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THE EFFECT OF AUSTENITIZING TEMPERATURE ON THE AMOUNT OF RETAINED AUSTENITE IN AISI TYPE 4340 STEEL

G. Y. Lai*, W. E. Wood†, R. A. Clark*,
V. F. Zackay* and E. R. Parker*

ABSTRACT

The effect of austenitizing temperatute on both the plane strain fracture toughness, K_{Ic}, and the microstructure of AISI 4340 was studied. Austenitizing temperatures of 870°C and 1200°C were employed. All specimens austenitized at 1200°C were furnace cooled from the higher austenitizing temperature and then oil quenched from 870°C. Transmission electron microscopy revealed an apparent large increase in the amount of retained austenite present in the specimens austenitized at the higher temperature. Austenitizing at 870°C resulted in virtually no retained austenite; only minor amounts were found sparsely scattered in those areas examined. A considerably altered microstructure was observed in specimens austenitized at 1200°C. Fairly continuous 100 to 200Å thick films of retained austenite were observed between the martensite plates throughout most of the area examined. Additionally, specimens austenitized at 870°C contained twinned martensite plates while those austenitized at 1200°C showed no twinning. Plane strain fracture toughness measurements exhibited an approximate 80 percent increase in toughness

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for specimens austenitized at 1200°C compared to those austenitized at 870°C. The yield strength was unaffected by austenitizing temperature. The possible role of retained austenite and the elimination of twinned martensite in the enhancement of the fracture toughness of these specimens austenitized at the higher temperature will be discussed.

INTRODUCTION

Most of the quenched and tempered low alloy steels in current use were developed decades ago by a basically trial and error approach. At high yield strengths (i.e., over 200,000 psi), these steels exhibit relatively low fracture toughness. Advancements in metallurgical techniques since the time these alloys were first introduced have been wide ranging. The profession is now capable of precisely relating alloy microstructure to mechanical properties.

Recent studies have shown that undesirable microstructural features in a secondary hardening 5Mo-0.3C steel can be minimized through austenitizing at a higher temperature[1]. The result of this microstructural improvement was a much higher fracture toughness. In other investigations the fracture toughness of several ultra high strength steels was improved by (a) the elimination of twinned martensite plates[2,3] and (b) by the utilization of a strain induced phase transformation[4,5].

The goal of the current investigation was to apply the understanding of these and related studies to a widely used low alloy quenched and tempered steel in an attempt to impart higher fracture toughness at yield strengths exceeding 200,000 psi.

EXPERIMENTAL PROCEDURE

This investigation focused on a commercial aircraft quality AISI 4340 Steel. This alloy, received in a fully annealed condition, had the following composition (in weight percent).

C	Mn	Cr	Ni	Mo	Si	Cu	S	P
0.40	0.80	0.72	1.65	0.24	0.24	0.19	0.01	0.004

Heat treatment initially involved use of several austenitizing temperatures and varied quenching media. Specimens were austenitized for one hour in an Argon atmosphere at 870°C, 1100°C, 1200°C or at 1200°C for one hour followed by cooling to 870°C and holding for one-half hour before quenching. After austenitizing the specimens were quenched into either room temperature oil, ice-water, or an agitated ice-10% brine solution. Thus numerous combinations of heat treatments were initially employed. From preliminary data on tensile strength and plane strain fracture toughness it was found expedient to concentrate on two heat treatments. The first treatment was similar to that used in commercial practice, austenitizing at 870°C then quench into room temperature oil. The second heat treatment involved a step quench, with specimens austenitized at 1200°C for one hour, then cooled to 870°C for one-half hour prior to being quenched in oil.

Mechanical properties were determined through tensile tests, plane strain fracture toughness tests, and Charpy V-notch impact tests, all conducted at room temperature (23°C). Tensile properties were determined on ASTM specified round tensile specimens of 0.357 in. gage diameter and 1.4 in. gage length. The specimens were pulled on a 300 kip MTS machine at a cross heat speed of 0.04 in./min. Standard sized ASTM Charpy V-notch specimens were broken in a pendulum type impact machine calibrated to a 60 ft-lb capacity. Plane strain fracture toughness, (K_{Ic}) determinations were conducted in accordance with ASTM specification E399-70T[6] and using compact tension specimens 0.605 in. thick. The method of calculating stress intensity recommended by Srawley and Gross[7], was used to determine K_{Ic} values. All specimens were fatigue precracked

at 6 cycles per second on the MTS machine. They were then pulled on a 11,000 lb. capacity Instron at a 0.04 in./min cross head speed. All mechanical properties specimens were machined slightly oversized prior to heat treatment. After heat treatment, specimens were ground to final dimensions using flood cooling to minimize heating. This procedure was used to eliminate possible effects due to surface decarburization of the specimens.

