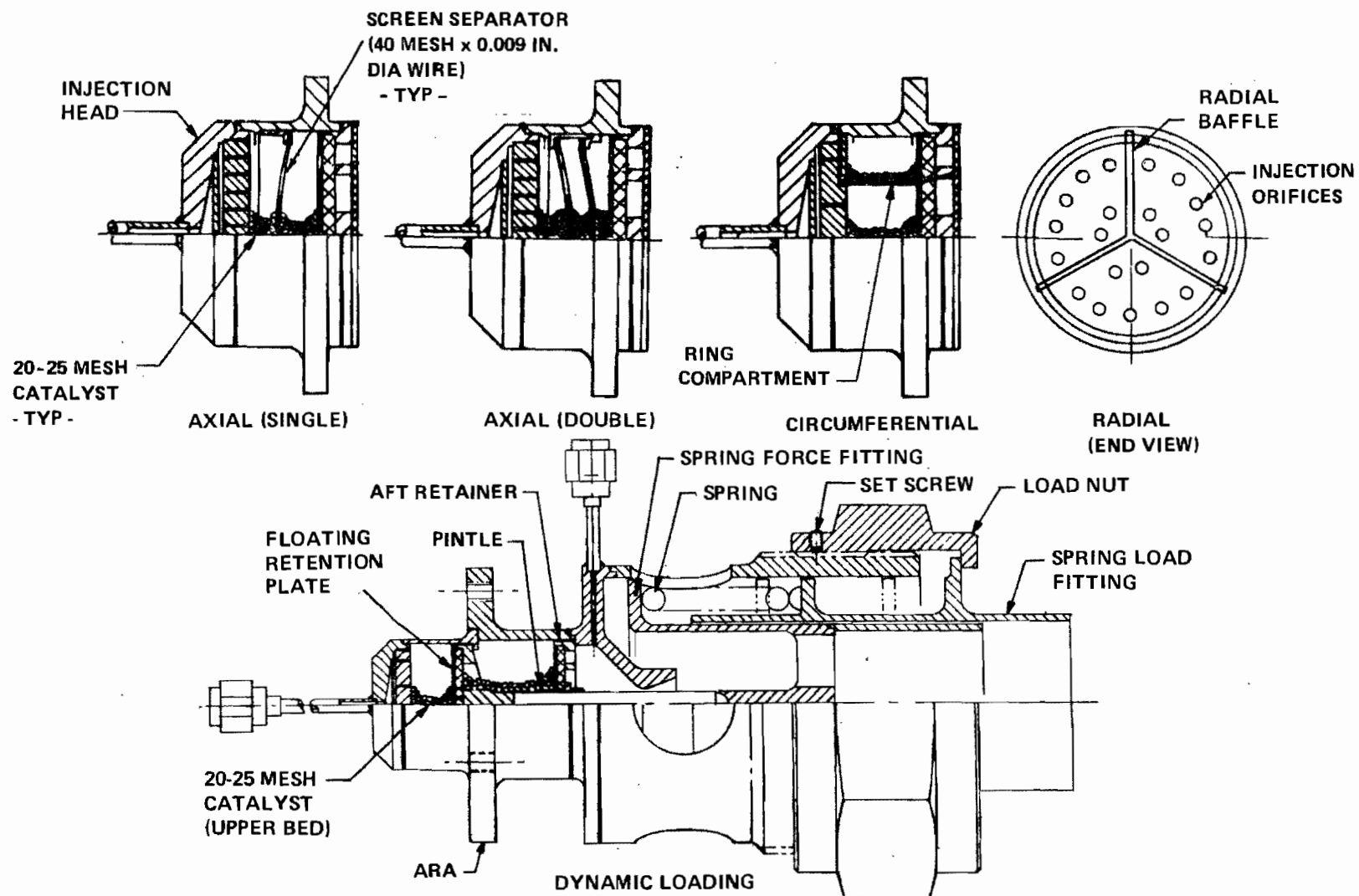


Figure 13. Catalyst retention hardware designs.

The circumferentially-compartmented SUB employed a thin concentric tubular baffle (extending the full length of the bed), similarly mounted. Some of the compartmented SUBs were modified to permit catalyst (20-25 mesh ABSG Shell 405) loading from the aft end. Separator screens were manufactured from Haynes 25 alloy (40 mesh x 0.009 inch diameter wire), while the various baffles were made from 304L stainless steel.

## 2.2 TEST PROCEDURE

Except for the attrition rate experiments, each ARA in a test block was subjected to a series of one-hundred cold starts.\* Eleven of these - the first and every multiple of 10 thereafter - were steady - state basepoint firings during which performance data were gathered; the remainder were isolated pulses as delineated in Table 1. As noted, both the steady-state and pulse durations varied with nominal ARA propellant flow, in order to maintain constant  $N_2H_4$  throughput.

For the attrition rate tests, each ARA was subjected to some fraction of a 41-sequence duty cycle, outlined in Table 2. Twenty-one of these - the first and every multiple of 10 thereafter - were combination pulse mode and steady-state firings; the remainder were sequences of isolated 200 msec pulses. Note that in the "A" sequence, the first train of pulses was delivered at 0.5 cps and the remaining four trains at 1.0 cps. Also the "ON" times in this sequence were 20, 35, 65, 100 and 200 msec - in that order - in the first such sequence and cyclically permuted in every A-sequence thereafter.

Since the test objectives mandated that the first firing of the test sequence be made on virgin units, prior calibration firings were not done. Consequently, the first basepoint firing also served as the "calibration firing" for the set of ARAs. Propellant tank pressure for the first pulse number was selected based upon best engineering judgement using calibration data for the cavitating venturis. The latter were sized to yield the requisite nominal flows, on the basis of prior water and nitrogen flow data for injectors and retainer/screen assemblies together with computed bed pressure drops. Based upon actual data from the first firing, propellant tank pressure was then adjusted to the best compromise value such that the propellant flow rates for all ARAs in the Test Block were as close as possible to their nominal values. Propellant flow rate and total propellant throughput were the primary control parameters and were maintained as constant as possible, irrespective of any variations induced in other performance parameters by so doing.

Since the majority of the time in these duty cycles was spent in cool down to the 50°F point, a "gang" firing arrangement was adopted. Up to sixteen ARAs were tested together with the system shown schematically in Figure 14. Note that flow to all the reactors was controlled by common cavitating venturis.\*\* Provision was made to test at two different flow rates within the same test block. Thermal control was provided by a recirculating water/glycol system. The thrusters were mounted to aluminum plates which were internally cooled by the water/glycol. Only natural conductive/radiative cool-down was permitted - purging and other forced cooling techniques were prohibited. Figures 15 and 16 present an installation drawing and a photograph of thrusters mounted to the cooling plates on the test stand. Before each firing, catalyst bed temperature was brought to  $50 \pm 10^\circ\text{F}$  as monitored on a probe thermocouple inserted in the lower bed. SUB skin temperature was also used as a check, but generally ran about 10°F lower than that of the catalyst bed. Propellant temperature was ambient (about 70°F).

---

\* Catalyst temperature  $50 \pm 10^\circ\text{F}$  at the inception of each pulse.

\*\* No cavitating venturis were used for the attrition-rate experiments.

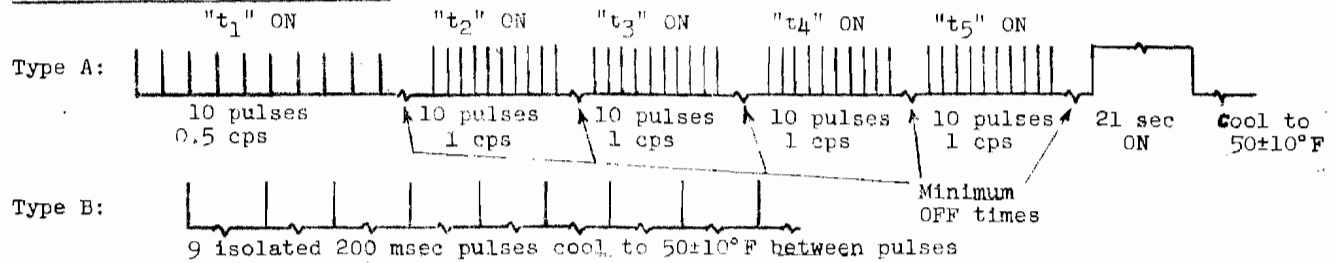
TABLE 1  
MULTIPLE COLD START DUTY CYCLE

| Pulse Number | Electrical Pulse Width (sec)<br>for steady-state propellant flow of: |              |              | Comments              |
|--------------|----------------------------------------------------------------------|--------------|--------------|-----------------------|
|              | 0.011 lb/sec                                                         | 0.022 lb/sec | 0.033 lb/sec |                       |
| 1            | 42.                                                                  | 21           | 14.          | Calibration/Basepoint |
| 2 thru 9     | 0.420                                                                | 0.210        | 0.140        |                       |
| 10           | 42.                                                                  | 21.          | 14.          | Basepoint             |
| 11 thru 19   | 0.420                                                                | 0.210        | 0.140        |                       |
| 20           | 42.                                                                  | 21.          | 14.          | Basepoint             |
| 21 thru 29   | 0.420                                                                | 0.210        | 0.140        |                       |
| 30           | 42.                                                                  | 21.          | 14.          | Basepoint             |
| 31 thru 39   | 0.420                                                                | 0.210        | 0.140        |                       |
| 40           | 42.                                                                  | 21.          | 14.          | Basepoint             |
| 41 thru 49   | 0.420                                                                | 0.210        | 0.140        |                       |
| 50           | 42.                                                                  | 21.          | 14.          | Basepoint             |
| 51 thru 59   | 0.420                                                                | 0.210        | 0.140        |                       |
| 60           | 42.                                                                  | 21.          | 14.          | Basepoint             |
| 61 thru 69   | 0.420                                                                | 0.210        | 0.140        |                       |
| 70           | 42.                                                                  | 21.          | 14.          | Basepoint             |
| 71 thru 79   | 0.420                                                                | 0.210        | 0.140        |                       |
| 80           | 42.                                                                  | 21.          | 14.          | Basepoint             |
| 81 thru 89   | 0.420                                                                | 0.210        | 0.140        |                       |
| 90           | 42.                                                                  | 21.          | 14.          | Basepoint             |
| 91 thru 99   | 0.420                                                                | 0.210        | 0.140        |                       |
| 100          | 42.                                                                  | 21.          | 14.          | Basepoint             |

- Ambient propellant temperature
- Hardware at  $50 \pm 10^{\circ}\text{F}$  at the inception of each pulse
- Cell equivalent altitude:  $\geq 100,000$  feet

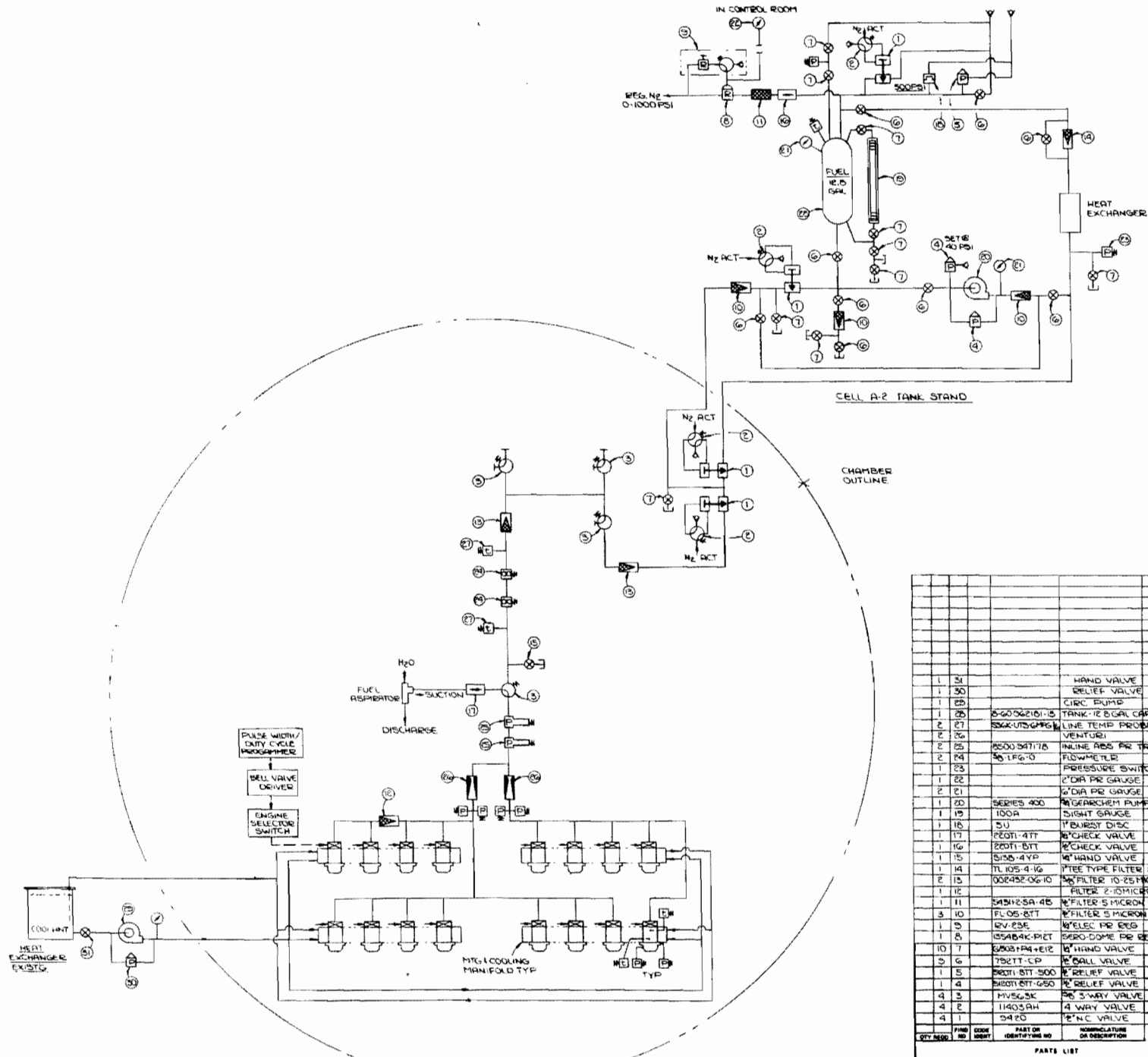
**TABLE 2**  
**PERFORMANCE-ATTRITION TEST DUTY CYCLE**

Definition of Sequence Types



| Sequence No. | Type | No. of Starts |       | ON Times      |               |               |               |               |                | Cumulative |             |                  |
|--------------|------|---------------|-------|---------------|---------------|---------------|---------------|---------------|----------------|------------|-------------|------------------|
|              |      | Cold          | Total | $t_1$<br>(ms) | $t_2$<br>(ms) | $t_3$<br>(ms) | $t_4$<br>(ms) | $t_5$<br>(ms) | Total<br>(sec) | Starts     | Cold Starts | On Time<br>(Sec) |
| 1            | A    | 1             | 51    | 20            | 35            | 65            | 100           | 200           | 25.2           | 51         | 1           | 25.2             |
| 2            | B*   | 8*            | 8*    |               |               |               |               |               | 1.6*           | 59         | 9           | 26.8             |
| 3            | A    | 1             | 51    | 35            | 65            | 100           | 200           | 20            | 25.2           | 110        | 10          | 51.0             |
| 4            | B    | 9             | 9     |               |               |               |               |               | 1.8            | 119        | 19          | 52.8             |
| 5            | A    | 1             | 51    | 65            | 100           | 200           | 20            | 35            | 25.2           | 170        | 20          | 78.0             |
| 6            | B    | 9             | 9     |               |               |               |               |               | 1.8            | 179        | 29          | 79.8             |
| 7            | A    | 1             | 51    | 100           | 200           | 20            | 35            | 65            | 25.2           | 230        | 30          | 105.0            |
| 8            | B    | 9             | 9     |               |               |               |               |               | 1.8            | 239        | 39          | 106.8            |
| 9            | A    | 1             | 51    | 200           | 20            | 35            | 65            | 100           | 25.2           | 290        | 40          | 132.0            |
| 10           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 299        | 49          | 133.8            |
| 11           | A    | 1             | 51    | 20            | 35            | 65            | 100           | 200           | 25.2           | 350        | 50          | 159.0            |
| 12           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 359        | 59          | 160.8            |
| 13           | A    | 1             | 51    | 35            | 65            | 100           | 200           | 20            | 25.2           | 410        | 60          | 186.0            |
| 14           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 419        | 69          | 187.8            |
| 15           | A    | 1             | 51    | 65            | 100           | 200           | 20            | 35            | 25.2           | 470        | 70          | 213.0            |
| 16           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 479        | 79          | 214.8            |
| 17           | A    | 1             | 51    | 100           | 200           | 20            | 35            | 65            | 25.2           | 530        | 80          | 240.0            |
| 18           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 539        | 89          | 241.8            |
| 19           | A    | 1             | 51    | 200           | 20            | 35            | 65            | 100           | 25.2           | 590        | 90          | 267.0            |
| 20           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 599        | 99          | 268.8            |
| 21           | A    | 1             | 51    | 20            | 35            | 65            | 100           | 200           | 25.2           | 650        | 100         | 294.0            |
| 22           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 659        | 109         | 295.8            |
| 23           | A    | 1             | 51    | 35            | 65            | 100           | 200           | 20            | 25.2           | 710        | 110         | 321.0            |
| 24           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 719        | 119         | 322.8            |
| 25           | A    | 1             | 51    | 65            | 100           | 200           | 20            | 35            | 25.2           | 770        | 120         | 348.0            |
| 26           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 779        | 129         | 349.8            |
| 27           | A    | 1             | 51    | 100           | 200           | 20            | 35            | 65            | 25.2           | 830        | 130         | 375.0            |
| 28           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 839        | 139         | 376.8            |
| 29           | A    | 1             | 51    | 200           | 20            | 35            | 65            | 100           | 25.2           | 890        | 140         | 402.0            |
| 30           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 899        | 149         | 403.8            |
| 31           | A    | 1             | 51    | 20            | 35            | 65            | 100           | 200           | 25.2           | 950        | 150         | 429.0            |
| 32           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 959        | 159         | 430.8            |
| 33           | A    | 1             | 51    | 35            | 65            | 100           | 200           | 20            | 25.2           | 1010       | 160         | 456.0            |
| 34           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 1019       | 169         | 457.8            |
| 35           | A    | 1             | 51    | 65            | 100           | 200           | 20            | 35            | 25.2           | 1070       | 170         | 483.0            |
| 36           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 1079       | 179         | 484.8            |
| 37           | A    | 1             | 51    | 100           | 200           | 20            | 35            | 65            | 25.2           | 1130       | 180         | 510.0            |
| 38           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 1139       | 189         | 511.8            |
| 39           | A    | 1             | 51    | 200           | 20            | 35            | 65            | 100           | 25.2           | 1190       | 190         | 537.0            |
| 40           | B    | 9             | 9     |               |               |               |               |               | 1.8            | 1199       | 199         | 538.8            |
| 41           | A    | 1             | 51    | 20            | 35            | 65            | 100           | 200           | 25.2           | 1250       | 200         | 564.0            |

\*Note First B sequence contains 8, instead of 9, pulses.



| QTY | NO. | CODE | PART OR IDENTIFYING NO. | NOMENCLATURE OR DESCRIPTION       | DRAWING OR SPECIFICATION | MATERIAL OR NOTE      | MA. TYP. | NO. | LO. | TO ADD | REV. | STATUS | DATE |
|-----|-----|------|-------------------------|-----------------------------------|--------------------------|-----------------------|----------|-----|-----|--------|------|--------|------|
| 1   | 31  |      |                         | HAND VALVE                        |                          |                       |          |     |     |        |      |        |      |
| 1   | 30  |      |                         | RELIEF VALVE                      |                          |                       |          |     |     |        |      |        |      |
| 1   | 29  |      |                         | CIRC. PUMP                        |                          |                       |          |     |     |        |      |        |      |
| 1   | 28  |      | 2-50526210-15           | TANK 12 GAL CAP ST STL 304 B.A.C. |                          |                       |          |     |     |        |      |        |      |
| 2   | 27  |      | 20KUT50610-1            | LINE TEMP. PROBE                  |                          | CONAX CORP.           |          |     |     |        |      |        |      |
| 2   | 26  |      |                         | VENTURI                           |                          |                       |          |     |     |        |      |        |      |
| 2   | 25  |      | 2500507175              | INLINE ABS PR. TRANSDUCER         |                          |                       |          |     |     |        |      |        |      |
| 2   | 24  |      | 25 LFQ-10               | FLOWMETER                         |                          | COX INSTR.            |          |     |     |        |      |        |      |
| 1   | 23  |      |                         | PRESSURE SWITCH                   |                          |                       |          |     |     |        |      |        |      |
| 1   | 22  |      |                         | 2" DIA. PR. GAUGE                 |                          |                       |          |     |     |        |      |        |      |
| 2   | 21  |      |                         | 2" DIA. PR. GAUGE                 |                          |                       |          |     |     |        |      |        |      |
| 1   | 20  |      | SERIES 400              | REARVIEW PUMP                     | ST STL                   | ECO ENGR.             |          |     |     |        |      |        |      |
| 1   | 19  |      | 100A                    | SIGHT GAUGE                       | ST STL                   | STRAHMAN              |          |     |     |        |      |        |      |
| 1   | 18  |      | 5U                      | 1" BURST DISC                     |                          |                       |          |     |     |        |      |        |      |
| 1   | 17  |      | 2201-4TT                | 1" CHECK VALVE                    | ST STL                   | BLACK SWALLS & BRYSON |          |     |     |        |      |        |      |
| 1   | 16  |      | 2201-5TT                | 1" CHECK VALVE                    | ST STL                   | JAMES POND CLARK      |          |     |     |        |      |        |      |
| 1   | 15  |      | 2155-4YP                | 1" HAND VALVE                     | ST STL                   | JAMES POND CLARK      |          |     |     |        |      |        |      |
| 1   | 14  |      | TL 105-4-16             | 1" FREE TYPE FILTER 2-5 MICRON    | NOM. ST STL              | REMCO                 |          |     |     |        |      |        |      |
| 2   | 13  |      | 002452-06-10            | 50" FILTER 10-25 MICRON NOM.      | FLUID DYNAMICS           |                       |          |     |     |        |      |        |      |
| 1   | 12  |      |                         | 1" FILTER 2-5 MICRON              | ST STL                   |                       |          |     |     |        |      |        |      |
| 1   | 11  |      | 24512-5A-4B             | 1" FILTER 5 MICRON NOM.           | ST STL                   | CUNO FILTERS          |          |     |     |        |      |        |      |
| 3   | 10  |      | FL 05-8TT               | 1" FILTER 5 MICRON NOM.           | ST STL                   | FLUID DYNAMICS        |          |     |     |        |      |        |      |
| 1   | 9   |      | 12V-23E                 | 1" ELEK. PR. REG.                 |                          | MARROTTA              |          |     |     |        |      |        |      |
| 1   | 8   |      | 155AB4K-PIET            | 2" DOME PR. REG.                  |                          | APCO                  |          |     |     |        |      |        |      |
| 10  | 7   |      | 2505144-1E2             | 1" HAND VALVE                     | ST STL                   | DAHL CO.              |          |     |     |        |      |        |      |
| 5   | 6   |      | 752TT-1P                | 1" BALL VALVE                     | ST STL                   | PETRO BALL VALVES     |          |     |     |        |      |        |      |
| 1   | 5   |      | 2201-8TT-500            | 1" RELIEF VALVE                   | ST STL                   | JAMES POND CLARK      |          |     |     |        |      |        |      |
| 1   | 4   |      | 2201-8TT-650            | 1" RELIEF VALVE                   | ST STL                   | JAMES POND CLARK      |          |     |     |        |      |        |      |
| 4   | 3   |      | 11556-5K                | 2" 5-WAY VALVE                    | ST STL                   | MARROTTA              |          |     |     |        |      |        |      |
| 4   | 2   |      | 11403-5A                | 4-WAY VALVE                       | BRASS                    | BARKSDALE             |          |     |     |        |      |        |      |
| 4   | 1   |      | 2420                    | 1" N.C. VALVE                     | ST STL                   | ANNIN                 |          |     |     |        |      |        |      |

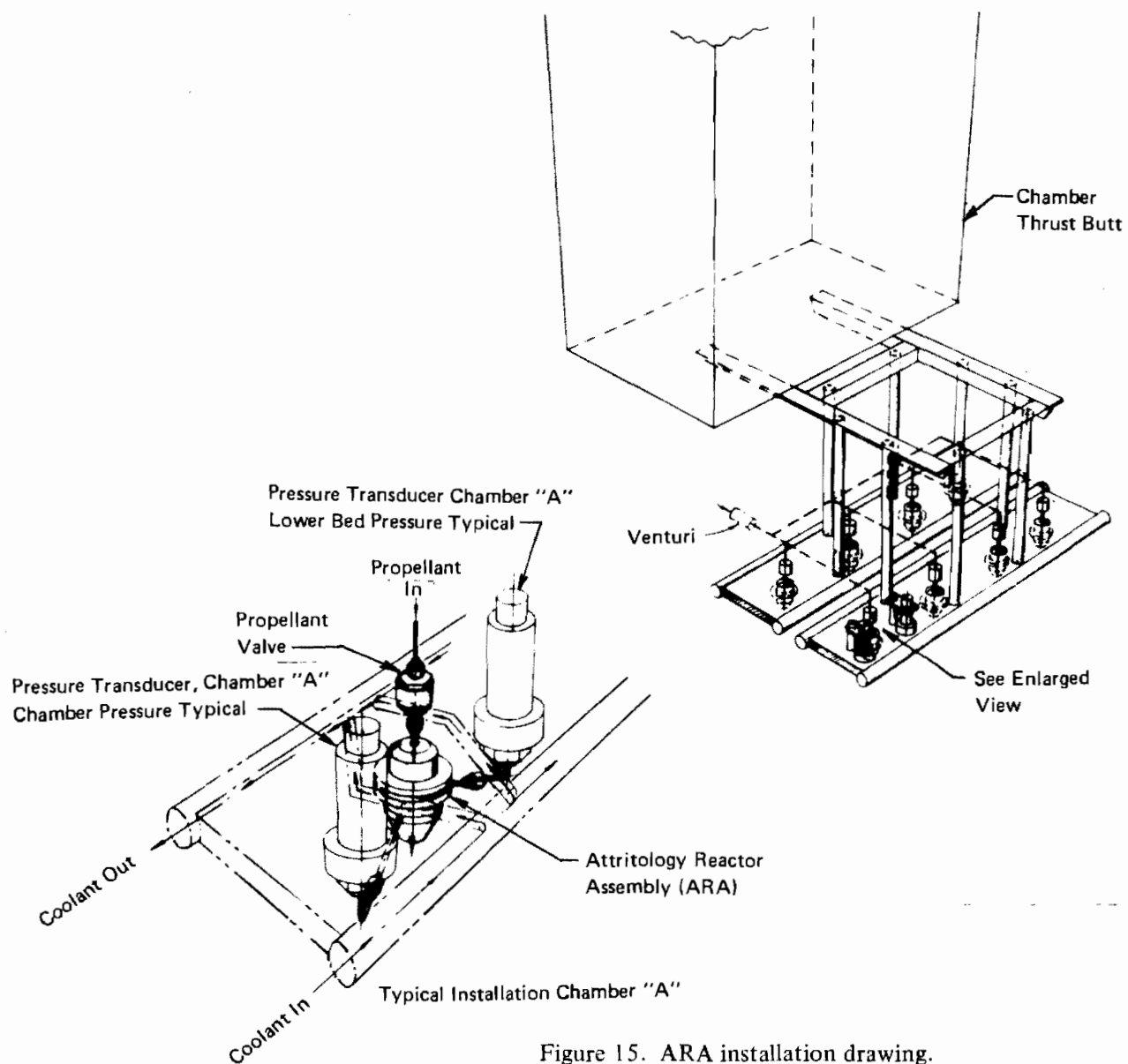


Figure 15. ARA installation drawing.

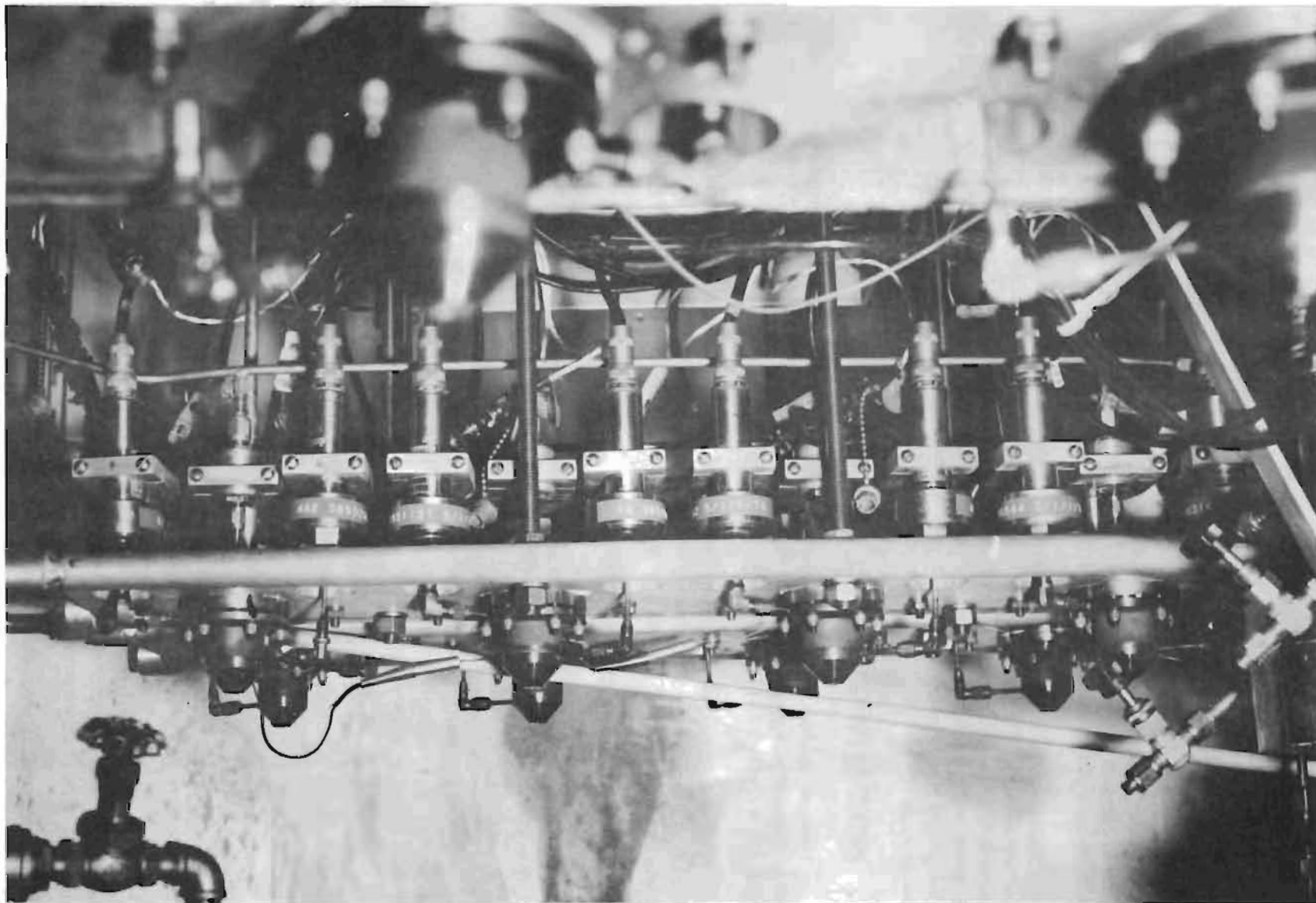


Figure 16. Thrusters mounted to cooling plates on test stand.

The thrusters in each test block were fired sequentially. Then all were cooled simultaneously. During this cool-down period, data from the previously completed pulse were reviewed. If it was an isolated pulse, only a cursory review of stripcharts was performed. If it was a basepoint firing, reduced steady-state data via a Beckman digital data system were evaluated. The key parameters in this evaluation were chamber pressure pulse shape, for a pulse; and characteristic velocity, upper catalyst bed pressure drop, chamber pressure and chamber pressure roughness, for a basepoint test. If one or more of the "incipient failure" criteria defined in Table 3 were met or exceeded by any thruster, the unit in question was assigned to "inactive" status for the remainder of the test sequence. It was then locked out (disarmed) on the engine selector/firing control panel.

The test cell was maintained under vacuum (equivalent altitude of  $> 100,000$  ft) during the entire test sequence. Measurements consisted primarily of chamber pressures, temperatures, and flow rates which were recorded on an oscillograph and Brush recorders for quick look purposes. All parameters were recorded on digital tape, and data from each basepoint firing were processed by a Beckman/IBM 360-44 data system. Only after the entire test sequence was completed, were all thrusters cooled to ambient temperature *in vacuo* prior to repressurizing the cell.

### 2.3 POST-TEST PROCEDURE

Upon completion of the test sequence, normal shutdown and securing procedures were employed. Extreme care was taken to avoid jarring or disturbing the catalyst beds. The units were not purged, nor were their respective propellant valves opened.

The test modules were carefully dismounted from the stand, and the valves and transducers removed. All thrusters were maintained in a vertical orientation at all times. The workhorse lower bed was removed and the SUB was then ready for the post-fire catalyst attrition evaluation.

### 2.4 ATTRITION MEASUREMENTS

Catalyst attrition was assessed both by measuring the reactor's performance during the test and by examining the catalyst bed after firing.

One basis for real-time measurement was that the attrition mechanism renders fines which tend to accumulate at the lower bed retainer and in the interstices between unfractured particles causing increased resistance to gas flow. This results in detectable (measurable) changes in bed pressure drop characteristics.

The void volume left by the displaced and fractured particles allows  $N_2H_4$  to accumulate and detonate. The resultant change in chamber pressure roughness was a second real-time attrition indicator. Gross attrition (with loss of catalyst) will also cause changes in  $C^*$  performance, which can be measured.

Each thruster's performance was, therefore, monitored during the entire duty cycle by recording individual pulses to look for gross pulse shape degradation and by monitoring steady-state firing data for changes in chamber pressure/roughness, reactor performance ( $C^*$ ), and for variation in upper catalyst bed pressure drop with accumulated pulses.

A typical computerized performance plot is shown in Figure 17 representing the variation of chamber pressure, characteristic velocity, roughness, and catalyst bed pressure loss with accumulated cold starts. The first two parameters were nondimensionalized by dividing each datum point by the first measurement. The roughness was defined as the half-amplitude roughness  $\left( \frac{P_{\max} - P_{\min}}{2P_{\text{mean}}} \right)$

TABLE 3  
INCIPIENT BED FAILURE CRITERIA

| Datum                                                                                                                                 | Failure Criterion                                                                                                                                                                                                                                                                                                                                                         |
|---------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Pulse Shape<br>$\frac{\delta P_c}{\bar{P}_c}$<br>$\left[ \frac{\partial (\delta P_c / \bar{P}_c)}{\partial n_s} \right]_{\text{avg}}$ | becomes grossly distorted and erratic<br>exceeds 50% in given basepoint firing<br>exceeds 30%/10 starts                                                                                                                                                                                                                                                                   |
| $\frac{\Delta P_{ub}}{\left[ \frac{\partial \Delta P_{ub}}{\partial n_s} \right]_{\text{avg}}}$                                       | exceeds 80 psid in given basepoint firing<br>exceeds 50 psid/10 starts                                                                                                                                                                                                                                                                                                    |
| $\frac{-\delta C^*}{C^*_o}$                                                                                                           | exceeds 25%                                                                                                                                                                                                                                                                                                                                                               |
| where:                                                                                                                                | $C^*_o$ = beginning-of-life characteristic velocity, ss (ft/sec).<br>$-\delta C^*$ = decrement in steady-state $C^*$ (ft/sec)<br>$\delta P_c$ = half-amplitude of steady state chamber pressure oscillation, mode value (psi)<br>$n_s$ = number of starts.<br>$\bar{P}_c$ = mean steady-state chamber pressure (psia)<br>$\Delta P_{ub}$ = upper bed pressure drop (psid) |

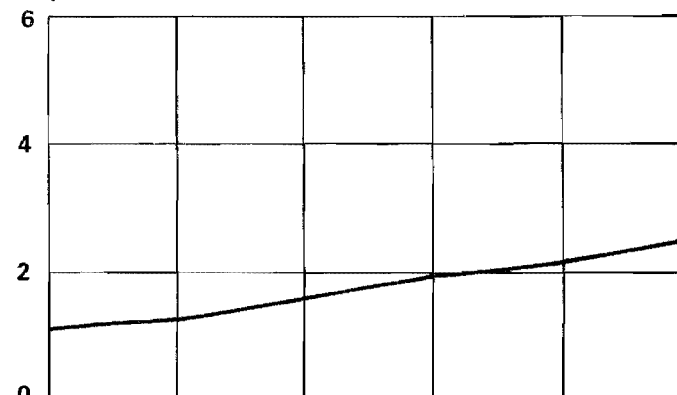
INJECTOR DATA:  
 SUB S/N AC01:  
 DASH NO. 27  
 ORIFICES 539  
 PATTERN U 100  
 MEAN DIAMETER 6.650 MIL  
 (FLOW/ORIFICE) AVERAGE 4.2770E-05 LB/SEC  
 G ORIFICE 1.2314 LBM/SQ. IN. - SEC

ARA ID NO. 3-B  
 MEAN FLOW RATE 0.0231 LBM/SEC  
 MEAN INJECTION VELOCITY 2.8632 FPS  
 MEAN PATTERN MASS FLUX 0.0381 LBM/SQ. IN. - SEC  
 CATALYST 20 - 25 (90% ATT)  
 CATALYST WEIGHT LOSS 11 %  
 AVERAGE PARTICLE DIA. CHANGE 0 %

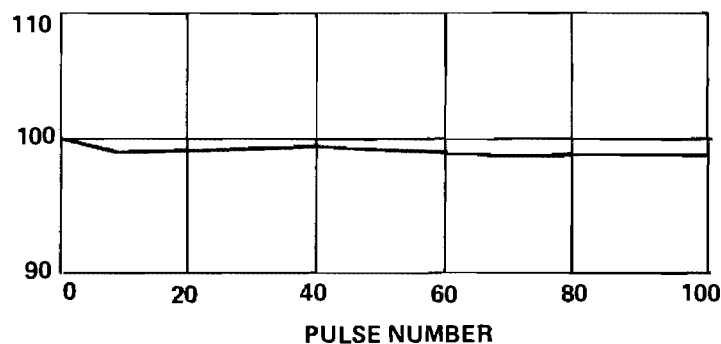
$P_c$  ROUGHNESS, %



PRESSURE LOSS  
INDEX, PLI



$P_c/(P_c)$  REF, %



$c^*/(c^*)$  REF, %

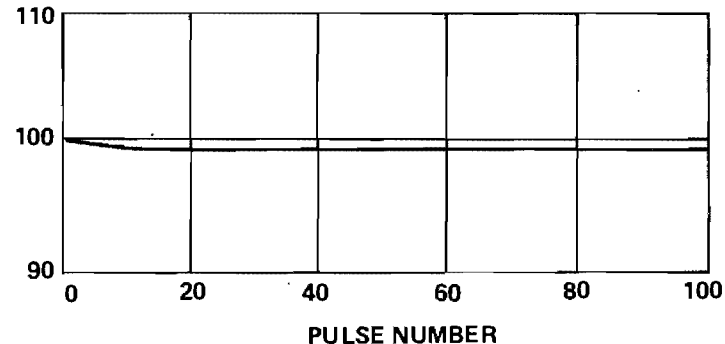


Figure 17. Typical ARA Performance Data Format

averaged over several successive time slices. The pressure loss index ( $I_{PL}$ ) is also a dimensionless parameter relating the flow rate, pressure drop and catalyst bed characteristics so that different catalyst beds under varying flow conditions can be properly compared.

It was derived by correlating a dimensionless pressure loss parameter ( $N_{PL}$ ) with the familiar Reynolds number ( $N_{RE}$ ). Basically, these parameters were derived from the conventional Hagen-Poiseuille equation, modified by assuming that a packed bed is merely a tube of a very complicated cross-section with a certain void fraction and wetted surface per unit volume. The following dimensionless parameters resulted, applicable to both liquid and low subsonic gas flow in the laminar and turbulent flow regime:

$$\text{Pressure Loss Modulus, } N_{PL} = \left( \frac{\epsilon}{1 - \epsilon} \right) \frac{g_c \rho \bar{D}_p^3 \Delta P}{\bar{\mu}^2 \cdot L}$$

$$\text{Modified Reynolds No., } N_{RE} = \frac{\bar{D}_p \cdot G_O}{\epsilon \bar{\mu}}$$

$\epsilon$  = void fraction

$g_c$  = gravitational constant

$\rho$  = average density of flowing medium

$\bar{D}_p$  = mean particle diameter

$\Delta P$  = pressure loss across catalyst bed

$\bar{\mu}$  = average viscosity of flowing medium

$L$  = bed length

$G_O$  = mass flux

These parameters were first calculated and plotted for each catalyst bed and flow condition. A parabolic correlation resulted yielding an empirical fit of the following expression:

$$N_{PL} = 27.45 (N_{RE})^{1.7}$$

We do not imply that these coefficients are necessarily the best fit since they were determined using only data of the first two test blocks (32 data points). But, for our purposes and range of data, this expression was a convenient and reasonable means of comparison.

In some test cases, the upper bed  $\Delta P$  measurements were inaccurate because they were obtained as the small difference between two much larger numbers (propellant inlet pressure less mid-bed chamber pressure). To overcome this, the SUB pressure drop was calculated for the first steady-state pulse (using the former expression) and the difference between this value and the one actually measured applied to every succeeding measurement. To be consistent, this normalization was then used for every test case regardless of ARA-type and flow rate. The flow/pressure data, in the

form  $PLI = N_{PL}/27.45 (N_{RE})^{1.7}$ , were then plotted against the number of cold starts for every test case; as typically shown in Figure 17.

The somewhat monotonic increase in roughness and bed pressure drop is evident in the figure, indicating the creation of fines and voids. You'll note that chamber pressure and characteristic velocity remains practically constant and will do so up to comparatively high levels of roughness (and catalyst weight loss). Of course, this is partially explained by the presence of the lower stabilizing catalyst bed. It appears that roughness will give the earlier alert to incipient bed failure.

The post-fire attrition assessment consisted of pre-/post differential mass determination and subsequent catalyst bed analysis. The former entailed first vacuum drying the SUB to remove fuel/moisture residue and then an accurate weighing of the entire unit (with the loaded catalyst). This weight was subsequently compared to the pre-test (loaded) SUB in order to determine the overall catalyst weight loss due to attrition. The bed was then vacuum-infiltrated with a pigmented, low-viscosity epoxy resin and cured (thereby "freezing" the particle configuration in space), appropriately sectioned radially and transversely, and polished. The sectioned SUB, typically shown in Figure 18, was then photographed, and the resulting 35mm positive transparency analyzed by a computerized Digital Image Processing technique. This basically, consisted of reconstructing the photograph in digital form as a binary map and analyzing it by generating the requisite particle size statistics to form a quantitative assessment of the degree of attrition. Figure 19 is a block diagram of the processing system. The 35mm positive transparency (the input) was first "read" into a 1024 x 1024 binary matrix by the flying spot scanner. The "picture" was then refined to account for touching particles, one or more particles that appear as a single particle because their boundaries are touching.

The refined matrix may be superimposed over the original picture on the video display to examine the quality of the binary representation. Figure 20 is an example of this. Note that a "box" has been written through the machine around the field to be studied. This field may be moved by the operator to include any area from the entire bed down to 1/274,674th of the frame. The smallest practical frame size was limited to about 5 x 5 particles, or in the order of 0.02 in.<sup>2</sup>.

The binary picture was stored on magnetic tape, the desired field was selected, and the data analyzed by the 2-dimensional (and 3-dimensional \* -optional) programs, which included statistical subroutines, generating the requisite particle size spectrum statistics, such as the equivalent diameter-, perimetral diameter-and shape factor distributions with their associated means and standard deviations. From the measured crosssectional area, A, and the perimeter, P, of the dissected particles an equivalent diameter,  $D_e$ , was calculated by equating those measurements to the area and perimeter of a circle. The resulting expression was  $4A/P$ . Similarly a perimetral diameter,  $D_p$  was established as  $P/\pi$ .

The shape factor was here defined as the ratio of the perimeter of the particle (P) to the perimeter of a perfect circle (P') having equal areas, or:

$$S = \frac{P}{P'}$$

For a circular particle, S is unity. S is greater than 1 for an irregular particle.

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\*The 3-D program can take the area and perimeter data from the 2-D data package and project these statistically into three-dimensional particle size distribution.

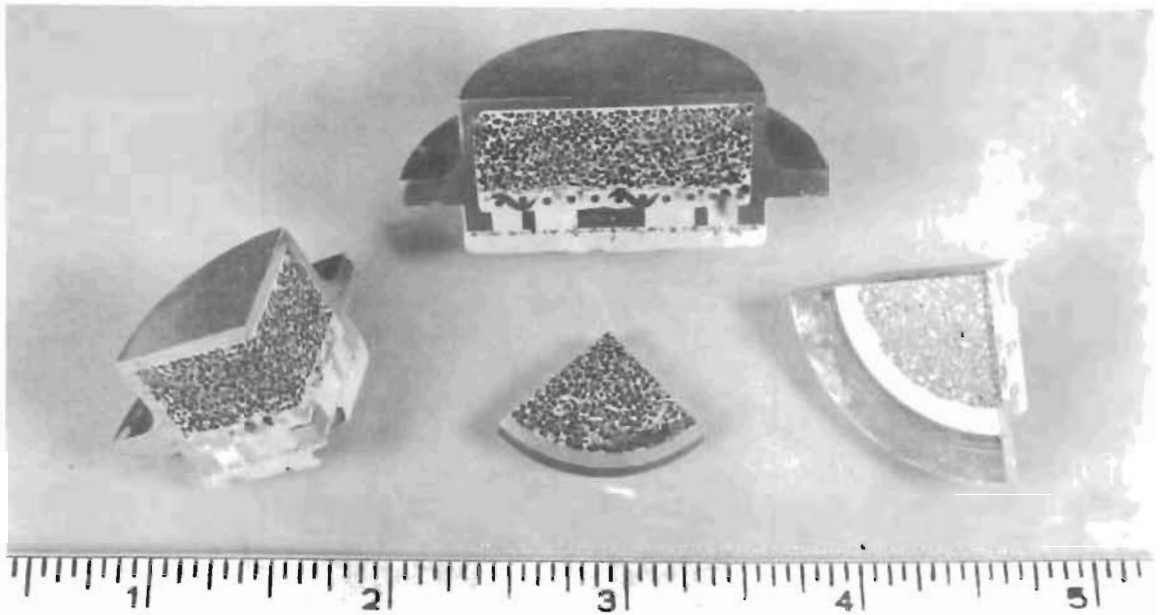


Figure 18. Photograph of typical sectioned SUB.

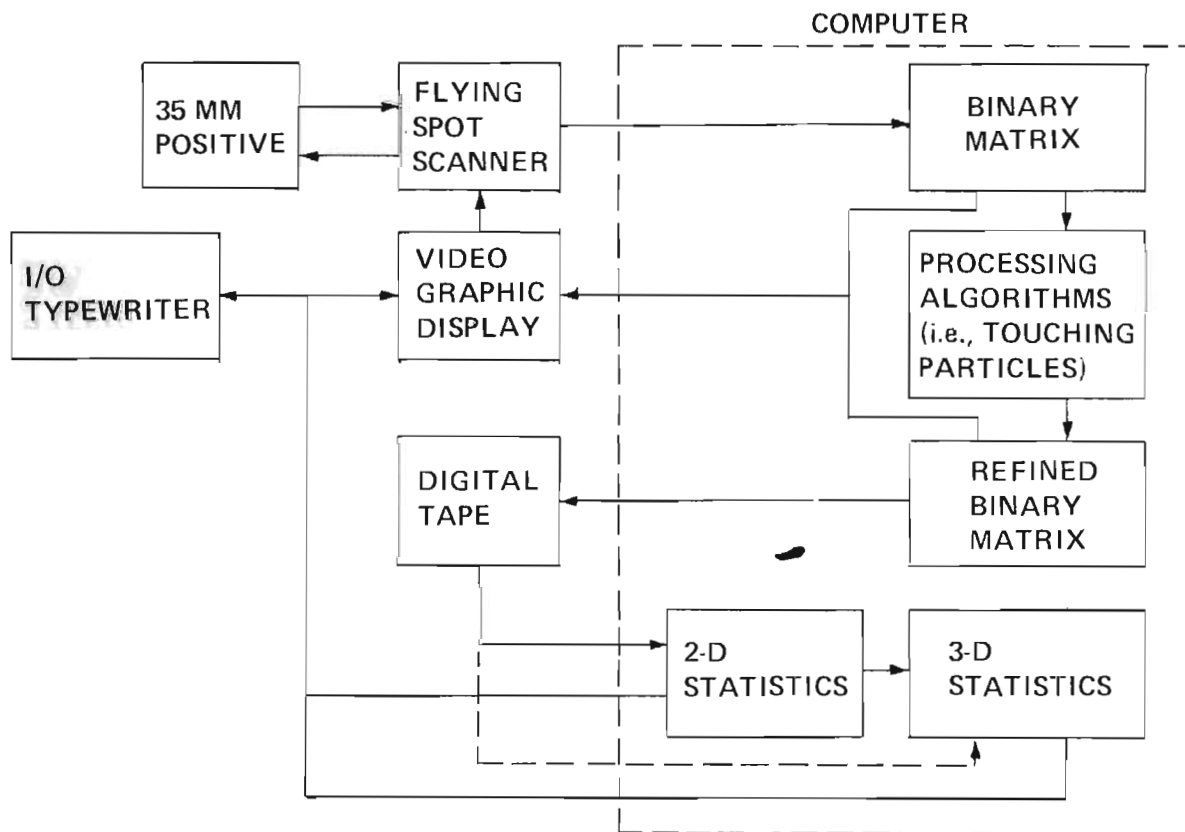


Figure 19. Block diagram of image processing system.

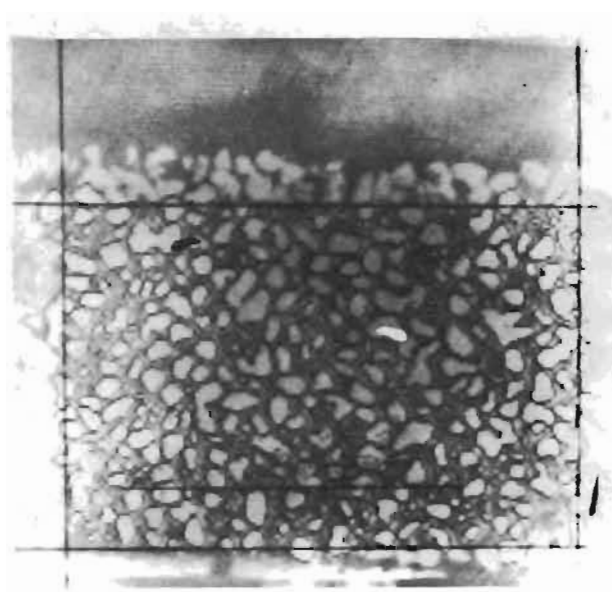


Figure 20. Refined binary picture over original image.

Comparing the particle size statistics between the unfired and fired catalyst bed thereby formed a quantitative basis to assess the degree of attrition - not only for the bed in toto, but also in specific subregions within.

For further details of the Digital Image Processing technique, Appendix A should be consulted.

## 2.5 DESIGN VARIABLE RESULTS

This section presents the results of the analysis of the data from the first five reactor design variable test blocks. The primary objective of this effort was to arrive at a mathematical description of the effects of the various engine design parameters on catalyst bed attrition as revealed by the more than seventy ARA's tested.

The attrition indicators which received the most attention were: catalyst bed (SUB) weight loss, changes in combustion roughness, and changes in bed pressure drop. The results from the Digital Image Processing system and pulse centroid variations were also studied.

The data set used is tabulated in Appendix B.

### 2.5.1 Summary and Conclusions

The results of the detailed statistical analysis are summarized in Table 4. Figure 21 presents, graphically, some of the trends. In reviewing the figure, bear in mind that it presents small groups of data outside of the overall context of the data set. The statistical treatment discussed below avoids this shortcoming and quantifies the significant trends. Regression equations were developed for the following relationships:

$\text{SUB Weight Loss} = f(\text{injection velocity, number of cold starts, and catalyst type})$

$\text{Average Rate of Pressure Loss Index Increase} = f(\text{injection velocity and catalyst type})$

$\text{Range of Pressure Loss Index} = f(\text{injection velocity and catalyst type})$

$\text{Range of Chamber Pressure Roughness} = f(\text{injection velocity, number of cold starts, number of injection orifices and catalyst type})$

The overall conclusions, for the parameter ranges investigated are:

- Injection velocity was influential, in particular when exceeding about 40 fps. Higher injection velocities caused increasingly poor performance.
- Roughness and catalyst weight loss increased significantly with number of cold starts and higher injection velocities.
- Of the five catalyst sizes and types tested, only 14-18 mesh ABSG revealed significantly lower attrition.
- The level of static preload on the catalyst bed had no consequential effect.
- Compartmentation was not beneficial.

**TABLE 4**  
**SUMMARY OF RESULTS**

| Independent<br>Parameter<br>Effect | On Dependent Parameter: |                    |                  |                    |
|------------------------------------|-------------------------|--------------------|------------------|--------------------|
|                                    | %Δ<br>Weight            | Mean PLI*<br>Slope | Range<br>of PLI* | Roughness<br>Range |
| Velocity                           | S                       |                    |                  |                    |
| Cat. Type:                         |                         |                    |                  |                    |
| -20-25                             | Baseline                |                    |                  |                    |
| -20-25/α                           |                         |                    | S ↑              |                    |
| -20-25/90%                         | S ↑                     |                    |                  |                    |
| -14-18                             | S ↓                     | S ↓                | S ↓              | S ↓                |
| -25-30                             |                         |                    |                  |                    |
| Pre-Load                           |                         |                    |                  |                    |
| Spring-Load                        | S ↓                     |                    |                  |                    |
| Distribution:                      |                         |                    |                  |                    |
| -U ≠ 100                           |                         |                    | S ↑ ?            |                    |
| -Clustering                        |                         |                    |                  |                    |
| -No. of Orifices                   |                         |                    |                  | S ↑                |
| -Penetrant                         |                         |                    |                  | S ↓                |
| -Rigimesh                          |                         |                    |                  |                    |
| -1 Spud                            |                         |                    | S ↓              |                    |
| -6 Spuds                           |                         |                    |                  | S ↑                |
| -Vortex                            |                         |                    |                  | S ↑                |
| -Hemisphere                        |                         |                    |                  | S ↑                |
| -Head Screen                       |                         |                    |                  |                    |
| Mass Flux                          |                         |                    |                  |                    |
| Compartments:                      |                         |                    |                  |                    |
| -1 Axial                           |                         |                    |                  |                    |
| -2 Axial                           |                         |                    |                  |                    |
| - Cylinder                         | S ↑                     |                    |                  |                    |
| - Radial                           |                         |                    |                  |                    |

\* PLI = Pressure Loss Index =  $f(\Delta P/\dot{\omega})$

S indicates statistically significant effect (arrows indicate trend where it is monotonic)

I indicates no significant effect

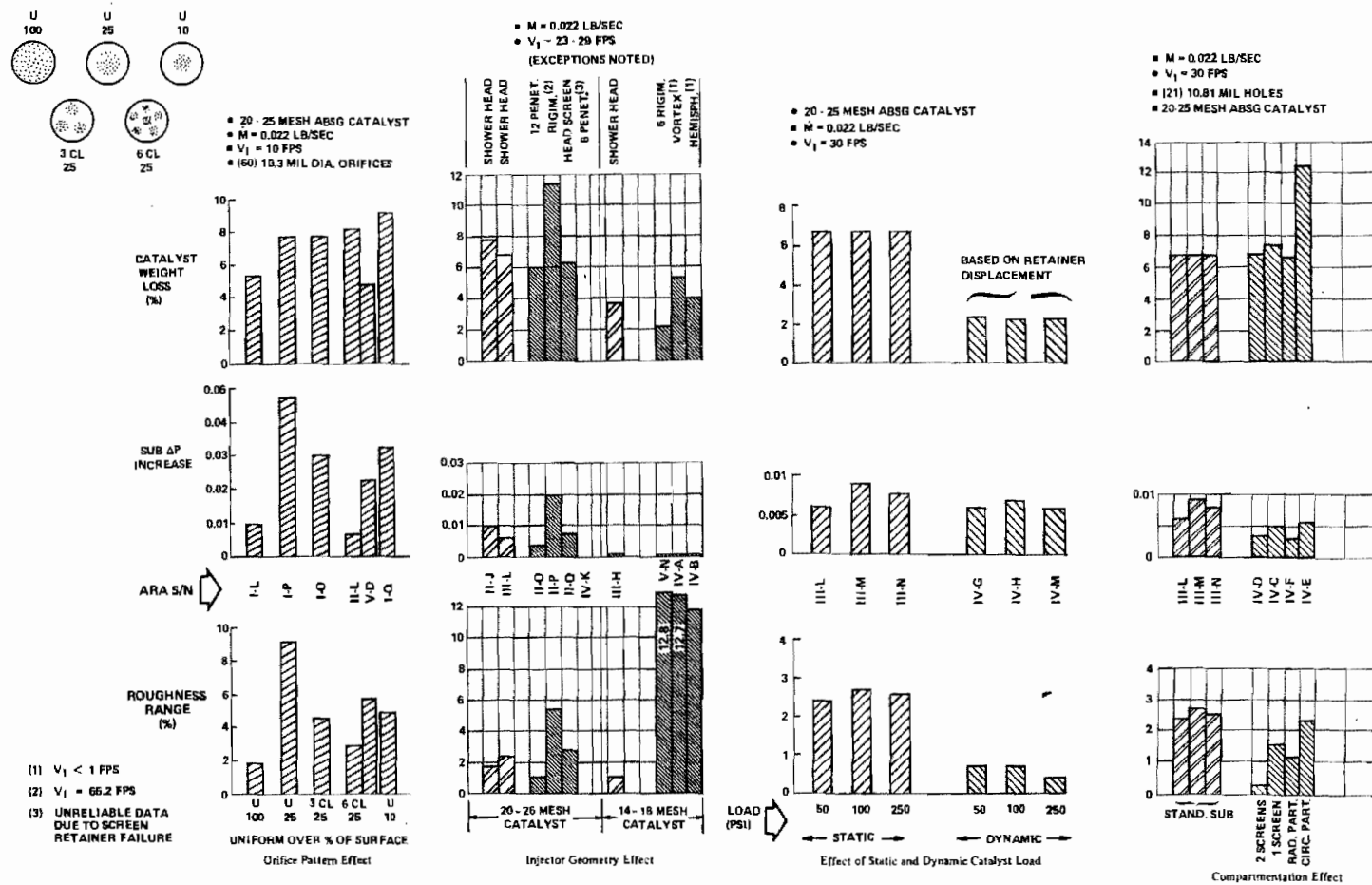


Figure 21. Design effects summary - non-statistical comparisons.

- Dynamic loading, regardless of magnitude, significantly reduced  $P_c$  roughness, bed pressure drop variation, and catalyst weight loss.
- No significant effects of propellant distribution in showerhead injectors was found.
- The non-showerhead units tested offered no consistent improvement.
- Pulse characteristic velocity ( $c^*$ ) was practically independent of injection velocity and number of cold starts. Impulse bit decreased slightly at higher velocities.
- Pulse centroid shifted in every case with increasing cold starts. This shift began earlier with high injection velocity, but the magnitude of the shift was greatest at intermediate (50 fps) injection velocity.
- Only a marginal shift in the particle diameter/frequency distribution of the catalyst bed resulted from firing exposure.

### 2.5.2 Discussion

The overall approach to the analysis was to withhold the data points which indicated significant effects due to variables that could not be easily characterized numerically, such as catalyst type, spring load and compartmentation. The remaining data were then modeled using a multiple linear regression computer program. The data withheld earlier were then checked against the regression model for significant effects. The model was modified to include the additional effects that were found.

