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Submitted to Materials Science and Engineering

LBL-3522
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Prepared for the U. S. Energy Research and Development Administration under Contract W-7405-ENG-48

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DESIGN OF Fe/4Cr/0.4C MARTENSITIC STEELS ELIMINATING QUENCH CRACKING

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SUMMARY

Inter-granular cracking in the as-quenched structures of vacuum melted Fe/4Cr/0.4C steel has been investigated in terms of the heat treatment conditions. It has been shown that amount of carbon in solution and martensite packet size are the two most important factors influencing cracking tendency. Multiple heat-treatments have been designed in order to take advantage of higher austenitizing temperature and fine austenite grain size. Elimination of quench cracking and good mechanical properties were obtained after these treatments.

1. OBJECTIVES OF THE RESEARCH

One of the most economical ways of increasing the strength of steel is to raise its carbon content, and utilize heat treatments to form martensite. Unfortunately, above about 0.3-0.35%C, depending on the total alloy content, such high carbon steels are very brittle and in many cases cannot be used because after austenitizing and quenching the steels are already cracked (quench-cracking). Further treatments do not allow crack healing and in any case tend to lower the strength.

In a continuing program on alloy design of experimental structural steels[1,2] we have investigated the problem of quench cracking in Fe/4Cr/0.4%C steels since a similar steel but with a lower carbon content viz., 0.35% exhibits excellent tensile strength and fracture toughness without even tempering[3]. The role of chromium is to achieve suitable hardenability. The increase of carbon from 0.35-0.4% has the potential of raising the yield strength to over 300,000 psi and the challenge is to achieve this whilst at the same time provide good toughness properties and elimination of quench cracking. In 0.4%C steels conventional martensitic heat treatments viz., austenitize and quench, invariably lead to quench cracking. This communication describes the heat treatments we have developed to overcome this problem.

2. PREVIOUS WORK ON QUENCH CRACKING

Before designing suitable heat-treatments it is important to identify the principal known factors which influence quench cracking. Quench cracking in ferrous martensites, and especially in high carbon steels, has been the subject of many recent investigations[4-8]. It has been established[4,6,8] that microcracking in as-quenched high carbon steels is

due to the stresses resulting from impingement of growing martensitic plates. Consequently, Brobst and Krauss[4] concluded that the micro-cracking tendency can be decreased by refining the prior austenite grain size so as to limit the martensitic plate length, a parameter which has been directly linked[6] to the impingement stresses. Similarly, it has been established[7] that the amount of carbon in solution influences the micro-cracking tendency viz., a higher carbon in solution resulted in more micro-cracking. However, there has been no such systematic investigation to study the influence of grain-size, dissolved carbon and quenching medium on the inter-granular quench cracking in the as-quenched structures of medium carbon steels. Clark et al[9] attributed the intergranular quench cracking as mainly arising from the segregation of impurities at the grain boundaries in a commercial low alloy En 30 steel. On this basis they proposed that a higher austenitizing temperature coupled with a fast quenching rate to suppress any segregation of embrittling constituents leads to a decrease or elimination of quench cracks. However, the authors did not investigate the influence of quenching medium nor grain-size on the cracking.

Finally, it is also well recognized that a mixed microstructure consisting of undissolved carbides is deleterious to toughness, particularly if the carbide phase is present as a network at the original austenite grain boundaries, or at interlath boundaries such as in upper bainite if the steel has inadequate hardenability.

In summary, it appears that in order to achieve desirable fracture toughness properties which allow the steels to be utilized in engineering applications, it is essential to austenitize such that all carbides go into solution and, at the same time, in order to avoid quench cracking at high levels of carbon in solution, it is necessary to obtain a fine grain size. Clearly for martensite this cannot be done in a single heat-treatment because in order to have all the carbon in solution it is

necessary to have a high austenitizing temperature and this leads to a coarse grain size. Alternatively, isothermal treatments to form lower bainite might be beneficial and experiments were done to study this[10]. The choice of high purity materials and vacuum melting minimizes problems encountered in current commercial steels arising from segregation of embrittling elements[11,12] e.g. S, P, C etc.