Microstructures were catagorized using both optical metallography and thin foil transmission electron microscopy. Optical metallography was employed to determine the presence and distribution of large inclusions. Also examination of prior austenite grain boundaries required optical examination. Transmission electron microscopy was used extensively for microstructural substructure identification, and especially in the determination of the existence and distribution of retained austenite. Foils for transmission work were prepared from slices carefully taken from previously tested fracture toughness (K_{Ic}) specimens. Slices 0.020 in. thick were mechanically sectioned using flood cooling to avoid tempering. They then were chemically thinned to 0.005 in. in an H_2O_2+HF solution. Electropolishing in a chromic-acetic acid solution using the window method completed foil preparation. All foils were examined in a Siemans Elmiskop IA operated at 100 kV.

All fracture surfaces of K_{Ic} specimens were examined using a JSM-U3 Scanning Electron Microscope operated at 25 kV in a back scattered electron mode. Fracture surfaces were protected with acetate replicating tape while the specimens were being cut to an acceptable size for viewing.

RESULTS

Mechanical Properties

The room temperature tensile properties are listed in Table I. From these data it is evident that varying the austenitizing temperature between 870°C and 1200°C has little effect on either the yield strength or the ultimate tensile strength. However, the ductility (elongation and reduction in area) was better with the conventional 870°C austenitizing temperature. There was no significant variation in the low Charpy V-notch impact resistance with austenitizing temperature, as shown in Table II. The results of the plane strain fracture toughness (K_{Ic}) tests are listed in Table III.

Several austenitizing temperatures and quenching media were employed on K_{Ic} specimens, as shown in Table III. The heat treatment conditions which afforded the best fracture toughness results were subsequently used for tensile and Charpy impact specimens. Several important observations can be made from the data in Table III. First, raising the austenitizing temperature from 870°C to 1200°C and oil quenching results in a substantial improvement in fracture toughness. Second, quenching directly from either the 870°C or 1200°C austenitizing treatment into water or iced-brine resulted in quench cracking. However, quenching into either water or iced-brine after the stepped 1200°C-870°C austenitizing treatment did not cause quench cracking. Furthermore, the stepped austenitizing treatment combined with an oil quench resulted in fracture toughness values of the same level as those exhibited by specimens oil quenched directly from 1200°C. It will also be noted in Table III that austenitizing at 1100°C and oil quenching resulted in toughness values intermediate to

those obtained with specimens austenitized at 870°C and 1200°C. In addition, one observes that with the aforementioned austenitizing treatment (1200°C) there is no apparent advantage accrued through quenching in a faster medium than oil. Thus it was decided to concentrate the investigation on the microstructures and related mechanical properties of specimens austenitized using the optimum stepped austenitizing procedure followed with an oil quench. In addition, specimens given the standard commercial procedure of austenitizing at 870°C and oil quenching were thoroughly examined in order to allow comparison and correlation of the microstructures and mechanical properties resulting from the different heat treatments.

Fractography

The fracture surfaces of two K_{Ic} fracture toughness specimens are shown in the SEM photomicrographs of Fig. 1. The photomicrograph in the upper panel (1a) is of a specimen austenitized at 870°C and oil quenched. The fracture surface is almost entirely quasi-cleavage, with scattered packets of ductile rupture. The "cleavage" was transgranular in nature and the area of each cleaved region approximates the size of the prior austenite grains. The photomicrograph in the lower panel (1b) is of the fracture surface from a specimen austenitized using the 1200°C/870°C/oil quench procedure. This surface exhibited extensive amounts of dimpled rupture, as well as some quasi-cleavage regions. The shallow dimples are typical of failures involving a void coalescence mechanism, as described by Low[8]. Comparing the two fractographs, the fracture surface in Fig. 1b is that of a specimen having much greater ductility, hence greater energy

absorption, than that shown in Fig. 1a which had the commercial heat treatment. An additional feature of the fractographs was the indication of MnS inclusions which were often located at the bottom of the dimples. It appears that inclusions may be responsible for crack nucleation by ductile tearing at the matrix-inclusion interface. It is obvious from the fractographs that the material step austenitized at 1200°C-870°C is capable of more ductility at the microscopic level than material austenitized at 870°C.

