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TITLE: Fracture Toughness and Stress Corrosion Characteristics of
High Strength Maraging Steel

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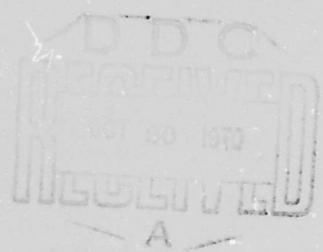
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BOEING | NO. D6-25459
PAGE 1



LIST OF ACTIVE PAGES

SECTION	PAGE NUMBER	REV SYM	ADDED PAGES						SECTION	PAGE NUMBER	REV SYM	ADDED PAGES					
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	1								26								
	2								27								
	3								28								
	4								29								
	5								30								
	6								31								
	7								32								
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REV SYM

BOEING NO. D6-25459
PAGE 3

→
6-7000

TABLE OF CONTENTS

	<u>Page</u>
List of Tables	5
List of Figures	6
Abstract	7
1.0 INTRODUCTION	8
2.0 ALLOY CONTENT	8
3.0 EXPERIMENTAL PROCEDURE	9
4.0 RESULTS	10
4.1 Effect of Solution Annealing	10
4.2 Effect of Aging Treatment	10
4.2.1 Tensile and Fracture Toughness Properties	10
4.2.2 Stress Corrosion Properties	12
5.0 DISCUSSION	14
6.0 CONCLUSIONS	16
7.0 REFERENCES	18

AD 1546 D

REV SYM

BOEING | NO. D6-25459
PAGE 4



LIST OF TABLES

<u>TABLE</u>		Page
I	CHEMICAL COMPOSITION (Wt. %)	20
II	MECHANICAL AND FRACTURE TOUGHNESS PROPERTIES	20
III	STRESS CORROSION CRACK VELOCITY DATA	21

AD 1548 D

REV SYM

BOEING | NO. D6-25459
PAGE 5



LIST OF FIGURES

<u>FIGURE</u>		<u>PAGE</u>
1	EFFECT OF SOLUTION TREATMENT (AGED 900°F/8HR)	22
1(a)	EFFECT OF FIRST SOLUTION ANNEALING TEMPERATURE (SECOND SOLUTION ANNEAL AT 1500°F)	22
1(b)	EFFECT OF SECOND SOLUTION ANNEALING TEMPERATURE (FIRST SOLUTION ANNEAL AT 1700°F)	22
2	EFFECT OF AGING FOR 3 HOURS	23
3	EFFECT OF AGING FOR 8 HOURS	24
4	INFLUENCE OF AGING TIME AT 900°F	25
5	RELATIONSHIP BETWEEN TENSILE STRENGTH AND TOUGHNESS	25
6	RELATIONSHIP BETWEEN AUSTENITE CONTENT AND PROPERTIES	26
7	EFFECT OF INTERRUPTED QUENCHING ON PROPERTIES	27
8	EFFECT OF AGING ON STRESS CORROSION PROPERTIES	28
9	EFFECT OF AGING TEMPERATURE ON STRESS CORROSION CRACK VELOCITY	29
10	RELATIONSHIP BETWEEN YIELD STRENGTH AND STRESS CORROSION CRACK VELOCITY	30
11	FRACTURE TOUGHNESS OF HIGH STRENGTH MARAGING STEELS	31
12	COMPARISON OF STRESS CORROSION CRACK VELOCITIES IN MARAGING AND LOW ALLOY STEELS	32

AD 1546 D



ABSTRACT

The effect of heat treatment on the tensile, fracture toughness, and stress corrosion properties of a high strength maraging steel (nominal composition 16.3 Ni-12.87 Co-4.98 Mo-0.78 Ti) is described. A maximum ultimate tensile strength of 323 ksi, combined with a fracture toughness K_{Ic} of 62 ksi $\sqrt{\text{in}}$, was achieved. This strength level appears to be the maximum which can be achieved in maraging type steels without decreasing the crack tolerance below that of currently used high strength low alloy steels. Reversion to austenite did not improve either the fracture toughness or stress corrosion resistance relative to completely martensitic microstructures with equivalent strength.

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REV SYM

BOEING

NO. D6-25459

PAGE 7



6-7000

1.0 INTRODUCTION

Since their introduction in 1960 the 18% Ni maraging steels have been increasingly used for a variety of aerospace and other applications. The commercially available 200, 250, and 300 grades contain 17-19% Ni, 7-9 Co., 3-5 Mo and titanium within the range 0.15 - 0.80 depending upon the strength-grade required⁽¹⁾. The highest strength which can be guaranteed for the 300 grade in heavy section form is 280 ksi. While higher strength grades have been developed^(2,3), the fracture toughness of these alloys has limited their structural application. The purpose of this study was to explore the feasibility of increasing the minimum tensile strength of 300 grade maraging steel by modifying the alloy content while maintaining an acceptable level of fracture toughness. In addition the effect of heat treatment on fracture toughness and stress corrosion was investigated.

2.0 ALLOY CONTENT

Previous studies have shown that titanium has a potent hardening effect on 18% Ni maraging steel (approximately 90 ksi/1% addition) but has an adverse effect on fracture toughness⁽¹⁾. Therefore, the titanium content in this program was restricted to the upper end of the 300 grade range; i.e., 0.8%. To achieve the desired strength the cobalt content was increased from 8.5 - 9.5% to 12-13%, thereby enhancing the age hardening response⁽⁴⁾. To maintain the Ms temperature at a similar level to the 300 grade (300-350°F) the nickel content was decreased by 2%.

A 200 lb. heat was vacuum induction melted by Carpenter Technology Corporation to this analysis and then remelted under vacuum into an

* Alloy content is given as wt.%

AD 1346 D

8 inch diameter ingot. The ingot was press forged to 5 inch square, then to 2-3/4 inch square, and finally to 1 inch square. Chemical analysis of the heat is shown in Table I, the composition being essentially that aimed for.

3.0 EXPERIMENTAL PROCEDURE

Tension specimens 0.25 in. diameter were machined from the 1" square bar. After heat treatment the specimens were tensile tested at room temperature at a strain rate of 0.005 in/in/min through to yield and 0.02 in/in/min. to fracture.

Single edge notch specimens (0.5 in. thick, 1.0 in. wide, 7.0 in. long) were also machined from the bar, and fatigue precracked after heat treatment. These were loaded in 3 point bending according to the ASTM recommended practice for plane strain fracture toughness testing⁽⁵⁾, and the results analyzed according to this procedure.

Standard size (0.394 in. square) Charpy specimens were used to determine the stress corrosion properties. These were fatigue precracked after heat treatment; the maximum stress intensity imposed during fatigue cracking was estimated to be less than $30 \text{ ksi}\sqrt{\text{in}}$. The specimens were subsequently loaded in cantilever bending to selected initial stress intensity levels using a hydraulic loading system. The stress intensity was calculated according to the relationship given by Brown and Srawley⁽⁶⁾ for pure bending. A supply of 3-1/2% aqueous sodium chloride was dripped into the notch, delivery being commenced just prior to load application. Specimens were exposed until either failure occurred or a minimum period of 100 hours elapsed. If fracture did not occur the specimens were rapidly broken open in laboratory air and

AD 1546 D

macroscopically examined for evidence of crack growth.

Austenite contents were measured by X-ray diffraction using the technique described by Lingren (7).

4.0 RESULTS

4.1 Effect of Solution Annealing

The effect of double solution annealing (1 hr + 1 hr) was investigated, followed by an aging treatment of 900°F/8hr. Results are shown in Fig. 1. Increasing the first annealing temperature slightly reduced the tensile properties but had no effect on the plane strain fracture toughness K_{Ic} . Optimum properties were achieved with a second solution annealing temperature of 1500°F; other temperatures decreased the tensile strength properties although there was no effect on fracture toughness. In view of these results all further tests were conducted after double solution annealing: 1700°F (1 hr) + 1500°F (1 hr).

