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SUPPLEMENTARY REPORT
MAY 1964
DOUGLAS REPORT SM-43105

MISSILE & SPACE SYSTEMS DIVISION
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EVALUATION OF MARAGING STEEL FOR APPLICATION TO SPACE LAUNCH VEHICLES

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DOUGLAS MISSILE & SPACE SYSTEMS DIVISION

ABSTRACT

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The principal objective of this investigation was to differentiate between the effects of retained austenite and titanium nitride inclusions on 18Ni-7Co-5Mo maraging weldmetal fracture toughness. Previously, it had been shown that normally aged weldmetal was characterized by an austenitic network laden with TiN. Weldmetal fractures propagated through these networks, preferentially, and resulted in fracture toughness properties which were substantially inferior to parent plate. The results of the present investigation demonstrated that the TiN inclusions alone (i.e., independent of the surrounding austenitic islands) were the primary cause of impaired toughness. This was determined by employing thermal treatments which eliminated austenite while leaving the nitrides substantially unaffected.

Additional observations suggested the possibility of nitride precipitation at elevated temperatures. Such a precipitation reaction, if indeed it exists, could under certain conditions promote further toughness degradation.

The presence of cracks in, or adjacent to, banded weld heat-affected zones was observed for the first time during this overall study. The possibility exists that such cracks represent a delayed cracking phenomenon.

Two areas deserve future study. First, nitrogen-free maraging alloys should be developed and thoroughly evaluated. Second, the phenomenon of banding cracks should be thoroughly investigated.

Author

I. INTRODUCTION

The initial work (Reference I) on the fracture toughness of 18-7-5 maraging steels (250 KSI grade) showed that plate is characterized by a banded structure. This banding seems to be related to the presence of angular inclusions which were tentatively identified as being titanium nitride. In aged weldmetal these inclusions appear to be segregated in the form of a semi-continuous network enveloped by clear-etching islands within a matrix which shows uniform precipitation. Electron fractographic studies showed that these titanium nitride inclusions tend to be associated with relatively flat fracture surfaces. The inclusions themselves appear to fracture by cleavage mode. Occasionally observed low fracture toughness in plate heat-affected regions as well as the inferior toughness of welds is apparently associated with an abundance of titanium nitride inclusions on the fracture surfaces.

The clear-etching islands almost always associated with the inclusions were hypothesised to be retained austenite. It was considered that the lowered fracture toughness of weldmetal could result from the presence of such a duplex structure. If the tentative identification of the inclusions as TiN was correct, the chemical nature of this compound would preclude its elimination by thermal treatment. However, if the clear-etching islands were retained austenite, thermal treatments could be designed for their elimination. Thus, if retained austenite played any part in the degradation of weldment properties, it's effect could be eliminated, and weldment properties improved.

This study represents an attempt to evaluate the fracture toughness of 18-7-5 maraging steel weldments in terms of the effects of a variety of

1. INTRODUCTION (Cont'd.)

thermal treatments upon their microstructures.

2. PROCEDURE

2.1 Test Samples

Test samples were acquired from a welded plate identified as No. 7C. This had been formed by welding specimens 5W6 and 5W8 during the initial program. This plate was from United States Steel, Heat No. 52911, designated Heat-B. Welding of the plate followed welding procedure W-7 which consisted of a two-pass MIG process. Complete history of this procedure is given in Reference 1. The specimen configuration for microstructure studies shown in Figure 1 was selected because it not only contained weldmetal, the primary object of this investigation, but also offered an opportunity to study the gross effects of various heat-treatments on both the parent plate and the heat-affected zone as well. Approximately the same mass was used for each specimen so that lag time during heating would not be a variable.

2.2 Heat Treatment

Several specimens were maintained in the as-welded condition. Each group of specimens which was subjected to a given thermal treatment contained one to which a thermocouple had been attached. Three temperatures, 1500, 1800 and 2100^{o}F (815, 982 and 1139^{o}C), were selected to study austenitization effects upon microstructure. Continuous temperature readings were made until the temperature stabilized. Thermal stabilization was reached after approximately 15 minutes; this provided the initial time for the isothermal treatments. The time at temperature was maintained at 16 hours

2.2 Heat Treatment (Cont'd.)

to permit the essential completion of the reaction at the given temperatures. One group of specimens was austenitized for 94 hours at 2100°F.

The specimens which were austenitized at 1500 and 1800°F were treated in an argon atmosphere. No protective atmosphere was employed in the heat treatment of specimens at 2100°F.

All subsequent aging was done at 900°F (482°C) for three hours. The complete thermal histories of all specimens for microscopic examination are given in Table I.

2.3 Austenite Content Determinations

2.3.1 X-Ray Diffraction

Determinations of austenite by X-ray methods were made on maraging steel weld samples representing seven different post-weld heat treatment combinations. It was found that conventional techniques are inapplicable to the present material because of preferred orientation and weak diffracting characteristics. As a result, it was necessary to develop a special technique. Some exploratory experiments and studies have indicated that several approaches are possible. Future work should consider the problem in detail, in order to evaluate the various approaches more thoroughly.

The standard Douglas procedure (DLP 13.811) for the determination of retained austenite, employs a basic method (Reference 2) which is quite widely accepted and applied. The Douglas procedure employs an X-ray diffractometer and a flat-surface reflection specimen, which is rotated in the plane of the diffraction surface. Integrated intensity measurements

2.3.1 X-Ray Diffraction (Cont'd.)

are obtained by fixed-time scans of several selected peaks for both the martensite (or ferrite) and austenite phases. The experimental intergrated intensities are weighted by those factors responsible for the differences in intensities among peaks of the same phase. This allows a quantitative estimate of retained austenite by ratioing the integrated intensity of an austenite peak to that of a martensite peak.

Although the routine application of the above technique is commonly employed with success to a variety of steels, application to maraging steel proved to be inappropriate. Two types of difficulties are responsible. The maraging steel samples employed were found to be poorly diffracting (more-so even than most steels). This results in broadening of peaks and reduced maximum peak intensities. Additionally, the degree of preferred orientation present in both the austenite and martensite phases was much more than could be "averaged-out" by simple spinning of the flat specimen in its own plane, as is usually done.

Determination of retained austenite was accomplished using a Norelco diffractometer, and an integrating pole figure device of the Schulz reflection design (Reference 3). A General Electric XRD-6 diffractometer was employed for a portion of the investigatory work. The present work employed both flat-reflection specimens mounted on the pole figure device, and filings mounted in the specimen spinner. The flat specimens were mounted in the Norelco diffractometer pole figure device. The diffractometer is set to automatically reverse the goniometer travel direction at both ends of a small angular interval, which covers both the (110)-martensite and (111)-austenite peaks (76 to 80 degrees 2θ for Cr K_α

2.3.1 X-Ray Diffraction (Cont'd.)

radiation). This allows a continuous repeating scan (at 1°, 2θ, per minute) of the two peaks. Simultaneously, the pole figure device is continuously rotating the specimen (20° per minute) in its reflecting plane, and tilting the specimen (1° per minute) with respect to the goniometer axis, up to 70°. As a result, the orientation of the planes in the specimen satisfying the diffraction conditions at a given time, trace out a spiral from the center out to 70° (the limit of the device) on a stereographic net.

The combination of continual repeat scanning and change in specimen orientation gives 18 (110-martensite and (111)-austenite (if present) peaks, for as many different portions of the stereographic net. For purposes of this project, the areas under the peaks (proportional to integrated intensities) were obtained by visual counting methods, with consideration given for over-lapping of peaks. Each peak was corrected for the usual factors, i.e. Debye-Waller temperature factor, multiplicity, volume of unit cell, structure factor per unit cell and Lorentz-polarization factor. Ratios of corrected integrated intensities (in arbitrary units) were then used to estimate volume fraction of austenite for each set of peaks. The resulting values were then averaged to give an approximate overall value. It is assumed for convenience that the average value for the 20° ring of the stereographic net (70° to 90°) will be approximately the same as that calculated for the central 70° region.

2.3.2 Magne-Gage Measurements

An effort to detect the transformation of austenite to martensite was made by use of an Aminco Brenner Magne-Gage, Model No. 2 using magnet No. 1.

