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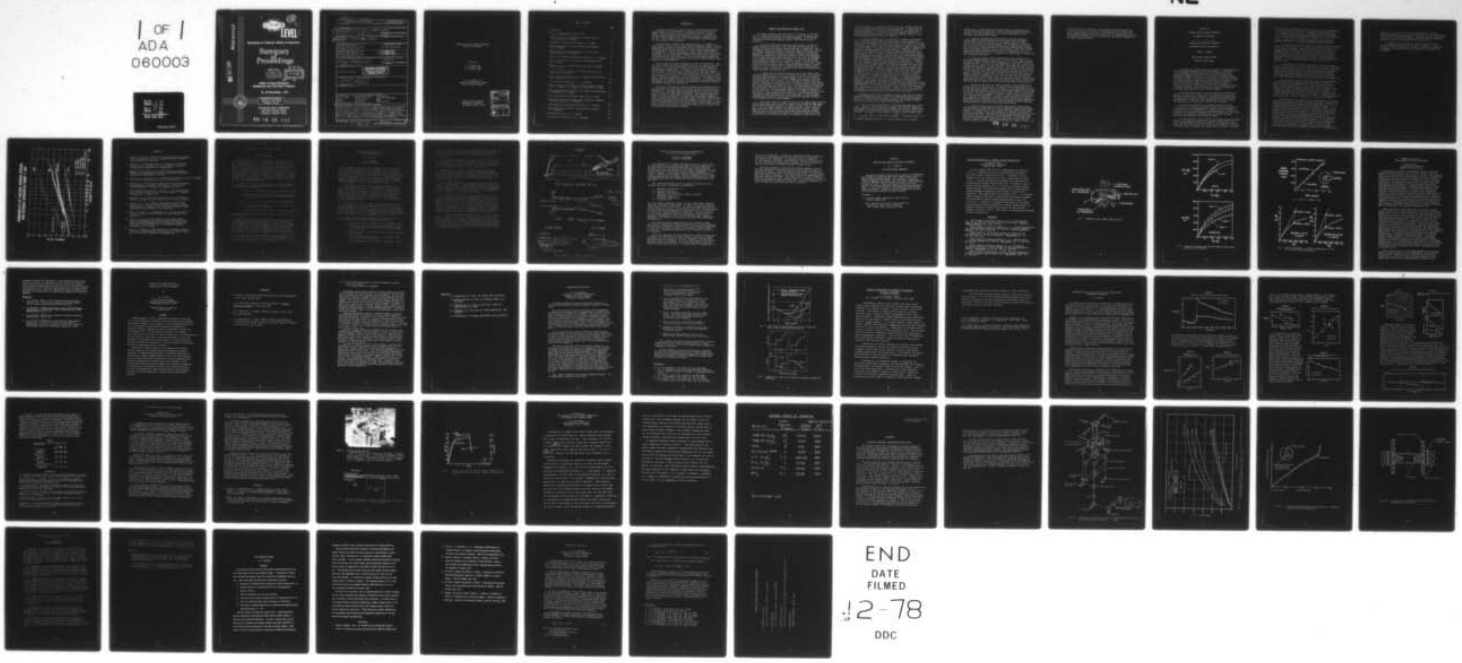

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Workshop on Thermal Shock of Ceramics:

Summary of Proceedings

Edited by:
P. F. Becher, NRL
S.W. Freiman, NRL
A.M. Diness, ONR

Office of Naval Research
Metallurgy and Ceramics Program

19, 20 December, 1977

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A workshop on thermal shock behavior of ceramic materials was held on December 19 and 20, 1977. Experts in the area discussed several aspects of thermal shock of ceramics, including theory and analysis, applications limitations and thermal shock test methods. In addition to 18 abstracts, a summary of the main points presented or discussed during this meeting is included in this report. A number of gaps in understanding of thermal shock behavior of ceramics and specific recommendations on how to overcome them are identified.

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19, 20 December 1977
Metallurgy and Ceramics Program
Office of Naval Research

Office of Naval Research
Department of the Navy
Arlington, Virginia 22217

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INTRODUCTION

In order to consider the state-of-the-art on understanding of thermal shock resistance and related matters for ceramic materials, a number of experts in the area were invited to participate in a discussion on the thermal stress and thermal shock resistance of ceramics. It was held at the Naval Surface Weapons Center, White Oak Laboratory on December 19 and 20, 1977. Special thanks are due to Mr. Frank Koubek of that laboratory for his assistance with this meeting.

Technical areas discussed at the meeting included: (1) theory and analysis (heat transfer, finite element methods, aerothermal analysis, thermal stress analysis, statistical approaches, fracture mechanics concepts, thermal-mechanical coupled failure, materials factors and microstructural aspects of the problem; (2) several applications in which thermal shock behavior is of concern (e.g., re-entry, electromagnetic domes, rocket nozzles and turbine components); (3) test technologies currently being used (such as quenching, cyclic flaws method, laser heating, disc tests, engine exhaust exposure and rig tests); (4) the comparision between calculated and observed thermal stress behavior for brittle materials.

Active discussion occurred throughout the course of this meeting. Important points brought out during the meeting, and during a summing-up session led by Dr. S. W. Freiman are presented in the section entitled "Summary of ONR Workshop on Thermal Shock". In addition to this Summary section, all of the abstracts submitted for the invited discussions have been compiled in this report. It was the sense of the meeting that there were several important areas in need of either further work by materials specialists or of more careful recognition by designers. They are presented in the summary section. Several important exchanges occurred during the meeting. For example, systems designers were provided with insight into current materials development efforts now underway, and materials developers and researchers heard about consideration systems designers are concerned with.

The results of the meeting revealed an apparent and important gap having to do with shock for thermal shock for ceramics and other brittle materials. Understanding of thermally caused failure has advanced since earlier views of the 1950's era. This newer understanding seems not yet to have had a significant influence on thermal shock test methods. Relationships between test methods and in-service conditions often has not been considered properly. Clearly, thermal shock failure is a complex field; theoretical simplifications have helped understand materials behavior somewhat. But real control of the difficulties encountered in service involves detailed consideration of a large number of conditions involved in each specific case. Such systematic studies cannot but help to define the limitations (and capabilities) of ceramics and related materials when thermally stressed.

SUMMARY OF ONR WORKSHOP ON THERMAL SHOCK

This summary represents the views of Drs. P. F. Becher, S. W. Freiman, and A. M. Diness, which evolved from discussions held at the ONR Workshop as well as follow-up discussions with several attendees. It is felt that the following are the most important points to have evolved from this Workshop.

1. The first priority need, especially from the point of view of the systems and material designer, is for a much more extensive data base on materials of interest for thermal shock applications. In particular, data is needed on the strength, thermal conductivity, thermal diffusivity, specific heat, thermal expansion coefficients, and elastic modulus as a function of temperature. If possible, strength data should be obtained from specimens removed from components of a particular material, in order to be representative of actual flaw distributions expected in practice. The size dependence of the strength of ceramics must be recognized. In addition, the strength data determinations should be of a statistical nature. It should be taken in an environment similar to that in which the component will be used. Crack growth data is also needed in order to determine possible effects of thermal cycling. The use of strain-to-failure rather than strength data may make computation easier. In addition, such data may be useful for materials which exhibit non-linear stress-strain curves.

2. It seems clear that ranking of materials as to their thermal shock resistance depends both on the test used to rank them as well as on the application for which the material is being considered. That is, the ranking of different ceramic materials will strongly depend on the severity of the heat flux which they will see in service. It is important to recognize that the ranking of materials in a particular heat flux situation such as a water quench may be quite different from the ranking in a less severe thermal stress environment such as that seen by a radome in flight. The possible change in ranking of various materials with changes in heat flux has not been sufficiently defined for many materials. Thus, the designer generally must depend on laboratory scale tests in order to judge whether a material is suitable for specific service applications. Except possibly in the reentry case, the correlation between laboratory and field experiments has not been performed. A test heirarchy of increasing sophistication is needed, which ranged from laboratory screening tests through to tests of actual components.

3. There were a number of laboratory scale tests for thermal shock discussed. These include quench tests, laser tests, flame tests (including the NASA wedge) and the Southern Research disk test. Of these, it appears that the flame test may, in fact, not measure thermal shock resistance at all, since it does not appear to put the sample through a large enough thermal gradient. The NASA wedge test may be a suitable thermal stress test provided that measurements of temperature distributions and temperature responses are known so that the stress distribution throughout the wedge can be matched to that which will be

seen by an actual part. It would also be useful if the residual strength of the material after a number of cycles could be measured. It appears that the Southern Research disk test would be quite expensive as a screening test for most ceramic materials. In addition, it does not seem that materials of different types such as graphite and Si_3N_4 can be compared, because of their very different responses to electrical induction.

The laser test appears to be a suitable test for thermal shock measurements in the laboratory provided that precautions are taken regarding the size of the beam compared to the area and thickness of the particular specimen being tested. For example, the beam area to sample area should be no less than 0.25 and no greater than 0.9, while the beam diameter to sample thickness should be no less than 2.0. An advantage of this test is the ability to control the thermal flux through control of the beam conditions. Otherwise, spurious results can be obtained and over-optimistic determinations of thermal stress resistance will result. To date, the quench test still seems to be the most useful thermal shock screening test for ceramic materials development purposes. Whether one measures the strength decrease after thermal shock or the internal friction changes in these specimens, similar results seem to occur although care must be taken in comparing these two types of measurements. In addition, care must clearly be taken in comparing materials with one another. In fact, they should not be compared based on the same size specimens but rather would best be compared based on samples of the same Biot's modulus (since the size dependence for different materials can be quite different and probably should be determined as well). One clear drawback of the use of this test is that it represents one of the most severe thermal shock situations. Extrapolation of these results to less severe cases could be quite misleading. While it would be possible to perform the quench tests in less severe environments, for instance, in a silicone oil rather than water, it is probable that many materials will not exhibit a decrease in strength up to quench temperatures above which results become quite questionable because of inaccuracies in temperature measurements. One should also recognize that a quench test imparts different stress distributions (and so activates a different population of flaws) than a heat-up test. The latter would tend to cause failure from internal processing defects rather than surface flaws. However, at this time, the quench test appears to be the best one available at least as a first screening test for new materials being considered for thermal stress resistance.

It appears that more work is necessary on many of these tests in order to accurately determine the stress distribution throughout the specimens during quenching or thermal shocking processes. This more quantitative treatment of the tests will better enable the system designer to use the data to predict behavior of the material in an actual service condition.

4. The use of thermal resistance "R" factors was discussed fairly extensively. However, it was pointed out by Professor Hasselman that there are a large number of R factors, each of which depends on a particular thermal stress environment, so that ranking of materials according to R factors can be quite dangerous. For example, at high fluxes, $R = \frac{\sigma (1-\nu)}{\alpha E}$, while at lower fluxes $R' = \frac{\kappa \sigma (1-\nu)}{\alpha E}$, where σ is the fracture strength, ν is Poisson's ratio, σ is the thermal expansion coefficient, E is Young's modulus and κ is thermal

conductivity. It was brought up that these R factors are dimensional and are thus affected by size and shape. These are not desirable from a design state point for ranking materials as the size and shape factors can be different, thus affecting the ranking.

5. Professor Hasselman discussed the idea of retained strength as a possible means of ranking materials for their thermal stress resistance. The question becomes one of whether or not a material that has a lower critical ΔT but loses a smaller percentage of its strength during the quench may, in fact, be a better candidate for a particular thermal stress environment than one which requires quenching from a higher temperature but undergoes nearly catastrophic failure as a result of that quench. It will be up to the designer to determine whether other stresses, e.g. aerodynamic, etc., are large enough such that any loss in strength due to thermal shock is unacceptable for a particular application.

6. The use of computer codes to determine the stress distribution in particular components appears to be useful. The use of these codes to analyze the thermal stress problem has proved valuable in the case of reentry nosetips. Nevertheless, while it is felt that the methodology in the code is likely to be correct, there are questions on the data input to the codes such that the stress distributions obtained for different materials, and therefore the way in which materials are ranked for thermal stress applications, may not in fact be correct. It should be made clearer what assumptions and what materials variables go into the use of the codes. In particular, the sensitivity of the final results to changes to material properties that are used to calculate the final stress distributions needs to be addressed. There is a clear need for more test data which can be correlated with predictions from the various types of thermal stress codes which are available. Some correlations between predictions and tests have been established for graphite but little has been performed on other material.

7. The use of proof tests as a means of assuring quality components for various applications was discussed. The general feeling seemed to be that for applications such as reentry nosetips, the expense of the final product, especially for carbon-based materials, may warrant the use and development of proof tests, but that for less expensive applications such as radomes, there is serious question whether it would be economically feasible to devise and use various proof tests to screen these components. In addition, there was clearly no easy way to test components of complex shape, and hence a complex stress field situation. There seems to be no indication at this time that non-destructive evaluation has reached the degree of sophistication to enable screening of ceramic components for flaws which will cause failure under the known stresses.

8. There was some discussion on possible techniques to improve thermal shock resistance of ceramic materials and to develop new materials with greater resistance to thermal stresses. One of these techniques, which has been demonstrated, is to increase the fracture toughness of the material by the use of microcracking phenomena. Other possible avenues of approach are through materials with lower elastic moduli, the discovery and development of materials having lower thermal expansion coefficients, and the tailoring of the thermal diffusivity of a material to meet the particular application; that is, there are situations where one prefers higher thermal diffusivity and those in which a low

thermal diffusivity is preferred. Also, the point was raised that the use of compressive layers on the surface of components appears to enhance their thermal shock resistance. The use of fiber composites might possibly also lead to greater thermal shock resistant materials. As demonstrated by the work on graphite, the microstructure of a material can also be quite important to its thermal shock resistance.

WORKSHOP ON

THERMAL STRESS AND SHOCK RESISTANCE

OF CERAMICS AND GRAPHITE

MATERIALS AND STRUCTURES TECHNOLOGY FOR REENTRY VEHICLE APPLICATIONS

ROBERT J. EDWARDS

NAVAL SURFACE WEAPONS CENTER

REENTRY SYSTEMS BRANCH

The significantly higher performance required of advanced ballistic (BRV) and maneuvering (MaRV) reentry vehicles places a high priority on the development of new materials which can withstand the benign and hostile environments dictated by such systems as extended range POSEIDON and TRIDENT. The materials and structural technology problems are however, intimately related and many phases of the materials development effort must be coordinated with a valid structural evaluation. Due to the complex behavior under aerothermal and dynamic loading of materials such as bulk graphites and carbon-carbon composites it is necessary to provide a parallel advance in structural technology in order to perform an accurate evaluation of material development efforts and to perform design analyses for future flight systems.

In the following sections the pacing technology problems will be discussed in three general areas: material characterization, material modeling and computer code development. The significant advances that have occurred in the past five years in thermostructural and materials technology will be indicated. This discussion will be directed toward the application of bulk graphites to reentry vehicle nosetips.