4.2 Effect of Aging Treatment

4.2.1 Tensile and Fracture Toughness Properties

The effect of aging temperature and time was initially examined using single specimens for both fracture toughness and tensile properties. Results of aging at temperatures within the range 800-1100°F for periods of 3 hr. and 8 hr. are shown in Figures 2 and 3, respectively. Tensile strength response was typical of age hardening type materials. For the 3 hr. aging period a peak tensile strength of 307 ksi was obtained after heat treatment at 950°F. After aging at 900°F for 8 hr. the strength increased to 318 ksi. Extending the aging period at 900°F to 16 hr. increased the tensile strength by 5 ksi (Fig. 4). These results were confirmed by triplicate tests on specimens aged at 900°F for 8 hrs. and 16 hrs. (Table II).

AD 1546 D

Subcritical crack growth occurred during the rising load fracture toughness tests on specimens aged at 800°F (3 hr. and 8 hrs) and 850°F (3 hrs). Cracking occurred in an intergranular manner and extensive branching occurred on a macroscopic scale. Because the branch cracks dissipate the applied load, a K_{Ic} value (for a single crack) could not be determined⁽⁸⁾. Similar behavior was reported for 350 grade maraging steel heat treated and tested in an identical manner⁽³⁾. This was attributed to a stress corrosion - hydrogen embrittlement crack growth mechanism, laboratory air being the corrosive mechanism. A similar mechanism would appear to explain the sub-critical crack growth discussed above. This phenomena was not observed when specimens were aged at temperatures of 900°F and higher.

As shown in Figs. 2 and 3 the fracture toughness increased with aging temperature. Aging at 850°F (8 hrs.) led to lower fracture toughness than at 900°F despite the 20 ksi greater tensile strength at the higher aging temperature. There was no significant effect of aging time at 900°F on the fracture toughness although the tensile strength was increased by 25 ksi when the time was increased from 3 hrs. to 16 hrs. (Fig. 4). This was confirmed by additional fracture tests on specimens aged for 8 hrs. and 16 hrs. (Table II). The relationship between strength and toughness is shown in Fig. 5. Aging at temperatures above 900°F gave the best combination of strength and toughness although maximum strength necessitated aging at 900°F.

The relationship between mechanical properties and retained austenite content is shown in Fig. 6. It is apparent that the decrease in strength following aging at temperatures exceeding 900°F can be associated with austenite reversion.

AD 1546 D

Fracture toughness increased with austenite content but the combination of strength and toughness was no better than can be achieved with other grades of maraging steel having a completely martensitic structure and fully age hardened to comparable strength. For example, the fracture toughness of 250 grade maraging steel is 80-100 ksi $\sqrt{\text{in}}$ combined with a tensile strength of approximately 260 ksi ⁽¹⁾. Thus, the deliberate reversion to austenite is not beneficial with regard to strength/toughness.

To investigate the influence of a duplex microstructure of martensite and unaged martensite on strength/toughness the following heat treatment study was performed. Single edge notch specimens were quenched from a solution annealing temperature of 1500°F to selected temperatures which encompassed the Ms-Mf temperature range. This provided a structure of martensite, and untransformed austenite. Specimens were held for 2 hrs. and then reheated to 900°F and held for 8 hrs. to age the martensite. Air cooling to room temperature transformed the residual austenite to martensite (unaged). The fracture toughness values obtained and the corresponding hardness values are shown in Fig. 7. An increase in toughness was only obtained at the expense of marked decrease in hardness and hence in strength. The strength/toughness combination obtained is no better than can be achieved from the commercial grades of maraging steel aged to peak strength in a conventional way.

4.2.2 Stress Corrosion Properties

Stress corrosion curves of initial stress intensity K_{Ic} versus time to failure are shown in Fig. 8 for four aging treatments. The

AD 1546 D

threshold stress intensity K_{Isc} below which stress corrosion cracking did not occur was approximately $10 \text{ ksi}\sqrt{\text{in}}$ for all heat treatment conditions except for the $850^\circ\text{F}/3 \text{ hr.}$ treatment which gave a K_{Isc} of less than $10 \text{ ksi}\sqrt{\text{in}}$. However, the time to failure at a given initial stress intensity level increased with aging temperature.

The stress corrosion curves show that the time to failure was essentially independent of initial stress intensity for K_{Ii} levels exceeding a value approximately $15 \text{ ksi}\sqrt{\text{in}}$ for the 850°F and 900°F aging treatments. This value increased to $20\text{-}25 \text{ ksi}\sqrt{\text{in}}$ at higher aging temperatures. Previous studies have shown that this time independent behavior can be correlated with an essentially constant rate of crack growth⁽⁸⁾. Also extensive crack branching was observed in all specimens; a constant crack velocity is an essential requirement for the occurrence of this phenomena. Since visual observation showed that the period for crack initiation was small compared to the total time to failure, an estimate of the stress corrosion crack velocity (for the stress intensity range from 15 or $20 \text{ ksi}\sqrt{\text{in}}$ to K_{Ic}) could be obtained by dividing the stress corrosion crack length by the time to failure. Velocities obtained in this way are presented in Table III, and summarized in Fig. 9. Velocity decreased by almost two orders of magnitude as the aging temperature increased from 850°F to 1050°F . This can be contrasted with the K_{Isc} parameter which was essentially independent of heat treatment. Therefore the increase in failure time with aging temperature noted above is due to the accompanying increase in K_{Ic} (Fig. 2,3), which dictates larger critical crack lengths for mechanical fracture, and the decrease in crack velocity.

AD 1546 D

The velocity data is compared with that reported for other grades of 18% Ni maraging steels in the stress intensity independent regime of growth in Fig. 10. It appears that there is an approximately linear relationship between the logarithm of crack velocity and yield strength when the aging temperature is not less than 900°F. It is interesting to note that the velocity estimated for the 1050°F/3hr. age condition containing 18% austenite is similar to that for a completely martensitic 250 grade maraging steel (aged 900°F/3hr) at a comparable strength level. On the other hand, the K_{Isc} associated with this 250 grade steel was 40 ksi $\sqrt{\text{in}}$ compared to 10 ksi $\sqrt{\text{in}}$ for the overaged steel. Thus, reverted austenite can be seen to have no beneficial effect on stress corrosion resistance when strength level is taken into account.

5.0 DISCUSSION

The relationship between strength and fracture toughness for various maraging steels with strength levels exceeding 300 ksi is shown in Fig. 11. The major question to be answered is what is the minimum toughness level required. Estimates of this can be made by means of fracture mechanics. Using the maximum stress to be applied in service and inspection capability for crack detection the minimum toughness required to avoid brittle fracture can be estimated. Alternatively, we can consider maintaining the critical flaw size at the same level as in currently used high-strength low alloy steels. The conditions for brittle fracture can be written as:

$$K_{Ic} = \sigma \sqrt{\pi c} \cdot S$$

where σ = Applied Stress (1)

C = Critical Crack Length

S = A factor depending on crack shape and loading conditions

Since design stresses are frequently based on the ultimate tensile strength (σ_u) the applied stress can be expressed as

$$\sigma = \sigma_u \cdot D \quad (2)$$

where: D is a design factor.

