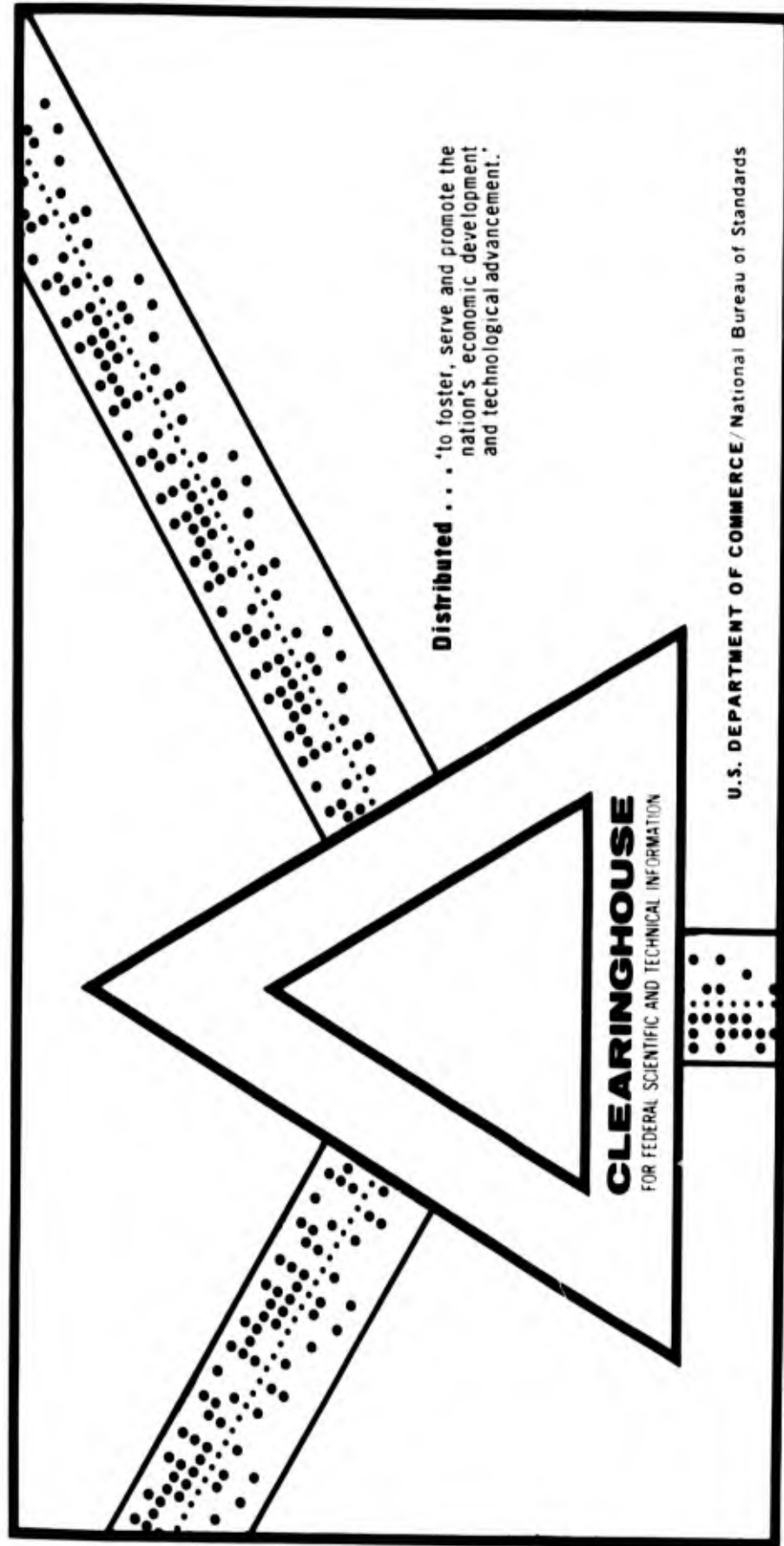


PRESSURE HULL CANDIDATE IONi DUAL STRENGTHENED STEEL

L. Schapiro

Lockheed Missiles and Space Company
Palo Alto, California

January 1969



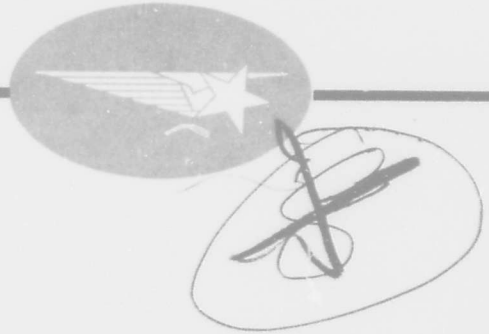
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PRESSURE HULL CANDIDATE
10Ni DUAL STRENGTHENED STEEL

by

L. Schapiro

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**PRESSURE HULL CANDIDATE
10Ni DUAL STRENGTHENED STEEL**

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**Materials Sciences Laboratory
Lockheed Palo Alto Research Laboratory
LOCKHEED MISSILES & SPACE COMPANY
A Group Division of Lockheed Aircraft Corporation
Palo Alto, California**

FOREWORD

The information and technical data contained in this report were developed during the conduct of two Lockheed Independent Development projects; "10 Nickel Hull Steel Characterization," and "10 Nickel Hull Steel Evaluation." The reported work is part of a continuing investigation to develop and advance the state-of-the-art in high strength, high toughness structural materials for deep submergence systems.