2.3.2 Magne-Gage Measurements (Cont'd.)

This proved to be ineffective and was abandoned.

2.4 Fracture Toughness Evaluations

Several untested partial thickness crack slab-coupons were available from the original program. These coupons were 8-inches long, 11/16-inch wide and 5/16-inch thick. The cracks were induced at the geometric center on the 11/16-inch flat and were propagated through the 5/16-inch thickness direction. Parent plate and weldmetal were evaluated. As stated before, these specimens were available from the original program; therefore, they represented several conditions. Some were as-received, some were heat treated, and some were heat treated and had cracks already induced. The prolonged austenitizing treatments of 1800°F (982°C) for 16 hours were considered sufficient to render any prior condition inconsequential. Any prior cracks were extended after heat treating so that a virgin crack front would be operative.

3. RESULTS

3.1 X-Ray Diffraction

Of the seven post-weld heat treat conditions represented by the various specimens, only #2, 9, 10 and 20 conclusively revealed the presence of austenite. Specimens for #4, 5, and 14 showed no evidence of austenite in any of the X-ray work. Judging from the values obtained, the lower level of detection probably is about 3 to 5%. Specimen 9 contained the highest amount of austenite, about 11 percent, while specimens 2, and 10 contained somewhat less, about 9 percent. Specimen #20, for which no flat specimen

3.1 X-Ray Diffraction (Cont'd.)

piece was run, revealed definite austenite peaks in the filings examined, generally comparable to those of #2, 9, and 10. The data for the retained austenite measurements are given in Table I.

3.2 Metallographic Observations

3.2.1 As-Deposited Weldmetal

Figure 2 illustrates the appearance of as-deposited weldmetal. Figure 2A shows a low magnification optical micrograph which exhibits the interpass fusion line. The microstructure apparently consists of as-quenched martensite and islands of retained austenite. The austenite occupies approximately 9% by volume according to X-ray diffraction data. Figure 2B shows the typical structure as revealed by electron microscopy at 6,500 X. Scattered inclusions of TiN (as identified in Reference 1) are also shown in the electron micrograph. These inclusions are commonly found in weld-metal.

3.2.2 Directly Aged Weldmetal

Figures 3A and 3B portray the typical appearance of directly aged weldmetal. Aging for 3 hours at 900°F was employed for the cases illustrated. However, this microstructure is typical after a wide range of aging temperatures. Figure 3A is an optical micrograph illustrating the typical semi-continuous austenitic network (approximately 11% by vol. according to X-ray diffraction data) in a matrix of aged martensite. Figures 3B and 3C represent typical electron micrographs of these regions. Probable austenitic islands are identified by the letter "A".

3.2.3 Austenitized-and-Aged Weldmetal

Austenitizing was performed at 1500, 1800, and 2100°F for times ranging from 16 to 94 hours. Subsequently the specimens were aged at 900°F for 3 hours. The weldmetal microstructure after some of these treatments changed considerably from that observed for directly aged weldmetal.

Austenitizing at 1500°F produced no obvious change in microstructure. Figure 3A and 3B, representing directly aged weldmetal, were found to be equally representative of microstructures after a 1500°F austenitizing treatment and aging, Figure 3C. Austenite content was found to be about the same as for the as-deposited and directly aged specimens (i.e., about 9 to 11% by volume).

Austenitizing at 1800 or 2100°F (followed by aging) produced striking microstructural changes. Figures 4A, 4B, and 4C illustrate the typical microstructures. Figure 4A shows an optical micrograph of aged weldmetal previously subjected to a 16 hour, 1800°F austenitizing treatment. The absence of the austenitic islands which are typical of as-deposited and directly aged weldmetal is to be noted. No austenite was detectable by X-ray diffraction in any of the three specimens subjected to austenitizing treatments at 1800 or 2100°F. The microstructure appears to consist of rather uniform aged Widmanstätten-like martensite. Also quite prominent are numerous particles of what is probably TiN. The arrows in Figure 4A indicate some of these randomly dispersed particles. Figures 4B and 4C are typical electron micrographs which respectively show nitrides and apparent subgrain structure in the aged martensitic matrix. The prior austenitic grain boundaries were also quite evident after 1800 and 2100°F austenitizing treatments and grain size appeared to range in size from

3.2.3 Austenitized-and-Aged Weldmetal

ASTM #4 to ASTM #6. The #4 size was more typical of the 16 hour 2100°F treatment, while #6 was more representative of the 16 hours, 1800°F treatment. Extreme grain coarsening occurred after 94 hours at 2100°F whereupon the prior austenitic grain size approached ASTM #2.

3.2.4 Banding in Parent Plate and Heat-Affected Zones

Frequently, during the course of this investigation several unusual observations were made pertaining to the banding that is prevalent in this alloy. The observations concerned (1) the apparent phase transformation of nitrogen-enriched austenite to a ferritic matrix containing nitrides and (2) the incidence of microcracks paralleling the band in heat-affected zones.

Figure 5 illustrates a series of electron micrographs portraying a typical banded region in mill-annealed and aged plate or "as-deposited" heat-affected zones. The microstructure of these regions appears to consist of aged martensite with light-etching islands of austenite and/or blocky ferrite. Blocky ferrite could result from the transformation of metastable austenite upon cooling from the aging temperature. Occasionally, nitrides are also observed in these regions. Specimens that were subjected to austenitizing treatments at 1800 and 2100°F and then aged, invariably exhibited a totally different microstructure within such bands. Figures 6A and 6B show the usual microstructure after such a treatment. The banded regions tend to darken and the TiN particles become very prominent. Frequently, nitrides were also observed along grain boundaries as illustrated in Figure 6C. These observations suggest, but do not prove, that nitrides may result from, or may enlarge from, the

3.2.4 Banding in Parent Plate and Heat-Affected Zones (Cont'd.)

decomposition of retained austenite.

Another unusual observation pertains to a rather high incidence of cracking in bands located within, or very close to, the heat-affected zones. Table I, under the remarks column describes the locations and conditions under which the cracks were observed. Figures 6D and 6E illustrate typical crack regions. Figure 6D indicates the most common location in banded heat-affected regions near the overlap of the two weld passes. One additional type of crack was observed, however in only one of the specimens examined. Figure 6F illustrates this unusual type which appears to be integranular in appearance and located adjacent to the weld fusion-line.

3.2.5 Apparent Nitride Formation When Austenitizing in Air

This section describes some observations which indicate that severe nitride concentrations may develop when maraging steel is exposed to air at elevated austenitizing temperatures. Such occurrences may be particularly important with respect to mill practices. Prior to ingot reduction prolonged, high temperature, soak times are usually employed to homogenize the ingot. The results described in this section indicate that severe surface nitriding could result from such a treatment.

The initial group of specimens austenitized at 2100°F for 16 hours were exposed to an air atmosphere. Upon removal of these specimens from the furnace, a heavy, loose scale was observed. A tightly adherent under-scale was found beneath the loose outer-scale. Figure 7 shows a typical cross-section. Below the adherent under-

3.2.5 Apparent Nitride Formation When Austenitizing in Air (Cont'd.)

scale the parent material (either plate or weldmetal) exhibited substantial penetration of a rather uniform dispersion of apparent TiN particles. Figure 7 also depicts this region of penetration. Needle-shaped nitrides are also prevalent closer to the scale interface.

Additional specimens were austenitized at 2100°F in a 25 micron vacuum. No attack of the type observed for air-treated specimens was found on the vacuum-treated specimens. Apparently, nitrogen from the air is capable of causing nitride formation in maraging steel exposed to elevated austenitizing temperatures.