The multiple regression technique evaluates the coefficients ( $C$ 's) of an equation of the form:  $Y = C_0 + C_1 X_1 + C_2 X_2 \dots$  and provides indications of the statistical significance of the overall equation and of the individual regression coefficients ( $C_1, C_2, \dots$ ). The  $X_i$ 's in turn, may be functions of the independent variables (e.g.,  $X_1 = w^2$ ,  $X_2 = w \cdot z$ ). The result of multiple regression is the best unbiased fit to the inputted data set using the functional form selected by the analyst.

The regression effort consisted of selecting the significant variables and their functional relationships, by educated trial-and-error, to result in a statistically significant and physically satisfying equation.

#### 2.5.2.1 Catalyst Weight Loss

The actual loss of catalyst from the bed during a firing duty cycle is a clear indication that a life-limiting process is taking place even though mission critical performance parameters may not be materially affected until gross weight loss attrition has occurred. The SUB's were weighed before and after being subjected to fire testing. Their change in weight, expressed as a percentage of the initial catalyst weight ( $\% \Delta W$ ), was taken as an attrition variable.

The obvious initial step in the selection of a data set for regression analysis was to confine attention to 20-25 mesh catalyst since it was used in most of the SUB's tested. This choice was modified by the results of an analysis of variance performed on the results from a set of 15 SUB's. This technique and data set allowed the significance of the effects of the 5 different catalyst sizes/treatments upon the dependent variable to be determined. Catalyst sizes/treatments which did not differ significantly in effect from 20-25 mesh were later included in the regression data set.

It was also clear from inspection that: 1) the three levels of bed preload tested had no measurable effect on weight loss; 2) the spring loaded units were significantly different from the units, but not from each other; and 3) that the flight type penetrating screen (Hamilton Standard), Rigimesh Spud (Rocket Research), and discrete stream open head space (TRW) type units fit into the general family.

The 45-unit regression data set then, excluded data from SUB's which had catalyst effects significantly different from those of 20-25 mesh, spring loaded beds, compartmented beds, or incomplete data. The first restriction eliminated the "improved injectors" of test block V since they all utilized 14-18 mesh catalyst. The three 20-25 mesh  $\alpha$  Phase units were added to the basic data set near the end of the effort, since their weight loss characteristics were statistically the same as the plain 20-25 mesh, according to the analysis of variance.

#### Regression Results

Over 100 regressions were performed on the data set and selected sub-sets. Much of this effort was expended in attempts to arrive at an equation which included a description of injection geometry as a significant term. Table 5 lists the 22 parameters that were tried as  $X_i$ 's in the regressions. Injection velocity and the number of cold starts were, however, found to be the only statistically significant factors influencing  $\% \Delta W$ . Figure 22 presents the regression relationship and the data points used in the fit. The  $T^{(1)}$  values for the regression coefficients are:

| Coefficient Of     | t                                                 |
|--------------------|---------------------------------------------------|
| $V \cdot \#CS$     | 2.61 $> t (\alpha = 0.01, \nu = 47) \approx 2.41$ |
| $V^2 \cdot \#CS^2$ | 4.01                                              |
| $\#CS^2$           | 6.15                                              |

The greatest error made by this fit occurs at ( $V = 38$  ft/sec,  $\#CS = 200$ ) where the regression value is 13% lower than the test value.

The regression results and the block V data points are plotted against number of cold starts in Figure 23. This presentation shows the nonlinear effect of increasing cold starts and the strong interaction with injection velocity. Figure 24 shows the higher velocity SUB's sectioned and potted after their fire test exposure.

#### Catalyst Effects

Fifteen of the tests formed a data set in which injection velocity ( $V$ ) was tested at three levels with each of the five types of catalyst and with all other factors held constant. A two-way analysis of variance was done on this set to identify catalyst and velocity effects. At the 95% confidence level, no overall velocity effects were found (the data set range, 3 to 24 ft/sec., is in a very shallow part of the  $\% \Delta W$  versus  $V$  curve), but overall catalyst effects did exist. The analysis of variance technique then, allowed the detail trends indicated in Table 4 to be found: 20-25 mesh, 20-25 mesh  $\alpha$  phase, and 25-30 mesh are about the same; 90% attrited 20-25 mesh catalyst had more, and 14-18 mesh catalyst less, weight loss than plain 20-25 mesh catalyst.

(1) "Student-t" statistic which measures the significance of a relationship between the given independent and dependent variable ( $\alpha$  is level of significance,  $\nu$  is degrees of freedom).

TABLE 5  
%ΔW TRIAL INDEPENDENT VARIABLE FORMS

| Symbol                                                                                         | Definition                                                                                                               |
|------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------|
| V, V <sup>2</sup> , LOG V                                                                      | - mean propellant injection velocity                                                                                     |
| #OR, LOG (#OR)                                                                                 | - number of injection orifices                                                                                           |
| %A                                                                                             | - proportion of injector face containing injection orifices.                                                             |
| φ, φ <sup>2</sup>                                                                              | - mass flux distribution parameter<br>$\phi = \frac{1}{r_w \dot{W}_T} \int_0^{r_w} \int_0^{2\pi} \dot{M} r^2 dr d\theta$ |
| #CS, #CS <sup>2</sup> ,<br>LOG (#CS)                                                           | - number of accumulated cold starts                                                                                      |
| V%A<br>V#CS<br>V <sup>2</sup> #CS<br>(V#CS) <sup>2</sup><br>Vφ<br>#CS/V<br>#CS <sup>2</sup> /V | Interactions                                                                                                             |
| $\dot{w}$                                                                                      | - test nominal flow rate (αG)                                                                                            |
| $\dot{w}/v, (\dot{w}/v)^2$                                                                     | - proportional to actual injection area                                                                                  |
| D <sub>o</sub>                                                                                 | - diameter of orifices                                                                                                   |

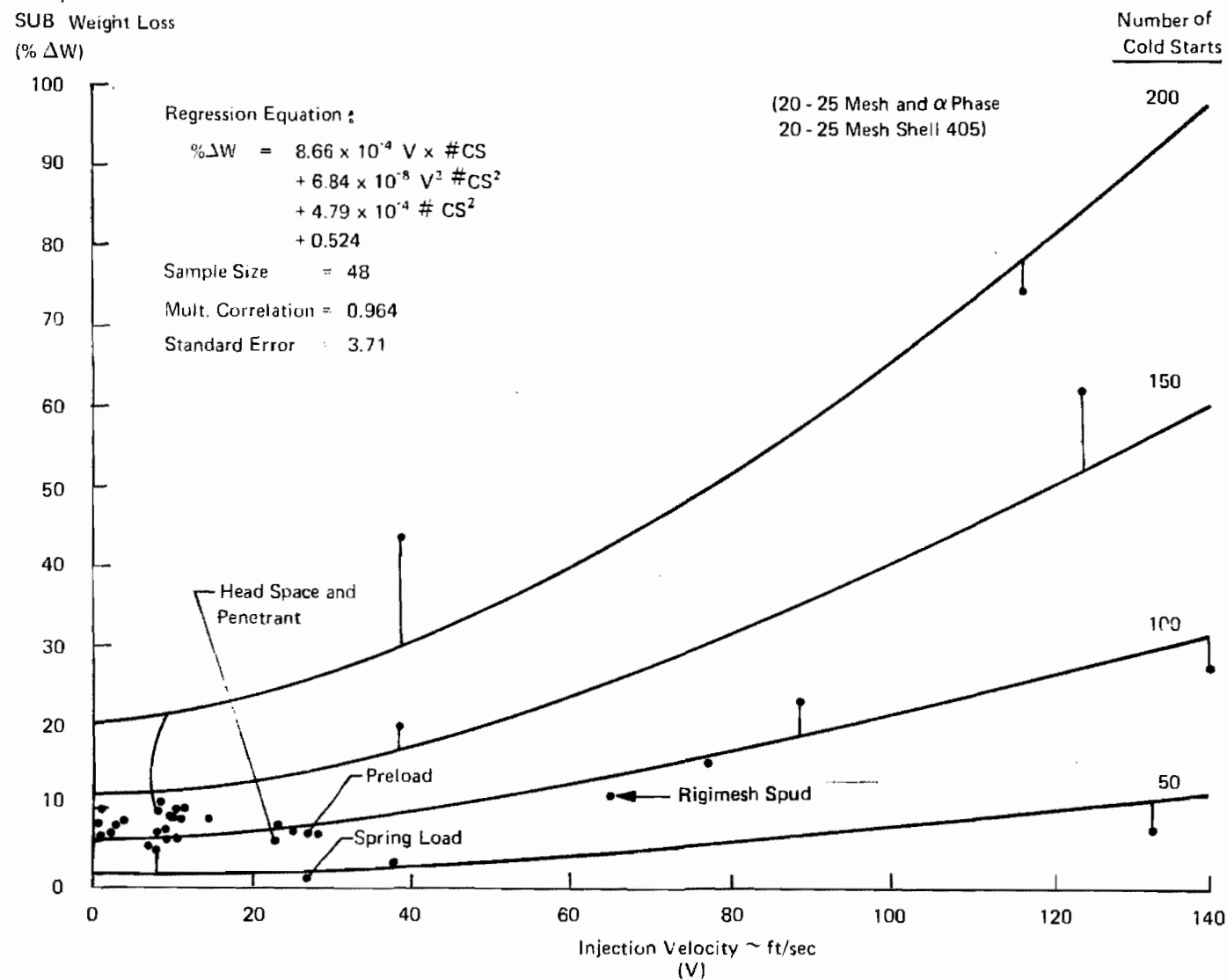


Figure 22. SUB weight loss versus injection velocity and cold starts.

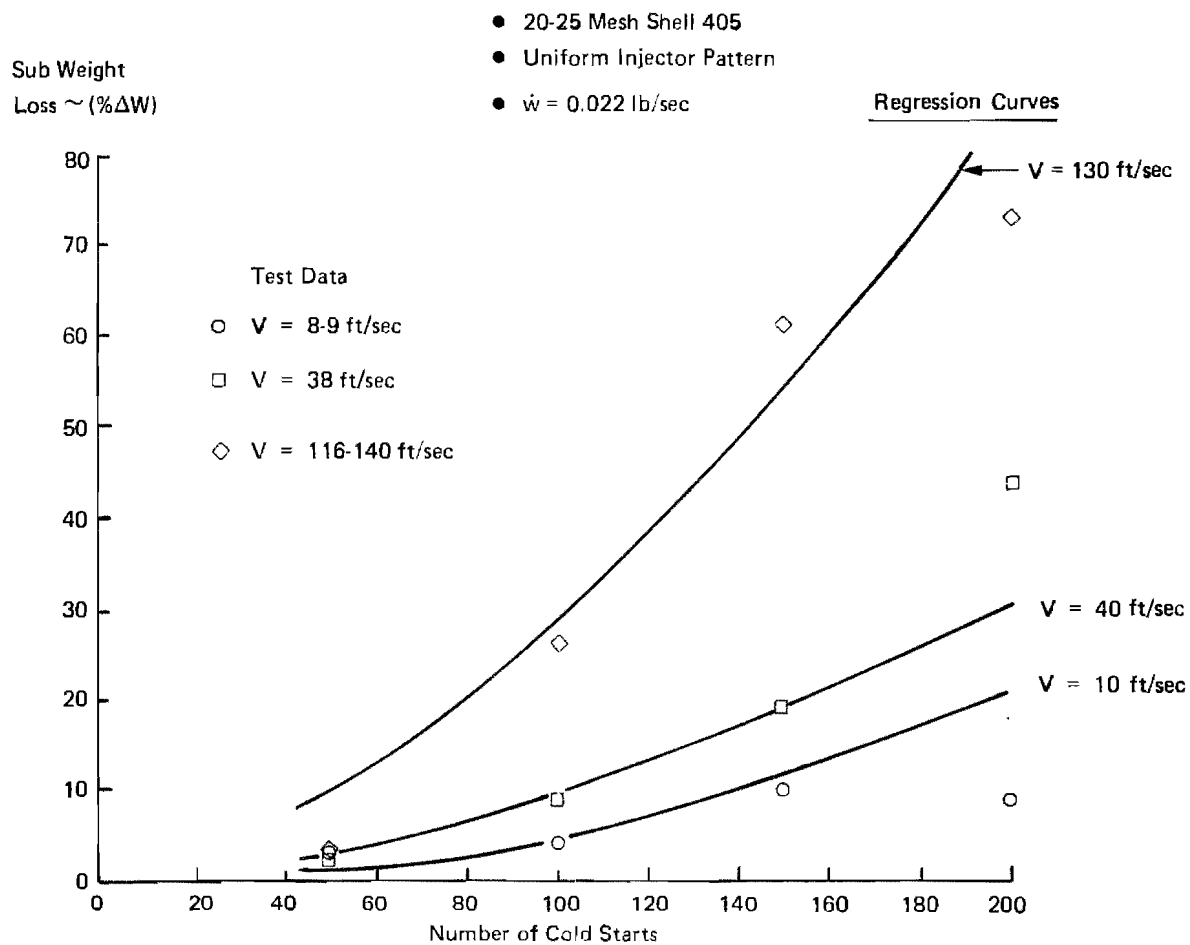
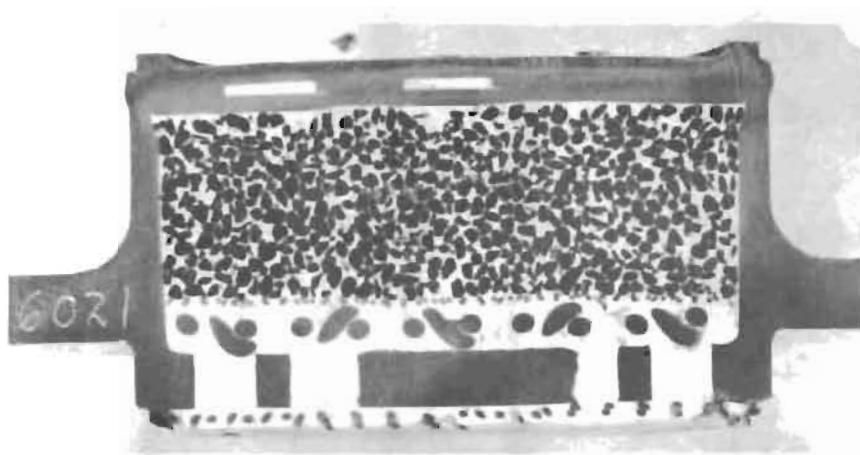
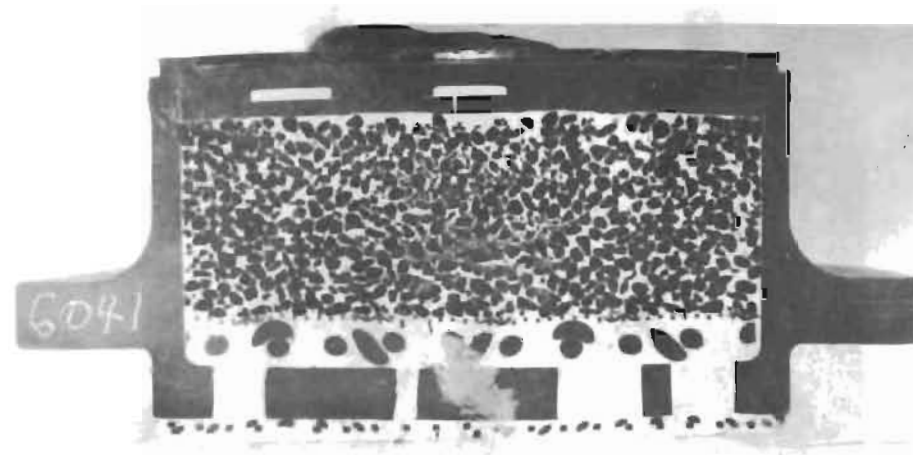


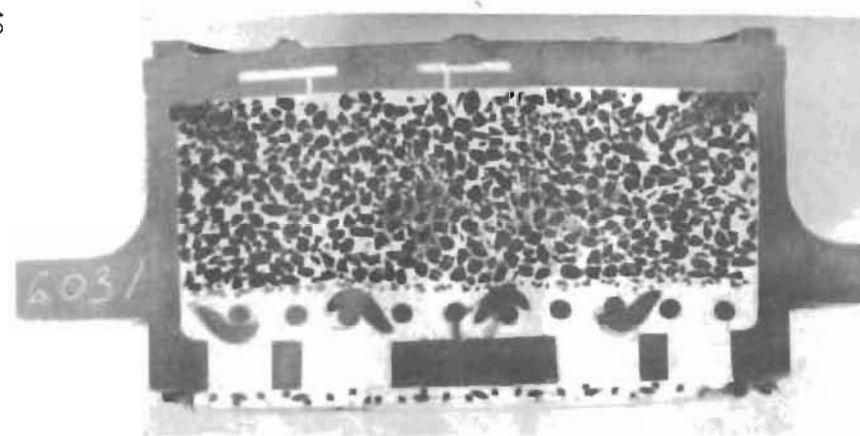
Figure 23. SUB weight loss versus cold starts and injection velocity.



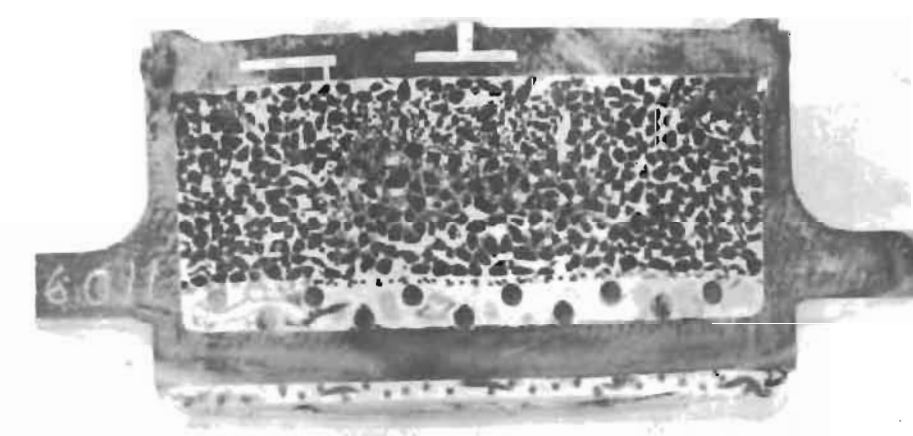
50 Cold Starts  
( $-\Delta W = 4.55\%$ )



100 Cold Starts  
( $-\Delta W = 4.68\%$ )

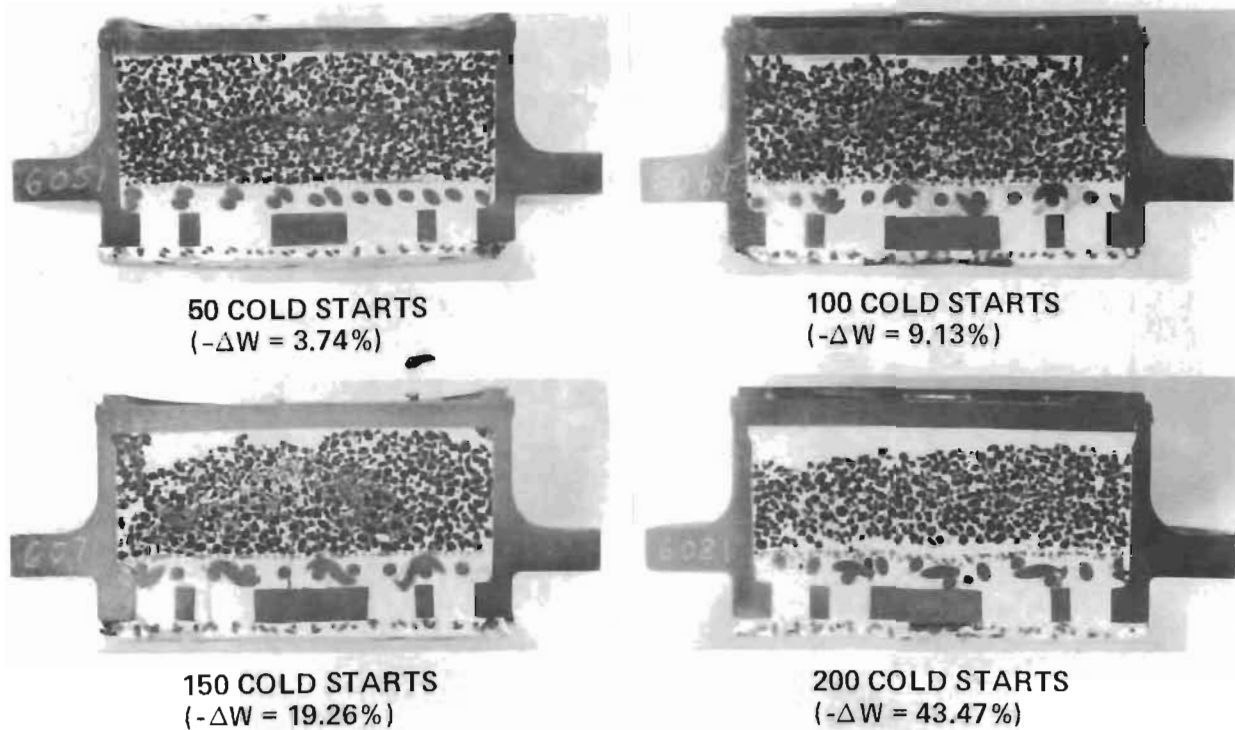


150 Cold Starts  
( $-\Delta W = 10.35\%$ )

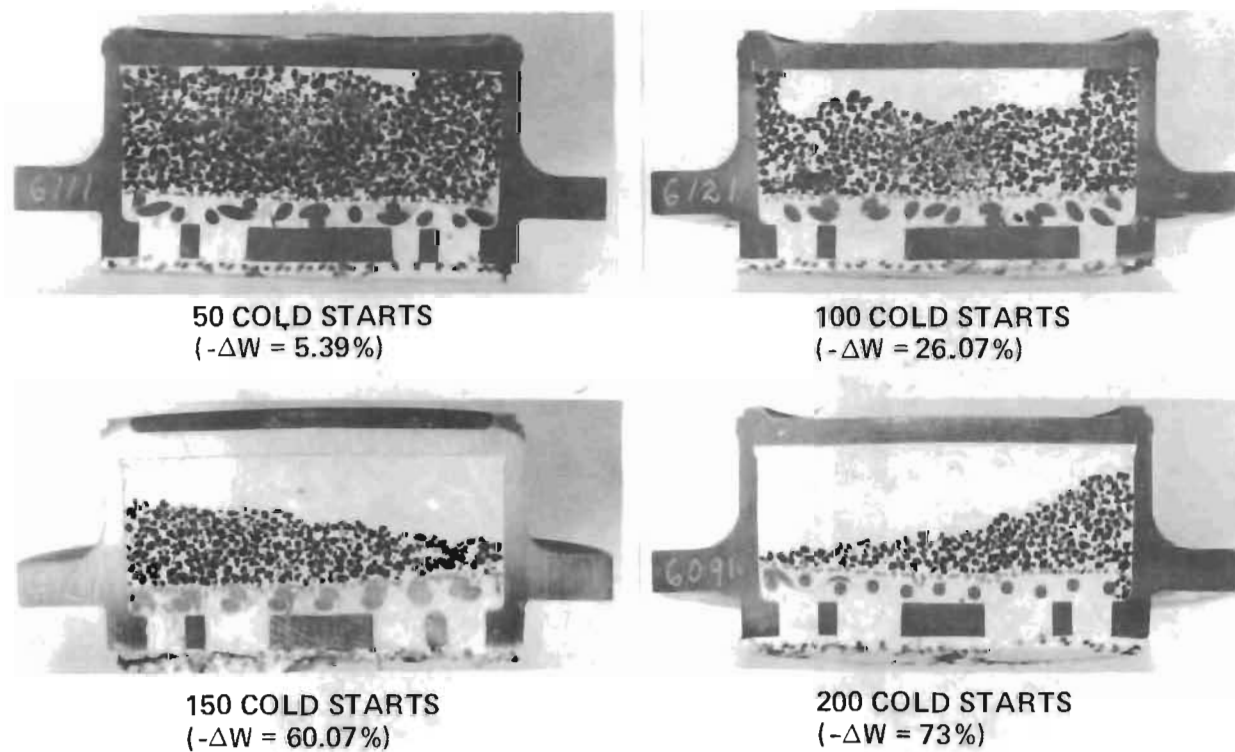


200 Cold Starts  
( $-\Delta W = 8.58\%$ )

Figure 24. Post test catalyst bed cross sections (1 of 2).



INJECTION VELOCITY = 38 FPS



INJECTION VELOCITY >100 FPS

Figure 24. Post test catalyst bed cross sections (2 of 2).

Based on these results, the intercept of the regression fit was modified to account for the significant catalyst effects. The resultant expression is:

$$\% \Delta W = 8.66 \cdot 10^{-4} V \cdot \#CS + 6.84 \cdot 10^{-8} V^2 \cdot \#CS^2 + 4.79 \cdot 10^{-4} \#CS^2 + C$$

$$C = 0.524 \text{ for 20-25 plain or 20-25 } \alpha \text{ phase}$$

$$= -2.307 \text{ for 14-18 mesh}$$

$$= 3.993 \text{ for 20-25 90\% attrited}$$

$$= -0.007 \text{ for 25-30 mesh}$$

These curves and the 15 data points are plotted in Figure 25. Note that the 90% attrited catalyst points appear to have a negative slope. Although this is not a significant trend over the velocity range tested, it is possible that the 90% attrited catalyst could show less weight loss than others at injection velocities greater than about 30 ft/sec. If so, this would be an example of an important interaction.

#### Injector Design Influences

As was stated above, injection velocity was the only design parameter which was found to correlate with  $\% \Delta W$  in the basic data set (which included all of the 20-25 mesh units except for those with spring loaded and compartmented beds). Non-velocity design variations were tested in both 20-25 mesh and 14-18 mesh catalyst. The results are discussed separately for each catalyst type.

#### 20-25 Mesh Results

The 95% upper and lower prediction intervals for the basic regression fit were computed based on the deviations from the regression for  $V \leq 30$ . Figure 26 is a plot of the resulting band with the non-U-100 injector results indicated. Points falling within the 95% prediction interval band may be considered to be part of the family of results described by the regression line. Only two designs appear to differ significantly: the spring loaded units, which had lower weight loss, and the cylindrically compartmented unit, which had greater weight loss. The Rigimesh spud type injector falls beyond the velocity scale of Figure 26 and is shown on Figure 22. It is also part of the family.

#### 14-18 Mesh Results

The very low-velocity injectors were tested with 14-18 mesh catalyst. Their results are compared with the basic regression result (modified for 14-18 mesh) in Figure 27. The 95% prediction tolerance used above is also shown for reference. These units do not differ significantly in  $\% \Delta W$  from the basic, U-100, units when injection velocity is taken into account. In fact, it is unlikely that the 14-18 mesh results differ from each other at all.

Although the prediction interval about the regression line includes zero, the regression values are statistically different from zero: the 95% confidence interval, which gives the likely limits for the true mean value of  $\% \Delta W$  as predicted by the regression, does not include zero.

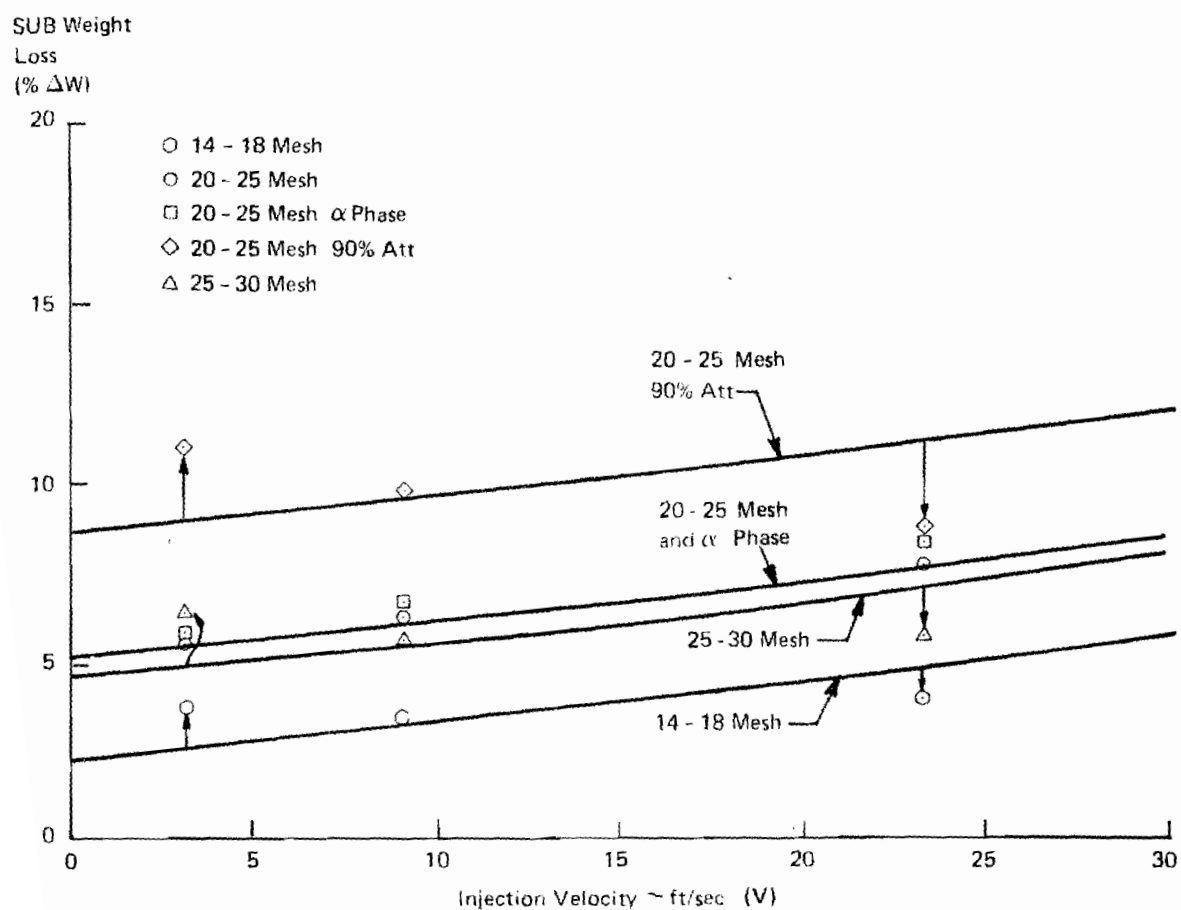


Figure 25. Catalyst size/treatment effect on weight loss (100 cold starts).

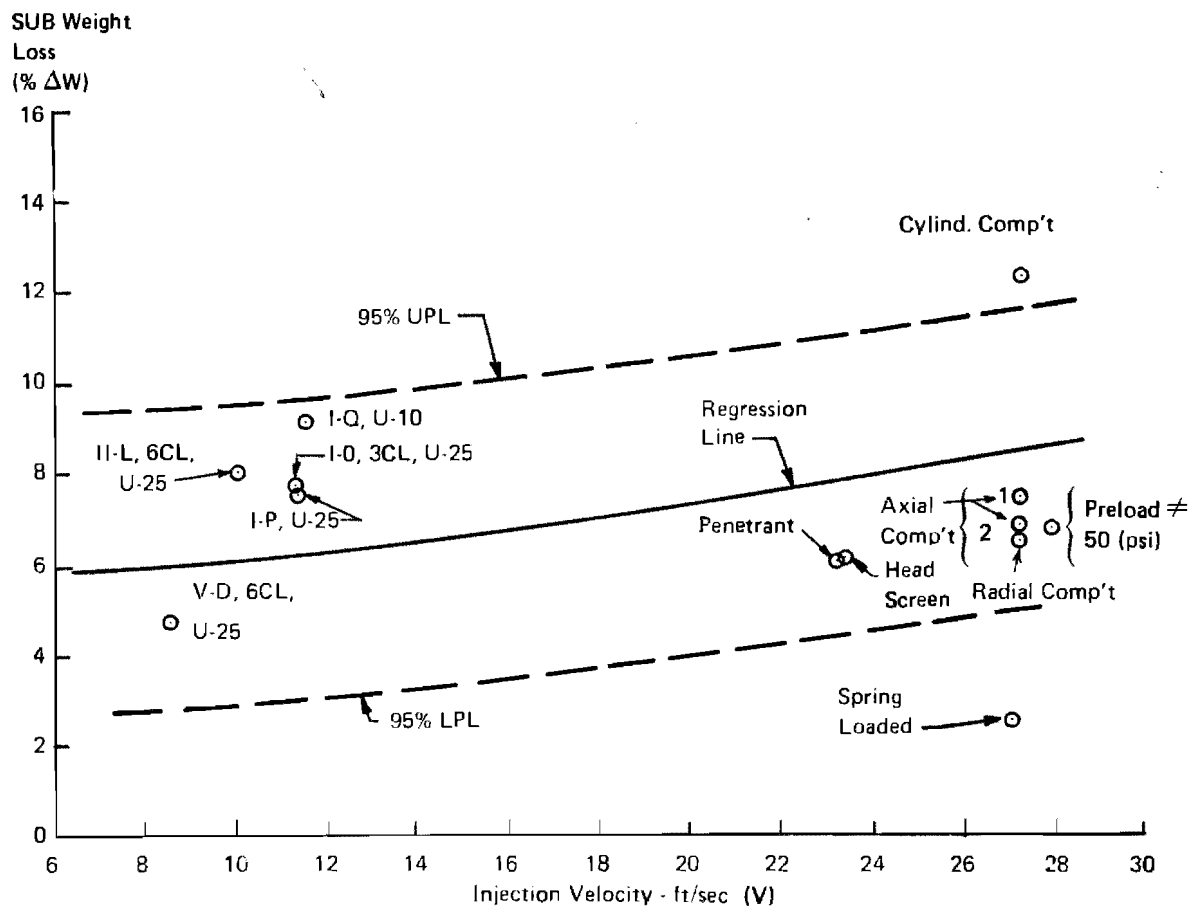


Figure 26. Injector design influences on SUB weight loss (20-25 mesh only, 100 cold starts).

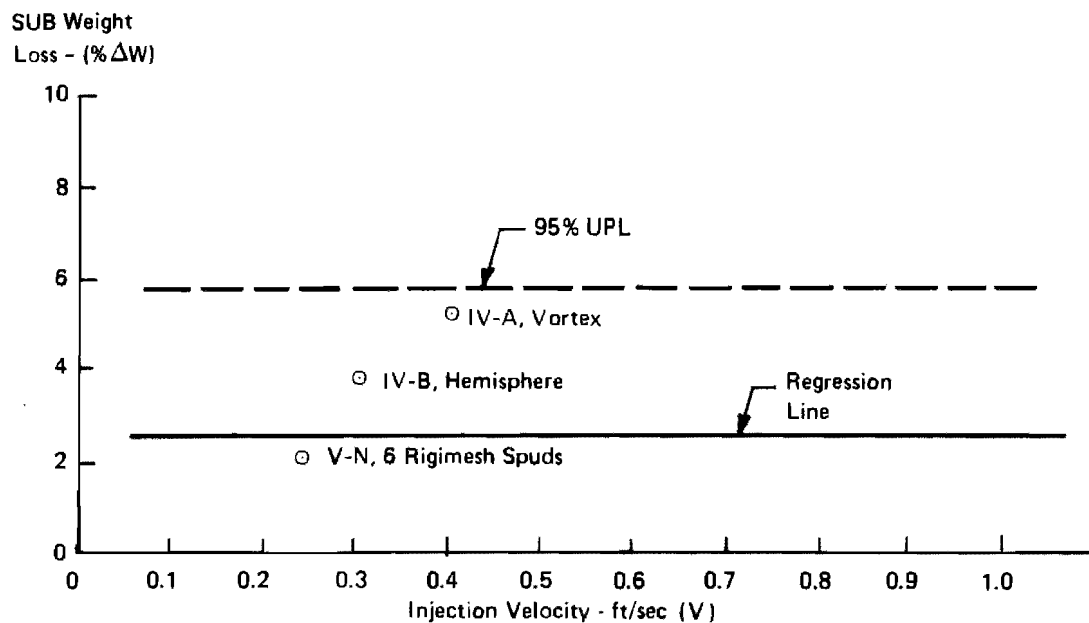


Figure 27. Injector design influences on SUB weight loss (14-18 mesh only, 100 cold starts).

### 2.5.2.2 SUB Pressure Loss Index

The SUB pressure loss index (PLI), defined in paragraph 2.4, is simply the catalyst bed pressure drop normalized for catalyst type and flow conditions. This parameter was evaluated during the steady state firings of the duty cycles for each SUB and plotted against the cumulative cold starts (#CS). (See Figure 17 for an example of such a plot.) Both the average slope and the total range of the resulting traces were considered as attrition parameters.

#### 2.5.2.2.1 Average Rate of PLI Increase

The average slopes of the PLI versus #CS plots were estimated as:

$$\overline{M}_{PLI} = \frac{PLI \text{ (last burn)} - PLI \text{ (first burn)}}{\#CS}$$

#### Regression Results

Initially, the data set for this parameter included the results from the same 45 units that were in the %ΔW data set. No useful correlation with #CS was found, however, so the data from Test Block V were deleted from the set. Review of the Block V PLI plots indicates that their characteristics are too complex to be described by  $\overline{M}_{PLI}$ . The data from the injector geometry series units were also deleted. The data set then contained 30 cases from 20-25 mesh, non-spring loaded, shower-head units - all of which had #CS = 100.

The regression results and the test data are shown in Figure 28. Note that the basic fit is parabolic with the minimum (at  $V=31.2$  ft/sec,  $\overline{M}_{PLI} = 0.0134$ ) within the range of interest.

#### Catalyst Effects

The two way analysis of variance of the  $\overline{M}_{PLI}$  data from the 15 point catalyst effects data set indicated that, as with %ΔW, the effect of velocity is negligible in the range of the set. Again significant catalyst effects were found: The 20-25 mesh α phase and the 14-18 mesh results differ from the 20-25 results while the 90% attrited and the 25-30 mesh catalyst were substantially the same as the plain 20-25 mesh results. The catalyst effects data are also plotted in Figure 28.

The indication of an effect due to 20-25 mesh α phase catalyst is only partly due to the relatively high value of  $\overline{M}_{PLI}$  obtained from the 23.5 ft/sec injection velocity unit (changing this value to be similar to the results from the two lower velocity units in the analysis of variance does not change the conclusion). Bearing in mind, therefore, that there may be no physical basis for the indicated trend; the basic regression equation was modified as shown in Figure 28 to pass through the 20-25 α phase data without altering its shape. The minimum of this curve is at the low velocity 20-25 α phase point ( $V=2.9$  ft/sec,  $\overline{M}_{PLI} = 0.0172$ ).

As the figure indicates, the individual PLI versus #CS curves for the 14-18 mesh units tested showed little or no permanent change in PLI over the 100 cold start duty cycle at any of the injection velocities.

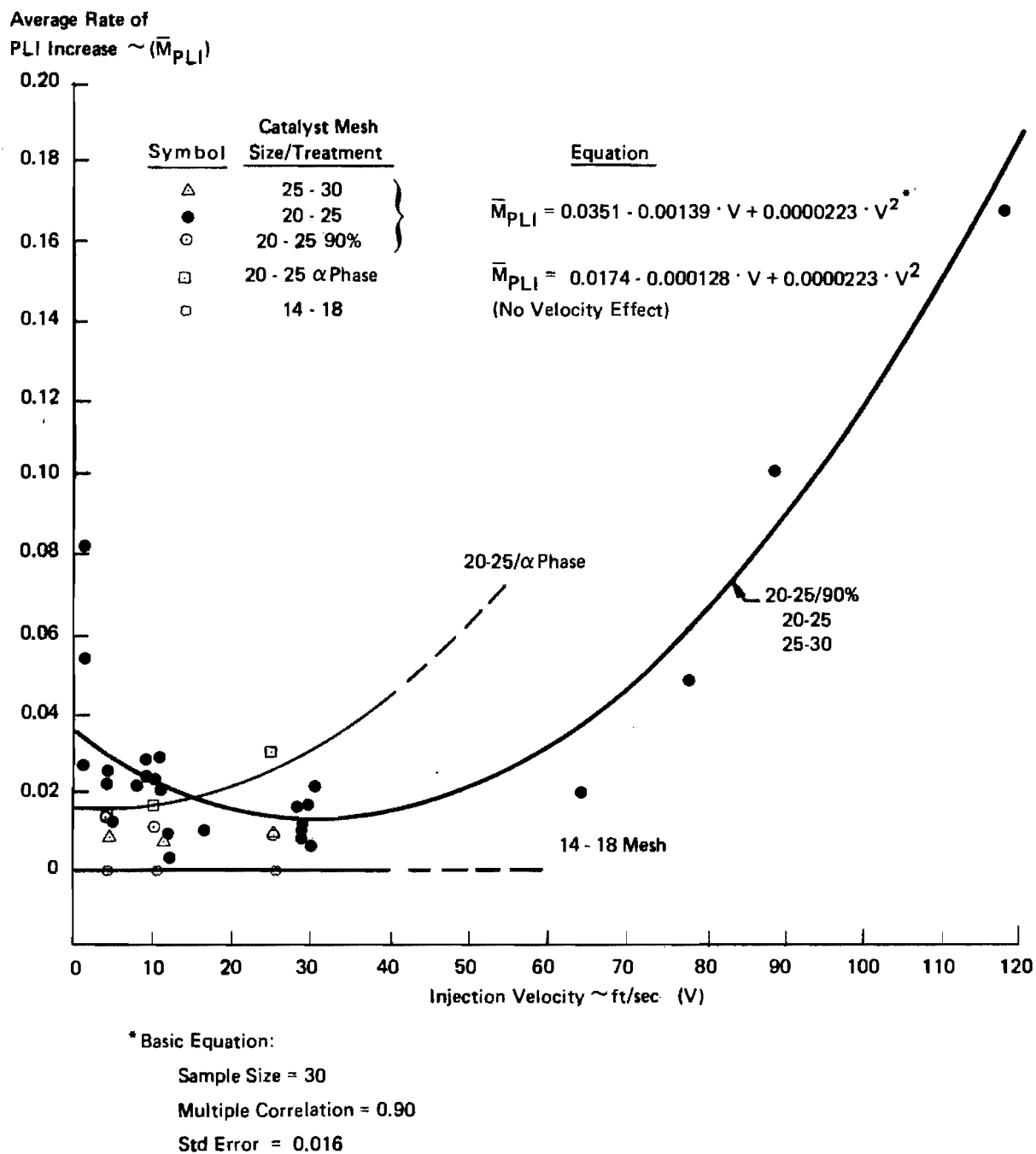


Figure 28. Average rate of pressure loss index increase versus injection velocity (100 cold starts).

### Injector Design Influences

As was noted above, none of the 14-18 mesh units exhibited lasting changes in PLI. This includes U-100 showerheads in the velocity range from 2.9 to 24 ft/sec and the three very low velocity (< 1 ft/sec) injectors.

The 20-25 mesh results are shown in Figure 29. The 95% upper prediction line for the basic regression line is also shown. It is clear that no significant deviation from the fit exists.

In Figure 29, as in Figure 27, the prediction interval includes zero. The same comment also applies: A confidence interval test indicates that the regression values are significantly different from zero.

#### 2.5.2.2.2 Range of PLI

The range covered by the PLI versus #CS plots was computed as:

$$R_{PLI} = PLI_{max} - PLI_{min}$$

### Regression Results

As with  $\overline{M}_{PLI}$ , a correlation of  $R_{PLI}$  with #CS was not obtained, consequently the regression data set for  $R_{PLI}$  was limited to the same 30 units as the  $\overline{M}_{PLI}$  set. In this case, however, it was necessary to use a  $1/V$  term in order to get a fit with a multiple correlation coefficient greater than about 0.8. The resulting expression shown in Figure 30 along with the data, fits the data well, but approaches  $\infty$  as  $V$  approaches 0.

### Catalyst Effects

The analysis of variance again indicated no velocity effect in the catalyst effects data set. The 20-25 mesh 90% attrited results were found to be the same as the plain 20-25 mesh results. The other catalyst sizes/treatments were found to differ significantly from those two.

The catalyst effect data are highlighted in Figure 30. The basic regression results were adjusted to pass through the  $\alpha$  phase 20-25 mesh and the 25-30 mesh data as indicated.

As with  $\overline{M}_{PLI}$ , the 14-18 mesh units had no change in PLI over the 100 cold start duty cycle. Compared to the plain 20-25 mesh results, the  $\alpha$  phase catalyst suffered greater  $R_{PLI}$  attrition, while the 25-30 mesh had slightly less. Again, however, the small number of data points for the  $\alpha$  phase and 90% attrited catalyst dictate caution in applying these results.

### Injector Design Influences

Figure 31 compares the design variations that were tested with 20-25 mesh catalyst with the regression results. Of the units shown, none exhibits significantly lower  $R_{PLI}$  attrition than the family. The Rigimesh Spud injector, which is shown in Figure 30, because of its high injection velocity, had a  $R_{PLI}$  value in the same range as those shown in Figure 31. Since it is lower than the 95% prediction line, this result may be considered statistically significant.

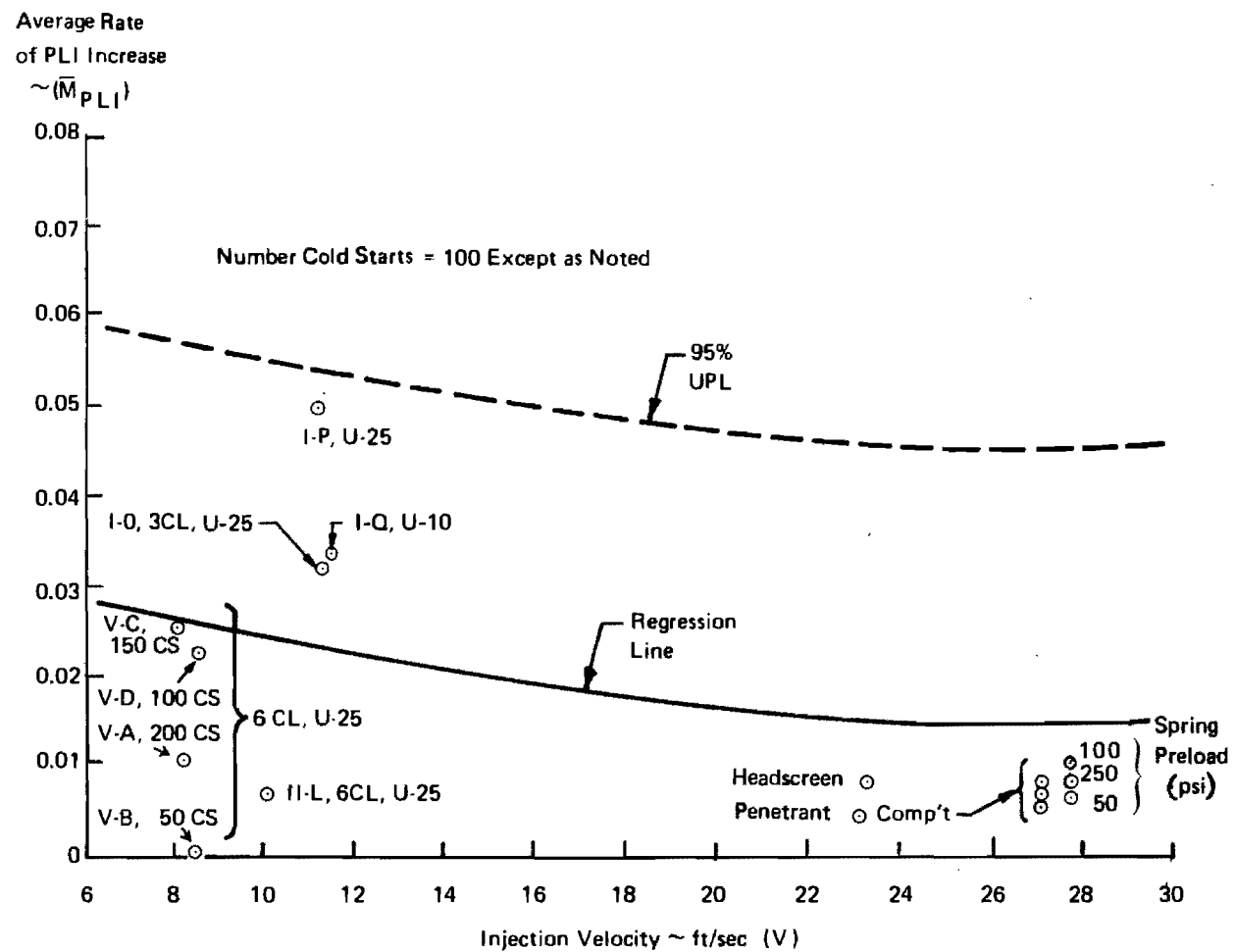
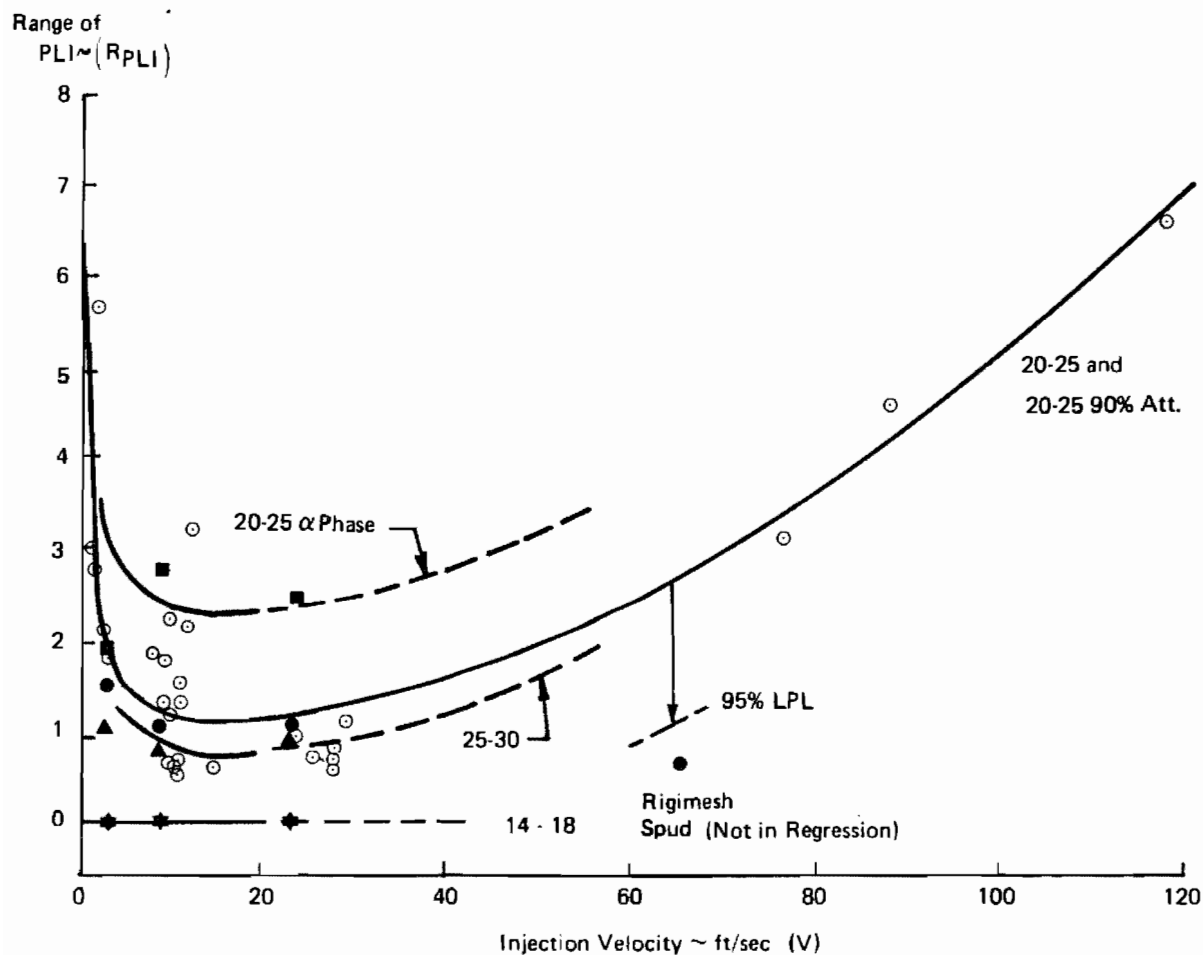


Figure 29. Injector design influences on SUB PLI increase (20-25 mesh).

| Catalyst<br>Mesh Size/<br>Treatment | Equation                                                        |
|-------------------------------------|-----------------------------------------------------------------|
| ○ 20-25                             | $R_{PLI} = 2.917/V + 4.285 \cdot 10^{-4} \times V^2 + 0.8671^*$ |
| ● 20-25 90% Att                     | $R_{PLI} = 2.917/V + 4.285 \cdot 10^{-4} \times V^2 + 0.8671^*$ |
| ■ 20-25 $\alpha$ Phase              | $R_{PLI} = 2.917/V + 4.285 \cdot 10^{-4} \times V^2 + 3.117$    |
| ▲ 20-30                             | $R_{PLI} = 2.917/V + 4.285 \cdot 10^{-4} \times V^2 + 0.5671$   |
| ★ 14-18                             | $R_{PLI} = 0$                                                   |



\* Basic Regression Equation:  
Sample Size = 30  
Multiple Correlation = 0.88  
Standard Error = 0.79

Figure 30. Range of pressure loss index over 100 cold starts versus injection velocity.

Number Cold Starts = 100 Except as Noted

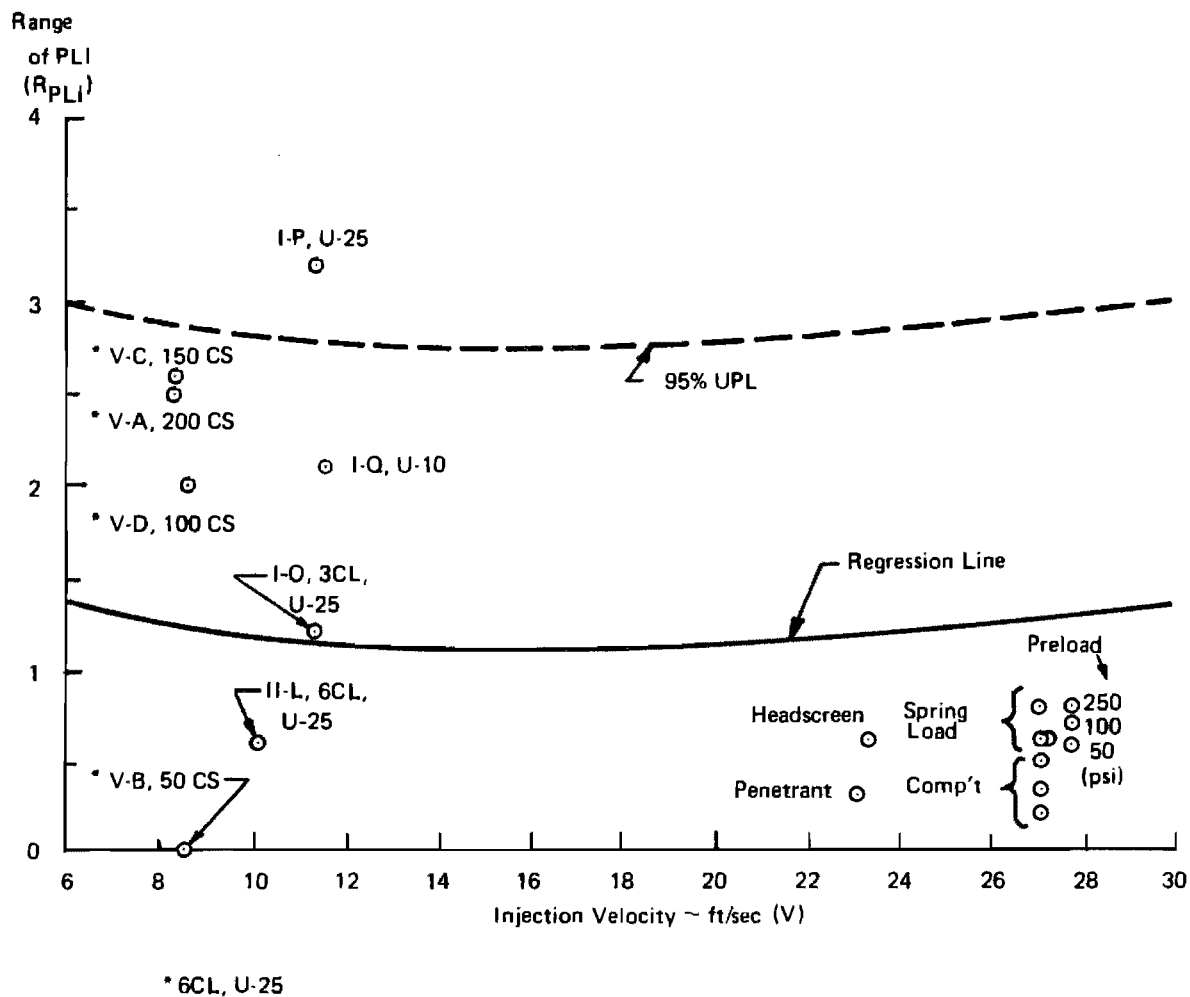


Figure 31. Injector design influences on range of PLI (20-25 mesh).

The 14-18 mesh results are tabulated below:

| Unit              | Injection Velocity | R <sub>PLI</sub> |
|-------------------|--------------------|------------------|
| III-F (U-100)     | 2.9                | -0-              |
| III-G (U-100)     | 9.0                | -0-              |
| III-H (U-100)     | 23.5               | -0-              |
| IV-A (Vortex)     | 0.4                | 0.1              |
| V-N (6 Rigimesh)  | 0.24               | 4.2              |
| IV-B (Hemisphere) | 0.3                | 0.8              |

Since no trend can be established for the U-100 injectors, it is not possible to assess the merits of any of the designs independent of injection velocity. The improved designs are characterized by low effective injection velocities and measurable R<sub>PLI</sub> but the basic curve fit, which may or may not apply to 14-18 mesh catalyst, predicts some increased attrition at these velocities.

#### 2.5.2.3 Combustion Roughness

The percent half amplitude lower bed chamber pressure roughness was computed for each data frame of the steady-state firings of the duty cycle as follows:

$$R = \left( \frac{P_c (\text{Max}) - P_c (\text{Min})}{2 \bar{P}_c} \right) \times 100$$

The parameters available for analysis from the individual R versus #CS plots are:

$$\Delta R = R (\text{first firing}) - R (\text{Max for the duty cycle})$$

and mean roughness;

$$\bar{R} = \frac{\Sigma R (\text{over the duty cycle})}{(\text{number of R samples})}$$

The mean roughness, while it should be considered in designing an injector, is not as sensitive an attrition parameter as the change in roughness. Thus  $\Delta R$  was studied and is discussed below.

#### Regression Results

The 2-way analysis of variance that performed on the catalyst effects data set indicates that only the 14-18 mesh data differed from the others. The regression data set therefore, consisted of the results from 42 U-100 injectors which did not contain 14-18 mesh catalyst.

The resulting best fit differs from the previous results in that the logarithm of the number of injection orifices (#OR) was found to improve the correlation. Roughness range is the only attrition parameter which has been found to be affected by a non-velocity injection parameter. The regression equation is:

$$\Delta R = 6.66 \cdot 10^{-6} V^2 \#CS + 2.22 \cdot 10^{-4} \#CS^2 \\ + 2.06 \text{ LOG}_{10} (\#OR) - 1.75$$

(N = 42, Multiple correlation = 0.86  
Standard error = 2.79)

On the velocity plane, this represents a family of parabolas with their minima at  $V = 0$ . The curves for various cold starts and  $\#OR = 6$  are shown in Figure 32 along with the comparable data points. The family for various numbers of orifices at  $\#CS = 100$  is shown in Figure 33.

#### Catalyst Effects

As was indicated above, the 14-18 mesh catalyst showerhead units were found to have significantly lower  $\Delta R$  attrition than those with the other catalysts. The 14-18 mesh data are plotted against  $\#OR$  in Figure 34. The equation for the line shown through the three U-100 points is the same as that presented above for the regression except that the constant has a value of -5.0.

