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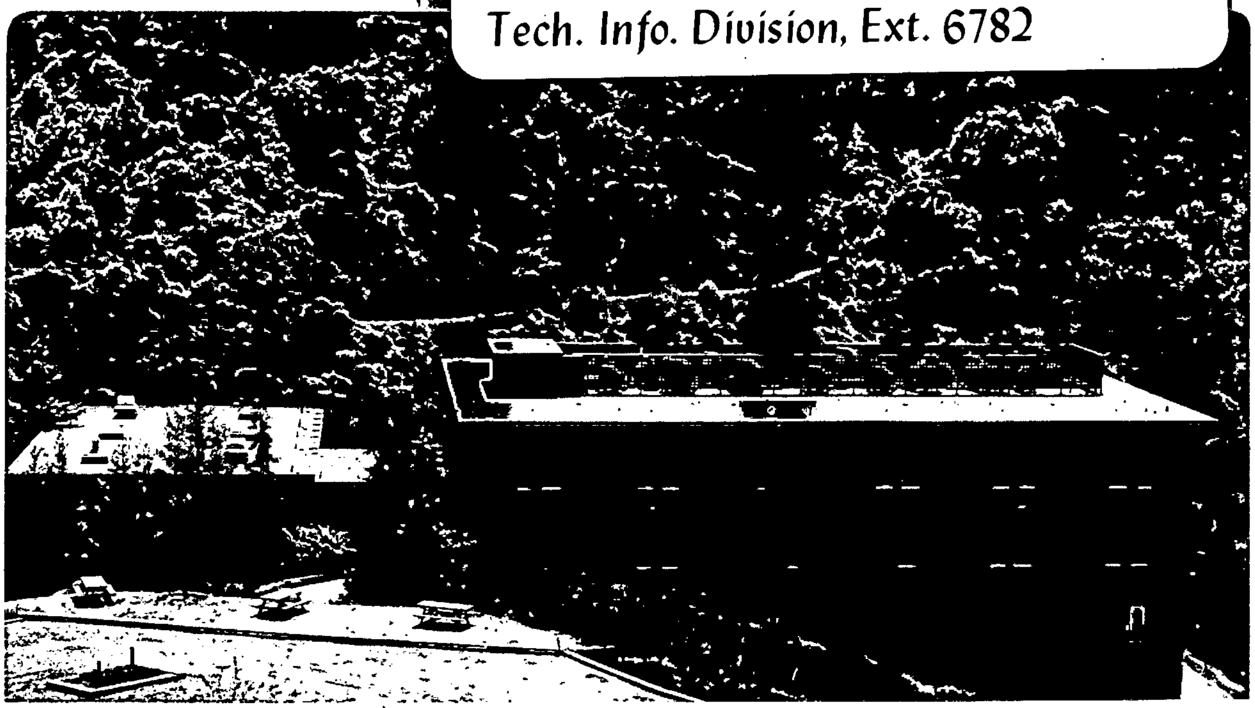

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ABSTRACT

Rapid solidification processing (RSP) has been carried out on an Fe/Cr/Mn/Mo/C alloy using both electron-beam melting and piston-and-anvil techniques. Preliminary TEM results show RSP produces a refined duplex microstructure of ferrite and martensite, with a typical ferrite grain size of 0.50 - 3.0 microns. This RSP microstructure is significantly different from that observed in the conventionally austenitized and quenched alloys--a lath martensitic microstructure with thin films of retained interlath austenite. The morphological change produced by RSP is accompanied by an increase in hardness from $48R_c$ to $61R_c$ (~ 480 to 720 VHN). It is intended to use electron-beam specimens to examine the potential beneficial effect of RSP upon sliding wear resistance and, by careful TEM studies, it will be possible to characterize the microstructure and its role in the hardness and wear behavior of the RSP alloy.