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Section 1
INTRODUCTION

The NAVSEC funded program which produced HY-80 and HY-100 steels for sea water pressure resisting use and progressed to the higher strength HY-140 steel has sought compositions suitable for the even higher strength designation HY-180. Starting with 18% nickel maraging steels and advancing to greater toughness and sea water stress corrosion resistance with decreasing nickel content, the Navy funded program in 1967 concentrated on a 10Ni-8Co-2Cr-1Mo dual strengthened composition.

In its DSSV preproposal activities, LMSC selected this 10Ni steel as the most desirable steel candidate for the forthcoming Navy competition and made overtures to the NAVSHIPS program controllers for the acquisition of such data as required by specification NAVSHIPS 0900-028-2010 for a Category III material. Approval from the NAVSHIPS funding office was needed for this earlier-than-planned advancement of the program to the needs of product application.

Total requirements for consideration of a Category III material in a Navy certified submersible necessitates acquisition of the following information:

- (a) Material chemistry requirements
- (b) Material mechanical properties
- (c) Basic process to be used in producing the material. Sufficient information is required to demonstrate that the procedures insure that repeatable material properties are obtainable by the process used
- (d) Lack of any susceptibility to failure when subjected to a dynamic shock wave such as that resulting from explosively jettisoning external equipment, implosion of a flotation sphere or any other airbacked component or equipment contained within or transported by the submersible
- (e) Effect of flaws, such as cracks or defects, on material performance

- (f) Effects of temperature on material performance and resistance to crack propagation
- (g) Results of tests to destruction of samples fabricated from the materials and comparison of these results with the design basis predictions of the failure point
- (h) Fatigue data in the high strain, low cycle range (less than 10,000 cycles) in environment (e.g., sea water, air HeO₂, etc.)
- (i) Data covering an extended time period justifying the adequacy of the material with respect to general corrosion and to stress-corrosion cracking in sea water in the presence of cracks, assuming the material is exposed to this type of environment
- (j) Proof of fabricability including data verifying the repeatability of results
- (k) Nondestructive test requirements to be applied to the base material and joints as appropriate
- (l) Hazards involved in fabrication or use of material with respect to toxicity or flammability

Our program for acquisition of mechanical properties [item (b)] selected 3-1/2-in. plate as the thickness to be evaluated since this thickness, at the reported material strength, would be required for a 20,000-ft operating depth submersible. For proof of fabricability [item (g)], our program included manufacture and depth testing of a subscale spherical pressure hull to illustrate material formability, weldability, and suitability of the weld quality for deep submergence service. As a step leading to Category II classification, our program also included explosive bulge testing of 2-in. plate weldments in conformance with NAVSHIPS 0900-005-5000.

Section 2
TEST PLAN

As funded by NAVSEC, U.S. Steel's development program with this 10Ni-8Co-2Cr-1Mo steel did not include production of 2-in. and 3-1/2-in. plate in 1968. Special approval was obtained from NAVSEC that permitted U.S. Steel to accelerate production of plate for this LMSC program. Accordingly, production heat L50967 was made as a vacuum induction melted 28-in. diameter 26,800-lb ingot and was vacuum arc remelted as a 30-in. diameter 23,500-lb ingot. This ingot was forged into two blooms: one, 14 by 50 by 60 in., was rolled to 3-1/2-in. plate for material properties characterization; the other, 8 by 44 by 60 in., was rolled to 1-5/8-in. plate for subscale pressure hull fabrication. From production heat L50881 comprising three 17-in. diameter 9,200-lb vacuum induction melted ingots which were vacuum arc remelted as three 20-in. diameter 8,500-lb ingots, one was converted to a 68 by 42 by 8 in. bloom which was rolled to 2 by 19 by 145-in. plate for explosive testing by NAVSEC.