Microstructure

Optical metallographic examination revealed that the prior austenite grain size of the step quenched (1200°C-870°C/oil) heat treatment was an order of magnitude larger than the grain size attained with a commercial heat treatment (870°C/oil). The specimens austenitized at 870°C had an average grain size of 0.015 mm, while the coarse grained material had an average diameter of approximately 0.15 mm. Optical microscopy did not reveal any ferrite or upper bainite for either heat treatment.

Transmission electron microscopy revealed that both heat treatments (1200°C-870°C, oil and 870°C, oil) resulted in mixed microstructures consisting of auto-tempered martensite, untempered martensite, and small amounts of lower bainite. Two types of martensite were present; large lenticular plates and bundles or packets of fine parallel laths. In the fine grained structure, the large martensite plates would often extend across an entire grain; this was never observed in the coarse grain structure. The large plates in the coarse grain structure originated at the prior austenite grain boundaries and protruded only partially across the grain.

The martensite exhibited a substructure which varied with the two heat treatments. The packets of lath martensite were very similar for both austenitizing treatments. The laths generally exhibited a high dislocation density; however, a few twinned laths were observed. The plate martensite in specimens austenitized at 870°C consisted of a mixture of dislocated plates and twinned plates. The dislocated plates were auto-tempered; the carbide precipitates were identified as ε-carbide. The twinned plates showed no auto-tempering. This suggests the twinned plates were the last to form, hence, at the lowest temperatures. Use of the high temperature step austenitizing treatment resulted in the elimination of twinned martensite plates. All the plate martensite in the specimens heat treated at the higher austenitizing temperature was dislocated and exhibited extensive auto tempering.

The most striking difference in microstructure resulting from the two different austenitizing temperatures was in the amount of retained austenite observed. The 870°C austenitization resulted in very little retained austenite, as observed in the bright field micrograph of Fig. 2a, and the corresponding dark field micrograph of an austenite reflection (Fig. 2b). Conversely, specimens given the step austenitizing treatment (1200°C-870°C) showed extensive networks of 100-200Å thick austenite films surrounding martensite plates and packets of laths. Figure 2c is a bright field picture and 2d is a dark field image of an austenite reflection for the stepped heat treatment. Bright field (Fig. 3a) and dark field (Fig. 3b), lower magnification electron micrographs of step austenitized material, reveal the extensive distribution

of the austenite films. Verification of this unusual type of microstructure was made by examining several foils. Diffraction patterns similar to that exhibited in Fig. 3c were used to determine the presence of retained austenite. The analysis of this pattern, given in the line drawing (Fig. 3d), showed that the orientation between austenite and martensite, exhibited the Kurdjumov-Sachs relationship, $\langle 111 \rangle_M \| \langle 110 \rangle_\gamma$. Another interesting observation that was made related to the apparent thermal stability of the retained austenite. Specimens refrigerated in liquid nitrogen (-196°C) for 1 hour directly after the initial oil quench exhibited no change in the amounts of retained austenite compared to non-refrigerated specimens. Correspondingly there was no difference in plane strain fracture toughness between refrigerated and non-refrigerated specimens. X-ray studies of the retained austenite were conducted, but it was not possible to obtain quantitative data on the volume fraction present.

The stability of the austenite with respect to plastic strain was considered, but because of the difficulties in transmission microscope foil preparation, the effect of plastic strain could not be determined.

DISCUSSION

Examination of the foregoing data has shown that a large improvement in plane strain fracture toughness results when 4340 steel is austenitized at 1200°C rather than at 870°C. The as-quenched yield and ultimate tensile strengths are not significantly changed by the higher austenitizing temperature, but the tensile test bar ductility is lowered by the 1200°C treatment. Concurrent with the improvement in fracture toughness, the

mode of fracture changed from a mixture of quasi-cleavage and dimpled rupture to a predominantly dimpled rupture fracture surface. Possible explanations for the improved fracture toughness appear to lie in two areas, the change of submicrostructure, i.e., less twinning, with the higher austenitizing temperature, and secondly, the large increase in retained austenite obtained with the higher austenitizing temperature.