3. EXPERIMENTAL PROCEDURE

The composition of the steel used in this investigation is 4.2% Cr, 0.42% C and the balance Fe. The alloy was prepared by vacuum melting. The measured Ms temperature for the steel was 320°C.

In order to observe quench cracking, optical microscopy of polished and etched surfaces and scanning microscopy of the fractured tensile specimen surfaces was conduted. In order to reveal the prior austenite grain-sizes, by light optical techniques, etching was carried out in an etchant of 5 gm. of picric acid in 100 cc of water saturated with dodecylbenzene-sulfonate. Transmission electron microscopy was carried out to document the substructural features.

All the heat-treatments were carried out in a vertical tube furnace under a constant flow of high purity argon gas. Isothermal transformations are carried out by directly quenching into a salt pot maintained at the required temperature at the end of austenitization treatment. Room temperature quenching was done in either agitated water or oil.

Mechanical properties were determined through tensile tests conducted on a 300 kip MTS machine using 1.25" gauge round specimens and through standard Charpy v-notch impact tests, all conducted at room temperature (23°C).

4. RESULTS AND DISCUSSION

Multiple heat treatments were devised to achieve the aims of fine grain size with all carbon in solution. Table I reports some of the results. In order to study the influence of austenitizing temperature and quenching medium, austenitizing at temperatures of 900°C, 1100°C, 1200°C, and 1290°C was carried out followed by quenching into either water or oil. Higher austenitizing temperatures (>1000°C) were found to be necessary[4] to keep all Cr and C (present as chromium carbide) in solution which thus ensures higher hardenability. 900°C is conventionally used in commercial austenitizing practice. The mechanical property data are given in Table II.

In all the treatments, the cracking observed was only intergranular with no evidence of intragranular micro-cracking normally associated with high carbon steels. Transmission electron microscopy indicated the transformation substructure to be mixed, i.e., both twinned and dislocated martensites were present, although their relative amounts varied with treatment (e.g., compare Fig. 1b and Fig. 2c). As should be expected, SEM fractographs on the fractured surfaces of quench-cracked specimens indicated extensive intergranular cleavage.

The high austenitizing temperatures used in this study resulted in a coarse grain size (ASTM grain size number <0.5). Intergranular quench cracking was observed in all the steels which had coarse austenite grain sizes when quenched to room temperature either in oil or water. Such cracking was widespread even in steels which were austenitized at 1200°C and 1290°C (Fig. 1a). Quenching in water resulted in more cracking than quenching in oil. Transmission electron microscopy on the steel austenitized at 1200°C resolved large amounts of twinned martensite (Fig. 1b) although

optical metallography revealed that the morphology is of lath type occuring in the form of packets[1] (Fig. 1a). Fractography of quench cracked specimens revealed an intergranular fracture mode (Fig. 2a). In order to understand the influence of re-austenitizing at the same temperature but for a shorter period on the quench cracking, a steel which was quenched in oil from 1100°C after an austenitizing treatment for 60 minutes was re-austenitized for 1 to 15 minutes at the same temperature and oil quenched. In all the steels irrespective of holding time during the second austenitizing treatment, quench cracking was present in the final structure (Fig. 2b). The striking feature of these treatments is that the quench cracks appeared to occur at the boundaries of the grains resulting from the first treatment. Furthermore, SEM fractographs revealed that most of the cracks after the first treatment did not heal during the second austenitizing treatment. However, there was a small increase in the failure stress after the second treatment. Concomitant with this, fractography revealed that there was a large increase in quasi-cleavage as compared to almost 100% cleavage (i.e. intergranular failure) in single-treated specimens (Fig. 2a and 2b). Marder and Benscoter[5] investigated the influence of tempering on the crack lengths in a 1.39 wt. pct. C steel, and concluded that the number of microcracks decreases sharply above a certain tempering temperature ostensibly due to the bridging of cracks by carbide nucleation. In contrast to this observation on microcracks, bridging of the intergranular quench cracks is very rare even when the specimen is re-austenitized. <u>Thus, intergranular quench cracking must be eliminated in the first cycle itself in a multicycle treatment</u>.

