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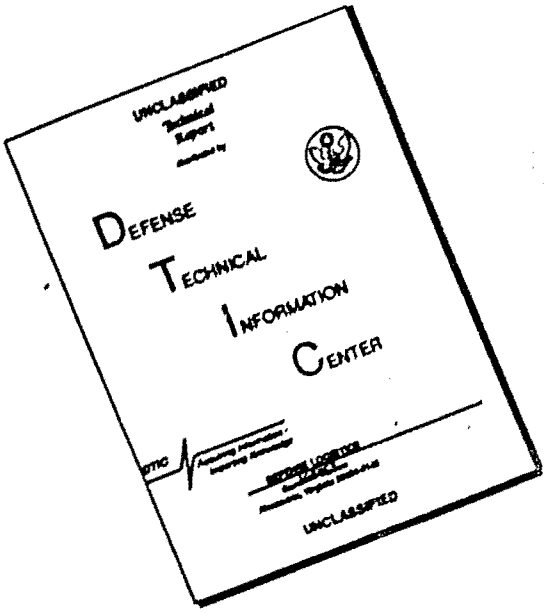

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THE EFFECT OF HEAT TREATMENT ON THE FRACTURE TOUGHNESS AND SUBCRITICAL CRACK GROWTH CHARACTERISTICS OF A 350-GRADE MARAGING STEEL

by

C. S. Carter

ABSTRACT

The heat treatment response of a 350-grade maraging steel, with the nominal composition 18.5 Ni, 12 Co, 4.6 Mo, 1.4 Ti, balance Fe, has been determined in billet and bar form. When aged at temperatures below 900°F, the material was very susceptible to subcritical crack growth, and premature brittle fracture occurred in unnotched tension specimens loaded at a slow strain rate in laboratory air. Fracture mechanics was used to interpret this behavior. The introduction of reverted austenite significantly decreased the strength level but had little effect on fracture toughness. The resistance to brittle fracture of this material is contrasted with that of high-strength steels currently used by the airplane industry.

INTRODUCTION

The continuing demand for ultra-high-strength steels prompted the recent development of two 350-grade maraging steels. The development and properties of one alloy—nominal composition 17.5 Ni, 12.5 Co, 3.8 Mo, 1.7 Ti, 0.15 Al, balance Fe—were recently described (1). Vasco Metals, a subsidiary of Teledyne, Inc., introduced a second alloy that is lower in titanium than the first to avoid segregation problems and has slightly higher nickel and molybdenum contents. In this paper, the effect of heat treatment on the fracture toughness and subcritical crack growth resistance of this second alloy is described.

MATERIAL

Samples from two heats of consumable-electrode, vacuum-remelted, 350-grade maraging steel were supplied by Vasco. The form and chemical analysis of these samples are shown in Table I. Most of this study was conducted on the 4-in.-square billet.

EXPERIMENTAL PROCEDURE

All specimens were machined in the longitudinal direction unless otherwise noted. To determine tensile properties, 0.25-in.-diameter tension specimens were loaded in an Instron machine at a strain rate of 0.005 in./in./min. Fatigue precracked, standard-size Charpy specimens were loaded in three-point bending to determine the plane strain fracture toughness K_{Ic}; the data were analyzed using the recommended ASTM procedure (2).

Similar specimens were used to establish the subcritical crack growth characteristics. These were deadweight loaded in cantilever bending

Table I. Material composition.

Heat	Form	Ni	Co	Mo	Ti	Al	C	S	P	Si	Mn	B	Zr	Ca
03412	4 in. sq. billet	18.61	11.91	4.64	1.36	0.13	0.005	0.005	0.002	0.01	0.01	0.001	0.010	0.05
03261	1-3/4- by 3/4-in. bar	18.48	11.92	4.63	1.48	0.09	0.004	0.004	0.002	0.01	0.03	0.001	0.010	0.05

using the technique described by Brown (3). The environment was either laboratory air or 3.5% aqueous sodium chloride solution, with the latter continuously dripped into the specimen notch. Stress intensity was calculated from the relationship given by Brown and Srawley (4) by assuming that cantilever bending corresponded to three-point loading with a span-to-width ratio of eight. The results of the tests were plotted as the initial applied stress intensity level K_{Ii} versus time to failure, and the plane strain* threshold stress intensity level K_{Iscc}, below which crack growth did not occur, was determined. Fatigue precracked single-edge-notched specimens 7.5 in. long, 1.5 in. wide, and 0.48 in. thick were used to determine the crack velocity characteristics. The crack length was measured optically, using low-power magnification, at selected time intervals. From these observations, a curve of crack length versus time was constructed. The slope of the curve, which represents the instantaneous crack growth rate, was determined graphically for various crack length values; from this, the relation between stress intensity and crack velocity was determined.

To determine the amount of austenite, the X-ray technique of Lindgren was used (5). This technique compares the integrated intensities of the (220) austenite line and the (200) martensite line and is claimed to give an accuracy of 1%.

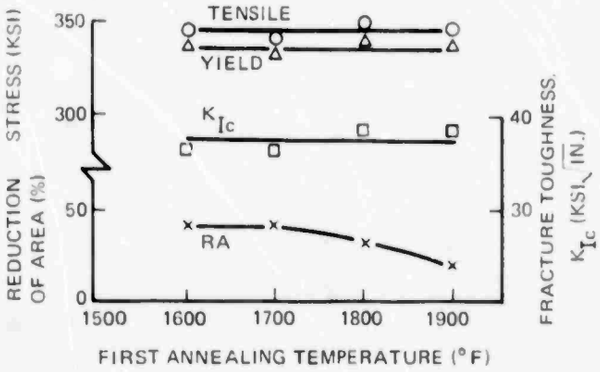


Fig. 1. Effect of first annealing temperature on mechanical properties (second anneal at 1500° F/1 hr and aged 900° F/8 hr).