#### Injector Design Influences

The non-velocity, non-orifice count, injector design effects are discussed by catalyst type below.

**Catalysts Other Than 14-18 Mesh** - Due to the number of parameters involved, a graphical comparison of the non-U-100 results with the regression predictions is difficult to present clearly. The comparison was made and it shows that only the penetrant type unit falls below the relevant prediction interval-indicating a significant improvement in roughness stability over the other types.

**14-18 Mesh** - As Figure 34 shows, the design variations tested with 14-18 mesh catalyst were significantly worse in roughness range than the basic U-100 design.

#### 2.5.2.4 Pulse Centroid

The block V ARA's were tested to a duty cycle intended to result in pulse data as well as accumulate cold starts. The resulting data were integrated and the pulse centroid values studied. The pulse widths used were 20, 35, 65, 100 and 200 ms. Only the two extreme values are discussed here since the trends observed on the others were identical.

The shift in pulse centroid (linear) is shown in Figure 35 for 0.020 and 0.200 second pulses as a function of number of cold starts and injection velocity. A significant centroid shift was observed for the 100fps units when the pulse train for the specific pulse width was begun at low temperature ( $50 \pm 10^\circ \text{F}$ ). This shift was not evident in the 10 fps and 50 fps data.

As expected, higher injection velocities initially produced lower centroids. However, as the duty cycle progressed, other trends evolved. The 100 fps units showed a rapid increase in centroid after 50 cold starts. This trend continued to about 100 cold starts, where the curve flattened to a level about 0.035 seconds greater than the initial value. Similarly, the 50 fps injector produced a centroid shift beginning at about 90 cold starts, but the shift was over twice the magnitude of that observed for the higher velocity injectors (0.070 sec). The shift in the centroid for the 10 fps injectors

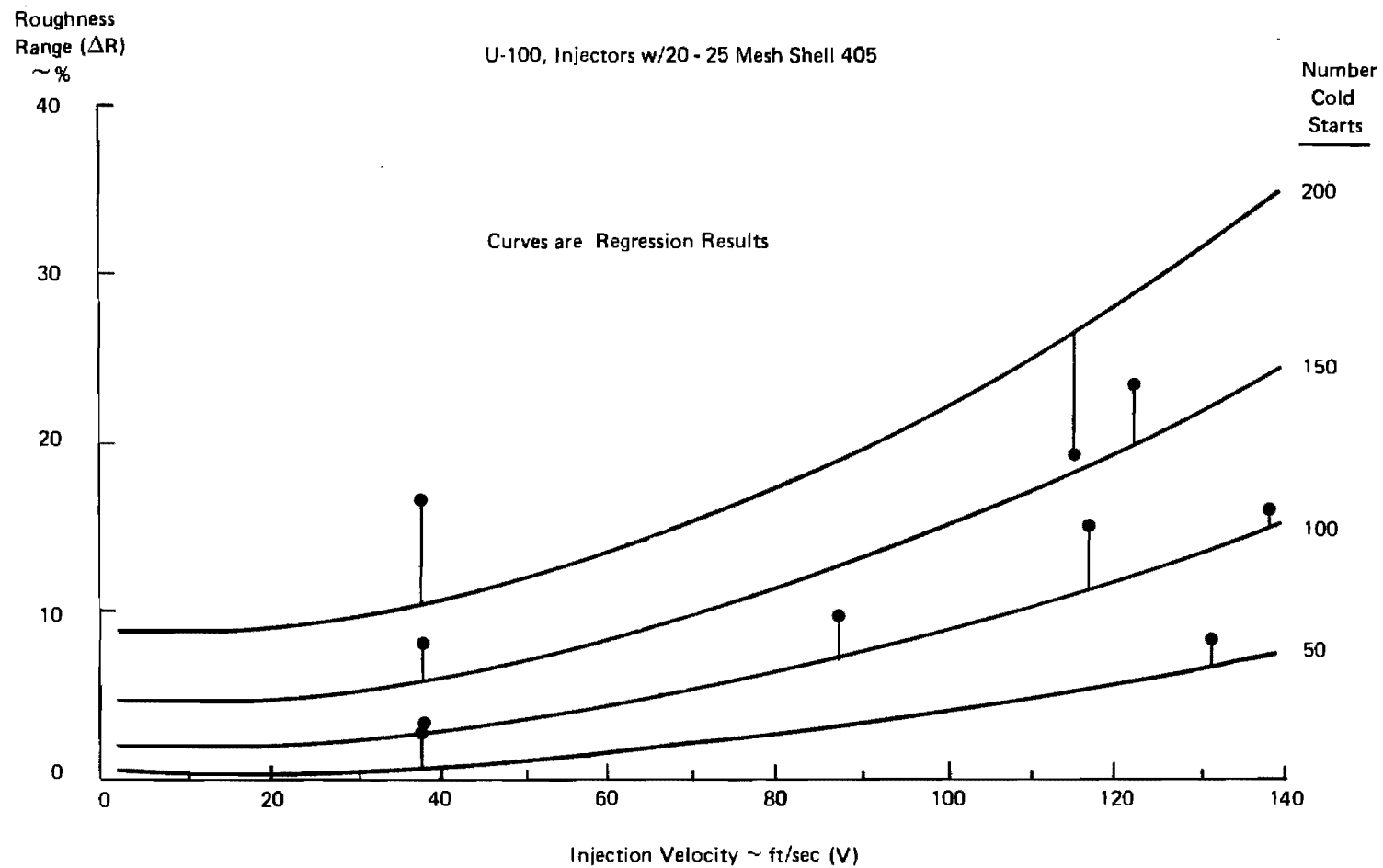


Figure 32. Roughness range versus injection velocity and cold starts (6-orifice injectors excluding 14-18 mesh Shell 405)

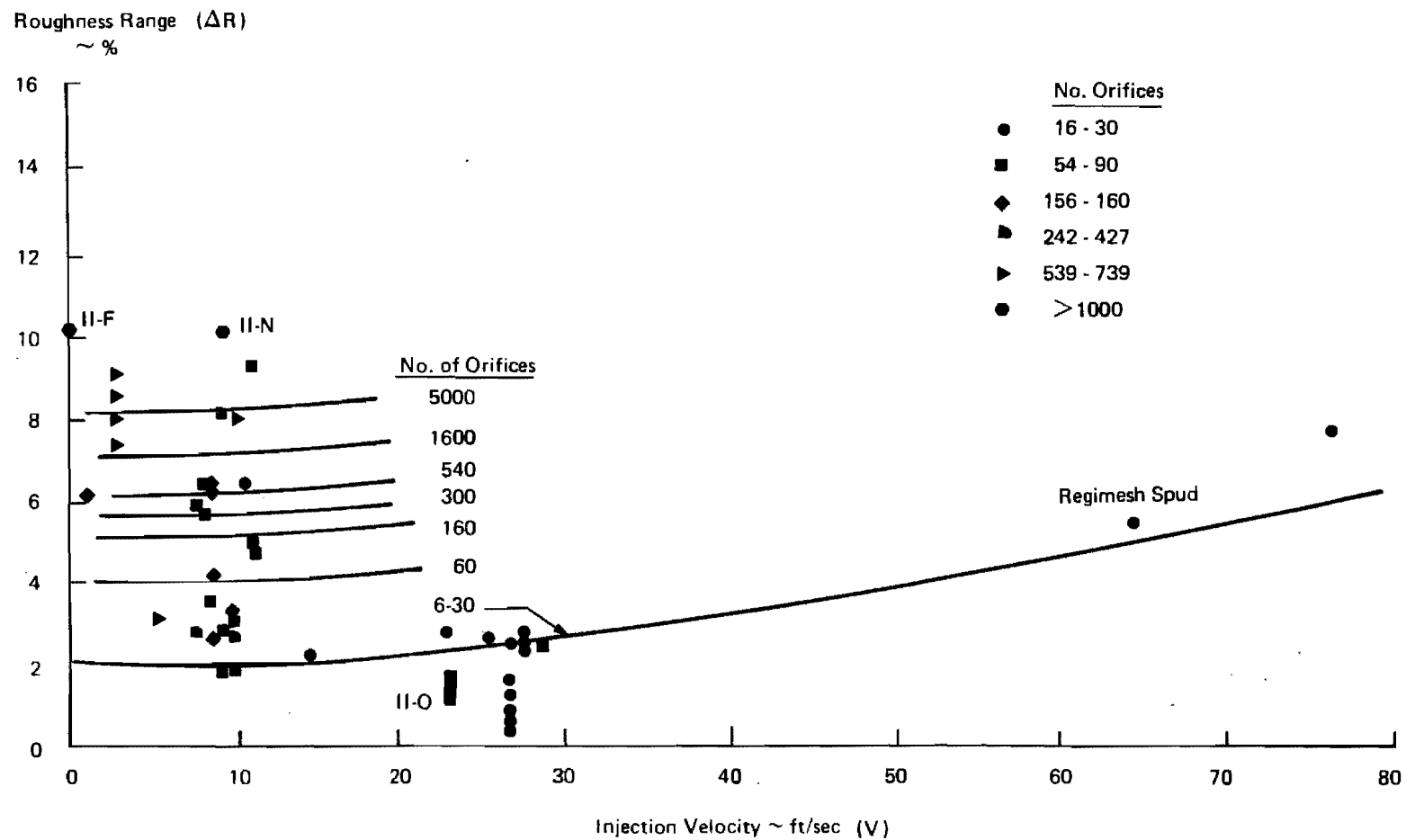


Figure 33. Roughness range versus injection velocity (excluding 14 - 18 mesh catalyst, 100 cold starts only).

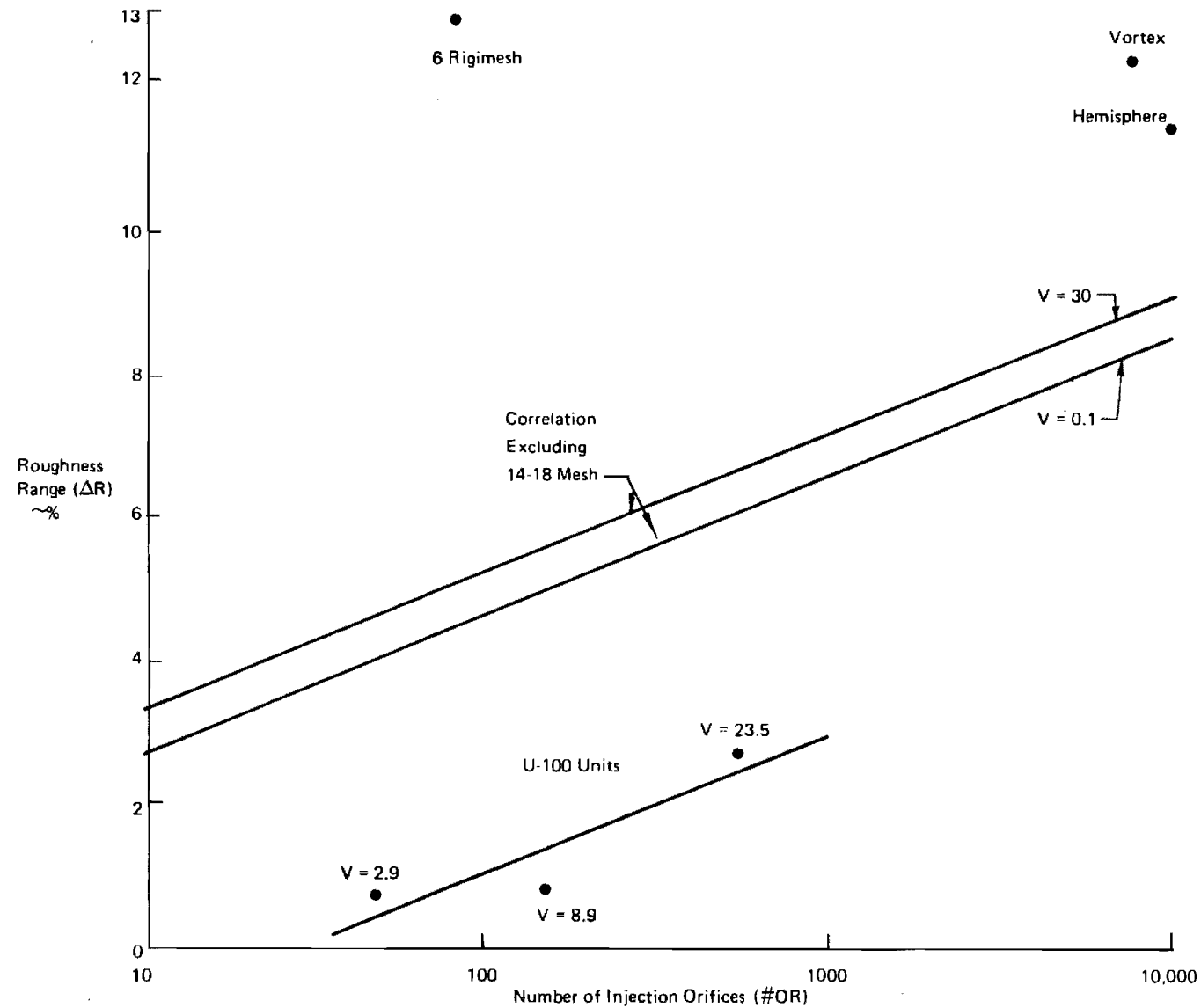


Figure 34. Roughness range versus number of injection orifices; 14-18 mesh data compared to others,(100 cold starts).

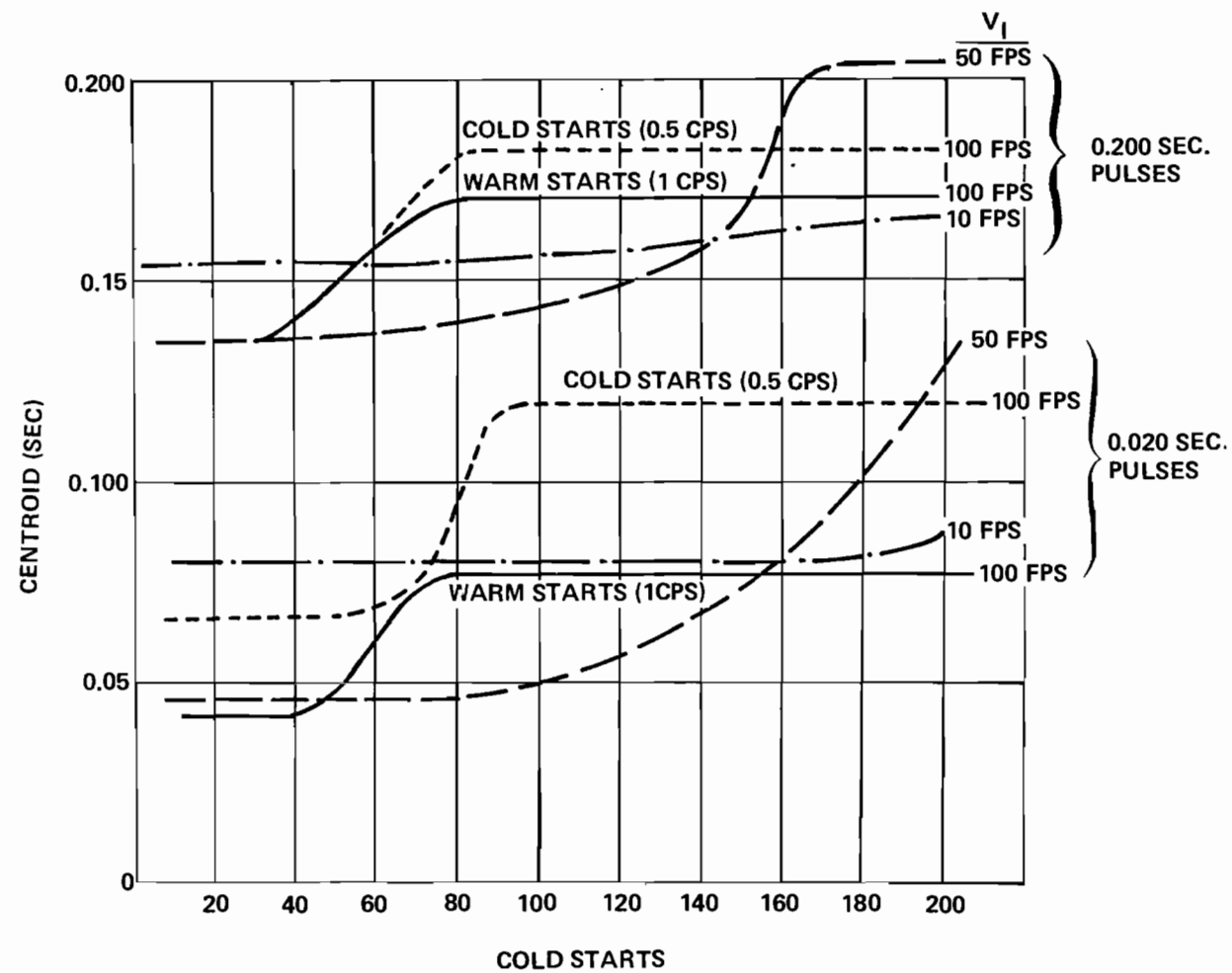


Figure 35. Effects of injection velocity and cold starts on mean linear centroid for first ten pulses.

was only 0.010 sec over the 200 starts, but we probably had not reached the level of catalyst attrition necessary to produce drastic changes in these low velocity units.

The larger magnitude of the centroid shift with the 50 fps injection velocity is probably associated with the propellant transport characteristics as voids are formed in the catalyst bed. If this is the case, one would expect the lower velocity injectors to exhibit the greatest shifts as the catalyst bed is depleted.

During these tests, no significant change in impulse bit was seen on the 10 fps units. Both the 50 fps and 100 fps units showed a decrease of some 12% in impulse bit. Characteristic velocity ( $c^*$ ) did not change significantly.

#### 2.5.2.5 Performance Reproducibility

Figure 36 typically shows the performance history of four identical SUB's (10 fps nominal injection velocity), each one exposed to an increasing number of cold starts. The interesting observation is the remarkable reproducibility, exhibited in all four measurements. This was repeatedly confirmed with other test cases.

#### 2.5.2.6 Digital Image Processing System Results

Digital Image Processing data on sectioned catalyst beds are presented in Figure 37, exhibiting the effects of injection velocity and flow rate on the mean "equivalent" particle diameter of different catalysts. This parameter is the arithmetic average defining the frequency distribution of the particle sizes in the sectioned bed ("two-dimensional distribution"). The "equivalent" particle diameter was calculated by equating the measured cross-sectional area,  $A$ , and the perimeter,  $P$ , of each dissected particle in the bed to the area and perimeter of a circle. The resulting expression was  $4 A/P$ . Included in the plotted data are the mean and standard deviation (measure of process variability) of particle size measurements performed on several unfired beds..

A Fisher Variance Ratio and Student's "t" test\* was applied to test whether a significant overall reduction in particle size occurred (within the random measurement error) due to firing exposure. Regardless of the independent parametric deviation, values of all 20-25 mesh fired beds were pooled and their statics tested. This somewhat gross statistical test disclosed that the variances are indifferent and there is only a marginal difference ( $t$ -calculated = 2.06,  $t$ -critical = 2.01 at  $\alpha = 0.05$ )\*\* between mean particle diameters of fired versus unfired beds. This could imply an overall deterioration due to some or all variables. Isolation of the attributable factors was subsequently carried out with the multiple regression analysis, which also treated the influence of catalyst size, catalyst bed retention, injector geometry, etc. The proper interpretation of these particle data must be tempered with consideration of inherent DIPS resolution. Visual observations repeatedly indicated a second population composed of fines too small to be treated by this equipment ( $< 7$  mils). This obviously biased the particle statistics, and mean parameters must be considered in a comparative frame.

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\*Even though the individual particle size distribution per catalyst bed sample was not normally distributed, means of large samples do approximate a normal distribution (Central Limit Theorem), so that conventional significance tests are applicable.

\*\*Differences in the mean equal to or greater than obtained could be expected to occur by chance only 5% of the time and can therefore be assumed to represent real changes.

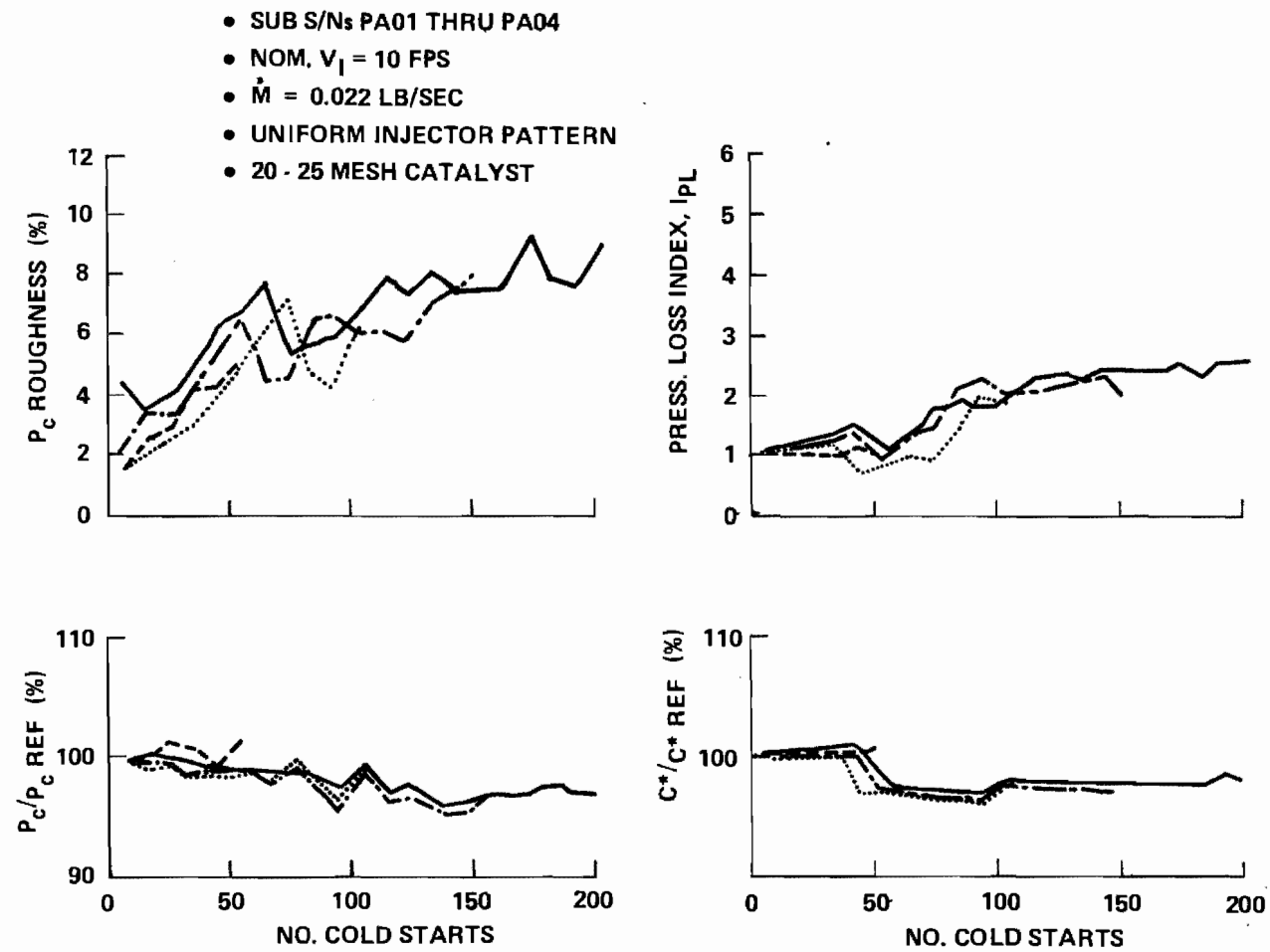


Figure 36. ARA performance reproducibility.

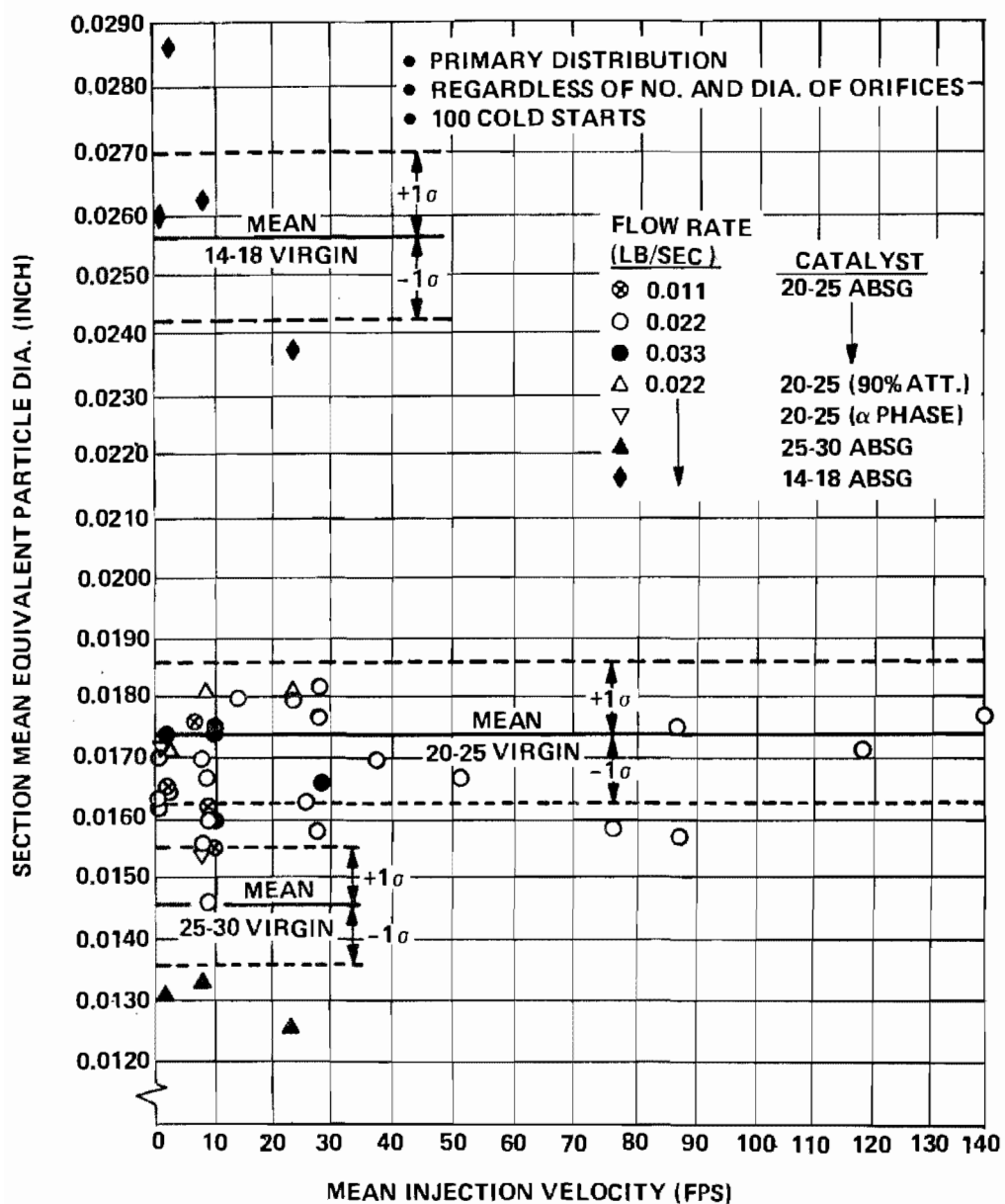


Figure 37. Velocity and flow rate effect on section mean catalyst particle diameter.

### 2.5.3 Future Efforts

In addition to analysis of the results from the upcoming test block, several areas for further effort present themselves.

A preliminary attempt has been made to correlate measured catalyst weight loss with the fire test attrition parameters. The result is promising, but further work is required.

More effort is planned in the areas of the Digital Image Processing System Data and the pulse performance results from Block V.

The pressure drop data will be studied to attempt to clarify the effect of various cold start accumulations.

## SECTION 3.0 MATERIALS EVALUATION

Most hydrazine reactors currently in use are fabricated from some combination of stainless steels, nickel alloys, or cobalt alloys. These metals are, to varying degrees, susceptible to nitriding and/or hydrogen embrittlement. Since the decomposition products of hydrazine are hydrogen, nitrogen, and ammonia, the latter tending to decompose further into the former two, the operating reactor environment is perfectly suited to produce both nitriding and hydrogen embrittlement in its structural members. Of the two, nitriding appears to be the more serious problem among candidate materials. Further, while the problem is of some concern for long-life reactors even in massive components, it is particularly severe in high surface-to-volume ratio components such as screens and fine injection orifices.

The purpose of this Phase, then, was:

- Acquisition of basic nitride depth and rate information;
- Evaluation of materials potentially superior to those currently in use; and
- Evaluation of surface-protective coatings/claddings for currently used materials.

### 3.1 APPROACH

The approach in this phase was to expose materials specimens to the decomposition products from a workhorse reactor. Two series of tests were made. In the first series, the dynamic exposure test, coupon samples of candidate materials were mounted in a heated, insulated, continuous flow test chamber fed from the workhorse reactor. Replicate sets of this assortment of materials, rationale for selection of which is presented in Paragraph 3.2, were exposed to the reactor effluent for varying periods of time, up to 10 hours. They were then subjected to bend tests and metallographic analyses, and their properties compared with those of unexposed specimens.

From this test series both absolute nitriding depth and rate of nitriding information was obtained, as well as a qualitative ranking of the candidates. These data were the basis for selection of the most promising high surface-to-volume ratio candidates (screens) for the next test series, the in situ reactor tests.

For the in situ tests, bed separator screens were fabricated from six candidates selected from the dynamic exposure tests.

### 3.2 MATERIALS SELECTED FOR EVALUATION

Materials were selected for evaluation on the basis of literature and experience, availability and cost, adaptability to reactor components, and, foremost, expected compatibility with the hydrazine decomposition products environment. We attempted to include materials representative of relevant classes. For example, platinum-10% rhodium was chosen as representative of the platinum alloy class.

Nineteen materials and coatings were selected for evaluation. These, together with their composition, are given in Table 6. The rationale for this selection follows. Note that this rationale was documented prior to testing and should not be considered as conclusions, particularly in the case of Nichrome V.

### 3.2.1 Base Metals

Thirteen base metals were selected for evaluation, with emphasis on covering all relevant classes of materials. (A fourteenth, Columbium 291, was not tested uncoated because previous work had shown the bare material to be unsatisfactory.) Nickel, cobalt, and molybdenum base alloys are generally resistant to nitriding in the service temperature range encountered in hydrazine engines. However, nickel and cobalt base alloys can be susceptible to hydrogen embrittlement. Tungsten and platinum offer excellent resistance and are included. One iron base alloy, GE 2541, was also included because of its unique surface passivation characteristic.

The Haynes alloys (25 and 188) are the best known cobalt base alloys for this environment.

Hastelloy B was chosen as representative of the Hastelloy family based on the published experience at Rocket Research.<sup>(1)</sup> In addition to being a reasonably resistant alloy, it has a favorable coefficient of thermal expansion ( $8.1 \times 10^{-6}$  in./in. °F compared to over  $9 \times 10^{-6}$  for Inconel and Haynes alloys).

Inconel alloys have been widely used in ammonia and nitrogen-hydrogen environments. They are reasonably inexpensive and available, and are resistant to nitriding and hydriding. Inconel 600 is the most readily available in various forms, including screens. Inconel 718 has better nitriding properties based on the literature. Inconel 750 should offer somewhat better hydriding resistance than 718 because of its high nickel content but may be somewhat more susceptible to nitriding.

Nichrome is highly resistant to hydriding but was not expected to be as good as some of the other alloys in nitriding.\* It is readily available and inexpensive.

Platinum is one of the best materials known in terms of resistance to nitriding. However, its strength and cost probably limit its use to screens or small internal parts. Platinum-10% rhodium was chosen in preference to pure platinum because it offers better mechanical properties without compromising resistance to nitriding. Screens of this alloy are commercially available. Also, pure platinum was tested as a plating.

Molybdenum and tungsten are among the best materials available in terms of both nitriding and hydriding resistance, but they both require protection from oxidation. In most hydrazine space system applications however, oxidation is not a problem. Therefore, tungsten and molybdenum alloys are good candidates. Moly-50 rhenium and TZM were chosen over pure molybdenum because of their better mechanical properties. Also, the TZM is a relatively inexpensive moly alloy.

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(1) Monopropellant Engine Investigation for Space Shuttle Reaction Control System, Sixth Monthly Report, Contract NAS 8-28950, Rocket Research Corporation, January 16, 1973.

\* The postulation that Nichrome V would be susceptible to nitriding was subsequently proven erroneous.

TABLE 6  
COMPOSITION OF TEST MATERIALS

| Material                                 | Constituents |         |          |      |           |         |         |          |           |                        |        |         |           |         |          |             |
|------------------------------------------|--------------|---------|----------|------|-----------|---------|---------|----------|-----------|------------------------|--------|---------|-----------|---------|----------|-------------|
|                                          | Chromium     | Nickel  | Tungsten | Iron | Manganese | Silicon | Cobalt  | Aluminum | Titanium  | Colum.<br>and<br>Tant. | Copper | Moly    | Zirconium | Rhenium | Platinum | Rhodium     |
|                                          | Cr           | Ni      | W        | Fe   | Mn        | Si      | Co      | Al       | Ti        | Nb and Ta              | Cu     | Mo      | Zr        | Ru      | Pt       | Rh          |
| 1. Haynes 25                             | 19-21        | 9-11    | 14-16    | 3    | 1-2       | 0.4     | Rem.    | -        | -         | -                      | -      | -       | -         | -       | -        | -           |
| 2. Haynes 188                            | 20-24        | 20-24   | 13-16    | 3    | 1.25      | 0.2-0.5 | Rem.    | -        | -         | -                      | -      | -       | -         | -       | -        | -           |
| 3. Hastelloy B                           | 1            | Rem.    | -        | 4-6  | 1         | 1       | 2.5     | -        | -         | -                      | -      | 26-30   | -         | -       | -        | V 0.2-0.4   |
| 4. Inconel 600                           | 14-17        | 71-80   | -        | 6-10 | 1         | 0.5     | 1       | 0.35     | 0.5       | 1                      | 0.5    | -       | -         | -       | -        | -           |
| 5. Inconel 718                           | 17-21        | 50-55   | -        | Rem. | 0.35      | 0.35    | 1       | 0.2-0.8  | 0.65-1.15 | 4.75-5.5               | 0.3    | 2.8-3.3 | -         | -       | -        | -           |
| 6. Inconel X-750                         | 14-17        | 70-77   | -        | 5-9  | 0.35      | 0.35    | 1       | 0.4-1.0  | 2.25-2.75 | 0.7-1.2                | 0.5    | -       | -         | -       | -        | -           |
| 7. Nichrome V                            | 20 Nom.      | 80 Nom. | -        | -    | -         | -       | -       | -        | -         | -                      | -      | -       | -         | -       | -        | -           |
| 8. Platinum-Rhodium                      | -            | -       | -        | -    | -         | -       | -       | -        | -         | -                      | -      | -       | -         | -       | 90 Nom.  | 10 Nom.     |
| 9. Molybdenum - Rhenium                  | -            | -       | -        | -    | -         | -       | -       | -        | -         | -                      | -      | 50 Nom. | -         | 50 Nom. | -        | -           |
| 10. Tungsten                             | -            | -       | 100      | -    | -         | -       | -       | -        | -         | -                      | -      | -       | -         | -       | -        | -           |
| 11. T2M Molybdenum<br>(Cb 291 Columbium) | -            | -       | -        | -    | -         | -       | -       | -        | 0.4-0.55  | -                      | -      | Rem.    | 0.06-0.12 | -       | -        | -           |
|                                          | -            | -       | 9-11     | -    | -         | -       | -       | -        | -         | -                      | -      | -       | -         | -       | -        | -           |
|                                          |              |         |          |      |           |         |         |          |           | 9-11<br>Cb<br>Rem.     |        |         |           |         |          |             |
| 12. GE 2541                              | 25 Nom.      | -       | -        | Rem. | -         | -       | -       | 4 Nom.   | -         | -                      | -      | -       | -         | -       | -        | Y<br>1 Nom. |
| 13. B1900                                | 8 Nom.       | Rem.    | -        | -    | -         | -       | 10 Nom. | 6 Nom.   | 1 Nom.    | Ta 4 Nom.              | -      | 6 Nom.  | 0.1 Nom.  | -       | -        | -           |
| Coatings                                 |              |         |          |      |           |         |         |          |           |                        |        |         |           |         |          |             |
| 14. Aluminide, Al-10 Si, on B1900        | -            | -       | -        | -    | -         | -       | -       | -        | -         | -                      | -      | -       | -         | -       | -        | -           |
| 15. Silicide R508C, on Cb 291            | -            | -       | -        | -    | -         | -       | -       | -        | -         | -                      | -      | -       | -         | -       | -        | -           |
| 16. Silicide R508C, on Cb 297            | -            | -       | -        | -    | -         | -       | -       | -        | -         | -                      | -      | -       | -         | -       | -        | -           |
| 17. Tungsten on Inconel 600              | -            | -       | -        | -    | -         | -       | -       | -        | -         | -                      | -      | -       | -         | -       | -        | -           |
| 18. Platinum on Nichrome V               | -            | -       | -        | -    | -         | -       | -       | -        | -         | -                      | -      | -       | -         | -       | -        | -           |
| 19. Rhodium on Nichrome V                | -            | -       | -        | -    | -         | -       | -       | -        | -         | -                      | -      | -       | -         | -       | -        | -           |

B-1900 and IN100 are both quite similar to the Inconels except for a somewhat higher cobalt content. Both are castable alloys, but B-1900 has much better welding properties. Therefore, B-1900 was chosen to represent this class.

The GE 2541 is an iron base alloy which contains aluminum and yttrium. These alloying elements both form very stable oxides. For example, the substrate for the Shell 405 catalyst is a form of aluminum oxide. When treated properly, then, the 2541 alloy may form a highly resistant coating on itself. Although it is a relatively new material, it could be produced in most forms at reasonable cost.

### 3.2.2 Platings and Coatings

Platings and coatings were selected based on the combination of their properties and those of the base metal. For example, there appears to be little reason to platinum plate the molybdenum alloys because the base material already has resistance comparable to the plating. Conversely, platinum plating materials which are susceptible to nitriding is logical because the platinum should isolate the base metal from the nitrogen environment.

Aluminide and silicide coatings have been used effectively in the past to prevent high temperature oxidation and, to a lesser extent, to prevent materials from nitriding and/or hydriding. B-1900 was chosen to be tested with the Al-10 Si aluminide because some protection was expected to be necessary on this castable nickel alloy. Columbium 291 with the Silicide coatings was chosen because of the extensive data base on these combinations. The columbium was not selected in the base coupon tests because it had already been shown to be unacceptable uncoated in Bell independent research testing.

As mentioned earlier, Inconel 600 was expected to be a poorer Inconel, but it is available in most common forms. Therefore, a coating of tungsten - a highly resistant material - was selected to protect the Inconel 600 from nitriding and hydriding. The tungsten can be applied either by vapor deposition or in a fused salt process, and could be applied to screens in the same manner.

Another inexpensive and readily available nickel base alloy, nichrome, is highly resistant to hydriding but was thought to be susceptible to nitriding. Therefore, by plating nichrome with platinum, the nitriding environment could be excluded.

Platinum is porous to hydrogen, but because of the hydriding resistance of the nichrome this was not expected to cause a problem. Rhodium is similar to platinum in its resistance to degradation but is somewhat less porous to the hydrogen. It should also provide good protection for the nichrome or similar hydriding-resistant alloys.

### 3.2.3 Screens

The selection of materials for further testing in screen form was based on the results of the Dynamic Exposure Test (discussed in Paragraph 3.3), constrained by the availability of materials in usable form.

The materials of particular interest because of their excellent durability in the earlier test were Nichrome V, platinum-10% rhodium, and tungsten. All of these could be obtained as screens and were selected for further testing.

Hastelloy B ranked slightly below the first group. This material is widely used, and was considered one of the better choices for structural parts.

A materials evaluation at Rocketdyne<sup>(1)</sup> (1000 hours at 1800°F, ammonia environment) showed Inconel 600 to be a good candidate. Our data corroborated this finding, and Inconel 600 was included as the Inconel family representative.

We had hoped to test Haynes 188 screen because it appeared to be superior to Haynes 25. However, it could not be obtained in screen form within a reasonable time. Haynes 25 was included, but more as a baseline for the test since it has been extensively used in various hydrazine applications.

### 3.3 DYNAMIC EXPOSURE TEST

The selected materials (Paragraph 3.2) were obtained, machined into 1/2 inch x 3 inch test coupons and plated/coated where applicable. These were generally 0.050 in. thick, but some deviation was allowed in cases where availability or cost (such as Pt-10 Rh) dictated. Four sets of coupons were made: One for a control, and one each for exposures of 1, 6, and 10 hours, respectively.

#### 3.3.1 Test Apparatus

The coupons were installed in three holders, one set per holder, as shown in Figure 38. These, in turn, were placed in the Materials Test Chamber (Figure 39). The materials test chamber was fitted with a sonic nozzle sized to produce chamber pressure of 100 psia at a nominal hydrazine flow rate of 0.022 lb/sec. Redundant hydrazine gas generators were welded to an inlet manifold and fed through a cavitating venturi from a conventional regulated nitrogen-pressurized hydrazine test stand.

An electric heater was installed around the chamber and set to maintain the gas generator outlet gas temperature, as measured in the manifold below the gas generators (Figure 40). Nominally, this temperature was 1400°F - somewhat lower than will be found inside a hydrazine thruster - because of the high ammonia dissociation (~75%) in the long catalyst bed of the reactor, which was designed as a tank pressurization gas generator. Still, the conditions of continuous replenishment of gases within the sample region at temperatures conducive to further ammonia dissociation and representative of nozzle entrance conditions in small hydrazine devices was maintained. The chamber and gas inlet manifold were wrapped with Fiberfrax insulation to minimize heat loss.

The gas inlet manifold was fitted with a helium purge to prevent exposure of hot samples to air and/or gases which might be reactive at elevated temperatures. This purge was also used as a means to accelerate cooling.

#### 3.3.2 Testing

The exposure of the coupons was accomplished by firing the gas generator for one hour, cooling with a helium purge, removing the first sample holder, firing for five more hours, cooling and removing the second sample holder, and then exposing the third sample set for the remainder of the ten hours.

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(1) Hydrazine Gas Generator Program, Monthly Progress Report No. 6, Contract NAS 9-13003, Rocketdyne, January 1973.

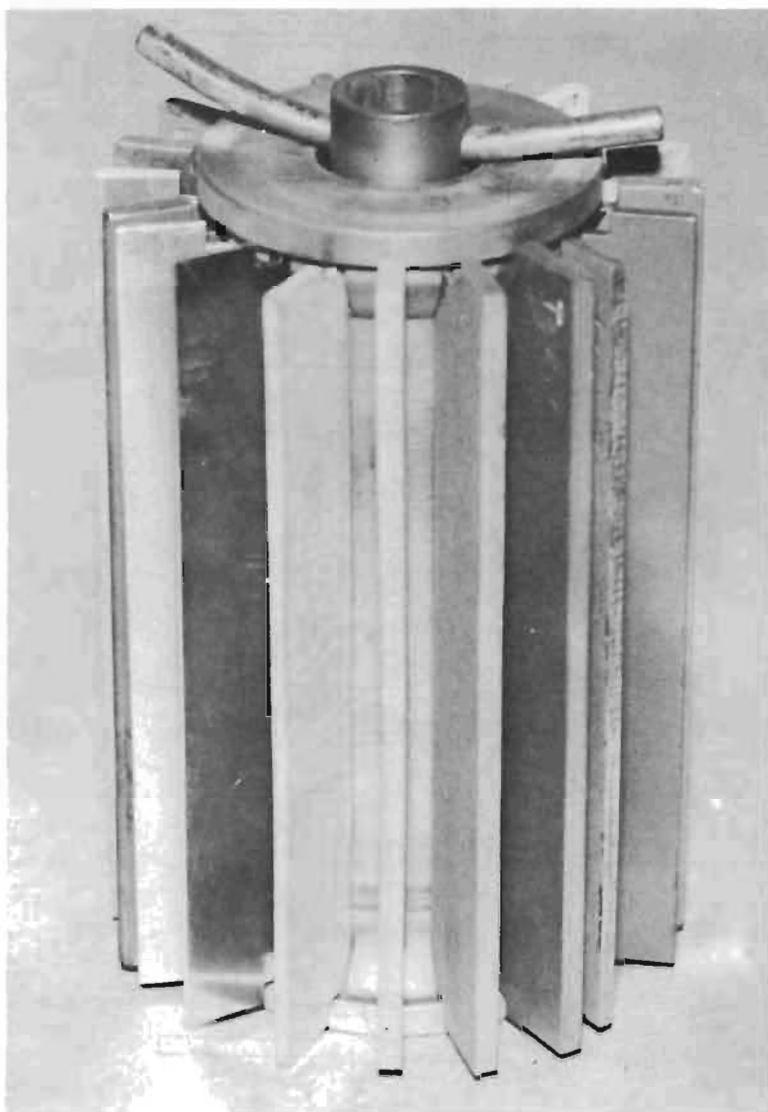


Figure 38. Sample holder with coupons installed (after 10-hour exposure).

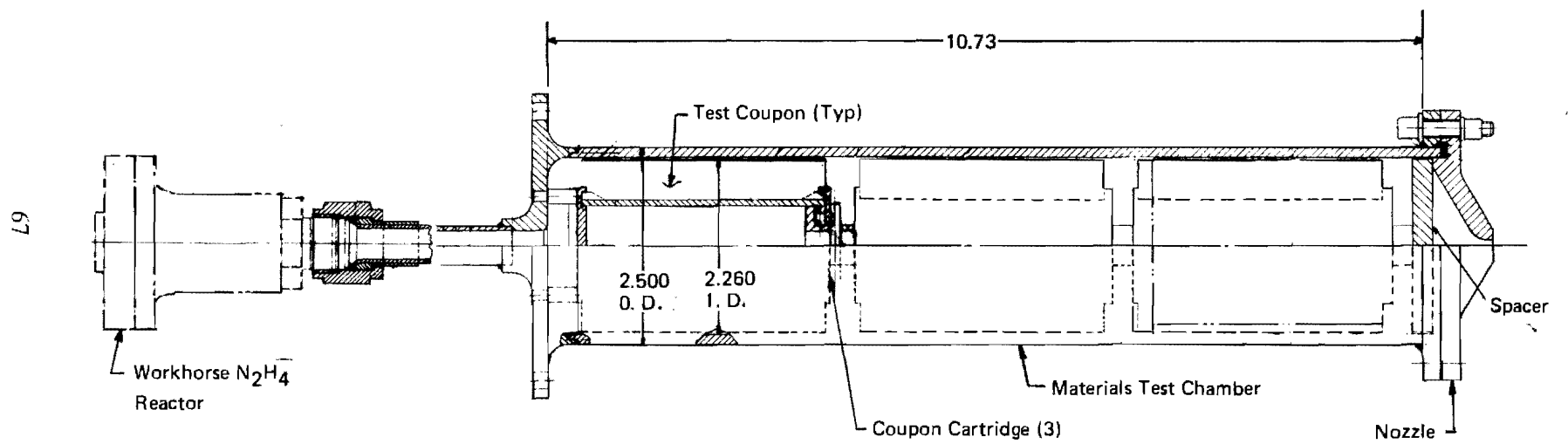


Figure 39. Materials test chamber.

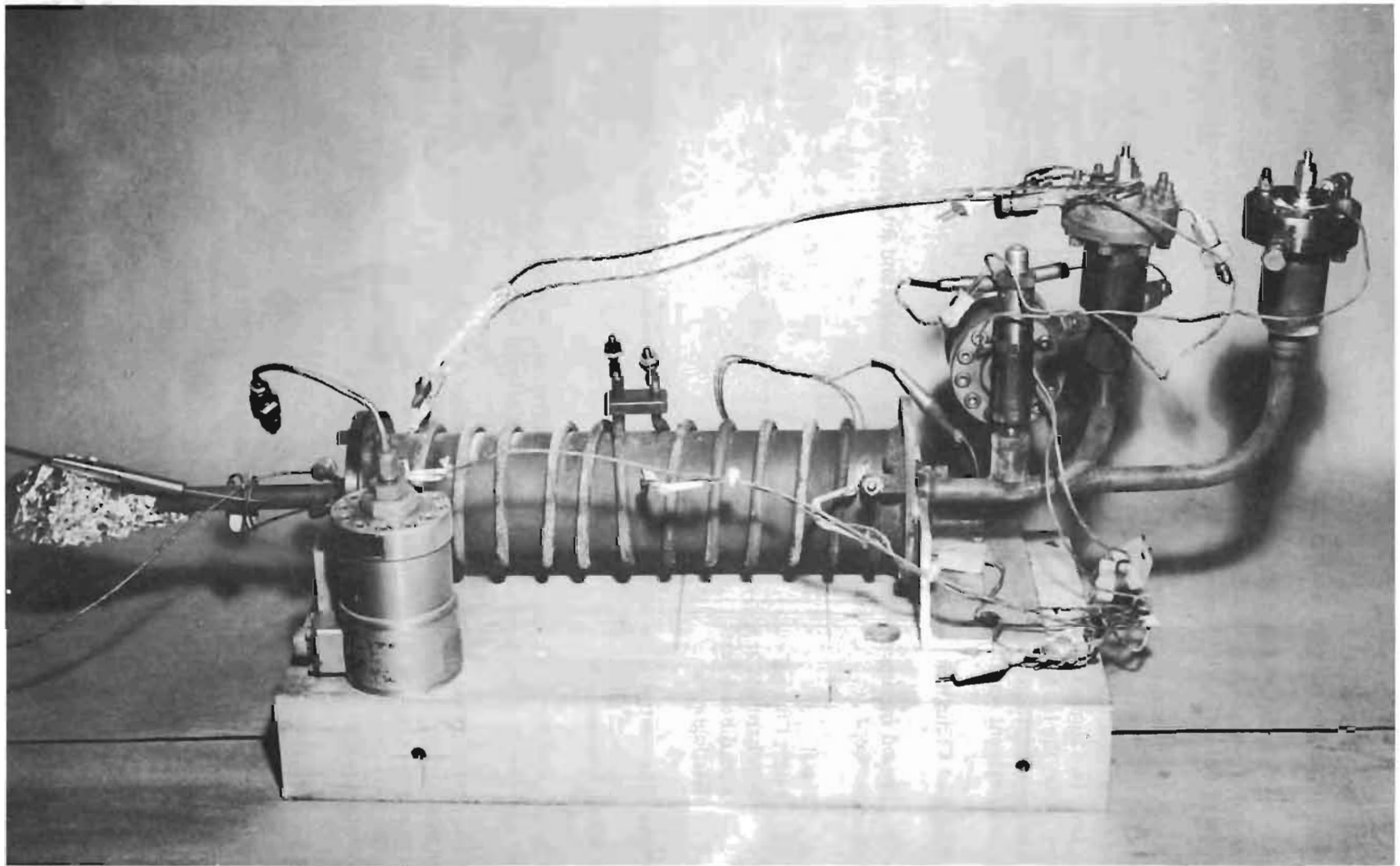


Figure 40. Materials test chamber with heater and gas generators (after test).

Pressure within the test chamber was 100 psia during firing, and temperature stabilized near 1400°F both within the chamber and on the electrically heated chamber walls.

The test was accomplished as planned, except for a leak which developed in the aft flange seal of the test chamber approximately 27 minutes into the second firing. It was necessary to stop the test, purge and cool with helium, and replace the seal. After this, normal operation was resumed.

Although redundant gas generators were provided to preclude interruption of the test in the event of a failure, only one of these was ever fired. The gas generator configuration was the same as that used for the in situ test discussed in Paragraph 3.4 and shown in Figure 56, except for the screens. Only two Haynes 25 screens - an aft bed retainer and a hemispherical injection diffuser - were used. After the ten hours of operation, the 14-18 mesh Shell 405 catalyst bed showed no visible damage or attrition but was mildly sintered. Precise pre-post weights were not taken because attrition measurement was not the purpose of this test.

### 3.3.3 Post Test Evaluation

The exposed specimens were removed from the test holder and subjected to the following evaluation sequence:

- a. Visual
- b. Weight Change
- c. Microhardness
- d. Guided Bend Test
- e. Metallographic

Concurrently, the control set of unexposed coupons was subjected to the same examination.

#### 3.3.3.1 Visual Examination

Table 7 and Figure 41 present the visual observations. With the exceptions of platinum-10% rhodium, moly-50 rhenium, tungsten, and TZM, all specimens tested exhibited a color change.

The coated/plated specimens all showed an effect due to poor adhesion. For example, the tungsten and platinum coatings peeled or blistered. This occurs when a poor bond exists. The environment provided conditions (thermal) which would weaken the bond and create the resultant effect. The metal beads noted in the R508C coated columbium specimen are a normal condition for the coating. These beads are silver, which is part of the coating.

#### 3.3.3.2 Weight Change

Most of the test specimens gained weight as a result of the exposure except for R508C silicide coated columbium, platinum-10% rhodium, tungsten coated Inconel 600, and rhodium plated Nichrome V. The R508C silicide coated columbium lost weight because the silver portion of the coating melted and dropped off in the form of spherical globules. Portions of the rhodium and platinum plated nichrome and tungsten coated Inconel 600 had blistered and flaked off. Of all the specimens tested, the platinum-10% rhodium and tungsten appeared least affected from a weight standpoint. Nichrome, moly-rhenium, TZM, and the aluminide coated specimen (Al-10 Si coated B-1900) were next. Table 8 gives the weight change of all specimens tested, normalized to the external surface area ( $\sim 21.3 \text{ cm}^2$ ).

**TABLE 7  
VISUAL OBSERVATIONS**

| Material             | Group No. 1 - One Hour Exposure |                                 | Group No. 2 - Six Hours Exposure |                                 | Group No. 3 - Ten Hours Exposure |                                 |
|----------------------|---------------------------------|---------------------------------|----------------------------------|---------------------------------|----------------------------------|---------------------------------|
|                      | Specimen No.                    |                                 | Specimen No.                     |                                 | Specimen No.                     |                                 |
| Haynes 25            | H5-1                            | Darkened                        | H5-2                             | Darkened                        | H5-3                             | Darkened                        |
| Haynes 188           | H8-1                            | Darkened                        | H8-2                             | Darkened                        | H8-3                             | Darkened                        |
| Inconel 718          | I8-1                            | Dark blue-black                 | I8-2                             | Dark blue-black                 | I8-3                             | Dark blue-black                 |
| Inconel 750          | IO-1                            | Darkened                        | IO-2                             | Darkened                        | IO-3                             | Darkened                        |
| Nichrome V           | N-1                             | Darkened (black)                | N-2                              | Darkened (black)                | N-3                              | Darkened (black)                |
| Pt-10 Rhodium        | PR-1                            | No change                       | PR-2                             | No change                       | PR-3                             | No change                       |
| Moly-50 Rhenium      | MR-1                            | No change                       | MR-2                             | No change                       | MR-3                             | No change                       |
| B-1900               | B-1                             | Darkened                        | B-2                              | Darkened                        | B-3                              | Darkened                        |
| GE 2541              | GE-1                            | Dark blue-black                 | GE-2                             | Dark blue-black                 | GE-3                             | Slight color darkening          |
| Tungsten             | W-1                             | No change                       | W-2                              | No change                       | W-3                              | No change                       |
| TZM                  | TM-1                            | No change                       | TM-2                             | No change                       | TM-3                             | No change                       |
| Inconel 600          | I6-1                            | Darkened                        | I6-2                             | Darkened                        | I6-3                             | Darkened                        |
| B-1900 Al-10 Si      | A-1                             | Dark blue-black                 | A-2                              | Dark gray                       | A-3                              | Dark gray                       |
| Cb 291 R512E         | S-1                             | Dark brown                      | S-2                              | Dark blue                       | S-3                              | Dark gray                       |
| Inconel 600/Tungsten | I6-5                            | No color change, coating peeled | I6-6                             | No color change, coating peeled | I6-7                             | No color change, coating peeled |
| Nichrome/Platinum    | NP-1                            | Blistered Plating-shiny         | NP-2                             | Blistered Plating-shiny         | NP-3                             | Darkened                        |
| Nichrome/Rhodium     | NR-1                            | No change                       | NR-2                             | Gray                            | NR-3                             | White film                      |
| Cb 291 with R508C    | R-2                             | No change                       | R-1                              | No change-metal beads           | R-3                              | No change-metal beads           |

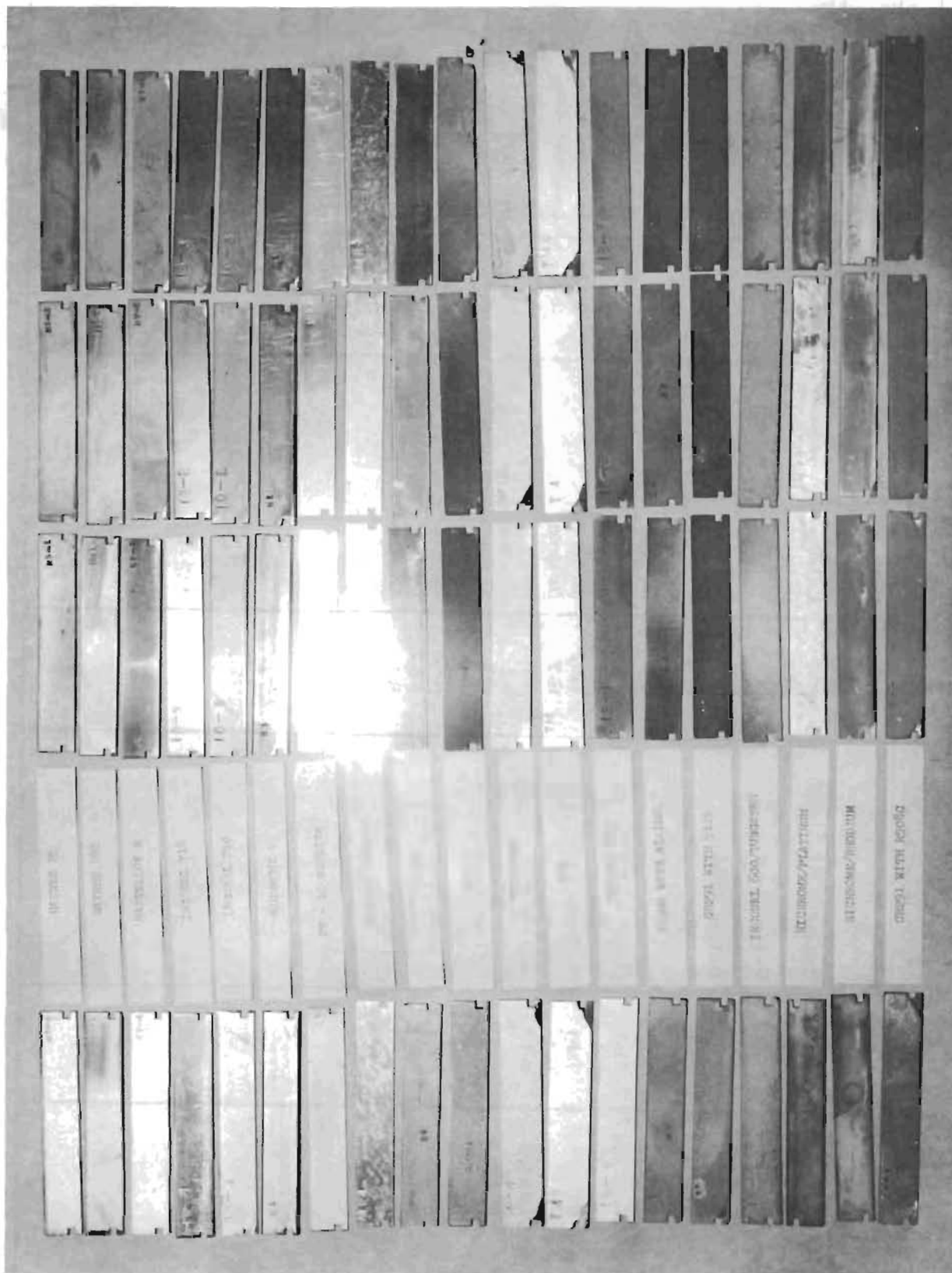


Figure 41. Sample coupons (control, 1-hour, 6-hour, and 10-hour exposure sets).