Special arrangements were made for U.S. Steel to perform all the material properties characterization tests. The following specific tests were enumerated by LMSC purchase order:

A. 2-in. Plate

1. Fabricate the standard set of six explosive bulge tests weldments and arrange for the explosive testing with NAVSEC.
2. From prolongations from the explosive bulge weldments, conduct the following tests:
 - (a) Tension at room temperature - 2 minimum
 - (1) Base metal (0.505-in. -diameter test specimen or larger); longitudinal and transverse directions at plate quarter-thickness
 - (2) Transverse to weld - full plate thickness specimens
 - (3) All weld metal (0.252-in. -diameter test specimen or larger)

- (b) Charpy vee-notch impact at $0^{\circ}\text{F} \pm 3^{\circ}\text{F}$
 - (1) Base metal - 2 minimum; longitudinal and transverse directions at plate quarter-thickness
 - (2) Transverse to weld - 3 minimum with notch normal to plate surface; top and bottom quarter-thickness
 - (c) Compression at room temperature with stress-strain curves
 - (1) Longitudinal and transverse directions at quarter-thickness
 - (2) Short transverse direction
- B. 3-1/2-in. Plate**
- 1. Tension at room temperature - 2 minimum (0.252-in. diameter specimen)
 - (a) Longitudinal and transverse directions at surface, quarter-thickness, and midthickness locations.
 - (b) Short transverse direction at two width locations (preferably near plate edge and near plate midwidth).
 - 2. Charpy vee-notch impact at $0^{\circ}\text{F} \pm 3^{\circ}\text{F}$ - 2 minimum
 - (a) Longitudinal and transverse directions at surface, quarter-thickness, and midthickness locations.
 - (b) Short transverse direction at two width locations (preferably near plate edge and near plate midwidth).
- C. K_{IC} and K_{ISCC} values on A and B material.**

Section 3 TEST RESULTS

Chemical compositions of the heats and plates making up this program are detailed in Table 1. Characterization of the tension and impact properties of 3-1/2-in. plate is detailed in Table 2. Comparable properties of 2-in. plate are detailed in Table 3, and of 2-in. plate weldments in Table 4. A representative stress-strain diagram for compression loading and strain recording with Huggenbergers is presented in Fig. 1. Fracture toughness values are presented in Table 5, as well as such stress corrosion threshold values in sea water as are completed at this date. When U.S. Steel completes those tests, the missing values will be added to this report.

Crack propagation rate during cyclic loading is a material property receiving much current interest in Navy circles. The fatigue crack propagation rate for this steel was evaluated by U.S. Steel and is presented in Fig. 2.

3.1 EXPLOSIVE BULGE TESTS

Spherical and cylindrical explosive bulge tests were performed on the 2-in. plate weldments by NAVSEC with stand-off distance of 17 in. , at 30° F. with 42-lb Pentolite charge.* Spherical bulge test results are presented in Table 6. Cylindrical bulge tests results will become available when the Navy report is issued. We are advised the results were satisfactory in both bulge tests and were better than the results with HP 9-4-20 steel in a companion test program.

3.2 SUBSCALE PRESSURE HULL

A 38-in. o.d. sphere was made, Fig. 3, using the 1-5/8-in. plate for hemispheres and 3-1/2-in. plate for insert and plug. The insert and plug - simulating a hatch

*For perspective comparison, HY-80 is tested with 24-lb charges at same stand-off distance.

Table 1
HEAT ANALYSES AND CHECK ANALYSES

Heat No. :	3-1/2- and 1-5/8-in. Plate		2-in. Plate		Weld Wire
	L50967		L50881		W8560
Element	Heat Analysis	Plate Check	Heat Analysis	Plate Check	Heat Analysis
C	0.118	0.12	0.106	0.11	0.10
Mn	0.09	0.052	0.13	0.04	<0.01
P	0.005	0.004	0.005	0.005	0.001
S	0.002	0.005	0.003	0.005	0.005
Si	0.03	0.038	0.08	0.09	0.12
Ni	10.11	10.0	10.05	10.0	10.2
Cr	1.99	1.99	2.19	2.20	2.1
Mo	1.05	1.00	1.01	1.00	1.0
Co	8.00	8.00	7.86	7.94	6.0
V	—	—	—	—	0.065
Ti	0.007	0.007	0.018	0.014	
Al ^(a)	<0.01	0.001	<0.01	0.005	0.023
N ^(b)	20	20	50	20	10
O ^(b)	21	5	21	10	—

(a) Total aluminum.
(b) Parts per million.

Table 2
3-1/2-IN. PLATE PROPERTIES^(a)

Location	Orientation	Tension (Room Temperature)				Charpy (0° ± 3°F)		
		Yield Strength (ksi)	Ultimate Strength (ksi)	Elong. in 2 in. (%)	Reduction of Area (%)	Energy Absorption (ft-lb)	Fibrous Fracture (%)	Lateral Expansion (mils)
Surface	Longitudinal	176	185	17.0	70.1	61	100	34
	Transverse	176	185	17.0	67.8	64	100	35
Quarter Thickness	Longitudinal	177	186	17.5	70.9	62	100	31
	Transverse	177	187	17.5	71.7	73	100	45
Midthickness	Longitudinal	173	189	17.5	68.9	53	100	26
	Transverse	177	191	17.5	69.7	54	100	25
Plate Midwidth	Longitudinal	173	187	18.0	71.1	56	100	31
	Transverse	176	189	18.0	71.1	56	100	32
Plate Edge	Longitudinal	171	187	17.5	69.4	55	100	26
	Transverse	173	190	17.5	69.6	57	100	23
	Thru Thickness ^(b)	176	190	17.5	70.6	58	100	31
	Thru Thickness ^(b)	173	187	17.5	71.1	62	100	36
	Thru Thickness ^(b)	173	189	16.0	67.6	47	100	22
	Thru Thickness ^(b)	173	187	16.5	68.1	48	100	23
	Thru Thickness ^(b)	170	184	15.5	62.3	32	100	15
	Thru Thickness ^(b)	169	184	16.0	63.6	32	100	13

(a) Austenitized 3 hr at 1650 F and water quenched, re-austenitized 3 hr at 1500 F and water quenched, and aged 16 hr at 950 F and water quenched.