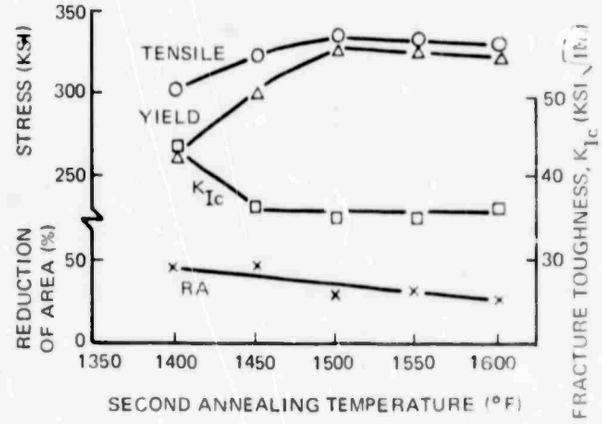


Fig. 2. Effect of second annealing temperature on mechanical properties (first anneal at 1700° F/1 hr and aged 900° F/8 hr).

RESULTS AND DISCUSSION

EFFECT OF SOLUTION ANNEALING TEMPERATURE

The effect of double solution annealing on the properties of the 4-in.-square billet was examined, and the results are shown in Figs. 1 and 2. The first annealing temperature had no significant effect on tensile properties except that the reduction of area was less when the annealing temperature exceeded 1700° F (Fig. 1). This could be associated with grain growth. Fracture toughness K_{Ic}, however, was independent of annealing temperature. As indicated in Fig. 2, the second annealing temperature should exceed 1450° F. Annealing below this temperature gave lower tensile properties since some austenite was present. Therefore, for the remainder of this study, the specimens were double solution annealed at 1700° F for 1 hr followed by 1500° F for 1 hr.

* The specimens were sufficiently thick that plane strain conditions existed (2).

EFFECT OF AGING

The hardness behavior of the 4-in.-square billet aged at various temperatures for times up to 100 hr is shown in Fig. 3. The curves are typical of precipitation hardening alloys, with maximum hardness being achieved after aging at the lower temperature for a longer time.

The effect of aging temperature on the tensile properties and fracture toughness of the same material is shown in Fig. 4. Significant differences in behavior were observed between specimens aged at less than 900° F and those aged at higher temperatures. Because of this, the two temperature regimes are discussed separately below.

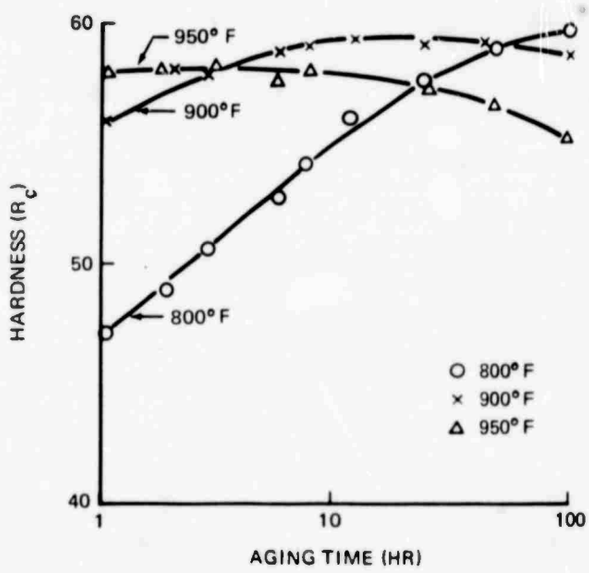


Fig. 3. Effect of aging on hardness (double solution annealed 1700°F/1500°F).

Aging Below 900° F

Premature brittle fracture occurred during loading of the tension specimens aged at 800° F for periods of 3, 6, and 8 hr. Similar behavior was exhibited by the specimens aged at 850° F for 3 and 6 hr but not after aging for 8 hr (Fig. 4). Failure usually occurred in the gage section, normal to the loading axis, but in a few tests a threaded end of the specimen broke. Those that failed in the gage section exhibited a bright, thumbnail-shaped region of fracture at the specimen circumference (Fig. 5). The chevron pattern in the remainder of the fracture face indicated that rapid brittle fracture initiated from this site. The dimensions of this region were measured from enlarged photographs, and a K_{Ic} value was determined for the initiation of brittle fracture using the stress intensity solution for a surface-cracked plate (6). Values for individual specimens are shown in Table II. A similar region was also observed at the thread roots in specimens that failed at the ends; K_{Ic} values were not determined for these specimens. This behavior did not depend on the position from which the specimens were removed from the billet and was also observed in similarly heat treated specimens taken from the 1-3/4-in. by 3/4-in. bar.

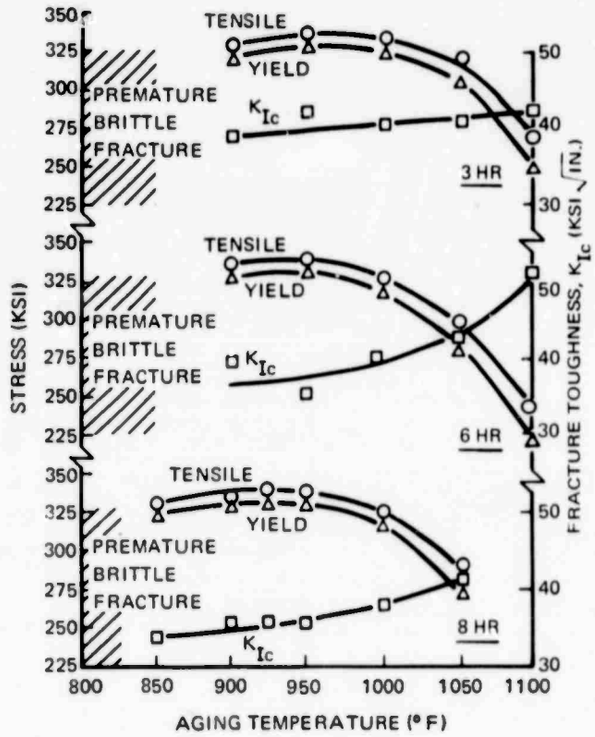
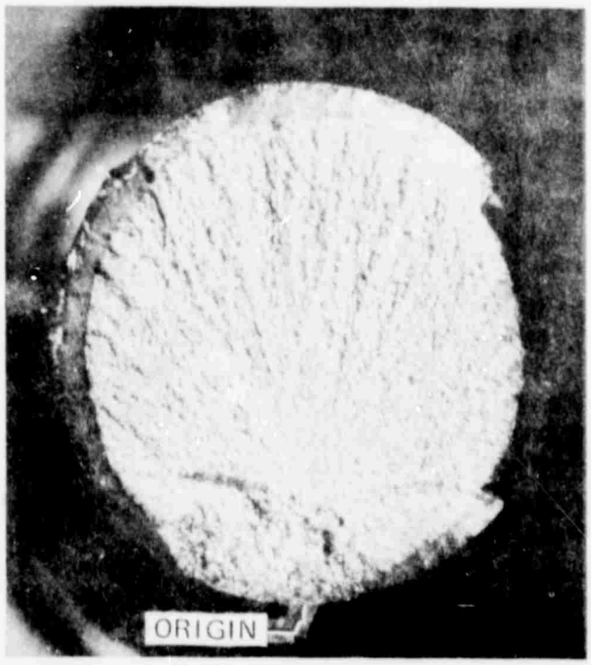