TABLE 8  
WEIGHT CHANGE

|                      | Weight Change - Milligram/cm <sup>2</sup> |         |          |
|----------------------|-------------------------------------------|---------|----------|
|                      | 1 Hour                                    | 6 Hours | 10 Hours |
| Haynes 25            | 0.367                                     | 0.632   | 0.816    |
| Haynes 188           | 0.261                                     | 0.427   | 0.568    |
| Hastelloy B          | -1.845                                    | 0.457   | 0.543    |
| Inconel 718          | 0.120                                     | 0.299   | 0.453    |
| Inconel 750          | 0.182                                     | 0.364   | 0.466    |
| Nichrome V           | 0.056                                     | 0.077   | 0.085    |
| Pt-10 Rhodium        | -0.014                                    | -0.014  | 0        |
| Moly-50 Rhenium      | 0.039                                     | 0.072   | 0.126    |
| B1900                | 0.271                                     | 0.480   | 0.653    |
| GE2541               | 5.250                                     | 11.538  | 14.912   |
| Tungsten             | 0.009                                     | 0.013   | 0.047    |
| TZM                  | 0.094                                     | 0.115   | 0.222    |
| Inconel 600          | 0.298                                     | 0.440   | 0.538    |
| B1900/Al-10 Si       | 0.027                                     | 0.040   | 0.053    |
| Cb 291/512E          | 0                                         | 0.085   | 0.196    |
| Inconel 600/Tungsten | -2.572                                    | -1.430  | -0.471   |
| Nichrome/Platinum    | 18.317                                    | 11.790  | -3.742   |
| Nichrome/Rhodium     | -1.461                                    | -5.246  | 1.794    |
| Cb 291/R508C         | -35.22                                    | -0.030  | -3.080   |

### 3.3.3.3 Bend Test

A guided bend test conducted per the requirements of ASTM-E-190 (specimen size waived) was performed on each coupon. Each was bent around a radius twice the sample thickness (~0.1 inch) through an angle of 180 degrees. This was done under ambient conditions except for tungsten, B-1900 and GE 2541. These three materials were tested at 500°F because their brittle nature does not permit bending at ambient temperature. The convex or tension surface of each bent specimen was examined for cracks and any other surface type defects. A summary of the data acquired appears in Table 9.

Candidate materials meeting all requirements of the bend test with no evidence of cracks or checking on the convex surface\* of either the control or exposed specimens were:

- a. Inconel 600/Tungsten
- b. Tungsten
- c. Platinum-10% Rhodium
- d. Nichrome V
- e. Nichrome/Rhodium
- f. Nichrome/Platinum

Candidate material specimens meeting the full 180-degree bend requirement with no evidence of cracks or checking of the control specimens, but whose exposed specimens exhibited checking were:

a. Hastelloy B - Figure 42 shows the observed checking on the convex surface of 10-hour exposure specimen HY-3. Detail A is a 32X magnification showing that the checking is a series of shallow cracks along the convex surface. Detail B increases the magnification to 200X indicating that the deepest "crack" is approximately 0.0023 inch.

b. Haynes 188 - Figure 43, Detail A, is a 32X record of the checked surface of the 10-hour exposure specimen (HB-3). Detail B expands the magnification to 200X showing that the depth of "cracking" is approximately 0.0017 inch. A distinct diffusion layer is also evident as a discoloration to a depth of 0.00066 inch.

c. Inconel 600 - Figure 44, Detail A, is a 32X record of the checked surface of 10-hour exposed specimen I6-3. Detail B expands the magnification to 200X showing that the depth of "cracking" is approximately 0.0013 inch. A 0.0006-inch deep discolored diffusion layer is present.

Control specimens of the following metals passed the 180-degree bend test with no evidence of cracks or checking, but 10-hour exposure specimens cracked or failed:

- a. Molybdenum-50% Rhenium
- b. TZM
- c. Haynes 25
- d. Inconel 718
- e. Inconel 750

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\* Except for expected cracking or checking of brittle coatings.

TABLE 9  
MATERIAL SELECTION TESTS  
LONG LIFE MONOPROPELLANT PROGRAM (Page 1 of 3)

| Material                   | Specimen Ident. | Condition   | Thickness (Inch) | Bend Radius (Inch) | Max. Load To Bend 180° (Pounds) | Failure   |                 | Remarks                                                                        |
|----------------------------|-----------------|-------------|------------------|--------------------|---------------------------------|-----------|-----------------|--------------------------------------------------------------------------------|
|                            |                 |             |                  |                    |                                 | Load (lb) | Angle (Degrees) |                                                                                |
| Inconel 600/<br>Tungsten   | I6-8            | Control     | 0.0728           | 0.150              | 100                             | —         | —               | Coating checked. ⚠                                                             |
|                            | I6-7            | 10 hr. exp. | 0.0710           | 0.150              | 98                              | —         | —               | Coating checked.                                                               |
|                            | I6-6            | 6 hr. exp.  | 0.0710           | 0.150              | 125                             | —         | —               | Coating checked.                                                               |
|                            | I6-5            | 1 hr. exp.  | 0.0731           | 0.150              | 120                             | —         | —               | Coating checked.                                                               |
| Tungsten                   | W-4             | Control     | 0.0934           | 0.125              | 1300                            | —         | —               | Tested at 500°F.                                                               |
|                            | W-3             | 10 hr. exp. | 0.0925           | 0.125              | 1400                            | —         | —               | No cracks or checking. ⚠                                                       |
|                            | W-2             | 6 hr. exp.  | 0.0930           | 0.187              | 1250                            | —         | —               | Broke away from bend.                                                          |
|                            | W-1             | 1 hr. exp.  | 0.0930           | 0.187              | 1258                            | —         | —               | No cracks or checking.                                                         |
|                            | (W-3 1/2)       | 10 hr. exp. | 0.0925           | 0.125              | 1300                            | —         | —               | No cracks or checking.<br>1/2 of above W-3 retested,<br>no cracks or checking. |
| Platinum-<br>10% Rhodium   | PR-4            | Control     | 0.0282           | 0.0625             | 35                              | —         | —               | Cracked ⚠                                                                      |
|                            | PR-3            | 10 hr. exp. | 0.0316           | 0.064              | 30                              | —         | —               | No cracks or checking.                                                         |
|                            | PR-2            | 6 hr. exp.  | 0.0286           | 0.064              | 22                              | —         | —               | No cracks or checking.                                                         |
|                            | PR-1            | 1 hr. exp.  | 0.0319           | 0.064              | 38                              | —         | —               | No cracks or checking.                                                         |
| Nichrome V                 | N-4             | Control     | 0.0929           | 0.192              | 179                             | —         | —               | No cracks or checking. ⚠                                                       |
|                            | N-3             | 10 hr. exp. | 0.0926           | 0.192              | 191                             | —         | —               | No cracks or checking.                                                         |
|                            | N-2             | 6 hr. exp.  | 0.0920           | 0.192              | 196                             | —         | —               | No cracks or checking.                                                         |
|                            | N-1             | 1 hr. exp.  | 0.0924           | 0.192              | 206                             | —         | —               | No cracks or checking.                                                         |
| Hastelloy B                | HY-4            | Control     | 0.0936           | 0.192              | 276                             | —         | —               | No cracks or checking. ⚠                                                       |
|                            | HY-3            | 10 hr. exp. | 0.0948           | 0.192              | 284                             | —         | —               | Surface checked.                                                               |
|                            | HY-2            | 6 hr. exp.  | 0.0940           | 0.192              | 275                             | —         | —               | Surface checked.                                                               |
|                            | HY-1            | 1 hr. exp.  | 0.0940           | 0.192              | 375                             | —         | —               | Surface checked.                                                               |
| Molybdenum-<br>50% Rhenium | MR-4            | Control     | 0.0293           | 0.0625             | 72                              | —         | —               | No cracks or checking. ⚠                                                       |
|                            | MR-3            | 10 hr. exp. | 0.0295           | 0.0625             | 61                              | —         | —               | Cracked.                                                                       |
|                            | MR-2            | 6 hr. exp.  | 0.0290           | 0.064              | 65                              | —         | —               | Surface checked.                                                               |
|                            | MR-1            | 1 hr. exp.  | 0.0290           | 0.064              | 70                              | —         | —               | Surface checked.                                                               |
| TZM                        | TM-4            | Control     | 0.0935           | 0.192              | 350                             | —         | —               | No cracks or checking.                                                         |
|                            | TM-3            | 10 hr. exp. | 0.0938           | 0.192              | —                               | 100       | 2               | Failed.                                                                        |
|                            | TM-2            | 6 hr. exp.  | 0.0938           | 0.192              | —                               | 128       | 2               | Failed.                                                                        |
|                            | TM-1            | 1 hr. exp.  | 0.0924           | 0.192              | —                               | 114       | 2               | Failed.                                                                        |
| Nichrome/<br>Rhodium       | NR-4            | Control     | 0.0938           | 0.192              | 191                             | —         | —               | No cracks or checking. ⚠                                                       |
|                            | NR-3            | 10 hr. exp. | 0.0934           | 0.192              | 194                             | —         | —               | Plating checked.                                                               |
|                            | NR-2            | 6 hr. exp.  | 0.0920           | 0.192              | 204                             | —         | —               | No checking.                                                                   |
|                            | NR-1            | 1 hr. exp.  | 0.0926           | 0.192              | 200                             | —         | —               | Plate checked and peeled.                                                      |

TABLE 9 (Page 2 of 3)

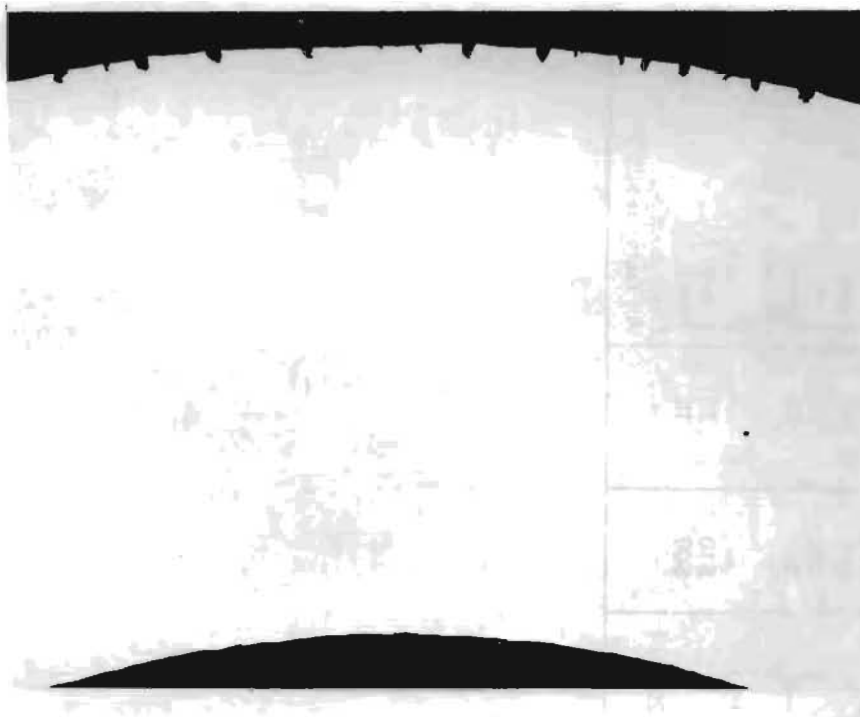
| Material              | Specimen Ident. | Condition   | Thickness (Inch) | Bend Radius (Inch) | Max. Load To Bend 180° (Pounds) | Failure   |                 | Remarks                     |
|-----------------------|-----------------|-------------|------------------|--------------------|---------------------------------|-----------|-----------------|-----------------------------|
|                       |                 |             |                  |                    |                                 | Load (lb) | Angle (Degrees) |                             |
| Nichrome/<br>Platinum | NP-4            | Control     | 0.0926           | 0.192              | 200                             | —         | —               | No cracks or checking. ⚠    |
|                       | NP-3            | 10 hr. exp. | 0.0925           | 0.192              | 192                             | —         | —               | Coating peeled.             |
|                       | NP-2            | 6 hr. exp.  | 0.0940           | 0.192              | 204                             | —         | —               | Coating peeled off.         |
|                       | NP-1            | 1 hr. exp.  | 0.0940           | 0.192              | 200                             | —         | —               | Coating peeled off.         |
| Haynes 188            | H8-4            | Control     | 0.0900           | 0.192              | 283                             | —         | —               | No cracks or checking. ⚠    |
|                       | H8-3            | 10 hr. exp. | 0.0892           | 0.192              | 300                             | —         | —               | Surface checked.            |
|                       | H8-2            | 6 hr. exp.  | 0.0880           | 0.192              | 304                             | —         | —               | Surface checked.            |
|                       | H8-1            | 1 hr. exp.  | 0.0896           | 0.192              | 309                             | —         | —               | Surface checked.            |
| Inconel 600           | I6-4            | Control     | 0.0643           | 0.125              | 203                             | —         | —               | No cracks or checking. ⚠    |
|                       | I6-3            | 10 hr. exp. | 0.0643           | 0.125              | 162                             | —         | —               | Surface checked.            |
|                       | I6-2            | 6 hr. exp.  | 0.0640           | 0.150              | 125                             | —         | —               | Surface checked.            |
|                       | I6-1            | 1 hr. exp.  | 0.0642           | 0.150              | 135                             | —         | —               | No cracks or checking.      |
| Cb 291/<br>R508C      | R-4             | Control     | 0.0911           | 0.192              | 145                             | —         | —               | Surface checked.            |
|                       | R-3             | 10 hr. exp. | 0.0965           | 0.192              | —                               | 148       | 45              | Failed.                     |
|                       | R-1             | 6 hr. exp.  | 0.0946           | 0.192              | —                               | 104       | 25              | Failed.                     |
|                       | R-2             | 1 hr. exp.  | 0.0892           | 0.192              | 135                             | —         | —               | Coating cracked and peeled. |
| Cb 291/<br>R512E      | S-4             | Control     | 0.0964           | 0.096              | 200                             | —         | —               | Surface checked.            |
|                       | S-3             | 10 hr. exp. | 0.0934           | 0.096              | —                               | 45        | 2               | Failed.                     |
|                       | S-2             | 6 hr. exp.  | 0.0940           | 0.192              | 167                             | —         | —               | Surface checked.            |
|                       | S-1             | 1 hr. exp.  | 0.0890           | 0.192              | 132                             | —         | —               | Surface checked.            |
| Haynes 25             | H5-4            | Control     | 0.0894           | 0.192              | 296                             | —         | —               | No cracks or checking.      |
|                       | H5-3            | 10 hr. exp. | 0.0895           | 0.192              | —                               | 307       | 70              | Failed.                     |
|                       | H5-2            | 6 hr. exp.  | 0.0896           | 0.192              | 353                             | —         | —               | Surface checked.            |
|                       | H5-1            | 1 hr. exp.  | 0.0896           | 0.192              | 350                             | —         | —               | Surface checked.            |
| Inconel 718           | I8-4            | Control     | 0.0914           | 0.192              | 243                             | —         | —               | No cracks or checking.      |
|                       | I8-3            | 10 hr. exp. | 0.0913           | 0.192              | —                               | 441       | 40              | Failed.                     |
|                       | I8-2            | 6 hr. exp.  | 0.0920           | 0.192              | 539                             | —         | —               | Surface checked.            |
|                       | I8-1            | 1 hr. exp.  | 0.0920           | 0.192              | 481                             | —         | —               | No cracks or checking.      |
| Inconel 750           | I0-4            | Control     | 0.0615           | 0.125              | 154                             | —         | —               | No cracks or checking.      |
|                       | I0-3            | 10 hr. exp. | 0.0615           | 0.125              | 250                             | —         | —               | Cracked.                    |
|                       | I0-2            | 6 hr. exp.  | 0.0620           | 0.125              | 215                             | —         | —               | Surface checked.            |
|                       | I0-1            | 1 hr. exp.  | 0.0620           | 0.125              | 220                             | —         | —               | Surface checked.            |
| B 1900/<br>Al-10 Si   | A-4             | Control     | 0.0648           | 0.125              | —                               | 157       | 10              | Failed.                     |
|                       | A-3             | 10 hr. exp. | 0.0612           | 0.125              | —                               | 127       | 5               | Failed.                     |
|                       | A-2             | 6 hr. exp.  | 0.0590           | 0.150              | —                               | 110       | 20              | Failed.                     |
|                       | A-1             | 1 hr. exp.  | 0.0695           | 0.150              | —                               | 150       | 20              | Failed.                     |

TABLE 9 (Page 3 of 3)

| Material | Specimen Ident. | Condition   | Thickness (Inch) | Bend Radius (Inch) | Max. Load To Bend 180° (Pounds) | Failure   |                 | Remarks                                                                                                                                                     |
|----------|-----------------|-------------|------------------|--------------------|---------------------------------|-----------|-----------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------|
|          |                 |             |                  |                    |                                 | Load (lb) | Angle (Degrees) |                                                                                                                                                             |
| B 1900   | B-4             | Control     | 0.0650           | 0.125              | —                               | 400       | 15              | Failed.<br>Failed.<br>Failed.<br>Failed.<br>All Tested at 500°F.         |
|          | B-3             | 10 hr. exp. | 0.0608           | 0.125              | —                               | 420       | 15              |                                                                                                                                                             |
|          | B-2             | 6 hr. exp.  | 0.0640           | 0.187              | —                               | 375       | 15              |                                                                                                                                                             |
|          | B-1             | 1 hr. exp.  | 0.0615           | 0.187              | —                               | 361       | 15              |                                                                                                                                                             |
| GE 2541  | GE-4            | Control     | 0.0918           | 0.125              | 810                             | —         | —               | Cracked.<br>Failed.<br>Failed.<br>Deep checking.<br>All Tested at 500°F  |
|          | GE-3            | 10 hr. exp. | 0.0950           | 0.125              | —                               | 510       | 10              |                                                                                                                                                             |
|          | GE-2            | 6 hr. exp.  | 0.0940           | 0.187              | —                               | 500       | 10              |                                                                                                                                                             |
|          | GE-1            | 1 hr. exp.  | 0.0928           | 0.187              | 725                             | —         | —               |                                                                                                                                                             |

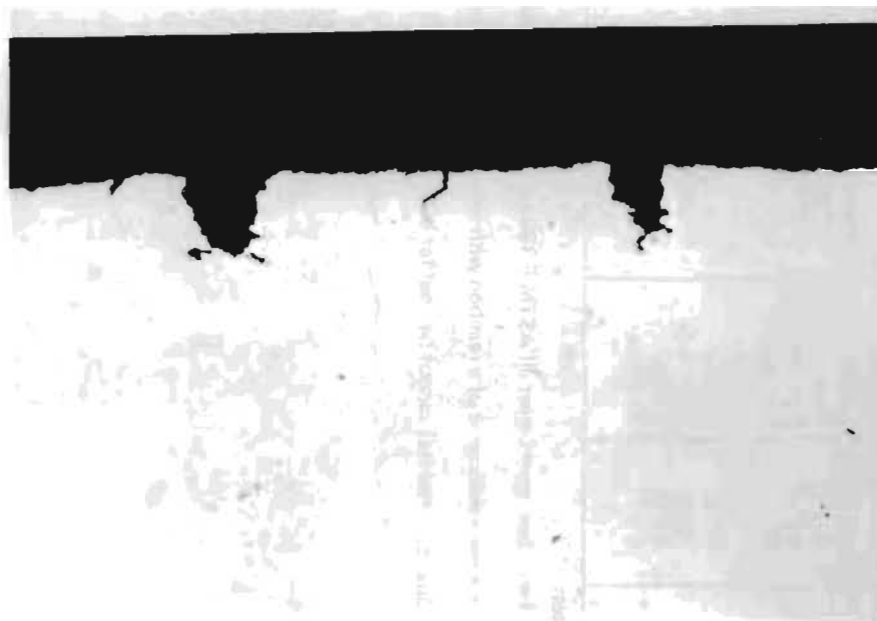
Notes:

-  Bend Test. Specimens meet all ASTM-E-190 bend test requirements.
-  A surface layer indicative of a reaction with the environment is evident at 500X.
-  Inconclusive. Bend test cannot be used for evaluation.



Detail A  
Hastelloy B  
post bend test,  
radius inspection.

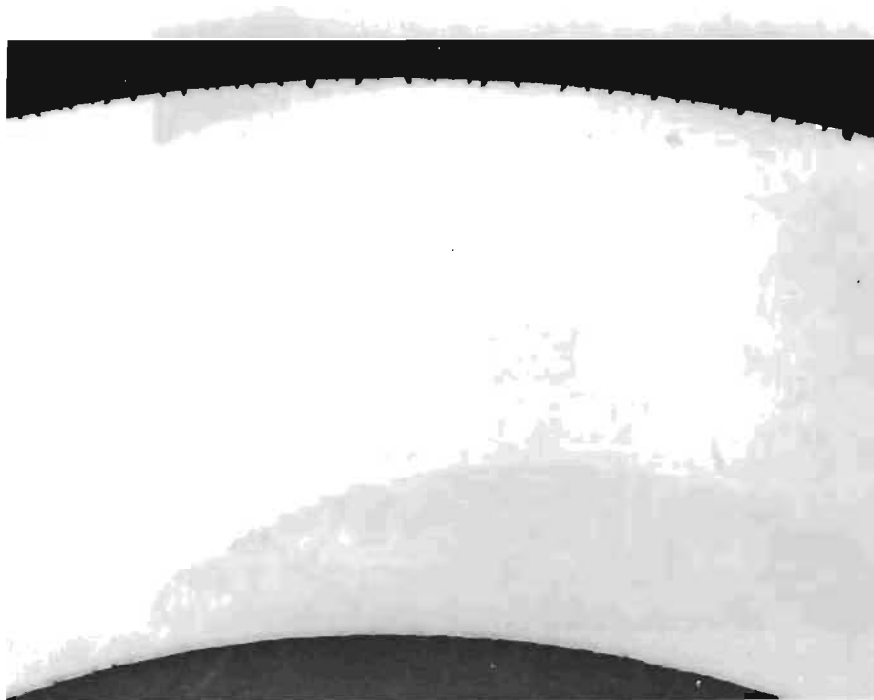
MAG: 32X



Detail B  
Hastelloy B  
post bend test  
convex surface.

MAG: 200X

Figure 42. Post bend test investigation of Hastelloy B exposed to hydrazine decomposition products for 10 hours at 1400°F.



Detail A  
Haynes 188  
post bend test  
radius inspection.

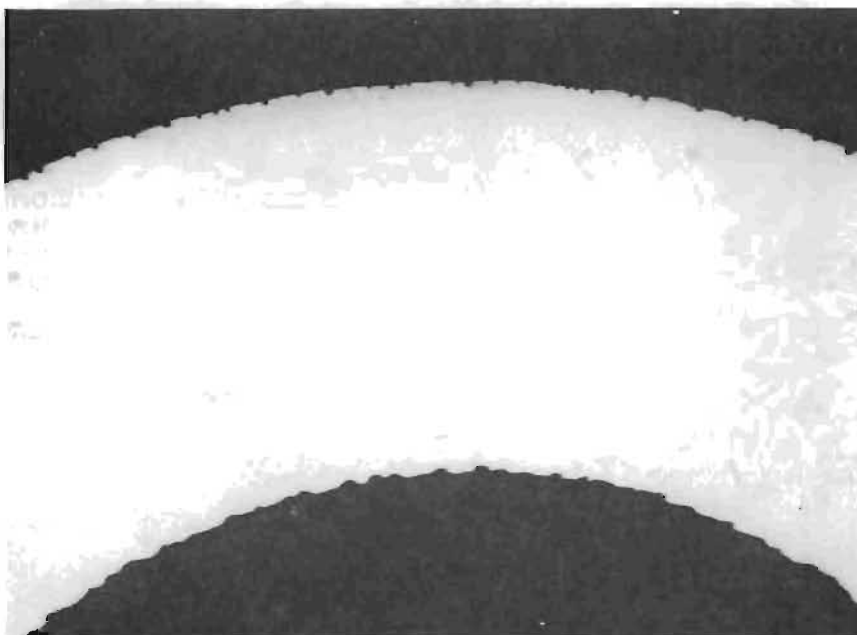
MAG: 32X



Detail B  
Haynes 188  
post bend test  
convex surface.

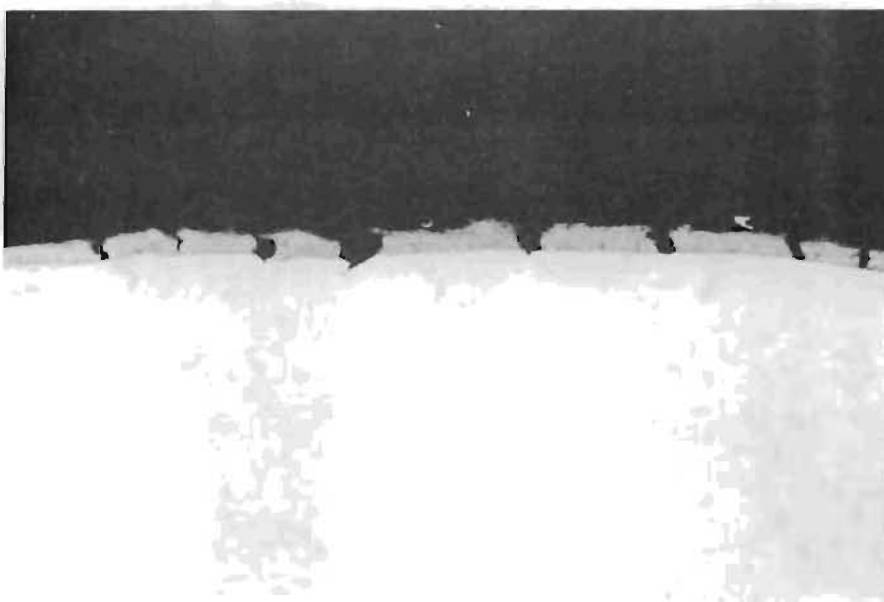
MAG: 200X

Figure 43. Post bend test investigation of Haynes 188 exposed to hydrazine decomposition products for 10 hours at 1400° F.



Detail A  
Inconel 600  
post bend test  
radius  
inspection.

MAG: 32X



Detail B  
Inconel 600  
post bend test  
convex surface.

MAG: 200X

Figure 44. Post bend test investigation of Inconel 600 exposed to hydrazine decomposition products for 10 hours at 1400°F.

Both coated columbium control specimens exhibited checking after bending 180 degrees, and 10-hour exposure specimens of these materials failed completely.

Candidate materials which could not be evaluated by a bend test since both the control and the exposed specimens failed to bend 180 degrees were:

- a. B-1900/Al-10 Si
- b. B-1900
- c. GE 2541

These materials were brittle to the extent that they could not be bent, even at 500°F.

#### 3.3.3.4 Microscopic Examination

The metallographic comparison of cross sections of the unexposed samples to the exposed ones was done to detect changes in the microstructural features of the material under the surface. Contamination depth was obtained by measuring depths of layers developed on the exposed samples under 200X magnification. The effect of the exposure on the base metal was further evaluated by utilizing microhardness numbers (Knoop 100 grams) to determine if there was a change in the material's resistance to deformation after exposure.

Materials exhibiting no evidence of contamination by this means were:

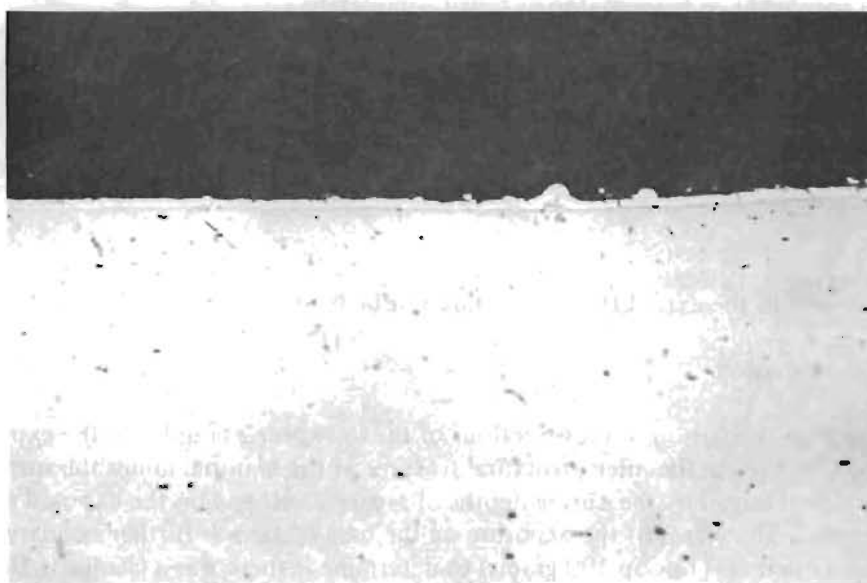
- a. Inconel 600/tungsten
- b. Tungsten
- c. Platinum-10% rhodium
- d. Nichrome V
- e. Hastelloy B
- f. TZM
- g. CB 291/R508C
- h. CB 291/R512E
- i. B-1900/Al-10 Si

Microstructural evaluation of the coated nichrome samples (nichrome-rhodium and nichrome-platinum), revealed a definite change in the coating and diffusion zones as a result of the exposure (Figures 45 and 46). No increase/decrease in Knoop (100 gram) hardness members was observed, however.

Reaction with the exposure environment was clearly discernable in the photomicrographs of Haynes 188, Inconel 600, Haynes 25, Inconel 718, Inconel 750, B-1900 and GE2541. Figure 47 shows the depth of the visible contamination layers (corrosion) in these materials as a function of exposure time. Table 10 tabulates these data and indicates average contamination rates. Note the generally increasing rates at the end of the test. This is indicative of a diffusion situation in which little or no surface passivation occurs.

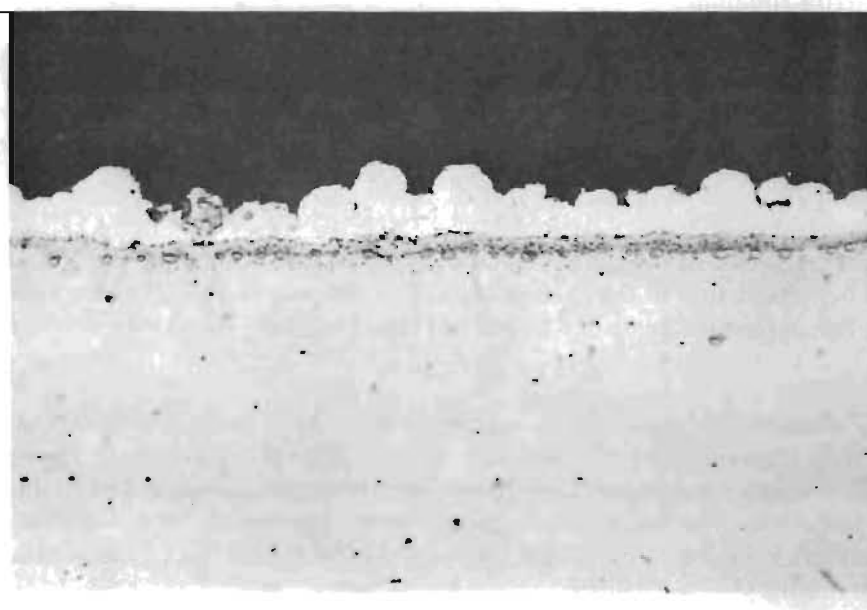
The Haynes 188 sections are compared in Figure 48. A diffusion-contamination layer 0.0007 inch deep is visible in the 10-hour exposure sample shown.

Figure 49 shows the 0.0006 inch deep contamination layer in Inconel 600. A similar layer 0.00087 inch deep was seen in Haynes 25 (Figure 50).



Detail A  
Nichrome/Rhodium  
Control  
Specimen NR-4  
Nominal material  
cross section.  
No indication of  
any discoloration  
at the Rhodium/  
Nichrome interface.

MAG: 500X



Detail B  
Nichrome/Rhodium  
10-hour exposure  
Specimen NR-3  
Coating build-up  
and a definite  
diffusion zone  
at the Nichrome/Rhodium  
interface is evident.

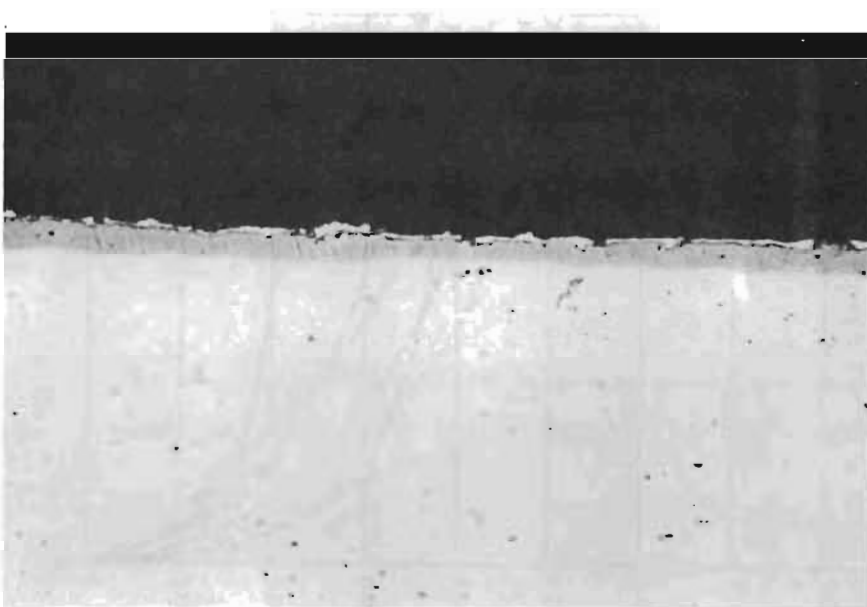
MAG: 500X

Figure 45. Nichrome/Rhodium - cross section comparison of control specimen and specimen exposed to hydrazine decomposition products for 10 hours at 1400°F.



Detail A  
Nichrome/Platinum  
Control  
Specimen NP-4.

MAG: 500X



Detail B  
Nichrome/Platinum  
10-hour exposure  
Specimen NP-3.

MAG: 500X

Figure 46. Nichrome/Platinum - cross section comparison of control specimen and specimen exposed to hydrazine decomposition products for 10 hours at 1400° F.

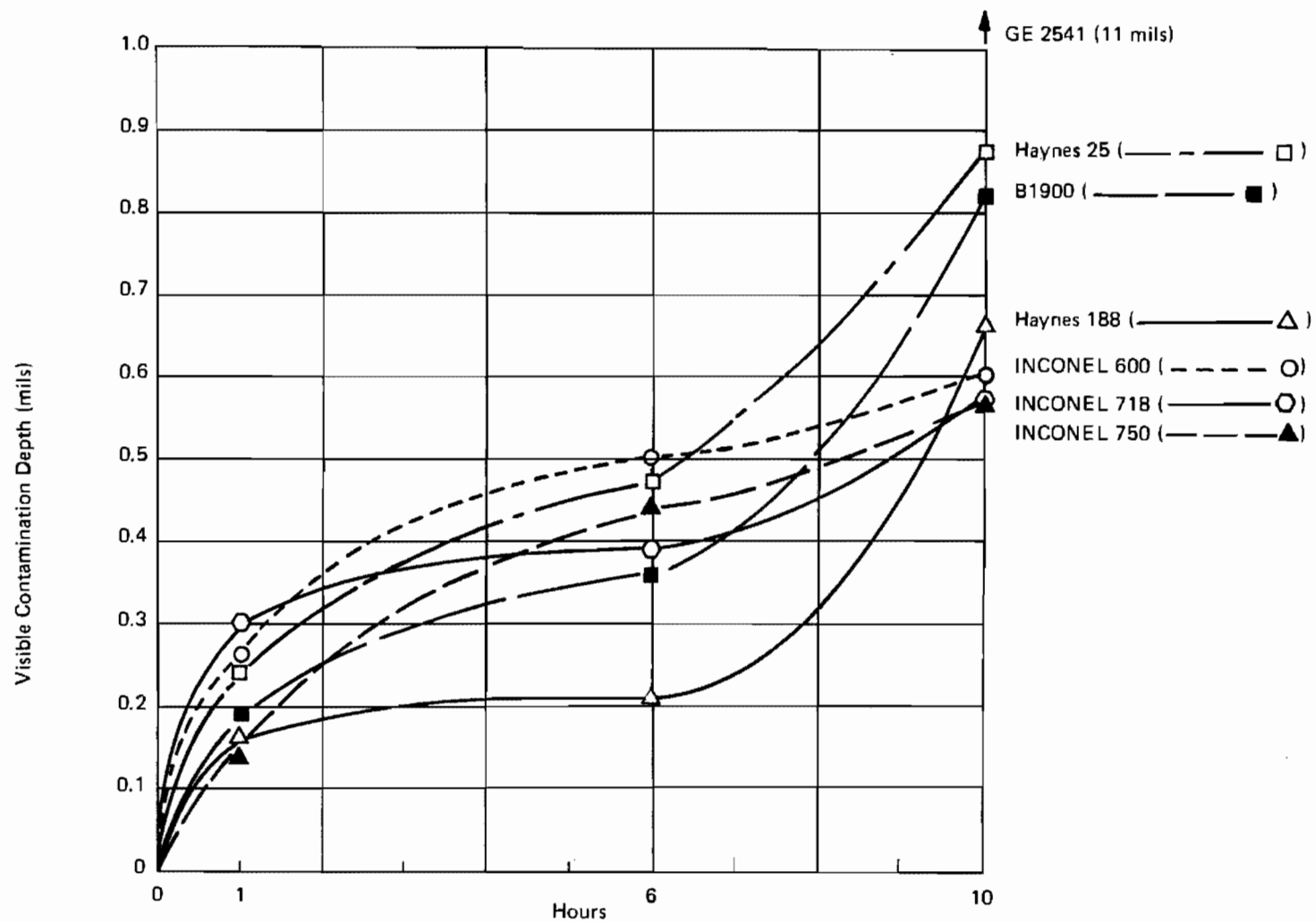


Figure 47. Corrosion rates of selected candidate materials.

TABLE 10  
DEPTH OF CORROSION AND CORROSION RATES OF  
CANDIDATE MATERIALS EXHIBITING A DIFFUSION/CONTAMINATION LAYER



| Material                   | 1-Hour Exposure<br>Depth and Rate (Mils<br>and/or Mils per hour<br>are the same) | 6-Hour Exposure |                     | 10-Hour Exposure |                     |
|----------------------------|----------------------------------------------------------------------------------|-----------------|---------------------|------------------|---------------------|
|                            |                                                                                  | Depth<br>(Mils) | Rate<br>(Mils/hour) | Depth<br>(Mils)  | Rate<br>(Mils/hour) |
| Haynes 188                 | 0.16                                                                             | 0.21            | 0.035               | 0.656            | 0.0656              |
| Inconel 600                | 0.26                                                                             | 0.50            | 0.084               | 0.60             | 0.060               |
| Haynes 25                  | 0.24                                                                             | 0.47            | 0.08                | 0.869            | 0.087               |
| Inconel 718                | 0.30                                                                             | 0.39            | 0.065               | 0.57             | 0.057               |
| Inconel 750                | 0.14                                                                             | 0.44            | 0.07                | 0.57             | 0.057               |
| B1900                      | 0.19                                                                             | 0.36            | 0.06                | 0.82             | 0.082               |
| GE 2541                    | 3.77                                                                             | 7.50            | 1.25                | 11.0             | 1.1                 |
| Molybdenum-<br>50% Rhenium | None<br>Visible                                                                  | None<br>Visible | —                   | 0.19             | 0.019               |

Notes:



Diffusion/contamination layer depth recorded is the maximum depth observed at 200X.



Diffusion/contamination layer is intermittent. See Figure 51.



Detail A  
Haynes 188  
Control Specimen

MAG: 200X



Detail B  
Haynes 188  
10-hour exposure  
Specimen.

MAG: 200X

Figure 48. Haynes 188 - cross section comparison of control specimen and specimen exposed to hydrazine decomposition products for 10 hours at 1400°F.



Detail A  
Inconel 600  
Control  
Specimen

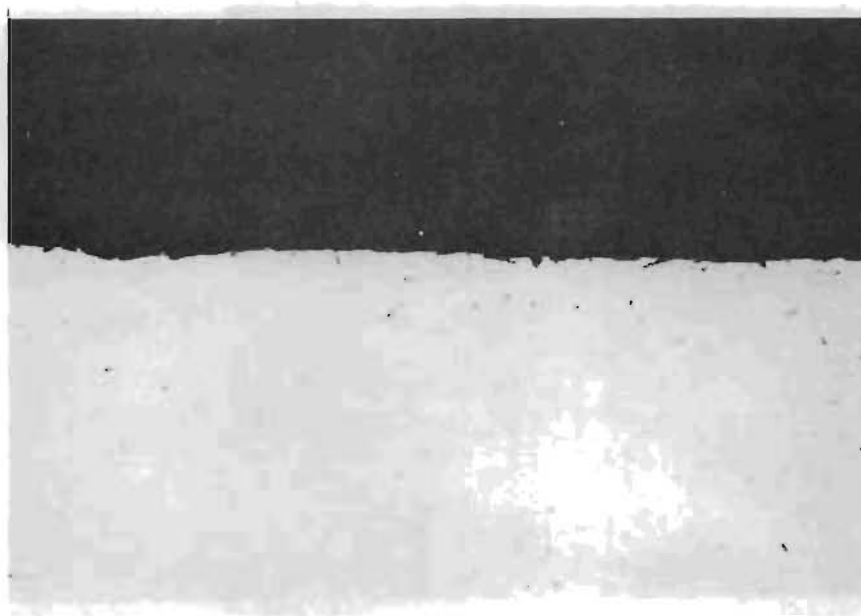
MAG: 200X



Detail B  
Inconel 600  
10-hour exposure  
Specimen  
(diffusion-contamination  
layer 0.0006 inch deep).

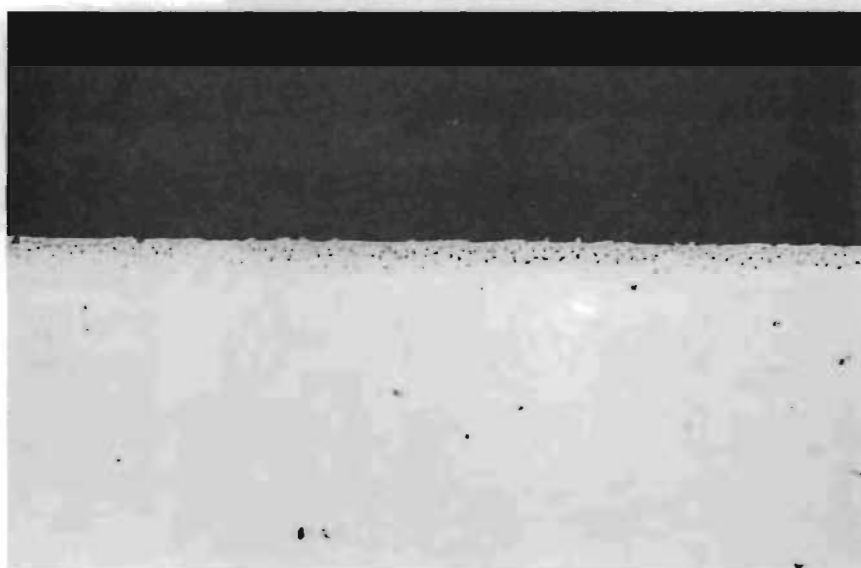
MAG: 200X

Figure 49. Inconel 600 - cross section comparison of control specimen and specimens exposed to hydrazine decomposition products for 10 hours at 1400° F.



Detail A  
Haynes 25  
Control Specimen

MAG: 200X



Detail B  
Haynes 25  
10-hour exposure  
specimen

(visible diffusion -  
contamination layer  
is 0.00087 inch deep).

MAG: 200X

Figure 50. Haynes Alloy No. 25 - cross section comparison of control specimen and specimen exposed to hydrazine decomposition products for 10 hours at 1400°F.

Inconel 718 and Inconel 750 both developed 0.00056 inch contamination layers, as shown in Figures 51 and 52.

A similar 0.0005 inch thick layer developed on the B-1900 (Figure 53). The surface features on the B-1900 control sample are normal for a casting material of this type and are not indicative of contamination.

GE 2541 was the poorest of the materials tested, showing a contamination layer an order of magnitude deeper (11 mils) than the worst of the others. Figure 54 shows this pronounced reaction to the 10-hour exposure.

The molybdenum-50% rhenium samples were only mildly affected. Figure 55 shows the 0.00019 inch layer barely visible on this material after 10 hours exposure. No layer was visible in the 1-hour and 6-hour samples.

#### 3.3.3.5 Microhardness Test

All specimens were evaluated for hardness change using Knoop (500 gram) microhardness. The hardness was measured at points 0.002 and 0.007 inch from both edges and in the center of the sectioned coupon. This hardness data is detailed in Table 11.

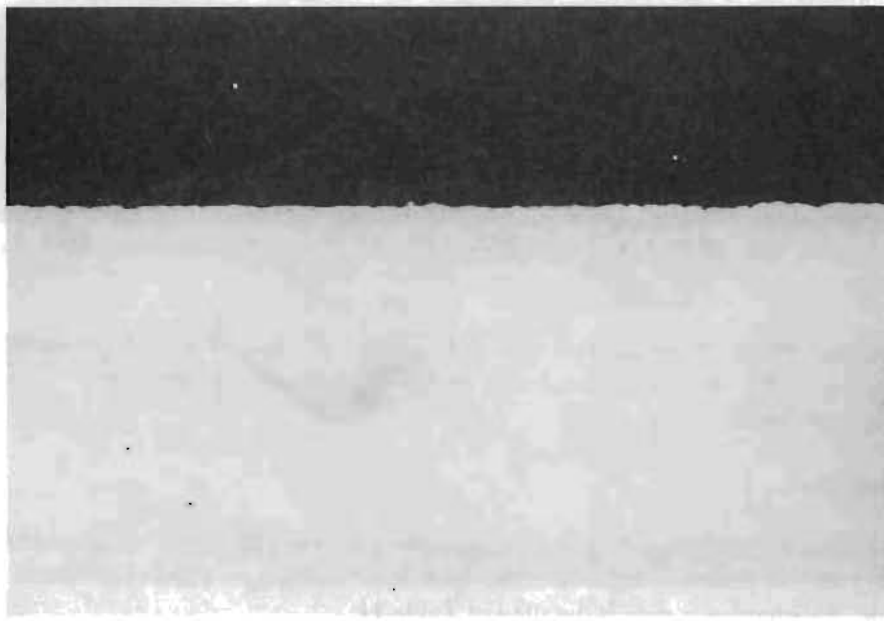
Materials which exhibited a large increase in hardness, indicating significant contamination, were Haynes 25, Inconel 718, Inconel 750, B-1900, and GE 2541. This is consistent with the visible contamination layers discussed in Paragraph 3.3.3.4, except for the absence of any appreciable increase in measured hardness of Haynes 188 and Inconel 600.

To reconcile this deviation, a Knoop 100 gram microhardness test was performed on all the materials with significant visible contamination layers. Measurements were made in the diffusion layers, 0.005 inch in from them, and at the center of the coupon section. These data are given in Table 12. All these materials showed the expected increased hardness in the contamination layer. Inconel 600 showed only a marginal increase, however. The very low hardness numbers obtained at one edge of the B-1900 were probably due to surface defects in the cast material.

The coated columbium showed a slight increase in hardness, indicating some contamination. The Inconel 600/tungsten, platinum-10% rhodium, and Inconel 600 actually showed a decrease in hardness. This is indicative of some degree of annealing, but the precise mechanism is questionable because of the relatively low (1400°F) exposure temperature. None of the other materials exhibited any appreciable change in hardness.

#### 3.3.4 Summary of Dynamic Exposure Test Results

Nichrome V passed all required tests except the initial visual inspection relative to surface discoloration/oxidation. The surface of the exposed nichrome specimens did exhibit a definite discoloration, but investigations of cross sections at 200X indicated no discernable depth to this layer. Further, these 200X investigations also indicated that there is no appreciable difference between the control specimen and the exposed specimens microstructurally. The ease of fabrication and relatively reasonable cost make Nichrome V an excellent material.



Detail A  
Inconel 718  
Control specimen

MAG: 200X



Detail B  
Inconel 718  
10-hour exposure  
specimen

(visible diffusion -  
contamination layer  
0.00056 inch deep).

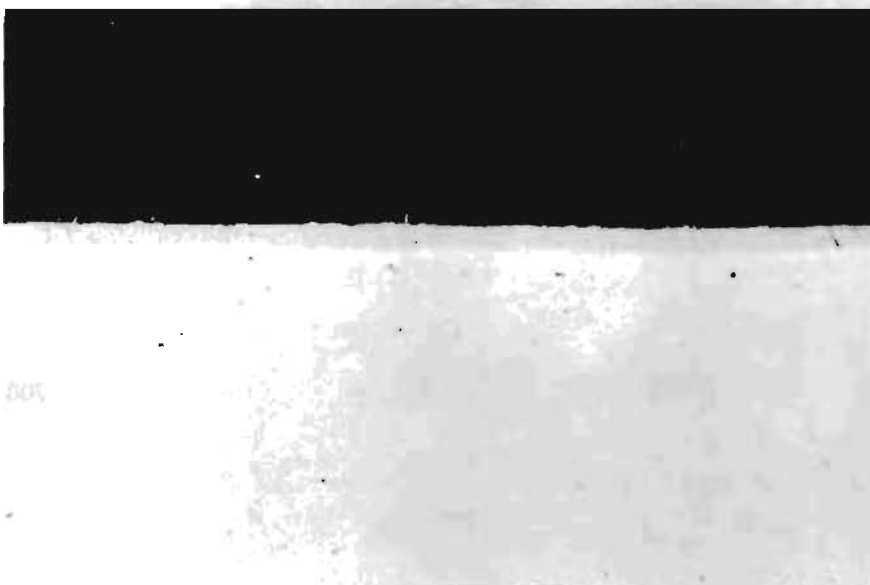
MAG: 200X

Figure 51. Inconel 718 - cross section comparison of control specimen and specimen exposed to hydrazine decomposition products for 10 hours at 1400°F.



Detail A  
Inconel 750  
Control Specimen

MAG: 200X

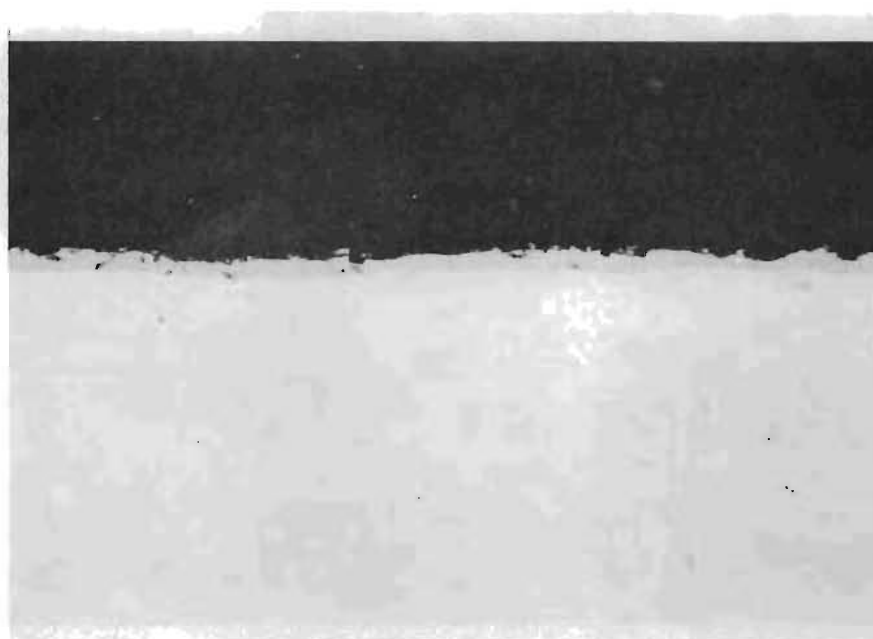


Detail B  
Inconel 750  
10-hour exposure  
specimen

(Visible diffusion-  
contamination  
layer is 0.00056  
inch deep)

MAG: 200X

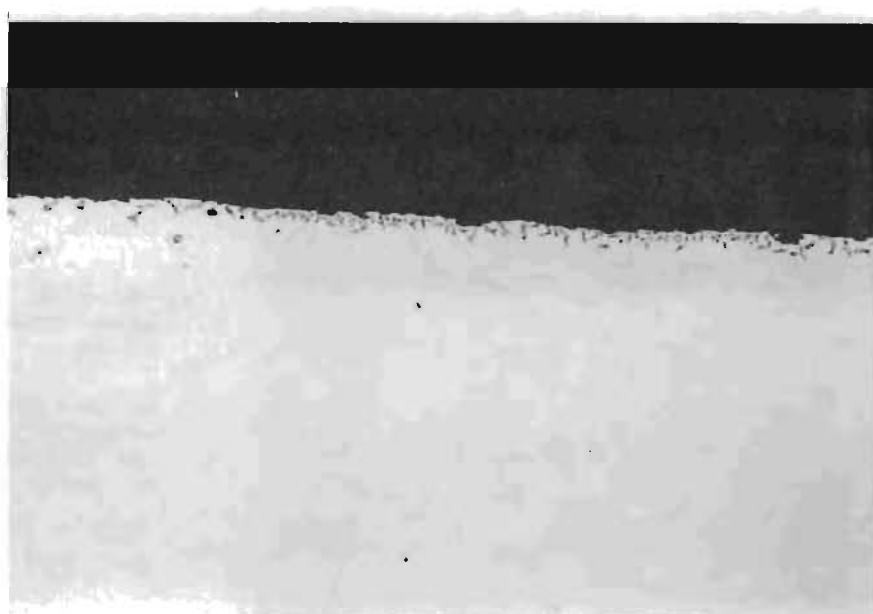
Figure 52. Inconel 750 - Cross section comparison of control specimen and specimen exposed to hydrazine decomposition products for 10 hours at 1400°F.



Detail A

B 1900  
Control specimen

MAG: 200X



Detail B

B 1900 10-hour  
exposure specimen

(Visible diffusion-  
contamination layer  
is 0.0005 inch deep)

MAG: 200X

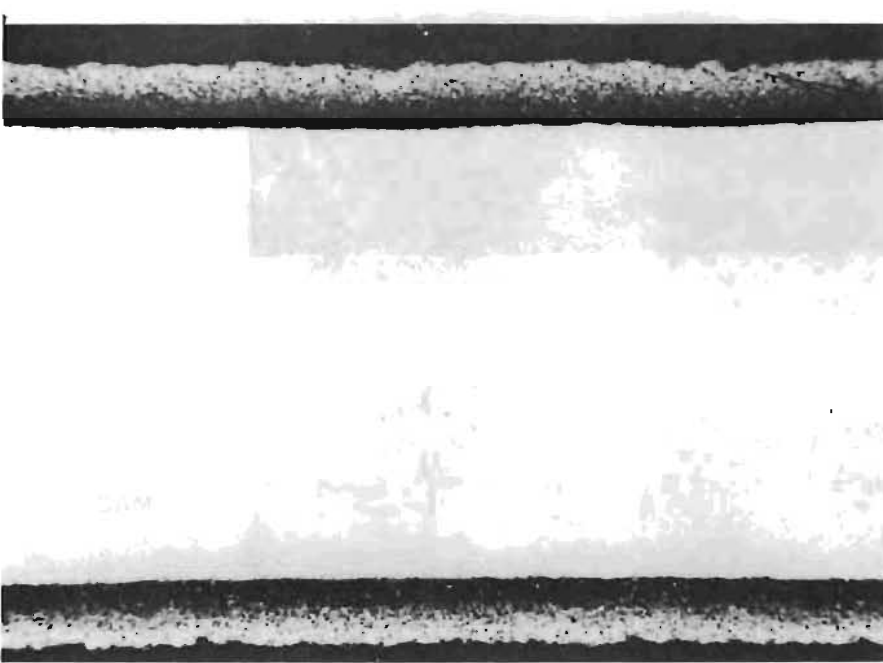
Figure 53. B 1900 - Cross section comparison of control specimen and specimen exposed to hydrazine decomposition for 10 hours at 1400°F.



Detail A

GE 2541  
Control specimen

MAG: 32X



Detail B

GE 2541 10-hour  
exposure specimen

(Visible diffusion-  
contamination layer  
0.011 inch deep)

MAG: 32X

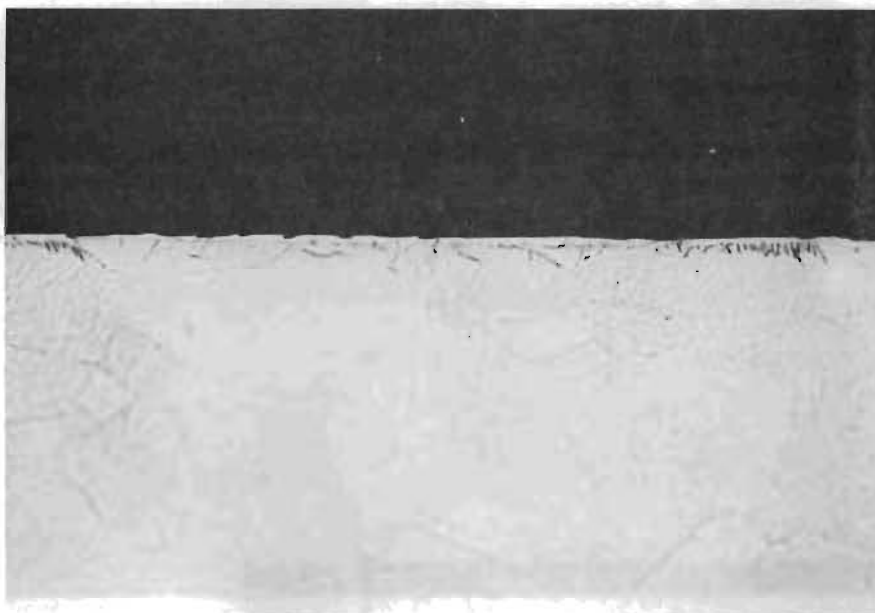
Figure 54. GE 2541 - Cross section comparison of control specimen and specimen exposed to hydrazine decomposition products for 10 hours at 1400°F.



Detail A

Molybdenum - 50%  
Rhenium  
Control specimen

MAG: 500X



Detail B

Molybdenum - 50%  
Rhenium  
10-hour exposure  
specimen

(Surface layer  
0.00019 inch  
deep)

MAG: 500X

Figure 55. Molybdenum - 50% Rhenium - Cross section comparison of control specimen and specimen exposed to hydrazine decomposition products for 10 hours at 1400°F.