In order to study the influence of grain-size alone on quench cracking while keeping as much of carbon and chromium in solution, two-cycle austenitizing treatments were planned which involve a structure after the first treatment which is almost completely free from quench cracking. During the first cycle high austenitizing temperature (1100°C) was employed in order to keep all Cr and C in solution while a low austenitizing temperature was employed during the second treatment to refine the grain size. Two heat treatments were designed for this purpose. One of the treatments consisted of an interrupted-quench wherein the steel from the high austenitizing temperature is quenched to 265°C which is below Ms but above Mf. After holding for 3 min. at this temperature, the steel is air cooled to room temperature. The second heat treatment consisted of isothermally transforming at a suitable temperature to obtain a 100% lower bainitic structure[10]. Research on the lower bainite in the same steel has shown that no quench cracking occurs by this method. Optical metallography did not reveal any grain-boundary grooving in steels transformed by interrupted quenching (Table I) However, small and discontinuous quench cracks could still be identified in this steel from fractography. Nevertheless, the cracking tendency had decreased significantly. The material exhibited plastic yielding followed by failure at 270 KSi stress, which indicates that the critical crack propagation stress lies above the yield strength but below the tensile strength of the steel. SEM fractography indicated a mixed fracture mode consisting of some dimpled rupture and widespread quasi-cleavage together with cleavage near the cracks at the boundary. As a result of the heat treatment investigations, the most successful results were obtained starting with one of the two initial micro-structures

described above and re-austenitizing at 900°C for 30 min. Rockwell hardness measurements and tensile strength data (Table II) indicated that most of the carbon is in solution after 30 min. holding time. The resulting grain size corresponded to ASTM grain size number 8 (Fig. 3b) and the as-quenched structures were free from intergranular cracking (Fig. 3a). An ultimate tensile strength of 335 KSI and a yield strength of 250 KSI (specimen C3) or 280 KSI (specimen D6) were obtained. The resulting microstructure is shown in Fig. 3c. It was also found that an optimum combination of strength and ductility could be obtained by employing interrupted quenching to 265°C after the second austenitizing treatment (i.e., specimen C7 or D5). After this treatment, the elongation and Charpy-V-notch impact energy values of the steel showed an improvement while there was a small drop in tensile strength. A mixed morphology consisting of a small number of plates extending right across the grains as well as parallel martensite laths were identified from the optical microstructures of all steels subjected to the two-cycle treatments. A detailed discussion of the microstructure-heat-treatment-mechanical properties including bainitic treatments will be given later.[13] For comparison purposes, mechanical properties of specimens subjected to a single treatment involving a 900°C austenitization followed by oil quenching to room temperature are also included in Table II. It is seen that the steel subjected to this treatment showed poor impact toughness and elongation while the strength level was about the same as the steels subjected to two-cycle treatments.

Fractographs of specimens C3, C7, D6 and D5 did not reveal any intergranular cracks. Extensive quasi cleavage fracture with a small amount of dimpled rupture was present in the specimens C3 and D6

(Fig. 3d). The amount of dimpled rupture increased in the specimens C7 and D5.

5. DEVELOPMENT OF A QUALITATIVE MODEL OF INTERGRANULAR CRACKING

Micro-cracking at impinging martensite plates in high carbon steels is totally absent in medium carbon steels (carbon content about 0.4%). Also, there was no intergranular quench cracking reported[3] in a steel containing essentially 4% Cr and 0.35%C when a high austenitizing temperature of 1100°C was used. The steel used in the present investigation which has about 20% more carbon when similarly heat treated resulted in extensive intergranular cracking. Thus, any explanation for quench cracking tendency should incorporate the amount of carbon in solution as one of the most important factors influencing the cracking tendency.