3.3 Fracture Toughness Tests

The microstructural observations on the effects of prolonged austenitizing treatments revealed that substantial microstructural changes in weldmetal could be produced. Austenitizing at 1800°F, followed by aging, produced a uniform aged-martensitic matrix containing a general dispersion of apparent TiN particles. No austenite was detectable by X-ray diffraction. Hence, compared to directly-aged weldmetal (containing about 11% austenite by vol.) a significant microstructural difference was developed. Testing specimens subjected to the 1800°F treatment would, therefore, permit the determination of the effect of measureable austenite content on fracture toughness. Weldmetal and parent-plate specimens were subjected to the 16 hour, 1800°F treatment, aircooled, and aged at 900°F for 3 hours. Partial-thickness crack (PTC) fracture toughness specimens were tested as described in Reference 1. The results are presented in Figures 8 and 9 for weldmetal and parent plate, respectively. The scatterband for directly-aged material (Reference 1 data) is shown for comparison. (Tabulated data are presented in Table II.) As may be seen, the fracture strengths of the austenitized-and-aged specimens generally fall within the scatterband for their directly-aged counterparts reported in Reference 1. The microstructural differences had no apparent effect on fracture toughness.

3.4 Electron Fractography

Several of the fracture toughness specimens were employed for electron fractographic evaluation. This work was done to determine microstructural relationships to fracture. Replicas of fracture surfaces were made by the plastic-carbon method (chromium shadowed at 45°) using a nitrocellulose substrate on the fracture surface. Figures 10A and 10B show typical weldmetal fracture surfaces of specimens subjected to the 1800°F austenitizing treatment followed by 900°F aging. Several small clusters of what was previously identified as TiN are apparent (Reference 1). However, the TiN dispersion seems to be somewhat finer than the more massive dispersions found in directly-aged weldmetal (Reference 1). No evidence of austenitic islands were observed on the fracture surfaces as had been previously found in directly-aged weldmetal. The appearance of the fractures shown in Figures 10A and 10B are consistent with the microstructural observations displayed in Figure 4. Significantly, the fracture surfaces exhibit substantial quantities of TiN and very flat features around the TiN inclusions. Clearly, the nitrides are promoting brittle fracture. The presence of austenite in the directly-aged weldmetal does not appear to be detrimental to fracture toughness. Titanium nitrides, 3.4 Electron Fractography (Cont'd.)

alone, appear to be the principal cause of the inferior fracture toughness of weldmetal.

Additional evidence on the brittle-fracture tendencies of the nitrides was gathered from heat-affected zone and parent plate fractures. Figure 11A shows an extensive flat-fracture region around nitrides on the surface of a heat-affected zone fracture. The dimpled structure shown in Figure 11B, typical of a ductile failure, was much more common on this specimen. Note the virtual absence of nitrides in the dimpled region. Figure 12 shows similar relationships for parent plate. In every instance, the nitrides were observed to promote the formation of localized, flat brittle-type fractures.

4. DISCUSSION AND CONCLUSIONS

The principal finding of this investigation pertains to the relative constancy of fracture toughness for markedly different weldmetal microstructures. Directly-aged weldmetal (i.e., deposited and aged for 3 hours at 900°F; Reference 1 data) was shown to have the same fracture toughness properties as those for austenitized-and-aged weldmetal (i.e., deposited, austenitized for 16 hours at 1800°F, air cooled, and aged for 3 hours at 900°F). The former treatment produced weldmetal containing a semi-continuous network of austenite in an aged martensitic matrix. The latter treatment resulted in a uniform aged martensitic microstructure with no detectable austenite. Both types of microstructures were characterized by numerous TiN inclusions, however, the dispersions appeared to be somewhat different. Electron fractography revealed high concentrations of TiN inclusions on the fracture surfaces of both types of weldmetal. Furthermore, the fracture surface immediately around such inclusions were characteristically flat; quite typical of brittle-type fracture. These results substantially support the conclusions offered in Reference 1: that the nitrides alone are responsible for the impairment of weldmetal toughness. The relative constancy of fracture toughness was previously explained on the basis of the thermal stability of the damaging TiN network. The present work effectively supports that same conclusion. The presence of retained or reverted austenite is apparently not a significant factor at least up to the volume concentrations measured (i.e., about 11%).

The precise mechanisms responsible for TiN embrittlement are open to conjecture. The dispersion of this brittle constituent undoubtedly lowers the strain energy required to create new free surfaces. In addition,

4. <u>DISCUSSION AND CONCLUSIONS</u> (Cont'd.)

the constituent would be expected to behave as an internal stress-raiser which would probably create a multitude of triaxially stressed zones. Such a dispersion would inhibit plastic flow and favor brittle failures. Whatever the mechanism, TiN inclusions are apparently the primary cause of the weldmetal toughness problem.

The observations concerning possible nitride growth or new formation in prior austentic bands is potentially important. These observations suggest the possibility that nitrides may precipitate from nitrogen-enriched austenite. Such a transformation may partially account for the general TiN dispersion observed in the 1800°F austenitized-and-aged weldmetal specimens rather than more clustered aggregates observed in directly-aged weldmetal. The general dispersion may have been fostered by the transformation of austenite in as-deposited weldmetal. Such austenitic regions would tend to be enriched with nitrogen (nitrogen solubility is substantially higher in austenite than in ferrite or martensite.) When the duplex martensitic-austenitic structure is heated to high austenitizing temperatures, nitrogen previously in interstial solution in austenite would tend to diffuse outward to minimize the nitrogen concentration gradient in the matrix. The nitrogen thereby made available could combine with titanium concentrated previously in the martensitic phase (where the solubility of titanium is much higher than in austenite,) and result in the ultimate formation of TiN. Once formed, TiN would not be expected to re-dessolve because of its strongly negative free energy of formation over a very wide temperature range (i.e., extremely high thermal stability). The observation of apparent surface nitride formation in specimens austenitized in air at 2100°F further supports the tentative observation that nitride formation

4. DISCUSSION AND CONCLUSIONS (Cont'd.)

can occur as a result of exposures at austenitic temperatures.

If such a mechanism is operative one might expect additional degradation of toughness under certain combinations of austenitizing time and temperature where the diffusion and solubility conditions could favor heavy nitride precipitation. Interestingly, work reported in Reference I presented an example of weldmetal toughness degradation caused by an austenitizing treatment. Data are insufficient at this time to determine whether or not the degradation resulted from pronounced nitride precipitation or some other cause.

Finally, the incidence of cracking within bands located adjacent to or within weld heat-affected zones may be quite significant. Such cracks had not been observed previously when cross-sections had been carefully examined microscopically or by Magnaflux. Their prevalence at this time, approximately 6 months since the last examinations, suggests the possibility of delayed cracking in banded regions. No definite conclusions may be drawn concerning the mechanisms of formation; however, further investigation of this phenomenon is unquestionably advisable.

5. RECOMMENDATIONS FOR FUTURE WORK

Two areas requiring future work are obvious from the results of this study. First, and foremost, an investigation of nitrogen-free maraging steel plate and weldmetal should be initiated. Overwhelming evidence essentially proves that nitrogen (particularly in the form of TiN) is responsible for damaging toughness degradation in maraging mill products and welds. The maraging alloys will never be used to their full potential until the nitride problems are overcome. Perhaps, development of nitrogen-

5. RECOMMENDATIONS FOR FUTURE WORK (Cont'd.)

free maraging alloys might also lead to their application over much wider temperature ranges than are now permissible (e.g., cryogenic applications).

A second area requiring future work is that pertaining to banding and the possibility of delayed cracking in or near banded heat-affected zone regions. Additional studies should be undertaken to define the exact nature of the problem and to achieve an eventual solution.

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TABLE I

Thermal Treatments Used, Results of X-Ray Diffraction on Austenite Determination, Hardness of Weldmetal and Location of Cracks on Specimens.