INTRODUCTION

Over the past few years, an extensive research program has been carried out at Berkeley on the microstructural and mechanical properties of Fe/Cr/Mn/C based alloys [1,2]. By optimizing both alloying element content and heat treatment, a refined microstructure has been produced with a corresponding high strength and high toughness. These properties are desirable for many engineering applications, in particular, mining and mineral processing, in which a strong, tough material is required with good wear resistance. The aim of the present work is to try and improve the sliding wear resistance of an Fe/Cr/Mn/Mo/C alloy, through RSP, and to characterize the wear resistance as a function of microstructure. This paper represents a preliminary transmission electron microscopy (TEM) examination of the RSP alloy.

EXPERIMENTAL DETAILS

The alloy, Fe-3wt%Cr-2wt%Mn-0.5wt%Mo-0.3wt%C which was supplied by Daido Steel company of Japan, was air melted into 20lb ingots and subsequently rolled to 1" thick by 25" wide bars. Homogenization of these bars occurred at 1200°C for 24 hrs. Specimens suitable for rapid solidification were cut from these bars. Prior to electron-beam melting, specimens were austenitized at 1100°C for 1 hr, oil-quenched and then tempered at 200°C for 1 hr with a final brine quench. This heat treatment was carried out in order for the substrate material to have the microstructure found previously to give the optimum combination of mechanical properties [2].

Rapid solidification was performed using a piston-and-anvil technique and electron-beam melting technique. Both techniques have been adequately described elsewhere [3,4]. The operational parameters for the electron-beam melting were 60 cycles beam frequency, 300 watts beam power, 5cm/sec linear velocity and 0.3mm focussed beam diameter.

Microhardness testing was carried out using a Leitz microhardness tester with a Vickers diamond indentor. The hardness values were converted to Rockwell hardness numbers, R_c, for ease of comparison with previous data obtained for the conventional material [2].

RSP specimens were mounted in thermosetting resin, polished and etched in 5% Nital, and examined optically by a Zeiss Metallograph microscope. Transmission electron microscopy was carried out on a Siemens 1A microscope on thin foils prepared by jet-polishing using a Chromic/Acetic Acid solution at room temperature.

RESULTS

Figures 1-4 show the typical microstructures observed in both conventionally heat-treated and RSP Fe/3Cr/2Mn/0.5Mo/0.3C alloy. In the conventional as-quenched alloy, the microstructure was dislocated lath martensite with fine films of interlath austenite [2]. The lath dimensions were ~0.5μm in width and ~25μm in length. The optimum prior austenite grain size (i.e. the smallest) after heat treatment was measured to be ~30μm. The as-quenched microstructure of the RSP alloy, however, was found to be a refined duplex microstructure of ferrite and martensite. The ferrite grain size was ~0.5-3.0μm in diameter, and the lath martensite was .1-.2μm in width and 0.5-5.0μm in length. The martensite packet size was generally observed to be equivalent to the prior austenite grain size (see Figure 3). Figure 2 shows a predominantly ferritic region in which the grains show evidence of deformation. This is not surprising since all the TEM foils were taken from RSP specimens solidified by the piston-and-anvil technique. Many of the ferrite grains also exhibited convoluted grain boundaries, and in some instances, facetting of the boundaries was observed. Similar types of grain-boundary configurations have been observed previously in both ferrous and non-ferrous RSP alloys [5,6]. A possible explanation for the presence of both convoluted and facetted grain-boundaries in RSP alloys is that the boundaries, which were very mobile at high temperatures, were "frozen-in" during the rapid cooling experienced, and therefore were unable to adopt a more uniform, low-energy configuration [6]. Figures 3 and 4 show examples of the martensitic microstructure found in the RSP steel. The microstructure was basically dislocated lath martensite. However, substructural twinning was observed within some laths and also apparently within the ferrite. The twinning was identified as $\{112\}_{\alpha}$ twinning by selected area diffraction. Similar microtwinning was found in the conventional material, as well as extensive formation of autotempered carbides, presumably because of the high M_s (320°C) of the alloy [2]. No such autotempered carbides were observed in the RSP alloy; this is almost certainly due to the relatively short time available during the rapid cooling for precipitation to occur. The martensite laths were generally parallel-sided, and although not enough TEM has been completed to confirm conclusively, it appears at present that a significant proportion of adjacent laths were either twin-related or separated by relatively high-angle boundaries. To date, no interlath austenite has been detected by TEM in the RSP alloy. There are two possible reasons for this: (i) The martensite microstructure was so fine that any austenite films present would have probably been less than 100Å thick, and therefore difficult to detect by conventional TEM techniques, and (ii) Many of the martensite laths were twin related and it has been shown previously that the interlath retained austenite occurs only between non-twin related laths [2].