Thus eq. (1) can be written as:

$$K_{Ic} = \sigma_u \sqrt{\pi c} \cdot D \cdot S \quad (3)$$

It can be seen from equation (3) that the fracture toughness must increase in direct proportion to the tensile strength to maintain the same critical crack size. The alloy 4340 has been widely used for airplane landing gear and other airframe applications at the 260-280 ksi strength level. At this strength level the plane strain fracture toughness is approximately $50 \text{ ksi}\sqrt{\text{in}}$ (10). Using the above approach the toughness required to maintain the critical flaw size at the same level as in 4340 can be determined. This minimum toughness level is compared with the maraging steel data in Fig. 11 and shows that the critical flaw size in the experimental maraging steel described in this report is maintained at the same level as in the 4340 steel in spite of the 60 ksi increase in tensile strength level. However, the crack tolerance of the higher strength maraging grades (350 and 400) is lower than that of 4340. Thus, 320 ksi appears to be the maximum tensile strength which can be achieved in the maraging system if crack resistance is to be maintained at least the same level as in low alloy steels.

AD 1546 D

Stress corrosion crack growth in low alloy high strength steels is stress intensity independent over a wide range of stress intensity in most heat treatment conditions⁽¹¹⁾. Such velocities for a number of these steels are compared with the maraging steel data in Fig. 12. At equivalent strength levels the stress corrosion crack velocity in maraging steel is slower than in the low alloy steels. Comparison with the 4340 steel data shows an order of magnitude difference. Wei and Landes⁽¹⁶⁾ have suggested that fatigue crack growth in an aggressive environment can be estimated by adding the sustained load influence of the environment to the fatigue crack growth rate in an inert environment on a cyclic basis. The fatigue crack growth rate characteristics of high strength low alloy and 18 Ni (400) maraging steels are reported to be similar in an inert environment⁽¹⁷⁾. Therefore, the Wei-Landes hypothesis combined with the data shown in Fig. 11 suggests that fatigue crack growth resistance of maraging steels in a sodium chloride environment will be significantly better than in low alloy steels of similar strength at low cyclic frequencies.

6.0 CONCLUSIONS

1. An ultimate tensile strength level of 320 ksi can be achieved in a maraging steel containing 16.3 Ni-12.87 Co-4.98 Mo - 0.78 Ti. The associated plane strain fracture toughness K_{Ic} was 62 ksi $\sqrt{\text{in}}$. It is shown that this combination of strength and toughness provides a similar resistance to brittle fracture as 4340 steel heat treated to a 260 ksi tensile strength level. Increasing the strength of maraging steels to higher levels results in a lower crack tolerance than in 4340.

AD 1546 D



2. Although reverted austenite reduced the strength, the fracture toughness and stress corrosion resistance were no greater than other grades of 18% Ni maraging steel in the fully martensitic condition at comparable strength levels.
3. A duplex microstructure of aged and unaged martensite did not offer any benefit with respect to strength/toughness.
4. Increasing the aging temperature from 850 to 1050°F significantly decreased the stress corrosion crack velocity in the region of stress intensity independent crack growth but had an insignificant effect on the threshold stress intensity K_{Isc} .
5. At equivalent strength levels the stress corrosion crack velocity in the stress intensity independent region is approximately an order of magnitude less in maraging steels than in 4340 type steels.

AD 1546 D

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BOEING | NO. D6-25459
PAGE 17

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7.0 REFERENCES

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AD 1546 D

REV SYM

BOEING

NO. D6-25459

PAGE 19



6-7000

TABLE I - CHEMICAL COMPOSITION (Wt.%)

Ni	Co	Mo	Ti	C	Mn	Si	P	S	B	O (ppm)	N (ppm)
16.30	12.87	4.98	0.78	0.004	0.01	0.01	0.005	0.003	0.0018	14	10

TABLE II - MECHANICAL AND FRACTURE TOUGHNESS PROPERTIES

Aging Treatment	Yield Strength (ksi)	Ultimate Strength (ksi)	Reduction of Area (%)	Fracture Toughness K_{Ic} ksi \sqrt{in}
<u>900°F/8 hr.</u>	315.8	321.8	N.M.	61.5
	309.6	312.6	60	63.0
	316.3	320.4	60	63.5
	(313.9)	(318.3)	(60)	(62.7)
<u>900°F/16 hr.</u>	316.2	325.4	N.M.	59.1
	317.0	321.6	55	62.2
	320.8	322.8	57	65.7
	(318.0)	(323.8)	(56)	(62.3)

Note: 1. NM - Not Measured
 2. () - Indicates Average

AD 1546 D

TABLE III - STRESS CORROSION CRACK VELOCITY DATA

Aging Treatment	Initial Stress Intensity (ksi $\sqrt{\text{in}}$)	Crack Velocity (in/min)
<u>850°F/3hr.</u>	14.2	1.2×10^{-3}
	25.0	1.6×10^{-3}
	40.0	<u>1.5×10^{-3}</u>
		(1.4×10^{-3})
<u>900°F/8hr.</u>	25.0	1.2×10^{-4}
	35.0	5.4×10^{-4}
	50.0	<u>2.2×10^{-4}</u>
		(2.9×10^{-4})
<u>900°F/16 hr.</u>	14.7	2.5×10^{-4}
	23.5	<u>2.8×10^{-4}</u>
		(2.7×10^{-4})
<u>1050°F/3hr.</u>	25.0	8.8×10^{-5}
	38.7	6.3×10^{-5}
	58.3	5.7×10^{-5}
	80.0	<u>6.3×10^{-5}</u>
		(6.9×10^{-5})

Note: () Indicates Average

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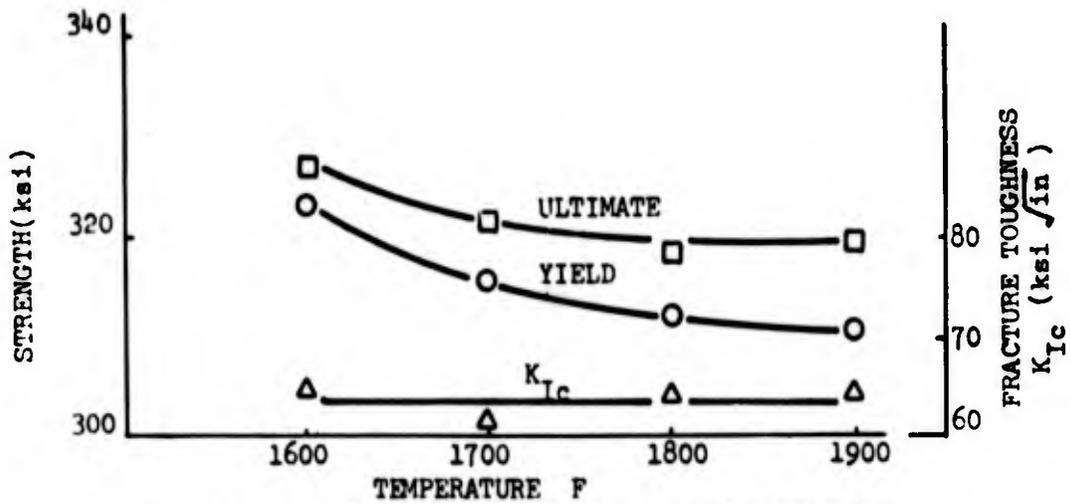
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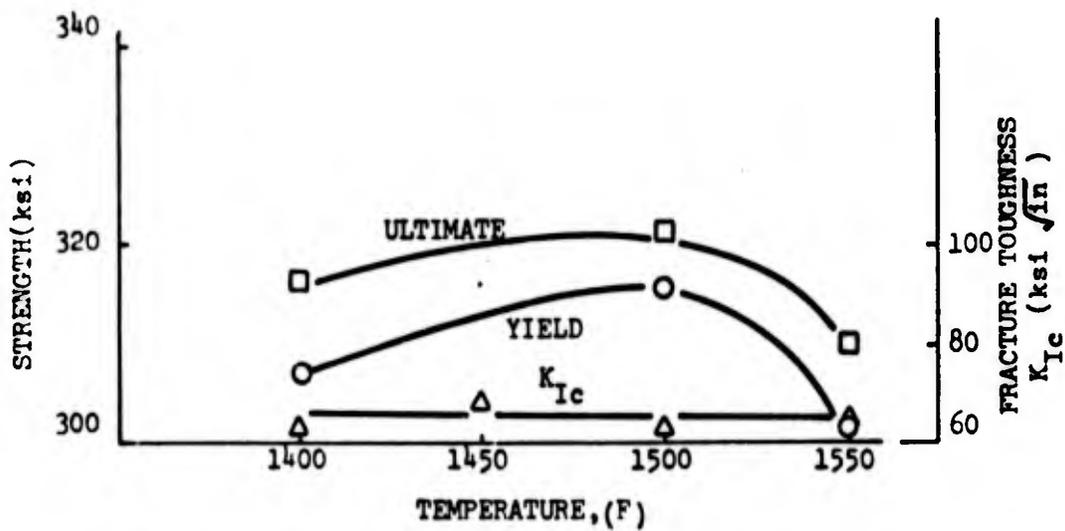
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(a) EFFECT OF FIRST SOLUTION ANNEALING TEMPERATURE
(SECOND SOLUTION ANNEAL AT 1500°F)