Fig. 4. Effect of aging on mechanical properties (double solution annealed 1700°F/1500°F).



X 12

Fig. 5. Tension specimen that prematurely failed in laboratory air (aged 850° F/3 hr).

This suggested that the thumbnail region was due to subcritical crack growth during the rising load tension test and not to preexisting defects. Tension specimens that had been aged at either 800° F for 8 hr or 850° F for 3 hr were therefore tested in two additional ways. In one test series, the specimens were rapidly tension loaded to failure in laboratory air at a crosshead travel speed of 2 in./min as compared with 0.005 in./min in the tests discussed above. All the specimens tested in this way broke in a ductile manner with a cup-and-cone-type fracture (Table III), thus confirming that the cracks were not preexisting but only developed after loading for a definite period of time. In the second series of tests, 0.4 in. of the 1.25-in.-long gage section of the tension specimens were surrounded with Seattle tap water and the specimens tension loaded to failure at a crosshead travel speed of 0.005 in./min. Premature brittle fracture occurred in the section exposed to tap water at a similar stress level and in an identical manner to the specimens tested in air. These results, included in

Table II. Fracture data for prematurely failed tension specimens.

Aging treatment	Environment	Fracture stress (ksi)	Critical crack size (in.)		K_{Ic} (ksi$\sqrt{in.}$)
			Depth	Length	
800° F/6 hr	Laboratory air	215.1	0.047	0.107	64.2
800° F/8 hr	Laboratory air	232.1	0.030	0.075	58.0
		247.0	0.045	0.120	79.8
		223.8	0.068	0.150	80.4
	Tap water	238.6	0.034	0.082	62.3
					(mean 70.1)
800° F/16 hr	Laboratory air	238.7	0.010	0.054	43.7
850° F/3 hr	Laboratory air	291.0	0.030	0.070	71.0
		215.0	0.051	0.102	62.6
	Tap water	259.0	0.021	0.055	60.0
					(mean 64.5)

Table III. Mechanical properties of tension specimens tested at a fast strain rate.

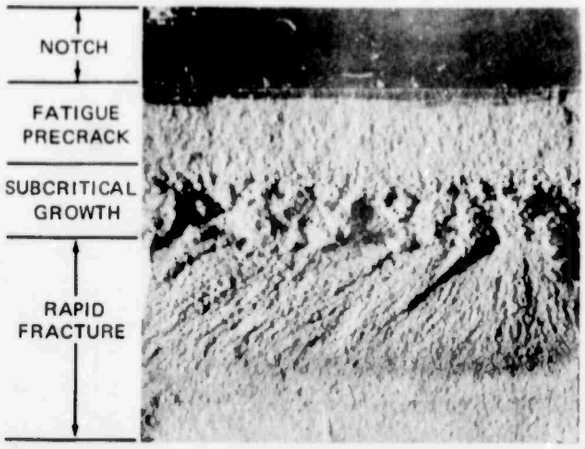
Aging treatment	Yield strength (ksi)	Ultimate tensile strength (ksi)	Elongation (%)	Reduction of area (%)
800°F/8 hr	299.0	302.3	9.6	45.7
850°F/3 hr	267.4	290.7	11.1	52.9

Table II, suggested that water (or water vapor in the air) was involved in the crack extension process.

Microexamination of sections taken from specimens exposed to both environments (air and water) revealed that the thumbnail regions had extended in a predominantly intergranular manner. In some specimens, secondary intergranular cracks extended parallel to the loading axis along dark etching bands, indicating that these discontinuities had very low crack growth resistance. Previous studies have shown that these bands are associated with nickel, molybdenum, and titanium segregation although the cause of the dark etching appearance is not known (7).

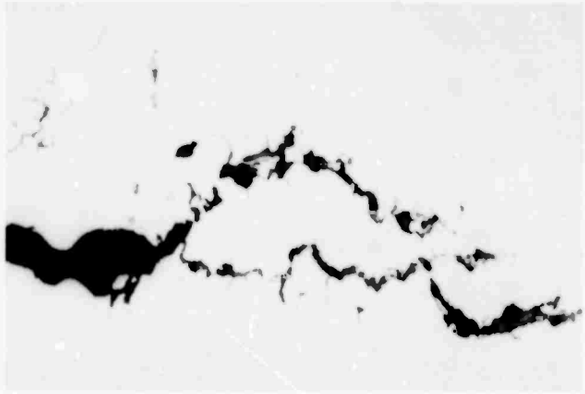
An identical phenomenon was observed in the fracture toughness tests. Subcritical crack growth commenced during loading, and the crack extended until rapid failure occurred at maximum load (Fig. 6). This was eliminated by increasing the loading rate (crosshead speed increased from 0.05 in./min to 2 in./min). In specimens exhibiting crack growth, K_{Ic} was computed from the maximum load and the distance from the surface containing the notch to the position where subcritical crack growth terminated. Values determined in this way were significantly higher than the K_{Ic} obtained by rapid loading (Table IV). This was unlikely to be a strain rate effect because other investigations have shown that the fracture toughness of maraging steel is essentially independent of strain rate (8).