Material characterization is an important problem area since it serves to link the material development efforts and the technologies of structures and aero/thermodynamics. Without a valid preliminary characterization it is impossible to determine whether new or "improved" materials offer significant advantages over existing materials, and further downstream, a comprehensive characterization is required to insure accurate design analyses for prototype development.

The characterization effort involves a wide range of tests, including physical and mechanical property tests, ablation tests, thermal stress tests, and establishment of failure statistics. The mechanical property tests, which must be conducted over a wide range of temperature, are used to provide a basis for elastic and non-linear material models. Statistical

scatter, material anisotropy, different moduli in tension and compression and non-linear behavior serves to further increase the data required and results in a large test matrix. The physical property tests (also at temperature) and the ablation tests provide a basis for the thermal model. Failure criteria are established through statistically significant numbers of uniaxial and multiaxial tests. Thermal stress testing serves as a screening tool in making comparative evaluations between candidate materials and is an aid in establishing failure criteria.

The thermal stress disc test developed at Southern Research Institute (SoRI) (Ref 1) has proven to be a valuable screening tool in the development of advanced graphites. New graphites such as Union Carbide's proprietary grades 994 (Ref 2) and CMT (Ref 3) and the non-proprietary GRAPHNOL N3M (Ref 4) have been developed within the past five years. The N3M graphite was developed at Oak Ridge National Laboratory under Naval Surface Weapons Center sponsorship (Ref 5). Figure 1 shows the improvement in the across-grain strain to failure at 2000°F that has been achieved over ATJ-S graphite which previously represented the state-of-the-art.

The development of constitutive equations for advanced graphites is an area that has received a lot of attention in recent years. Non-linear theories have been developed which address transverse isotropy, different stress-strain behavior in tension and compression, temperature-dependent properties and biaxial softening (larger strains for biaxial stress states than for uniaxial states at the same stress level). The Jones-Nelson model (Ref 6) the Batdorf model (Ref 7) and the Cyr-Chinn model (Ref 3) have been shown to correlate well with ground test data (Refs. 3, 8).

The other area of material modeling in which significant progress has been made is that of failure criteria development. The PFAIL model (Ref 9) and the STARVE model (Ref 3) have both been successfully used to predict observed fracture rates of full scale nosetips in rocket nozzle environments which simulate quite well the thermal strains developed in flight. Both models employ Weibull type theories for defining the distribution of tensile failure strains or stresses. Such factors as material anistropy, statistical variability, volume effects, biaxial softening, non-linear stress-strain behavior, temperature dependence and general stress states are accounted for in the failure modeling.

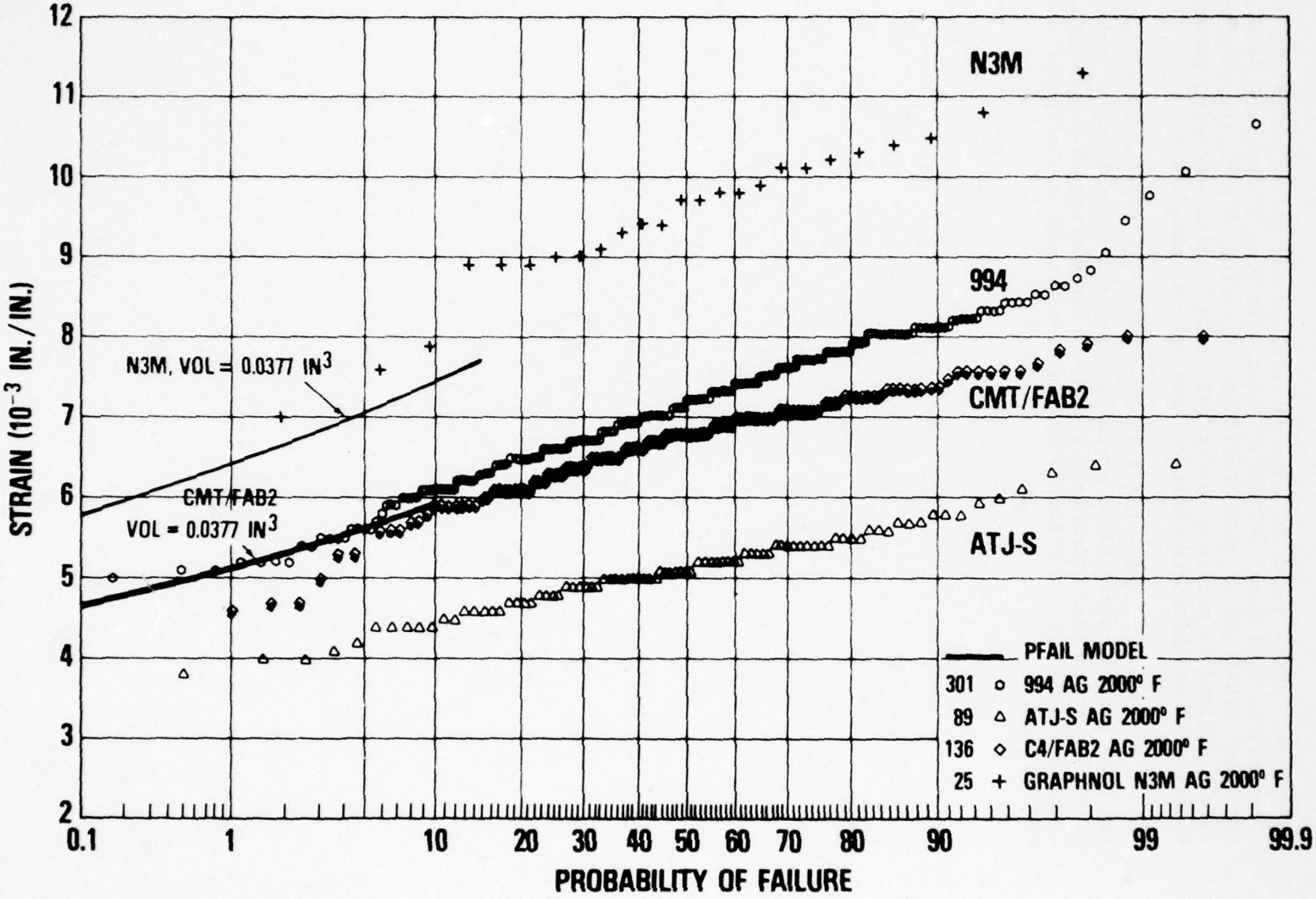
The SAAS-III (Ref 10), DOASIS (Ref 11) and FANCAP (Ref 3) codes are examples of the degree of analytical sophistication usually applied to the design/analysis of graphite nosetips for BRV applications. One or more of the nonlinear material models previously discussed have been incorporated in these codes and they model the geometric aspects of the problem extremely well within their range of applicability (e.g. 2D axisymmetric or plane strain/stress solids). A conservative prediction of thermal strain results if a linear analysis is conducted with different elastic moduli in tension and compression (which is accounted for by iteration of the elastic solution) and also accounting for temperature dependent, transversely isotropic material response. If the PFAIL or STARVE failure criterion is entered with the strain state predicted by a linear, multi-modulus analysis the resulting forecase of probability of survival is also conservative (Refs 3 and 9). The most accurate analysis that can currently be performed is to use one of the newly developed nonlinear models in conjunction with the PFAIL or STARVE failure

criterion. (Refs 3 and 9). The material model/failure criterion combinations that have correlated best with ground tests are Jones-Nelson/PFAIL and CYR-CHINN or BATDORF/STARVE. Classical plasticity theories such as are applied to metals do not predict the biaxial softening phenomenon observed in graphite and therefore are not as satisfactory.

In summary, during the last five years the state-of-the-art has been greatly advanced in graphite materials technology, in materials screening and characterization, in non-linear constitutive modeling, in development of failure criteria and in thermostructural computer code development.

FIGURE 1

PROBABILITY OF FAILURE VERSUS STRAIN FOR SEVERAL GRAPHITES (2000° F, AG)



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AEROHEATING OF TACTICAL MISSILE DOMES

19 December 1977
J.B. Patton - NWC

Missile seeker domes are an integral part of the missile airframe as well as a window for the seeker. Aerodynamic heating can severely effect dome performance. Thermal stresses sufficient to cause a structural failure may be generated; if not, thermal distortion may degrade optical or electromagnetic transmission to the seeker. As with all systems, performance requirements are increasing. Missiles will be launched at greater and greater speeds. Propulsion systems will be more energetic, imparting a greater total impulse to the missile. Increased speed means increased aerodynamic heating. Current analytical methods although theoretically adequate are limited by inadequately characterized materials. These material behavior deficiencies must be corrected if we are to design with confidence.

In concept, the thermal structural analyses of a missile dome is quite simple. Once a flight trajectory has been selected,a sequential series of analyses are performed:

i) characterize the flow field (inviscid and boundary layer)

ii) characterize the dome heating environment (heat transfer coefficient distribution and recovery temperature) based upon flow field

iii) predict dome transient indepth temperature distribution

iv) using predicted temperatures at a selected time, calculate dome structural response

v) apply appropriate failure criteria to structural response

The structural and failure analyses are repeated for other selected times of interest.

In general, current analytical techniques employed are limited to axisymmetric geometry and loading; angle of attack is assumed to be zero. State-of-the-art techniques are used to predict the flow field, heating environment, and indepth temperature distributions. Good agreement between predicted and measured temperatures has been obtained. Conventional, linear finite element methods are used to predict structural response. Finally statistical failure theories are applied to predicted stress distributions to determine probabilities of survival.

Unfortunately, the ceramic materials used have not been well characterized. Mechanical properties have been measured using materials that are not representative of the as-fabricated dome material. Insufficient test data exist to verify any failure theory.

GRAPHITE APPLICATIONS IN TACTICAL MISSILE ROCKET NOZZLES

19 December 1977

J.E. PATTON
NAVAL WEAPONS CENTER

Bulk graphites, because of their relatively low cost, good erosion resistance, and strength at elevated temperatures, are used as nozzle throat materials in many tactical missile rocket propulsion systems. Heat transfer and consequently the mechanisms for material removal are in general, most severe at the nozzle throat. Surface recession (erosion) at the throat decreases the exit velocity of the propellant gases and the specific impoulse. Bulk graphites, for many missions, can provide adequate erosion resistance; the problem then becomes one of designing the nozzle components to avoid a thermostructural failure.

Several different tactical nozzles are shown in Figures 1, 2, and 3. The ALVRJ nozzle (Figure 1) is fully submerged with the graphite insert heated on all but one side. Interior tensile stresses caused a failure and necessitated changing to a better grade of graphite. The SRB graphite throat piece (Figure 2) because of an inability to control the bond gap between the outside diameter and the phenolic support caused a compression failure on one firing and a tensile failure on a second. Subsequent redesign allowed better control of the bond-gap and has been successfully tested.

The original Agile nozzles (Figure 3) performed adequately in the early development phase. However, when a total impulse of the motor was increased twenty-five per cent, the three G-90 graphite rings failed. Subsequent development, first replaced the G-90 with ATJ-5; finally the three throat pieces and two of the phenolic throat supports were replaced with a single piece of ATJ-5. Both fixes were successful. Detailed thermal and structural analyses were performed on all three nozzles.

In concept, the thermal structural analyses of a rocket nozzle is quite simple; a sequential series of analyses are performed:

i) characterize the flow field (inviscid and boundary layer)

ii) characterize the nozzle heating environment (heat transfer coefficient distribution and recovery temperature) based upon flow field

iii) predict surface recession and transient in-depth temperature distribution

iv) using predicted temperatures at a selected time, calculate dome structural response

v) apply appropriate failure criteria to structural response

The structural and failure analyses are repeated for other selected times of interest. However, there are many sources of uncertainty in this analysis. In many instances, results are used qualitatively, not quantitatively.

Erosion predictions within ten per cent are considered excellent, within twenty per are considered excellent. In-depth temperature predictions are also influenced by surface thermo-chemistry. Unfortunately, only the external temperature response of a nozzle is measured, these data are not of much use in validating our thermal models.

Graphite nozzle components are generally supported by phenolic materials. Phenolics decompose and char at elevated temperatures; their mechanical properties are a function of temperature and heating rate. An accurate prediction of the behavior of these materials is required in order to model the support provided to the graphite. This capability does not exist.

In these multi-component systems, even if the support materials could be accurately modeled, bond gaps between components are difficult to hold to close tolerances. Small changed in bond gaps significantly change stress levels. Also the adhesives used in these gaps eventually decompose, supporting the graphite in compression but not tension or shear - a non-linear contact problem with sliding.

The majority of bulk graphites are not well characterized. This is not unexpected when one realizes that mechanical properties are required between room temperature and 5000°F of a transversely isotropic material with different behavior in tension and compression. Bulk graphites also exhibit large variations of properties within a billet and from billet to billet. It should be also mentioned that the high temperature compressive behavior is highly non-linear; nozzle graphite material at high temperature and in compression is the most significant contributor to thermal stresses. High thermal expansion on the inside diameter results in hoop tensile stresses on the outside diameter.

In conclusion, the thermal structural analysis of rocket nozzles is far from an exact science and is really an engineering approach that bounds the extremes of the problem. Predictions of very high or very low factors of safety are meaningful. If the margins are close, however, most designers prefer to fall back upon experience. Successful design of tactical rocket nozzles requires the judicious application of available design tools and data, tempered with experience and understanding of the rocket nozzle problem.