(b) EFFECT OF SECOND SOLUTION ANNEALING TEMPERATURE
(FIRST SOLUTION ANNEAL AT 1700°F)

Figure 1 - EFFECT OF SOLUTION TREATMENT (AGED 900°F/8HR)

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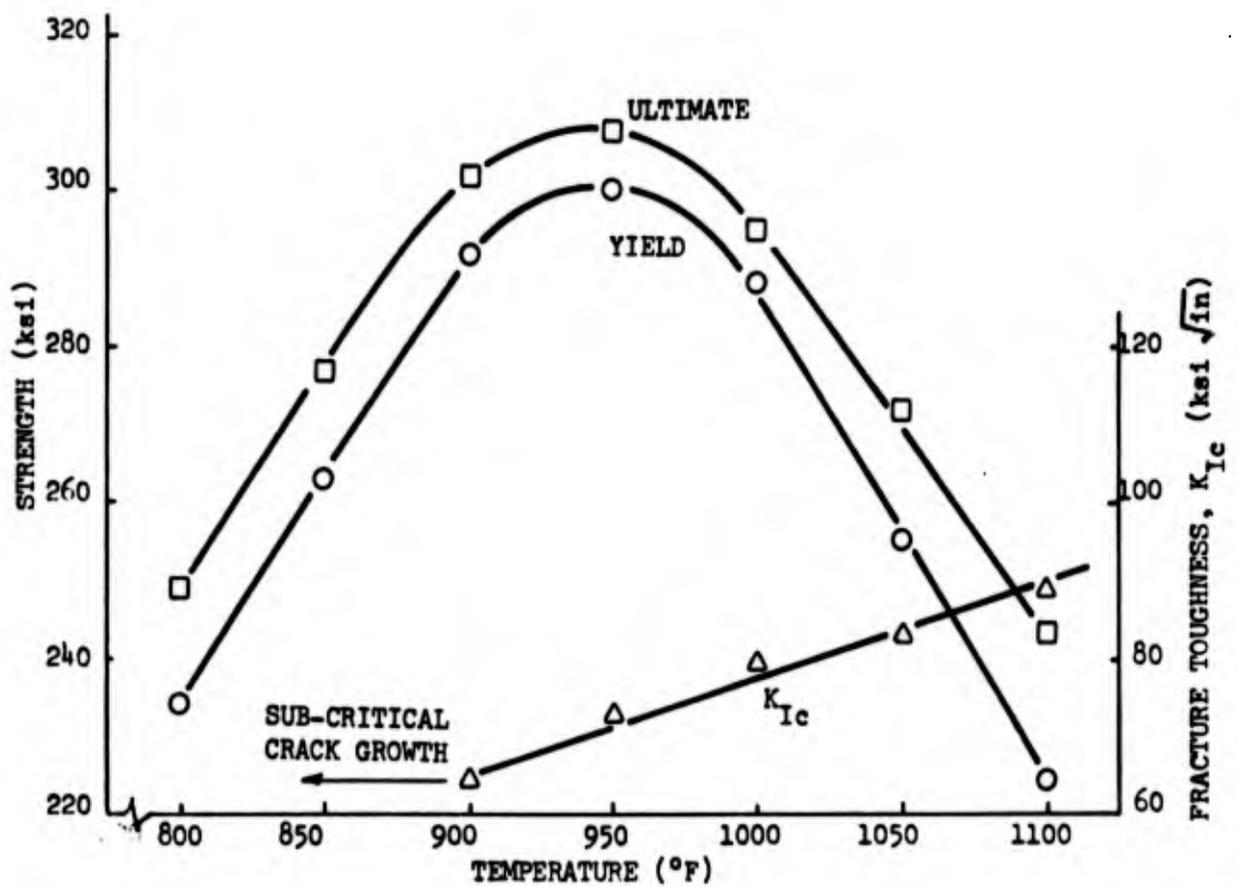


Figure 2 - Effect of Aging for 3 Hours

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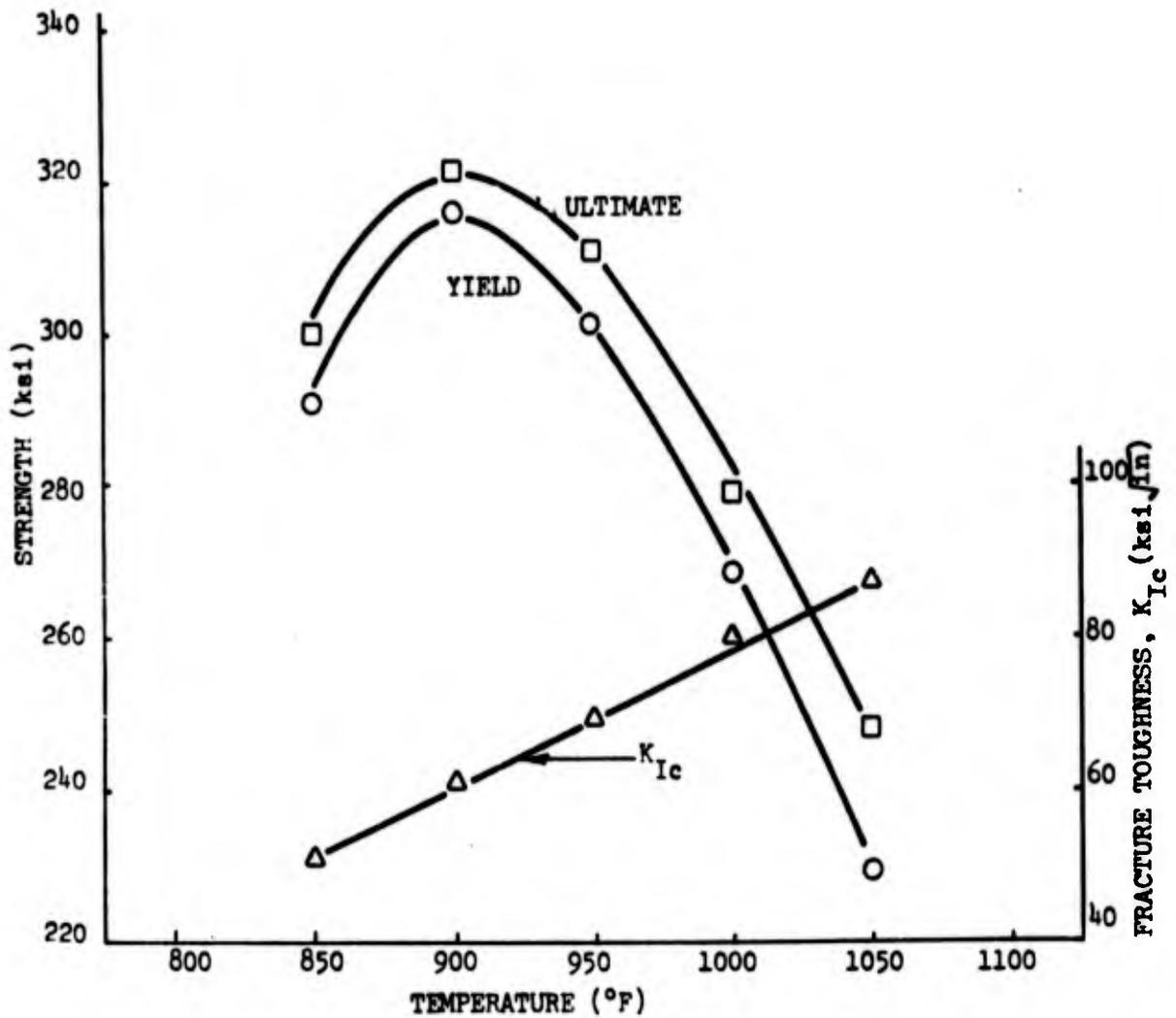