As described previously, the 870°C austenization resulted in a fine grain size. These grains were often traversed by large lenticular martensite plates. Some of the martensite plates were dislocated; however, others were twinned. The specimens austenitized at 1200°C did not exhibit any twinned plates. Furthermore, none of the dislocated plates were large enough to extend across the larger grains resulting from the higher temperature. Several previous investigators[2,3,9] have associated a drop in fracture toughness with the presence of increasing amounts of twinned martensite. Kelley and Nutting[10] attribute the loss in toughness to a decrease in the number of available slip systems in twinned plates by a factor of four. They suggested that the slip system must be such that both the slip plane and the slip direction are common to both the twin and the matrix. Bevis, et al[11] and Krauss et al[12] have indicated that twinned martensites are more likely to deform by mechanical twinning than by slip. Furthermore, Gilbert et al[13] have observed that twins induce cracks in certain instances. Thus, considering the fact that twinned martensite plates often traverse entire grains in the small grained material, while no twinned plates exist when the grains were larger, the possible correlation between transformation twinning and the lowered

fracture toughness in 870°C austenitized material must be considered.

The other prominent microstructural change was a large increase in the amount of retained austenite as the austenitizing temperature was raised from 870°C to 1200°C. A similar effect has been observed by Webster[14-16] in several studies. In his papers, Webster associates the increase in retained austenite with an increased solution of carbides and intermetallic precipitates at higher austenitizing temperatures. This, he reasoned, resulted in a lower M_s temperature, due to increased alloy concentration in the matrix, which in turn caused the increase in retained austenite. In order to test this hypothesis for our alloy system, M_s temperature measurements were conducted on specimens austenitized at 870°C and 1200°C[17]. It was found that the higher austenitizing temperature resulted in an increase of the M_s temperature of approximately 10°C, with no apparent effect on M_f. It is believed that the increase is due to the large grain size obtained by austenitizing at 1200°C. This would be in agreement with conclusions reached by Breinan and Ansell[18] on the effect of austenite yield strength on M_s temperature. In addition, the higher austenitizing temperature did not affect yield or ultimate tensile strength, which again suggests that the precipitate or inclusion density has not changed. Also, no undissolved carbides other than those due to auto-tempering were found in transmission or optical micrographs of specimens austenitized at 870°C then oil quenched.

The presence of retained austenite has been shown by Webster[16] to improve toughness through its crack arresting ability. In his paper Webster mentions how high magnification movie techniques were used to

follow crack propagation. Cracks propagating through martensite would be stopped upon intersecting a region of retained austenite. With further loading the cracks would branch out and grow around the austenitic area. This sort of crack motion would necessarily involve more energy absorption than straight propagation through martensite plates. Thus, the increased austenite content could certainly help explain the greater fracture toughness of the specimens austenitized at 1200°C.

Webster's explanation of enhanced toughening was based upon the assumption that the retained austenite was stable with respect to stress and strain. Recent studies by Gerberich et al[4] and Antolovich et al[5] suggest that an enhancement of the fracture toughness could also occur if austenite is converted to martensite by a strain (or stress) induced transformation. These workers showed that about five times more energy is absorbed by this type of transformation than for the plastic deformation of a stable metal matrix. In this investigation it was not possible to determine whether or not the retained austenite was being transformed in the fracture toughness test.

Ritchie, Knott, and Geniets[19] have proposed that high austenitizing temperatures can alter the distribution of impurities in steel and thereby affect the fracture toughness. They postulate that segregation of impurities (Sb in their studies) in prior austenite grain boundaries can be minimized by high temperature austenitization followed by fast quenching. The resultant dispersal of impurities changes the mode of fracture, and therefore the toughness from a low energy absorbing mode such as intergranular failure to a high energy one, i.e., dimpled rupture. In this

study no intergranular fractures were observed in the as quenched specimens regardless of austenitizing temperatures. The predominant mode of fracture of the brittle specimens was by cleavage.