**TABLE 11**  
**KNOOP MICROHARDNESS - 500 GRAM LOAD (Page 1 of 3)**

| Specimen                         | Distance From Edge |           | Center | Distance From Other Edge |           |
|----------------------------------|--------------------|-----------|--------|--------------------------|-----------|
|                                  | 0.002 In.          | 0.007 In. |        | 0.007 In.                | 0.002 In. |
| Haynes 25                        |                    |           |        |                          |           |
| Control                          | 293                | 306       | 307    | 317                      | 293       |
| 1 hr.                            | 287                | 286       | 312    | 306                      | 277       |
| 6 hr.                            | 277                | 307       | 312    | 306                      | 315       |
| 10 hr.                           | 385                | 325       | 317    | 344                      | 400       |
| Haynes 188                       |                    |           |        |                          |           |
| Control                          | 257                | 293       | 267    | 290                      | 267       |
| 1 hr.                            | 257                | 293       | 286    | 286                      | 267       |
| 6 hr.                            | 266                | 271       | 286    | 302                      | 286       |
| 10 hr.                           | 304                | 304       | 312    | 296                      | 304       |
| Hastelloy B                      |                    |           |        |                          |           |
| Control                          | 233                | 246       | 224    | 264                      | 219       |
| 1 hr.                            | 233                | 253       | 267    | 268                      | 234       |
| 6 hr.                            | 244                | 247       | 255    | 246                      | 246       |
| 10 hr.                           | 244                | 253       | 246    | 264                      | 210       |
| Inconel 718                      |                    |           |        |                          |           |
| Control                          | 215                | 257       | 236    | 258                      | 206       |
| 1 hr.                            | 406                | 436       | 414    | 415                      | 406       |
| 6 hr.                            | 414                | 446       | 414    | 446                      | 426       |
| 10 hr.                           | 390                | 428       | 390    | 408                      | 409       |
| Inconel 750                      |                    |           |        |                          |           |
| Control                          | 203                | 223       | 199    | 213                      | 193       |
| 1 hr.                            | 357                | 367       | 367    | 363                      | 337       |
| 6 hr.                            | 374                | 375       | 375    | 390                      | 378       |
| 10 hr.                           | 327                | 358       | 332    | 374                      | 348       |
| Inconel 600                      |                    |           |        |                          |           |
| Control                          | 272                | 220       | 188    | 200                      | 275       |
| 1 hr.                            | 202                | 184       | 172    | 192                      | 206       |
| 6 hr.                            | 207                | 182       | 177    | 181                      | 212       |
| 10 hr.                           | 217                | 188       | 178    | 185                      | 224       |
| Aluminide,<br>Al-10 Si on B-1900 |                    |           |        |                          |           |
| Control                          | 407                | 407       | 421    | 366                      | 367       |
| 1 hr.                            | 326                | 333       | 363    | 373                      | 354       |
| 6 hr.                            | 367                | 364       | 414    | 415                      | 406       |
| 10 hr.                           | 414                | 408       | 402    | 332                      | 338       |
| Silicide 512E<br>on Cb291        |                    |           |        |                          |           |
| Control                          | 145                | 163       | 158    | 154                      | 150       |
| 1 hr.                            | 170                | 172       | 168    | 168                      | 175       |
| 6 hr.                            | 177                | 182       | 181    | 185                      | 182       |
| 10 hr.                           | 185                | 178       | 192    | 212                      | 194       |
| Tungsten on<br>Inconel 600       |                    |           |        |                          |           |
| Control                          | 205                | 159       | 138    | 238                      | 232       |
| 1 hr.                            | 147                | 147       | 152    | 152                      | 137       |
| 6 hr.                            | 142                | 142       | 163    | 147                      | 152       |
| 10 hr.                           | 168                | 176       | 185    | 192                      | 192       |

TABLE 11 (Page 2 of 3)

| Specimen                    | Distance From Edge |           | Center | Distance From Other Edge |           |
|-----------------------------|--------------------|-----------|--------|--------------------------|-----------|
|                             | 0.002 In.          | 0.007 In. |        | 0.007 In.                | 0.002 In. |
| Platinum<br>on Nichrome V   |                    |           |        |                          |           |
| Control                     | 162                | 200       | 168    | 198                      | 162       |
| 1 hr.                       | 173                | 183       | 176    | 187                      | 168       |
| 6 hr.                       | 186                | 202       | 182    | 192                      | 172       |
| 10 hr.                      | 168                | 188       | 188    | 198                      | 182       |
| Rhodium on<br>Nichrome V    |                    |           |        |                          |           |
| Control                     | 201                | 216       | 186    | 214                      | 189       |
| 1 hr.                       | 166                | 211       | 177    | 200                      | 184       |
| 6 hr.                       | 197                | 193       | 175    | 176                      | 186       |
| 10 hr.                      | 182                | 202       | 177    | 196                      | 214       |
| GE 2541                     |                    |           |        |                          |           |
| Control                     | 260                | 280       | 235    | 305                      | 275       |
| 1 hr.                       | 805                | 245       | 235    | 225                      | 745       |
| 6 hr.                       | 790                | 690       | 230    | 610                      | 855       |
| 10 hr.                      | 750 to<br>800      | 585       | 240    | 595                      | 505       |
| Silicide<br>R508C on Cb 291 |                    |           |        |                          |           |
| Control                     | 158                | 158       | 159    | 159                      | 160       |
| 1 hr.                       | 192                | 189       | 192    | 189                      | 186       |
| 6 hr.                       | 177                | 177       | 162    | 177                      | 177       |
| 10 hr.                      | 188                | 185       | 178    | 188                      | 177       |
| Platinum-Rhodium            |                    |           |        |                          |           |
| Control                     | 276                | 276       | 290    | 282                      | 235       |
| 1 hr.                       | 172                | 183       | 176    | 188                      | 178       |
| 6 hr.                       | 188                | 201       | 183    | 194                      | 172       |
| 10 hr.                      | 99                 | 154       | 125    | 124                      | 130       |
| Molybdenum-50%<br>Rhenium   |                    |           |        |                          |           |
| Control                     | 358                | 372       | 378    | 379                      | 360       |
| 1 hr.                       | 358                | 376       | 390    | 366                      | 315       |
| 6 hr.                       | 264                | 376       | 405    | 356                      | 390       |
| 10 hr.                      | 373                | 363       | 394    | 395                      | 354       |
| Tungsten                    |                    |           |        |                          |           |
| Control                     | 456                | 486       | 514    | 538                      | 518       |
| 1 hr.                       | 528                | 538       | 548    | 512                      | 504       |
| 6 hr.                       | 495                | 512       | 494    | 495                      | 504       |
| 10 hr.                      | 456                | 502       | 494    | 495                      | 486       |
| TZM Molybdenum              |                    |           |        |                          |           |
| Control                     | 292                | 344       | 338    | 312                      | 324       |
| 1 hr.                       | 325                | 328       | 316    | 336                      | 312       |
| 6 hr.                       | 312                | 335       | 312    | 312                      | 320       |
| 10 hr.                      | 300                | 358       | 308    | 324                      | 338       |
| Nichrome V                  |                    |           |        |                          |           |
| Control                     | 178                | 192       | 168    | 202                      | 178       |
| 1 hr.                       | 182                | 182       | 164    | 182                      | 185       |
| 6 hr.                       | 182                | 196       | 164    | 178                      | 178       |
| 10 hr.                      | 188                | 185       | 189    | 191                      | 197       |

TABLE 11 (Page 3 of 3)

| Specimen | Distance From Edge |           | Center | Distance From Other Edge |           |
|----------|--------------------|-----------|--------|--------------------------|-----------|
|          | 0.002 In.          | 0.007 In. |        | 0.007 In.                | 0.002 In. |
| B-1900   |                    |           |        |                          |           |
| Control  | 320                | 384       | 396    | 390                      | 379       |
| 1 hr.    | 374                | 396       | 420    | 402                      | 402       |
| 6 hr.    | 396                | 404       | 420    | 402                      | 402       |
| 10 hr.   | 441                | 441       | 441    | 421                      | 420       |

**TABLE 12**  
**SUMMARY OF HARDNESS NUMBERS ON MATERIAL EXHIBITING**  
**A DIFFUSION/CONTAMINATION LAYER**

Knoop 100 Gram Microhardness<sup>(1)</sup> Traverse

| Material    | Sample Ident. | In Diffusion Layer      | 0.005 in. from Diffusion Layer | Center | 0.005 in. from Diffusion Layer | In Diffusion Layer |
|-------------|---------------|-------------------------|--------------------------------|--------|--------------------------------|--------------------|
| Haynes 188  | H8-3          | 661.0<br>644.0          | 364.2                          | 331.6  | 358.4                          | 702.9<br>633.0     |
| Inconel 600 | I6-3          | 290.0                   | 233.2                          | 206.0  | 216.3                          | 259.0              |
| Haynes 25   | H5-3          | 863.2<br>715.6          | 395.2                          | 453.6  | 430.4                          | 806.8<br>854.8     |
| Inconel 718 | I8-3          | 715.6<br>735.2          | 488.0                          | 422.0  | 479.2                          | 665.2<br>684.8     |
| Inconel 750 | I0-3          | 470.4<br>438.0          | 422.8                          | 408.8  | 402.2                          | 558.0<br>536.0     |
| B-1900      | B-3           | 317.0<br>370.2          | 488.0                          | 497.2  | 462.0                          | 715.6<br>569.2     |
| GE 2541     | GE-3          | 772.0<br>788.0<br>804.5 | 588.0                          | 246.2  | 599.4                          | 502.2              |

**Notes:**

- (1) All data displayed in this table are for material exposed to hydrazine decomposition products for 10 hours at 1400°F.

The tungsten specimens passed all required tests. The 10-hour exposure specimen did break when bending 180 degrees, but the break was not in the bend radius. One half of the broken specimen was retested with fully acceptable results. Tungsten requires heating to 500°F in order to attempt to bend this normally nonductile material; therefore, any conclusions based on bend test evaluation of tungsten must also include the material's ductility as a variable. Although presenting some fabrication difficulties, tungsten also appears to be an acceptable material.

The platinum-10% rhodium control specimen cracked and therefore actually failed the ASTM-E-190 Bend Test. Further investigation into this anomalous behavior utilizing Knoop 100 gram microhardness traverses showed that the control specimen was significantly higher in hardness than the exposed specimens. These findings indicate that the exposed specimens were annealed by the high temperature exposure test. Thus, the exposed specimens were able to pass the 180-degree bend test while the unannealed control specimen was not. The exposed specimens demonstrated no evidence of any corrosive attack; therefore, platinum-10% rhodium is an acceptable material.

Haynes 188 exhibited a definite contamination layer during exposure to the decomposed hydrazine. However, at times less than 10 hours exposure, it was the thinnest layer of any of the materials which showed a contamination layer, and it did not cause any failure in the bend test evaluation. The layer did check or surface crack in the bending but the cracks did not penetrate into the uncontaminated interior of the Haynes 188. The depth of contamination layer is increasing at a faster rate at the 10-hour exposure time, which may indicate that for lives longer than 10 hours, Haynes 188 may not be as desirable. But within the exposure time studied, this alloy is the best of those which showed a contamination layer. It should also be noted that this alloy shows the highest strength at 1400°F of any of the materials which show satisfactory bend test performance.

The Inconel 600 showed a definite, but thin, contamination layer from exposure to the decomposed hydrazine. This layer, with an expected higher hardness and increasing depth with time of exposure, caused some checking or surface cracking during the bend test. Even with the surface checking in the contaminated layer, no cracking extended into the unaffected interior portion of the material. The ductility and toughness of the Inconel 600 prevented any serious degradation of the material. The Inconel 600 should be considered a candidate material for this application provided this surface contamination penetration is allowed for. Thin sections, such as screens using thin wire, could be a marginal application for this alloy, but thicker sections could be used.

Hastelloy B exhibited no visible contamination layer from exposure to the decomposed hydrazine. There was also no significant increase in hardness 0.002 inch below the surface. There was, however, definite surface cracking or checking in a layer up to 0.003 inch below the surface during the bend testing of the exposed samples. As with Haynes 188, these surface cracks or checks did not extend into the interior and did not significantly degrade the bend test performance. On the basis of this test, Hastelloy B, Haynes 188, and Inconel 600 could be considered equal for this type of application.

Inconel 600 coated with tungsten passed all required tests. Therefore, this is a satisfactory candidate material. However, the chance of a coating failure would always be present.

B-1900/Al-10 Si is a satisfactory candidate. The bend test cannot be used to judge its eligibility because it is inherently nonductile, but Knoop 500 gram microhardness traverses indicate no increase in hardness. Further, 200X investigations show no contamination layer or any other

microstructural change to indicate that this material has been adversely affected by the corrosive environment. However, bare B-1900 is susceptible to corrosive attack (see below), and, in the event of a coating failure, this material could be attacked.

The control specimens of molybdenum-50% rhenium and TZM (molybdenum - 0.5% Ti-0.1% Zr) met the full 180-degree bend, but the exposed specimens in each case either cracked or completely failed. Microhardness comparisons showed no change even very close to the surface. A very thin diffusion layer was observed on the molybdenum-50% rhenium. Both materials had definite surface reactions from the exposure.

Cb 291/R508C, Cb 291/R512E, Haynes 25, Inconel 718, and Inconel 750 control specimens met a full 180-degree bend, but the 10-hour exposure specimens in each case either cracked or completely failed. Knoop (500 gram) microhardness comparisons of each material indicated that the 10-hour exposure specimens were harder than the control specimens (although in the case of the Cb 291 specimens, the increase is small).

The bend test data for B-1900 and GE 2541 is inconclusive due to inherent limited ductility. Knoop (500 gram) microhardness readings show that the 10-hour exposure specimens were significantly and uniformly higher in hardness than the control specimens. Investigations of typical cross sections at 200X showed a discoloration layer in the exposed specimens of each material and these layers were significantly higher in hardness.

Nichrome/Rhodium and Nichrome/Platinum platings reacted to the thermal exposure conditions of this test. The bare nichrome performed better, indicating that the effort to develop reliable platings for this material is probably not justified.

#### 3.4 IN SITU SCREEN TESTS

The most severe condition encountered by materials in a hydrazine reactor is exposure of a relatively large surface (such as screens) to repetitive pulsing within the catalyst bed or decomposition region. The logical next step, then, was to subject the better materials to such a condition.

The selected materials (see Paragraph 3.2) were obtained commercially in screen form in the mesh and wire sizes indicated in Table 13. This table also summarizes the operating conditions and duty cycle used.

##### 3.4.1 Test Apparatus

One of the workhorse gas generators used for the Dynamic Exposure Test was rebuilt with the test screens in the positions shown in Figure 56. The chamber was fitted with a sonic nozzle sized to produce 100 psia chamber pressure at a hydrazine flow rate of 0.022 lb/sec.

The screens within the catalyst bed were not attached to the chamber wall, being spaced and supported by the 14-18 mesh Shell 405 catalyst. The lower bed retainer screen (Haynes 25) was spot welded to the retainer plate. The hemispherical diffuser screens were attached to the injector with AWS-ASTM Classification BAu-4 (82% Au, 18% Ni) braze.

A Wright Components solenoid propellant valve was used and, together with a cavitating venturi, controlled the propellant flow. The reactor was fired vertically downward with standard MIL-P-26536C hydrazine from a conventional nitrogen-pressurized feed system.

**TABLE 13**  
**IN SITU SCREEN TEST MATERIALS REACTOR**

- Constant Operating Conditions:
- Bed Exit  $P_c \approx 100$  psia
  - $N_2H_4$  Flowrate = 0.022 lbm/sec
  - 14-18 Mesh Shell 405 ABSG Catalyst
  - Sea Level

| Reactor Position (1) | Type                                     | Material      | Mesh    | Wire Dia. |
|----------------------|------------------------------------------|---------------|---------|-----------|
| 1                    | Diffuser <sup>(2)</sup><br>(Head Screen) | Nichrome V    | 20 x 20 | 16 mil    |
| 2                    | Diffuser <sup>(2)</sup><br>(Head Screen) | Pt-10 Rh      | 45 x 45 | 6 mil     |
| 3                    | Separator                                | Tungsten      | 16 x 16 | 8 mil     |
| 4                    | Separator                                | Inconel 600   | 40 x 40 | 10 mil    |
| 5                    | Separator                                | Hastelloy B   | 10 x 10 | 41 mil    |
| 6                    | Separator<br>(Aft Retainer)              | Haynes 25 (3) | 24 x 24 | 14 mil    |

- (1) Numbered sequentially from injector station.  
 (2) Numbers 1 and 2 are formed together and are in direct contact.  
 (3) Control Specimen.

Duty Cycle

- 5 sec steady-state, cool to ambient with He-purge.
- 199,998 pulses: 50 msec ON/450 msec OFF.
- 5 sec steady-state.

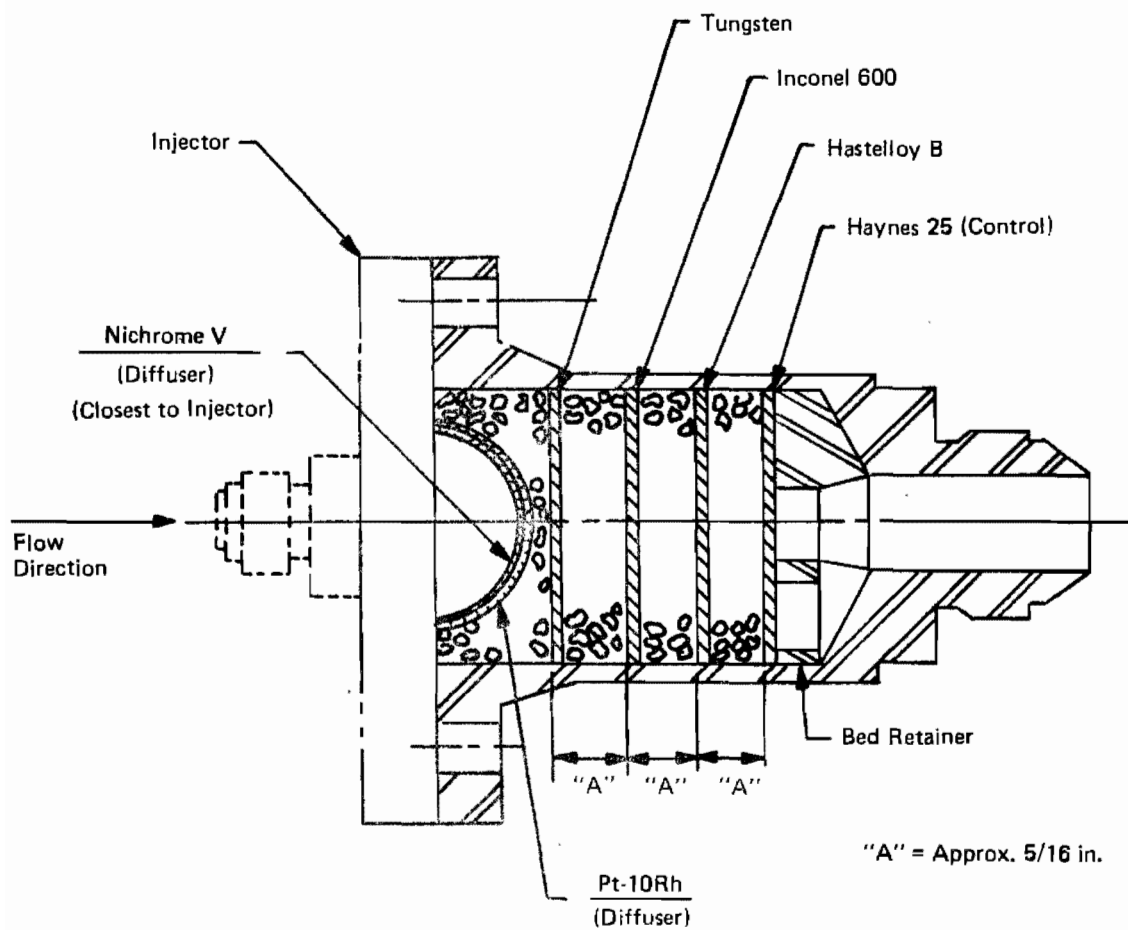


Figure 56. Relative position of screen types in catalyst bed of *in-situ* reactor.

### 3.4.2 Testing

The reactor, containing the test screens, was check-fired for five seconds to assure that the introduction of the extra screens did not affect performance. Operation was very smooth and stable, and the test sequence was continued.

199,998 fifty-millisecond pulses were fired at a rate of two pulses per second. Only one interruption occurred in this sequence. It was necessary to reset the electronic control system after 99,999 cycles because this was the limit of the unit. At that time, as at all other times when the reactor was shut down for over 0.5 second, a helium purge was used to prevent any uncontrolled environment from developing in the test region while specimen temperatures exceeded 200°F.

After completion of the pulsing duty cycle, a 5-second check firing showed no change in reactor operation from the initial baseline. Upon disassembly, all screens were observed to be intact, and no shifting of the unattached screens had occurred.

The catalyst was in very good condition, again with only mild sintering observed. Quantitative weight and mesh size measurements were not made, but catalyst attrition appeared to be minimal.

### 3.4.3 Metallurgical Evaluation

#### 3.4.3.1 Screen Condition and Surface Appearance

All six screens after the test firing were intact and there was no evidence of gross damage. The exposed screens, and samples of unexposed screen for all materials are shown in Figure 57. The platinum-10% rhodium screen, which formed the outer layer (catalyst side) of the two-screen diffuser, had suffered mechanical damage before final assembly. The peak of the hemispherical screen was scraped and some wires were broken mechanically. However, this mechanical damage was not aggravated in the exposure test.

A photograph of the diffuser screens after firing is shown in Figure 57, with views of the individual screens, after disassembly of the diffuser, shown in Figure 58. There was a light "tarnish film" on the Nichrome. The platinum was bright and unchanged from the control sample appearance. The tungsten wire screen was also bright and unchanged from the as-received condition.

The last three screens, Inconel 600, Hastelloy B and Haynes 25, (Figures 56 and 57), all showed definite surface discoloration. The Haynes 25 and Hastelloy B showed rather uniform discoloration appearance, while the Inconel 600 screen had small spots that were still bright and apparently unaffected. Based on the macroscopic examination (10-20X), none of these three screens showed a degradation beyond the surface discoloration. However sectioning and microscopic examination, showed that this preliminary examination did not reveal the true nature of these three screen materials, particularly the Haynes 25 screen.

#### 3.4.3.2 Metallographic Examination

Samples of each screen material, in both the exposed and the as-received condition, were metallographically prepared to examine the cross section of the screen wires. In all cases, the cross sections were first examined in the as-polished (unetched) condition for evidence of cracks or other defects and contamination layers or other degradation from the test exposures. After examining and

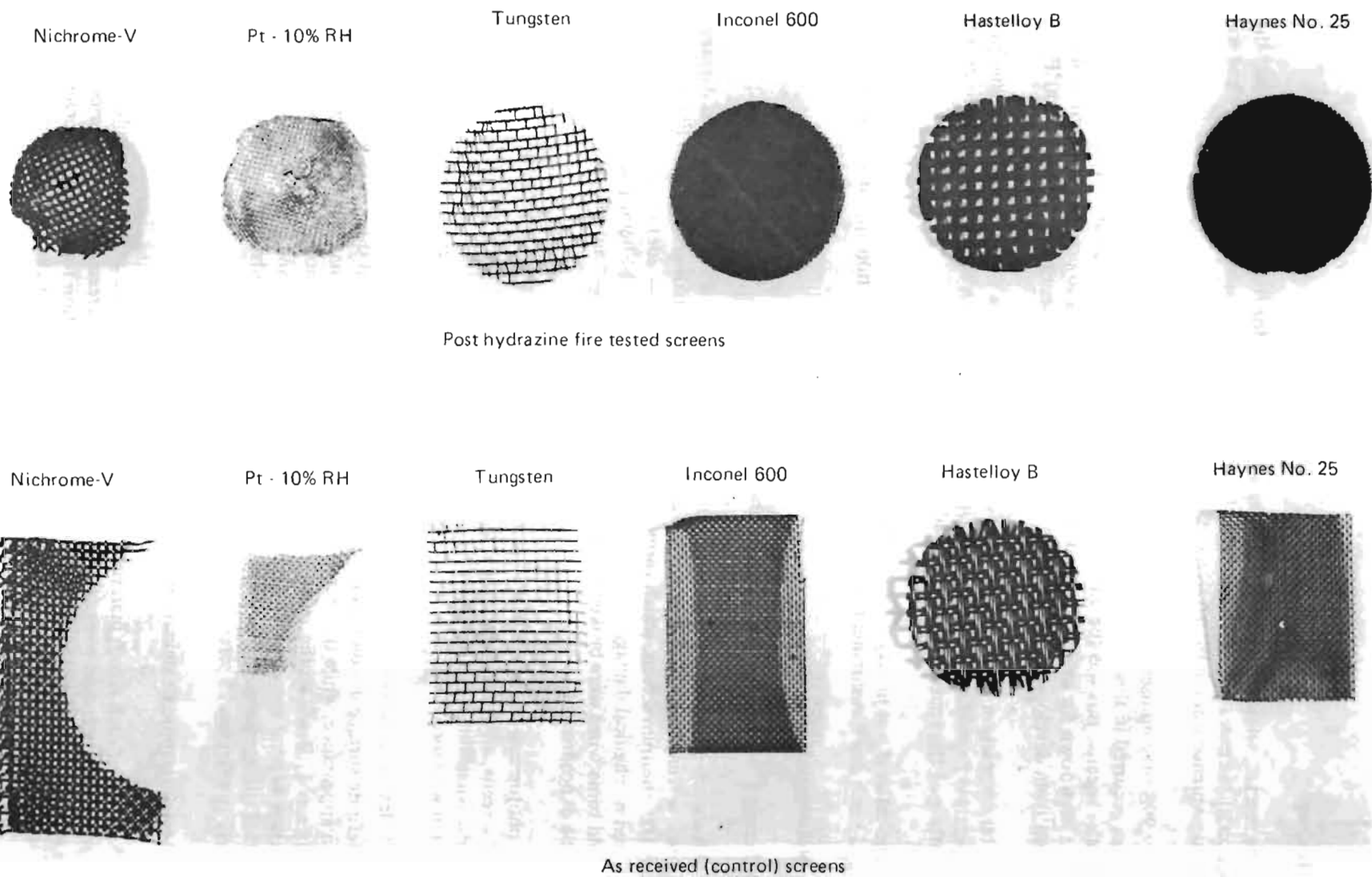
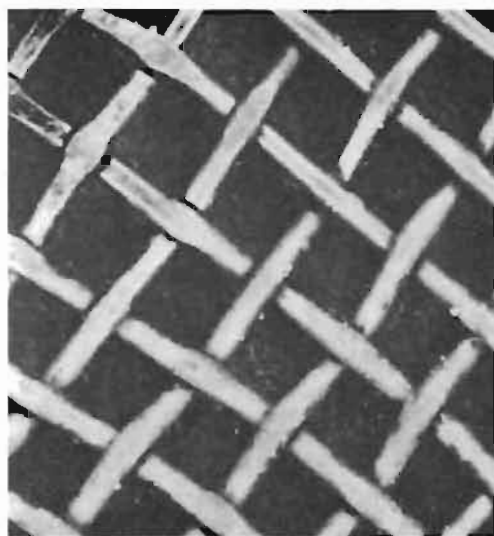
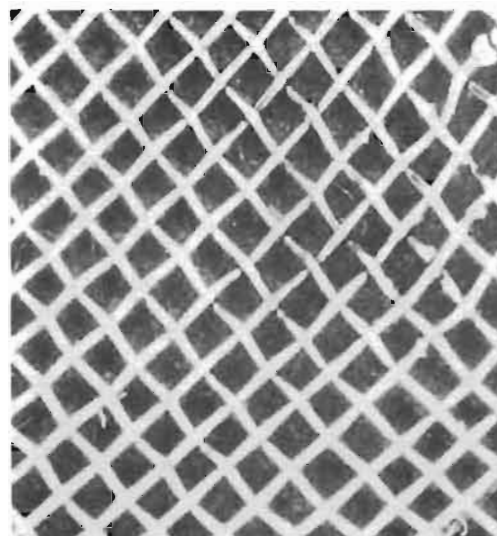


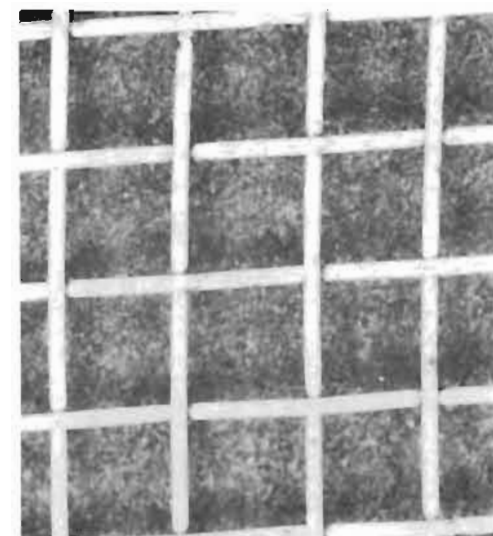
Figure 57. Comparative views of various screen materials before and after firing.



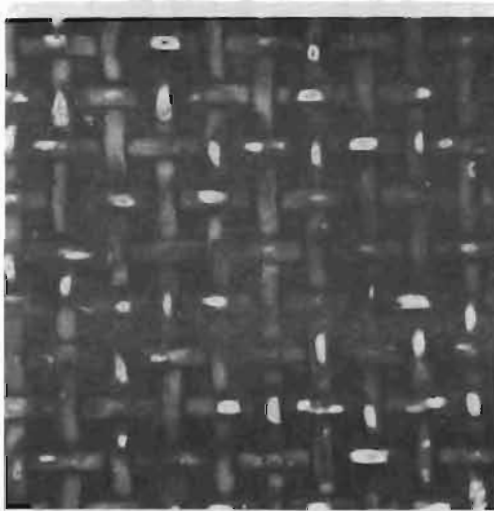
A. Nichrome V



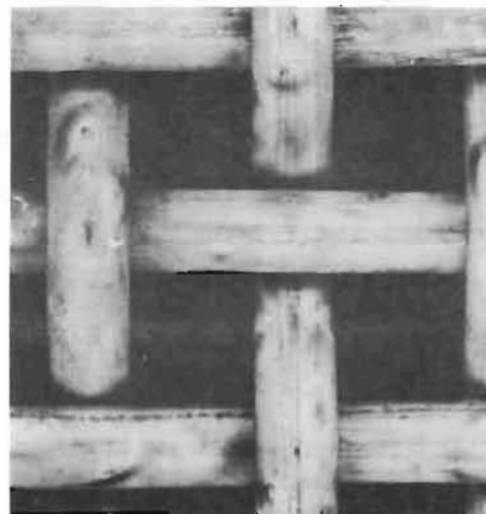
B. Platinum - 10% Rh



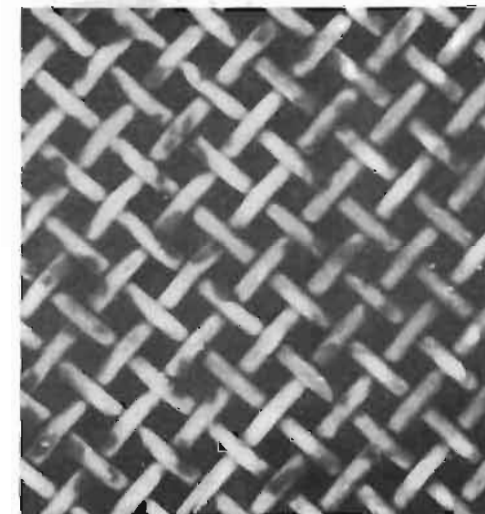
C. Tungsten



D. Inconel 600



E. Hastelloy B.



F. Haynes 25

All Views: MAG: 10X

Figure 58. Macrophotographs of catalyst retainer screens after firing tests.

documenting the as-polished condition, the samples were etched in an appropriate manner to bring out the grain structure of the screen material for further examination of the material and any environmental effects.

The Nichrome V screen, both control and exposed sections, is shown in Figure 59. After exposure, a very thin (0.00012 inch) contamination layer was visible around the surface wires examined. The contamination layer contained microcracks in several locations around the periphery on most wire sections examined. However, these extremely small microcracks did not extend into the matrix in any location observed. In the etched condition, Figure 60, the Nichrome V was found to have a normal and desirable microstructure and grain size (ASTM 6) after exposure. It should be noted that in addition to the thermal exposure in the test firing, this Nichrome screen (and the platinum screen) was subjected to a high temperature (2100°F) vacuum brazing cycle. This caused no abnormalities (grain growth) or other deleterious effects.

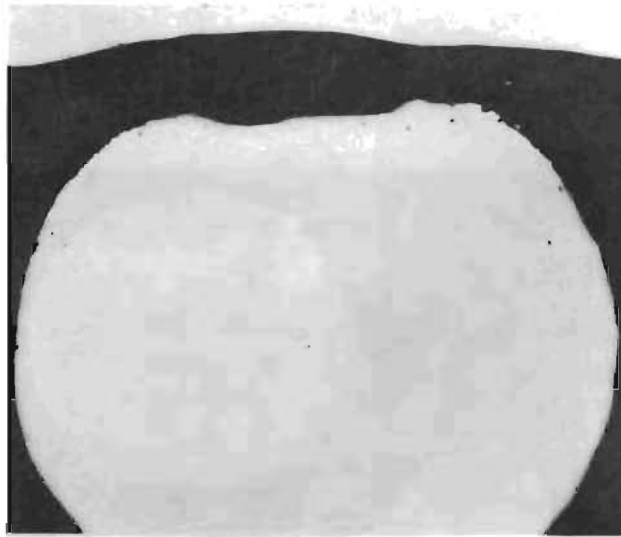
In order to verify that the microcracks in the surface contamination layer of the Nichrome V did not degrade the screen, a segment of the exposed screen was bent through a 180-degree angle. No evidence was seen of any opening or propagation of these extremely shallow cracks in the contaminated surface layer. The bent screen is shown in Figure 61. It would appear that although there was a very thin contamination zone with microcracks in this Nichrome V material after exposure, the presence of the zone with microcracks did not affect the overall bend ductility of the alloy.

Sectioning and microstructure examination of the platinum-10% rhodium screen showed absolutely no evidence of contamination or other environmental effects from the exposure or prior brazing cycle. Views of the platinum screen wire after exposure in both unetched and etched conditions are shown in Figure 62.

The tungsten wire screen provided the same observations as the platinum. There was no evidence of any surface attack, formation of surface contamination layer or any other degradation of the tungsten, as shown in Figure 63. The microstructure in the etched condition was indistinct, indicating that the thermal exposure of the firing cycle had not caused recrystallization and/or grain growth in the tungsten. This is a desirable condition (lack of recrystallization/grain growth) for the tungsten, since as-worked and stress relieved tungsten possesses better properties (some ductility and toughness) than tungsten with large grains.

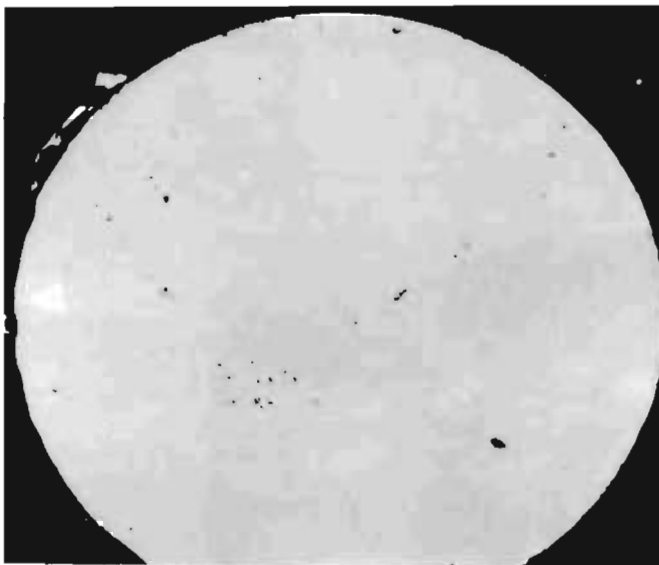
The Inconel 600 screen material showed a definite contamination layer after the exposure, as seen in Figure 64. This layer, approximately 0.0005 inch thick, was uniform around the wire circumference, except at the contact points of the cross wires in the woven screen where the wires protected each other. The contamination layer consisted of a discoloration and hardening of the material, but did not appear to result in the formation of new phases, since the grain boundary pattern in the contaminated layer was the same as in the core of the wire.

The Hastelloy B showed a 0.001 to 0.002 inch thick layer on the surfaces of the exposed screen, barely visible in Figure 65. The layer was not discolored, compared to the interior, and did not respond to chemical etching in a manner differing from the interior. The microhardness evaluation, discussed below, verified the presence of the contaminated layer in this Hastelloy B, but this layer is evidently of a subtle nature, different from the more obvious layers of the Inconel 600 and Haynes 25 alloys.



A. Nichrome V wire as-received

MAG: 200X

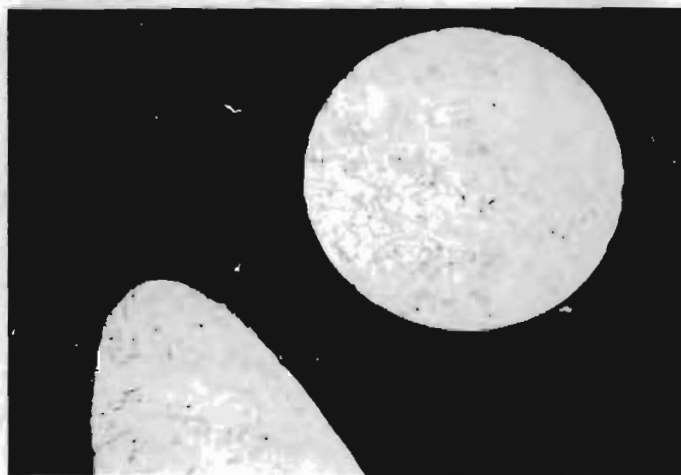


B. Exposed screen (contaminated surface layer 0.00012 inch deep)

Mag: 500X

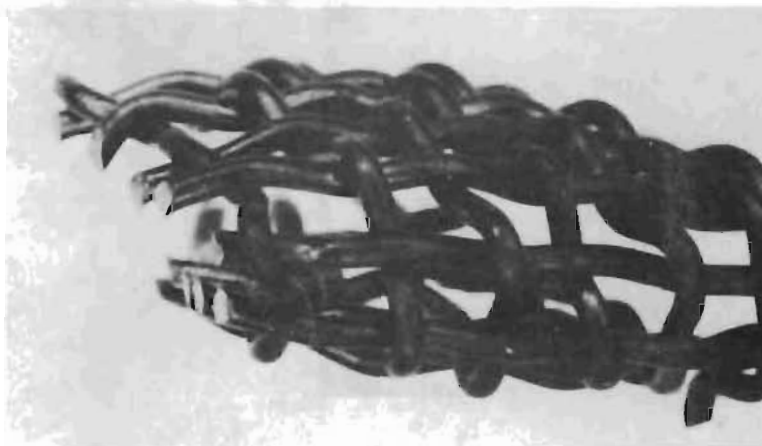
MAG: 200X  
As-Polished-Unetched

Figure 59. Cross sections of wires from Nichrome V screen.



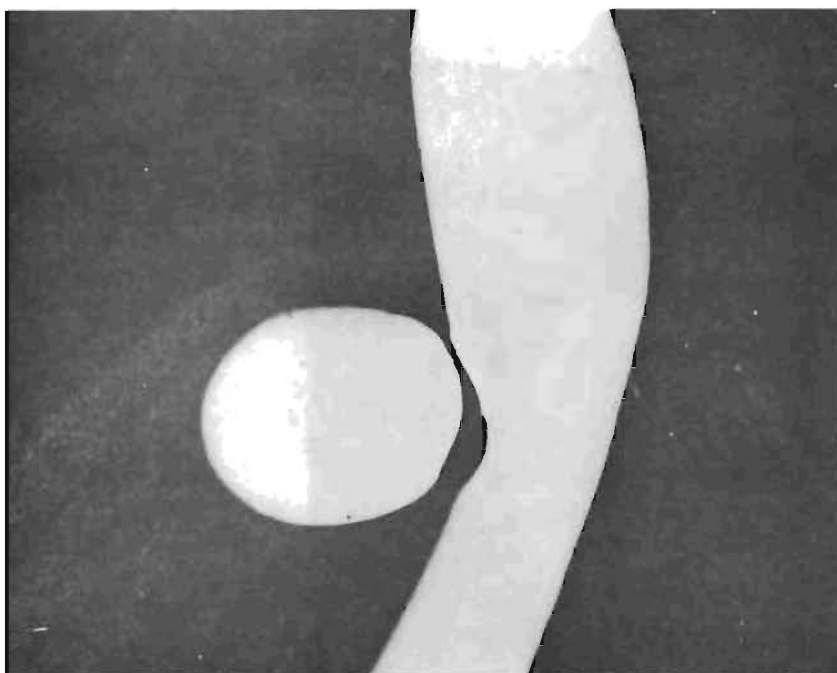
MAG: 100X  
 Etchant-92% HCL 5% H<sub>2</sub>SO<sub>4</sub>  
 3% HNO<sub>3</sub> Immersion

Figure 60. Microstructure of exposed Nichrome V screen.



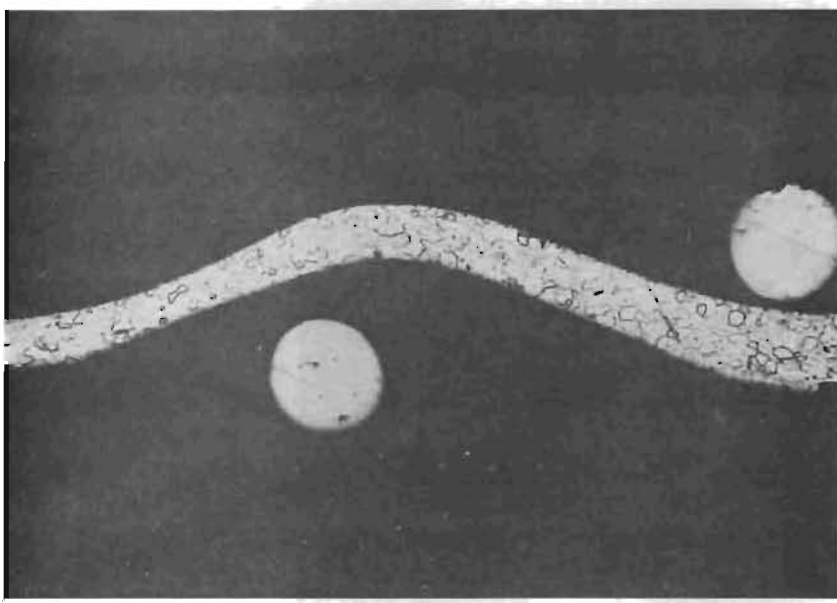
MAG: 10X

Figure 61. Bend test of exposed Nichrome V screen.



(A) Pt-10% Rh wire in as-polished condition after exposure.

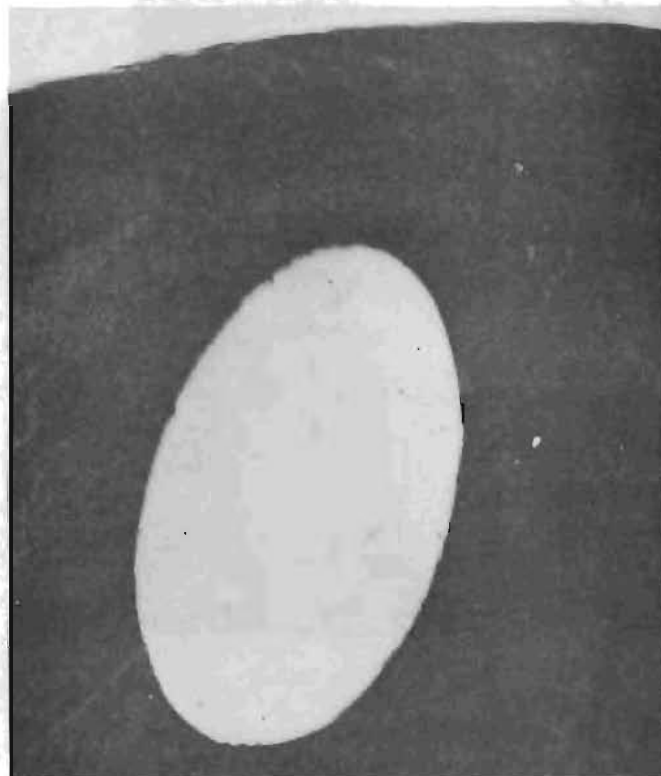
MAX: 200X



(B) Pt-10% Rh wire polished and etched.

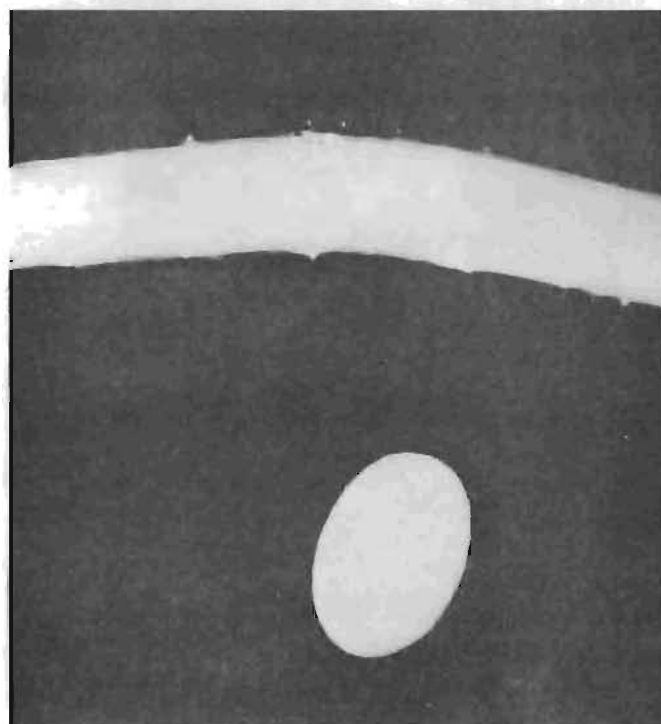
MAG: 100X  
ETCHANT: 3 Parts  
Hcl 1 Part H<sub>2</sub>O  
electrolytic.

Figure 62. Photomicrographs of platinum 10% rhodium screen after exposure.



(A) As-polished condition  
after exposure

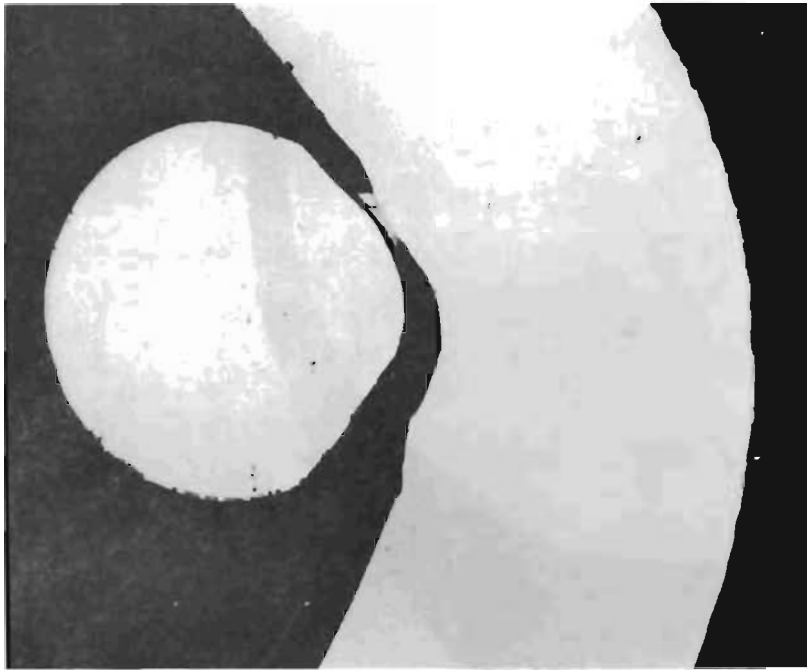
MAG: 200X  
UNETCHED



(B) Polished and etched.

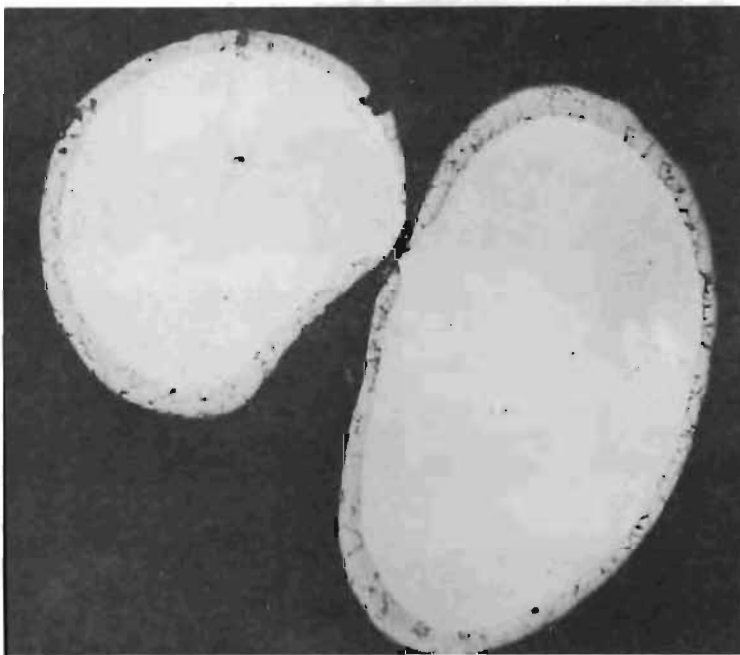
MAG: 100X  
ETCHANT:  
10% NaOH  
ELECTROLYTIC

Figure 63. Cross sections of tungsten screen wires after exposure.



(A) Inconel 600 wires  
As-received

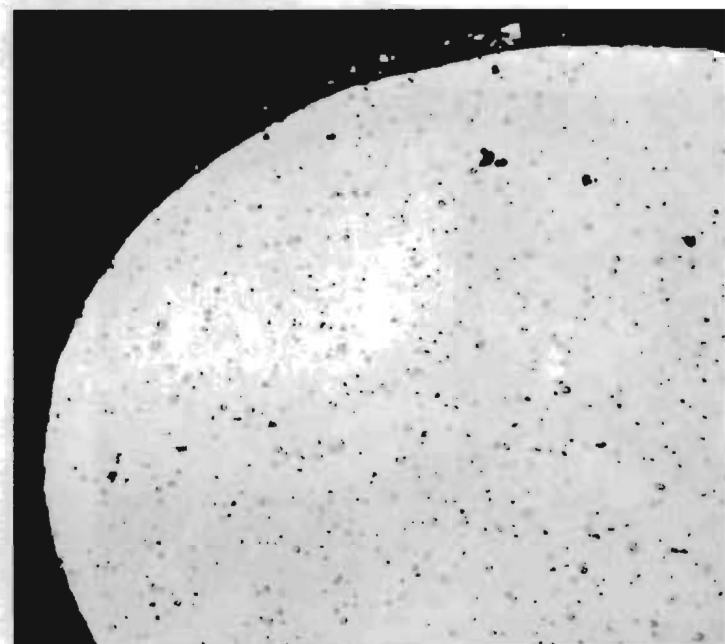
MAG: 200X  
UNETCHED



(B) Inconel 600 wires.  
Exposed screen.  
(Surface layer  
0.0005 inch deep)

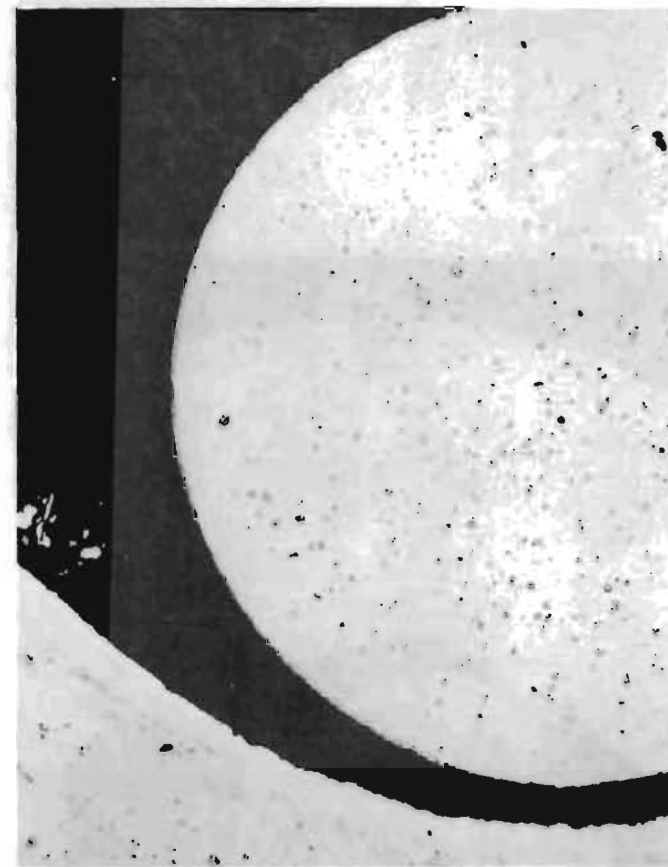
MAG: 200X  
UNETCHED

Figure 64. Photomicrographs of Inconel 600 screen before and after exposure.



(A) Hastelloy B  
control screen wire.

MAG: 100X  
UNETCHED



(B) Hastelloy B  
wire after exposure.

MAG: 100X  
UNETCHED

Figure 65. Photomicrographs of Hastelloy B screen before and after exposure.

The Haynes 25 as-received screen material was fabricated from wire that contained extensive longitudinal cracks extending into the core 1/10 to 1/3 of the diameter. Every as-received cross section contained evidence of at least one of these cracks and often showed two or three (see Figure 66, Detail A). After exposure, a pronounced contamination layer, generally 0.0006 inch in depth was found on all wire surfaces (Figure 66, Details B and C). In addition, there was penetration of the contamination layer much deeper into the core, where the longitudinal cracks had existed in the original material. This produced a large amount of contamination throughout the cross section of the exposed Haynes 25 screen wires. In a few instances new cracks were formed in the contaminated layer which extended into the core of the exposed wire, indicating embrittlement of the core, as well as the surface. The extent of this internal contamination is illustrated by the views of the wire cross section in the etched condition, Figure 66, Details D and E. The exposed sample in Figure 66, Detail E shows obvious alteration of the microstructure, particularly outlining of the grain boundaries through the entire cross section. Although the pre-existing longitudinal cracks in the Haynes 25 aggravated the situation, it is evident that there is a basic incompatibility between Haynes 25 and the decomposed hydrazine environment to which it was exposed.

#### 3.4.3.3 Microhardness Evaluation

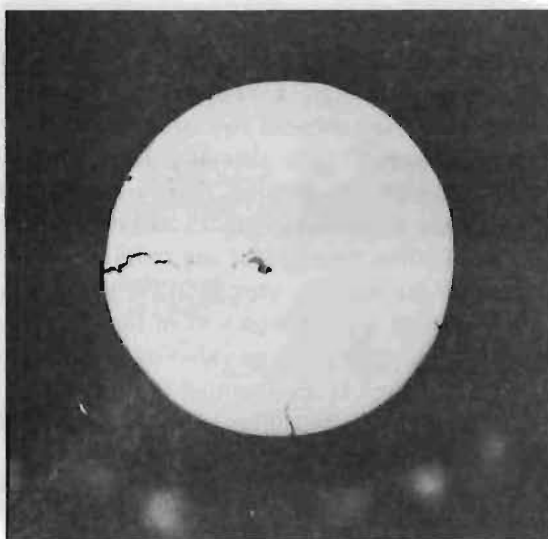
The polished cross sections representing all screen materials, both as-received and after exposure, were evaluated for microhardness. One reading was obtained near the surface (approximately 0.001 inch below surface) and two readings were obtained in the core for each sample. Because of its importance as the most promising, yet economical candidate material, several additional sets of readings were taken on the Nichrome V material. The average hardness number data for all of these screens are presented in Table 14.

The data for the Nichrome V, as-received, shows a slight but definite hardening of the surface compared to the interior, probably as the result of wire drawing effects. The exposed sample shows essentially the same increase between interior and surface, but the hardness numbers are 100 points lower than in the unexposed sample. This indicates that a definite annealing (softening) of the Nichrome has occurred due to the thermal effects of the brazing cycle plus test exposure. Since the interior to surface hardness increase of the exposed sample is slightly less than the corresponding increase in the control sample, it is evident that there was no degrading effect of the test exposure on the Nichrome V.

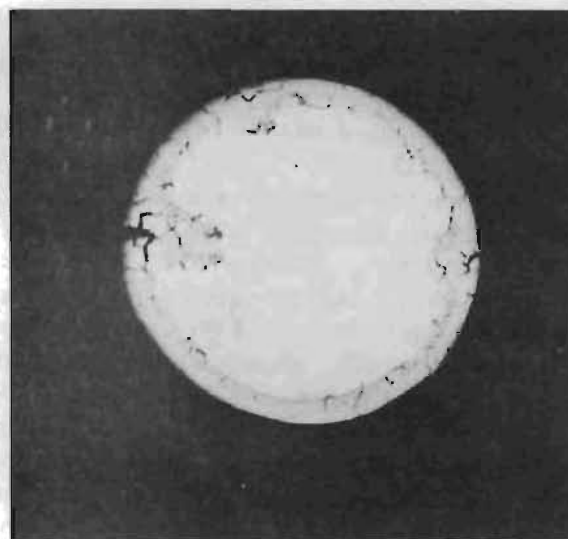
The platinum-10% rhodium is a very soft alloy, and it was necessary to restrict the hardness test to a very light load, 25 grams, to maintain a minimum indentation size. There is a slight evidence of an increase in surface hardness versus the interior of the exposed sample. These differences of 10 points of Knoop hardness are quite small, and represent two points or less of difference in a Rockwell scale. Based on this observation, the platinum-10% rhodium material is considered to have an insignificant hardness change due to the exposure.

The tungsten wires are, as expected, quite hard. The center of the control compared to the exposed sample shows essentially identical hardness numbers. The surface showed a slight increase, but this change in surface hardness is not considered significant.

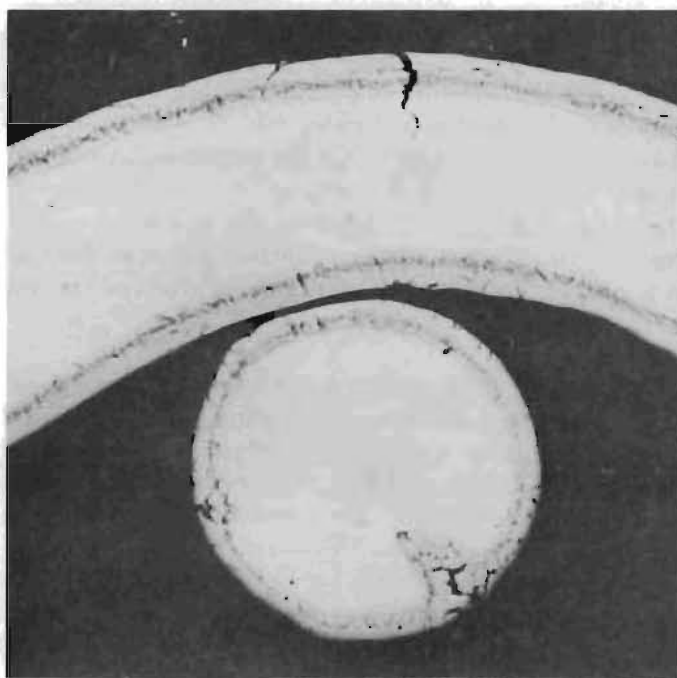
The Iconel 600 shows essentially equivalent interior and surface readings between the control and exposed sample. This material exhibited a pronounced surface contamination layer, and the hardness within this layer is very high. Since the hardness immediately under this layer is not



(A) Haynes No. 25 control  
screen  
MAG: 200X

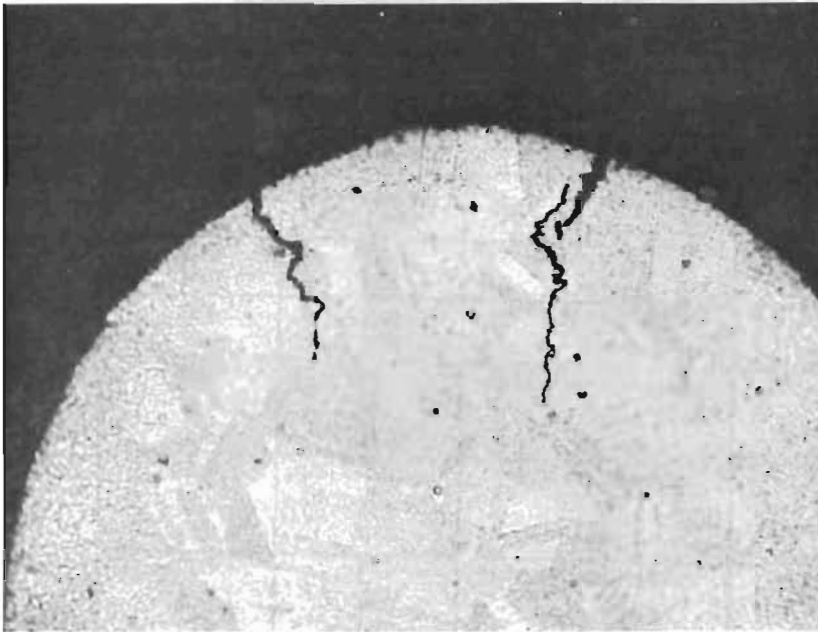


(B) Wire cross section from  
exposed screen illustrating  
the extensive contamination  
observed both under surface  
and along the pre-existing  
cracks. MAG: 200X



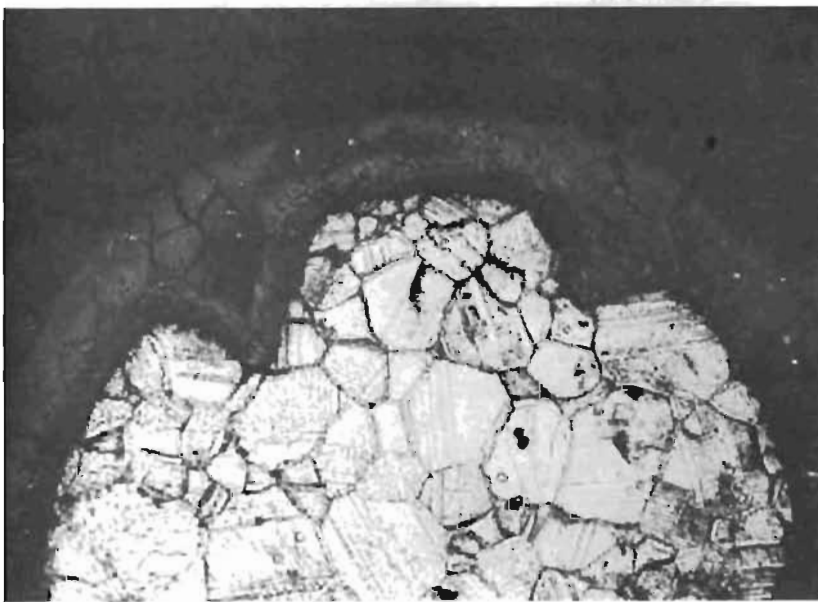
(C) Cross section of wire  
from exposed screen  
illustrating the  
extensive surface  
contamination and  
formation of crack  
into interior of wire.  
MAG: 200X

Figure 66. Cross sections from Haynes 25 catalyst bed retainer screen before and after exposure. (Sheet 1 of 2)



- (D) Cross section of wire from Haynes No. 25 control screen polished and etched illustrating at higher magnification the depth of cracking in the as-received material.