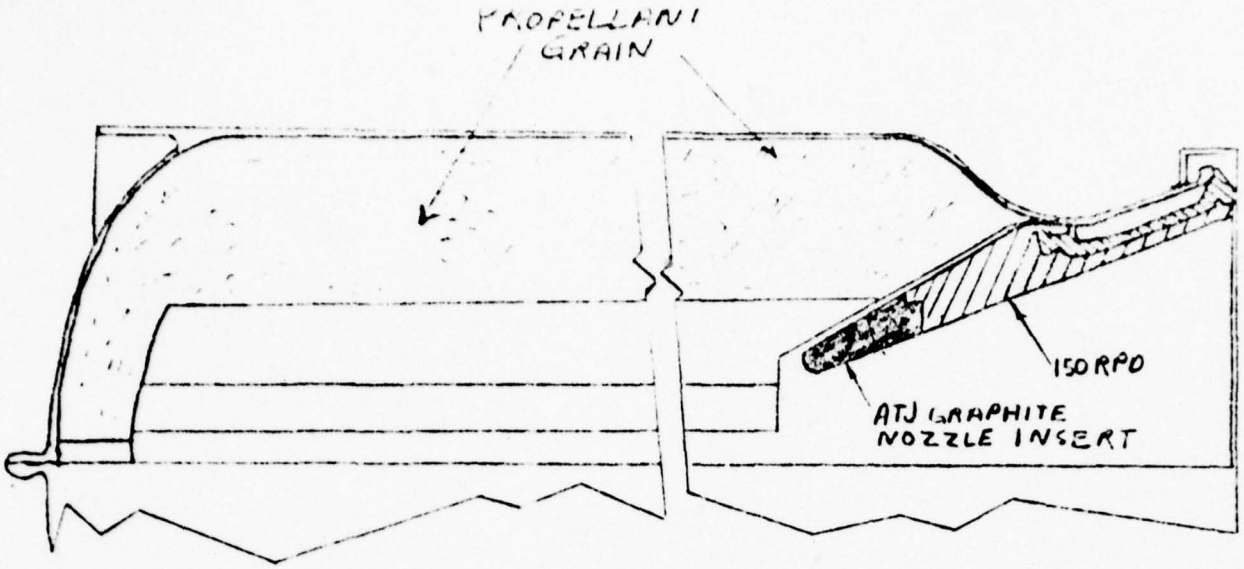

FIG. 1 ALVRJ BOOST MOTOR

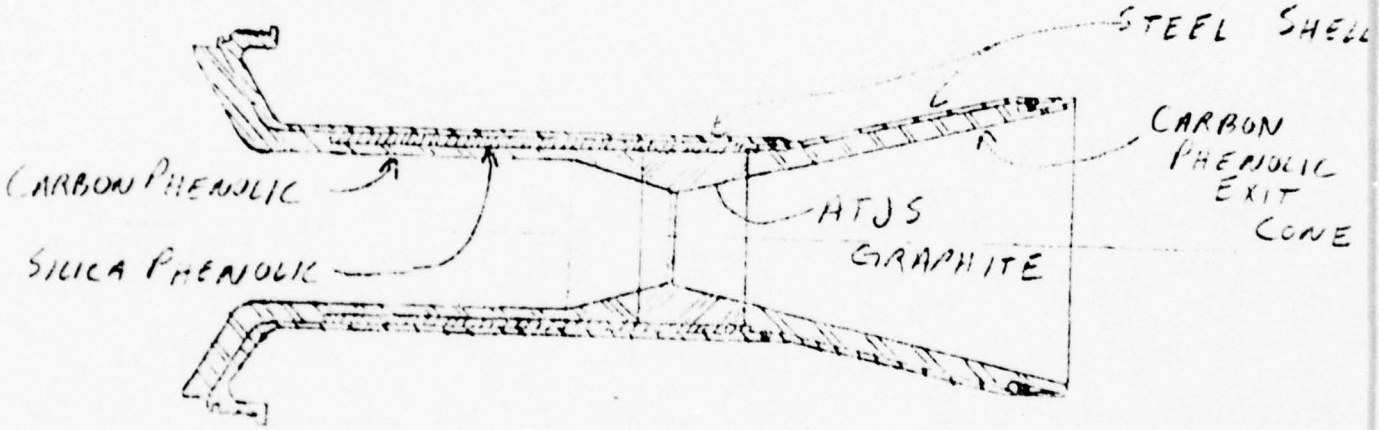

FIG. 2 SRB NOZZLE ASSEMBLY

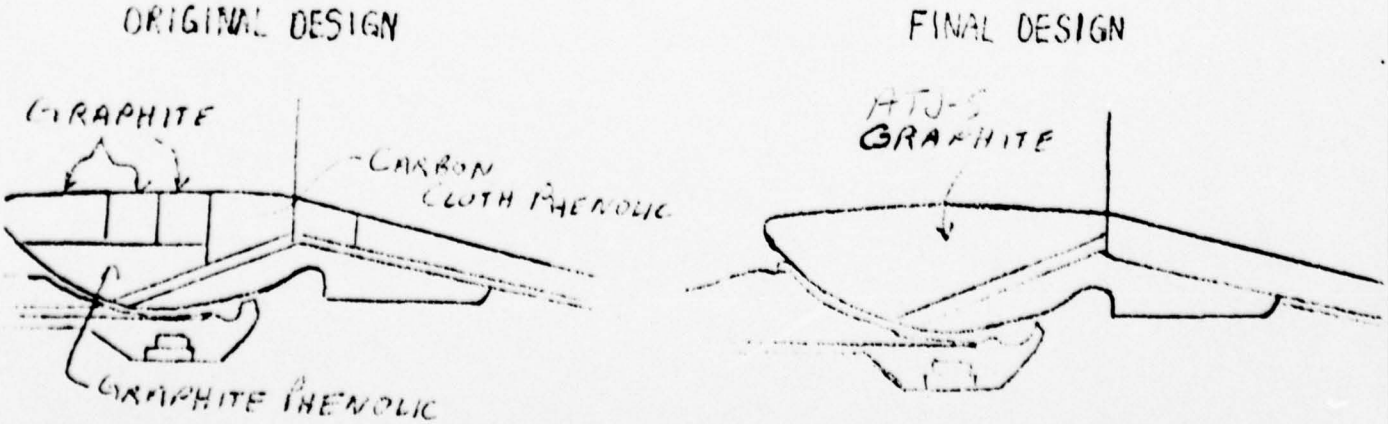

FIG 3. AGILE TVC NOZZLE

Thermal Fatigue Data Design Requirements for Gas Turbine Applications

David W. Richerson
Garrett AiResearch

The ability of a ceramic material to survive in most gas turbine components is largely dependent upon the capability of the material to withstand thermal shock or thermal fatigue. However, little or no thermal fatigue data is available for use in the design and life prediction of ceramic components. To be directly useable in analytical design, data must consist of either 1) direct strain versus time and temperature or 2) temperature distribution and change (thermal response) versus time and temperature plus the thermal and elastic properties of the material.

The following thermal shock or thermal fatigue test approaches have been considered at AiResearch:

1. Cascade rig or engine rig
2. Torch/air quench
3. Combustor/air quench
4. Strength versus ΔT water quench to produce Hasselman type curve
5. Induction heated ring
6. Fluidized bed

The first three approaches appear to have significant disadvantages for obtaining design data. In order to achieve stresses comparable to engine conditions, the test specimen must have an airfoil shape or a taper. This specimen must then be instrumented to obtain the precise temperature distribution as a function of time during an engine-simulating transient. The heat sources for the three approaches cannot be adequately controlled to guarantee reproducible conditions and the specimens cannot be adequately instrumented to precisely determine the temperature distribution.

The fourth approach produces data that permits a semi-quantitative comparison between materials and is a good approach for ranking or screening. However, the heat transfer conditions in this test are quite different from those in a gas turbine. In addition, temperature distribution as a function of time is not measured.

The fifth approach has the potential to produce design data. It permits the application of pure thermal strain and a direct measurement of the strain to failure. However, this approach also has potential problems. It can be used easily for graphite specimens since the graphite can be used directly as the susceptor for the induction coil. Si_3N_4 and most SiC materials do

not act as a susceptor. Therefore a separate susceptor such as graphite or molybdenum must be used and the heat transferred to the ceramic test specimen. Two questions must be answered:
(a) Can adequate gradients be produced to fracture the specimen?
(b) Can conditions be controlled to permit cyclic testing at a subcritical strain to evaluate thermal fatigue life?

The final approach, fluidized bed testing appears to have the most promise for obtaining thermal fatigue data suitable for gas turbine design. Fluidized beds provide rapid heat transfer under well controlled and instrumented conditions. Ceramic specimens have been successfully instrumented and the temperature/time/position response recorded. This type data can then be used to analytically design a test specimen that will simulate in fluidized beds the thermal stresses predicted for a specific component in a gas turbine during maximum transient condition.

ABSTRACT

PRACTICAL HEAT EQUATION SOLUTIONS FOR CERAMICS

W. T. LAUGHLIN

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The causes of thermal stress failure of ceramics are reviewed and the inadequacies of applying conventional thermal shock criteria to ceramic design problems discussed. The importance of detailed transient temperature and thermal stress solutions is emphasized to answer the important question of under what thermal stress conditions a ceramic will fail. Useful analytical solutions to the heat equation for predicting transient temperature response are presented (Refs 1, 2) for a wide variety of surface boundary conditions and thermal environments. Examples of some series analytical solutions are given and their numerical evaluation discussed.

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NONLINEAR DEFORMATION OF A THERMALLY STRESSED GRAPHITE DISK

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An ATJ-S graphite annular disk of a wedge-shaped cross section as in Fig. 1 is subjected to outside to inside temperature gradients of up to 3000°F from an induction heating coil. The change in internal diameter of the disk is measured with a laser tracking device as shown in Fig. 1. The disk deformations are predicted with a modification of the Jones-Nelson nonlinear material model [1-5]. This model is shown to be valid for ATJ-S graphite, which is a transversely isotropic granular composite material with stress-strain behavior that is highly nonlinear, strongly dependent on temperature, and different in tension than in compression. These characteristics are displayed in Fig. 2. The predicted diameter changes at three times in the same test agree with the averages of two perpendicular diameter change measurements within about 3% as seen in Fig. 3. A measure of the degree of nonlinearity of the problem is obtained by observing the substantial differences between elastic and nonlinear solutions for stresses and strains at the inner and outer diameters of the disk in Fig. 4. These results are discussed more completely by Jones and Starrett [6].

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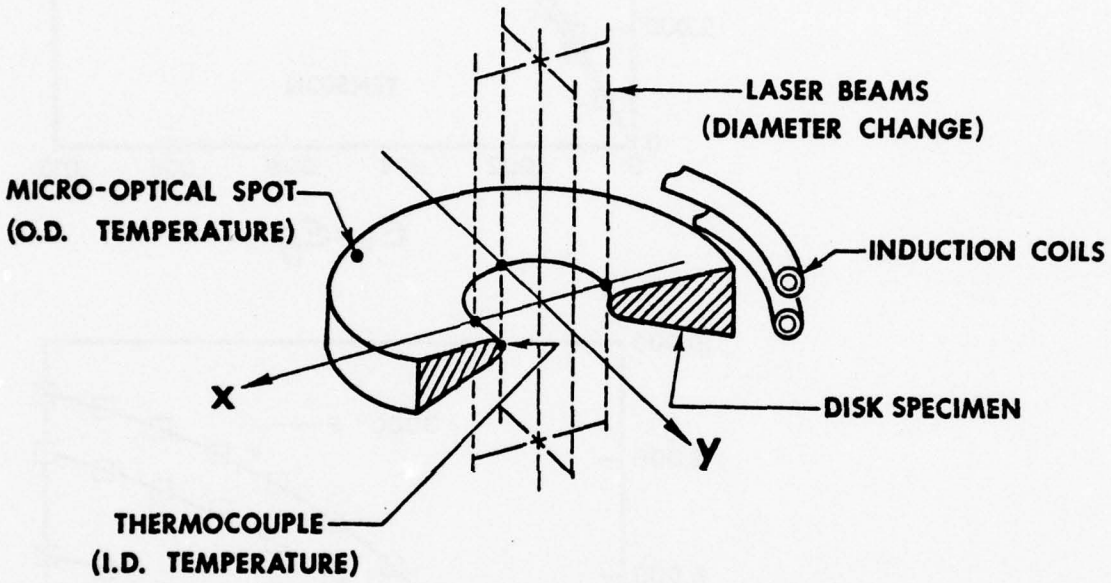

FIG. 1 SCHEMATIC OF SoRI THERMAL STRESS DISK TEST

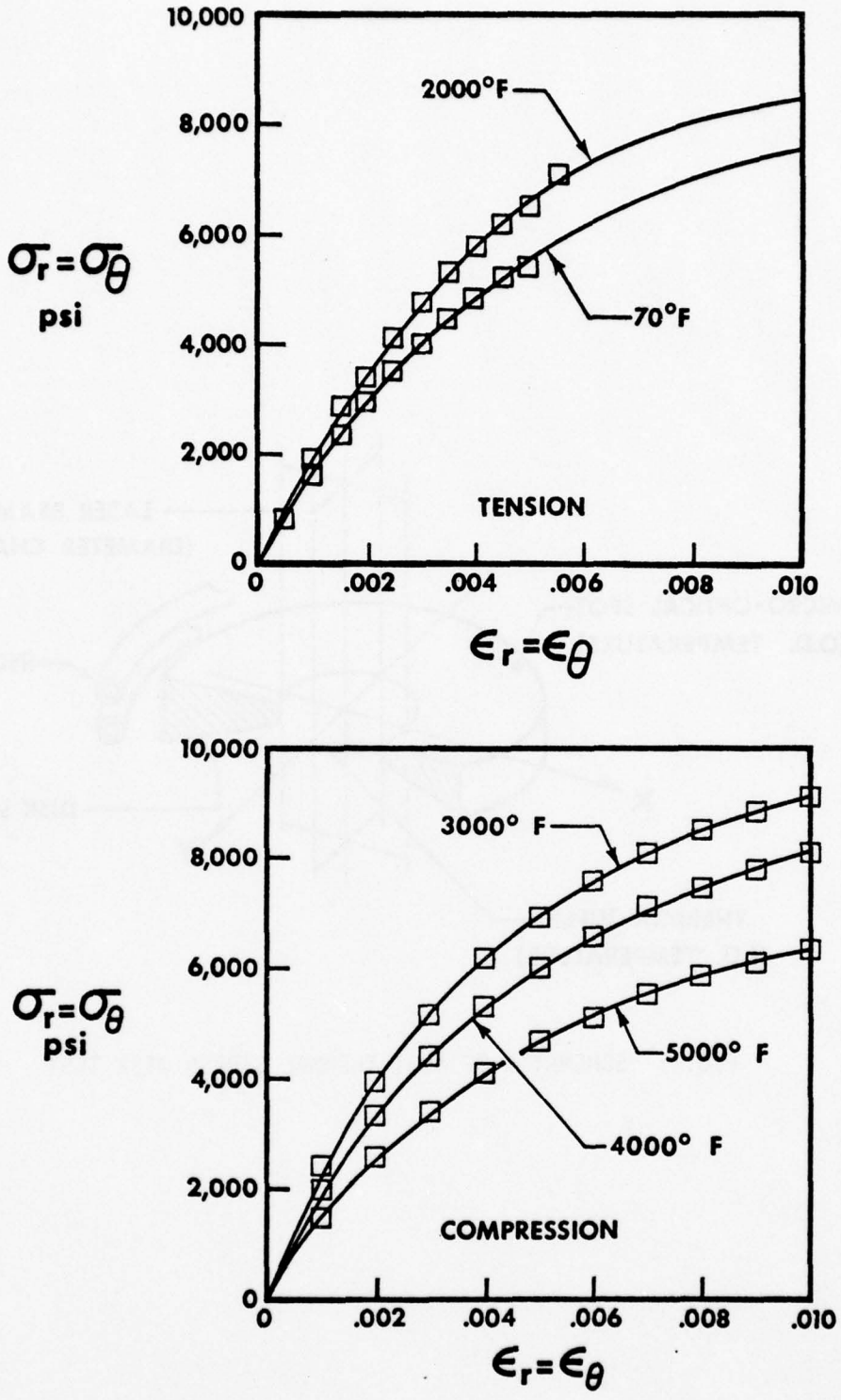

FIG. 2 TEMPERATURE-DEPENDENT NONLINEAR MULTIMODULUS STRESS-STRAIN BEHAVIOR OF ATJ-S GRAPHITE.