Nevertheless, X-ray diffractometry had shown that austenite was present on a level of approximately 5% in the RSP specimens--whether as small, isolated, untransformed areas or as ultra-thin interlath films, it has not as yet been possible to determine. Microhardness testing of the RSP foils revealed a hardness of 720 $\pm$ 60 VHN (~ 61R_c) as compared to a hardness of ~480 VHN (48R_c) for the conventional austenitized and quenched alloy.

Recently rapid solidification through electron-beam melting (EBM) was carried out on the Fe/3Cr/2Mn/0.5Mo/0.3C alloy. Figure 5 shows an optical micrograph of the as-glazed surface and Figure 6 shows a cross-section through an EBM specimen. The main relief features of the as-glazed surface were surface martensite, slip-lines, and periodic undulations produced during pass overlap. The melt zone depth of the EBM specimens ranged between 60-120 microns. The microstructure of this region was predominantly martensitic with apparently only a small amount of microstructural refinement. It is possible, however, that SEM and TEM will show that significant refinement has occurred, albeit on a smaller scale than that produced by piston-and-anvil technique.

DISCUSSION AND CONCLUSIONS

RSP of Fe/3Cr/2Mn/0.5Mo/0.3C results in a refined duplex microstructure of ferrite and martensite. This differs from the conventional austenitized and quenched microstructure of dislocated lath martensite with fine interlath films of retained austenite. Such changes in the as-quenched microstructure are often found in RSP, and are a direct consequence of the high rate of solidification and subsequent solid-state cooling [7]. In the present case it is not obvious through TEM results whether the ferritic phase is the high temperature form (δ) or the low temperature form (α). Figure 7 is a binary cross-section of the ternary Fe/Cr/C system in which the chromium content is five weight percent. This level is similar enough to that in the alloy studied to allow the use of this binary section in understanding the solidification and cooling reactions. However, any extrapolations drawn are limited by the knowledge that Figure 7 is an equilibrium phase diagram and that RSP is a process far from equilibrium. On cooling from the liquid, an Fe/5Cr/0.3C alloy would first pass through the $\gamma + L$ two phase region, with possibly some δ-ferrite forming, too. Once below ~1400°C, the alloy would be in the Y-stable region until ~800°C. Below this, ferrite becomes stable once more with martensite transformation occurring at 320°C. It is well-known that RSP can produce large undercoolings below the liquidus temperature (up to 200°C), often large enough for hypercooling to occur [8]. Hypercooling is the condition that exists when the undercooling is far enough below the solidus temperature to ensure that any temperature increase during recalescence will not bring the solid/liquid interface temperature up to that of the solidus. When this occurs, solidification takes place without diffusion, i.e. a "massive" transformation. When considering the Fe/Cr/C system, it is likely that an undercooling of ~200°C would be sufficient for hypercooling to occur, resulting in the formation of massive austenite. How the austenite eventually transforms to both α-ferrite and martensite, however, is difficult to understand. It will be necessary to use more sophisticated TEM techniques to obtain detailed information of the microstructure. It should be possible using convergent beam and high-voltage electron microscopy to detect the presence and morphology of the austenite and by using scanning-transmission electron microscopy, to determine the elemental partitioning. This knowledge should provide a good clue to what transformations are indeed taking place during the rapid solidification of Fe/3Cr/2Mn/0.5Mo/0.3C. The increase in hardness produced by RSP has often been observed and is generally considered to be a result of the microstructural refinement, in addition to the deformation produced during piston-and-anvil RSP. The EBM results are still somewhat tentative, but it has been shown previously that self-substrate RSP of ferrous alloys produce similar microstructural changes and increased hardness as that obtained by other RSP techniques [9], [7]. It is hoped that the future planned work in this area will show a similar trend, and that these properties will have a beneficial effect on the sliding wear behavior of the material.