Metallographic examination of the subcritical crack growth region revealed an intergranular mode of cracking with the cracks tending to branch on a microscopic scale (Fig. 7). Possibly these microscopic branch cracks were sufficient to dissipate the applied load and effectively blunt



X 6

Fig. 6. Subcritical crack growth in a notched bend specimen tested under rising load in laboratory air (aged 800° F/3 hr).



X 430

Fig. 7. Microscopic crack branching in a notched bend specimen tested under rising load in laboratory air (aged 800° F/8 hr).

[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
800° F/8 hr	Slow[a]	52.7, 49.8 (mean 51.2)	Crack growth
	Fast[b]	30.8	No crack growth
800° F/16 hr	Slow[a]	42.6	Crack growth
850° F/3 hr	Slow[a]	49.8, 48.4, 72.0, 84.8 (mean 63.8)	Crack growth
	Fast[b]	30.5	No crack growth

[a]Crosshead speed 0.05 in./min [b]Crosshead speed 2 in./min

the crack tip. This would be most apparent in brittle materials with small plastic zones. In tougher materials, the larger plastic zone would override the influence of the crack morphology. A comparison of Tables II and IV indicates that the K_{Ic} values obtained from the tension and slowly loaded notched bend specimens were similar. There was some scatter in both test series, which is attributed to variations in crack morphology.

To quantitatively measure the resistance to crack growth, precracked Charpy specimens were cantilever loaded to selected stress intensity levels in laboratory air and the time to failure established. The results, shown in Fig. 8, indicate that crack growth did not occur below a stress intensity level of 15 ksi$\sqrt{\text{in.}}$ This information can be used to explain the mechanics of fracture in the tension specimens.

To estimate the depth of flaw required for cracking during tension loading, a typical fracture stress value of 230 ksi (Table II) and a threshold value of 15 ksi$\sqrt{\text{in.}}$ were substituted into the stress intensity equation for a surface crack (6). This led to a critical crack depth of 0.001 in., for a crack with a length-to-depth ratio of 10. However, the fracture stress was used rather than the lower (unknown) stress at which crack growth initiated, so the actual critical depth would be less than 0.001 in.

As discussed previously, the dark etching bands were very susceptible to crack growth. Since these bands were oriented parallel to the loading axis, and their thickness was typically 0.0005 to 0.002 in., they probably provided the initiation site. The short failure times of the bend specimens loaded to stress intensity levels above 20 ksi$\sqrt{\text{in.}}$ indicate a negligible incubation period and rapid crack propagation, thus explaining the extent of crack growth observed in the tension specimens (Table II).

Resistance to crack growth was similarly determined in 3-1/2% aqueous sodium chloride solution (Fig. 8). Threshold stress intensity was

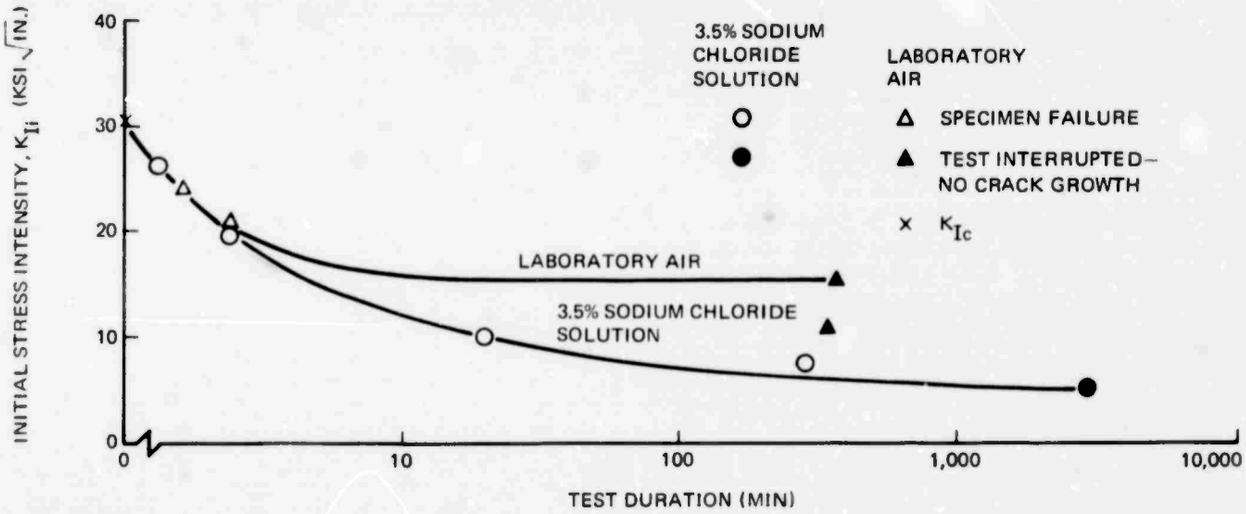


Fig. 8. Subcritical crack growth resistance (aged 800° F/8 hr).

reduced to 5 ksi $\sqrt{in.}$ in this environment although the times to failure at stress intensities exceeding 20 ksi $\sqrt{in.}$ were similar to those recorded in air. Crack growth measurements revealed that crack velocity was directly proportional to applied stress intensity (Fig. 9).

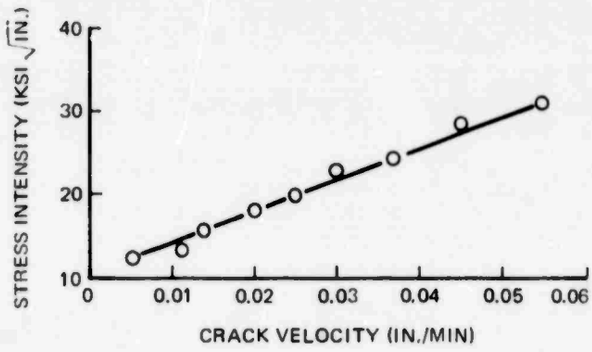


Fig. 9. Relationship between stress intensity and crack velocity (aged 800° F/8 hr).