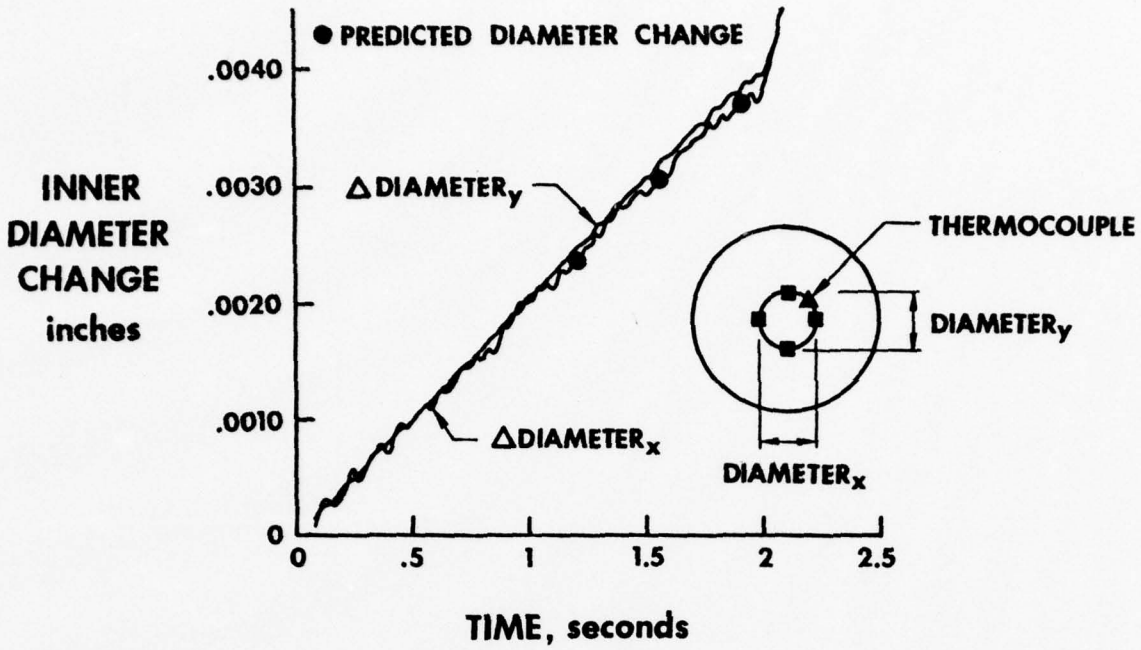

FIG. 3 DISK I.D. CHANGE VS TIME

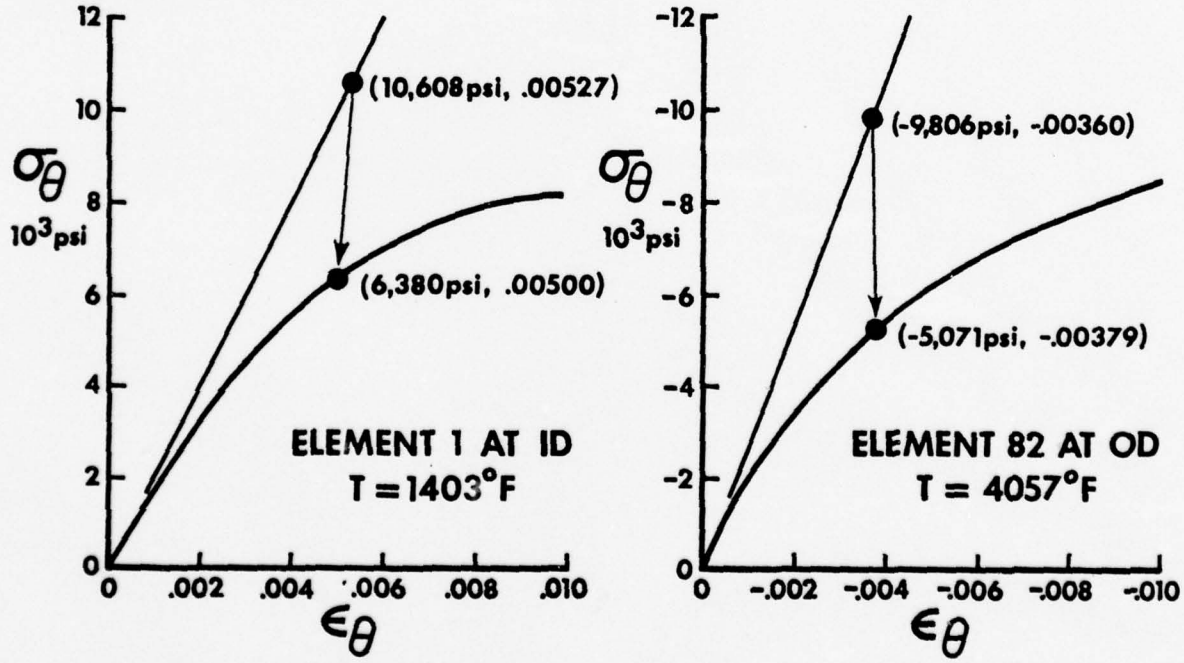

FIG. 4 DEGREE OF NONLINEAR σ-ε BEHAVIOR; COMPARISON OF I.D. AND O.D. ELASTIC AND NONLINEAR RESULTS

AEROTHERMAL ANALYSIS OF NAVAL SURFACE-TO-AIR MISSILE RADOMES

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Johns Hopkins University
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Assessing the suitability of a particular material for use on supersonic missiles requires knowing at least two basic items: The first item is the flight environment, namely altitudes and Mach numbers, and the second involves the thermomechanical properties of the material in question. The characteristics of importance to ceramic radomes include thermal shock resistance and limited distortion of the radar seeker beam. Also important, but less often a problem during flight are resistance to melting and structural strength sufficient for handling and high-g maneuvering. Determining these characteristics over the range of flight paths for a missile system depends on predicting the aerodynamic heating to the radome and calculating the resultant structural temperatures, boresight errors, and thermal stresses. The URLIM computer code (Ref. 1) performs these calculations and suitably edits the data for ready interpretation. The thermal stress calculations used by URLIM are performed assuming a thick-wall cylinder whose expansion and elastic modulus properties may vary with temperature (and hence thru the radial thickness) of the wall section (Refs. 2 & 3). This thermal stress prediction technique along with the aerothermal calculations have been verified by numerous experiments (Ref. 4) and are felt to be reasonable engineering tools.

Recent applications of the URLIM code have included a radome material screening for an improved version of the long range Standard Missile (SM-2). In this effort six materials (Pyroceram 9606 and 960X, fused silica, Rayceram, beryllium oxide, and reaction sintered silicon nitride) were evaluated over the "improved" trajectories and rated for their performance in thermal stress. The results showed that all of the materials would withstand the thermal shock and that the most readily available material (i.e. Pyroceram 9606) would be the radome of choice. In connection with this study a survey of available wind tunnel facilities capable of simulating the aerodynamic environment experienced by the improved SM-2 radome was made. The URLIM program was instrumental in providing the correlation between facility capability (Mach number, total pressure and total temperature) and flight induced thermal shock. In connection with another aspect of the Standard Missile program, the URLIM program has been used to simulate the detailed thermal response of a flight test radome. The thermal results from the URLIM program are being used as input to another program to evaluate the boresight error characteristics of the radome during flight. It is anticipated that these results will be correlated with telemetered data about the seeker's behavior. The overall result of this effort will be to quantify the effect of aerodynamic heating on boresight errors.

The accuracy of results of any radome material evaluation using URLIM are highly dependent on the thermomechanical characterization of the material. Specifically these properties include: thermal conductivity, specific heat, density, IR emissivity/absorptivity, elastic modulus, free linear thermal expansion, Poisson's ratio,

dielectric constant, and loss tangent. All of these quantities should be given as functions of temperature up to the maximum use temperature. Other crucial parameters that depend as much on the missile system specifications and radome geometry as on the intrinsic material properties are the maximum design stress for thermal shock and the maximum allowable boresight error slope. Only when these detailed material properties are available can the suitability of a material for radome use be confidently assessed.

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THE ROLE OF FRACTURE STATISTICS IN THERMAL SHOCK FAILURE OF CERAMICS

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ABSTRACT

Ceramics generally contain a nonuniform distribution of flaw sizes which causes variability or scatter in fracture strengths and a dependence of strength on specimen size and geometry. Several distribution functions have been suggested to quantitatively describe the consequences of nonuniform flaw sizes including the Weibull distribution,[1] extreme value distributions,[2,3] etc. Most distributions are based on the "weakest link" concept which states that the entire structure will fail catastrophically when the stress in any element of the body is sufficient to cause crack propagation of the largest flaw in that element. No single distribution function is universally acceptable for all brittle materials in describing the observed statistical effects of fracture.

Application of fracture statistics for components with thermally induced stresses is more involved than for most mechanically loaded components. As shown by Manson and Smith,[4] the highest probability of failure during a thermal transient does not occur at the time of maximum stress but at some time shortly after when the tensile stresses are more uniformly distributed over a larger volume of the component. Once a particular distribution function is chosen and the statistical parameters are determined for the material of interest, then fracture statistics are easily incorporated into finite element thermal and stress analyses to predict the maximum probability of failure during a given thermal transient.

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II (f) Analysis Problems Related to Fracture Toughness of Concrete and ATJ-S Graphite

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The general term, fracture toughness, has been defined as a measure of the resistance to crack estension. In these terms the fracture toughness of concrete and of ATJ-S graphite have similarities. In both materials crack extension is somewhat difficult to follow because fine scale cracking gives a diffuse character to the leading edge of the separation process. The crack tip zone of partially stress relieved material thus generated can, however, be treated for analysis purposes in a manner similar to that used for crack tip plastic zones in metals. The process of developing representations of fracture toughness using currently familiar methods of fracture mechanics involves oversimplifications. Nevertheless, the available results appear to be useful and extensions of these results would be of interest.

There has been limited interest in fracture toughness of concrete. Civil engineers assume this material to have zero tensile strength in practical applications. The writer is not familiar with applications of the statistical, worst-flaw viewpoint in studies of concrete tensile strength although such a viewpoint must have applicability. The discussion here is illustrated from work by Kaplan (at NBS) and Glucklich (at U. of Illinois) in the period 1958-1961. From these one can estimate a toughness values, $\mathcal{G}_c$ (0.11 lbs/in., 19 N/m) and a nominal crack-tip non-linear zone size, $2r_y$ (0.4 in., 10 mm).

With ATJ-S graphite, the statistical viewpoint has for some years furnished helpful estimates of tensile strength size effects. More recently fracture toughness studies by Starrett and Cull (at So. Research Inst.) permitted estimates of $\mathcal{G}_c$ and K_c at room temperature. Data reported by Hettche and Tucker (NRL) indicated a positive trend of fracture toughness with increase of test temperature. The reported toughness values correspond in semi-quantitative fashion with the influence of fine scale defects on the tensile strength.

With the ATJ-S graphite as well as with concrete, a more complete representation of fracture toughness could be obtained using the resistance-curve (R-curve) viewpoint which has been quite successful in applications to metallic materials. The R-curve method employs effective rather than physical crack size as a basis for resistance computations. Inferentially the justification for use of effective crack size depends upon concepts which retain a substantial degree of meaning inside of the crack tip zone of non-linear behavior. Currently these are the J and δ concepts. The applicability of the J and δ concepts to materials of the kind discussed here deserves additional study. Concepts of this nature would seem to be necessary as a means for assisting our understanding of the relationship between separational behaviors at fine scale and macroscopic fracture properties.

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Thermal-Mechanical Failure

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Two material design and selection criteria are currently in vogue for high-temperature structural components subjected to thermal stress.

The first criterion is based on the concept that the component is designed in such a manner that the maximum value of thermal stress does not exceed the strength of the material. The second criterion is based on the practical fact that the thermal environment usually encountered in high-temperature technology is so severe that failure as defined by the onset of crack propagation cannot be avoided. Under these conditions crack arrest governs the ability of the material to withstand failure by severe thermal stress. This latter design criterion is not commonly found in most engineering disciplines and may well be unique to engineering design involving severe thermal stress environments.

In this presentation the general results of a simple fracture-mechanical analysis of crack-propagation in thermal stress fields will be presented. This analysis was carried out by the present writer (1,2) in order to uncover the physical principals which underlie crack propagation under conditions of thermal stress and to establish the proper material selection and design rules, in order to minimize the extent of crack propagation.

This analysis basically follows the approach of Berry (3). In the absence of external body forces crack propagation under conditions of thermal stress (strain) mechanically is analogous to crack propagation under conditions of constant displacement. The mechanical model chosen for the analysis consists of a thin plate uniaxially constrained from thermal contraction when subjected to uniform cooling over a temperature difference ΔT. The plate contains N cracks per unit area. Crack interaction is assumed to be absent. The effect of the cracks is taken into account by means of an expression for the effective Young's modulus in terms of the crack size and crack density. The conditions for crack instability in terms of the critical temperature difference of cooling of the plate, ΔT_c, can be obtained in a manner identical to the derivation of the Griffith criterion. The reader is referred to the original papers for the mathematical details.

Fig. 1 shows a schematic for the crack propagation behavior. The following general conclusions can be drawn:

1. ΔT_c for $\ell_o << \ell_m$ is proportional to $\ell^{-\frac{1}{2}}$.

2. ΔT_c for $\ell_o >> \ell_m$ is proportional to $\ell^{3/2}$. This conclusion reflects the effect of the cracks on the compliance of the plate at the higher values of crack length.

3. For $\ell_o << \ell_m$ and $\Delta T \geq \Delta T_c$ crack propagation occurs in an unstable manner. The length of the crack which results is inversely proportional to the crack length prior to propagation. This also implies that the strength retained after crack propagation and arrest is an inverse function of the original strength.

4. For $\ell_o >> \ell_m$ crack propagation occurs in a stable manner. For a given final value of crack length, ΔT_c for stable crack propagation exceeds ΔT_c for unstable crack propagation.

5. For $\ell_o > \ell_m$, ΔT_c is a function of the number of cracks participating in the fracture process.

6. A material can fail in an unstable or stable mode, depending on the number of cracks participating in the fracture process.

7. Materials with high densities of micro-cracks should exhibit excellent thermal stress resistance.

Fig. 2 compares the anticipated crack propagation and relative crack propagation and relative strength behavior of a brittle material as a function of severity of thermal stress as measured by a temperature difference.

The above analytical results are illustrated by literature data (4,5,6) and discussed in terms of the criteria for material selection for thermal environments involving severe thermal stress. Future work will consist of computer simulation of dynamic crack propagation in thermal stress fields.