MAG: 500X  
 ETCHANT: 90%-HCL  
 10%-(30%)  $H_2O_2$



- (E) Cross section of wire from Haynes No. 25 exposed screen polished and etched illustrating at higher magnification the observed contamination.

MAG: 500X  
 ETCHANT: 90%-HCL  
 10%-(30%)  $H_2O_2$

Figure 66. Cross sections from Haynes 25 catalyst bed retainer screen before and after exposure. (Sheet 2 of 2)

**TABLE 14**  
**HARDNESS OF SCREENS BEFORE AND AFTER EXPOSURE**

| Screen Material      |                    | Microhardness Numbers<br>(100 gm Knoop Readings) |         |
|----------------------|--------------------|--------------------------------------------------|---------|
|                      |                    | Control                                          | Exposed |
| Nichrome V           | edge               | 396                                              | 290     |
|                      | center             | 363                                              | 266     |
| Platinum-10% Rhodium | edge               | 241*                                             | 254*    |
|                      | center             | 233*                                             | 244*    |
| Tungsten             | edge               | 830                                              | 890     |
|                      | center             | 870                                              | 872     |
| Inconel 600          | contaminated layer | —                                                | 870     |
|                      | edge               | 308                                              | 337     |
|                      | center             | 331                                              | 305     |
| Hastelloy B          | contaminated layer | —                                                | 536     |
|                      | edge               | 454                                              | 402     |
|                      | center             | 353                                              | 333     |
| Haynes 25            | contaminated layer | —                                                | 890     |
|                      | edge               | 424                                              | 569     |
|                      | center             | 399                                              | 472     |

Notes: Edge Readings: are 0.001 inch or less from surface but are not in any visible contaminated layer.

Nichrome: data is the average of 3 readings (edge) and 6 readings (center). All other material data is the average of 1 or 2 data points.

\*Because of extreme softness of Pt readings are 25 gm Knoop.

significantly different from the control sample, it is evident that the contamination observed has not affected any portion of the Inconel except the obvious surface contamination layer.

The Hastelloy B screen exhibited a very subtle, but definite, contamination layer in visual examination which showed significant hardness increase. The wire material underneath this contamination layer, showed a small decrease in hardness from control to exposed sample, both near the surface and in the interior. This would indicate that an annealing effect was taking place in the Hastelloy B wire during the exposure, with hardening due to contamination only occurring in the 0.001 to 0.002 inch thick visible surface contamination layer.

The hardness testing of the Haynes 25 showed that the entire wire was hardened by contamination effects in the exposure. The visible contamination layer had very high hardness, as would be expected. The metal immediately under that layer also had relatively high hardness. Even the interior of the Haynes 25 wire was substantially higher in hardness than the control, indicating general contamination of the entire wire cross section.

#### 3.4.4 Summary of In Situ Screen Test Results

Platinum-10% rhodium and tungsten showed no effect from the exposure. There was no visible contamination on the surface or in the subsurface layers, nor any significant hardness change. The extreme softness of the platinum and tendency toward brittleness of the tungsten make them somewhat less desirable materials of construction, but they do represent the best material choices from a resistance to environmental attack basis.

Nichrome V showed a light tarnish film on the surface and an extremely thin contamination layer in the cross section analysis. However, there was no subsurface contamination (hardness increase) of the wire material itself, and bend test after exposure showed it to retain its excellent ductility. Therefore, Nichrome V is recommended as a very good material of construction for screens or similar use in this environment.

Inconel 600 and Hastelloy B showed essentially identical behavior with a definite contamination layer (~0.002 inch deep) formed on all exposed surfaces. This layer was quite hard and could be expected under some conditions to degrade the integrity of screens or similar structures exposed in this type of environment. The interior material of the test screens was unaffected (or annealed) by the exposure, indicating that the contamination was limited to the visible surface layer.

Haynes 25 screen wire was seriously contaminated through its entire thickness by the decomposed hydrazine exposure, with a surface layer of high hardness, and internal contamination of the matrix. This material was the least acceptable of those tested. It should be noted, however, that extensive lateral cracking, probably introduced in manufacture of the wire, was found in the Haynes 25 control sample. This is probably responsible to some degree for the depth of contamination.

### 3.5 CONCLUSIONS

The materials tested can be divided into four groups, as indicated in Table 15. The materials within any group are roughly comparable, but resistance to the hydrazine decomposition environment is progressively poorer from group to group. Group three is divided to indicate a distinctly greater but similar reaction of the second part to the environment.

TABLE 15  
RANKING OF MATERIALS TESTED

| Group | Material                                                                                                                                                                                                                        | Comments                                                |
|-------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------|
| 1     | Platinum 10% Rhodium<br>Tungsten                                                                                                                                                                                                | Unaffected                                              |
| 2     | Nichrome V                                                                                                                                                                                                                      | Slight Surface Discoloration                            |
| 3     | Haynes 188<br>Hastelloy B<br>Inconel 600<br>Tungsten on Inconel 600<br>Al-10 Si on B-1900<br><br>Molybdenum-50% Rhenium<br>TZM<br>R512 E on Columbium 291<br>R508 C on Columbium 291<br>Haynes 25<br>Inconel 718<br>Inconel 750 | Measurable Contamination<br>Layer<br>Increased Hardness |
| 4     | B-1900<br>GE 2541<br>Rhodium on Nichrome<br>Platinum on Nichrome                                                                                                                                                                | Not Recommended                                         |

No visible or measurable change was found in platinum-10% rhodium or tungsten under these test conditions. Nichrome V was only mildly affected and was rated the best choice where high temperature strength is needed.

Haynes, Hastelloy, and Inconel alloys were generally considered acceptable but suffered visible degradation and measurable surface hardening, probably nitriding. B-1900 and GE 2541 were hardened throughout and are not recommended for this service.

The aluminide and silicide coatings were effective, as was tungsten. Platinum and rhodium plating on nichrome served no purpose and may have contributed to embrittlement below the plating. Platings and coatings are not generally recommended but can be useful in specific cases, provided coating integrity can be maintained.

## SECTION 4.0 THERMAL MANAGEMENT

Thermal management is recognized as one of the areas critical to the design of monopropellant devices because of the characteristic thermal instability of this type of propellant. Vaporization of hydrazine in confined passages can cause significant impulse bit and total impulse/pulse shifts; and, in the extreme, premature thermal decomposition can produce damage to the thruster.

Transient behavior is of particular interest because, for the numerous pulse-mode applications of monopropellant hydrazine devices, the majority of the operation is nonequilibrium. A short pulse will not bring the unit to steady-state operating conditions thermally; and as the wide temperature gradients in the thrust chamber/catalyst bed region begin to normalize after the pulse, heat soak-back to the propellant valve and vehicle and radiation to the surroundings ensues.

Multinodal thermal models are commonly used to predict and analyze these transients. Some of these models assume the catalyst bed to be a single node, i.e., a mass with a uniform temperature and constant specific heat. While this is acceptable for first order approximations, it is not adequate for situations requiring precise predictions or for defining conditions within zones of a catalyst bed.

As we began to study this problem, we realized that no reliable data were available on thermal conductivity of Shell 405 catalyst and effective conductivity to chamber walls. Further, even measured bulk specific heat values were not published. Hence, these measurements were made and were used to predict thermal gradients within catalyst beds.

To verify the predictions and to obtain a better understanding of steady-state thermal gradients within catalyst beds, two instrumented thrusters were tested. A series of tests was also done to determine if there were significant effects from detonations within propellant manifolds on catalyst attrition.

This section presents the data obtained from these basic measurements and discusses their relevance to thruster design.

### 4.1 CATALYST THERMOPHYSICAL PROPERTIES

A series of laboratory measurements of the "effective" thermal conductivity,  $K_e$ , and specific heat,  $C_p$ , of shell 405 catalyst were made to assist the development of a more fine-grained thermal model of the catalyst bed. The tests were carried out by Dynatech R/D Co. under subcontract.

#### 4.1.1 Thermal Conductivity

The "effective" conductivity, which includes radiative transfer between particles, was determined for several particle sizes as a function of temperature and compaction pressure.

##### 4.1.1.1 Testing

The measurements were carried out in vacuum in a standard guarded hot plate apparatus illustrated in Figure 67, wherein a cylindrical wafer of catalyst particles was confined and electrically heated at one face with a heat sink at the opposite face. The guard heaters prevent heat losses from

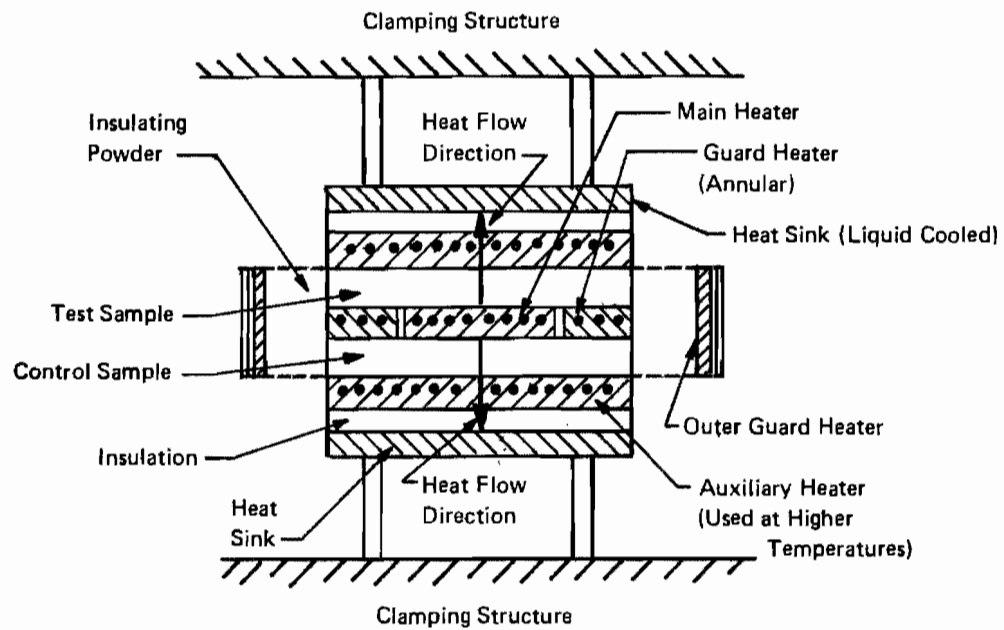


Figure 67. Schematic of guarded hot plate.

the source and from the cylindrical edge, guaranteeing unidimensional heat flow. With the input power ( $q$ ), cylinder height ( $\Delta X$ ) and cross section ( $A$ ), and source-sink temperature differential ( $\Delta T$ ) known, the effective conductivity is then:

$$k_e = \frac{q\Delta X}{A\Delta T}$$

The catalyst sample occupied one of the sample positions indicated in the figure, and was 3.6 inches in diameter and 0.4 inch thick. The catalyst was retained by a thin annular ring of a very slightly compressible, fiber insulation. The other sample position was occupied by a piece of insulation material of known thermal conductivity.

One sample of each of three mesh sizes was tested at temperatures ranging from about 400°F to about 1500°F at each of three compressive loads. A gradient of about 50°F was maintained across the catalyst sample during the tests.

Each test series began with the highest main heater temperature to account for "sintering" effects of the catalyst carrier. Also, for each mesh size the lowest compressive load, 30 psi, was applied first then increased to 150 psi and finally to 250 psi. Under each load, the compressed sample height was accurately measured and reported.

All of the tests were conducted in a vacuum of less than  $10^{-3}$  torr, and were in compliance with ASTM C177-71.

#### 4.1.1.2 Results

Table 16 presents the conditions and results of the 24 tests that were made. Figure 68, details A, B and C, plot the conductivity values for each mesh size against temperature. The trend lines are replotted in Figure 69 to allow comparison of the mesh sizes.

The measured sample thickness values ( $\Delta X$ ) are presented in Table 17. It is seen that only minor crushing took place. A small amount of sintering was noted in samples 1 and 2 after the high pressure tests, but the particles separated very easily when the apparatus was dismantled; sample 3 exhibited no sintering.

#### 4.1.1.3 Conclusions

One of the key questions to be answered by these measurements was the degree of dependency of  $k_e$  upon compaction pressure. As is seen in Figure 68, the highest pressures resulted in  $k_e$  values that were consistently slightly higher than the lower pressure results. However, this difference can be neglected for most practical applications.

#### 4.1.2 Specific Heat

Catalyst specific heat was determined, as a function of temperature, for a single sample of 20-30 mesh catalyst, vacuum sealed inside a container of known mass and specific heat. Unlike thermal conductivity, it is believed that the data from one sample is of sufficient accuracy for adequate thermal modelling. The influence of particle size, if any, should be second order if specification metal-loading tolerances are maintained in the catalyst.

TABLE 16  
THERMAL CONDUCTIVITY OF SHELL 405 ABSG CATALYST  
(AS A FUNCTION OF TEMPERATURE AND COMPRESSIVE LOAD)

| Sample No.              | Mean Temp (°F) | $K_e$ (BTU hr <sup>-1</sup> ft <sup>-1</sup> °F <sup>-1</sup> )<br>At Applied Load |                       |                       |
|-------------------------|----------------|------------------------------------------------------------------------------------|-----------------------|-----------------------|
|                         |                | 30 (psi)                                                                           | 100 (psi)             | 250 (psi)             |
| 1<br>(20-30 mesh)       | 1544 ± 18      | 0.139                                                                              | 0.144                 | 0.153                 |
|                         | 1177           | —                                                                                  | 0.116                 | —                     |
|                         | 959 ± 13       | 0.095                                                                              | 0.092                 | 0.104                 |
|                         | 766            | —                                                                                  | 0.081                 | —                     |
|                         | 374 ± 9        | 0.046                                                                              | 0.052                 | 0.058                 |
|                         | Vacuum*        | <3 × 10 <sup>-4</sup>                                                              | <5 × 10 <sup>-4</sup> | <5 × 10 <sup>-4</sup> |
| 2<br>(14-18 mesh)       | 1551 ± 7       | —                                                                                  | 0.191                 | 0.196                 |
|                         | 1175           | 0.144                                                                              | —                     | —                     |
|                         | 977            | —                                                                                  | 0.130                 | —                     |
|                         | 788            | —                                                                                  | —                     | 0.115                 |
|                         | 568            | 0.072                                                                              | —                     | —                     |
|                         | 392            | —                                                                                  | 0.061                 | —                     |
|                         | Vacuum*        | <7 × 10 <sup>-4</sup>                                                              | <5 × 10 <sup>-4</sup> | <8 × 10 <sup>-4</sup> |
| 3<br>(1/8 inch pellets) | 1580           | —                                                                                  | 0.292                 | —                     |
|                         | 970 ± 2        | 0.168                                                                              | 0.170                 | 0.176                 |
|                         | 378            | 0.064                                                                              | 0.064                 | —                     |
|                         | Vacuum*        | <8 × 10 <sup>-4</sup>                                                              | <7 × 10 <sup>-4</sup> | 10 <sup>-3</sup>      |

\*torr  
—47-50°F Gradient Across Sample

TABLE 17  
MEASURED SAMPLE THICKNESS

| Sample                 | $\Delta X$ (inch) at<br>Applied Load (psi) |       |       |
|------------------------|--------------------------------------------|-------|-------|
|                        | 30                                         | 100   | 250   |
| 1<br>(20-30 mesh)      | 0.364                                      | 0.362 | 0.356 |
| 2<br>(14-18 mesh)      | 0.411                                      | 0.407 | 0.402 |
| 3<br>(1/8 in. pellets) | 0.374                                      | 0.370 | 0.368 |

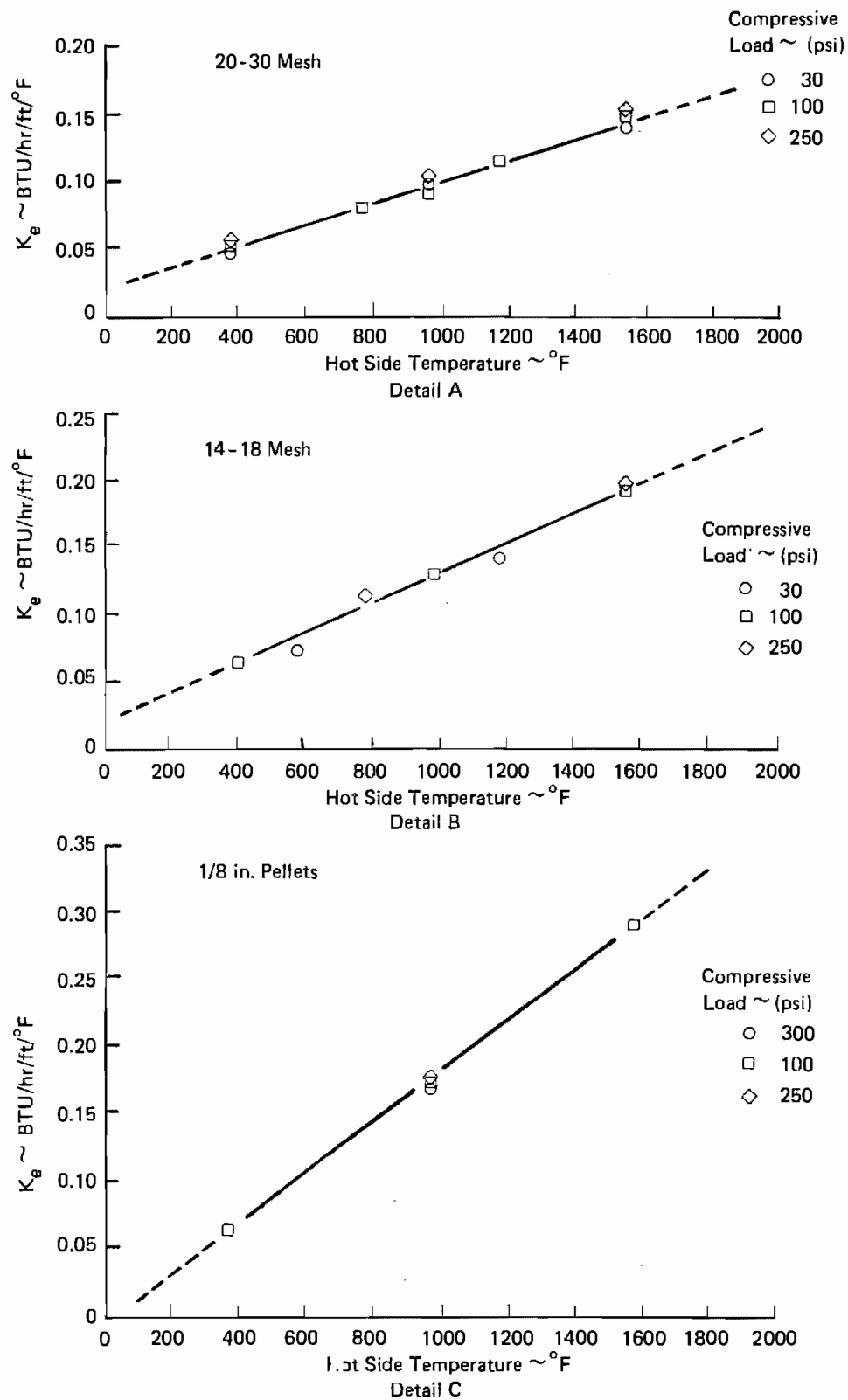


Figure 68. Thermal conductivity of Shell 405 ABSG catalyst (47-50°F gradient)

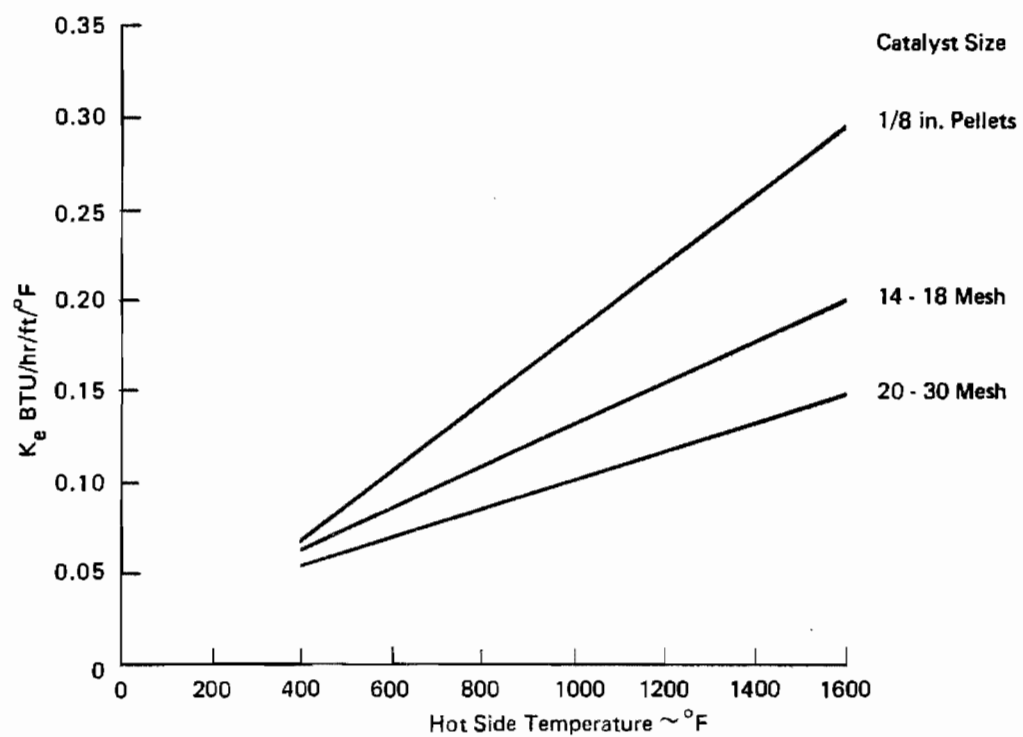


Figure 69. Catalyst size effect on thermal conductivity.

#### 4.1.2.1 Test Method

The test equipment (a standard drop calorimeter) consists of a furnace for heating the specimen, an insulated, calibrated receiver (copper chamber), a drop mechanism which holds the specimen in the furnace and drops it through a connecting tube into the receiver. The connecting tube has "flapper" radiation shields which prevent heat loss from the receiver and the furnace. Control and temperature measurement equipment is also provided.

The specimen was heated to the required temperature and dropped into the copper receiver. The temperature increase of the receiver was measured. Since the mass and specific heat of the receiver was known, the heat content of the test specimen can be determined from the temperature increase of the receiver. The specific heat is determined by measuring the slope of the heat content - temperature curve at different temperatures:

$$H = MC_p (T_F - T_{RI})$$

where:

H = Specimen heat content from initial specimen temperature to final equilibrium temperature (BTU).

M = Mass of receiver (lbs).

C<sub>p</sub> = Specific heat of receiver (BTU/lb°F)

T<sub>F</sub> = Final equilibrium temperature of specimen - receiver system (°F).

T<sub>RI</sub> = Initial receiver temperature (°F).

The test sample consisted of 36.4 grams of 20-30 mesh Shell 405 vacuum packed into a columbium (Cb-291) container. Prior to loading, the container's specific heat was determined at Dynatech by the identical procedure that was subsequently used on the loaded unit.

#### 4.1.2.2 Results

The resulting specific heats are presented in Table 18 and are plotted in Figure 70. The regression curve presented has the following statistical parameters:

|                      |                                                                        |
|----------------------|------------------------------------------------------------------------|
| Multiple Correlation | = 0.98                                                                 |
| Standard Error       | = 0.0028 $\left( \frac{\text{BTU}}{\text{lb } ^\circ\text{F}} \right)$ |
| Sample Size          | = 8                                                                    |

As a verification of these results, specific heat values corresponding to two of the tested temperatures were computed by appropriately weighting published values of the specific heats of the catalyst constituents. Good agreement was obtained.

TABLE 18  
SPECIFIC HEAT OF 20-30 MESH SHELL 405 ABSG  
CATALYST AT VARIOUS TEMPERATURES

| Temperature<br>(°F) | C <sub>p</sub><br>(BTU/lb/°F) |
|---------------------|-------------------------------|
| 212                 | 0.170                         |
| 392                 | 0.180                         |
| 572                 | 0.190                         |
| 752                 | 0.201                         |
| 932                 | 0.203                         |
| 1112                | 0.205                         |
| 1292                | 0.205                         |
| 1472                | 0.208                         |

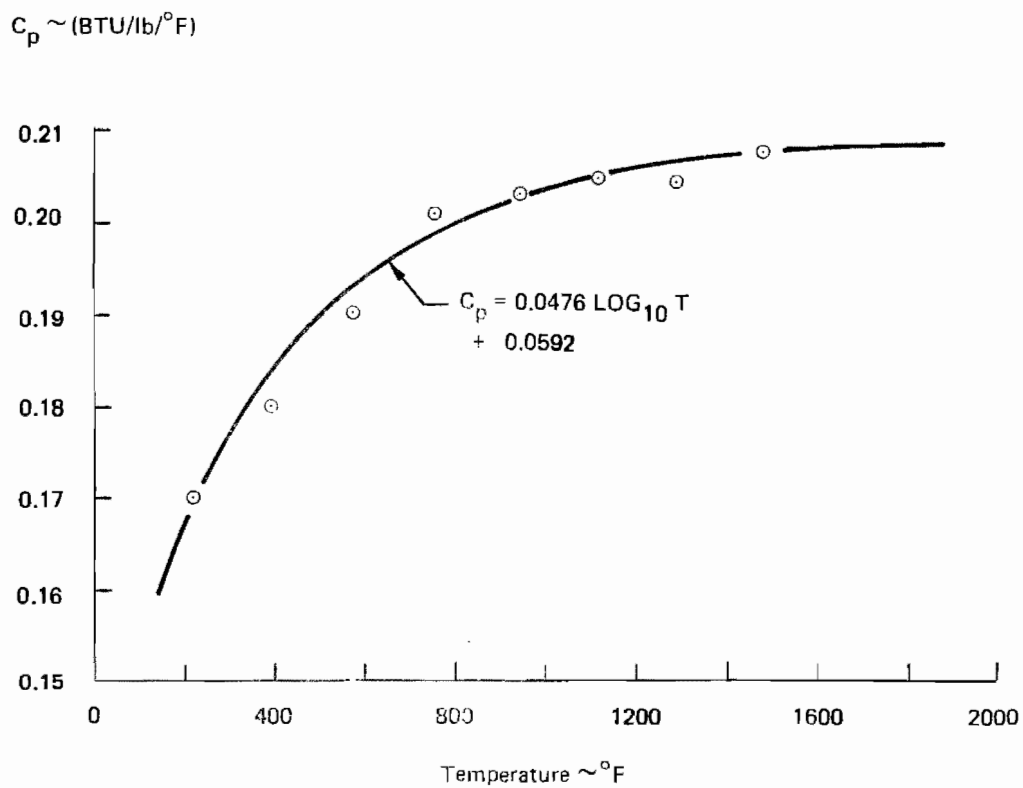


Figure 70. Specific heat of 20-30 mesh Shell 405 ABSG catalyst versus temperature.

## 4.2 EFFECTS OF DETONATIONS IN PROPELLANT MANIFOLDS

As a part of the injector design evaluation, a series of tests to evaluate the effect of detonations occurring in propellant manifolds was undertaken. A study of the potential energy available from decomposition of hydrazine available in the injector manifold of a 5 lbf thruster led to the conclusion that the maximum conceivable level was about 400 ft-lbs. Combining this, then, with an over-pressure of about 20:1 - - a level at which most flight weight manifolds would have failed - - should produce a severe if not extreme test of the importance of this mechanism.

### 4.2.1 Approach and Test Equipment

A Pulse Unit, wherein small powder charges, simulating hydrazine, were deliberately detonated upstream of the injector of an experimental upper bed assembly, containing 20-25 mesh Shell 405 ABSG, was conceived. The configuration of this unit is shown in Figure 71. It consisted essentially of a heavy-weight housing and detonator assembly, which fitted around and sealed against a SUB\* with the propellant manifold removed. To determine effects of injector orifice size, an injector containing 0.0036, 0.0063, and 0.0103 inch diameter orifices in three sectors, as shown in Figure 72, was used.

The shock-pulse from the detonated powder charge was admitted into, and traveled transversely across, the cavity behind the injector. Provision was made, via a threaded plunger and spacers of differing lengths, to vary the volume of the cavity over a 5:1 ratio. This permitted adjustment of the overpressure level achieved. The resultant pressure surge was monitored by a high-response transducer close-coupled through the sensing port. Facility provisions (not shown in the illustration) were made to admit water to the SUB, for filling it to a level upstream of the injector face, when desired, and to vent the cavity below the SUB.

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\* Sacrificial Upper Bed, See Section 2. for a general description.

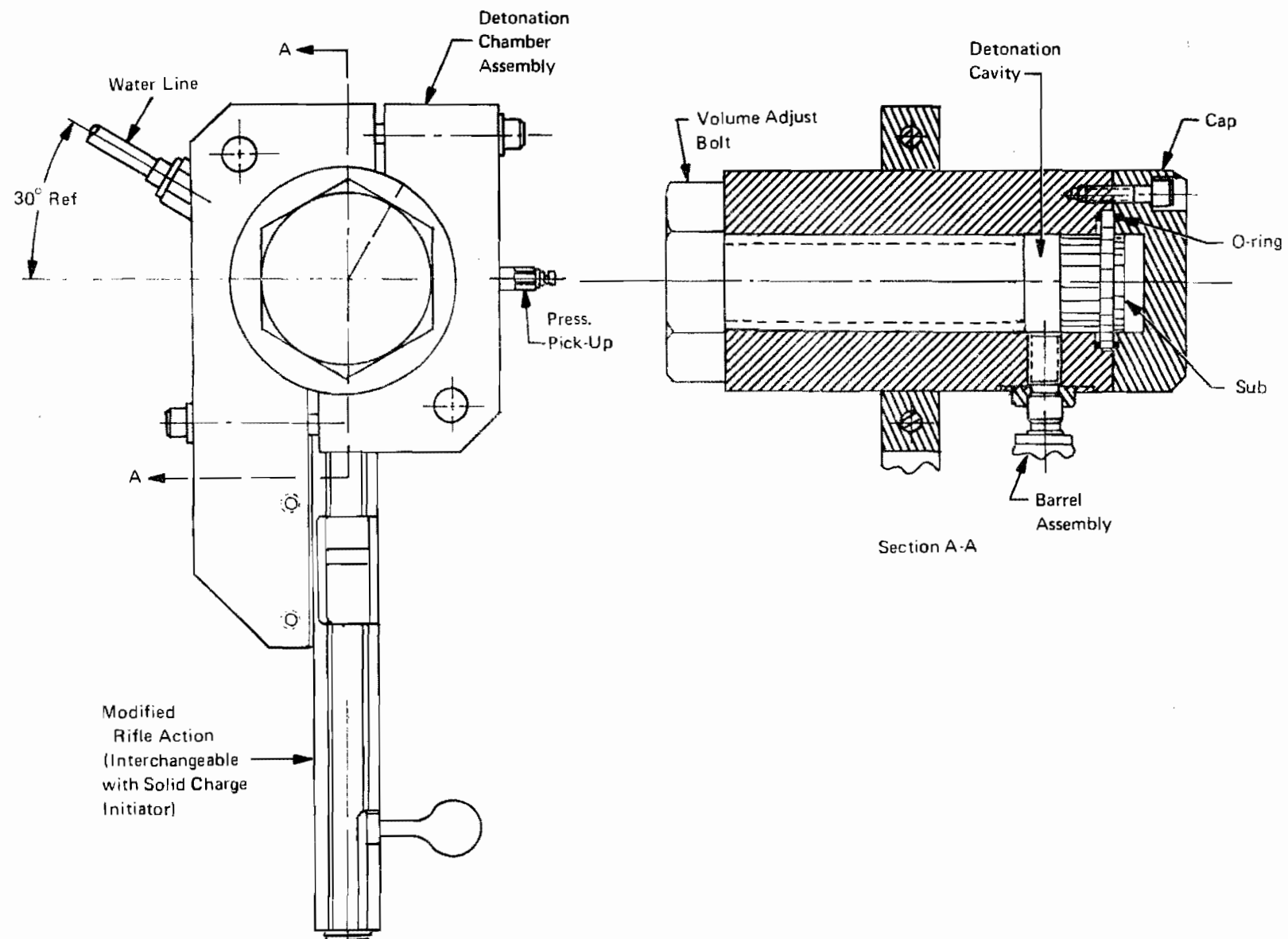


Figure 71. Pulse unit assembly

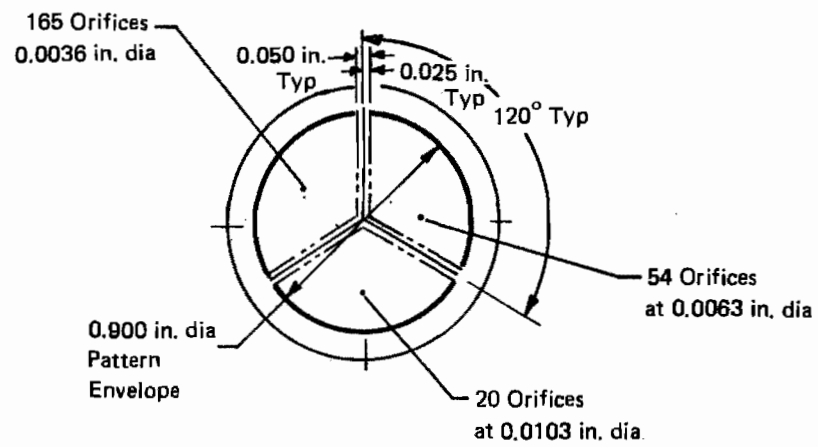


Figure 72. Injector plate (uniform orifice distribution within each 120° sector pattern envelope).

Detonation energy was determined by the size and composition of the Powder charge employed. Commercially available 0.22 calibre Ramset ammunition provided seven repeatable charge levels between 55 ft-lb and 368 ft-lb. This was consistent with the energy levels which could conceivably occur in the propellant manifold of a 5 lbf thrust hydrazine reactor.

#### 4.2.2 Manifold Detonation Tests

A series of up to ten tests was planned, as shown in Table 19. The first two tests were done at the maximum energy level (368 ft-lb), one on a "dry" SUB and the other with the SUB filled with water to simulate the hydraulic condition of liquid propellant within the injector. Figure 73 shows a typical manifold pressure trace produced by these changes. Peak pressure, as measured by a high-response Kistler transducer, was reached in about 0.1 millisecond.

The ten pulses at an energy level of 368 ft-lb and an average overpressure of 3,778 psi did little damage to the catalyst in a dry chamber. Weight loss was less than 1%, (within measurement accuracy) and no significant voids or fracturing was observed after sectioning.

However, the second unit, exposed to the same conditions,\* but with injector and bed filled with water, showed a weight loss of 2.8%. Large voids beneath the injector face and a significant degree of particle breakup were observed. The degree of attrition, as seen in the sectioned bed, was much greater below the 0.0103 inch diameter orifices (Figure 74).

It is interesting that the breakup seen in this test is not characteristic of that in the firing tests, the difference being in particle sizes. A definite bimodal particle size distribution exists in fired beds, but those subjected to the "cold" overpressure did not tend to break down to the very small particles (50-80  $\mu$ ) seen in that secondary distribution.

The third manifold detonation test was done at an energy level of 55 ft-lb - a level much more representative of actual conditions - to see if this was below a threshold level. A "wet" bed was used since this had been determined to be the worst condition.

Ten pulses at the 55 ft-lb energy level produced an average overpressure of 221 psi and resulted in no visible catalyst damage or significant catalyst weight loss (0.2%). Figure 74 compares the damage from the 368 ft-lb test with this similar test at 55 ft-lb. The large cavity toward the right in the lower photograph was situated under the large (10.3 mil) orifices.

The fourth test was done at an intermediate value of 212 ft-lb to better define the attrition threshold. Ten pulses at this level produced an average overpressure of 864 psi and no visible damage to the catalyst. A 70 milligram weight increase was measured. But this was insignificant - 0.08% in 9.16 gram catalyst weight.

It was concluded that further testing was not justifiable in that the threshold of damage to the catalyst bed was well above the level where damage to flight weight hardware would be expected.

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\*Average overpressure was 3849 psi, 2% higher than the first test because of small volume changes caused by the water and variability in the Ramset ammunition.

**TABLE 19**  
**MANIFOLD DETONATION TESTS**

Constant Operating Conditions:

- Ramset Powder Charge
- 20-25 Mesh Shell 405 ABSG
- Ambient Conditions

| Test No. | SUB S/N | Injector/Catalyst Condition       | Energy Level, ft-lb | No. of Pulses |
|----------|---------|-----------------------------------|---------------------|---------------|
| 1        | MD-1    | Dry                               | 368                 | 10            |
| 2        | MD-2    | Wet                               | 368                 | 10            |
| 3        | MD-3    | Wet*                              | 55*                 | 10            |
| 4        | MD-4    | Wet*                              | 212*                | 10            |
| 5        | MD-5    | Deleted based on first four tests |                     | 1             |
| 6        | MD-6    |                                   |                     | 3             |
| 7        | MD-7    |                                   |                     | 10            |
| 8        | MD-8    |                                   |                     | 1             |
| 9        | MD-9    |                                   |                     | 3             |
| 10       | MD-10   |                                   |                     | 10            |

\*Decided on basis of earlier test.

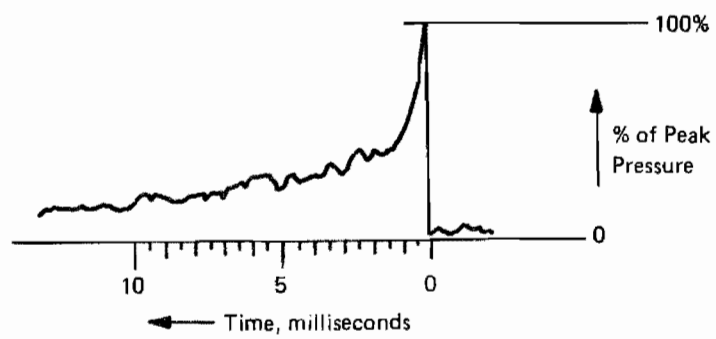
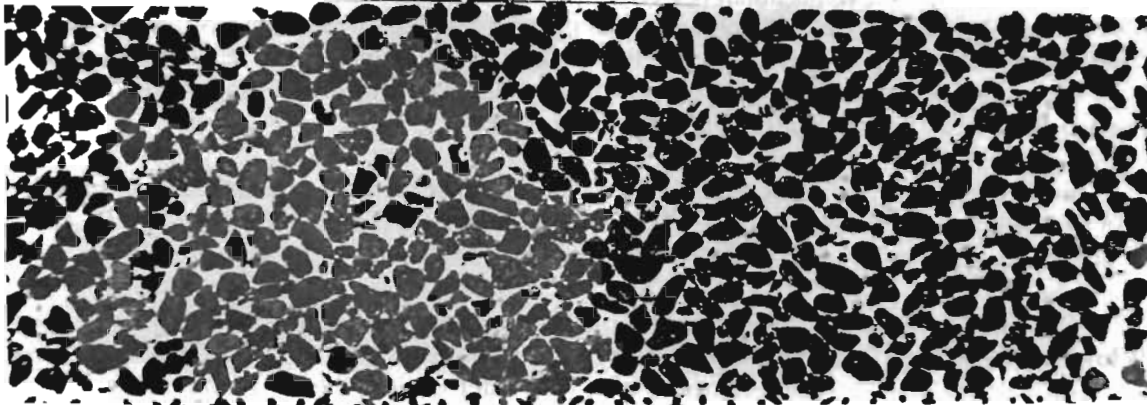


Figure 73. Typical manifold pressure trace produced by Ramset charges.

(Injector End)



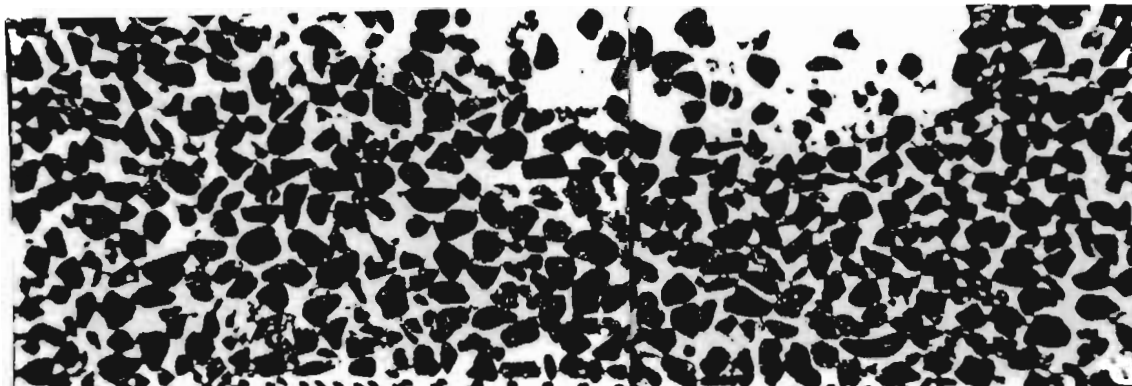
Energy = 55 ft-lb

Average Peak Pressure = 236 psia

0.0036" Orifices

0.0103" Orifice

(Injector End)



Energy = 368 ft-lb

Average Peak Pressure = 3849 psia

Figure 74. Catalyst Bed Cross-Sections after High and Low Energy - Pulse Exposures (Wet).

#### 4.2.3 Observations and Conclusions

The 368 ft-lb level seems much too severe for realistic thruster designs. An energy release of this magnitude in a confined volume such as an injector manifold would almost certainly cause hardware damage to a degree that damage to the catalyst would be incidental. Further, it would require the release of the total available energy in about 0.01 cubic inch (the order of the hold-up volume in 5 lbf class thrusters) of liquid hydrazine at 70°F.

Since detonation or propagation of detonation in liquid hydrazine seems highly improbable, the 55 ft-lb condition is more reasonable. This case approximates the situation where a 0.01 cubic inch mixture containing 85% saturated hydrazine vapor at 450°F and 275 psia is detonated.

The effect of orifice size is apparent at the high energy level, but any influence at lower levels is below the resolution of this experiment. The relationship shown in Figure 75 is indicated. However, within the region where the hardware would withstand a detonation, the situation is academic in that no significant catalyst attrition is expected from this mechanism.

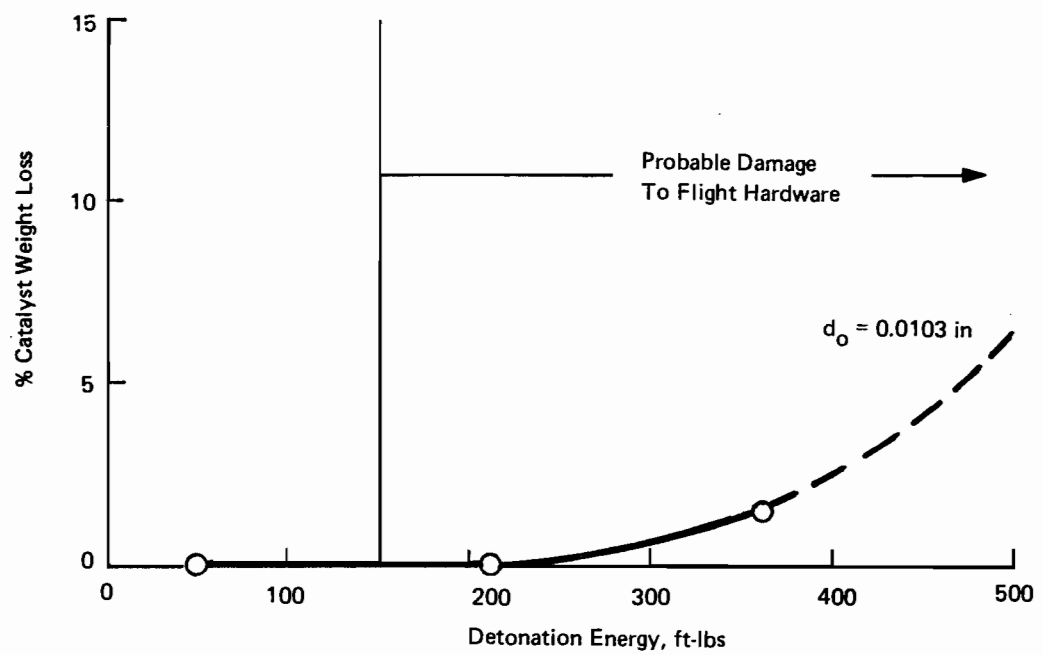


Figure 75. Effect of propellant detonation in a liquid-filled injector manifold.

### 4.3 Predicting Temperature Distribution Within a Catalyst Bed

In order to understand the thermal behavior of hydrazine catalyst bed reactors (i.e., hardware and catalyst temperature response during firing), two temperature-instrumented reactors of different sizes were thermally modeled and test fired.

To obtain more accurate temperature measurements in the axial and radial direction of the catalyst bed, the first reactor—called the Conductometric Reactor Assembly (CRA)—was large, with a bed diameter and bed length approximately twice that of the standard SUB. This reactor was of a flight-type design having approximately a 25 lbf - thrust level. Transient and steady-state temperature data, obtained on this CRA were then employed to correct a thermal model for true reactor effects.

Subsequently a second thermal reactor, designated TM01, was fabricated and tested. It was essentially a standard ARA (5 lbf thrust level) containing a temperature - instrumented upper catalyst bed (SUB). It operated under the same conditions of mass flux, injection velocity and number pressure as the first reactor. This unit was fired to obtain temperature measurements to verify the predictions of the upgraded thermal model.

Both units were subjected to a firing of duration sufficient to allow steady-state temperatures to be reached. Heat soakout data was taken after the firings.

The thermal models of the reactors were established using a generalized three-dimensional multi-material transient heat transfer program. A description of this program is given in Appendix C.

The program requires that the analyst subdivide the structure being analyzed into elements or nodes and input the volumes, thermal connections between nodes, material properties, and boundary conditions. This is also described in detail in the Appendix.

Thermal property measurements on Shell 405 catalyst, as obtained by Dynatech, was described in Section 4.1. The following section discusses the boundary conditions assumed for the thermal modeling, and summarizes the test conditions and results. Extensive graphical presentations of the measure temperature - time histories throughout the catalyst bed, and at the injector face and retainer plate, during and immediately after the firing (heat-soak) are presented. Test results will be compared with the analytical predictions.

#### 4.3.1 Boundary Conditions

The major problem with thermal modeling of the hydrazine catalyst bed is establishing the boundary conditions produced by the decomposed hydrazine, i.e., the driving temperature and heat transfer coefficients:

##### 4.3.1.1 Heating of the Catalyst

Theoretical analysis of catalyst bed reactors,<sup>(1)</sup> indicate that as the fuel progresses through the bed length, it changes to the vapor phase and decomposes in a distance of less than 0.15 inch. Further travel

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(1) Kesten, A.S., "Analytical Study of Catalytic Reactors for Hydrazine Decomposition," UARL F910461-12 May, 1967.

through the bed causes a slow increase in ammonia dissociation with resulting bed temperature decrease (due to this exothermic reaction). Hence the axial temperature distribution in the catalyst bed is not uniform. Also, tests have shown that the radial temperature distribution is nonuniform. As illustrated later, higher temperatures were recorded near the chamber walls. Therefore, in order to model this system an arithmetic average temperature was selected at each station. Figure 76 shows the assumed axial temperature profile; also illustrating the known "quenching" effect, that occurs immediately after shut-down (dashed line). This effect results when hydrazine vaporizes within the bed, and is more pronounced near the injector face where a sudden, drastic drop in temperature occurs. This was the "initial condition" assumed for the heat-soak cycle.

The heat transfer coefficient between the hydrazine gases and the catalyst is determined from the expression<sup>(1)</sup>:

$$h_c = 0.74 (G/A_p \mu)^{-0.41} (\bar{C}_p G)$$

where

$h_c$  heat transfer coefficient - BTU/in.<sup>2</sup> sec °F

$G$  flow rate per unit area - lb/in.<sup>2</sup> sec

$\bar{C}_p$  specific heat - BTU/lb °F

$A_p$  catalyst surface area per unit volume - 1/in.

$\mu$  viscosity - lb/in. sec

Incidentally, using the heat transfer coefficients obtained from this equation and the driving temperature given in Figure 76, it was later found that, in order to match the response of the bed thermocouples, the effective area for heat transfer to the bed material was only 8.3% of the total surface area based on the values of  $A_p$ .

#### 4.3.1.2 Reactor Walls

The heating rates to the reactor walls were determined by assuming 50% of the heat transfer coefficient which was predicted between the hydrazine decomposition gases and the catalyst. Local temperatures, as shown in Figure 76, were again assumed.

#### 4.3.1.3 Retainer Plate

The retainer plate receives heat from the combustion gas flow through the orifices. First, the heat transfer coefficient through an equivalent pipe diameter was calculated:

$$h_c = 0.023 (W^\circ/A)^{0.8} 1/D^{0.2} \frac{\bar{C}_p^{0.4} K^{0.6}}{\mu^{0.4}}$$

(1) Kesten, A.S., "Analytical Study of Catalytic Reactors for Hydrazine Decomposition," UARL F910461-12 May, 1967.

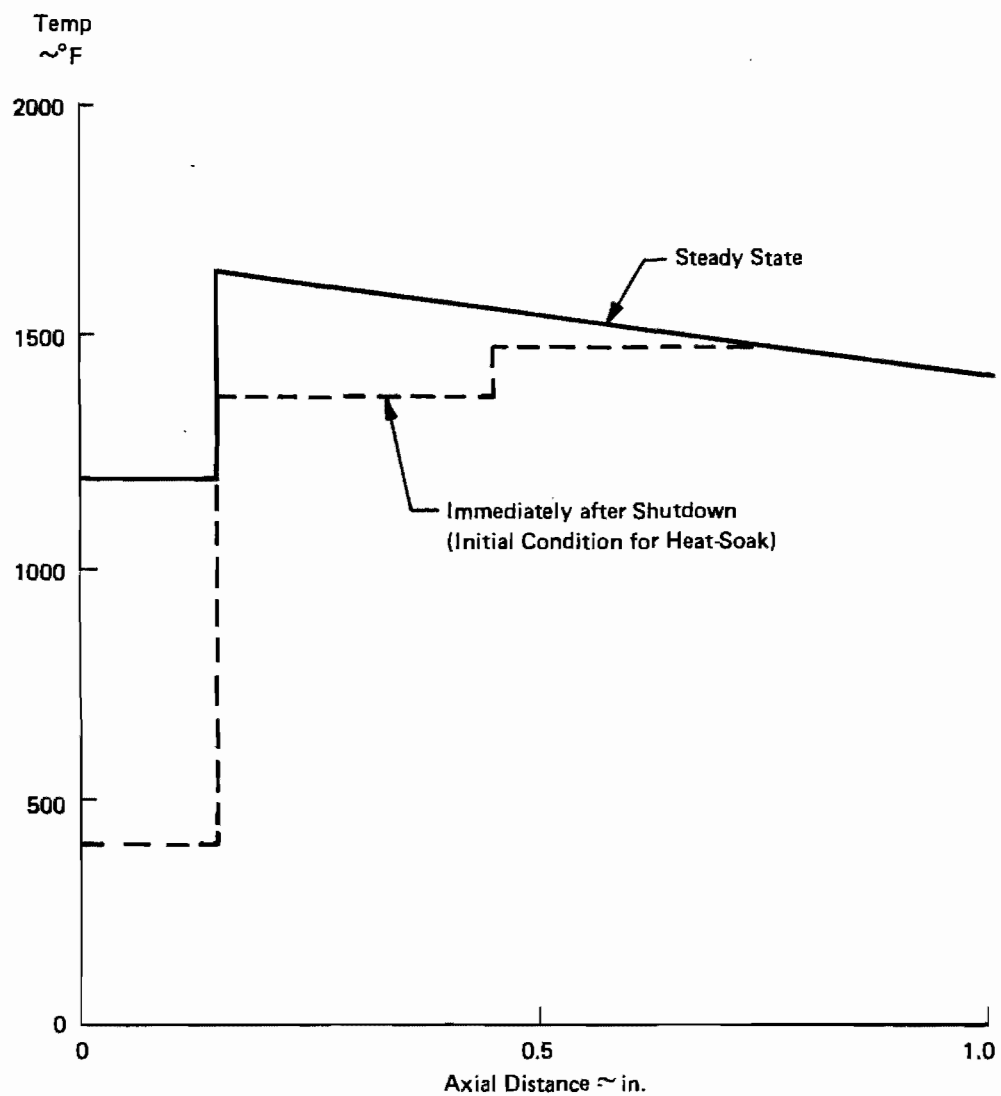


Figure 76. Assumed driving temperature for thermal model.



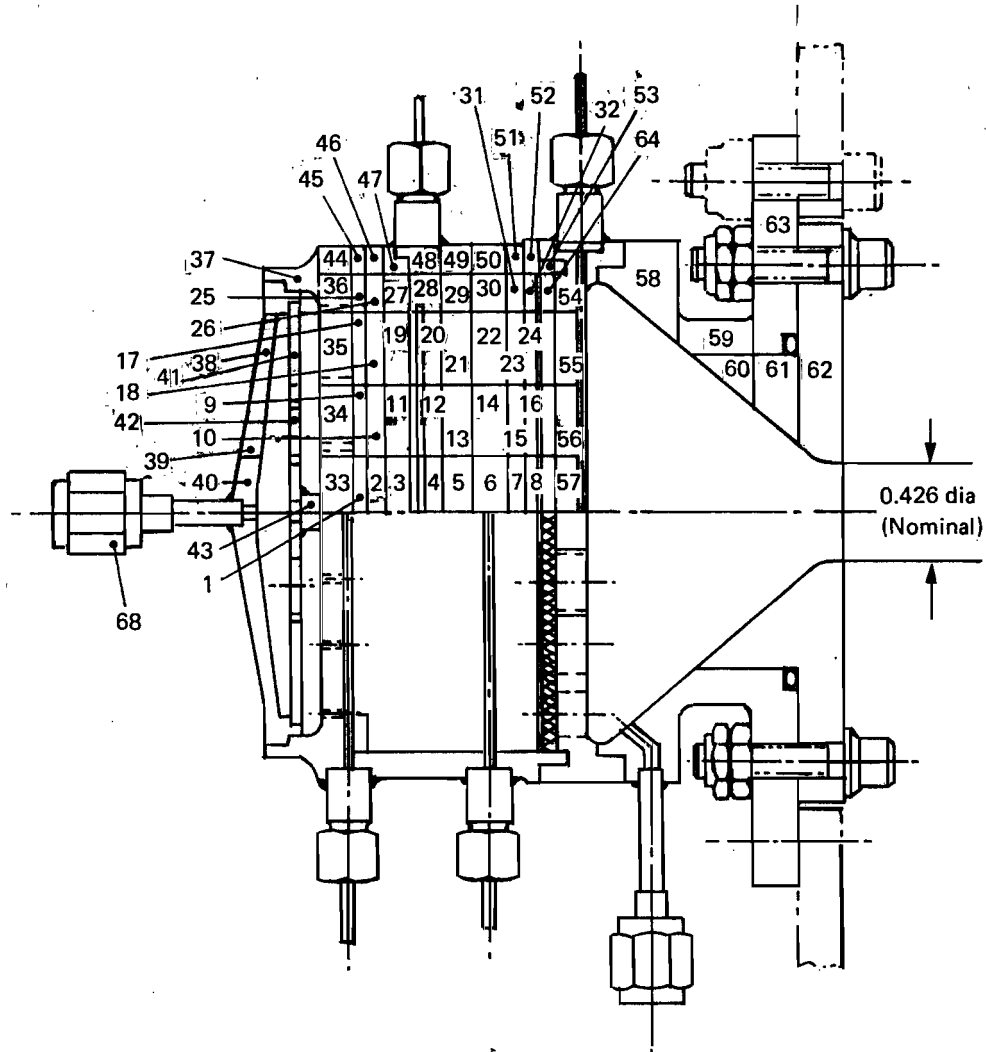


Figure 77. Node locations for thermal model of the CRA reactor.