Quench cracking persisted even in specimens subjected to very high temperature austenitizing, viz., at 1200°C and 1290°C followed by fast quenching in water to room temperature. Such a practice was considered to discourage any segregation of embrittling constituents at the grain boundary and the consequent intergranular cracking[9]. Fast quenching apart from discouraging segregation of embrittling constituents also results in an increased amount of carbon in solution by reducing the carbon segregation to defects in the parent austenite[15]. So also, fast quenching results in decreased carbon precipitation in martensite, i.e., less auto-tempering by limiting the time for such process. The results suggest that segregation of embrittling constituents at the grain boundary can not be construed as the main reason for intergranular cracking in the present steel.

Davies and Magee[6] considered qualitatively the mechanisms of micro-cracking in detail and explain that micro-cracking results from the accommodation of strain at the tip of the impinging martensite plate. The authors argue that the strain energy due to the volume change accompanying the austenite-martensite transformation increases as the transformed volume [proportional to $(\text{length})^2 \times$ thickness of plate]. In contrast to plate martensite in high carbon steels, martensite in medium carbon steels occurs predominantly in the form of laths, particularly when high temperature austenitizing is employed. The martensite laths in a packet are all parallel and the laths from one packet may not be parallel to the ones from a different packet. It is observed that the packet size is strongly dependent on the prior austenite grain size. Thus, in a steel austenitized at 1200°C, the average packet size is much larger than the average packet size obtained after a 900°C austenitizing treatment.

Thus, it is possible to identify the two most important factors which influence intergranular cracking, viz., (i) carbon in solution (in austenite) and (ii) the average packet size of the martensite. By analogy with micro-cracking in high carbon steels, continuous intergranular cracking in medium carbon steels is then proposed to occur as a result of accommodation of strain due to the impingement of one growing packet on the other at grain boundaries. Whereas the strain and the corresponding stress due to transformation are highly localized in the case of plate martensite, this strain in the case of packet martensite is distributed throughout the packet boundary and thus, results in continuous cracking. These strains are dependent essentially on two

parameters; (i) dilatation of the lattice accompanying transformation, and (ii) the volume of the transformed product. Carbon in solution before transformation determines the magnitude of the first parameter while the packet size determines the second parameter. If the carbon in solution is fixed, then, the cracking tendency can be decreased by limiting the packet size, i.e., prior austenite grain size.

Thus, specimens C3 and D6 which had most of the carbon in solution showed no evidence of intergranular cracking because of their much reduced packet size as compared to specimen A. Finally, it is suggested that inter-granular cracking is a manifestation of transformation strains and the resultant impingement stresses of two growing martensite packets and other proposed parameters like severity of quench and segregation of embrittling constituents can only aggravate but can not form the basis for such cracking.

CONCLUSIONS

(i) It has been established that carbon in solution and the martensite packet size are the two most important factors influencing quench-cracking. An increase in either of them increases the probability of such cracking.

(ii) Based on this research, heat treatments have been developed which ideally combine the beneficial effects of higher austenitizing temperature and fine grain size. In addition to eliminating quench cracking, good mechanical properties are also obtained utilizing these treatments.

(iii) Conventional grain refining involving repeated austenitizing and quenching cannot be used to refine the grain size since intergranular cracks produced after the first cycle cannot be eliminated during subsequent heat treatment.

(iv) A heat treatment which involves interrupted quenching to a temperature in the martensite transformation range appears to yield relatively better ductility and impact toughness without exhibiting a drastic drop in tensile strength, as compared to conventional treatments.

ACKNOWLEDGMENTS

This work is supported by the U.S. Energy Research and Development Administration and is being done in partial fulfillment of the M.S. degree at Berkeley (B.V.N.R.). We acknowledge helpful discussions with Dr. R. O. Ritchie. The alloy steel used in this investigation was supplied by Republic Steel Research Center.

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TABLE CAPTIONS

Table I. Inter-granular quench cracking characteristics of various heat treatments.

Table II. Mechanical properties of some of the heat treatments.

FIGURE CAPTIONS

Fig. 1a: Coarse prior austenite grain size and the resultant intergranular cracking when quenched to room temperature in water following 50 min. austenitization at 1200°C. Note large martensite packets.