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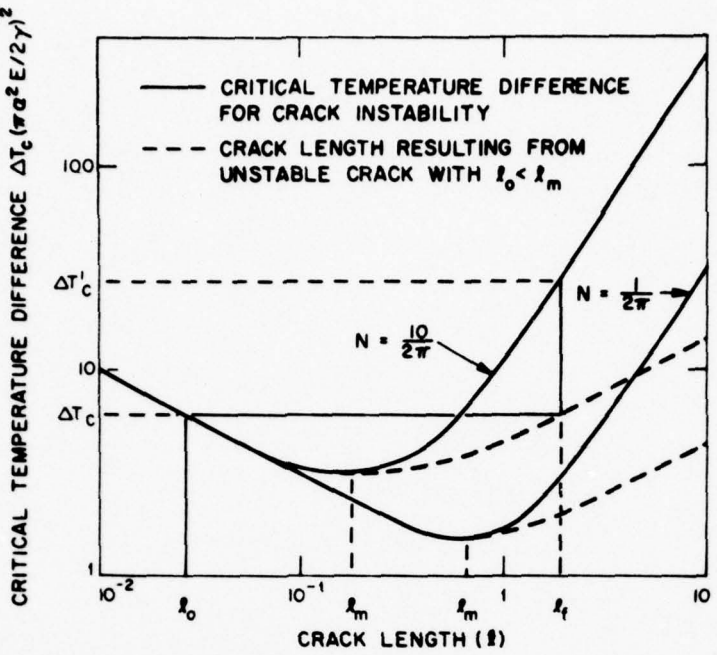

Fig. 1. Crack stability and propagation in axially constrained plate cooled by temperature difference ΔT.

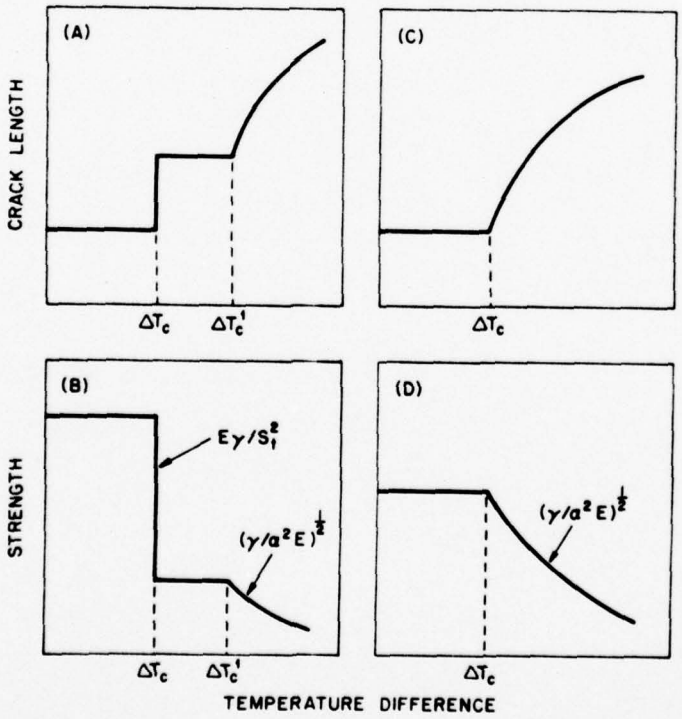

Fig. 2. Dependence of crack size and relative strength on temperature difference.

THERMAL STRESS/SHOCK RESISTANCE OF CERAMICS: MATERIALS FACTORS

G.C. Walther (IITRI)
S.W. Freiman, P.F. Becher, and R.W. Rice (NRL)

Experimental thermal shock results from water-batch quenches were compared to calculated thermal stress resistance parameters for several aluminas, silicon nitrides, and silicon carbides. The degree of damage was monitored by internal friction and/or residual strength measurements (both 3 and 4 point flexural). Agreement between experimental and calculated results for resistance to thermal fracture initiation (R) was good for thicker samples. For thinner samples ($\leq$3mm) higher ΔT_c values ($\geq 1200^{o}C$) were obtained, suggesting that the water quenches were not as severe as expected. Examination of possible quench severity factors ($\beta = r_m h/k$) further supported this. Boiling water films may also have decreased quench severity with increasing quench temperature difference. Results of R^{1} and R^{11} parameters for milder quench conditions showed satisfactory qualitative ranking of the various materials.

Comparison of strength changes with thermal shock damage resistance parameters also agreed qualitatively when actual strength loss was used, while results in terms of percentage strength change were less satisfactory. This implies the nature and influence of flaw structure on strength before and after shocking needs further attention.

Although all the properties needed to determine the appropriate resistance parameters can be determined as a function of temperature, the choice of which particular values to use is still not well established. Variability in these measured properties from sample to sample is fairly small, except for strength and fracture toughness determinations. These two properties are weak links in attempts to quantitatively compare

experimental and calculated results because of this variability.

The influence of sample flaw structure, stress distribution during testing , and test environment on measurements of strength, fracture initiation, and crack propagation and arrest should be more fully studied to provide better materials property information for thermal stress resistance analysis.

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MICROSTRUCTURAL ASPECTS CONTROLLING THE THERMAL STRESS RESISTANCE OF GRAPHITE

C. R. Kennedy

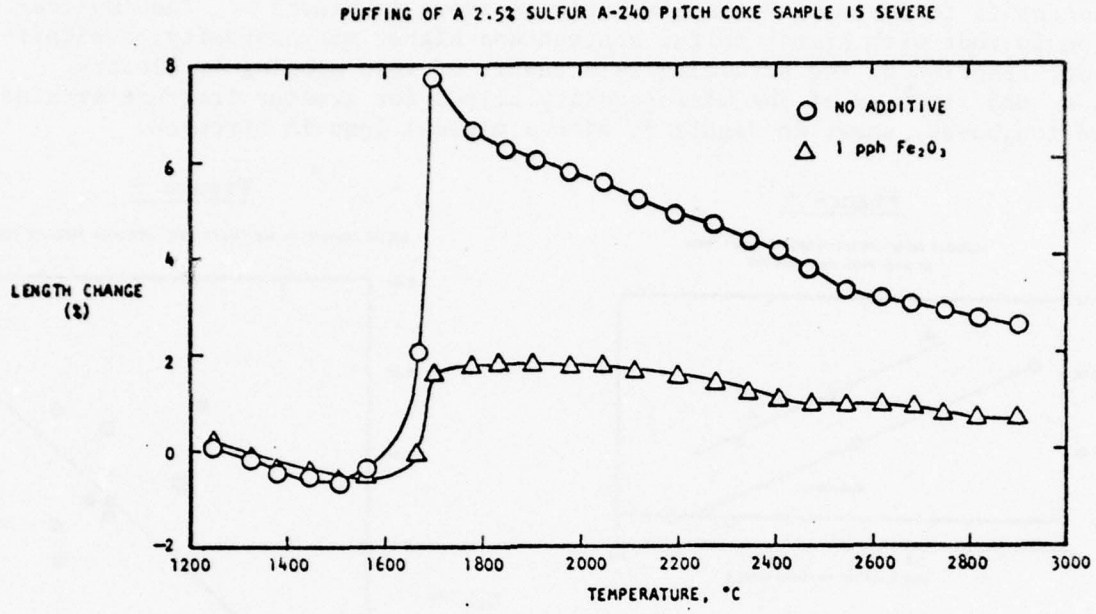
The thermal stress resistance of graphite can be improved significantly by structural control during fabrication. The gain is accomplished by the reduction of the critical defect size, increased resistance to crack propagation, and by the introduction of a fine, homogeneously distributed microporosity within the structure. At the Oak Ridge National Laboratory (ORNL) we have demonstrated several procedures to accomplish these goals culminating in the formulation of a new thermal-stress-resistant graphite, GRAPHNOL N3M.

Graphite is a porous material with two distinctive defect distributions to consider in the continuum mechanical approach to fracture. The first is the large pore associated with the particle boundaries that form the critical defect eventually leading to catastrophic failure. The second is the microporosity existing on a very fine scale homogenously distributed within the particulate and crystallites. The generally accepted model is that the pores initiate a crack that propagates from pore to pore normal to the stress axis. This may be along optical domain boundaries or along the boundaries of misaligned particles. Microcracking begins well below the failure stress and increases in number with increasing applied stress. The magnitude and ease of microcracking is determined by the microporosity pre-existing within the crystallites. The ability of graphite to achieve large strains to failure is limited by the ability of the structure to accommodate homogeneous microcracking without resulting in rapid crack propagation to failure. The microcracking is actually a stress-relief system which distinguishes graphite from classically brittle ceramic materials.

Graphites made by conventional methods generally do not have efficient bindering and the crack path is preferentially along the particle-particle boundary. An ORNL process, developed initially for neutron irradiation resistance, utilizes a green filler coke with a plasticized surface to obtain a very efficient bindering system. The resulting contiguous structure eliminates the boundary as an easy path for crack propagation forcing the crack to travel more tortuously within the particles. The size of the pore or the critical defect is minimized by reducing the particle size to the point of where the fines begin to agglomerate. The pore size is then proportional to the agglomerate size rather than particle size. In our case, we found that a mean particle size of 35 μm was a practical lower limit.

One method of regulating the microporosity is through the use of sulfur to cause an irreversible expansion during the final heat treatment to graphitization, commonly called "puffing". The puffing reaction is a result of a sudden release of sulfur creating sufficient internal strains to cause a significant increase in the micropore density. An example of the puffing reaction is given in Figure 1. This increase in volume due to the microporosity results in lower final bulk densities and alterations in both the thermal and mechanical properties of graphite. It is primarily through the regulation of the puffing reaction that the desired structural modifications

Figure 1



for improved thermal-stress resistance can be made. While the effect of sulfur puffing is to increase the total porosity, the large pores defining the critical defect are neither increased in size or number. As expected, the bulk compressibility is markedly increased by the increased total porosity (shown in Figure 2) but the strength of the higher sulfur lower density graphites is slightly reduced. However, the fracture strain will be increased as seen in Figure 3. Another effect of increased sulfur

Figure 2

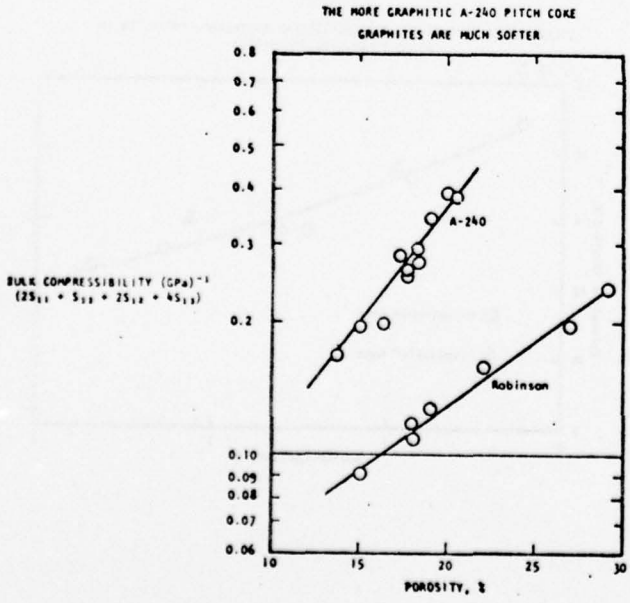
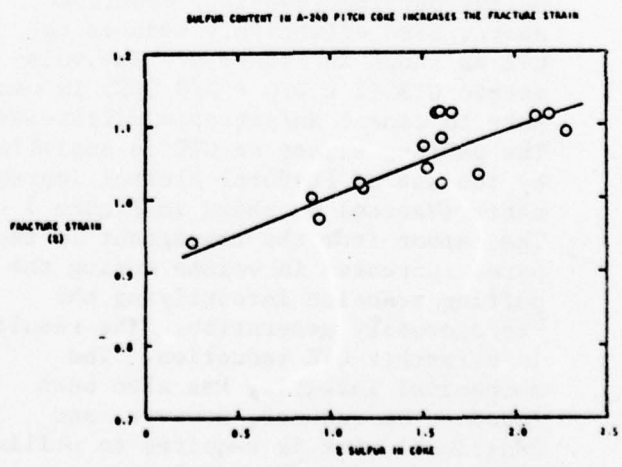


Figure 3



content is to decrease Poisson's Ratio as shown in Figure 4. The implication is that with higher sulfur content and higher microporosity, a significant fraction of the straining is a result of void opening or closure. Thus, the fineness of the microporosity allows for greater fracture strains and toughness, shown in Figure 5, with a minimal loss in strength.

Figure 4

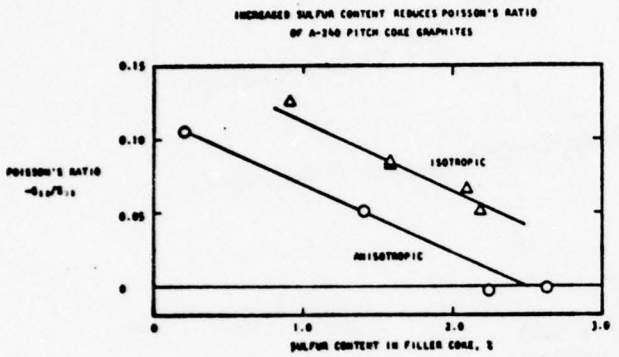
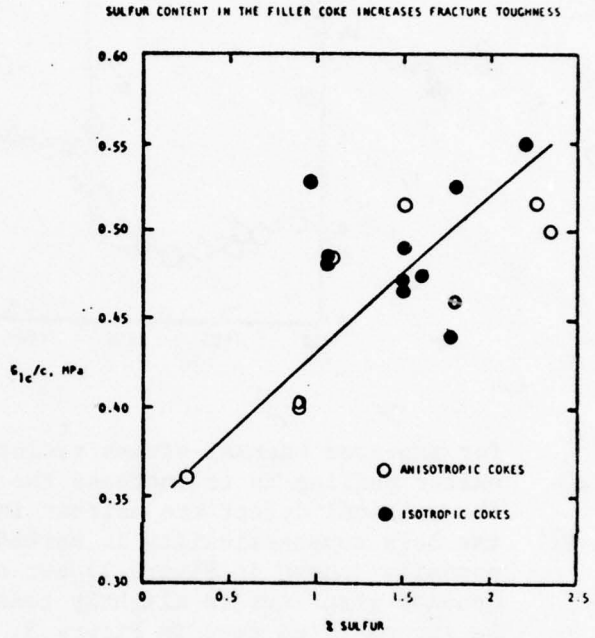


Figure 5



The coefficient of thermal expansion (CTE) of a graphite is almost entirely a function of the defect structure normal to the c-axis of the individual crystallites within the filler particle. The use of green filler cokes precludes the normal development of structural defects by calcination heat treatments and alternate procedures had to be developed. The microporosity generated by the sulfur puffing reaction, described above, also effectively reduces the CTE as shown in Figure 6. The volumetric CTE (2 x W/G + A/G CTE) is used here to cancel anisotropic differences. The puffing effect on CTE is amplified by the use of Furfural alcohol impregnants (Varcum) as shown in Figure 7. The carbon from the impregnant in the pores increases in volume during the puffing reaction intensifying the microporosity generation. The result is a further CTE reduction. The mechanical integrity has also been found to be reduced, however, and additional work is required to utilize this procedure effectively.