Figure 3 - Effect of Aging for 8 Hours

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PAGE

24



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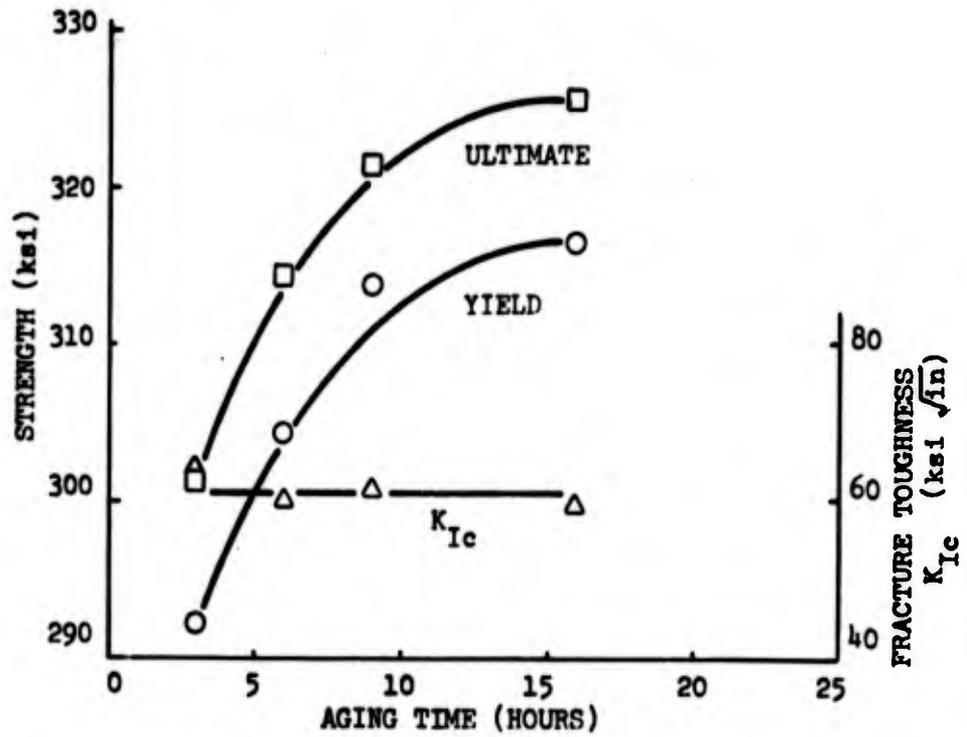


Figure 4 - Influence of Aging Time at 900°F

- △ AGED BELOW 900°F
- AGED 900°F
- AGED ABOVE 900°F

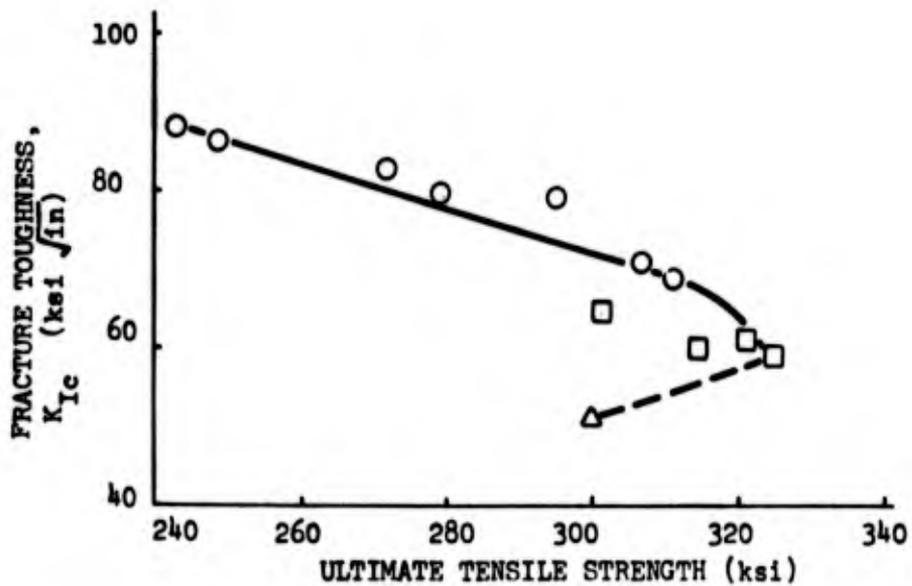


Figure 5 - Relationship Between Tensile Strength and Toughness

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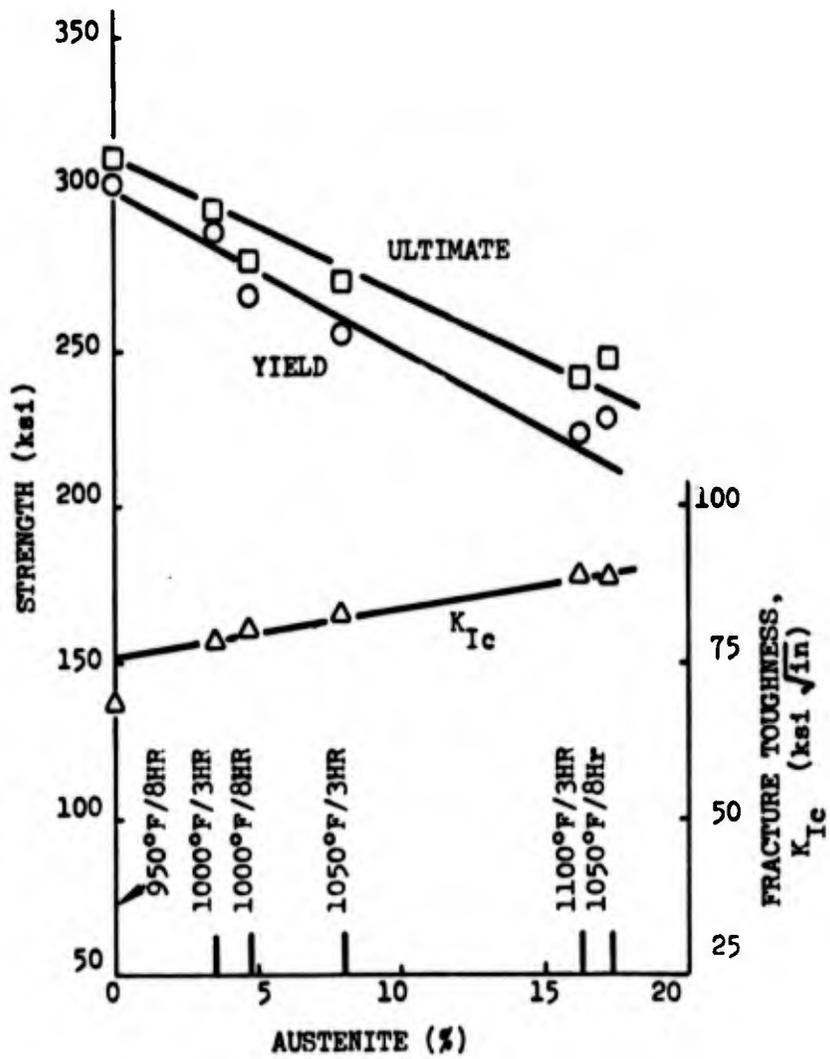