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References

[1] G. Thomas, Met. Trans. 2, 2373 (1972).

[2] B. V. N. Rao and G. Thomas, Proceedings of the Int. Conf. on Martensitic Transformations, Cambridge, Mass. (1979).

[3] B. G. Lewis, D. A. Gilbert and P. R. Strutt, Proceedings of 2nd Int. Conf. on Rapid Solidification Processing, Reston, Virginia, March 1980 (Claitor's Publishing Division, Baton Rouge) p. 221.

[4] P. Duwez, Progress in Solid State Chemistry, (Pergamon Press, New York 1966), Vol. 3, p. 377.

[5] J. J. Rayment, O. Ashiru and B. Cantor, Proceedings of Int. Conf. on Solid-Solid Phase Transformations, Pittsburgh, PA, Aug. 1981. LBL No. 13212

[6] J. Perkins, J. J. Rayment, B. Cantor and R. W. Cahn, Scripta Met. 15, 771 (1981).

[7] J. J. Rayment and B. Cantor, Met. Trans. 12A, 1557 (1981). LBL No. 12121.

[8] M. Cohen, B. H. Kear and R. Mehrabian, see Ref. [3] p. 1.

[9] M. Tuli, P. R. Strutt, H. Nowotny and B. H. Kear. Proceedings of the 1st

Int. Conf. on Rapid Solidification Processing, Reston, Virginia, November 1977 (Claitors Publishing Division, Baton Rouge), p. 112.

[10] Metals Handbook, Vol. 8, Amer. Soc. Metals.

Figure Captions

Fig. 1. The lath martensitic microstructure of conventionally heat-treated Fe/Cr/Mn/Mo/C alloy. [Courtesy of M. Sarikaya].

Fig. 2. The ferritic microstructure observed in RSP Fe/Cr/Mn/Mo/C alloy.

Fig. 3. The lath martensitic microstructure of RSP Fe/Cr/Mn/Mo/C alloy; a martensite packet is observed to be equivalent to a prior austenite grain.

Fig. 4. The lath microstructure of RSP Fe/Cr/Mn/Mo/C alloy; the laths are sometimes twin-related and often contain sub-structural twinning.

Fig. 5. The as-glazed surface of electron-beam melted Fe/Cr/Mn/Mo/C alloy.

Fig. 6. A cross-section of electron-beam melted Fe/Cr/Mn/Mo/C alloy.

Fig. 7. The pseudo-binary phase diagram of Fe/Cr/C [10].