(b) Tension specimens 0.252-in. diameter with 0.1-in. gage length for elongation percent.

Table 3
2-IN. PLATE PROPERTIES(a)

Location	Orientation	Tension (Room Temperature) (0.505-in. diameter)			Charpy (0° ± 3°F)			Compression (Room Temperature)		
		Yield Strength (ksi)	Ultimate Strength (ksi)	Elong. in 2 in. (%)	Reduction of Area (%)	Energy Absorption (ft-lb)	Fibrous Fracture (%)	Lateral Expansion (mils)	Yield Strength (ksi)	Elastic Modulus (10 ⁶ psi)
Quarter Thickness	Longitudinal	183	192	16.5	70.2	73	100	38	193	29.42
	Transverse	184	197	17.0	68.1	82	100	38	194	
Midthickness (b)	Transverse	182	192	17.0	72.6	76	100	37	193	29.55
	Longitudinal	184	193	17.0	72.5	80	100	39	194	
	Transverse	189	206	17.0	70.2	72				
	Thru Thickness	190	206	16.0	70.7	57				
	Thru Thickness	150	193		68.0				189	
									191	

(a) Austenitized 2 hr at 1500° F and water quenched. Aged 10 hr at 950° F and water quenched.

(b) Specimen diameter 0.252, gage length 1 in., aging 5 hr.

Table 4
2-IN. PLATE WELDMENTS^(a)

Location	Orientation	Tension (Room Temperature)				Charpy (0° ± 3°F)		
		Yield Strength (ksi)	Ultimate Strength (ksi)	Elong. (%)	Reduction of Area (%)	Energy Absorption (ft-lb)	Fibrous Fracture (%)	Lateral Expansion (mils)
Top Quarter Thickness	All Weld Metal ^(b)	189	192	17.0	73.4	102 ^(c)	100 ^(c)	
Bottom Quarter Thickness	All Weld Metal ^(b)	189	196	15.7	73.3			
Top Quarter Thickness	Transverse to Weld	191	193	16.5	72.0	111 ^(c)	100 ^(c)	
Bottom Quarter Thickness	Transverse to Weld	189	196	16.5	71.7			49
Full Plate Thickness	Transverse to Weld	184	190	15.0 ^(d)	66.0	101	100	53
		183	190	14.9 ^(d)	59.0	111	100	55
						102	100	50

- (a) Heat treatment same as in Table 1 with welding after austenization and before aging.
 (b) 0.357-in. diameter specimens, elongation in 1.54-in. gage length in conformance with G. L. = 4.5 area.
 (c) Average of triplicate tests.
 (d) Elongation in 5.45 in. gage length.