Specimen No.	Austenitizing Temperature °F (16 Hours)	Age 900°F (3 Hours)	Austenite Content of Weldmetal % by Vol. By X-Ray Diff.	Hardness Weldmetal (R_C)	Prior Austenitic Grain Size ASTM Number: Plate	Prior Austenitic Grain Size ASTM Number: Weldmetal	Remarks (Section 3.2.4)
1	1500	No		27.8			Crack in HAZ Band
2	1500	Yes	9	49.2			Crack in HAZ Band
3	1500	No		28.8			Crack in HAZ Band
4	1800	Yes	****	51.0	5	6	Crack in HAZ Band
5	2100	No	****	28.1			Crack in HAZ Band
6	2100	Yes		51.2			Crack in HAZ Band
7	2100*	No		22.3			
8	2100*	Yes		47.4			Possible Crack in HAZ Band
9	0	Yes	11		4	7	3 Cracks in Parent Plate Band
10	0	No	9	30.5			
11	1500	No		27.9			2 Cracks in HAZ Band
12	1500	Yes		50.7			
13	1800	No		29.6			3 Cracks in HAZ Band
14	1800	Yes	****	51.2			3 Cracks in HAZ Band
15	2100	No		27.0			2 Cracks in Parent Plate Band
16	2100	Yes		43.4	1+	4	3 Cracks in HAZ Band
17	2100*	No					
18	2100*	Yes		38.8	1+	2	
19	0	No		30.9			
20	0	No	9-11	50.6			5 Cracks in HAZ and Parent Plate Bands
		No					
		Yes					
21	0	Yes		30.6			
22	0	No					
23	0	Yes		49.7			
24	0	Yes		29.5			Crack in HAZ Band
25	0			29.0			
27	2100**	No					
28	2100**	Yes					2 Cracks in Haz Band
29	2100***	No					
30	2100***	Yes					Crack in HAZ Band

* 94 hours, ** 6 hours in vacuum, *** 8 hours in air, **** less than limit of detectability

TABLE II

Fracture Toughness Properties of 18Ni-7Co-5Mo Steel. Parent Plate and Weldments, Austenitized at 1800°F (982°C), Air Cool, Aged 3 Hours at 900°F (482°C)

PARENT PLATE

Specimen No.	Crack Length (inches)	Crack Depth (inches)	Gross Fracture Stress (KSI)	Net Fracture Stress (KSI)	% Elongation (2" gage)
PP-19			246.6		8.0
PP-20	0.050	0.025	260.1	*	
PP-2 [1,2,3]	0.085	0.039	251.3	158.0	
PP-4 [2,3]	0.197	0.075	244.3	258.0	
PP-15 [2,3]	0.272	0.101	197.0	218.0	
PP-17	0.342	0.121	154.1	191.5	
PP-18	0.369	0.127	151.6	193.2	

WELDMENTS

Specimen No.	Crack Length (inches)	Crack Depth (inches)	Gross Fracture Stress (KSI)	Net Fracture Stress (KSI)	% Elongation (2" gage)
4G6[3]			248.5	--	2.0
7A16			249.1	--	BOGM**
7B1			248.6	--	6.0
4E6[3]	0.040	0.020	263.9	*	
4A6	0.072	0.030	258.4	*	
4A4	0.077	0.032	256.3	258.5	
4A10[1]	0.100	0.050	143.6		
4A11	0.097	0.040	240.2	244.0	
4A12	0.125	0.048	215.9	220.0	
4A8	0.151	0.056	167.3	173.5	
A24	0.172	0.064	171.2	178.3	
4A9	0.167	0.069	147.4	153.5	
4A2	0.191	0.072	142.9	150.0	

1 = austenitize only, 2 = prior age, 3 = prior notch

* = failed away from crack

** = broke outside gage marks

HOW MICROSTRUCTURE STUDY SAMPLES WERE REMOVED
FROM WELDED PLATE

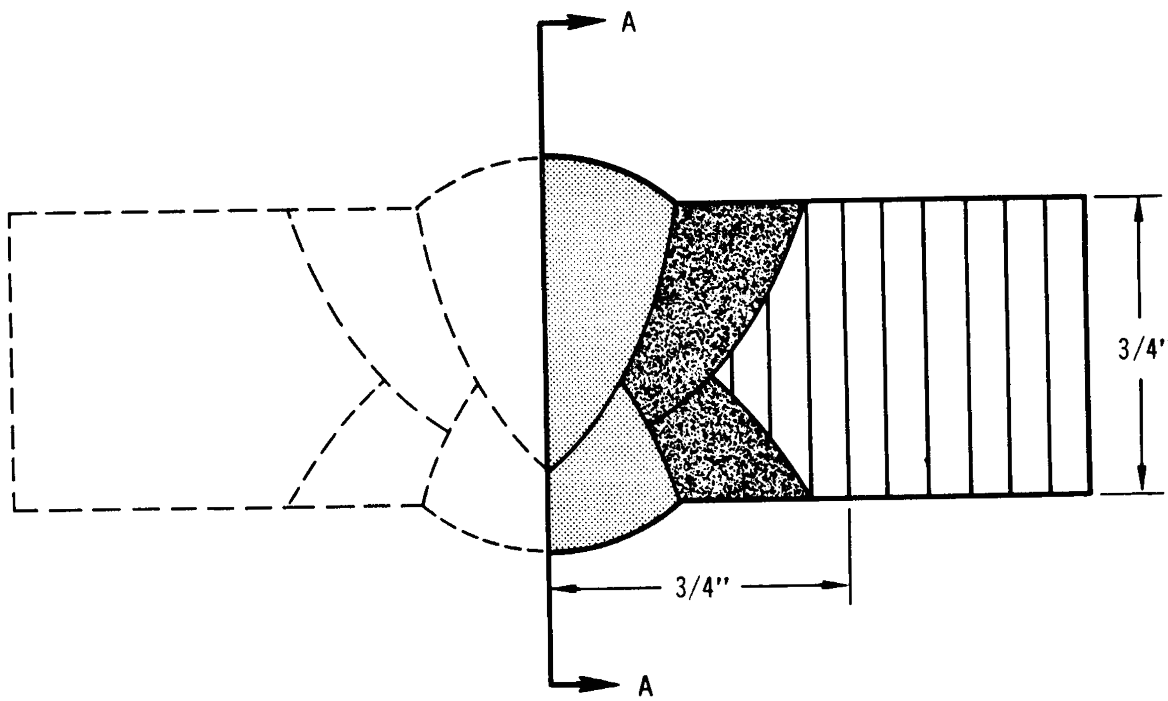
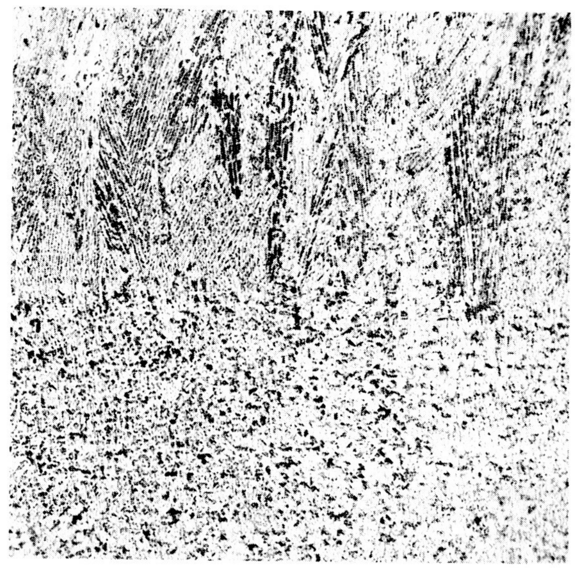


FIGURE 1

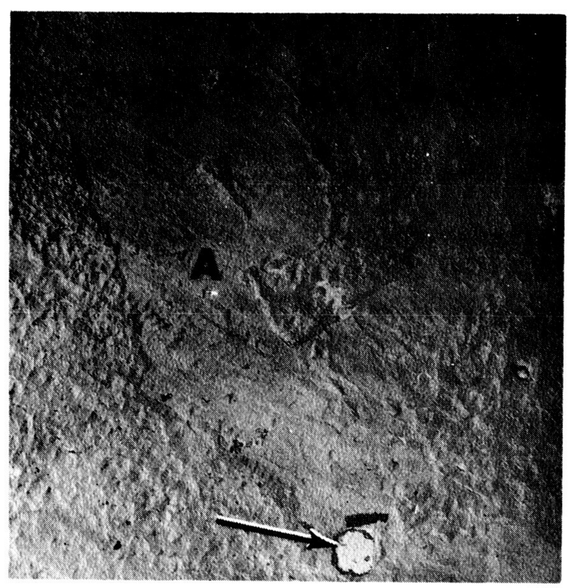
APPEARANCE OF AS DEPOSITED WELDMETAL
ETCHED IN CARAPELLA'S REAGENT



MAGN. 250X M18198

A.