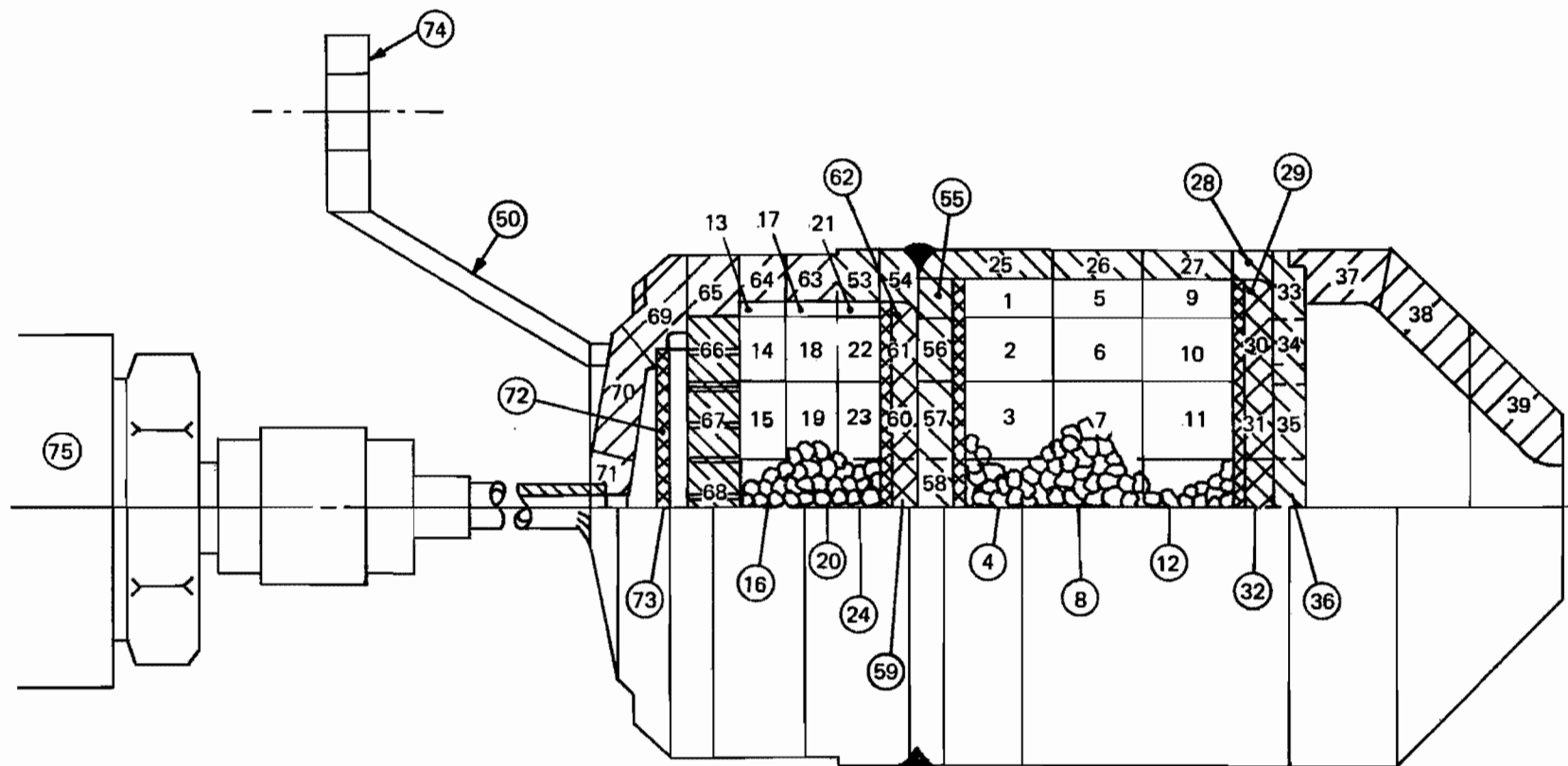


Figure 78. Node locations for thermal model of the TM01 reactor.

#### 4.3.2.1 Test Hardware and Instrumentation

The CRA reactor was of flight-type design (thin walls, welded construction), having a 25-lbf thrust level at a bed-exit chamber pressure of 100 psia. The injector was of a flat-face design, embodying a uniform pattern of thirty-six 0.031-inch-diameter orifices. It had a superficial mass flux of 0.02 lbm/in<sup>2</sup> sec at a nominal flowrate of 0.11 lbm/sec.

The catalyst bed consisted of 20-30 mesh Shell 405 ABSC, contained with the injector and an aft retainer plate/screen assembly, as indicated in Figure 79. Also illustrated in this figure is the 4 x 4 radial/axial matrix of the thermocouple locations, inside the bed and on the outside of the chamber and injector. It should be noted that four thermocouple junctions were at the injector face: (1) Three tack welded between injector and screen and the fourth also at the injector, but with no screen present (a small section, a "window," of the screen was removed for that purpose). The intent was to check for screen effects on the catalyst/injector heat transfer.

#### 4.3.2.2 Operating Conditions

The unit was fired in the altitude chamber, evacuated to an equivalent altitude of 100,000 ft. The test sequence consisted of a steady-state firing, for at least 100 seconds, so that all thermocouples had reached steady state. After shutdown, the reactor was allowed to cool down naturally in vacuum, during which time all reactor thermocouples (bed and skin) continued to monitor temperatures, gathering a heat-soak time history.

#### 4.3.2.3 Thermal Test Results and Predictions

The thermal test data and the predictions are presented as radial temperature-time histories at the selected axial stations along the catalyst bed; starting at the injector face. Subsequently, the respective temperature - time histories (sampled data) during engine shutdown will be discussed.

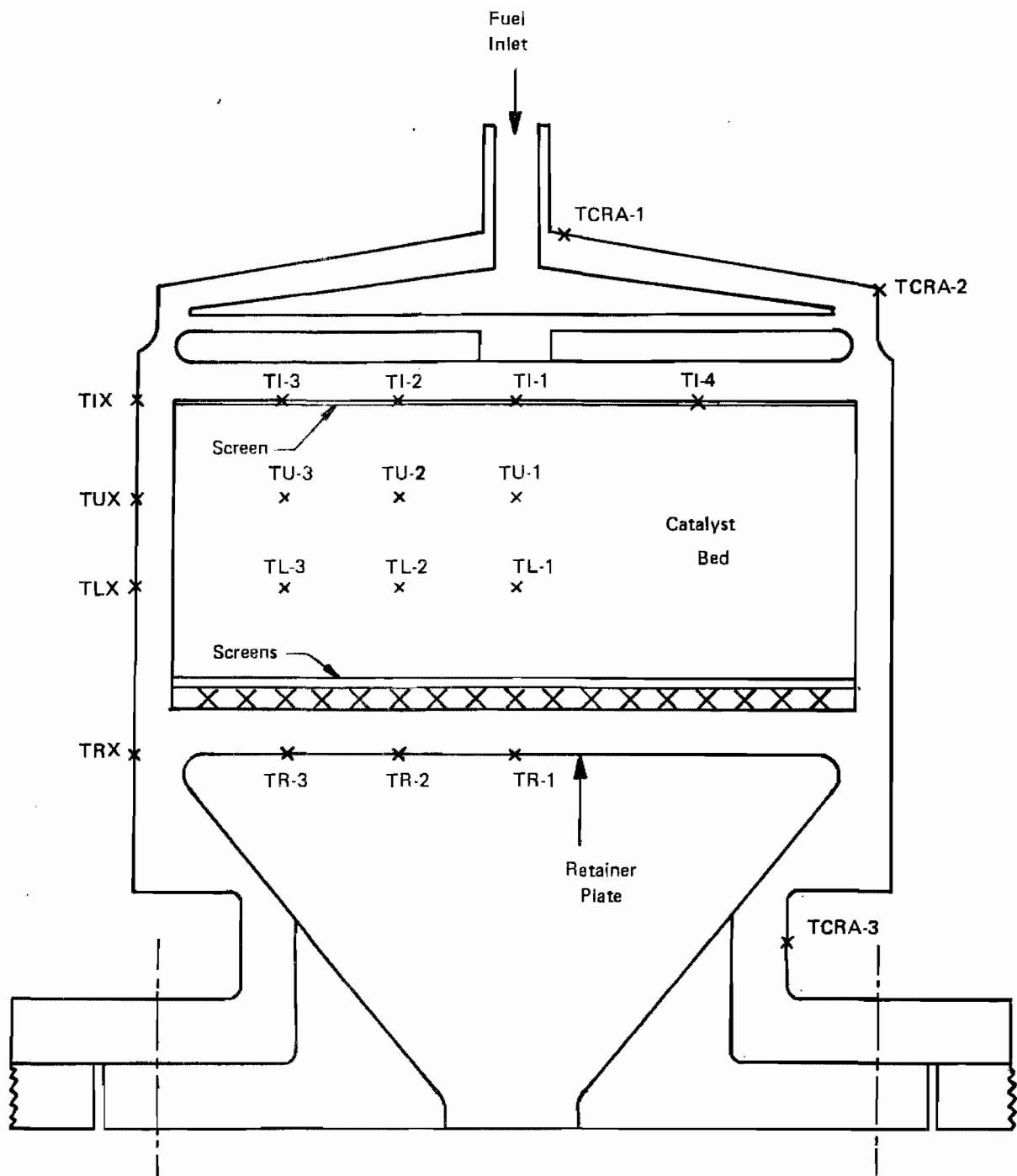
Figure 80 shows the temperature response of thermocouples at the injector from test no. 995 (102 seconds of firing). The data from that test compares very well with that from test no. 994 (a 68 second checkout firing).

As noted in Figure 80 the thermocouples come to an equilibrium temperature plateau of about 270°F in less than 1 sec - this may be explained by considering the thermocouple to be heated by recirculation gases and cooled by conduction to the injector. The thermocouple (TI-4)\*, which had the wire mesh screen cut-out at this location, read approximately 60°F lower than the other three injector thermocouples. This may be caused by this particular thermocouple receiving less combustion gas heating and thus achieving lower equilibrium temperature. At some point in the firing cycle, this thermocouple may have broken contact with the injector face and began to measure the local gas temperature.

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(1) These thermocouples were mounted in radial slots on the injector face and were bent so that their junction was firmly pressed against the surface.

\* Not plotted



Scale 4/1

Figure 79. Conductometric reactor assembly thermocouple locations.

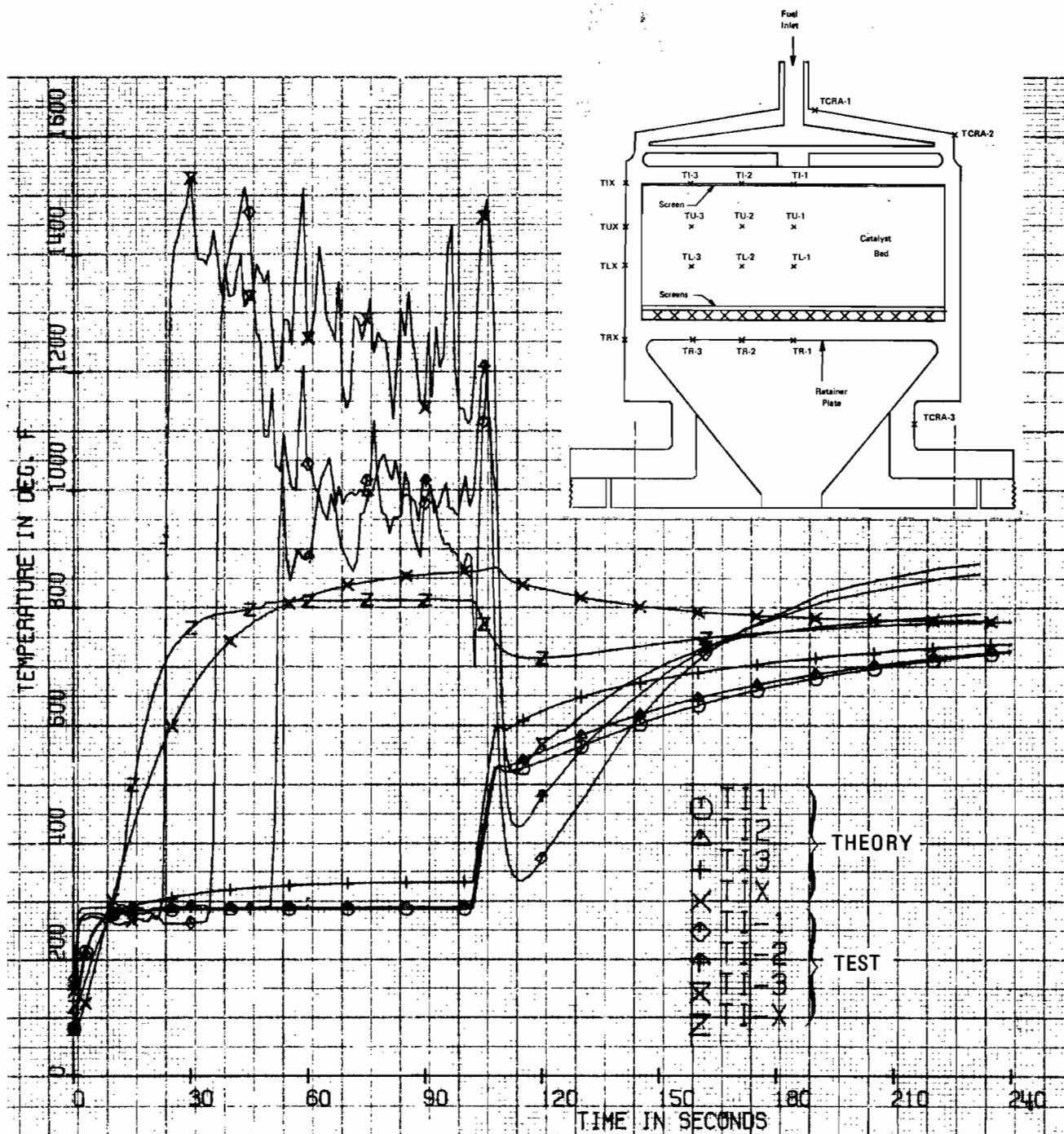


Figure 80. Temperature time history of selected nodal points  
CRA Run No. 995-Injector.

Thermocouple TI-3 suddenly rises to 1500°F after 22 seconds of firing, followed by TI-1 and TI-2 at 35 and 50 seconds, respectively. Thermocouple TI-4\* rises at 90 seconds to 700°F. During the course of firing, the TI-thermocouples show a gradual reduction in average temperature readings. Upon shutdown at 102 seconds, the thermocouples register a heat pulse and quickly decay to measure the injector temperature. The injector temperature has risen to between 320 to 520°F during the 102 second heating period. During the previous test (994), a similar rise in temperature was experienced for the 69-second firing. Thus the injector has come to an equilibrium temperature at some time less than 68 seconds. In terms of radial temperature distribution, the outer third of the injector is hottest and the center is coldest. Upon shutdown the injector temperature rises to a peak value of 890°F at the TI-1 location, at 260 seconds and then slowly decays. The thermocouple at the TI-4 location (without screening) reads nominally 260°F less than the other thermocouple locations on the injector face. This, in itself, would infer that the heat transferred from the catalyst bed to the injector is reduced, or possibly, that the heat radiated from the surroundings in a near vacuum in comparison to the heat conducted through the thermocouple leads, thus causing the thermocouple to read low.

Also shown in Figure 80 is the theoretical temperature response of the injector nodes corresponding to the test data. Since there is a question as to what the thermocouple measured at the injector face, a second figure, Figure 81, was plotted showing the comparison of the same injector thermocouples with the theoretical catalyst bed temperature closest to the injector, i.e., at a distance of approximately 0.035 inch from the screen.

The temperatures in the catalyst bed achieve 90% of the steady-state values in approximately one second.

Figure 82 shows the temperature response of the "U-location" thermocouples in the catalyst bed. The temperatures peak after two seconds at about 1700°F and drop as much as 250°F from the peak value. The catalyst bed temperatures approach a steady-state reading after 50 seconds. The data show that the outer segment of the bed is hottest and the bed center is coldest. Upon reactor shutdown the temperatures within the bed drop steeply (300°F in 7-seconds) - this is indicative of a quenching effect caused by the stored propellants upstream of the injector being vaporized and decomposed at a different rate. For comparison, the theoretical temperature response (based on the preliminary thermal model) at the "U-location" nodes is also included in Figure 82. The disagreement is obvious, which probably indicates inaccurate assumption in the input to the thermal model.

The experimental and predicted temperature response at the lower region of the catalyst bed ("L-location") is presented in Figure 83. It can be seen that the catalyst temperatures rise to within 1400 - 1550°F in two seconds. Upon shutdown the bed temperature drops sharply by 100-200°F, which is less than at the "U-location." You will also note that a better agreement between the predicted and experimental profile was obtained.

Figure 84 shows the response of the retainer plate thermocouples which rise to 1420°F and slowly decay upon shutdown as heat is conducted to the cooler nozzle section.

Good agreement between the experimental and predicted temperature response was achieved at this location. However, a poorer agreement resulted with the temperatures on the outside wall locations (i.e., TCRA-1, -2, -3): as shown in Figure 85; yet the overall trend in temperature/time response was

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\* Not plotted

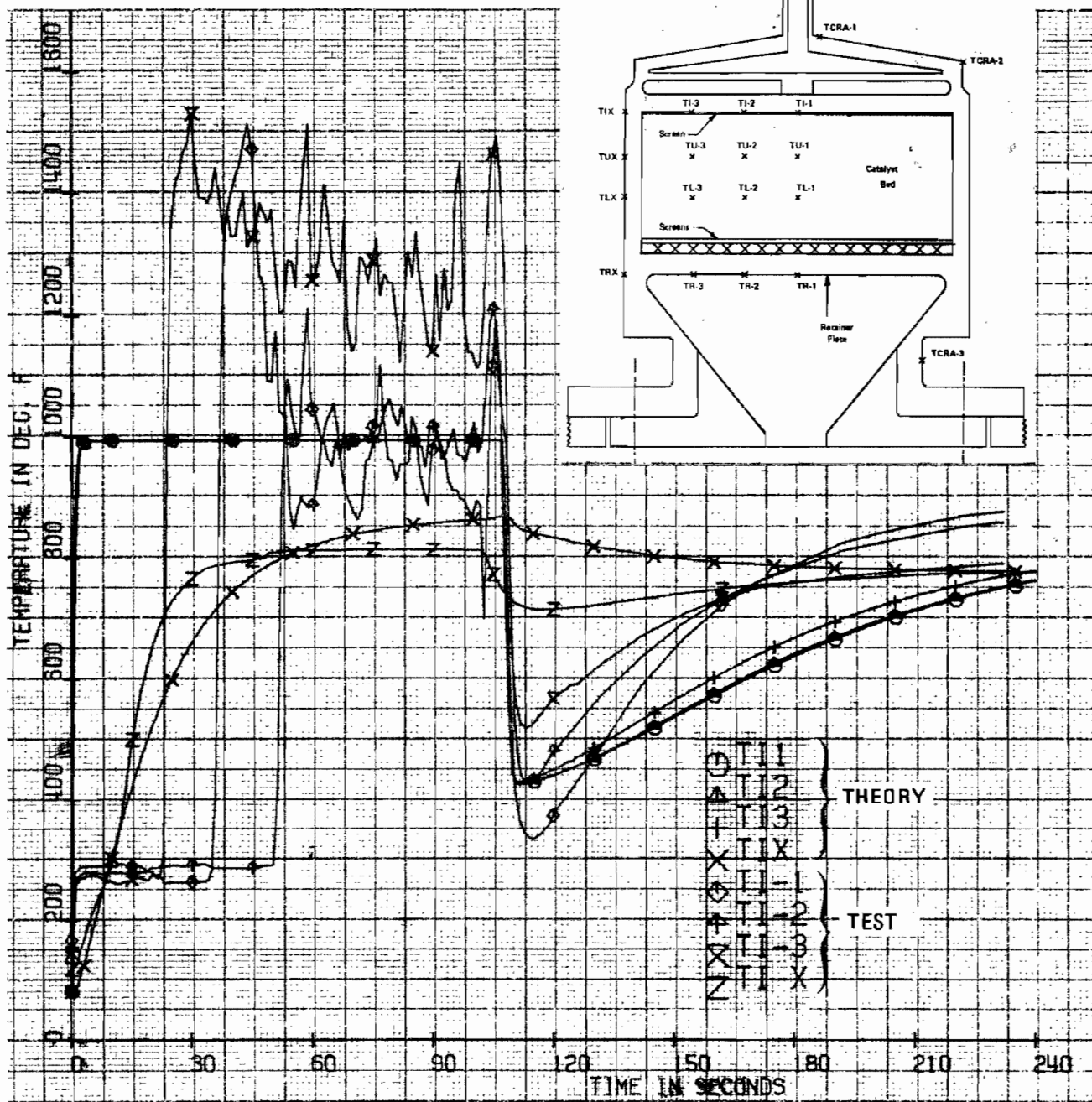


Figure 81. Temperature time history of selected nodal points.  
CRA Run No. 995 - Injector/Bed

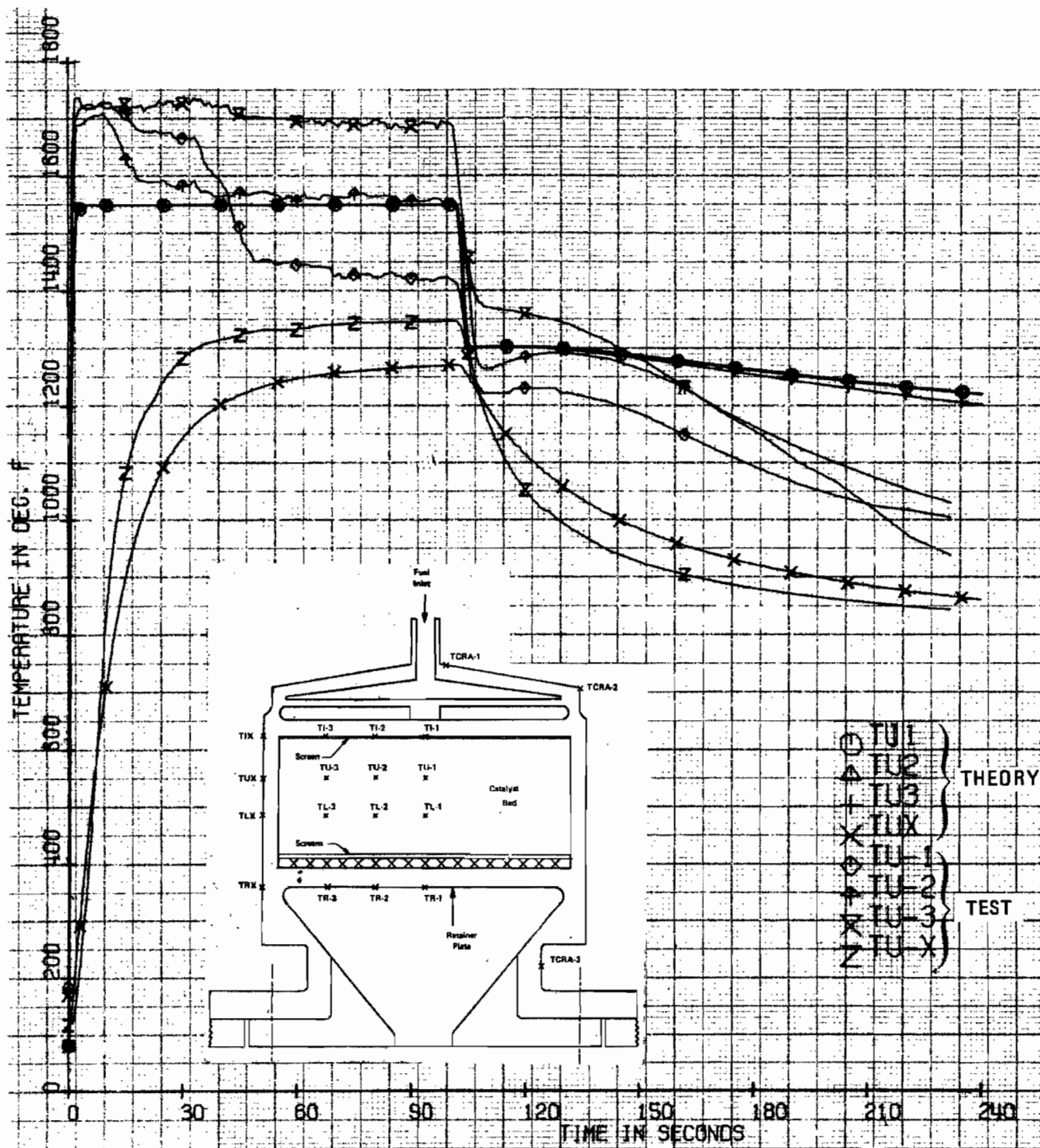


Figure 82. Temperature time history of selected nodal points.  
CRA Run No. 995 - Upper Bed Location

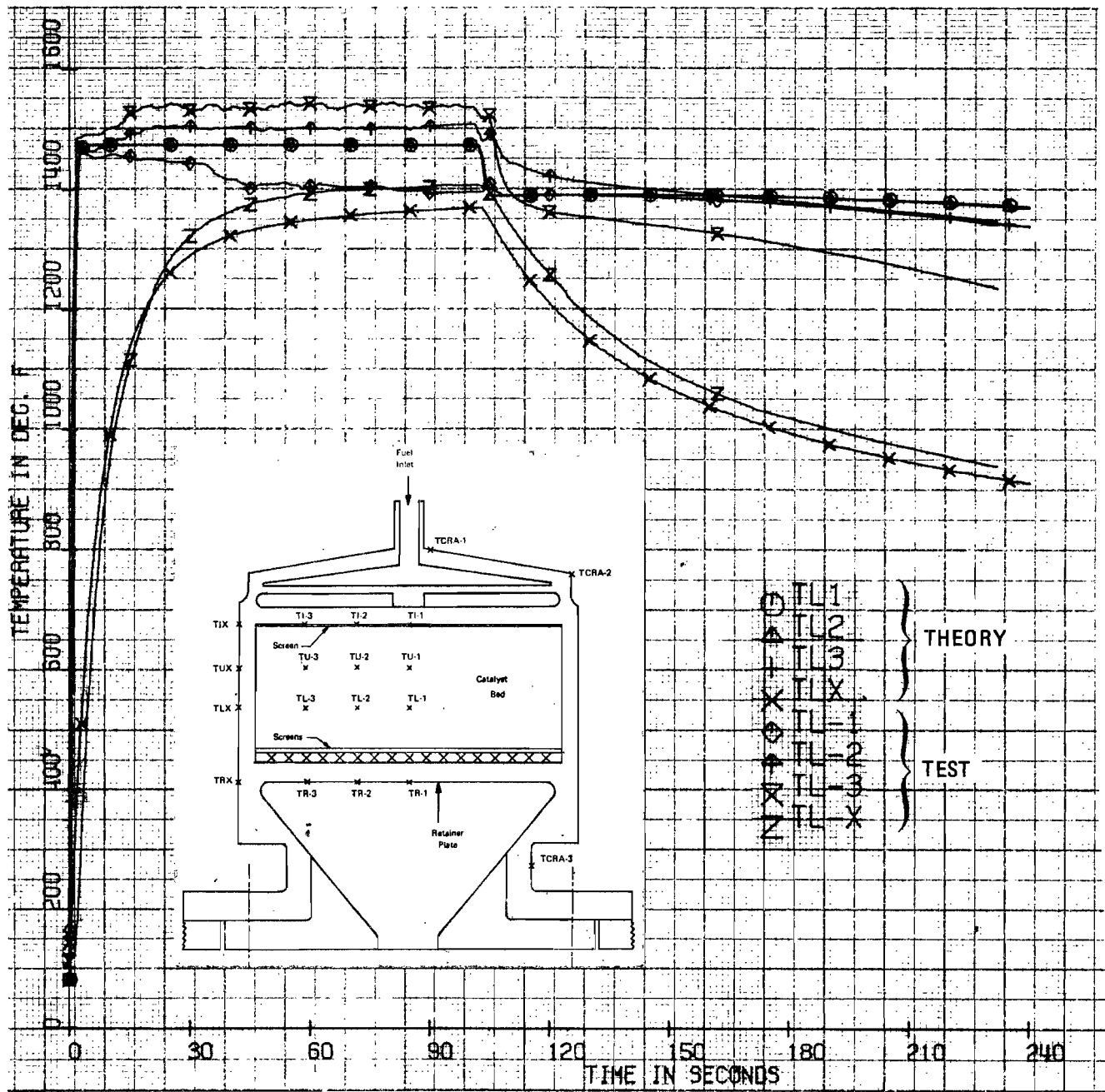


Figure 83. Temperature time history of selected nodal points.  
CRA Run No. 995 - Lower Bed Location

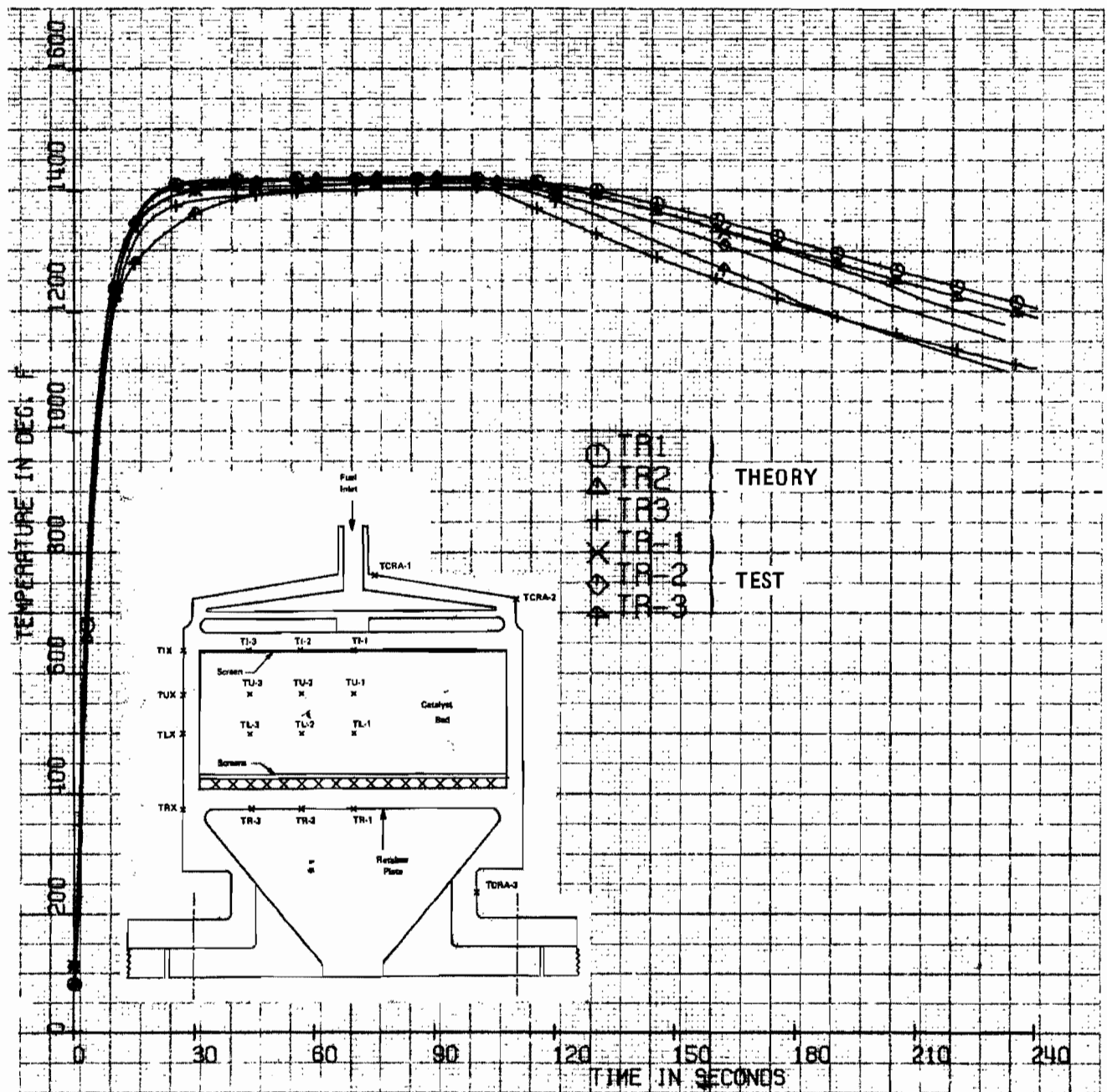


Figure 84. Temperature time history of selected nodal points.  
CRA Run No. 995 - Retainer Plate

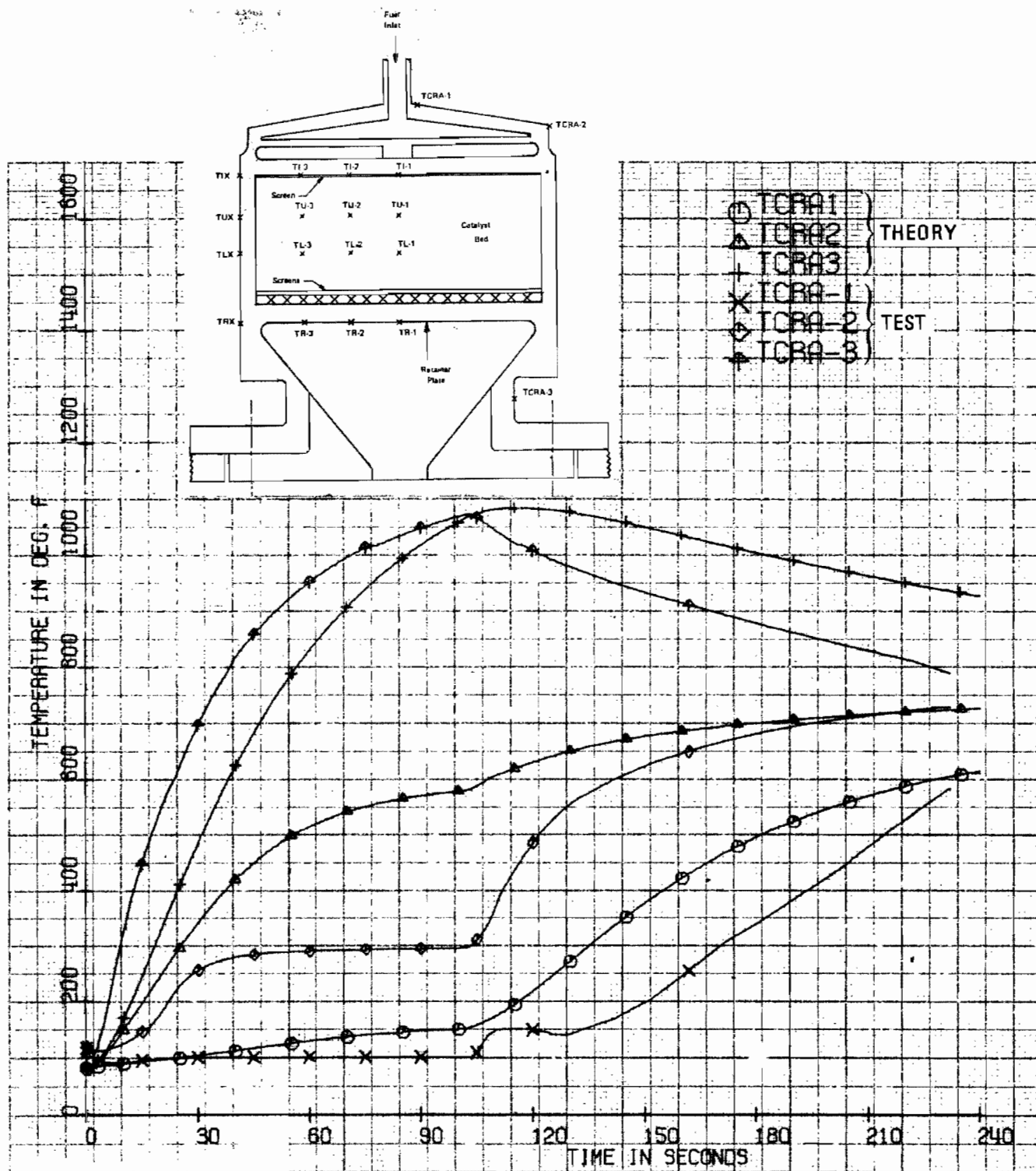


Figure 85. Temperature time history of selected nodal points.  
CRA Run No. 995 - Outer Thermocouples

similar. Thermocouple TCRA-1 (mounted on the back of injector, close to the fuel inlet tube), measured 100°F which is 25°F above the fuel inlet temperature. The temperature measured by TCRA-2 is 290°F (located on the shoulder of the injector) - this temperature is a result of heat soak from the catalyst bed. The measured temperature at the throat (TCRA-3) is 1070°F at 102 seconds and is increasing at the rate of 2° per second. This temperature will continue to rise until equilibrium is achieved between the convective heating of the nozzle and conduction to the mounting plate.

In order to get a composite map of the catalyst bed temperature distribution, the matrix in Figure 86 was prepared. This temperature distribution represents the steady-state temperature distribution towards the end of the firing time. The peak catalyst temperature occurs at 2/3 bed radius in the bed U-location (1700°F), the axis of the bed runs coolest (1430 to 1410°F). Theoretical data predict that 1700°F is the maximum bed temperature at 100 psia chamber pressure and a mass flux of 3.0 lb/ft<sup>2</sup>-sec (see Reference 1, Figure 21). The average mass flux for the CRA is 3.0 lb/ft<sup>2</sup>-sec; however, if the hydrazine flow is not evenly distributed, the specific flow in the outer section of the bed may be much higher. An injector design with a larger number of closely spaced hole patterns should provide a more even distribution of flow across the bed and hence a more uniform temperature distribution within the bed.

The reactor wall temperatures (TUX and TLX) are considerably lower than the catalyst bed temperatures because of radiation losses to the environment and conduction to cooler parts of the hardware.

Figures 87 through 91 show the heat-soak data to 15 minutes for Test 995. Although the outer portions of the catalyst bed were initially hotter than the core, the bed comes to equilibrium quickly. The outer portions of the bed cool much faster than would be expected. Figure 87 shows that the injector peaks at about 900°F during shutdown. Figure 88 shows that up to 300 seconds there is a steep drop in bed temperature at the U-location and then a slower temperature decay. This can possibly be attributed to initial rapid heat loss to the colder portion of the bed near the injector, which was "quenched" on shutdown.

Figures 89 and 90 show a steady uniform temperature decay at the lower catalyst bed and retainer bed locations. Figure 91 shows that thermocouple TCRA-2 peaks out at 750°F (this thermocouple is mounted on the injector shoulder). The thermocouple mounted at the fuel inlet tube also peaked out at 750°F - this is the driving temperature which heats the valve body during soak-back. No thermocouple was located on the valve body, however, the theoretical study indicates the temperature rise to be 1°F in 140 seconds of heat soak-back.

#### 4.3.3 Summary of Thermal Tests With the TM01 Reactor

This section will compare the measured temperature data on the TM01 engine with the temperature predictions from the up-dated Thermal Model. This second thermal reactor was essentially a standard ARA (5-lbf thrust size), containing a temperature-instrumented SUB. It was fitted with thermocouples in a 3 x 3 radial/axial matrix; analogous to the CRA pattern. Figure 92 presents the overall configurations and the thermocouple locations. This unit operated at the same nominal mass flux, injection velocity and chamber pressure as the CRA. The upper catalyst bed contained 20-30 mesh Shell 405 ABSG, while 14-18 mesh was loaded into the lower bed.

The thermocouples mounted on the injector face are TI-1 and TI-3, their response is shown in Figure 93 for a 100-second firing time and subsequent heat soak. The temperatures from thermocouple TI-1, located at the center of the injector face, fluctuate between 400 and 800°F, whereas the TI-3 reading

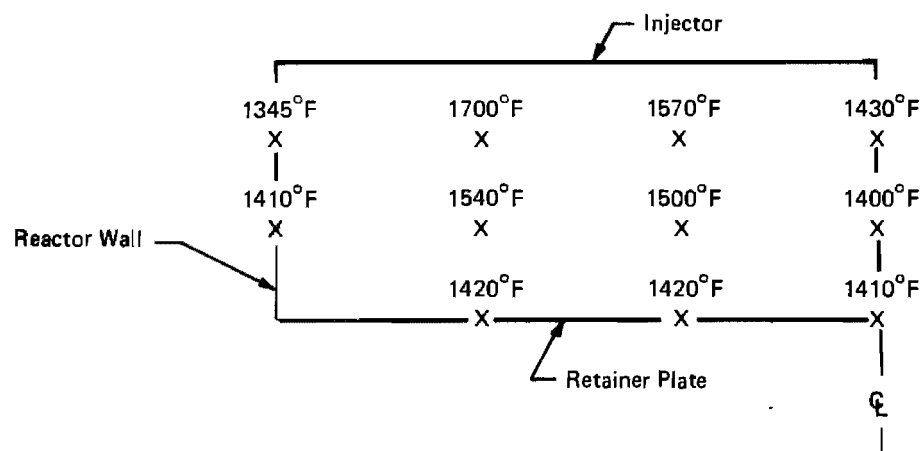


Figure 86. Catalyst bed temperature matrix

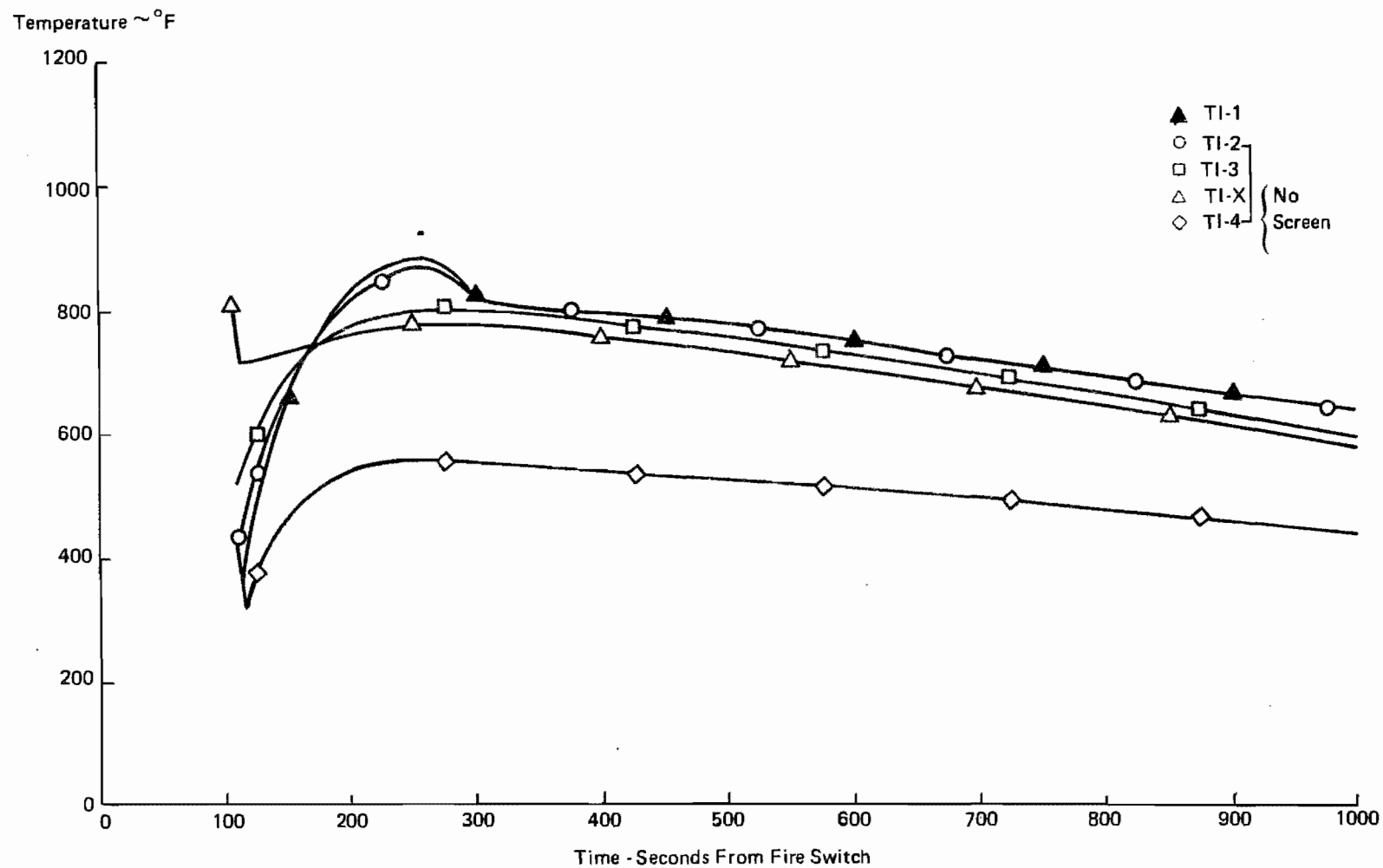


Figure 87. Temperature-time history during shutdown.  
CRA Run No. 995 - Injector

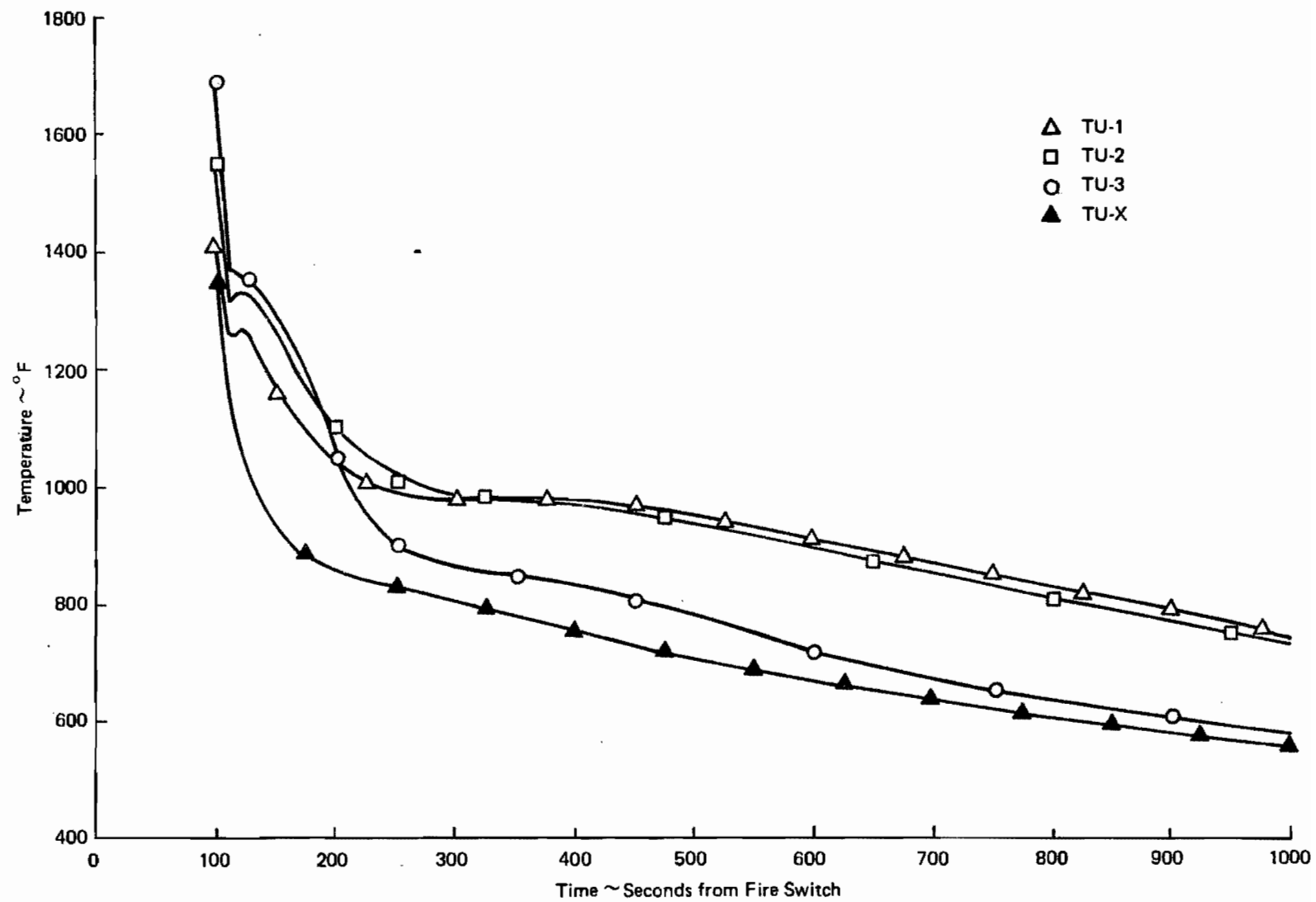


Figure 88. Temperature-time history during shutdown.  
CRA Run No. 995 - Upper Location

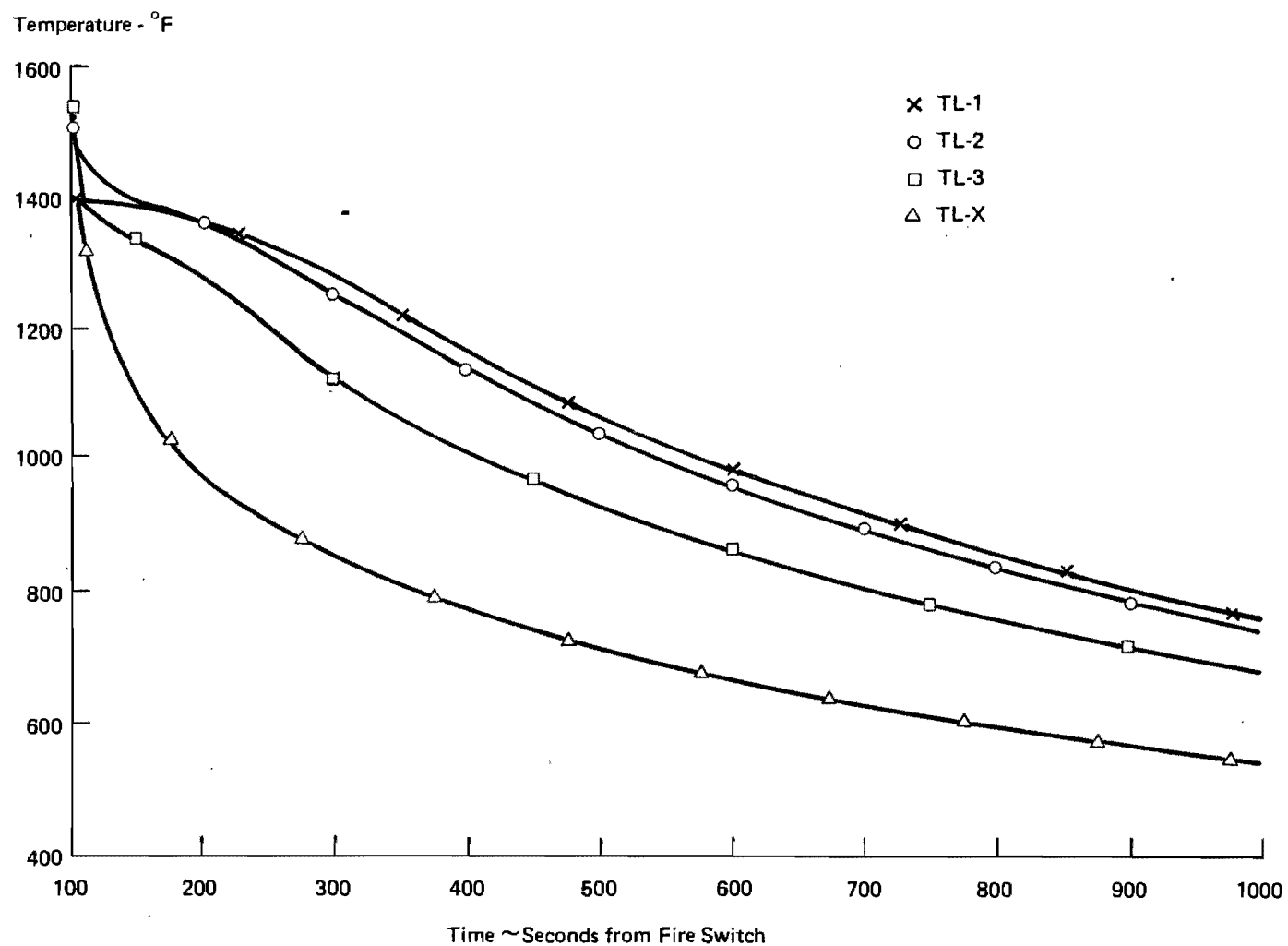


Figure 89. Temperature-time history during shutdown.  
CRA Run No. 995 - Lower Bed Location

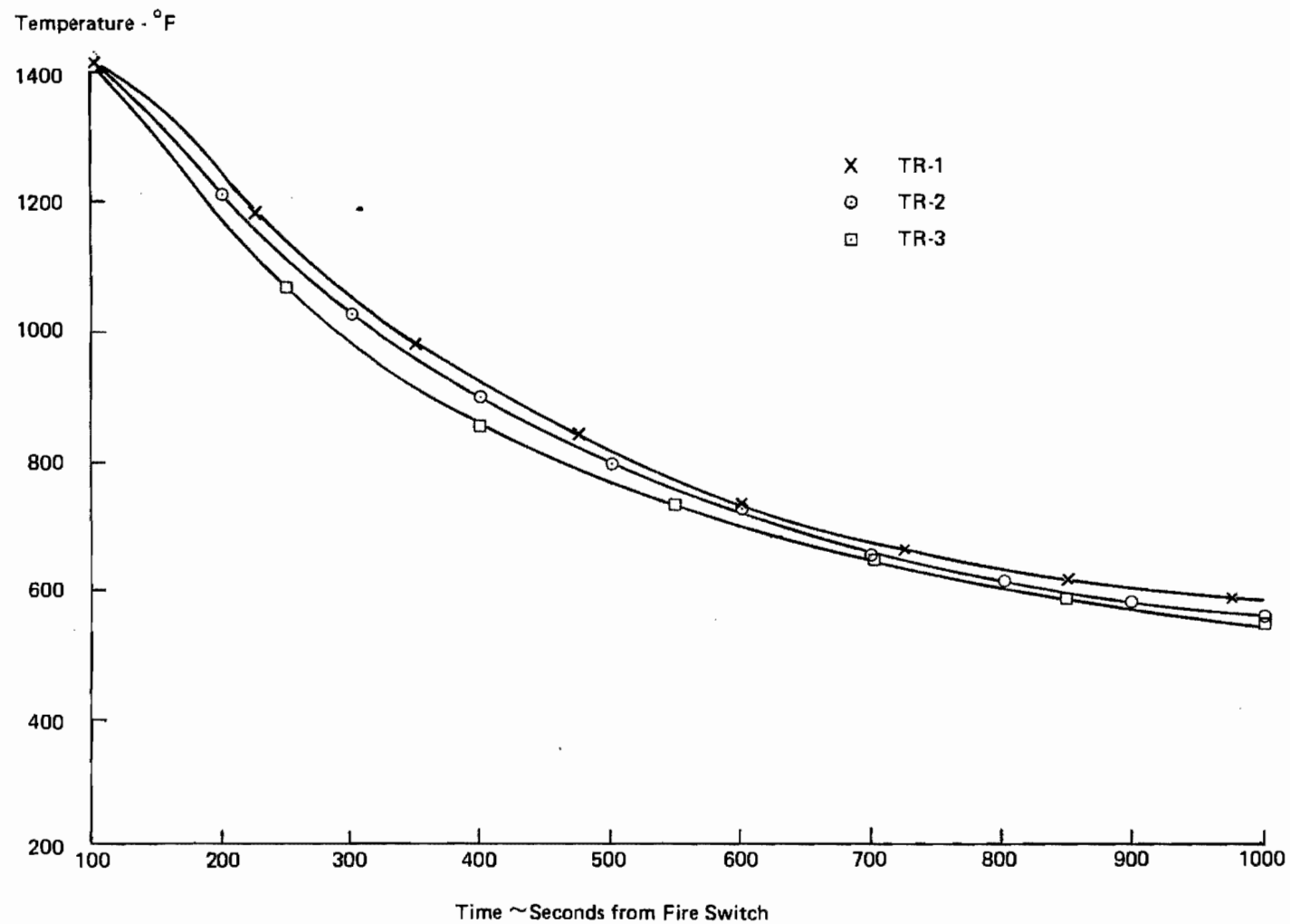


Figure 90. Temperature-time history during shutdown.  
CRA Run No. 995 - Retainer Plate

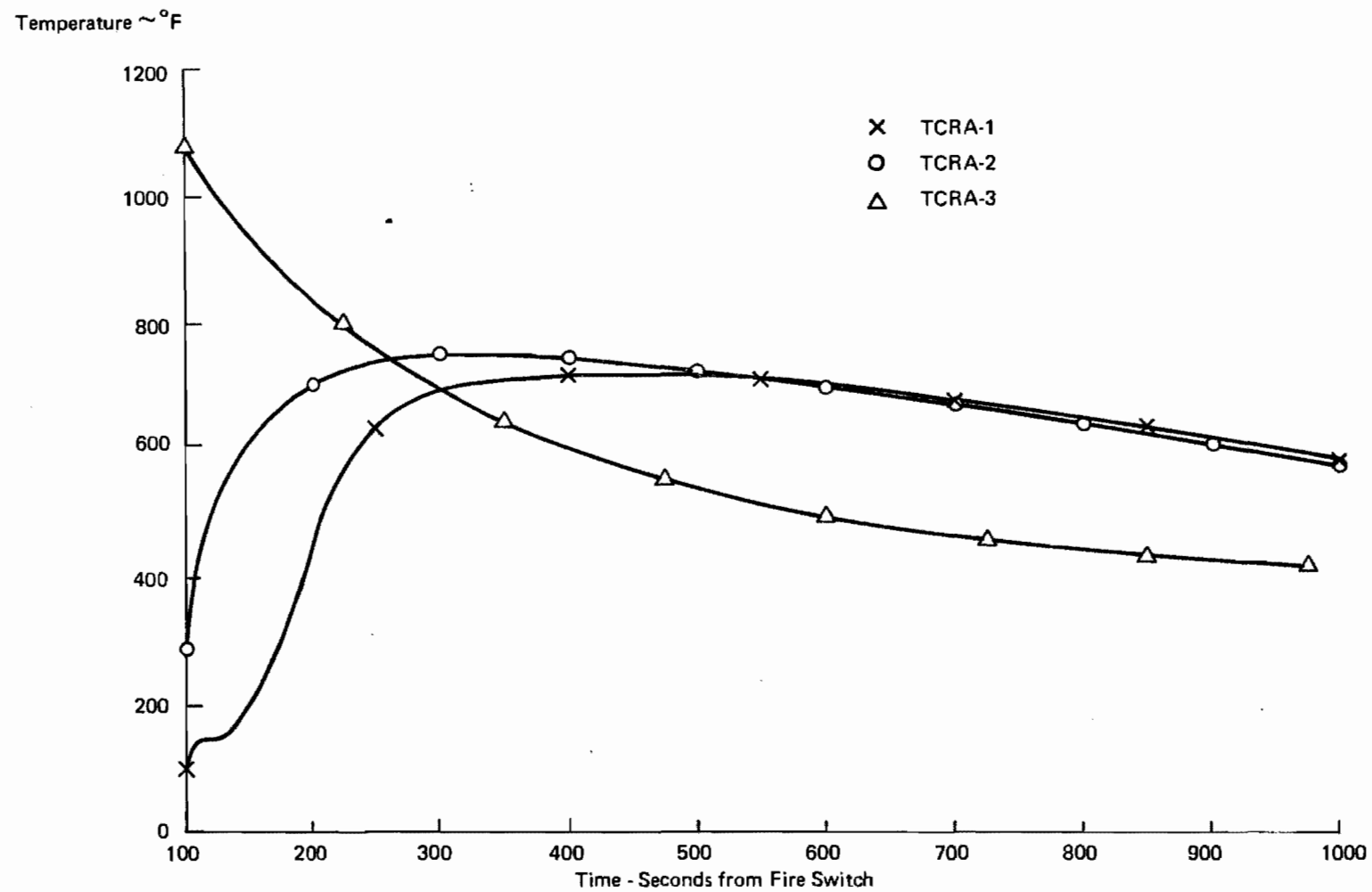


Figure 91. Temperature-time history during shutdown.  
CRA Run No. 995 - Outer Hardware

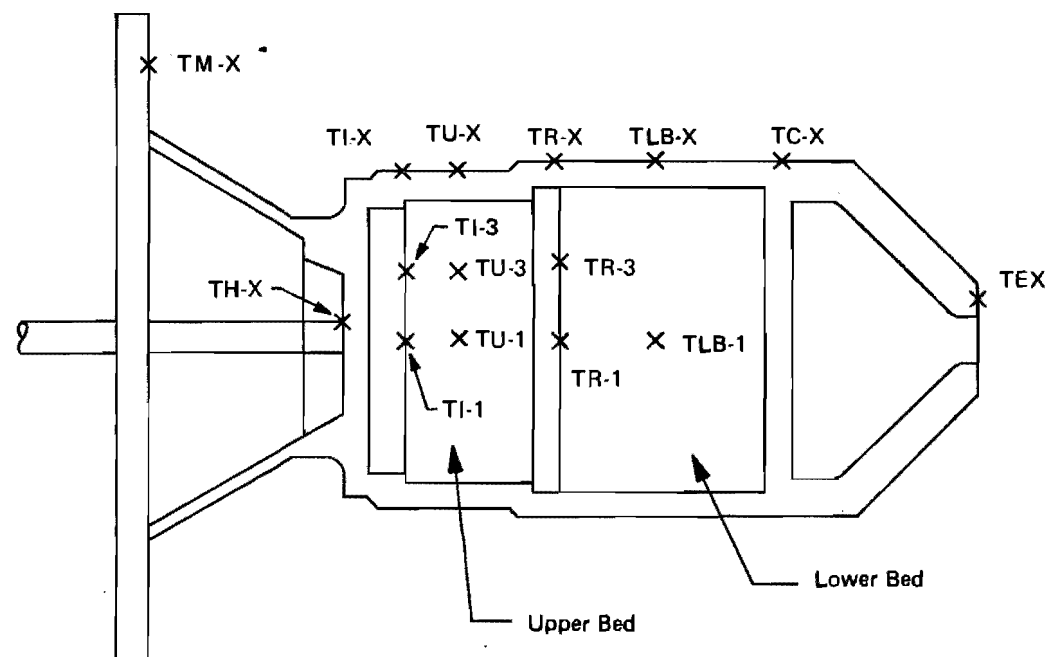


Figure 92. Thermal test reactor - thermocouple locations.