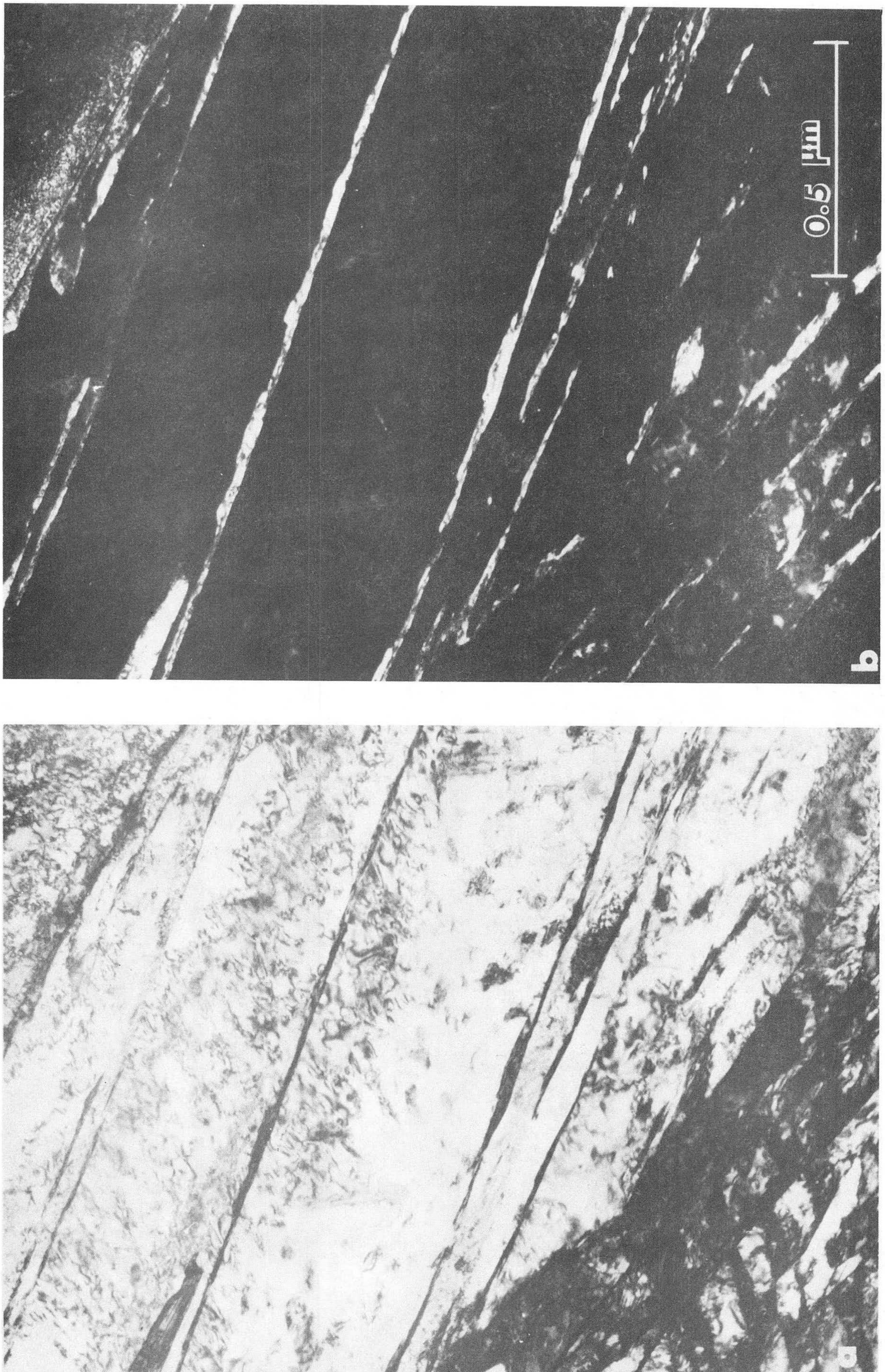

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Fig. 1

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Fig. 2

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Fig. 3

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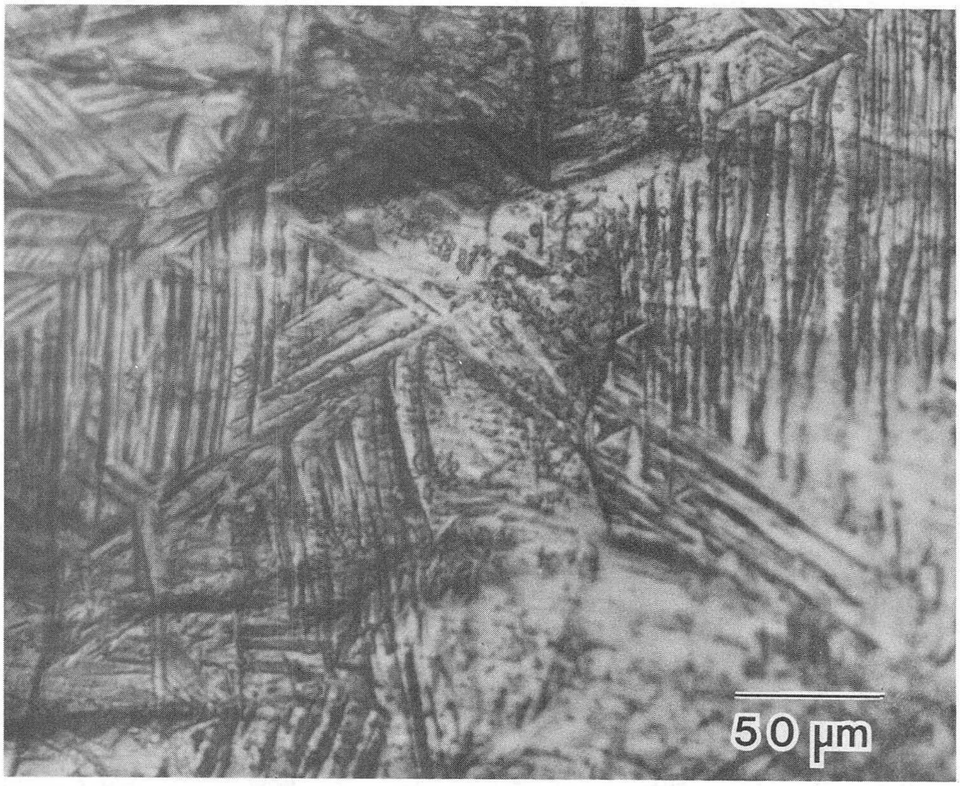
Fig. 4