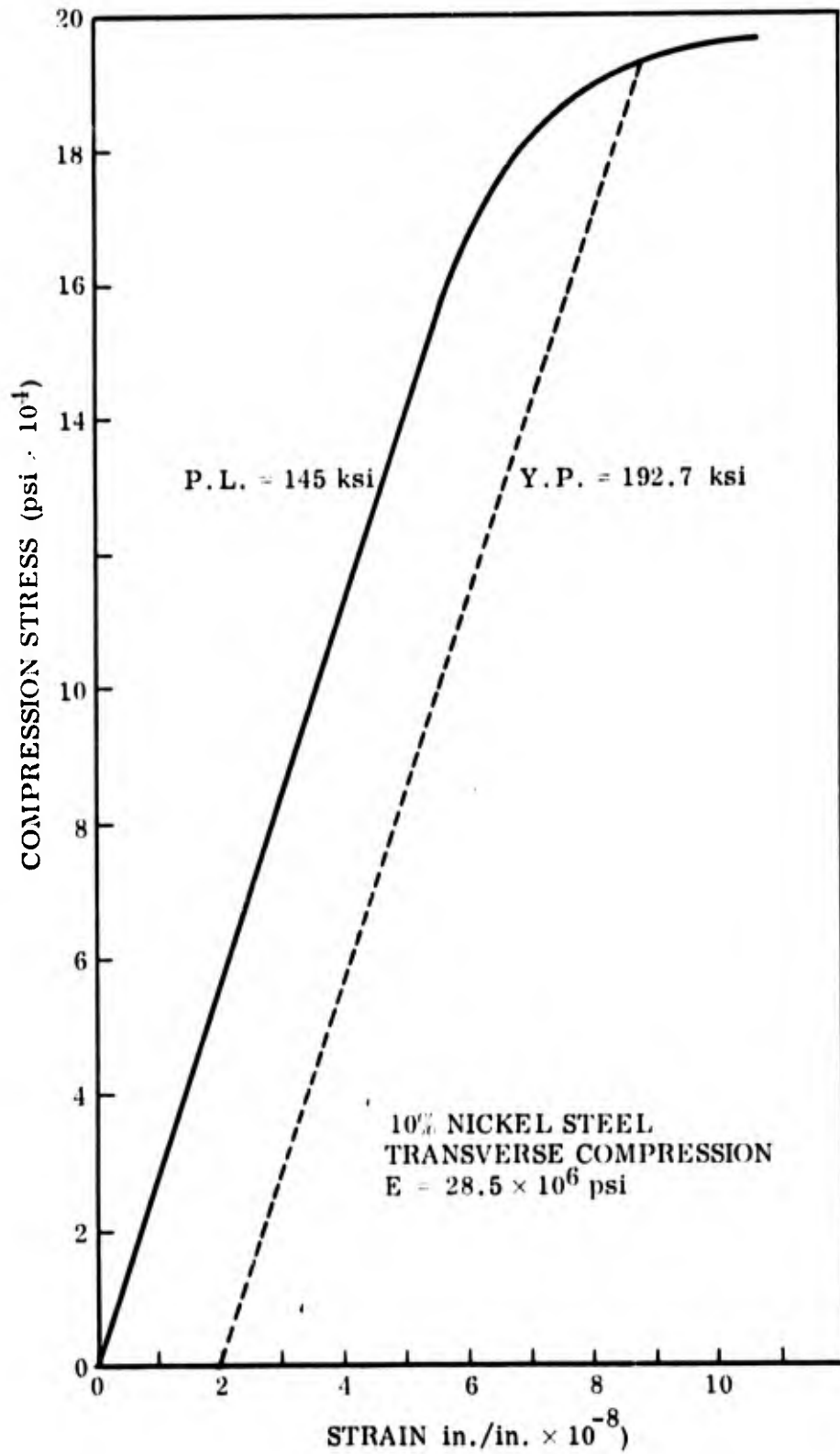


Fig. 1 Representative Stress-Strain Diagram for Compression Loading and Strain Recording

Table 5
TOUGHNESS DATA

	K_{IC}	$C_{V_{0^{\circ}F}}$	K_{ISCC}
<u>2-in. Plate</u>			
20-in. ingot, VAR			
Parent	202(?)	80	170
Weld	280(?)	100	In test 178(?)
20-in. ingot, \overline{VAR}^2			
Parent	300(?)	168	In test
<u>3-1/2-in. Plate</u>			
30-in. ingot, VAR			
Parent	170	60	In test

(?) Test invalid; 2- by 8-in. specimen used; a larger one needed.
 \overline{VAR}^2 double vacuum arc remelting.