Fig. 1b: Electron micrograph of the same steel showing mixed, i.e. twinned and dislocated substructures.

Fig. 2a: SEM fractograph of specimen A (see Table I) showing intergranular fracture.

Fig. 2b: SEM fractograph of specimen B (see Table I) revealing quasi-cleavage fracture and unhealed intergranular crack from the first treatment.

Fig. 3a: Optical micrograph of specimen D6 (see Table I) showing a mixed morphology of martensite. A small number of plates extending across the grain can be seen. There is no evidence of intergranular cracking. Nital etch.

Fig. 3b: Optical micrograph of the same specimen (D6) but etched in picric acid in order to reveal the grain boundaries. The measured average grain diameter was 23 microns.

Fig. 3c: Electron micrograph of specimen C3 (see Table I) showing predominantly dislocated lath martensitic structure. A very small percent of twinning has been observed. Thin films of retained austenite (stabilized) have been identified at interlath boundaries. Extensive auto-tempering has also been detected.

Fig. 3d: SEM fractograph of the same specimen (C3) showing extensive quasi-cleavage fracture.

TABLE I

Specimen	Heat-treatment	ASTM grain size no.	Presence of g.b cracking (Nital etch)	Fractography
A	1100°C-60min→oil	<0.5	yes	Intercrystalline cleavage.
B	A+1100°C-15min→oil	-	yes	Some quasi-cleavage
F	1200°C-50min→oil	<0.0	yes	-
G	1200°C-50min→water	<0.0	yes	-
E2	900°C-60min→oil	6.5	no	-
C1	1100°C-60min-265°C-3min-Aircool	<0.5	no grooving was observed	small gr.bd. cracks are detected
C3	C1+900°C-30min→oil	8	no	largely quasi-cleavage
C7	C1+900°C-30min→ 265°C- 3min-Aircool	8	no	quasi-cleavage + some dimpled rupture
D6	1100°C-60min-360°C-15min-Aircool-900°C-30min→oil	8	no	largely quasi-cleavage
D5	1100°C-60min-360°C-15min-Aircool-900°C-30min-265°C-3min-Aircool	8	no	quasi-cleavage + some dimpled rupture

TABLE II

Specimen	0.2% offset yield strength KSI*	UTS KSI*	% Elongation (1.25in gauge length)	CVN Impact Energy Ft.lbs°	Rc Hardness
A	200	← quench cracked →			60
B	235	← quench cracked →			-
C1	210	270	1.0□	9.4	53
C3	250	335	6.0	6.5	58
C7	225	300	7.0	10.5	53
D6	280	335	2.5□	9.0	57
D5	225	310	7.0	10.5	53
E2	270	330	2.0□	4.0	58

* 1 KSi = 6.89 MNm^{-2}

° 1 Ft-lb = 1.36 Nm

□ Failed before the onset of necking.