The response to variations in strain rate and the intergranular mode of fracture suggest that a hydrogen embrittlement mechanism was responsible for the subcritical crack growth. Hydrogen could have entered the steel during processing or as a result of crack tip corrosion reactions with water (e.g., water vapor in the air). The actual source has not been definitely established, but three observations indicate a corrosion reaction. First, the cracks invariably initiated at the tension specimen surface, suggesting that access to the environment was required. Second, the tension specimens fractured in the limited region exposed to tap water. Finally, aging at 800° to 850° F might be expected to "bake out" hydrogen remaining from earlier processing.

The detrimental effect of aging at temperatures less than 900° F also is reflected in other grades of maraging steel. Brisbane et al. (9) reported that the delayed failure resistance of precracked sheets of 300-grade maraging steel, tension loaded in distilled water, was reduced by lowering the aging temperature from 900° to 750° or 800° F. Schapiro (10) reported that the threshold value of 200-grade maraging steel aged at 840° F for 4 hr was 48 ksi $\sqrt{in.}$ in sea water, whereas, values exceeding 100 ksi $\sqrt{in.}$ have been obtained for similar material aged at 900° F(11).

This indicates that some critical change in the precipitation hardening reaction in maraging steels occurs at 850° F. Peters (12) has suggested that below 850° F there are two forms of precipitate, a dislocation nucleated precipitate

and a matrix precipitate. At higher temperatures, precipitation occurs only at dislocations. Such changes could significantly affect the deformation processes or the availability of sites for crack nucleation within the crack tip plastic zone.

Aging Above 850° F

Peak strength was achieved by aging at 925° F for 8 hr. However, the 350-ksi ultimate tensile strength level was not achieved; typical values obtained by aging at 900° to 925° F for 8 hr were within the range 335 to 347 ksi (Figs. 1 and 4). The fracture toughness K_{Ic} for this strength range was 35 to 40 ksi $\sqrt{in.}$

Aging at temperatures above 1000° F led to a marked reduction in tensile properties (Fig. 4). The amount of reverted austenite retained after aging at various temperatures for 3 hr is shown in Fig. 10, and comparison with Fig. 4 shows the decrease in strength could be associated with reverted austenite; however, some of the austenite formed during aging may have transformed to virgin (unaged) martensite upon cooling to room temperature and would also contribute to the observed strength reduction. Unfortunately, the reverted austenite only slightly improved the fracture toughness (Fig. 11). This may be contrasted with martensitic stainless steel in which retained austenite significantly improves fracture toughness (13). This steel is austenitized and incompletely transformed to martensite, so that retained austenite is dispersed between the martensite plates. On the other hand, maraging steel reversion commences at the martensite platelet boundaries, and a lamellar-type structure is subsequently formed (14). It would appear that this structure is a much less effective crack arrestor than the stainless steel with its intimate dispersion of austenite.

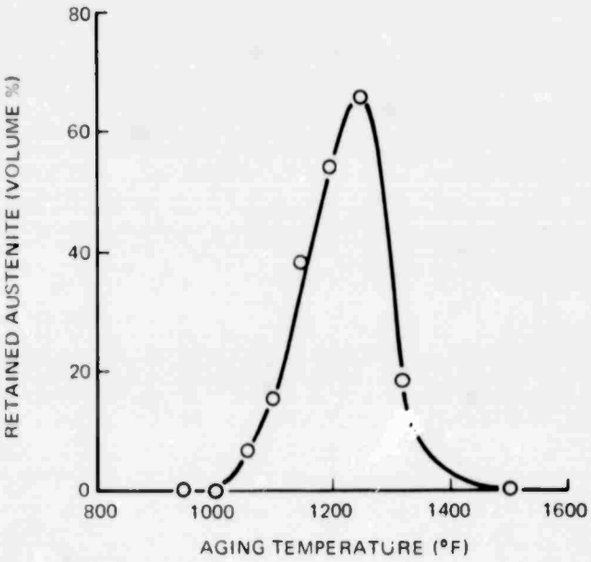


Fig. 10. Austenite retained at room temperature after aging for 3 hr (bar material).

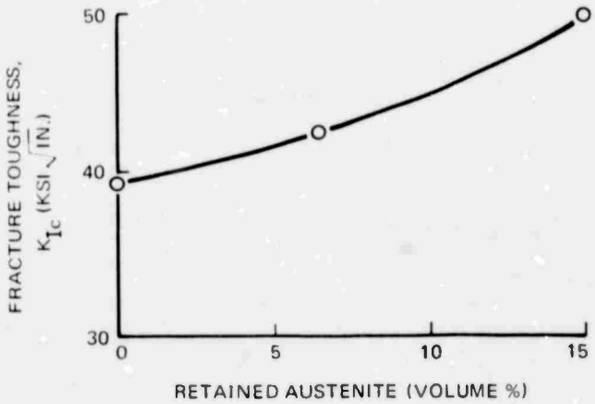


Fig. 11. Influence of retained austenite on fracture toughness.

Fracture toughness tests were conducted on specimens aged at 900° F for 8 hr either before or after an aging treatment at 800° F for 8 hr. In both cases, there was no evidence of subcritical crack growth, and the fracture toughness was typical of a single 900° F age. Therefore, crack-growth-susceptible microstructures, which could be present in the heat-affected zones of welds in solution-annealed material, will be removed upon aging to peak strength.

The crack growth resistance of 900° and 950° F aged specimens, statically loaded in 3.5% aqueous sodium chloride solution, is shown in Fig. 12. The threshold stress intensity was 10 ksi$\sqrt{in.}$ and the crack velocity was constant (independent of stress intensity) at approximately 0.0006 in./min, for both heat treatment conditions. Both the threshold level and the time to failure at a given initial stress intensity were increased relative to the 800° F aged condition, which is shown for comparison in Fig. 12. It was not apparent why the crack velocity changed from a stress intensity dependancy to a constant, and much slower, rate when the aging temperature was increased from 800° to 900° or 950° F; the fracture mode was intergranular for all heat treatments.