AS DEPOSITED WELDMETAL SHOWING FUSION
LINE BETWEEN PASSES



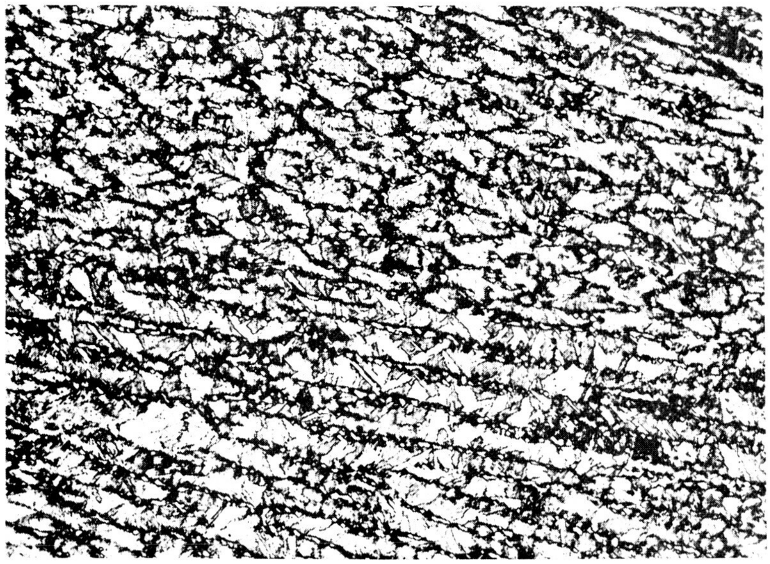
6450X E7716

B

AS DEPOSITED WELDMETAL. A INDICATES POSSIBLE
AUSTENITE IN MARTINSITIC MATRIX. ARROW
INDICATES TiN INCLUSION. PCR Cr @ 45°

FIGURE 2

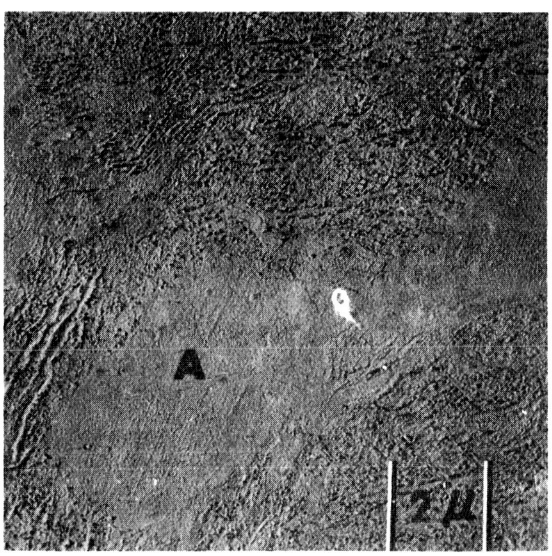
APPEARANCE OF AGED WELD METAL. "A" INDICATES PROBABLE AUSTENITE. ARROW INDICATES TiN INCLUSION. ETCHED IN CARAPELLA'S REAGENT



MAGN 100X M18341

DIRECTLY-AGED WELDMETAL

3A

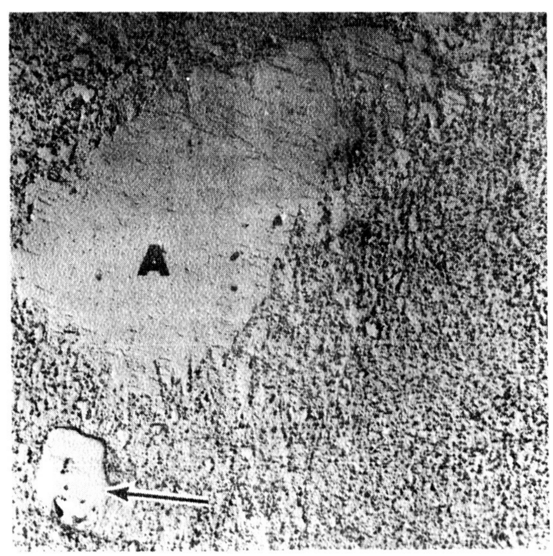


MAGN. 6500X E7898

DIRECTLY-AGED WELDMETAL

PCR Cr @ 45°

3B



MAGN 6500X E7757

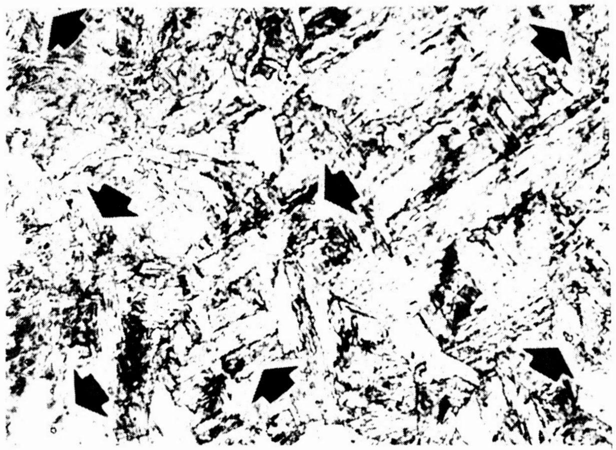
AUSTENITIZED (1500°F, 16 HRS)-AND-AGED (900°F, 3 HRS)

PCR Cr @ 45°

3C

FIGURE 3

APPEARANCE OF WELDMETAL AFTER AUSTENITIZING AT 1800°F FOR 16 HOURS, AGED AT 900°F FOR 3 HOURS. CARAPELLA'S ETCH



MAGN. 250X M18357

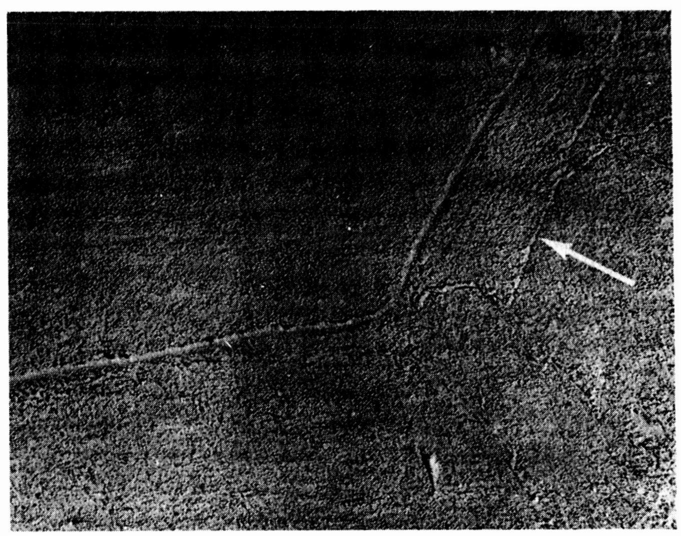
A

ARROWS INDICATE SOME TiN INCLUSIONS

MAGN 6500X E7799

B

TWO INCLUSIONS, PROBABLY TiN
FINE LINES ARE PROBABLY SUB-GRAIN BOUNDARIES. PCR Cr @ 45°



MAGN 6500X E7761

C

SUB-GRAIN BOUNDARY (ARROW) PCR Cr @ 45°

FIGURE 4

APPEARANCE OF BAND IN AGED PARENT PLATE

BAND CONTAINS AGED MARTINSITE (M) AND POSSIBLY AUSTENITE AND/OR FERRITE (A,F) THIS SERIES OF ELECTRON MICROGRAPHS WERE SEPARATED BY THE WIDTH OF THE SCREEN THEREFORE DO NOT MATCH EXACTLY. ETCHED IN CARAPELLA'S REAGENT, PCR Cr @ 45° 6500X.