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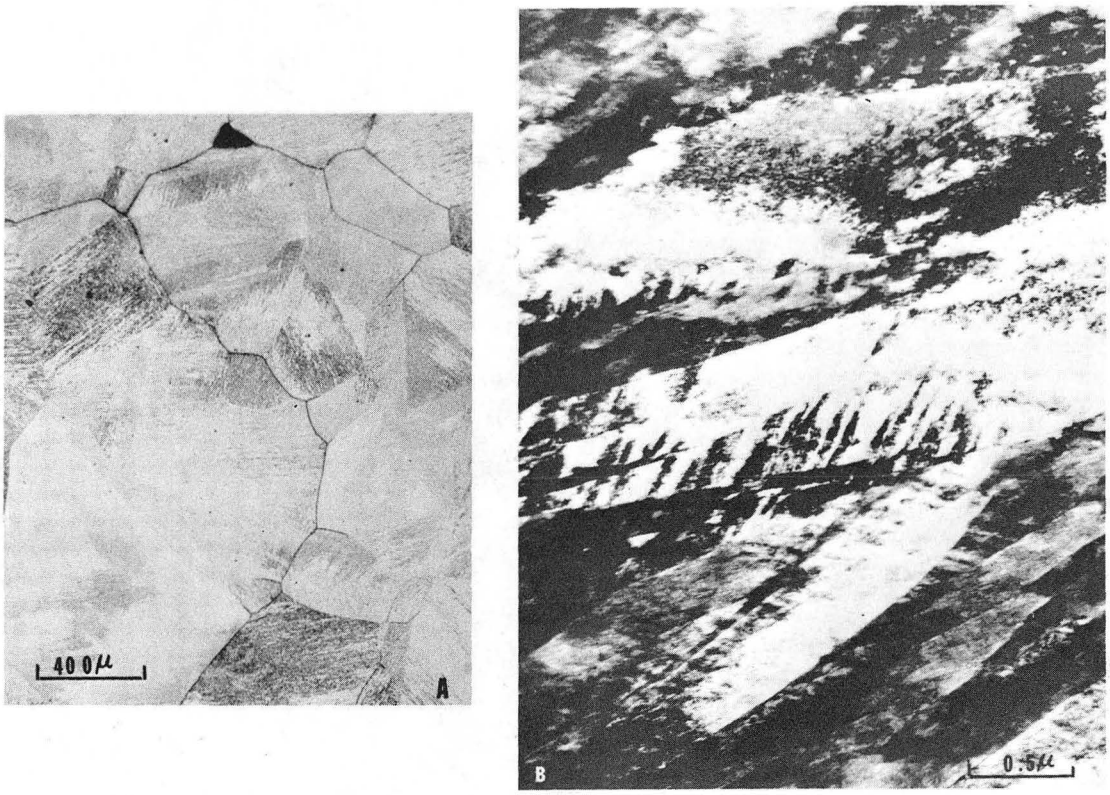

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Fig. 1.

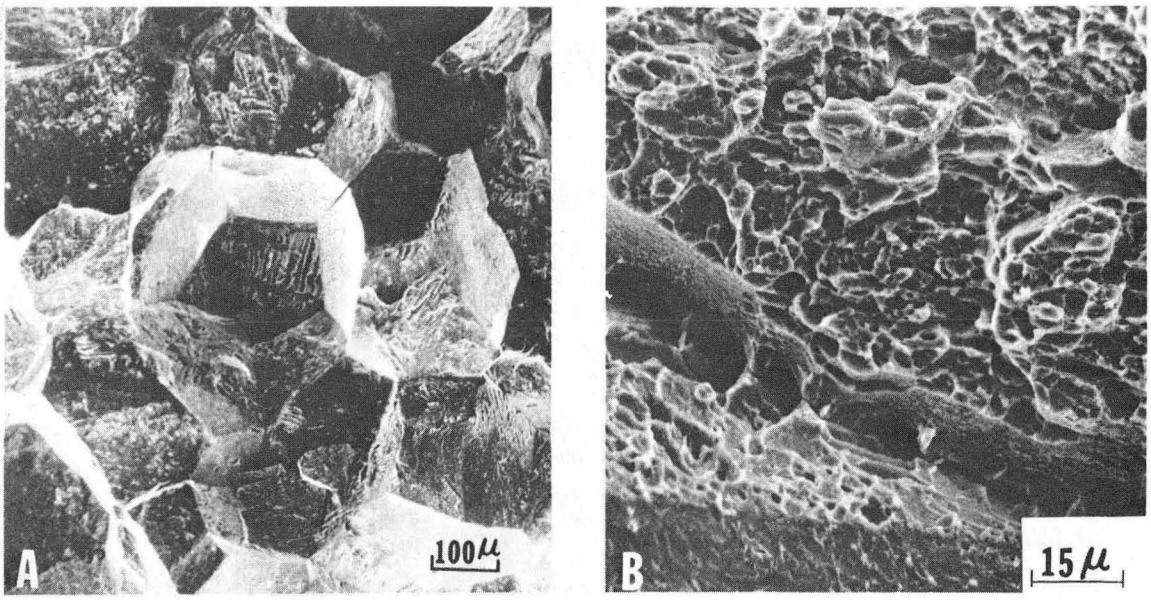

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Fig. 2.