Figure 6 - Relationship Between Austenite Content and Properties

AD 1546 D



SPECIMENS AUSTENITIZED AT 1500°F AND
 QUENCHED TO TEMPERATURE SHOWN

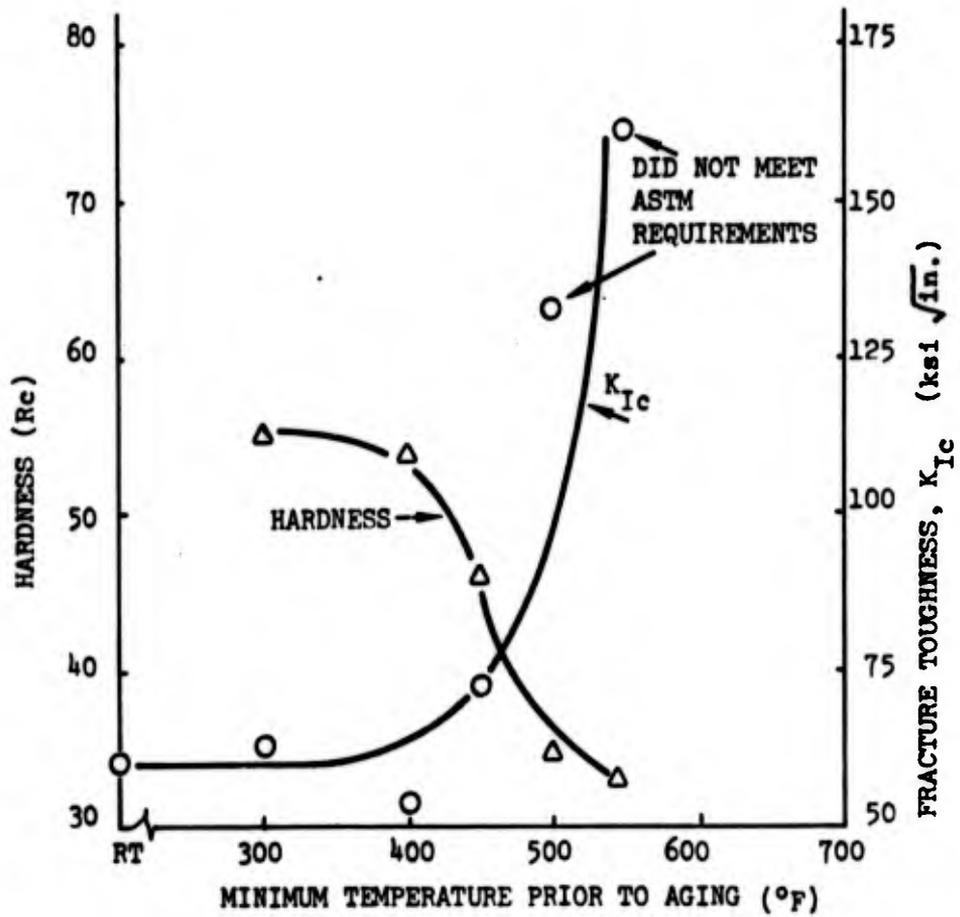


Figure 7-Effect of Interrupted Quenching on Properties

AD 1546 D

AD 1546 D

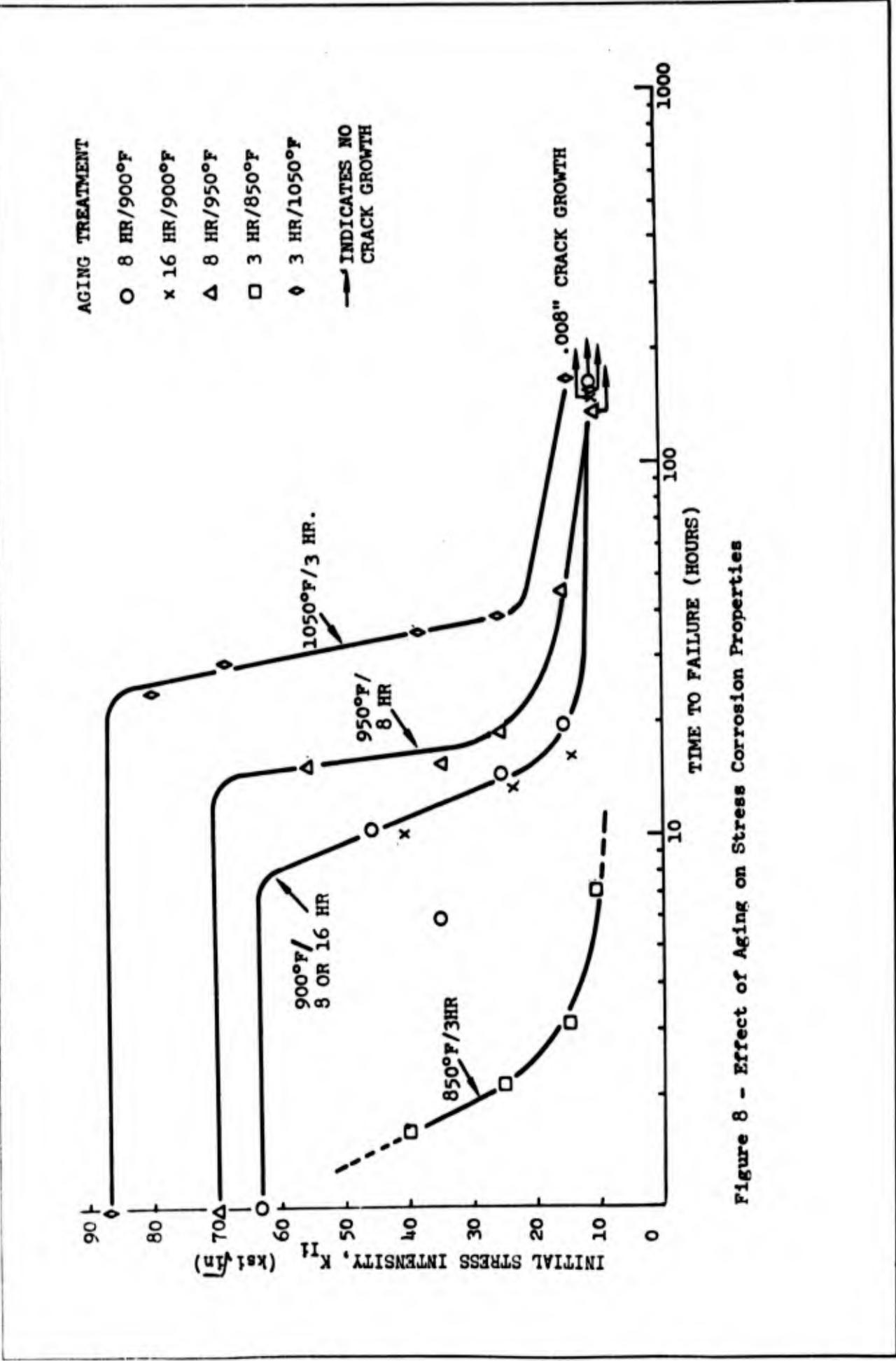
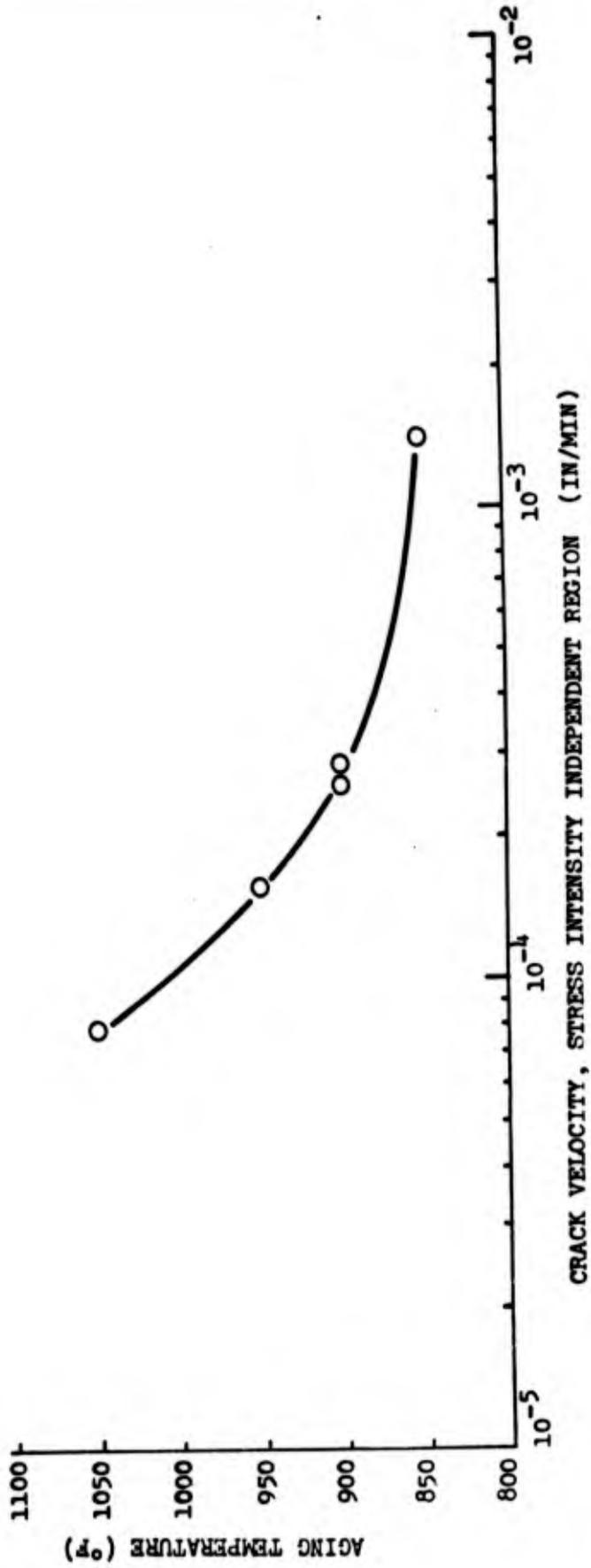