The time to failure at a given initial stress intensity level was increased by raising the aging temperature from 900° to 950° F. This can be attributed to an increase in the incubation time for crack initiation since the crack velocity and fracture toughness (hence the critical crack length for ligament rupture) were similar for both heat treatment conditions. It has been proposed that the incubation period can be explained as either the time for the environment to permeate a crack tip film or diffusion of the active species to a critical site in the crack tip region (15). The latter seems to be unlikely because the crack velocities were identical for both heat treatment conditions and, therefore, suggests the crack-tip film was more impervious in the 950° F aged condition.

The cracks were extensively branched on a macroscopic scale in both heat treatment conditions (Fig. 13). This can be contrasted with 800° F aged specimens in which there was no evidence of macroscopic branching and the crack velocity was directly proportional to stress intensity. As discussed elsewhere, branching only occurs when the crack velocity is essentially constant and a critical stress intensity level is attained (16).

PROPERTIES OF BAR MATERIAL

A similar study on 1-3/4- by 3/4-in. bar material yielded almost identical results to those discussed above for the 4-in. billet. In particular, stable crack growth was observed after aging below 900° F, and the 350-ksi strength level could not

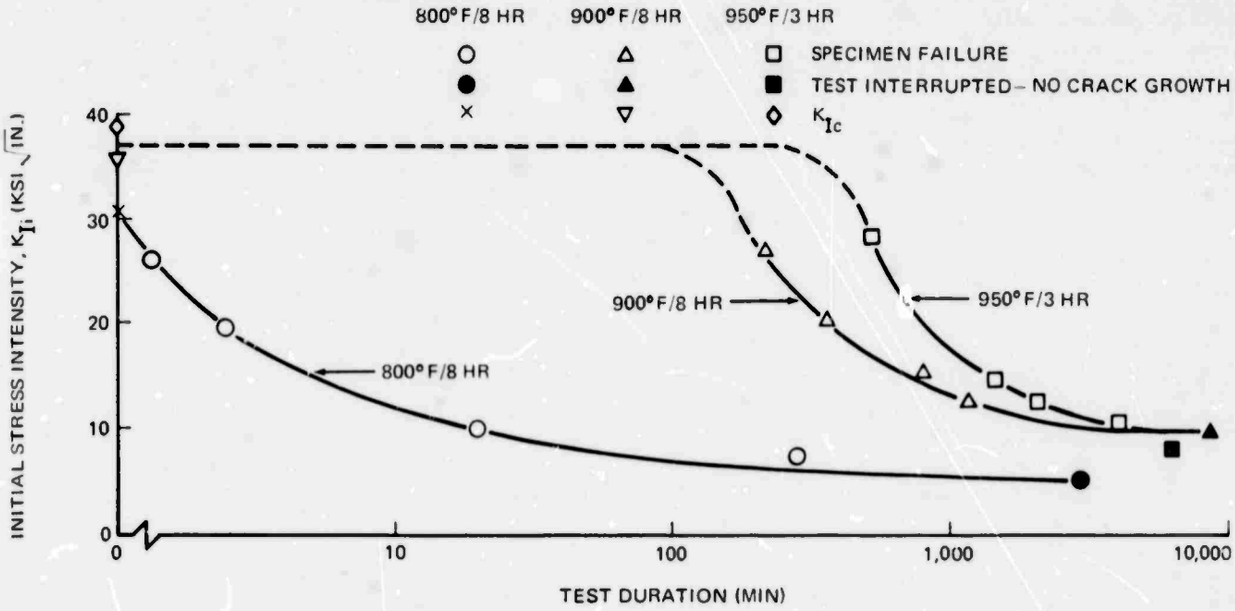
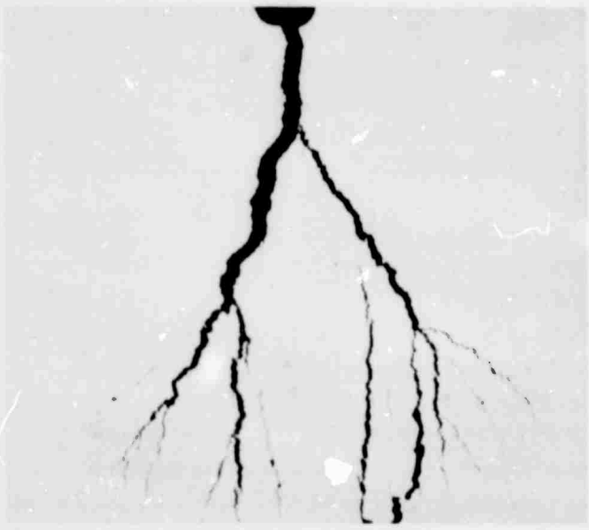


Fig. 12. Effect of aging treatment on subcritical crack growth resistance in 3.5% aqueous sodium chloride solution.



X 10

Fig. 13. Macroscopic crack branching in notched bend specimen loaded to K_{Ii} = 27 ksi$\sqrt{in.}$ in 3.5% aqueous sodium chloride solution (aged 900°F/8 hr).

be achieved. Therefore, the individual results are not recorded here, but the relationship between strength and K_{Ic} for material aged at temperatures in the range 900° to 1100°F is shown in Fig. 14 for both billet and bar material. The poor tradeoff between decreasing strength and toughness is apparent and emphasizes the ineffectiveness of reverted austenite as a crack stopper.

EFFECT OF SPECIMEN ORIENTATION

The results obtained from tension specimens machined in the transverse direction from the midsection of the billet are compared with typical longitudinal properties in Table V. Low elongation and reduction of area characterize the transverse properties. Examination of the broken specimens revealed that the fracture path was associated with the dark etching bands described earlier (Fig. 15). However, the K_{Ic} values were similar in the longitudinal (RT) and transverse (TW and TR) directions. Apparently the notch/precrack plane was not aligned with the arrays of dark etching bands, consequently, the toughness of the matrix was measured. These observations may be contrasted with those made by Salmon Cox et al. (7), which showed that banding in 250-grade maraging steel plate could be detected by fracture toughness tests.