CONCLUSIONS

The use of a higher austenitizing temperature for AISI 4340 steel results in a significant increase in fracture toughness with no loss in strength. Microstructural observations revealed that the higher austenitizing temperature resulted in both an increase in the amount of retained austenite and the elimination of twinned martensite. Either of these microstructural features by itself could enhance the toughness. In this investigation it was not possible to establish uniquely the role of each on the mechanical properties.

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TABLE I

Room temperature longitudinal tensile properties for alloy 4340 in the as-quenched condition

Austenitizing Temperature °C	Quench	Test Specimen Number	0.2% Yield Strength ksi	Ultimate Tensile Strength ksi	Elongation %	Reduction of Area %	True Fracture Strain %
1200	Oil	13	220	326	6.9	5.5	0.04
1200-870	Oil	7	230	318	2.6	6.7	0.06
		8	232	318	3.7	8.8	0.08
870	Oil	1	237	323	10.6	17.0	
		2	225	320	7.4	44.6	0.18

TABLE II

Room temperature Charpy V-notch impact properties of alloy 4340 (as-quenched)

Austenitizing Temperature °C	Quench	CVN Impact Energy (ft-lbs)
870	Oil	7.5
		6.1
1200	Oil	5.7
		5.4
1200-870	Oil	5.4
		6.0
1200-870	IBQLN*	3.6
		3.7

*Ice brine quench, liquid nitrogen refrigeration.

TABLE III

Room temperature fracture toughness properties for alloy 4340 in the as-quenched condition

Austenitizing Temperature °C	Quench	Test Specimen Number	$\frac{P_m}{P_Q}$	Plane Strain Fracture Toughness K_{Ic} ksi-in.$^{1/2}$	Critical Stress Intensity K_{crit} ksi-in.$^{1/2}$	$\frac{K_{Ic}}{\sigma_{ys}}$
1200	Oil	1	1.12	65.3	81.8	0.29
		2	1.15	60.9	88.9	0.27
		39	1.08	49.6	62.8	0.22
1200	Water	5	quench cracked			
		6	quench cracked			
1200	IBQLN	3	quench cracked			
		4	1.19	49.8	51.7	0.22
1100	Oil	41	1.07	40.0	45.3	0.18
1200-870	Oil	9	1.02	63.8	69.4	0.29
		10	1.01	66.6	68.7	0.29
		35	1.11	52.0	65.3	0.23
		46	1.00	61.2	61.2	0.26
1200-870	Oil+LN	43	1.07	55.6	63.7	0.25
1200-870	Water	11	1.14	59.3	74.6	0.26
		12	1.31	59.8	97.5	0.27
1200-870	IBQLN	7	1.14	60.5	84.5	0.27
		8	1.24	52.2	85.4	0.23
870	Oil	15	1.08	31.1	38.8	0.14
		16	1.00	39.0	39.4	0.17
		34	1.00	32.3	32.3	0.14
870	Oil+LN	45	1.00	33.1	33.1	0.14
870	Water	17	quench cracked			
		18	quench cracked			
870	IBQLN	13	quench cracked			
		14	quench cracked			
1200-870-870*	Oil	37	1.08	38.9	46.5	0.17

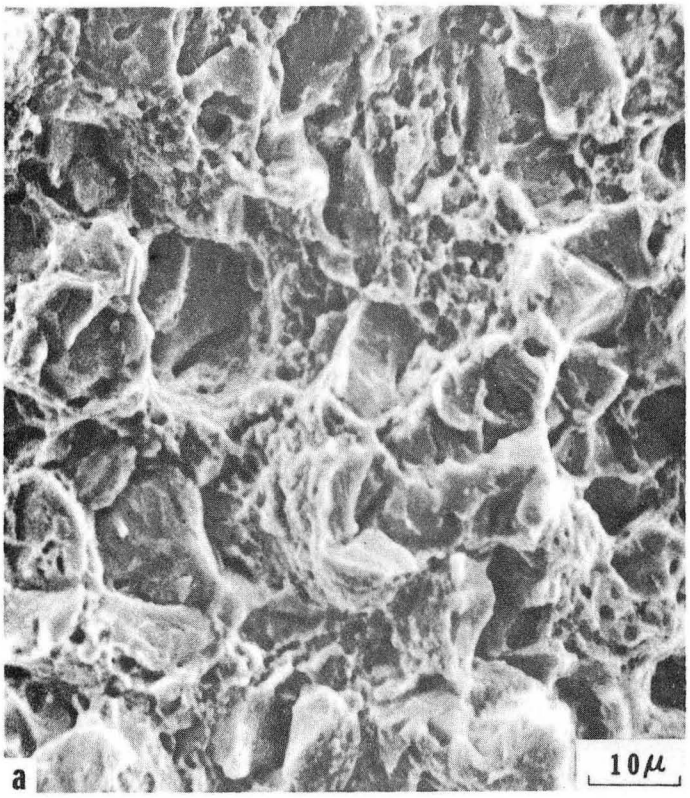
*Step quenched into oil (1200-870) and then re-austenitized at 870°C for one hour and oil quenched.