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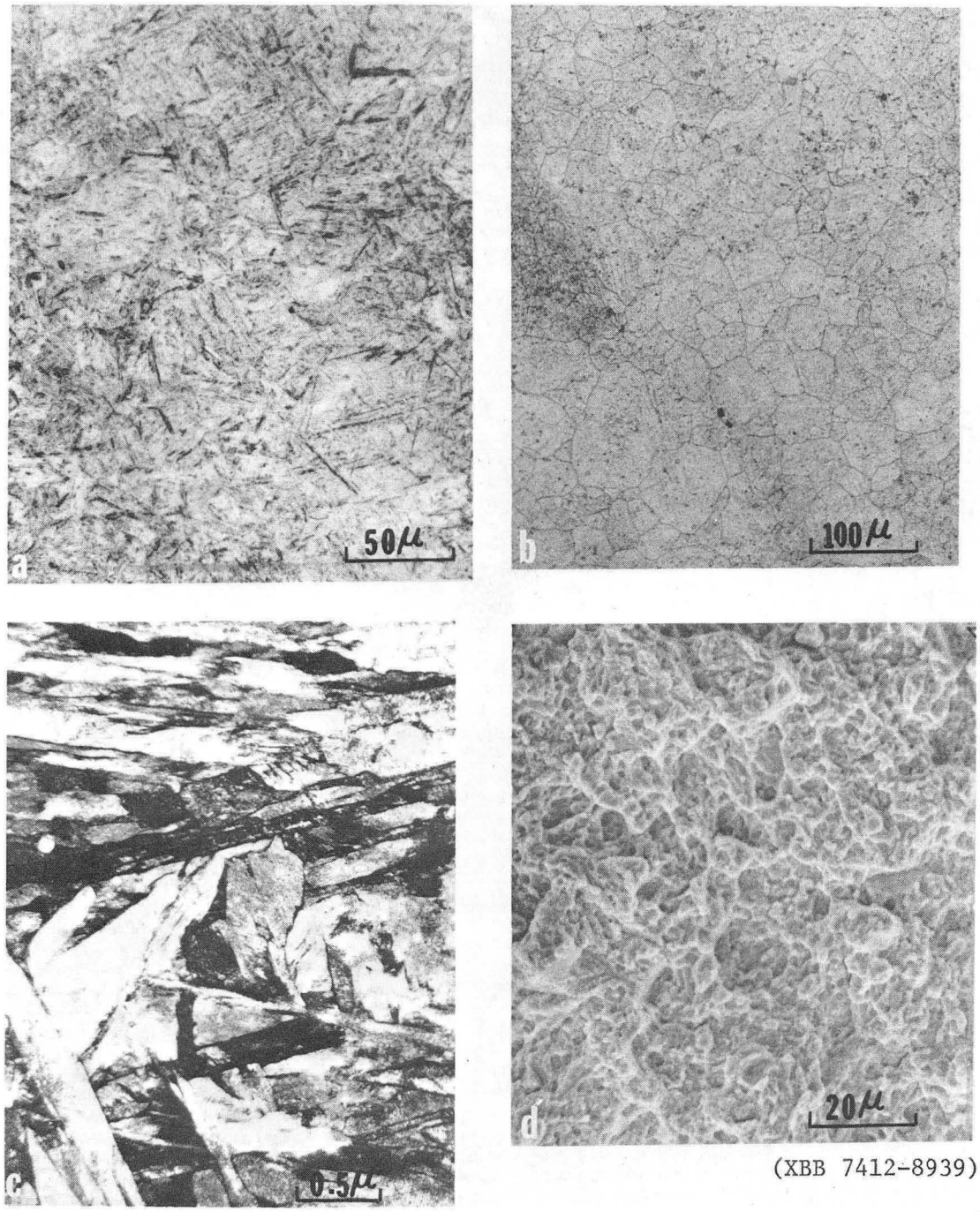

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Fig. 3.

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