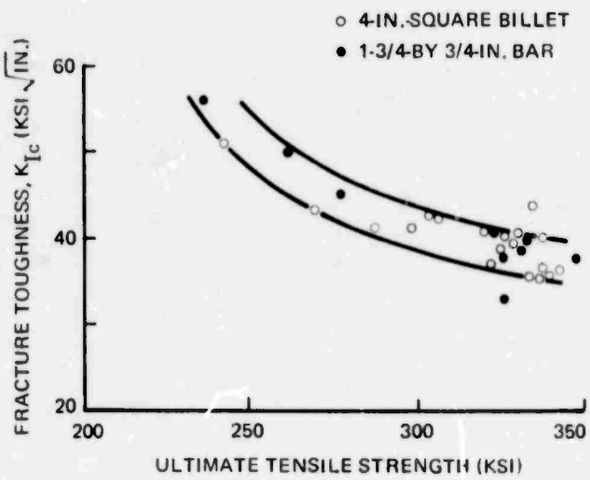


Fig. 14. Relation between strength and toughness for bar and billet (aged at various temperatures above 850°F).

Table V. Influence of specimen orientation[a].

Orientation	Yield strength (ksi)	Ultimate tensile strength (ksi)	Elongation (%)	Reduction of area (%)	K_{Ic} (ksi$\sqrt{in.}$)
Transverse	337.9	346.9	1.8	5.5	36.9 (TW) 36.9 (TR)
Longitudinal	334.4	342.3	6.8	33.6	36.1 (RT)

[a]Data are mean of duplicate tests on specimens aged at 900°F for 8 hr.

X 100

Fig. 15. Section through fracture face of transverse tension specimen showing fracture along dark etching bands.

Because of the heavier reduction in the plate, the bands would be more closely spaced than in the billet and, hence, a greater probability of notch alignment existed. Also, the stress field at the tip of a precrack in the matrix could be about 10 times more extensive in the tougher 250-grade material than in the 350 grade. This would be more likely to cause fracture along neighboring discontinuities. These observations point out that the conventional tension test has a distinct advantage over notch-type tests in the quality control testing (i.e., detection of discontinuities) of thick sections of relatively brittle materials.

EFFECT OF TITANIUM CONTENT IN MARAGING STEELS

In the tensile strength range 180 to 350 ksi, the strength level of maraging steels is primarily increased by raising the titanium content. The effect of this element is shown in Fig. 16 using typical values of strength and titanium content for each grade. It is apparent that titanium is much less effective in strengthening when the content exceeds 1%, particularly when it is remembered that the cobalt content of the 350 grade is approximately 4% higher than in the other grades.

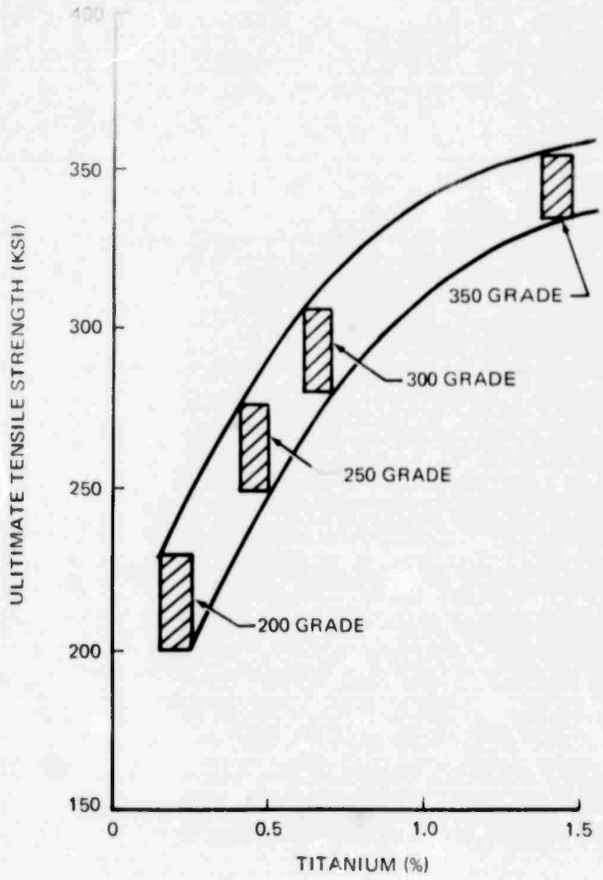


Fig. 16. Influence of titanium content on ultimate tensile strength.

The relationship between strength and fracture toughness (Fig. 17) indicates that K_{Ic} rapidly decreases with strength level in the range 300 to 350 ksi. This can be associated with the high titanium contents (1.0% to 1.5%) in this strength range because, as shown in Fig. 18, the fracture toughness is inversely related to the titanium content.

STRUCTURAL APPLICATION CONSIDERATIONS

Brittle fracture resistance is a primary consideration in the application of ultra-high-strength steels. Using fracture mechanics analyses, an

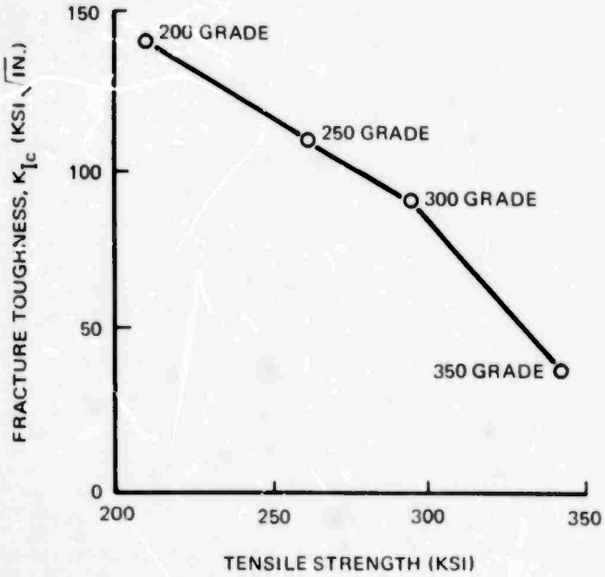


Fig. 17. Relationship between tensile strength (typical) and fracture toughness (optimum).