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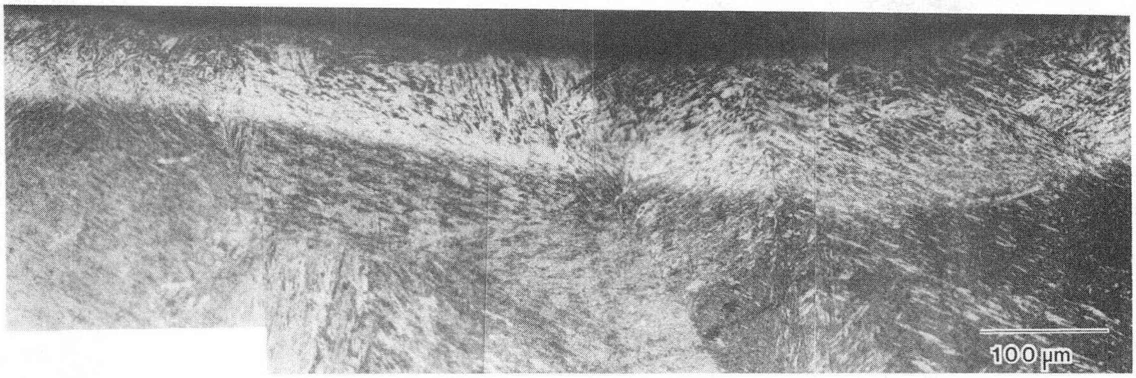

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Fig. 6

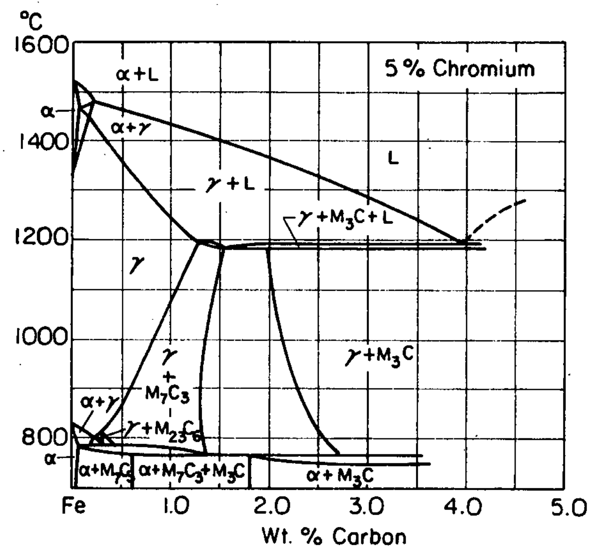

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Fig. 7
ref(10)

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