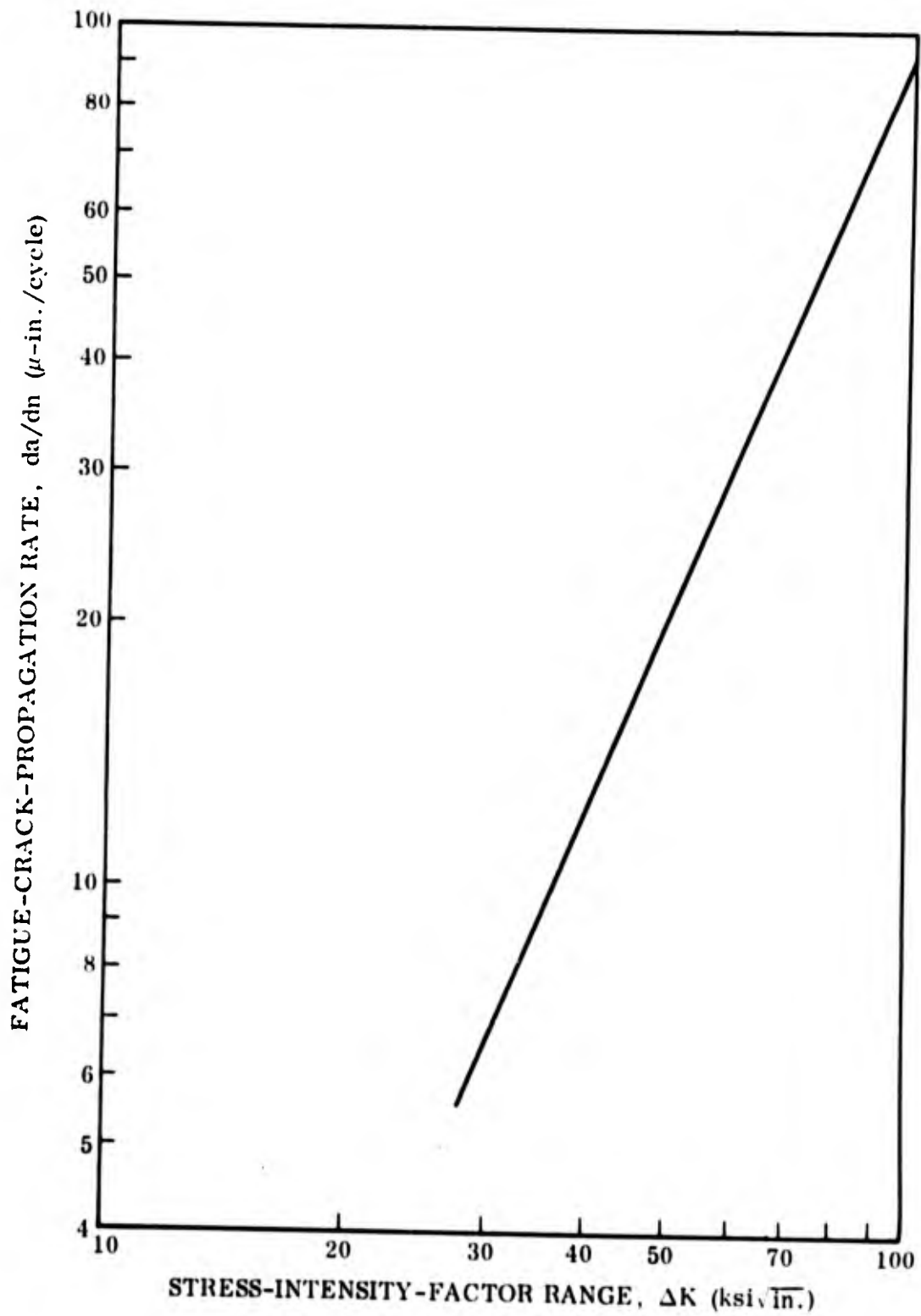


Fig. 2 Fatigue Crack Propagation in 10Ni-2Cr-1Mo-8Co Steel

Table 6
RESULTS OF 10Ni-Cr-Mo-Co EXPLOSION-BULGE TESTS

Type Specimen	Shot No.	Thickness Reduction (%)	Bulge Depth (in.)	Remarks
Crack starter	1	1.53	1.1	Superficial cracks around crack-starter beads
	2	4.9	2.2	Superficial cracks around crack-starter beads
Bulge	1	2.1	1.0	No visible cracks or tears
	2	4.1	1.9	No visible cracks or tears
	3	8.0	2.7	No visible cracks or tears
	4	13.1	3.2	No visible cracks or tears
	5	14.1	3.8	No visible cracks or tears
	6	16.1	4.3	8-in. -long crack in over-aged region of HAZ
Bulge	1	1.8	1.1	No visible cracks or tears
	2	4.7	2.0	No visible cracks or tears
	3	8.2	2.6	No visible cracks or tears
	4	11.0	3.5	No visible cracks or tears
	5	15.7	3.8	No visible cracks or tears
	6	19.4	4.6	No visible cracks or tears