Figure 8 - Effect of Aging on Stress Corrosion Properties



CRACK VELOCITY, STRESS INTENSITY INDEPENDENT REGION (IN/MIN)

Figure 9 - Effect of Aging Temperature on Stress Corrosion Crack Velocity



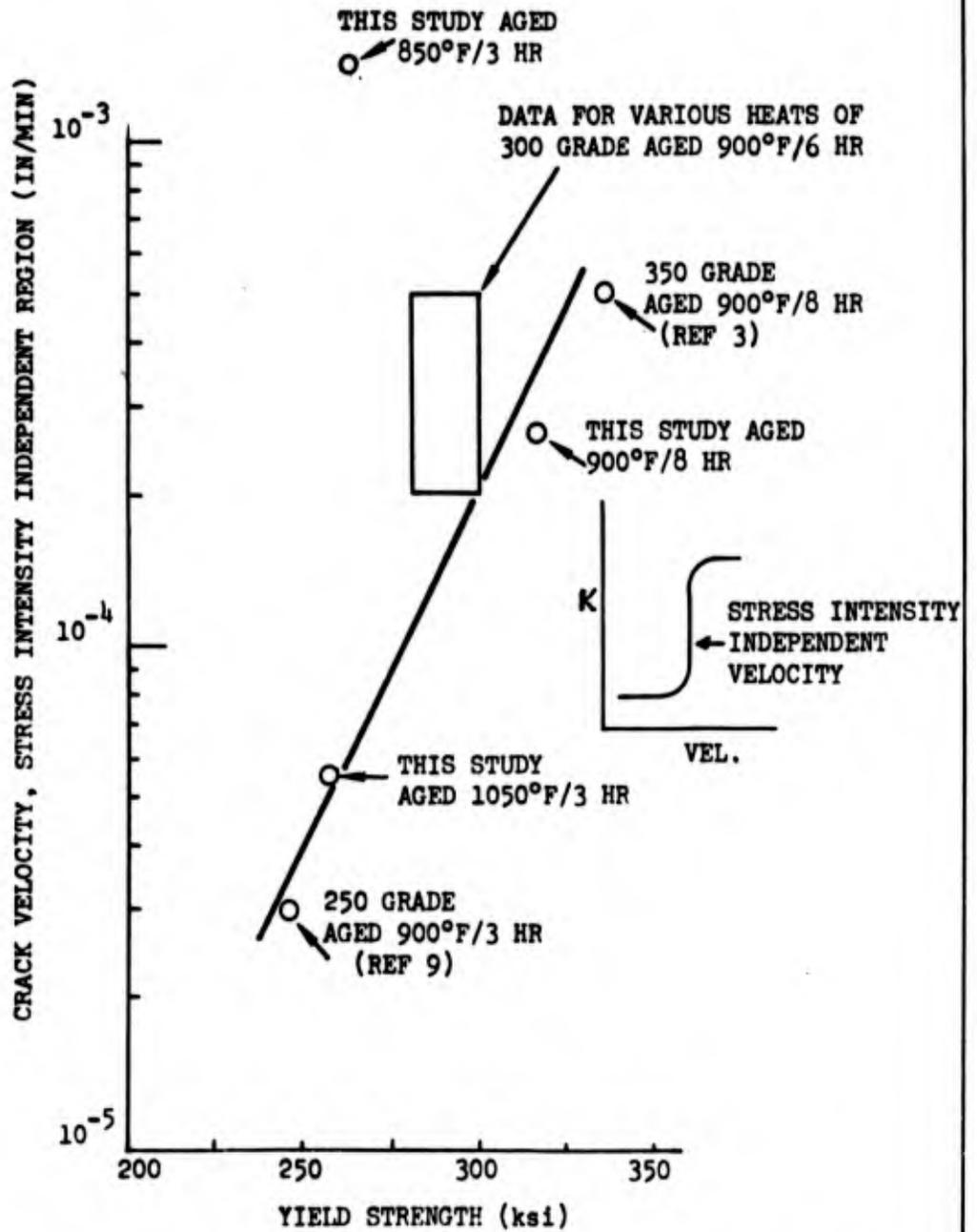


Figure 10 - Relationship between yield strength and stress corrosion crack velocity