Figure 6

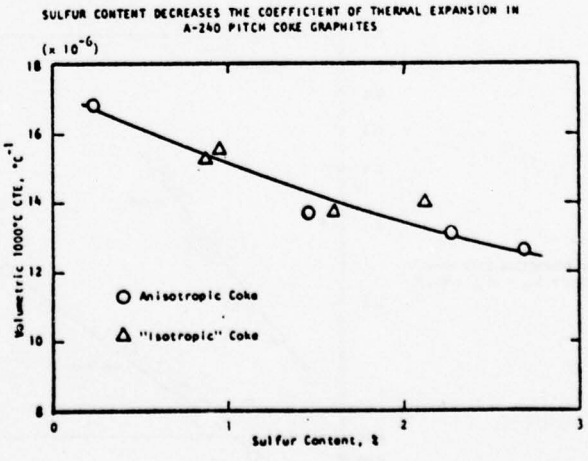


Figure 7

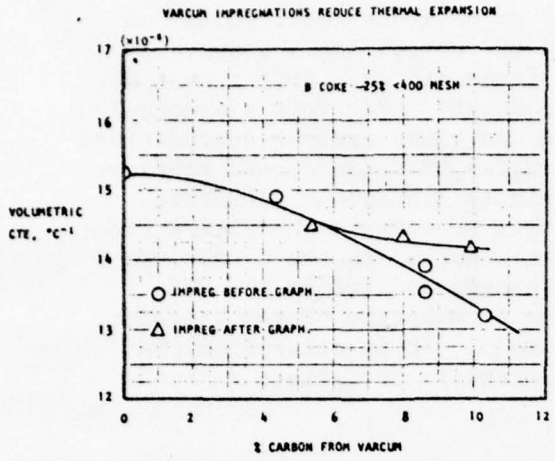
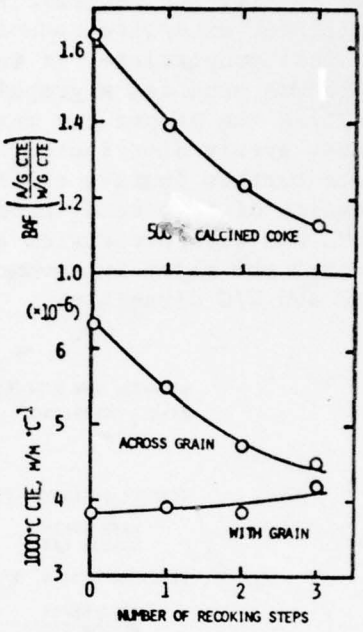


Figure 8

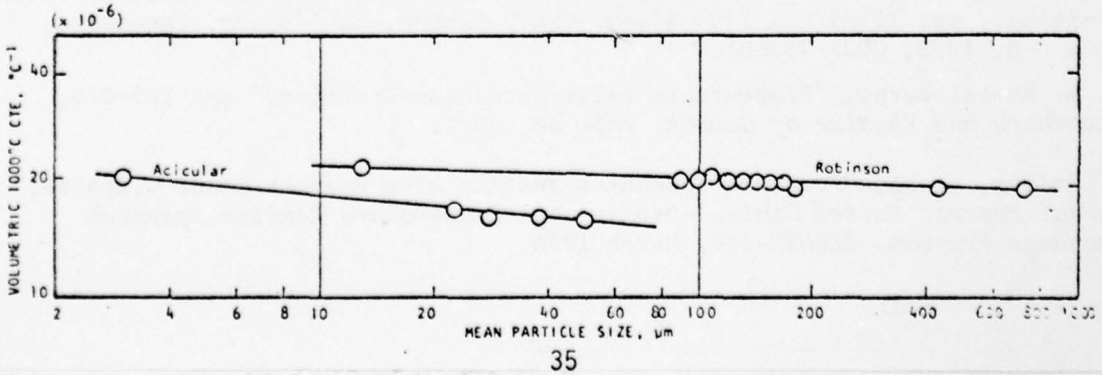
RECOKING BOTH REDUCES THE COEFFICIENT OF THERMAL EXPANSION AND INCREASES ISOTROPY.



Additional procedures were also developed to both randomize the structure as well as introduce the desired defect structure. We have found that prolonged heat treatments at 500°C are effective in reducing the CTE, as shown in Figure 8, while retaining the necessary chemical reactivity for efficient bindering. Each recoking step represents an additional 24 hours at 500°C heat treatment for a mixture of filler coke and pitch. Not only does this process reduce the CTE but significantly improves the isotropy of the graphites (BAF → 1). While the mechanism for defect formation and CTE reduction is not completely clear, it is certainly associated with the condensation and evolution of gases in the dehydrogenation process. The uncertainty is in whether the internal strains necessary for the defect formations are generated by the shrinkage of the lattice by hydrogen evolution or by the pressure generated by entrapped methane within the lattice. It is likely that both processes are active to generate the necessary microporosity. One additional note on controlling the CTE by a fabrication process--reducing the particle size will also reduce the defect concentration within the particle and increase the CTE. This is a result of reducing the particle size by breaking the particle through existing defects. The increase in CTE is shown in Figure 9.

Figure 9

THE HIGHER CTE AND LOWER SLOPE REFLECTS FILLER MATERIALS WITH FEWER DEFECTS



In general, it can be concluded that significant improvements in thermal stress resistance can be achieved by controlling the overall defect morphology. It is clear that the large critical defect must be reduced in size and neutralized by microporosity. Particle size should be small, but excessive reduction in size can cause serious degradations in physical properties. It was shown that the ORNL green coke fabrication process produces a graphite with a highly contiguous structure. Integrating the proper raw materials to this process to generate a fine porosity, evenly distributed, and orientated normal to the c-axis was found to further improve the fracture toughness and reduce the CTE. The results of this study have yielded the formulation of the graphite GRAPHNOL N3M with properties given in Table 1. It is clear from the table that the major improvement is in the strain to failure in both the A/G and W/G directions.

Table 1

GRAPHNOL N3M OFFERS A SIGNIFICANT IMPROVEMENT IN THERMAL STRESS RESISTANCE:

	ATS-5	GRADE 994	N3M
TENSILE STRENGTH, PSI			
WITH GRAIN	5150	5900	6400
ACROSS GRAIN	4250	4190	5680
FRACTURE STRAIN, %			
WITH GRAIN	0.44	0.55	1.07
ACROSS GRAIN	0.55	0.71	1.04
1000°C CTE, $^{\circ}C^{-1} \times 10^{6}$			
WITH GRAIN	3.5	3.4	4.0
ACROSS GRAIN	4.9	4.8	4.9
SORI THERMAL STRESS RESISTANCE, KW			
WITH-WITH GRAIN	18	29	>35
WITH-ACROSS GRAIN	11	21	30

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CYCLIC FLAME TESTING OF STRUCTURAL CERAMICS

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A prerequisite to the use of ceramics in structural applications at high temperature is the determination of thermal fatigue sensitivity. Whereas tests such as a water quench may simulate unusually severe thermal shock leading to immediate failure, repeated shocking in a less severe environment may cause iterative damage leading to failure.

In an effort to highlight potential thermal fatigue problems of ceramics for gas turbines, a thermal fatigue apparatus similar to that described by Johnson and Hartsock (Ref. 1) was constructed at AMMRC. (See fig. 1) Ceramic bend bar specimens are heated rapidly to 2500°F in a flame station and then indexed to a quench station where a compressed air jet cooled them to room temperature. The flame was generated by a heating torch and was directed at the midspan of the sample creating a zone of relatively uniform temperature (Fig 2.) The quench jet was directed at the hottest portion of the sample when the sample arrived in the quench station. Details of the construction and the operation of the AMMRC rig can be found in reference 2.

Heatup and cooldown rates were set comparable to small turbine components (fig 3.). The bend bar was chosen arbitrarily for its simplicity of machining and testing (flexural strength and resonance measurements). Although larger wedge shaped specimens would undoubtedly develop higher stresses, the difficulty of post exposure analysis made the bend bars more attractive.

For a variety of the newer structural ceramics, sixteen, twelve and four bend bars were exposed to 0, 1000, and 10,000 thermal cycles respectively. In addition a group of samples were flame heated only (no quench) for an exposure equivalent to that which the 1000 shock group experienced. This group was not cycled, but slowly heated in a flame station to give a cumulative time-temperature exposure of 1000 times a single cycle. The intent was to distinguish any possible thermal fatigue damage from flame exposure effects. Bend bar size was .080 x .125 x 2." with machining parallel to the long axis and edges chambered.

For most materials specimens survived the test proceedures intact, but showed marked surface attack. A gradient in damage usually was evident which corresponded to the temperature gradients which existed during heating (Fig 2.). This results in gradients in oxidation-flame exposure and also thermal shock severity. All specimens were tested in flexural resonance for determination of the elastic modulus and internal friction. These results showed little variation with test exposure when compared

with the control group. The resonance techniques measure properties averaged over the entire specimen volume and the data does not reflect a change in the specimen bulk.

The flexural strength test on the other hand is surface sensitive. Nearly all materials exhibited strength degradation after thermal cycling. Materials tested included: Norton NC-350 reaction sintered silicon nitride, Norton NC-132 hot pressed silicon nitride, Norton hot pressed silicon nitride/yttria (formerly known as NC-136), Norton hot pressed silicon carbide NC-203, Norton NC-435 recrystallized silicon carbide, Coors AD-99 alumina and a variety of experimental grade materials. The alumina was chosen intentionally for its poor thermal shock resistance, and samples shattered after only a few cycles. After 1000 shocks a 20% degradation of flexural strength was experienced by the NC-132 samples, 38% after 10,000 cycles. The flame exposure samples showed a strength loss nearly identical to the 1000 cycle group, indicating the damage was probably due to flame exposure or oxidation rather than due to thermal shocking. For some of the other materials, a similar comparison suggests the thermal shocks did contribute to degradation. Further analysis and metallographic inspection are being performed. However, it seems that the thermal cycling damages protective glass oxidation layers rather than the matrix itself.

An attempt was made to analyze the thermal stressing that resulted from the experimental exposures. It was apparent that due to the transient conditions with significant conduction convection and radiation heat transfer as well as variable material properties a computer-numerical analysis was necessary. A finite difference heat transfer computer program was developed and coupled with a plain strain numerical analysis. At present only a two dimensional analysis has been performed, but the results have been illuminating. The maximum tensile stress developed on heatup of NC-132 samples is estimated to be 1000 psi. The maximum stress on cooldown, is 5,400 psi. Different stresses will develop for the various other materials as a result of their properties, but it has become evident the stresses are usually quite low compared to the strength of the materials tested (There are exceptions). It is not surprising therefore that little thermal fatigue damage has been detected. The same computer technique is now being used to explore alternate specimen geometries that may develop more meaningful thermal stress.

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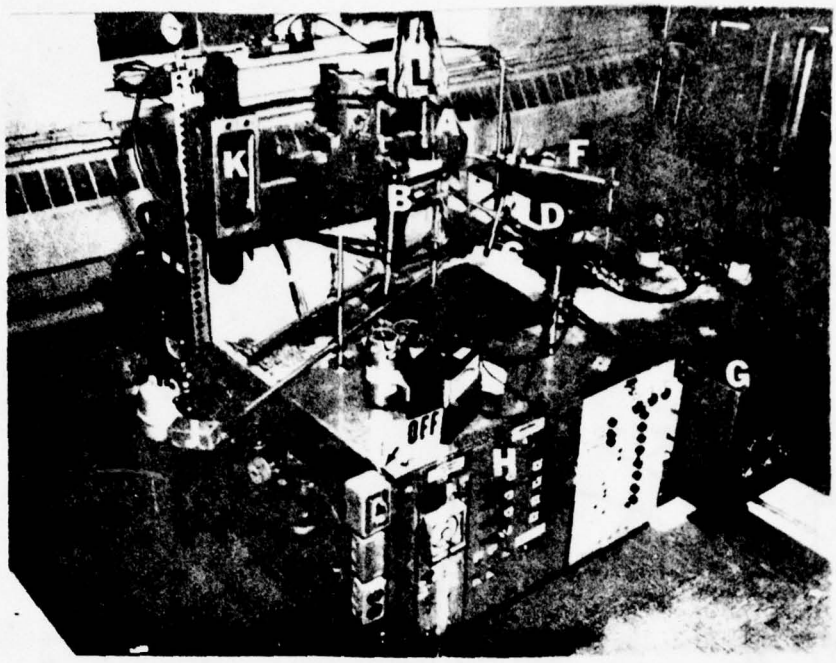

Figure 1. Thermal Fatigue Apparatus
A. Heating station. Four samples are on turntable. B. Quench station, C. Heating Torch, D. Microoptical pyrometer, E. Automatic pyrometer, F. Falme sensor, G. Safety control, H. Specimen failure monitor, I. Turntable rotation control, J. Quench line, K. Flowmeter, L. Exhaust duct for hot gases.

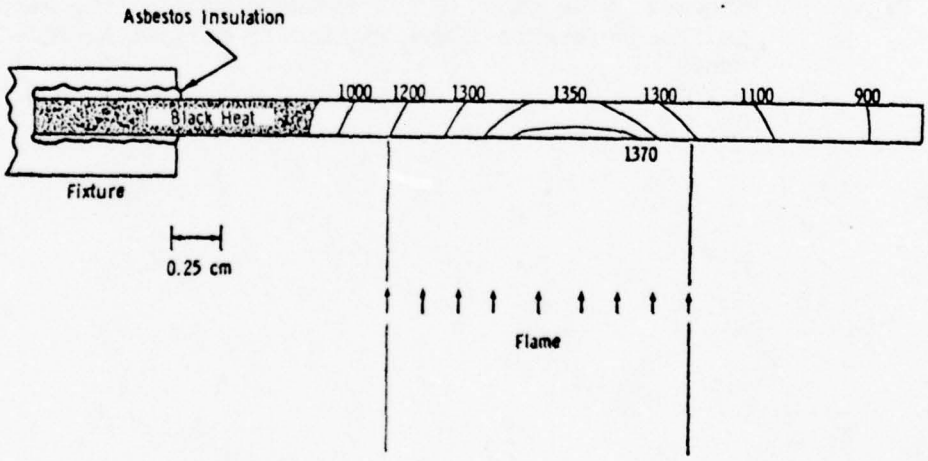

Figure 2. Steady state temperature distribution on NC-132 bend bar specimen. Side view. Isotherms are in degrees centigrade.