FIGURE CAPTIONS

Fig. 1 Scanning electron micrographs showing fracture surfaces of plane-strain fracture toughness specimens: austenitized at 870°C, oil quenched (a); austenitized 1200-870°C, oil quenched (b).

Fig. 2 Transmission electron micrographs of as-quenched AISI 4340: bright field (a) and dark field of austenite reflection (b) of 870°C/oil specimen; bright field (c) and dark field of austenite reflection (d) of 1200-870°C/oil specimen.

Fig. 3 Low magnification bright field (a) and dark field of austenite reflection (b) transmission electron micrographs. Diffraction pattern (c) showing austenite spots and corresponding explanatory line drawing (d).



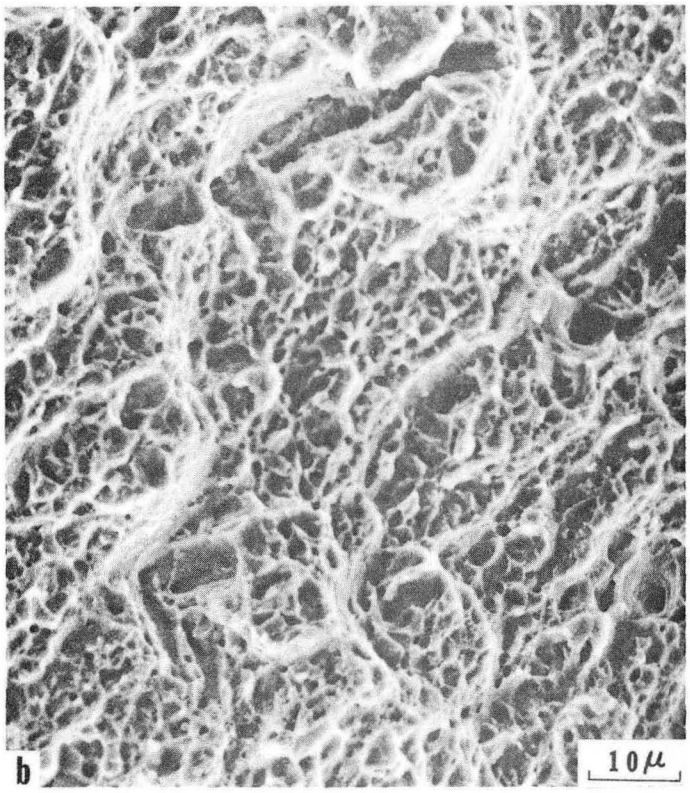


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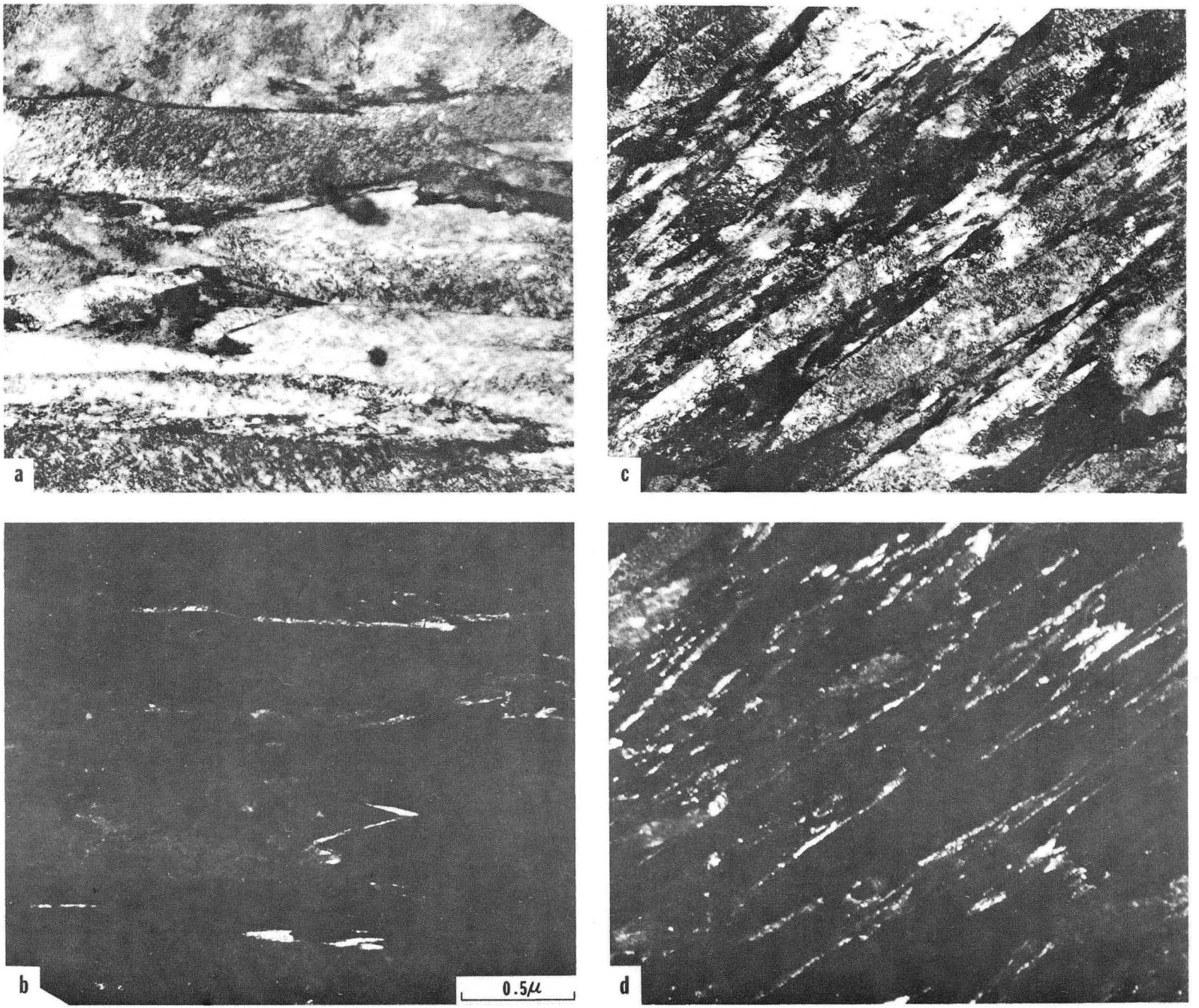
Fig. 1



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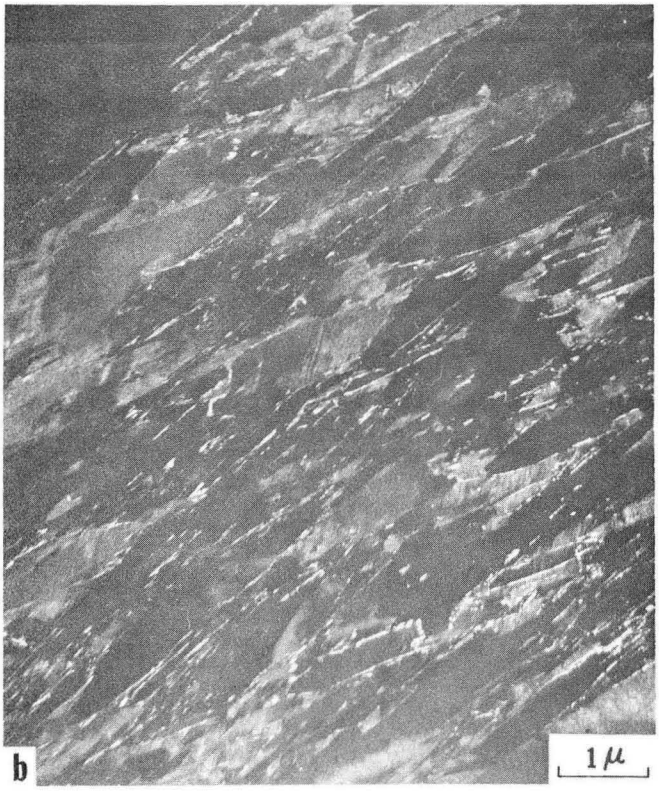