AD 1546 D

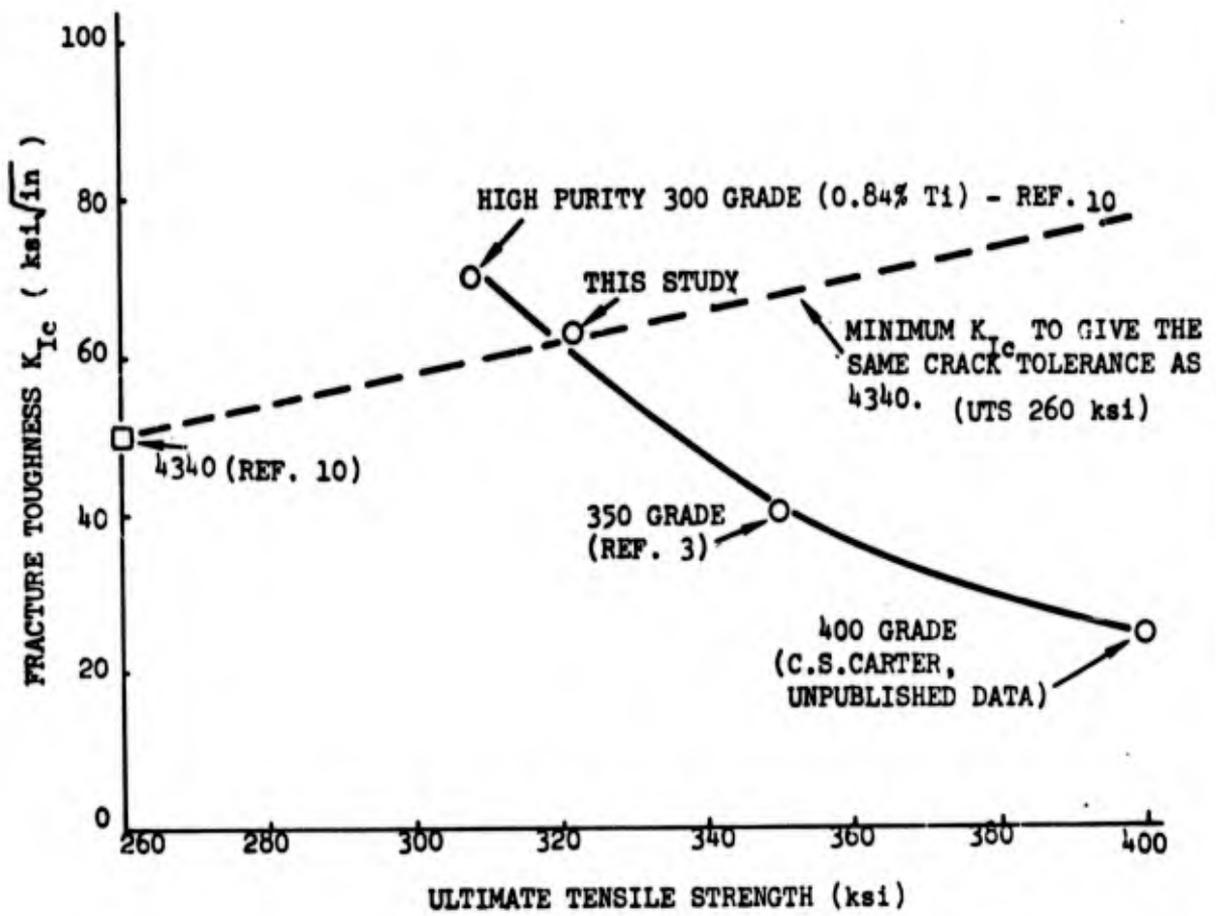


Figure 11 - Fracture toughness of high strength maraging steels.

AD 1546 D

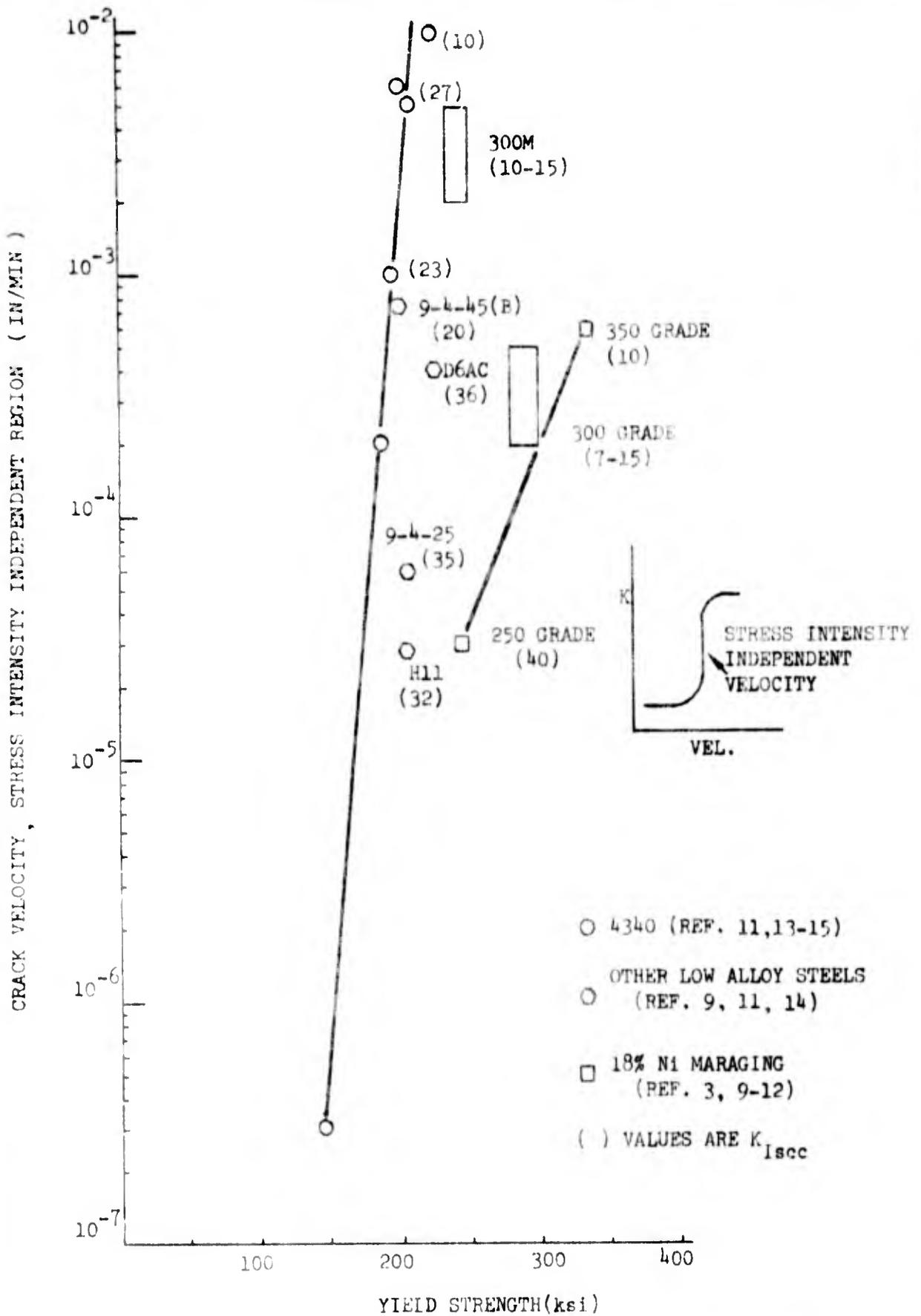


Figure 12 - COMPARISON OF STRESS CORROSION CRACK VELOCITIES IN MARAGING AND LOW ALLOY STEELS

AD 1546 D