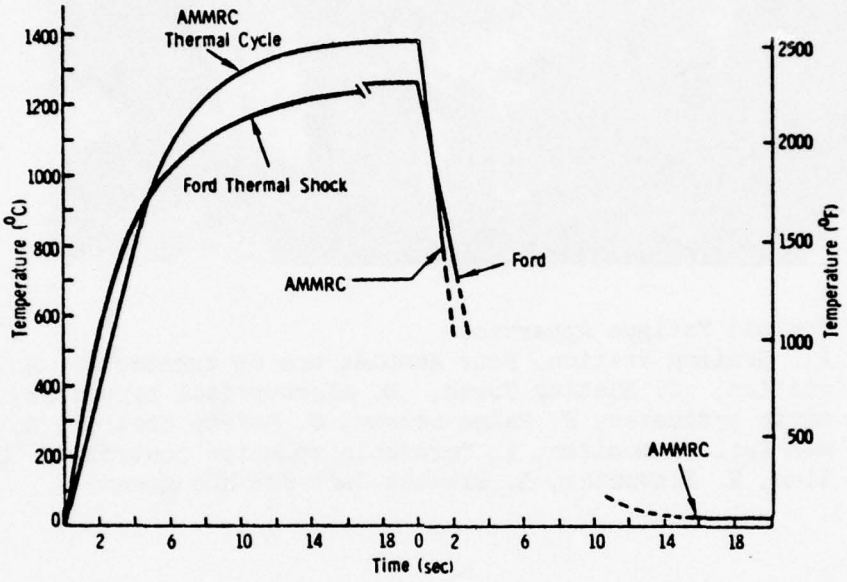

Figure 3. Thermal cycle on NC-132 bend bar samples. The Ford cycle is from reference 1 and applies to silicon nitride stator vanes.

A SUMMARY OF THE COMPARISON OF CW LASER IRRADIATION OF CERAMICS WITH QUENCH TESTS

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An analysis of thermal stress theory shows that the approaches to analyzing continuous wave (cw) laser irradiation and thermal quench data are basically the same. High intensity ($\tilde{>}$ 1 kw/cm^2) cw laser irradiation involves a large heat transfer coefficient, $h \tilde{>} 10 \frac{cal}{sec\,^oC\ cm^2}$, whereas the water quench test depends on the exact condition of heat transfer and varies from h = 0.1 to 1.0 $\frac{cal}{sec\,^oC\ cm^2}$. However, for most materials this difference is not significant.

Ranking of materials for resistance to quench (down) thermal stress fracture is generally made on the basis of the maximum temperature difference, ΔT_c, that can be tolerated such that the original room temperature strength, σ_i, is maintained. An important consideration to these tests is the residual strength, σ_r, that is left after the critical ΔT is reached. Depending on the particular application, ΔT_c and/or $\sigma_i - \sigma_r$ may be important. Rapid heating (quench up) is analytically similar to a quench down, except that the location of the maximum stress is on the inside of the sample instead of the exterior as in the quench down, and the magnitude of the stress is not as severe for the same ΔT. Ranking of materials for resistance to thermal stress failure from laser irradiation can be made on the basis of time to failure (fracture or burnthrough). The time to reach .95 of the maximum stress in a thermally quenched

sample is equivalent to the time to fracture under high intensity irradiation. The difference between the two cases is that the maximum stress reached in the quench test will not always result in a degradation of strength, or fracture because the maximum ΔT_c has not been reached, whereas in the cw laser irradiation case, for the materials that fracture, we irradiate until the fracture stress is reached, provided burnthrough does not occur first.

A comparison between ranked resistance to water quench and cw laser irradiation is given in Table I. The conditions for the quench (down) and laser irradiation experiments were selected such that Biot's modulus was approximately equivalent for the two cases. Notice that the rankings are equivalent if one compares the time to fracture to the most commonly used criterion, ΔT_c, but are not necessarily equivalent if $\frac{\sigma_r}{\sigma_i}$ is used as the criterion for the quench. In general, laser irradiation may be used to rank materials, and these rankings can be compared to other heating environments provided that the geometry (i.e. primarily thickness), wavelength (i.e. opaque or transparent) and heating rates are equivalent in the two cases, e.g. by comparison of Biot's modulus.

THERMAL SHOCK OF CERAMICS

Material	Laser Induced $t_{f(sec)}$	$\frac{\sigma_r}{\sigma_i}\left(\frac{ksi}{ksi}\right)$	(Water) Quench ΔT_c (^{o}C)
50BN-50 Al_2O_3	20*	12/12	1200
70BN-30 Al_2O_3	15*	6/25	950
SCFS	8*	4/8	850
Pyroceram 9606	3	5/35	600
H P Si_3N_4	1.5	60/140	600
R.S. Si_3N_4	1	17/30	350
Alumina	0.3	15/45	225
MgF_2	0.1	12/26	150

*Burnthrough time

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ABSTRACT

SOUTHERN RESEARCH TEMPERATURE/STRESS TEST

The thermal stress test developed at Southern Research has been used to rank relative thermostructural performance of materials under development, to measure strains to failure in the development of design criteria, and to calibrate and correlate analytical techniques used to model thermostructural loadings. Materials which have been evaluated to date in the test are polygraphites, carbon-carbon composites, graphite-carbide composites and refractory metals such as tungsten.

The test utilizes a disc-shaped specimen with a central hole. The specimen is heated on the OD by induction in the frequency range of 200-300 kHz. Figure 1 is a schematic of the test set-up. For the materials tested to date, this frequency range concentrates the induced currents near the OD of the specimen. The result of this current concentration is that the specimen is heated in the outer 0.1 in. simulating a surface heat flux. The typical time of a test is on the order of 2 to 5 seconds; thus, the ID remains relatively cool. Temperature gradients in the range of 3000°F from OD to ID are common for polygraphites and carbon-carbon composites. Some ID and OD temperature time curves for a 1.75 in. OD specimen heated at several power levels is shown in Figure 2.

Instrumentation for the test includes an optical temperature measurement on the OD, a temperature measurement using a thermocouple at the ID, and independent tracking of two axes of the diameter of the central hole. The schematic in Figure 1 shows the layout of the rapid tracking strain analyzer used to measure the changes in the internal diameter which occur during a test. A typical record of diametral change versus time for a graphite specimen is shown in Figure 3. This record, plus the record of the temperature measured at the ID permits the calculation of a strain time plot from which the strain at failure may be taken. For graphites, this strain has been

correlated with strains measured in gas-bearing tensile tests, and predicted in ground tests on full scale hardware. This has led to confidence in analytical prediction procedures, and the development of a strain based design criteria.

Notched specimens have also been used in the T/S test. The data from these tests were compared with the results from a K-calibration of the notched specimens. Qualitative correlation between the test results and the analytical prediction gave agreement within 15-20 percent. Qualitatively, the analytical procedure predicted ranges of notch sizes for which rapid and slow crack propagation would occur. The experimental results verified these predictions within measurement accuracy.

Although most of the work to date has been on electrically conductive materials, there has been some limited test on non-conductive materials. For these tests, a graphite susceptor is heated inductively, and heat is transferred to the specimen by radiation. Heat flux densities on the order of 0.5-1.0 BTU/sec-in^2 can be obtained by this method depending on the emissivity of the specimen material. Figure 4 shows a schematic of this test set-up.

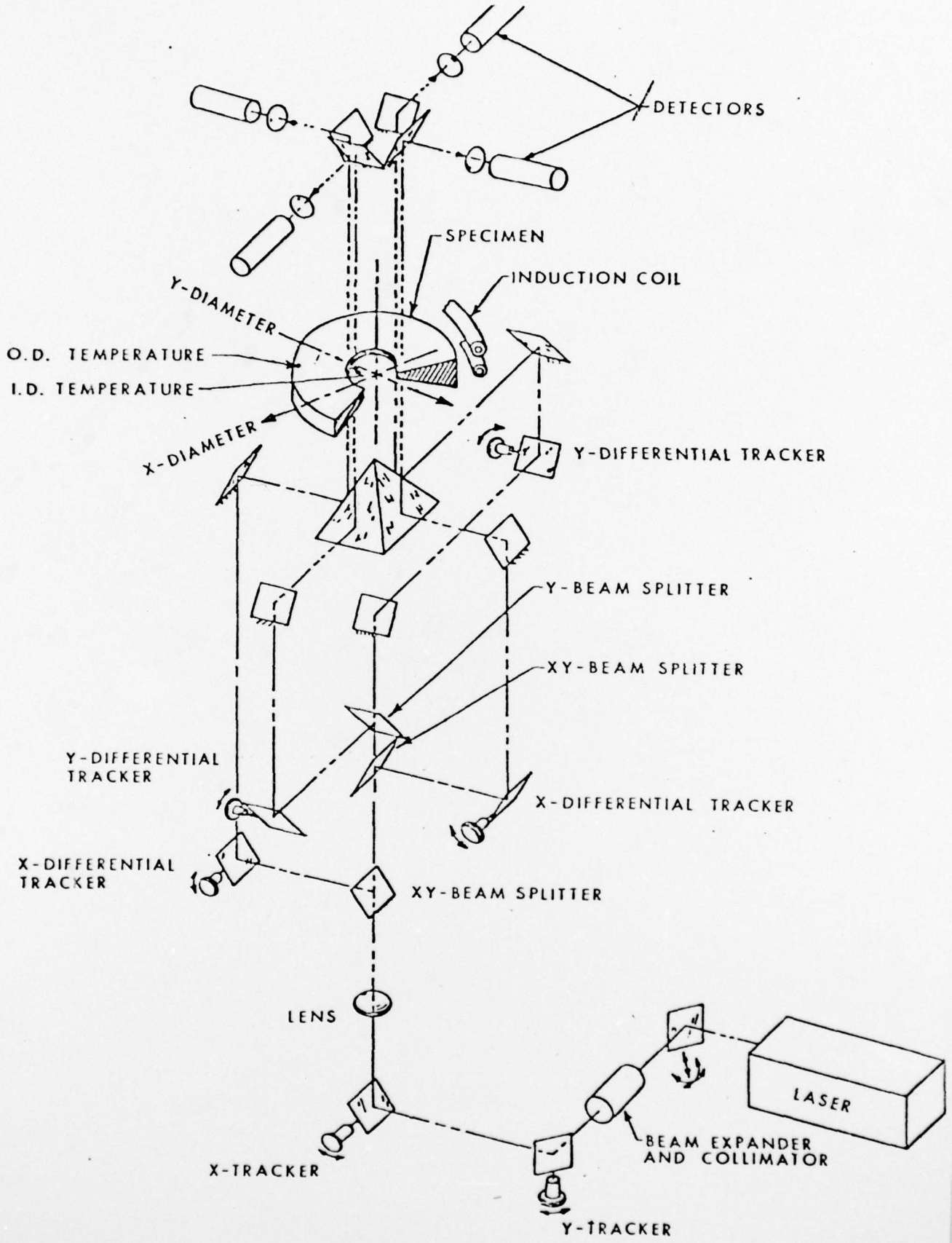

Figure 1. Schematic of Southern Research Temperature/Stress Test with Rapid Strain Analyzer

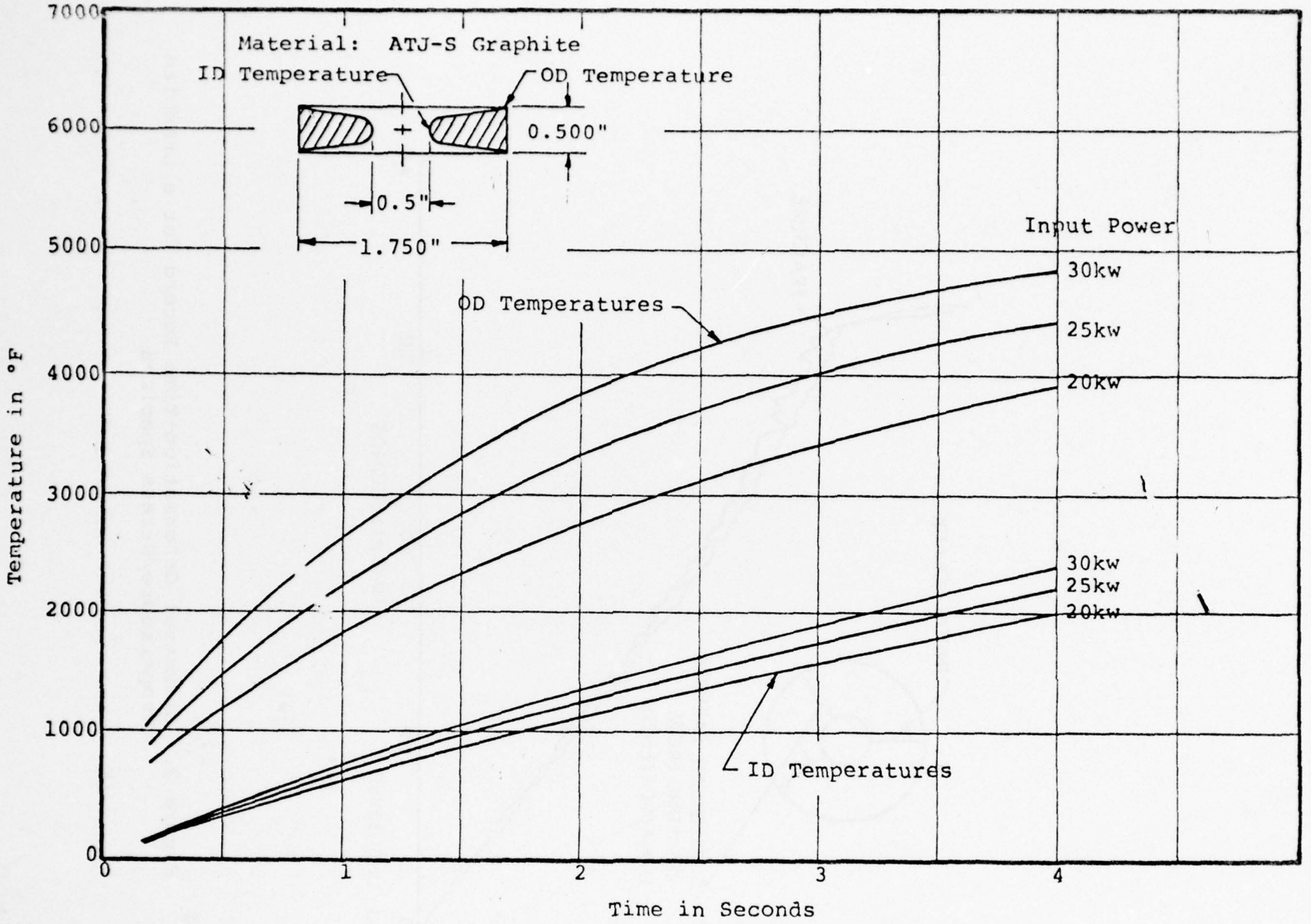

Figure 2. OD and ID Temperature-Time Curves for a 1.75 Dia ATJ-S Graphite Temperature/ Stress Specimen at Several Input Power Levels