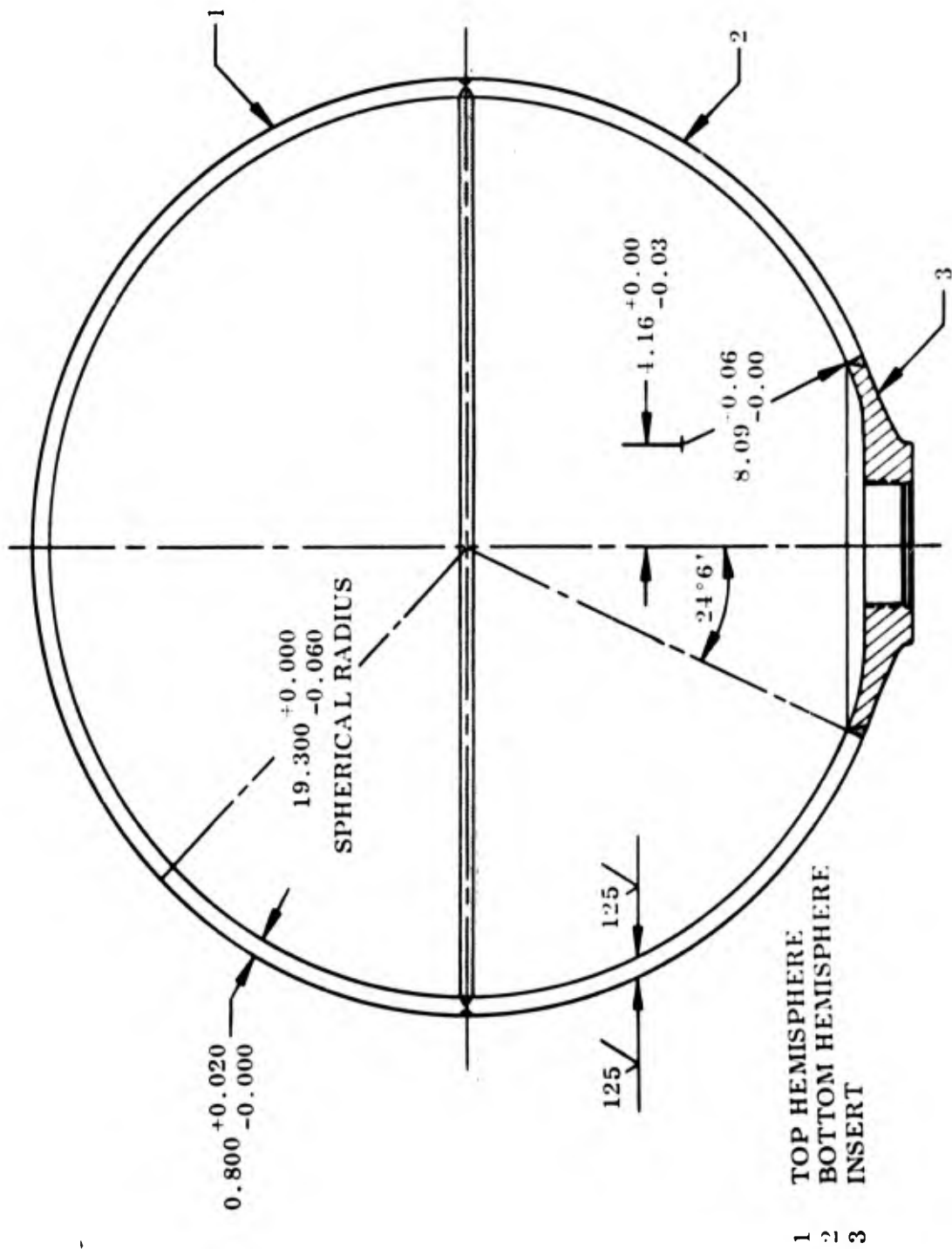


Fig. 3 Subscale Pressure Capsule

and cover – were machined from 3-1/2-in. plate which had been austenitized but incompletely maraged. Maraging treatment was completed when the fully assembled sphere was maraged. Properties associated with this treatment are presented in Table 7. The hemispheres of this sphere were austenitized after hemisphere forming. Plate trim stock of 1-5/8-in. thickness accompanied the hemispheres through the austenitization heat treatment and accompanied the assembled sphere during maraging. Tension and impact tests from this test plate (test results are in Table 8) are not in agreement with the properties of this steel established by all other tests. Differences have definitely been established as resulting from the very slow heating rate of the furnace used for maraging the assembled (welded) sphere.

Precipitate nucleation occurring well below aging temperature with slow heating presumably is of such microscopic particle size that customary time at aging temperature does not produce the same result as the same aging time and temperature with rapid heat-up in air circulating ovens. The net result of such nucleation and incomplete growth is a higher strength (yield and ultimate) with a lower charpy toughness than customarily is associated with a specified maraging cycle. A 20,000-psi strength increase with a 20-ft-lb toughness decrease seems to be associated with such slow heating coupled with normal maraging.

Inside strain gages were applied to the sphere by Southwest Research of San Antonio in accordance with the planned placement shown in Fig. 4, on a meridian passing through the point of maximum mismatch at the insert weld as determined by sphericity measurements performed by Exceleco. Sphericity data appear in Table 9.

External strain gaging was applied at NSRDC-Carderock by their personnel. The sphere will be given the following tests in accordance with NSRDC-Carderock desires:

- Preliminary hydrostatic pressure tests in oil, three runs to pressures equivalent to depths of approximately 10,000, 20,000, and 24,000 ft.
- Cyclic tests with inside and outside surfaces exposed to salt water, 3,000 cycles to a 22,000-ft depth and 3,000 cycles to a 24,000-ft depth at 2 min per cycle.
- Salt water pressurize to maximum facility capability.

Results of NSRDC-Carderock depth testing will be reported separately.