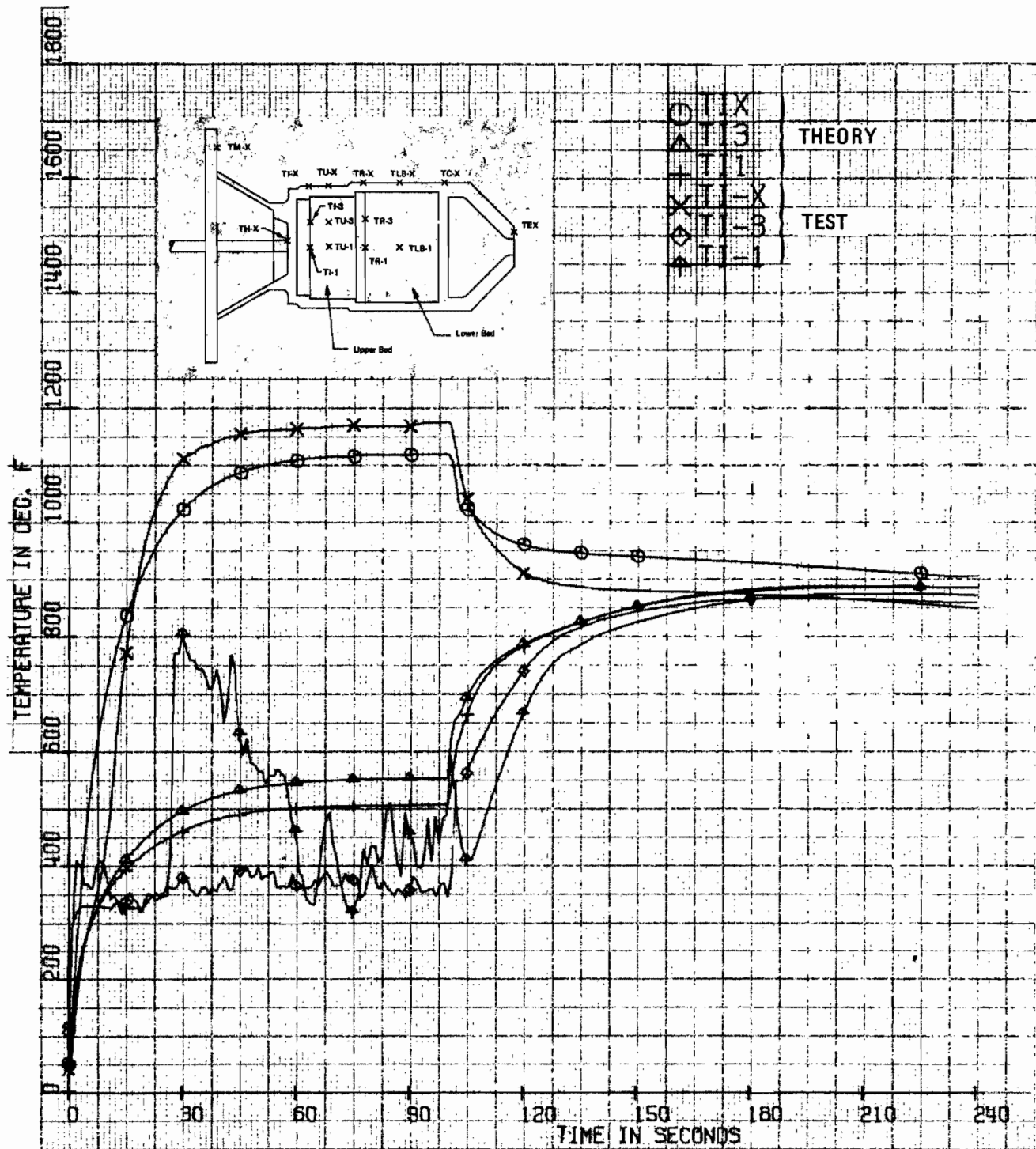


Figure 93. Temperature time history of selected nodal points. Thermal ARA Run No. 1197 - Injector Location

approximates 360°F (the saturation temperature of hydrazine is 360°F at 100 psia). It is thus apparent that the center thermocouple is immersed in hydrazine vapor and the TI-3 thermocouple is affected by decomposed hydrazine and vapor. The outer wall temperature (TI-X) rises to a value of 1170°F in comparison to the predicted value of 1120°F. The driving gas temperature at this location is 1200°F. Upon shutdown the test data and theoretical data converge to 880°F.

Figure 94 shows the response of the upper catalyst bed (TU-1 and TU-3). The predictions and the test data agree very well, falling within  $1640 \pm 50^\circ\text{F}$ .

Figure 95 shows that the heating rate to the retainer plate is higher than predicted. The equilibrium temperature is approximately 1680°F, about 200°F higher than expected at this location.

Figure 96 shows excellent agreement between theory and test for the lower bed thermocouples (TLB-X and TLB-1).

Figure 97 shows good agreement for the thermocouples located at the outer wall, lower bed retainer plate and the nozzle exit temperature (TC-X and TE-X). Thermocouple TH-X located on the rear of the injector levels off at 170°F compared to 180°F theoretically. Thermocouple TM-X, located on the stand-off mounting flange, indicates that more heat is conducted to the flange suggesting that the thermal stand-off is not as good as predicted but does come to an equilibrium temperature of 120°F.

The data from test No. 1198 compare very favorably with the data from test 1197. Test No. 1198 had a firing time of almost 150 seconds.

#### 4.3.4 General Observations and Conclusions

Significant temperature gradients were evident within the catalyst beds of the reactors tested. These gradients were similar, and are typified by the values shown in Figure 98.

Both reactors exhibited approximately 100°F cooler operation in the center of the catalyst bed as opposed to the chamber wall. The precise reason for this is not known, but the repeatability from unit to unit indicates that, at least for these uniform propellant distribution showerhead injector configurations, the trend is real.

Upon shutdown, a rapid temperature decrease ( $\sim 300^\circ\text{F}$  in 7 seconds) occurred in the catalyst bed below the injector. This indicates that a net cooling effect occurs when hydrazine in the region between the injector face and the propellant valve is vaporized out into the bed. The extent of this cooling is probably a function of the quantity of propellant ("holdup volume"). The units tested were designed with relatively large holdup volumes as a compromise to assure good propellant injection distribution.

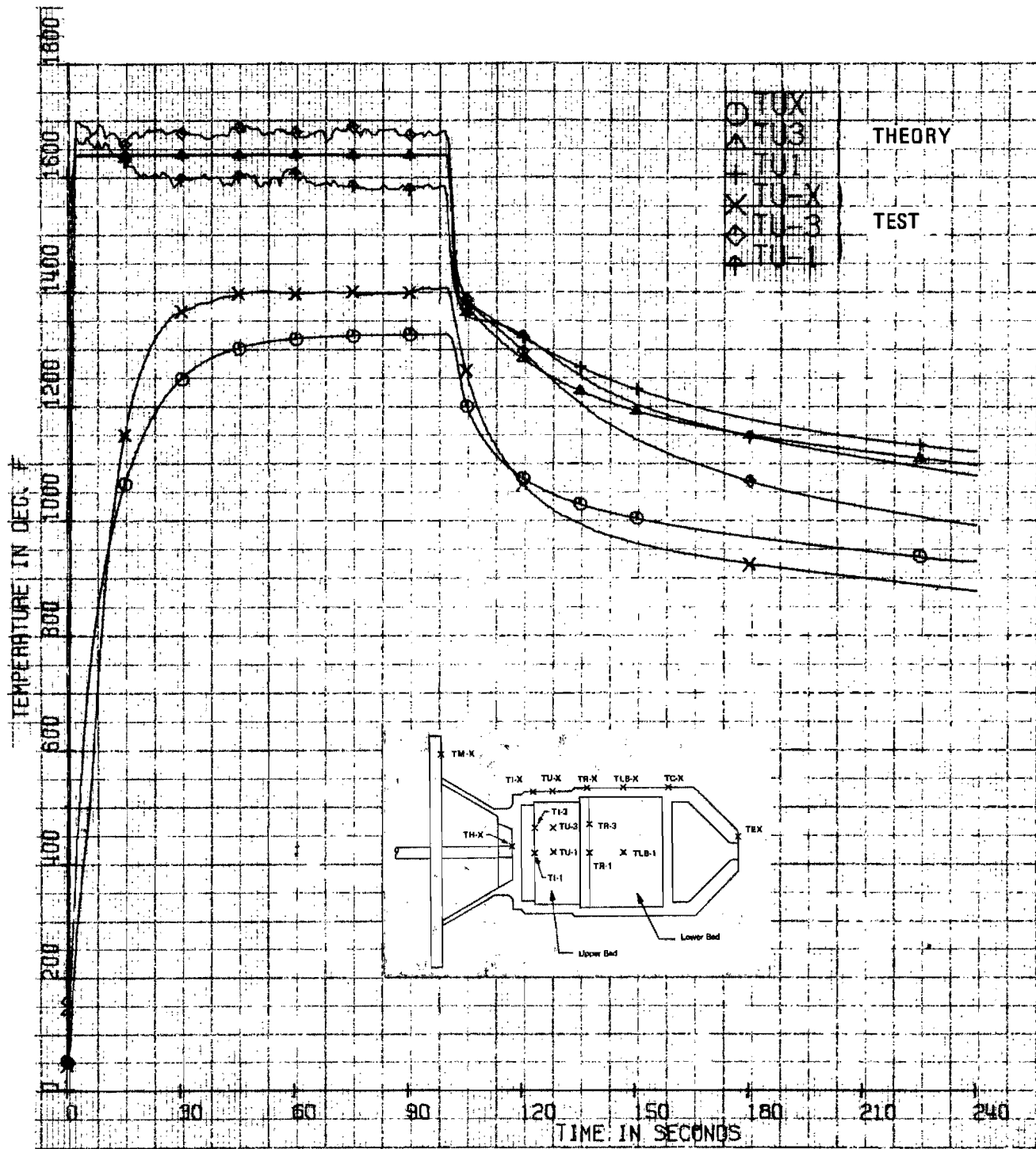


Figure 94. Temperature time history of selected nodal points. Thermal ARA Run No. 1197 - Upper Bed Location

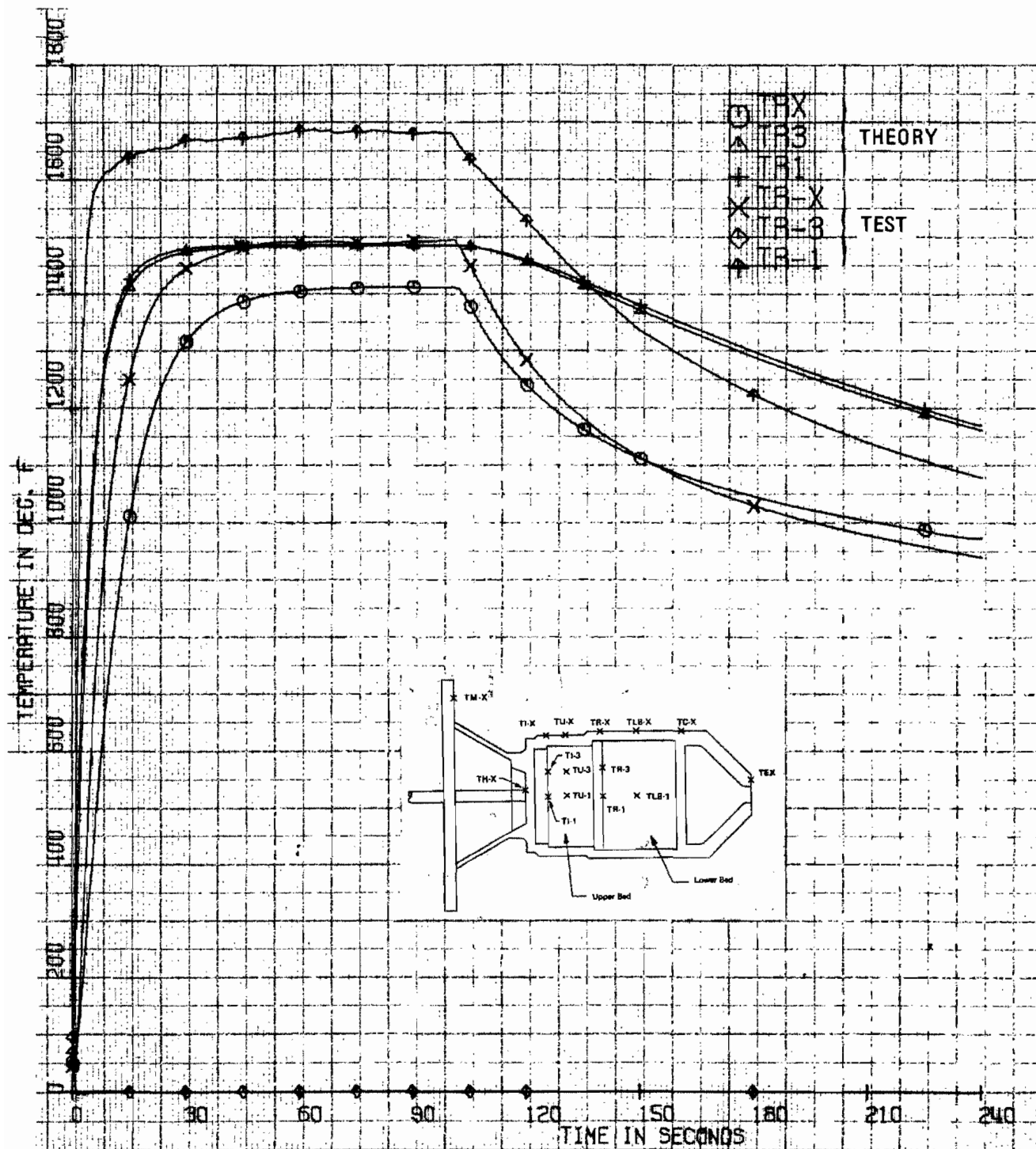


Figure 95. Temperature time history of selected nodal points. Thermal  
ARA Run No. 1197 - Retainer Plate Locations

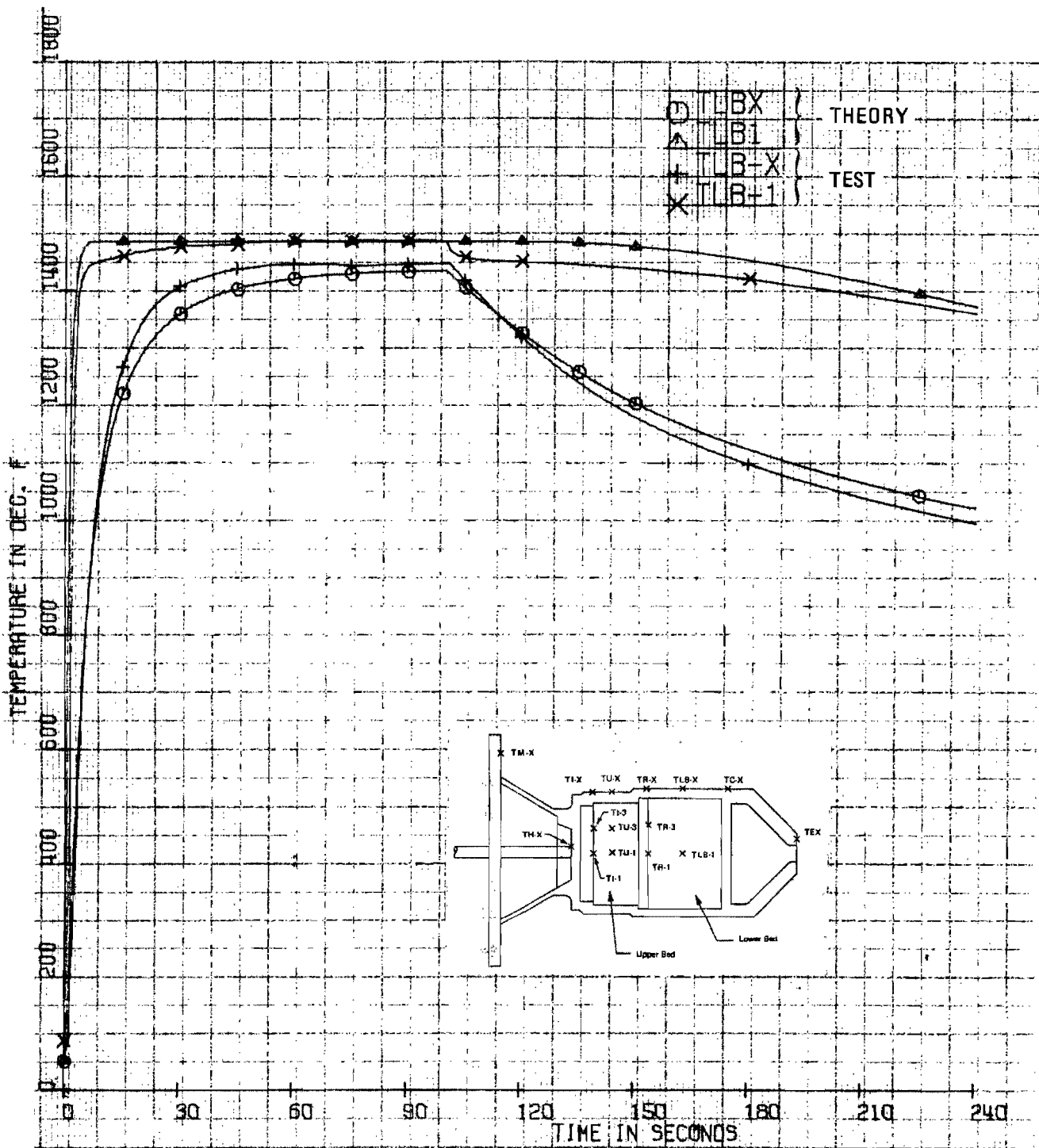


Figure 96. Temperature time history of selected nodal points. Thermal  
ARA Run No. 1197 - Lower Bed Location

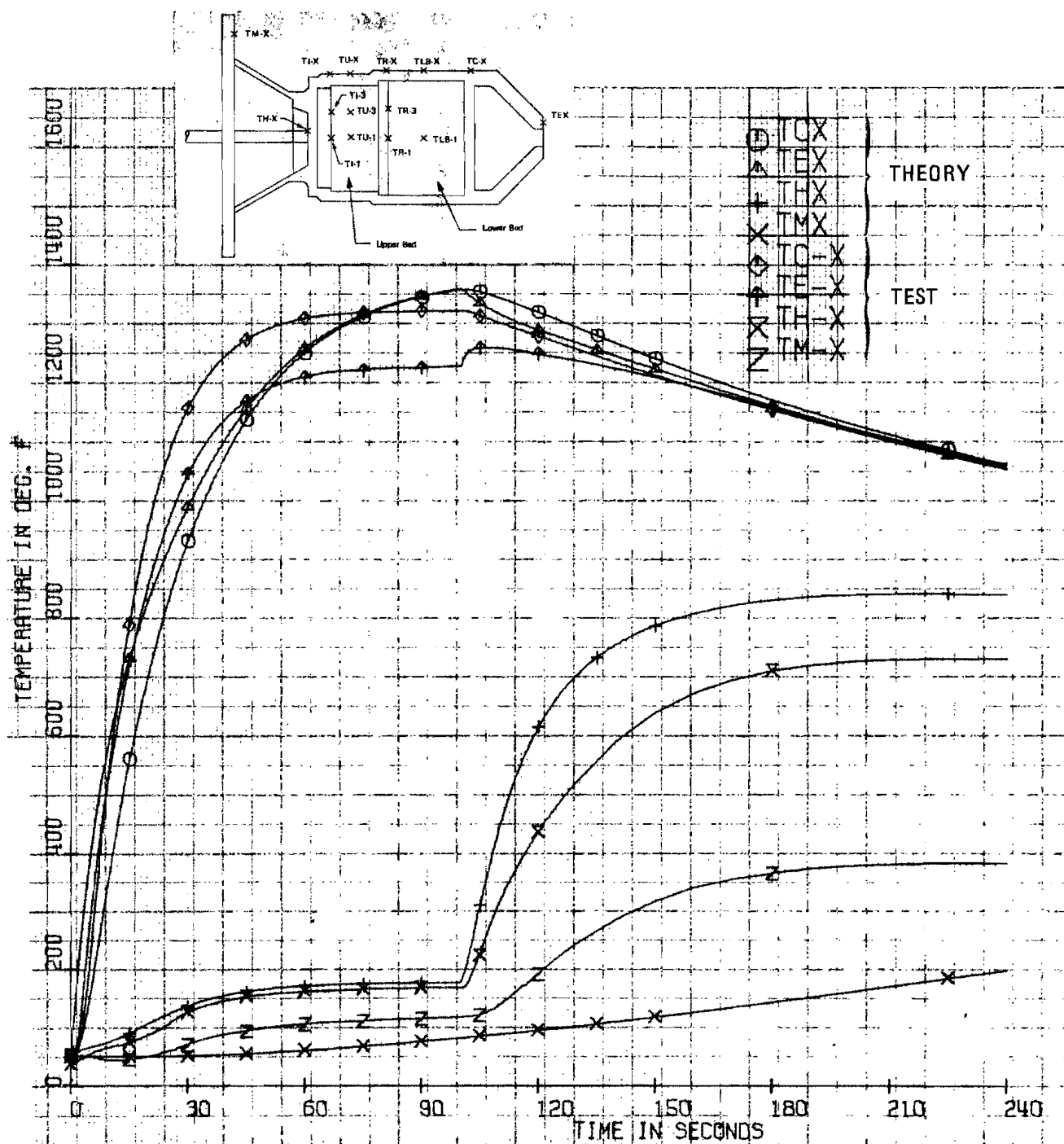


Figure 97. Temperature time history of selected nodal points. Thermal ARA Run No. 1197 - Outer Thermocouples

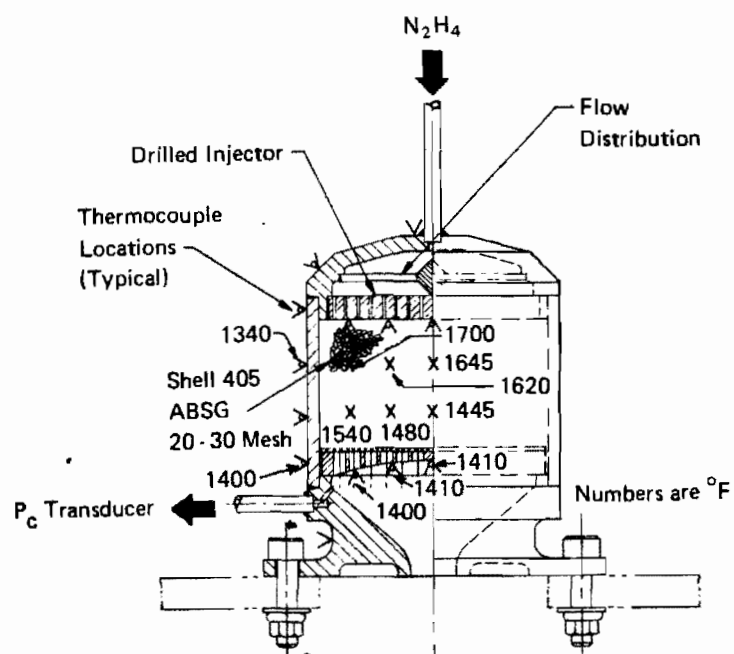


Figure 98. Conductometric reactor assembly steady-state operating temperature.

## APPENDIX A

### DEVELOPMENT OF AN IMAGE PROCESSING TECHNIQUE FOR CATALYST ATTRITION ANALYSIS

Two basic approaches were originally considered to examine a catalyst bed for attrition:

1. To disassemble the bed post-exposure and sieve the catalyst to ascertain the variation in mean particle size and distribution.
  2. To carefully infiltrate the post-fired bed with epoxy resin and study the particle statistics in planar sections thereof. The former approach, although more straightforward from the standpoint of obtaining particle size statistics, has the overriding disadvantage of destroying the spatial configuration of the bed, thereby obviating the possibility of correlating observed attrition with orifice configuration. The latter approach was therefore selected. This decision mandated development of an automated technique for analyzing the "potted" and sectioned catalyst beds.
- a. Approach

Stated in simplest and most general terms, the problem is this: Given a planar sample of closely-packed and contiguous, but not overlapping, "blobs" (sections through irregular but roughly spheroidal catalyst particles), to

- (1) Determine the size and shape of the blobs in the section;
- (2) Compute the appropriate frequency distributions; and
- (3) Using existing statistical techniques, calculate 3-dimensional distribution characteristics from the 2-dimensional sectional data.

The BAC Digital Image Processing System (DIPS) was selected as the best approach to this task. The input device is a flying spot scanner (FSS), presently adapted for scanning a 35 mm transparency. The first step in processing the "raw data" (the polished sections of the infiltrated beds) was to transpose them into this format.

To enhance contrast in the photographic image, the epoxy infiltrant employed was "loaded" with a white titanium oxide pigment which rendered interstitial areas an opaque light beige color. A data assembly was prepared for photographing, consisting of the polished sections of the catalyst bed to be evaluated, together with a similar section of a virgin bed containing the same catalyst, and a calibration device. The latter contained two fiducial marks, a known distance apart, which set the absolute scale of the photographic image, and a standard photographic gray scale. This data assembly was then shot on fine grain panchromatic film. The negatives thereby produced were, in turn, contact printed on high-contrast 35 mm film to produce maximum contrast positive transparencies. This positive film strip was then the input to DIPS.

The major elements of DIPS, besides the FSS, include a control/display console, and an on-line Varian 620i computer with associated storage units and operator I/O access via teletypewriter. The functions of the DIPS are to

- Reduce the input images to binary maps;
- Analyze these maps, and generate 2-dimensional statistics therefrom; and
- Prepare an input tape for the 3-dimensional analysis program, which is executed elsewhere on a high-speed IBM 360-65 computer.

b. DIPS Software

The DIPS software consists of two major programs, **TRACER** and **GRAPHER**, executed in that sequence. **TRACER** is an interactive on-line program, involving a sequence of operator-machine interactions, which "finds" each particle, traces its perimeter, and writes its boundary locations on tape. Each frame of the input data film strip is read in, via the FSS, as a two-dimensional array of locations, each having a potential opacity (gray level) of one of 256 possible values.

The first frame on any film strip is the calibration frame wherein, given approximate starting coordinates from operator review of the displayed image, **TRACER** finds the exact distance in FSS units between the fiducial marks and thus generates and retains a scale factor.

For the subsequent frames containing the bed sections, each frame is initially read in as a 256 x 256 picture element ("pel") array, and written out on the display. In passing through the processor, a frequency histogram of the opacities is generated and displayed superimposed on the image itself. If the photo image was properly prepared, this histogram will be essentially bimodal. On the basis of this histogram the operator selects and inputs a threshold gray level just above the foot of the "particle" (dark) mode. This threshold is the basis for a subsequent dichotomous decision by **TRACER** as to whether a given point is a particle point (1) or background point (0) in forming the binary map and is perhaps the most sensitive control parameter. The objective is to guarantee a faithful reproduction of the particle boundary while maintaining adequate sensitivity to separate particles along narrow cracks and fissures.

Also displayed with the image and histogram is an X-Y grid scaled in picture units. Using this grid the operator also selects and inputs four (X, Y) coordinate pairs representing the coordinates of the apices of a quadrilateral which bounds the region of interest (ROI) in the frame. All subsequent processing is limited essentially to the contents of the ROI.

Next the DIPS re-scans the image point-by-point at a resolution of 1024 x 1024 lines. Core limitations of the central processor, however, permit only a 256 x 256 bit binary map, hence a 16:1 compaction algorithm operates in the digitization. Using the aforementioned threshold, **TRACER** currently assigns the 16-point "superbit" a zero (background) value if 3 or more points are zero, otherwise it is taken as unity (particle). This conservative ground rule tends to ensure that only interior points are ascribed to a particle and aids separation of touching particles. Unfortunately, it also shrinks particles slightly. During this operation, the total number of pels inside the ROI is noted, as well as those having a value of 1, giving a measure of void fraction in the ROI.

Now with the binary image stored, processing of the matrix begins. The objective is to separate the array into two arrays, one containing only boundary points and the other only interior points. The processing algorithms operate on each bit as affected by its eight neighbor bits in the 256 x 256 array. Through the operation of these algorithms, isolated 0's and 1's are eliminated, narrow (one bit) necks are cut through, and the result is the desired pair of arrays. The "Interiors"

array contains a shrunken version of the binary image, while the "Edges" array contains boundary points and particles small enough to have "Interiors." Either or both arrays may be displayed.

Next, a tracking routine traces around the outside of each particle in the "Interiors" array twice, noting its path each time. The second tracing effectively replaces area lost in the thresholding/separation operations on the particle and also forms the basis for perimeter calculation. The finalized tracking data, together with the "particles-with-no-interiors" points, and other appropriate coding information are then output on tape for each particle in the ROI.

The second on-line interactive program, GRAPHER, then comes into play to analyze the data generated by TRACER. Particle section perimeters and areas computed from the number and orientation of unity bits are converted via the scale factor from pel units to inch units. A shape factor,  $f_s$ , is also generated for each blob based upon its area,  $a$ , and perimeter,  $p$ , defined as

$$f_{s_i} = \frac{p_i}{\sqrt{4 \cdot a_i}} \quad (i = 1, 2, \dots, \text{no. of particles})$$

which is the ratio of the perimeter of the blob to that of a perfect circle of equal area. It is the reciprocal of circularity, the two-dimensional analog of sphericity. Thus  $f_s$  has a minimum value of unity for circular blobs, and increases as they become more distended or irregular. GRAPHER computes frequency histograms for the equivalent  $\bar{r}$ , perimetral, diameter, and  $f_{s_i}$  and their means and standard deviations.

Finally, a summary of the data including the means and deviations is printed out via the teletypewriter, while, at operator's option, the histograms can be displayed on the console and/or printed on 35 mm film.

This completes the DIPS processing of the two-dimensional (sectional) data. Extrapolation to three-dimensional distributions can be accomplished by processing the data tape generated by TRACER with the IBM 360-65 using statistical techniques.

### c. Conclusions

The DIPS technique developed does a creditable job of generating particle spectrum statistics for sections of catalyst beds. The technique is of far more general utility however, being applicable to any planar array of contiguous, close-packed, non-overlapping particles against a contrasting background. A shortcoming of the present system is core limitation in the central processor. This may be easily overcome by either expanding the present core, or providing a data link to a higher capacity machine.

## APPENDIX B

### ATTRITION RESULTS LISTED BY ARA S/N

Table B-1 reproduces the main data file which was the resource for the computerized analyses discussed elsewhere in this report, Table B-2 presents the results from the partitioned chambers - which were not included in the computer file.

The data values are in the FORTRAN "E" format, a variation of scientific notation. In the table, up to five significant digits are presented as a decimal fraction. The number following the letter "E" is the exponent of 10 that is associated with the decimal fraction. For example, column 1 row 1 reads "0.10800E 01." This is the number "1.08" a value of "0.0" indicates no data has been entered in that location (i.e., it was lost or invalid).

Each of the 65 rows contains the results from one ARA. Each of the 15 columns contains a parameter as indicated below:

| Col. | Label      | Units          | Text Symbol/Comment                                                                                                                                                      |
|------|------------|----------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1    | ARA S/N    | —              | All are numbers from 1 to 10: integral part is coded roman numeral; 1 = I, 2 = II, . . . , fraction is coded letter; 0.01 = A, 0.02 = B, . . . (e.g., 1.08 means I - H). |
| 2    | INJ. VEL   | ft/sec         | V                                                                                                                                                                        |
| 3    | W DOT      | lbm/sec        | $\dot{w}$                                                                                                                                                                |
| 4    | CATALYST   | —              | Coded catalyst size/treatment:<br>1 = 20 - 25 mesh<br>2 = 20 - 25 mesh<br>90% attritted<br>3 = 20 - 25 mesh<br>$\alpha$ phase<br>4 = 25 - 30 mesh                        |
| 5    | PHI        | —              | $\phi$ - propellant distribution parameter                                                                                                                               |
| 6    | INJ. AREA  | —              | Fraction of injector face which contains injection orifices<br>1.0 = 100%<br>0.25 = 25%, etc.                                                                            |
| 7    | # ORIF's   | —              | # OR                                                                                                                                                                     |
| 8    | = OR DI    | $10^{-3}$ inch | - Diameter of circular injection orifices or of circular orifice of same area as non-circular orifice                                                                    |
| 9    | AVG RGHN   | %              | $\bar{R}$ , mean roughness                                                                                                                                               |
| 10   | DEL RGHN   | %              | $\Delta R$ , Roughness range                                                                                                                                             |
| 11   | PLI - MBAR | —              | $\bar{M}_{PLI}$ - average rate of PLI increase                                                                                                                           |

| <u>Col</u> | <u>Label</u> | <u>Units</u> | <u>Text Symbol/Comment</u>                                              |
|------------|--------------|--------------|-------------------------------------------------------------------------|
| 12         | PLI - RANG   | —            | R <sub>p</sub> LI - range of PLI                                        |
| 13         | DEC WGT      | %            | % ΔW - sub weight loss                                                  |
| 14         | # C.S.       | —            | # CS - number of cold starts.                                           |
| 15         | DE Bar       | inch         | - Section mean particle diameter (from digital image processing system) |

TABLE B-1

## ATTRITION RESULTS DATA FILE

| COL. --- 1 | 2           | 3           | 4           | 5           | 6           | 7           | 8           | 9           | 10          | 11          | 12          | 13          | 14          | 15          |             |
|------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| ROW        | ARA E/M     | INJ. VEL    | F DOF       | CATALYST    | PHI         | INJ AREA    | # ORIF'S    | = OR. DI    | ANG RGMN    | DEL RGMN    | PLT-MBAR    | PLT-RANG    | DEL MGT     | F C.S.      | DF BAR      |
| 1          | 0.10800E 01 | 0.11200E 01 | 0.23200E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.15910E 04 | 0.62000E 01 | 0.10600E 02 | 0.48000E 01 | 0.55000E-01 | 0.28000E 01 | 0.92000E 01 | 0.10000E 03 | 0.17100E-01 |
| 2          | 0.11300E 01 | 0.10500E 01 | 0.23200E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.15600E 01 | 0.20020E 02 | 0.14350E 02 | 0.61000E 01 | 0.42000E-01 | 0.57000E 01 | 0.60000E 01 | 0.10000E 03 | 0.16300E-01 |
| 3          | 0.20600E 01 | 0.86500E 00 | 0.23100E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.49400E 04 | 0.40000E 01 | 0.69000E 01 | 0.12200E 02 | 0.27600E-01 | 0.30000E 01 | 0.72000E 01 | 0.10000E 03 | 0.16400E-01 |
| 4          | 0.20200E 01 | 0.27300E 01 | 0.10900E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.27000E 03 | 0.66100E 01 | 0.25000E 01 | 0.18000E 01 | 0.22800E-01 | 0.21000E 01 | 0.67000E 01 | 0.10000E 03 | 0.16600E-01 |
| 5          | 0.20800E 01 | 0.28700E 01 | 0.23100E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.53900E 03 | 0.66500E 01 | 0.49000E 01 | 0.74000E 01 | 0.13700E-01 | 0.15000E 01 | 0.56000E 01 | 0.10000E 03 | 0.16500E-01 |
| 6          | 0.30200E 01 | 0.28600E 01 | 0.23100E-01 | 0.20000E 01 | 0.54550E 00 | 0.10000E 01 | 0.53900E 03 | 0.66500E 01 | 0.86500E 01 | 0.91000E 01 | 0.14500E-01 | 0.15000E 01 | 0.11000E 02 | 0.10000E 03 | 0.17200E-01 |
| 7          | 0.31400E 01 | 0.28600E 01 | 0.23000E-01 | 0.30000E 01 | 0.54550E 00 | 0.10000E 01 | 0.53900E 03 | 0.66500E 01 | 0.65000E 01 | 0.86000E 01 | 0.15600E-01 | 0.19000E 01 | 0.57000E 01 | 0.10000E 03 | 0.17200E-01 |
| 8          | 0.30300E 01 | 0.28600E 01 | 0.23000E-01 | 0.40000E 01 | 0.54550E 00 | 0.10000E 01 | 0.53900E 03 | 0.66500E 01 | 0.58000E 01 | 0.80000E 01 | 0.10200E-01 | 0.10200E 01 | 0.63000E 01 | 0.10000E 03 | 0.13200E-01 |
| 9          | 0.30600E 01 | 0.28600E 01 | 0.23000E-01 | 0.50000E 01 | 0.54550E 00 | 0.10000E 01 | 0.53900E 03 | 0.66500E 01 | 0.24000E 01 | 0.28000E 01 | 0.0         | 0.0         | 0.37000E 01 | 0.10000E 03 | 0.28800E-01 |
| 10         | 0.10200E 01 | 0.10200E 01 | 0.33700E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.73900E 03 | 0.66500E 01 | 0.11050E 02 | 0.51000E 01 | 0.25700E-01 | 0.18000E 01 | 0.70000E 01 | 0.10000E 03 | 0.17500E-01 |
| 11         | 0.20100E 01 | 0.77800E 01 | 0.10900E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.24700E 03 | 0.41000E 01 | 0.18500E 01 | 0.27000E 01 | 0.22500E-01 | 0.17000E 01 | 0.46000E 01 | 0.10000E 03 | 0.17700E-01 |
| 12         | 0.20300E 01 | 0.93200E 01 | 0.10900E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.80000E 02 | 0.65700E 01 | 0.18500E 01 | 0.29000E 01 | 0.19000E-01 | 0.12000E 01 | 0.67000E 01 | 0.10000E 03 | 0.16200E-01 |
| 13         | 0.20500E 01 | 0.10700E 02 | 0.10800E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.30000E 02 | 0.10000E 02 | 0.40000E 01 | 0.64000E 01 | 0.21800E-01 | 0.13000E 01 | 0.53000E 01 | 0.10000E 03 | 0.15600E-01 |
| 14         | 0.21300E 01 | 0.95800E 01 | 0.23100E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.16060E 04 | 0.21100E 01 | 0.62000E 01 | 0.10800E 02 | 0.29600E-01 | 0.22000E 01 | 0.82000E 01 | 0.10000E 03 | 0.16700E-01 |
| 15         | 0.10900E 01 | 0.92300E 01 | 0.23200E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.42100E 03 | 0.42000E 01 | 0.61500E 01 | 0.61000E 01 | 0.26200E-01 | 0.18000E 01 | 0.43000E 01 | 0.10000E 03 | 0.16000E-01 |
| 16         | 0.20700E 01 | 0.89700E 01 | 0.23100E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.16000E 03 | 0.69000E 01 | 0.31000E 01 | 0.62000E 01 | 0.12300E-01 | 0.13000E 01 | 0.62000E 01 | 0.10000E 03 | 0.15700E-01 |
| 17         | 0.30900E 01 | 0.09700E 01 | 0.23100E-01 | 0.20000E 01 | 0.54550E 00 | 0.10000E 01 | 0.16000E 03 | 0.69000E 01 | 0.38000E 01 | 0.64000E 01 | 0.11700E-01 | 0.11000E 01 | 0.98000E 01 | 0.10000E 03 | 0.18700E-01 |
| 18         | 0.31500E 01 | 0.89500E 01 | 0.23000E-01 | 0.30000E 01 | 0.54550E 00 | 0.10000E 01 | 0.16000E 03 | 0.69000E 01 | 0.51000E 01 | 0.62000E 01 | 0.18000E-01 | 0.28000E 01 | 0.65000E 01 | 0.10000E 03 | 0.15500E-01 |
| 19         | 0.11100E 01 | 0.94300E 01 | 0.23100E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.60000E 02 | 0.11000E 02 | 0.30500E 01 | 0.19000E 01 | 0.10000E-01 | 0.70000E 00 | 0.53000E 01 | 0.10000E 03 | 0.16800E-01 |
| 20         | 0.21000E 01 | 0.14890E 02 | 0.23100E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.16000E 02 | 0.16950E 02 | 0.27000E 01 | 0.22000E 01 | 0.10000E-01 | 0.60000E 00 | 0.75000E 00 | 0.10000E 03 | 0.18100E-01 |
| 21         | 0.30400E 01 | 0.89500E 01 | 0.23000E-01 | 0.40000E 01 | 0.54550E 00 | 0.10000E 01 | 0.16000E 03 | 0.69000E 01 | 0.23000E 01 | 0.26000E 01 | 0.72900E-02 | 0.80000E 00 | 0.55000E 01 | 0.10000E 03 | 0.13400E-01 |
| 22         | 0.30700E 01 | 0.89500E 01 | 0.23000E-01 | 0.50000E 01 | 0.54550E 00 | 0.10000E 01 | 0.16000E 03 | 0.69000E 01 | 0.13500E 01 | 0.00000E 00 | 0.0         | 0.0         | 0.33000E 01 | 0.10000E 03 | 0.26300E-01 |
| 23         | 0.10100E 01 | 0.10110E 02 | 0.33800E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.73800E 03 | 0.36600E 01 | 0.60000E 01 | 0.80000E 01 | 0.30000E-01 | 0.15000E 01 | 0.60000E 01 | 0.10000E 03 | 0.16100E-01 |
| 24         | 0.30100E 01 | 0.10580E 02 | 0.33200E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.24200E 03 | 0.62000E 01 | 0.21500E 01 | 0.27000E 01 | 0.45800E-02 | 0.50000E 00 | 0.90000E 01 | 0.10000E 03 | 0.17500E-01 |
| 25         | 0.10500E 01 | 0.10200E 02 | 0.33700E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.90000E 02 | 0.10400E 02 | 0.25000E 01 | 0.18000E 01 | 0.88700E-02 | 0.70000E 00 | 0.62000E 01 | 0.10000E 03 | 0.17600E-01 |
| 26         | 0.20400E 01 | 0.25740E 02 | 0.10900E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.27000E 02 | 0.68000E 01 | 0.23000E 01 | 0.26000E 01 | 0.12300E-01 | 0.80000E 00 | 0.69000E 01 | 0.10000E 03 | 0.16400E-01 |
| 27         | 0.20900E 01 | 0.23530E 02 | 0.23100E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.54000E 02 | 0.73400E 01 | 0.16500E 01 | 0.17000E 01 | 0.10000E-01 | 0.10000E 01 | 0.78000E 01 | 0.10000E 03 | 0.18100E-01 |
| 28         | 0.31000E 01 | 0.33600E 02 | 0.23100E-01 | 0.20000E 01 | 0.54550E 00 | 0.10000E 01 | 0.54000E 02 | 0.73400E 01 | 0.12500E 01 | 0.11000E 01 | 0.10000E-01 | 0.11000E 01 | 0.88000E 01 | 0.10000E 03 | 0.18100E-01 |
| 29         | 0.31600E 01 | 0.23430E 02 | 0.23000E-01 | 0.30000E 01 | 0.54550E 00 | 0.10000E 01 | 0.54000E 02 | 0.73400E 01 | 0.25000E 01 | 0.14000E 01 | 0.30200E-01 | 0.25000E 01 | 0.69000E 01 | 0.10000E 03 | 0.16500E-01 |
| 30         | 0.31100E 01 | 0.27820E 02 | 0.23100E-01 | 0.10000E 01 | 0.67400E 00 | 0.10000E 01 | 0.21000E 02 | 0.10810E 02 | 0.25000E 01 | 0.24000E 01 | 0.60000E-02 | 0.60000E 00 | 0.68000E 01 | 0.10000E 03 | 0.18300E-01 |
| 31         | 0.31200E 01 | 0.27810E 02 | 0.23100E-01 | 0.10000E 01 | 0.67400E 00 | 0.10000E 01 | 0.21000E 02 | 0.10810E 02 | 0.21500E 01 | 0.27000E 01 | 0.32400E-02 | 0.80000E 00 | 0.68000E 01 | 0.10000E 03 | 0.15900E-01 |
| 32         | 0.31300E 01 | 0.27800E 02 | 0.23100E-01 | 0.10000E 01 | 0.67400E 00 | 0.10000E 01 | 0.21000E 02 | 0.10810E 02 | 0.26000E 01 | 0.26000E 01 | 0.77800E-02 | 0.70000E 00 | 0.68100E 01 | 0.10000E 03 | 0.17800E-01 |
| 33         | 0.30500E 01 | 0.23440E 02 | 0.23000E-01 | 0.40000E 01 | 0.54550E 00 | 0.10000E 01 | 0.54000E 02 | 0.73400E 01 | 0.15000E 01 | 0.14000E 01 | 0.94300E-02 | 0.00000E 00 | 0.58000E 01 | 0.10000E 03 | 0.12000E-01 |
| 34         | 0.30800E 01 | 0.23460E 02 | 0.23100E-01 | 0.50000E 01 | 0.54550E 00 | 0.10000E 01 | 0.54000E 02 | 0.73400E 01 | 0.14000E 01 | 0.80000E 00 | 0.0         | 0.0         | 0.39000E 01 | 0.10000E 03 | 0.23800E-01 |
| 35         | 0.10400E 01 | 0.28910E 02 | 0.33700E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.80000E 02 | 0.65700E 01 | 0.24000E 01 | 0.28000E 01 | 0.22000E-01 | 0.11000E 01 | 0.63000E 01 | 0.10000E 03 | 0.16700E-01 |
| 36         | 0.11000E 01 | 0.77190E 02 | 0.23200E-01 | 0.10000E 01 | 0.54550E 00 | 0.10000E 01 | 0.16000E 02 | 0.74500E 01 | 0.52000E 01 | 0.76000E 01 | 0.40400E-01 | 0.33000E 01 | 0.14400E 02 | 0.10000E 03 | 0.16000E-01 |
| 37         | 0.21200E 01 | 0.88300E 02 | 0.23200E-01 | 0.10000E 01 | 0.59470E 00 | 0.10000E 01 | 0.60000E 01 | 0.11370E 02 | 0.61000E 01 | 0.98000E 01 | 0.12100E 00 | 0.48000E 01 | 0.22000E 02 | 0.10000E 03 | 0.15300E-01 |
| 38         | 0.40900E 01 | 0.11855E 03 | 0.21800E-01 | 0.10000E 01 | 0.60610E 00 | 0.10000E 01 | 0.60000E 01 | 0.95300E 01 | 0.24000E 01 | 0.15200E 02 | 0.17000E 00 | 0.68000E 01 | 0.22100E 02 | 0.10000E 03 | 0.17300E-01 |
| 39         | 0.40700E 01 | 0.27130E 02 | 0.22500E-01 | 0.10000E 01 | 0.67400E 00 | 0.10000E 01 | 0.21000E 02 | 0.10810E 02 | 0.11000E 01 | 0.60000E 00 | 0.60000E-02 | 0.60000E 00 | 0.23000E 01 | 0.10000E 03 | 0.16800E-01 |
| 40         | 0.40800E 01 | 0.27140E 02 | 0.22500E-01 | 0.10000E 01 | 0.67400E 00 | 0.10000E 01 | 0.21000E 02 | 0.10810E 02 | 0.10000E 01 | 0.80000E 00 | 0.60000E-02 | 0.80000E 00 | 0.23000E 01 | 0.10000E 03 | 0.16500E-01 |
| 41         | 0.41200E 01 | 0.27130E 02 | 0.22500E-01 | 0.10000E 01 | 0.67400E 00 | 0.10000E 01 | 0.21000E 02 | 0.10810E 02 | 0.80000E 00 | 0.40000E 00 | 0.52000E-02 | 0.60000E 00 | 0.23000E 01 | 0.10000E 03 | 0.15700E-01 |
| 42         | 0.11400E 01 | 0.11280E 02 | 0.23100E-01 | 0.10000E 01 | 0.60000E 00 | 0.25000E 00 | 0.60000E 02 | 0.10060E 02 | 0.00000E 00 | 0.47000E 01 | 0.31600E-01 | 0.13000E 01 | 0.77000E 01 | 0.10000E 03 | 0.17000E-01 |
| 43         | 0.11500E 01 | 0.11270E 02 | 0.23200E-01 | 0.10000E 01 | 0.40910E 00 | 0.25000E 00 | 0.59000E 02 | 0.10150E 02 | 0.10000E 02 | 0.93000E 01 | 0.48900E-01 | 0.32000E 01 | 0.76000E 01 | 0.10000E 03 | 0.17200E-01 |
| 44         | 0.11600E 01 | 0.11460E 02 | 0.23200E-01 | 0.10000E 01 | 0.25910E 00 | 0.10000E 00 | 0.59000E 02 | 0.10060E 02 | 0.77000E 01 | 0.90000E 01 | 0.33300E-01 | 0.21000E 01 | 0.91000E 01 | 0.10000E 03 | 0.16800E-01 |
| 45         | 0.21100E 01 | 0.10010E 02 | 0.21100E-01 | 0.10000E 01 | 0.58180E 00 | 0.25000E 00 | 0.60000E 02 | 0.10670E 02 | 0.33000E 01 | 0.30000E 01 | 0.67800E-02 | 0.60000E 00 | 0.80000E 01 | 0.10000E 03 | 0.17200E-01 |
| 46         | 0.50200E 01 | 0.85100E 01 | 0.22400E-01 | 0.10000E 01 | 0.53140E 00 | 0.25000E 00 | 0.60000E 02 | 0.11400E 02 | 0.34000E 01 | 0.36000E 01 | 0.0         | 0.0         | 0.45500E 01 | 0.50000E 02 | 0.17800E-01 |
| 47         | 0.50400E 01 | 0.84600E 01 | 0.22300E-01 | 0.10000E 01 | 0.53140E 00 | 0.25000E 00 | 0.60000E 02 | 0.11400E 02 | 0.44500E 01 | 0.57000E 01 | 0.22000E-01 | 0.20000E 01 | 0.46800E 01 | 0.10000E 03 | 0.17100E-01 |
| 48         | 0.50300E 01 | 0.82100E 01 | 0.21600E-01 | 0.10000E 01 | 0.53140E 00 | 0.25000E 00 | 0.60000E 02 | 0.11400E 02 | 0.54000E 01 | 0.64000E 01 | 0.25000E-01 | 0.26000E 01 | 0.10350E 02 | 0.15000E 03 | 0.16500E-01 |
| 49         | 0.50100E 01 |             |             |             |             |             |             |             |             |             |             |             |             |             |             |

TABLE B-2  
ATTRITION RESULTS FROM PARTITIONED CHAMBERS

| ARA<br>SN<br># | V     | W      | Cat.  | $\phi$ | Inj.<br>Area<br>(%) | #<br>Or | Equiv.<br>Or. Dia.<br>(10 <sup>-3</sup> in.) | $\bar{R}$ | $\Delta R$ | $\bar{M}_{PLI}$ | $R_{PLI}$ | %<br>$\Delta W$ | #<br>CS | $\bar{D}_E$ |
|----------------|-------|--------|-------|--------|---------------------|---------|----------------------------------------------|-----------|------------|-----------------|-----------|-----------------|---------|-------------|
| IV-C           | 27.12 | 0.0225 | 20-25 | 0.674  | 100                 | 21      | 10.81                                        | 1.6       | 1.6        | 0.0050          | 0.50      | 7.42            | 100     | NA          |
| IV-D           | 27.14 | 0.0225 | 20-25 | 0.674  | 100                 | 21      | 10.81                                        | 0.85      | 0.3        | 0.0033          | 0.20      | 6.8             | 100     | NA          |
| IV-E           | 27.10 | 0.0225 | 20-25 | 0.674  | 100                 | 21      | 10.81                                        | 3.4       | 2.4        | 0.0055          | 0.50      | 12.26           | 100     | NA          |
| IV-F           | 27.13 | 0.0225 | 20-25 | 0.674  | 100                 | 21      | 10.81                                        | 1.4       | 1.2        | 0.0308          | 0.35      | 6.55            | 100     | NA          |

\*Partition Configurations

IV-C — Single Axial Screen  
IV-D — Two Axial Screens  
IV-E — Circular Partition  
IV-F — Radial Partition

## APPENDIX C

### THREE DIMENSIONAL, MULTI-MATERIAL TRANSIENT HEAT TRANSFER PROGRAM

Bell Aerospace Company Program XY 6217 is a generalized heat transfer program for computing the three-dimensional transient temperature distribution in an arbitrary physical system. It employs a mathematical model which can consist of as many as 500 discrete elements or network nodes. Any node may be involved in a transfer of heat by conduction and/or radiation interchange with any other node in the network. Temperature-dependent multimaterial thermal properties may be functionally inputted. Boundary conditions are imposed by relating nodes in the network to control nodes by conduction, convection, and/or radiation. Radiation to space and incident solar and/or nocturnal radiation may also be included. For the control nodes, the known temperatures and, for convection, the heat transfer film coefficients may be held constant or may vary with time. The film coefficients may be dependent upon the resulting temperatures of the related network nodes. Known levels of heat flux versus time, either adding heat to or removing heat from selected network nodes, may also be considered. An additional option allows the setting of prescribed temperature limits versus time for selected network nodes. In this option, the temperature of the governed nodal points are computed and compared with the limits. The temperatures then assigned to these nodes will be less than, greater than, or equal to the limits, depending upon the instructions. The program output includes the network node temperatures at selected time intervals and, if desired, the values of heat flux by the various modes of heat transfer to or from selected nodal points in the network and to or from selected control nodes.

#### PROGRAMMED HEAT TRANSFER EQUATIONS AND NOMENCLATURE

At the outset of an analysis, the object being studied is subdivided into several elements and the mass centroid (node) of each element is located. These nodes, comprising a network, are numbered, however, in the equations which follow, the network nodes will be designated as either  $i$  or  $j$ . Node  $i$  is that node for which the temperature or heat flux is being computed at a point in time and node  $j$  is any of the other nodes related to node  $i$  by conduction and/or radiation. The last node or the total of the nodes is referred to in the equations as  $N$ .

The boundary conditions of various modes of heat transfer are imposed on the network nodes by means of control nodes. In an actual problem, each control node within a specific mode of heat transfer is numbered but is represented in the equations by one of the following subscripts:

- $w$  — A control node conducting heat to or from node  $i$  where the known control temperature may vary with time.
- $W$  — The total number of conduction control nodes.
- $x$  — A control node accounting for heat transfer by convection to or from node  $i$  where the known control temperature and/or film coefficient may vary with time.
- $X$  — The total number of convection control nodes.
- $y$  — A control node radiating heat to or from node  $i$  where the known control temperature may vary with time.

- Y — The total number of radiation control nodes.
- z — A control node wherein the heat transfer convective film coefficient to node i is dependent upon the temperature of node i and the known control temperature is constant with time.
- Z — The total number of temperature-dependent convection control nodes.

In addition, time varying levels of heat flux,  $q_c$ , may be applied directly to any network nodes.

The equation for describing the time derivative of the temperature for node i is as follows:

$$\begin{aligned}
 \frac{dT_i}{d\theta} = & \frac{1}{K_1 \rho_i c_{p_i} A_i} \left\{ \sum_{j=1}^N k_{ij} \frac{b_{ij}}{a_{ij}} (T_j - T_i) \right. \\
 & + \sum_{j=1}^N c_i \mathcal{F}_{ij} \left[ (T_j + 460)^4 - (T_i + 460)^4 \right] \\
 & + s_i \left[ -\sigma \epsilon_i (T_i + 460)^4 + G \right] \\
 & + \sum_{w=1}^W k_{iw} \frac{b_{iw}}{a_{iw}} (T_w - T_i) + \sum_{x=1}^X h_x \left( \frac{h_i}{h} \right) b_{ix} (T_x - T_i) \\
 & + \sum_{y=1}^Y c_i \mathcal{F}_{iy} \left[ (T_y + 460)^4 - (T_i + 460)^4 \right] \\
 & \left. + \sum_{z=1}^Z h_z \left( \frac{h_i}{h} \right) b_{iz} (T_z - T_i) + \sum_{c=1}^C q_c \left( \frac{q_i}{q} \right) b_{ic} \right\} \quad (1)
 \end{aligned}$$

where:

|          |                                                                                                 |                                                                                                                         |
|----------|-------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------|
| T        | node or control temperature                                                                     | °F                                                                                                                      |
| $\theta$ | time in units prescribed in the input                                                           | sec, min, or hr                                                                                                         |
| $K_1$    | constant - if time is in seconds, $K_1 = 518400$<br>minutes, $K_1 = 8640$<br>hours, $K_1 = 144$ | in. <sup>2</sup> sec/ft <sup>2</sup> hr<br>in. <sup>2</sup> min/ft <sup>2</sup> hr<br>in. <sup>2</sup> /ft <sup>2</sup> |
| $\rho$   | material density                                                                                | lb/in. <sup>3</sup>                                                                                                     |
| $c_p$    | material specific heat                                                                          | BTU/lb°F                                                                                                                |

|                   |                                                                                                 |                                                                |
|-------------------|-------------------------------------------------------------------------------------------------|----------------------------------------------------------------|
| k                 | material thermal conductivity                                                                   | BTU in./ft <sup>2</sup> hr°F                                   |
| A                 | volume of node i                                                                                | in. <sup>3</sup>                                               |
| a                 | distance between nodal points i and j or control node                                           | in.                                                            |
| b                 | surface area for heat flow between nodes i and j or control node                                | in. <sup>2</sup>                                               |
| c $\mathcal{F}$   | surface area times gray body radiation interchange factor between nodes i and j or control node | in. <sup>2</sup>                                               |
| $\sigma$          | Stefan-Boltzman constant                                                                        | $1.73 \times 10^{-9}$<br>BTU/ft <sup>2</sup> hr°R <sup>4</sup> |
| $\epsilon$        | surface emissivity                                                                              |                                                                |
| s                 | external surface area of node i                                                                 | in. <sup>2</sup>                                               |
| G                 | constant heat flux                                                                              | BTU/ft <sup>2</sup> hr                                         |
| h                 | heat transfer film coefficient                                                                  | BTU/ft <sup>2</sup> hr°F                                       |
| q                 | heat flux                                                                                       | BTU/ft <sup>2</sup> hr                                         |
| $h_i/h$ & $q_i/q$ | ratio of the local heating to a node to the value input for the control                         |                                                                |
| H                 | joint conductance                                                                               | BTU/ft <sup>2</sup> hr°F                                       |

If the physical system being studied is comprised of more than a single material, the network nodes are doubly subscribed and the corresponding thermal properties, i.e., thermal conductivity, specific heat and density are used in the solution of Equation 1. In the case of conduction across a material interface, the conductance ratio  $b_{ij}/a_{ij}$  is computed by

$$b_{ij}/a_{ij} = \frac{b_{ij}}{\frac{a_{ij}}{k_i} + \frac{a_{ij}}{k_j} + \frac{1}{H_{ij}}} \quad (2)$$

#### MANNER OF SOLUTION

After Equation 1 has been used to determine the time derivative of the network nodes at some time  $\theta$ , the temperatures after a finite time interval ( $\Delta\theta$ ) are determined employing the first two terms of a Taylor's series, i.e.:

$$T_{i(at \theta + \Delta\theta)} = T_{i(at \theta)} + \Delta\theta \frac{dT_i}{d\theta(at \theta)} \quad (3)$$

This procedure is repeated until the time histories of the temperatures have been obtained for the required time duration. The increment of time ( $\Delta\theta$ ) used in the computations must satisfy the criteria for obtaining a stable solution to the finite difference equations; therefore, an allowable  $\Delta\theta$  is computed for each node and the smallest  $\Delta\theta$  is retained for the temperature computations.

## PROGRAM OUTPUT

Results of the calculations are output at prescribed print intervals. These print intervals may be changed during the run. In addition to printing the temperatures of all nodes at each print time, the values of the heat flux contributed to each node from adjacent nodes and from the related control nodes are available. The heat content (BTU) versus time for any node or group of nodes is also available as well as the average temperature of the entire network accounting for the thermal mass of the nodes.

To facilitate operations with the computer, temperatures may be read in or out on tape and tapes can be generated for obtaining plots of temperatures versus time using the autoplottter.