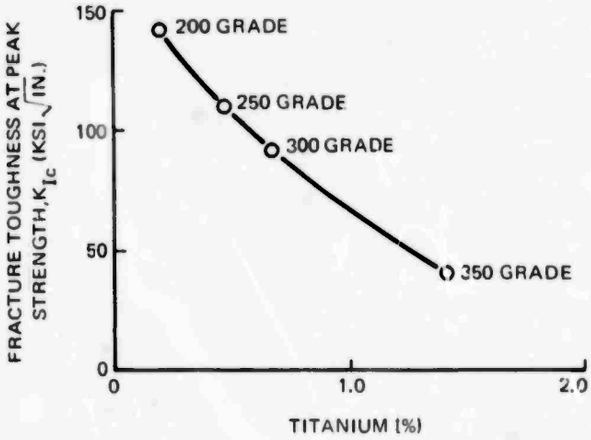


Fig. 18. Influence of titanium content (typical) on fracture toughness (optimum) values.

estimate can be made of the critical crack size as a function of applied stress. Such a plot is shown in Fig. 19 for 350-grade maraging steel (at maximum strength) and compared with similar relationships

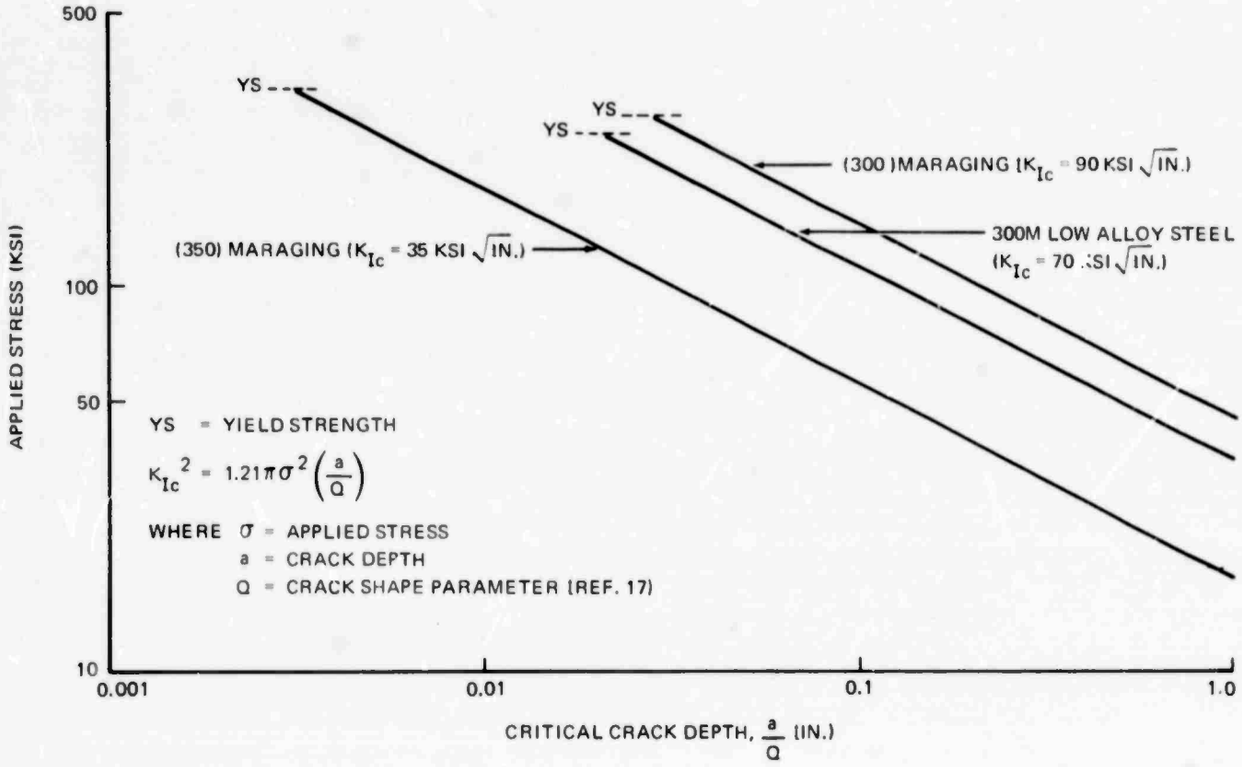


Fig. 19. Comparison between critical crack sizes in 350-grade maraging steel and other high-strength steels.

for 300-grade maraging and 300M low-alloy steels heat treated to the 280 ksi ultimate tensile strength level. For a given applied stress level, the critical crack size is significantly less than the lower strength steels. Furthermore, it must be remembered that design stresses are often a function of ultimate tensile strength. Allowing for such an increase in applied stress indicates that the critical crack size in 350-grade steel will be approximately five times less than in 300M (a widely used landing gear alloy). This does not of course mean that brittle fracture will occur in structural components made from this maraging steel, but this significant decrease in critical crack size must be carefully considered in relation to the structural weight saving offered.

CONCLUSIONS

1. The 350-grade maraging steel is particularly susceptible to subcritical crack growth in air when aged at temperatures below 900°F; this has been attributed to a hydrogen embrittlement mechanism. Aging at 900° and 950°F improved threshold stress intensity and changed the crack velocity from a stress intensity dependency to a constant, and much slower, rate. Reduced crack-growth resistance in the low-temperature-aged condition appears to be a characteristic of the 18% Ni maraging system.

2. The plane strain fracture toughness of 350-grade maraging steel, and hence the critical crack size for the initiation of brittle fracture, is significantly lower than the high-strength steels currently used by the aerospace industry.

3. Reverted austenite significantly decreased tensile strength but had little influence on fracture toughness.

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13. ABSTRACT

The heat treatment response of a 350-grade maraging steel, with the nominal composition 18.5 Ni, 12 Co, 4.6 Mo, 1.4 Ti, balance Fe, has been determined in billet and bar form. When aged at temperatures below 900° F, the material was very susceptible to subcritical crack growth, and premature brittle fracture occurred in unnotched tension specimens loaded at a slow strain rate in laboratory air. Fracture mechanics was used to interpret this behavior. The introduction of reverted austenite significantly decreased the strength level but had little effect on fracture toughness. The resistance to brittle fracture of this material is contrasted with that of high-strength steels currently used by the airplane industry.

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