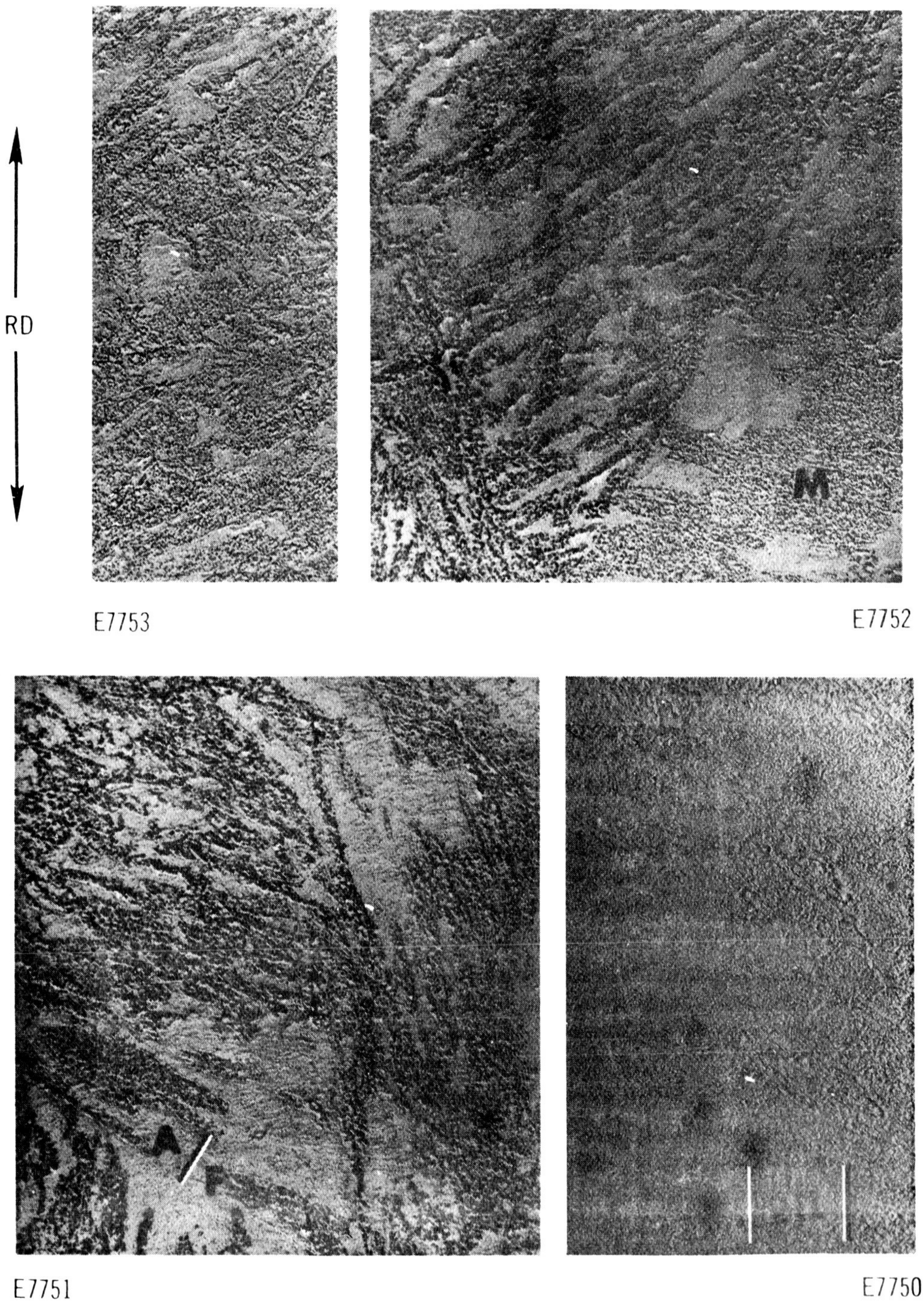
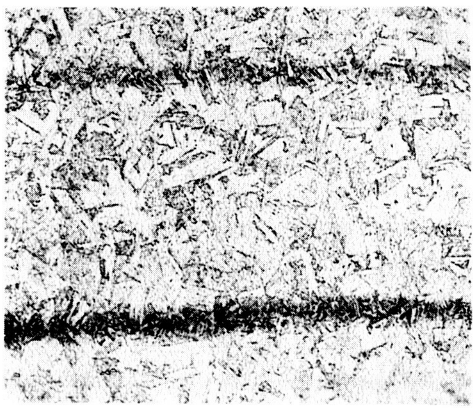


FIGURE 5

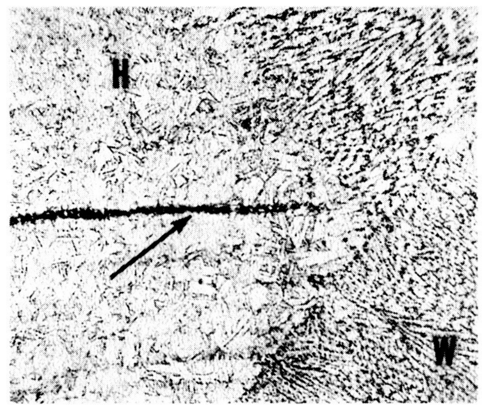
A, B AND C REPRESENT THE DEVELOPMENT OF NITRIDES IN BAND. AUSTENITIZE AT 1800°F FOR 16 HOURS THEN AGED. D AND E SHOW WHERE TYPICAL CRACKS DEVELOPED. CRACK IN HAZ(H). ILLUSTRATION F SHOWS INTERGRANULAR CRACKS ADJACENT TO FUSION LINE.

CARAPELLAS ETCH

(W ≡ WELDMETAL)



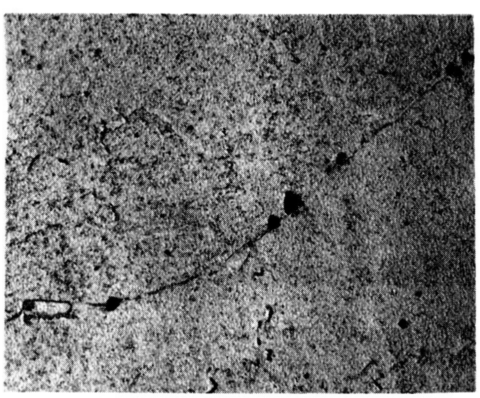
100X A M18525



50X D M18233

250X B M18522

100X E M18225



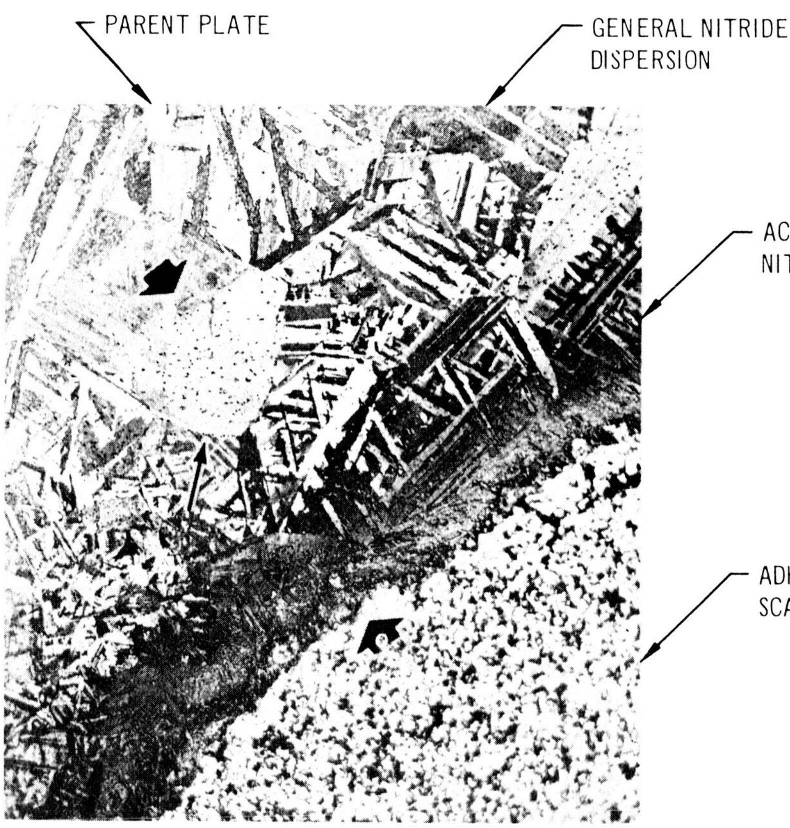
6500X C E7710

DCR CHROME SHADOWED AT 45°

50X F M18235

FIGURE 6

SURFACE NITRIDE FORMATION AFTER AIR EXPOSURE AT 2100°F 96 HOURS, 3 HOURS 900°F. LARGE ARROWS INDICATE AN AREA WHICH SHOWS NITRIDE DISPERSION. SMALL ARROW SHOWS NITRIDE FROMING ALONG A GRAIN BOUNDARY. LOWEST ARROW IS LOCATED ON DEEPEST PENETRATION OF OUTSIDE COATING.