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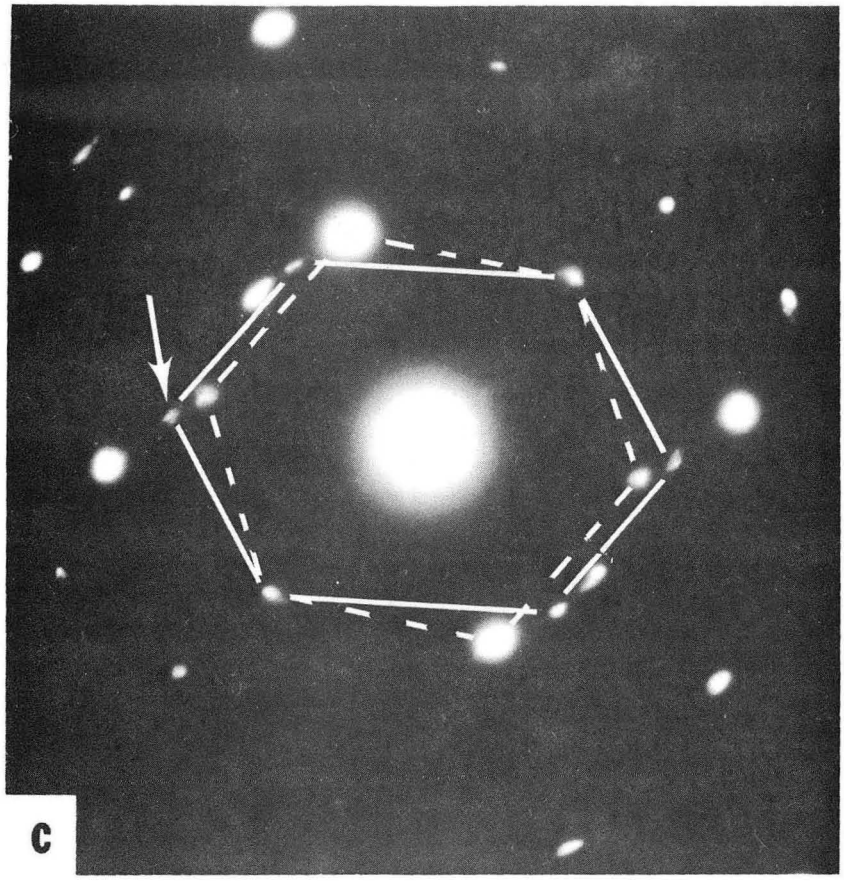
Fig. 3



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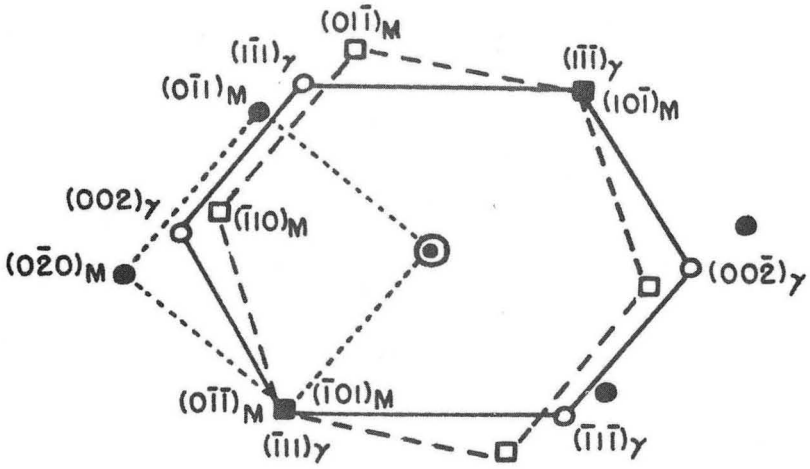

d

□ [111] MARTENSITE SPOT

○ [110] AUSTENITE SPOT

● [100] MARTENSITE SPOT

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Fig. 3 continued

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