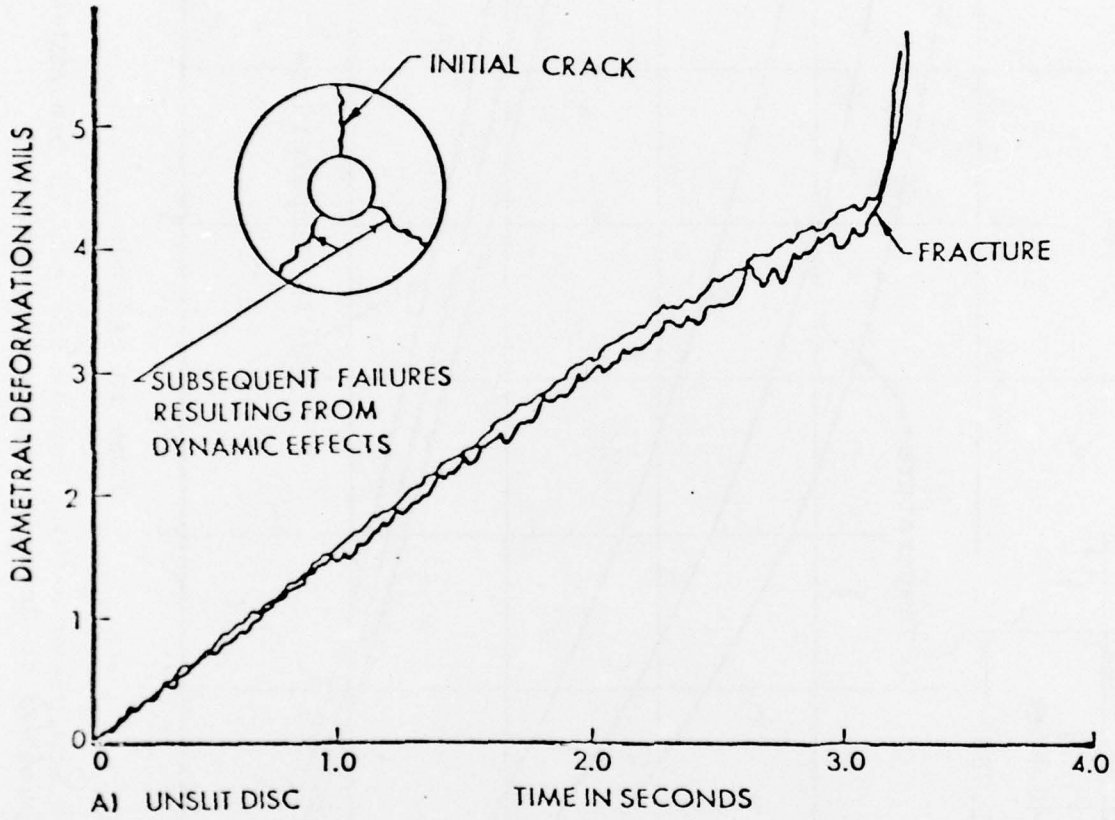

Figure 3. Diametral Deformation-Time Record for a Graphite Temperature/Stress Specimen

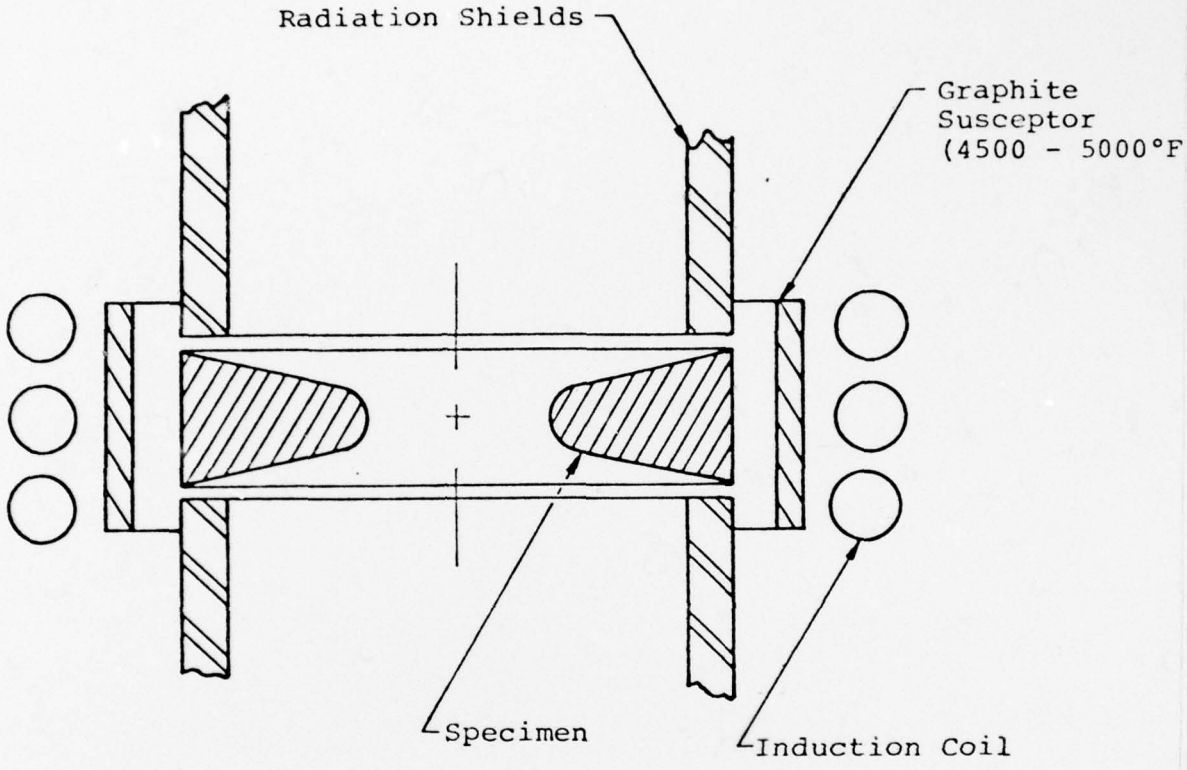

Figure 4. Schematic of a Temperature/Stress Setup for Nonconducting Materials

AEROTHERMAL TESTING AT THE NWC T-RANGE HOT GAS FACILITY

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A key step in the development and evaluation of high speed missile components such as seeker domes is aerothermal testing. To obtain meaningful results, such testing must realistically simulate the total temperatures, heating rates, and aerodynamic pressures occurring in flight. The Naval Weapons Center T-Range Air Augmentation Facility is capable of providing hot gases for free jet or connected pipe testing. Originally developed for testing of ramjet combustion chambers, T-Range has recently been used for supersonic free jet testing of missile radomes and IR domes (References 1 and 2).

Flow parameters that can be controlled are total temperature, total pressure, flow rate, flow velocity, Mach number, density, and, to some extent, chemical composition. The latter becomes particularly important when testing materials which undergo thermochemical decomposition. These parameters may be held constant or varied throughout the test to simulate a particular flight trajectory.

The temperature capability of the facility is provided by a SUdden Expansion (SUE) burner. This is an axisymmetric, water-cooled, propane-fueled combustor and mixing chamber. Compressed air from a high pressure storage system is delivered into the SUE burner. Propane is injected at the rate required for the desired temperature and oxidized in the air stream. Make up oxygen is introduced downstream of the burner to replace that consumed during combustion. The hot gases are then expanded through a nozzle to the atmosphere, thus, producing a supersonic free jet in which the test specimens are immersed.

Test capabilities include temperatures from ambient to 5000°R and mass flow rates from 2 lb/sec to 200 lb/sec. Durations range from several seconds at high flow rates to approximately two hours at the lower flow rates. The exit Mach number of the free jet is determined by the nozzle design.

Specific components which have been tested at T-Range include quartz/polyimide ogive radomes and magnesium fluoride hemispherical IR domes. Prior to testing of the actual seeker domes pressure and temperature rakes are used to map the flow and instrumented steel calorimeter domes are tested to determine surface heating conditions.

The purposes of the quartz/polyimide radome tests have been to evaluate the performance of the design under severe aeroheating conditions and to determine thermostructural limits. Test conditions are selected to simulate aerodynamic heating at speeds from Mach 2.5 to over Mach 4. Gas total temperatures have typically ranged from 800°F to over 1500°F.

Several magnesium fluoride IR domes have been tested to destruction in T-Range in order to provide data for evaluation of statistical failure theories. Temperatures measured on the inner wall of the domes have agreed well with those predicted for the flight trajectory being simulated.

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NASA BURNER RIG TESTS

J. R. Johnston

ABSTRACT

High velocity burner facilities have been used extensively over the past twelve years at the Lewis Research Center. The majority of tests have involved the oxidation and salt corrosion of superalloys and coatings. Other more specific applications have been as follows:

a. evaluation of thermal-barrier coatings for cooled blades (ref. 1).
b. thermal fatigue of simulated airfoils for life prediction studies (ref. 2).
c. effect of fuel/air ratio on salt corrosion.
d. correlation with furnace oxidation tests of superalloys (ref. 3).
e. effect of alternate fuels such as shale-oil on corrosion.
f. evaluation of ceramic materials for oxidation and thermal fatigue resistance (refs. 4, 5, 6).

Two basic types of burners are used at LeRC: three large Mach 1 burners designed at Lewis Research Center and ten smaller Mach 0.3 burners from a modified P&W design. The Mach 1 burners have usually been used for oxidation and thermal fatigue tests while the Mach 0.3 units typically have been used for hot-salt corrosion studies. Both burners can heat rotating groups of specimens to 2200°F and stationary

specimens to 2500°F without extensive heat shields or special baffles.

Several programs have been conducted to evaluate the oxidation and thermal fatigue resistance of ceramic materials using the Mach 1 burner. The most recent investigation is a cooperative program between AFML, IITRI, and NASA. In this program, specimens fabricated from NC132 ($HPSi_3N_4$), NC435 (siliconized SiC), NC350 (RSSN), and KBI RSSN were exposed to the burner exhaust at temperatures from 2200° to 2500°F and cooled in still air. The specimens were cycled 4 times per hour between the test temperature and room temperature until failure occurred or a total of 250 cycles was reached. In resistance to thermal fatigue, the materials were ranked roughly in order of strength. The strongest material (NC132) had no failures while for the weakest material (KBI RSSN) only one out of nine specimens survived the 250 cycle test.

The ability of the burner tests to separate materials as noted indicates that the burner subjects the specimens to reasonable stress levels although use of forced air cooling may change this conclusion. In future tests it is planned to measure transient temperature, compute thermal stress in the test specimen, and correlate failures with computed stress levels for various temperatures and cycles. These tests should permit optimization of the specimen shape and assure more meaningful comparison of rig test results with actual use conditions.

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Correlation - Prediction

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This presentation will address itself to the selection rules for the optimum ceramic material to be subjected to thermal stress in a given thermal environment, performance criterion and failure condition. Subsequently, the results of comparisons between calculated and observed thermal stress behavior are discussed in detail.

Brittle ceramics may be expected to withstand severe thermal stress under a wide variety of thermal environments such as steady-state or transient heat transfer, convection or radiation or other conditions. Thermal stress resistance may be measured in terms of temperature differences, heat fluxes, heating rates, time-to failure, thermo-elastic stability and strength loss due to crack propagation. All these conditions and criteria are governed by a large number of individual material properties. The relative role of each property depends on the specific thermal environment, performance and failure criteria.

In order to establish the proper selection rules, it has been the practice in ceramic technology to derive simple analytical expressions for the conditions of the thermal stress resistance of a ceramic in terms of the particular heat transfer condition, and pertinent physical properties as decided by the performance criterion and failure condition (1-7). From the expressions obtained, so-called thermal stress resistance parameters can be derived, on the basis of which the most promising material can be selected for the environment, etc., to which the material is to be subjected. To this time some nearly thirty (30) such non-redundant expressions have been derived which lead to some twenty (20) different thermal stress resistance parameters.

As an example for the above approach, the expression for the maximum temperature difference ΔT_{max}, which can be tolerated across the wall thickness of a hollow tube subjected to radially inward or outward heat flow is:

$$\Delta T_{max} = \sigma_f(1 - \nu)C/\alpha E \qquad (1)$$

where σ_f is the tensile fracture stress
ν is Poisson's ratio
α is the coefficient of the exp.
E is Young's modulus
and C is a geometric factor.

Eq. 1 can be rewritten in terms of the maximum heat flow per unit length given by:

$$Q_{max} = \sigma_f(1 - \nu)KC^1/\alpha E \qquad (2)$$

where C^1 is another geometric constant and K is the thermal conductivity.

Eqs. 1 and 2 lead to the thermal stress resistance parameters:

$$R = \sigma_f(1 - \nu)/\alpha E \text{ and } R^1 = K\sigma_f(1 - \nu)/\alpha E$$

Similar expressions presented or to be presented in the literature for other heat transfer conditions, etc., are reviewed briefly. The appropriate thermal stress resistance parameters are presented collectively in Table I.

Comparisons of the predicted and experimental thermal stress resistance, carried out by the present writer and co-workers are discussed in detail (8-10). These studies include single-cycle and multicycle thermal shock with or without the presence of slow crack growth. For fatigue crack growth a numerical program was developed for the computer simulation of thermal fatigue. The results of these studies indicate that in general the thermal stress resistance of brittle ceramics can be carried out successfully. This, however, requires a detailed knowledge of the heat transfer conditions, extensive evaluation of physical property data, as well as extensive analytical or numerical analysis.

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SUMMARY OF THERMAL STRESS RESISTANCE PARAMETERS

A. Steady-State heat flow:

$S_t(1-\nu)/\alpha E$; $S_t(1-\nu)K/\alpha E$; $S_t(1-\nu)/\alpha\eta$; $S_t(1-\nu)K/\alpha\eta$

B. Thermal Buckling:

α^{-1}; $(S_t^2/\alpha^2 E)^{1/2}$; $(S_t/\alpha^3 E)$; $(S_t K/\alpha^3 E)$; $(S_t/\alpha^3 E)^{1/2}$

C. Transient Heat Transfer:

$S_t(1-\nu)\alpha E$; $S_t(1-\nu)K/\alpha E$; $(S_t(1-\nu)K/\alpha E\varepsilon)^{1/4}$; $(S_t(1-\nu)K/(1-F_{\lambda_o})\alpha E\varepsilon)^{1/4}$; $S_t(1-\nu)a/\alpha E$, a^{-1}

D. Crack Propagation:

$(G/\alpha^2 E)^{1/2}$; $(GK^2/\alpha^2 E)^{1/2}$; GE/S_t^2; GE/S_t