MAGN 350X

M18230

CARAPELLA'S ETCH

FIGURE 7

COMPARISON OF DIRECTLY-AGED WELDMETAL, REFERENCE 1,
WITH WELDMETAL AUSTENITIZED AT 1800°F 16 HOURS,
AIR COOLED THEN AGED (900°F, 3 HOURS)

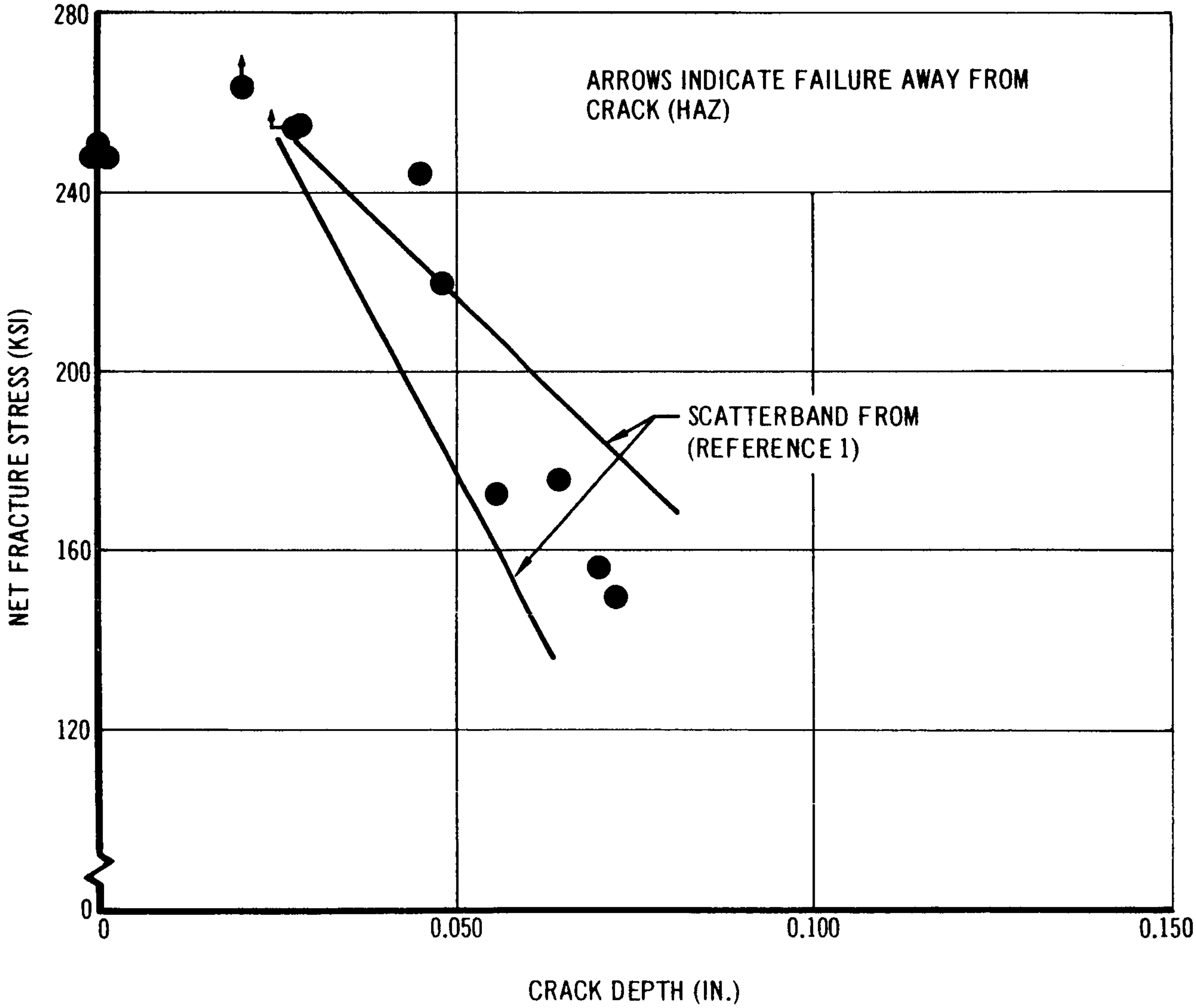


FIGURE 8

COMPARISON OF DIRECTLY-AGED PLATE, REFERENCE 1, WITH PLATE AUSTENITIZED AT 1800°F, 16 HOURS, AIR COOLED THEN AGED (900°F, 3 HOURS)

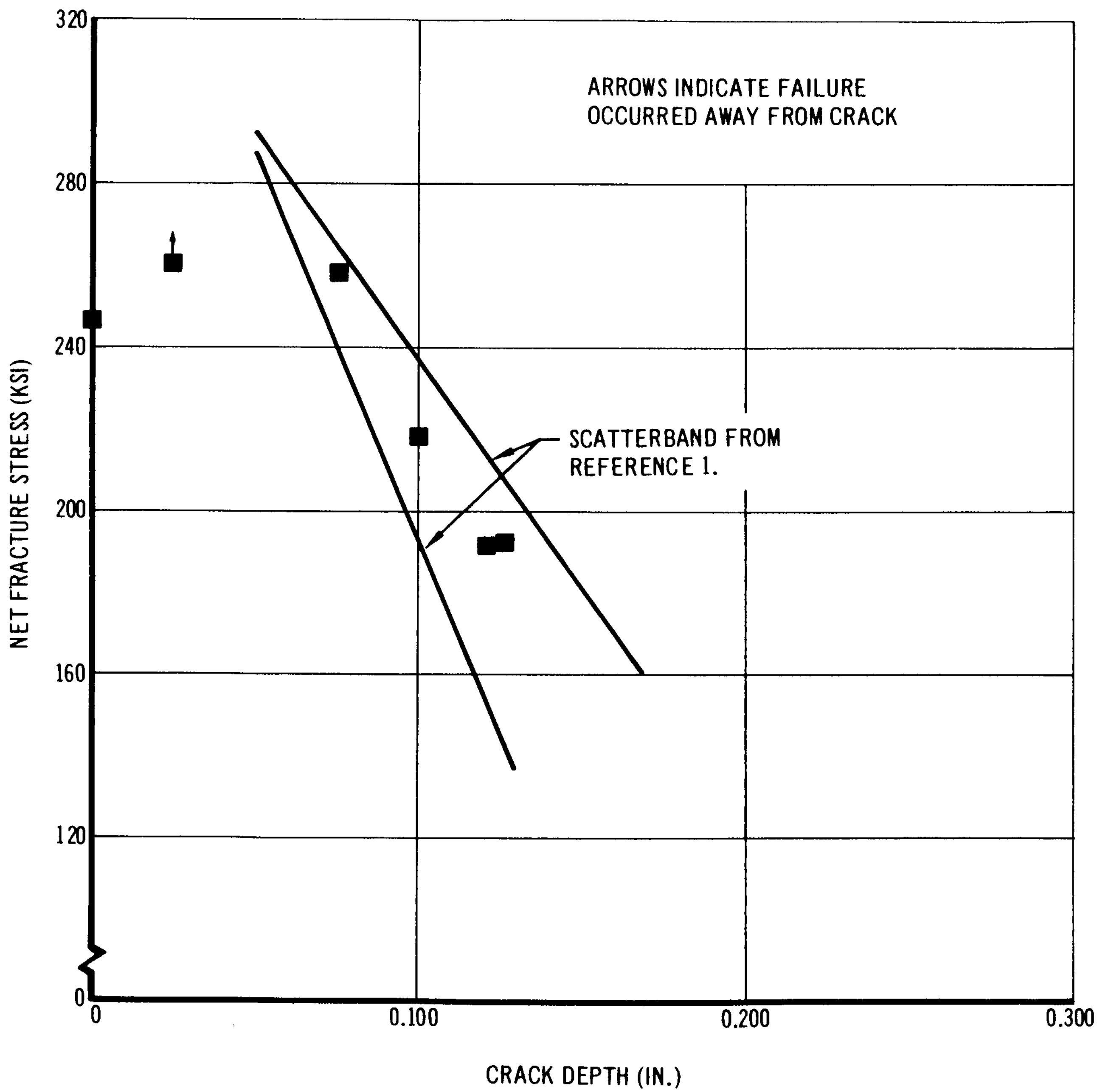
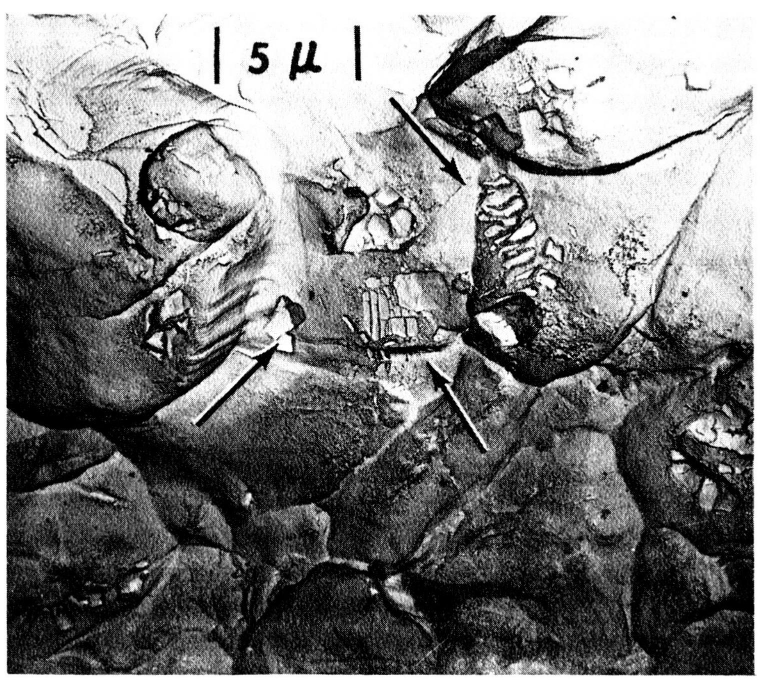