Table 7

3-1/2-IN. PLATE PROPERTIES IN SUBSCALE SPHERE

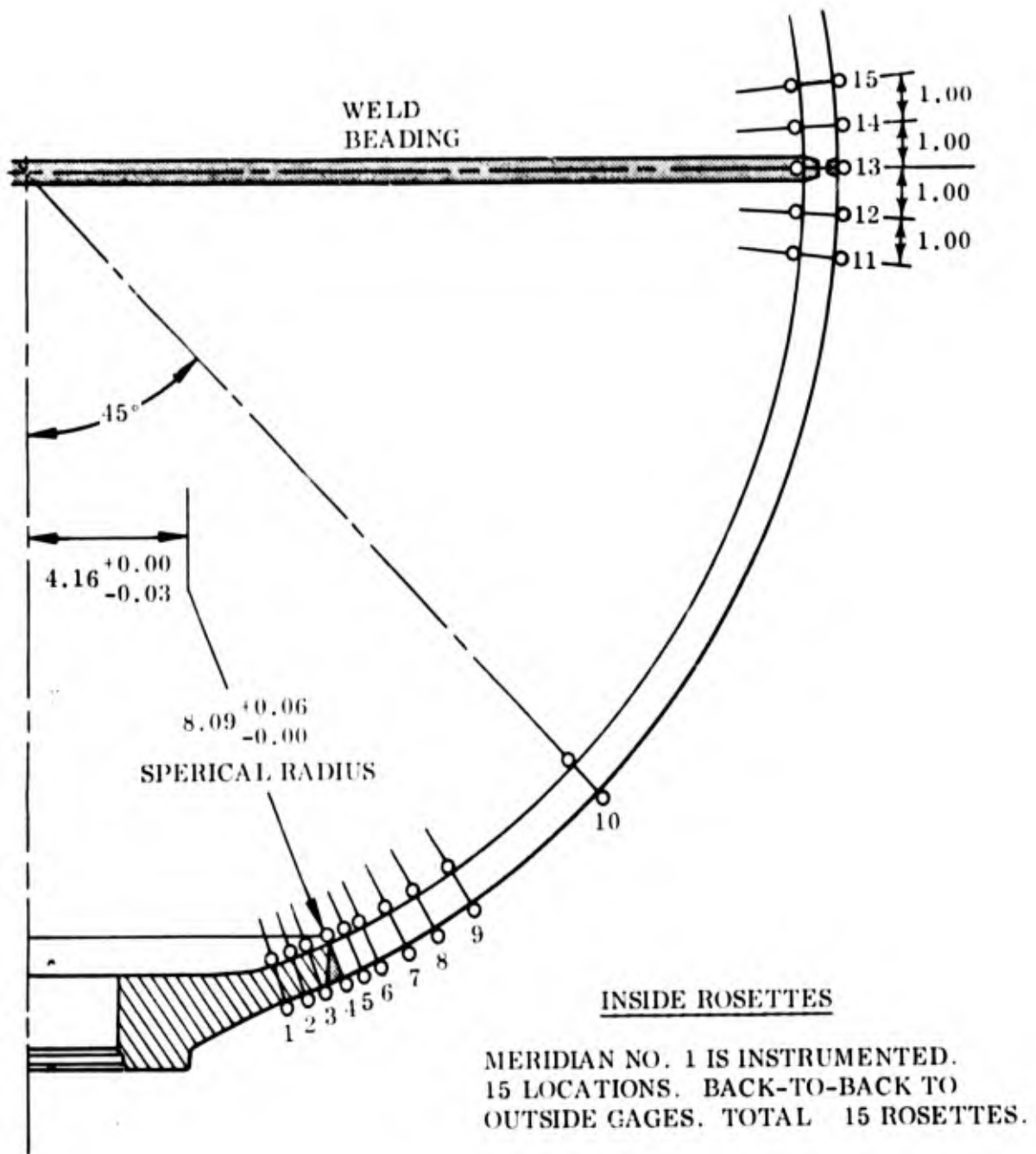
Furnished to Exello austenitized 3 hr at 1650° F and water quenched, re-austenitized 3 hr at 1500° F and water quenched, and partially aged 10 hr at 950° F and water quenched. Longitudinal properties as furnished.

Specimen Location	Yield Strength (0.2% Offset) (ksi)	Tensile Strength (ksi)	Elongation in 1 in. (%)	Reduction of Area (%)	Hardness, R _C	Charpy V-Notch Energy Absorption at 0° F (ft-lb)
Surface	181	200	17.5	71.9	43.0	55
Quarter Thickness	176	202	17.0	68.0	43.0	47
Midthickness	180	203	16.5	67.7	43.0	50
Properties expected after the additional 10 hr aging at 950° F given to assembled (welded) sphere.						
Surface	175	187	15.0	71.5	40.0	74
Quarter Thickness	175	189	18.0	70.0	40.5	63
Midthickness	170	187	18.0	70.5	40.5	60

Table 8
1-5/8-IN. PLATE PROPERTIES

	Tension (Room Temperature)				Charpy (0' ± 3' F)
	Yield Strength (ksi)	Ultimate Strength (ksi)	Elong. in 2 in. (%)	Reduction of Area (%)	Energy Absorbed (ft-lb)
Maraged ^(a) with subscale sphere					
	Parent Material	203.6 209.3	221.9 222.1	21.0 20.1	66.5 67.0
Weldment					
		197.2 199.2	209.0 212.0	18.0 19.0	73.8 72.0
Maraged ^(b) by U. S. Steel	184.0	199.0	17.0	70.2	70

- (a) Maraging cycle was 10 hr at 950° F. Heating was very slow in a noncirculating furnace.
 (b) Same cycle but with normal heating rate.



LOCATE MERIDIAN NO. 1 AT THE PLACE
WHERE MAXIMUM INSERT MISMATCH OCCURS.

Fig. 4 37-in. Diameter, 10Ni Steel Pressure Vessel Test Instrumentation