FIGURE 9

FRACTURE SURFACE OF WELDMETAL. AUSTENITIZED 1800°F 16 HOURS, AIR COOLED, AGED 900°F 3 HOURS. ARROWS INDICATE SOME TIN INCLUSIONS. PCR CR AT 45°

CARAPELLA'S ETCH

3800X A E 8016



3800 X B E 8015

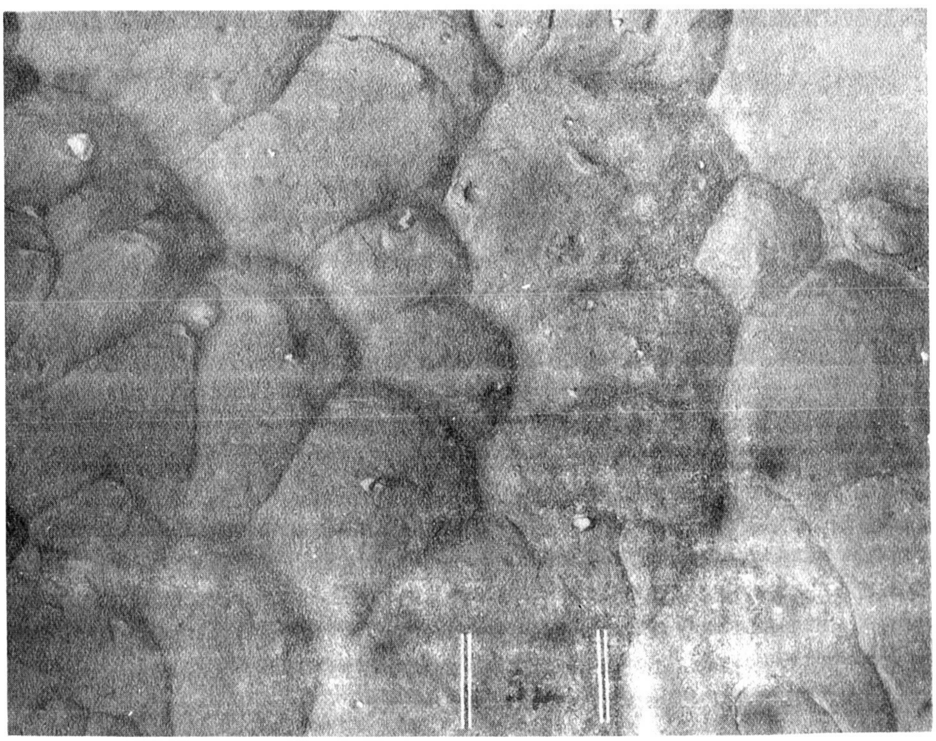
FIGURE 10

FRACTURE SURFACE OF HEAT-AFFECTED-ZONE FAILURE AUSTENITIZE 1800°F 16 HOURS, AIRCOOLED, AGED 900°F 3 HOURS PCR AT 45°

NO ETCH

3790X A E7847

MASSIVE TiN INCLUSION. FLAT, BRITTLE FAILURE

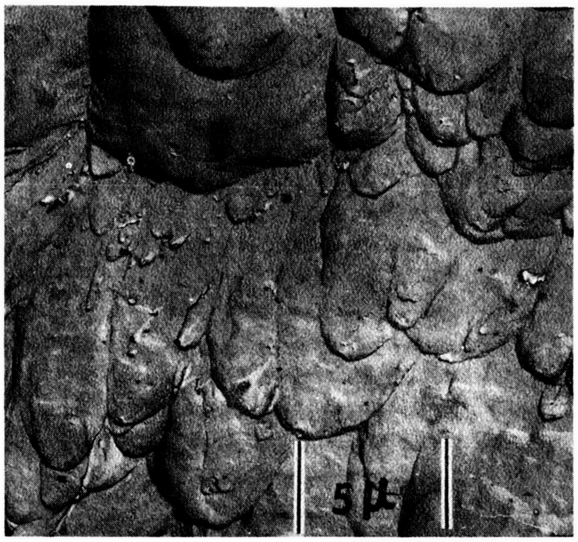


3790X B E7846

DUCTILE FAILURE

FIGURE 11

BRITTLE FRACTURE AREA CAUSED BY NITRIDES (ARROWS) NOTE DIMPLES IN LOWER RIGHT CORNER. AUSTENITIZED 1800°F, 16 HOURS, AIRCOOLED, AGED 900°F 3 HOURS. PCR CR AT 45°
NO ETCH



3790X E7850

DIMPLES INDICATING DUCTILE FAILURE

1930X B E7848

NOTE DIMPLES IN LOWER RIGHT CORNER

FIGURE 12

ERRATA SHEET

DOUGLAS REPORT SM-43105

EVALUATION OF MARAGING STEEL FOR APPLICATION TO SPACE LAUNCH VEHICLES

Page 21 - Table II, Parent Plate

Specimen No.	Crack Length (inches)	Crack Depth (inches)	Gross Fracture Stress (KSI)	Net Fracture Stress (KSI)	% Elongation (2" gage)
PP-2 [1,2,3]	0.085	0.039	251.3	158.0	

Should read:

Specimen No.	Crack Length (inches)	Crack Depth (inches)	Gross Fracture Stress (KSI)	Net Fracture Stress (KSI)	% Elongation (2" gage)
PP-2 [1,2,3]	0.085	0.039	158.0	162.0	

Page 21 - Table II, Weldments

4G6 [3]	--	--	248.5	--	2.0
7A16	--	--	249.1	--	BOGM**
7B1	--	--	248.6	--	6.0

Should read:

4G6 [3]	--	--	259.7	--	2.0
7A16	--	--	262.1	--	BOGM**
7B1	--	--	263.5	--	6.0

(Figures 8 and 9, Pages 29 and 30, should be corrected to reflect these changes for the zero cracked specimens)