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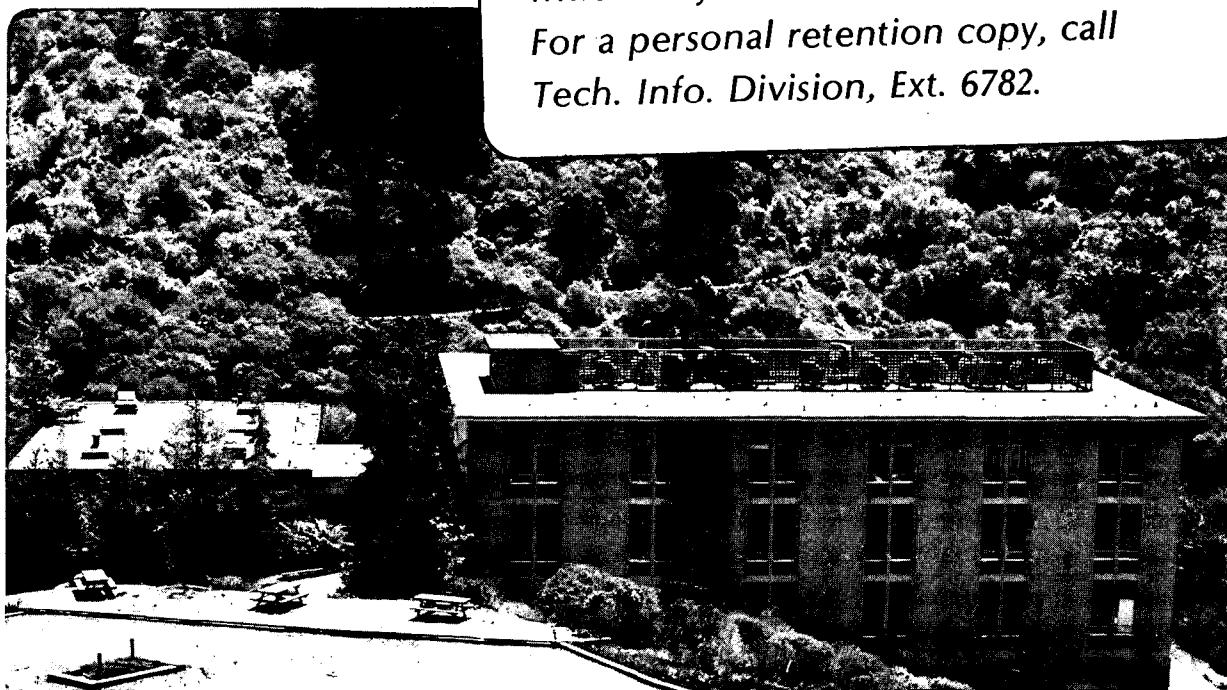
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N.J. Kim and G. Thomas

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Structure and Properties of As-Hot-Rolled Fe/1.5Mn/.06C Steel:
Potential for Line Pipe Application

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An investigation has been made to develop a simple duplex steel for line pipe application. Controlled rolling of an Fe/1.5Mn/.06C alloy followed by direct quenching produced a duplex ferrite-bainite structure in which the coarse upper bainite regions were uniformly distributed within a fine grained ferrite matrix. The microstructure and mechanical properties of this duplex structure were strongly influenced by the processing variables. Decreasing the finish rolling temperature improved both tensile and impact properties. This was due mainly to a refinement of the ferrite grain size. There was an abrupt increase in the yield strength and a small increase in the ductile-to-brittle transition temperature as a result of cold working (pipe forming) because of the steel's continuous yielding behavior and high initial work hardening rate. The mechanical properties attained in this duplex steel are attractive for low temperature line pipe applications.

INTRODUCTION

The discovery of oil and natural gas in severe environments such as arctic areas and offshore has resulted in more demanding mechanical property requirements for line pipe steels. These steels should have not only high strength but also good low-temperature toughness. Moreover, the improvement in mechanical properties should not impair the weldability of line pipe steels. This requires the judicious control of all possible strengthening mechanisms since each has a different influence on toughness and weldability.

Controlled rolled steels having ferrite-pearlite microstructures have been used for line pipe because it is possible to attain a relatively good combination of these properties. Such conventional steels attain their strength mainly from grain refinement, solid solution hardening, and precipitation hardening resulting from microalloying additions^{1,2}. However, with the exception of grain refinement, most of the routes taken to increase strength have tended to diminish toughness. Also, consideration of weldability puts an upper limit on carbon and alloying contents. These facts bring about a difficulty in obtaining higher strength with suitable toughness and weldability in conventional ferrite-pearlite steels. One of the answers to this problem is the use of transformation strengthening such as in acicular ferrite or bainitic steels. These steels have a good combination of strength and toughness due to a high dislocation density and fine effective grain size of the matrix phase. Another possible approach might be the use of the concept of duplex steels in that separate constituents are responsible for different property requirements. "Dual-phase" or "duplex" steels are a relatively new class of high strength low alloy (HSLA) steels

containing two or more phases, predominantly ferrite and martensite with small amounts of bainite and/or retained austenite depending on alloy content and processing^{3,4,5}. Compared with conventional ferrite-pearlite HSLA steels, the incorporation of a strong second phase in the soft ferrite matrix results in important changes in the mechanical behavior of the material: namely, continuous yielding, a high initial work hardening rate and excellent combinations of ultimate tensile strength and ductility. Although most of the interest in duplex steels to date has related to automotive applications, the stress-strain characteristics of duplex steels are also of great merit for general structural applications such as line pipe. The work reported here is aimed at developing the duplex structure by a controlled rolling process and investigating its mechanical properties for possible low temperature line pipe application. The basic concept underlying this research is that good impact toughness can be obtained primarily by grain size control while maintaining the desired duplex structure for high strength.

MATERIALS AND EXPERIMENTAL PROCEDURES

The chemical composition of the alloy used in this investigation is given in Table I. The alloy was air melted in the form of a 34kg ingot. The ingot was sand-blasted and homogenized in argon at 1200°C for 24 hours. It was upset and cross-forged at 1100°C into 30 mm x 64 mm slabs. All of the above treatments were followed by air cooling. The 3cm thick slabs were soaked at 1100°C for 45 minutes, hot rolled 35% in one pass at 1000°C and then rolled 50% in one pass in the temperature range of 750°C to 950°C and directly quenched into water. The temperature was monitored by thermocouples inserted at the midpoint of the plate thickness.

The room temperature tensile properties were determined using cylindrical tensile specimens of 13 mm gauge length and 3 mm gauge diameter. The loading direction was kept consistent with the rolling direction of the original plate. Testing was done on an Instron machine with a cross-head speed of 0.5 mm/min. Total elongations were determined by measuring the 13 mm marks on the gauge before and after testing. Other mechanical properties were determined from the stress-strain curves obtained from the Instron recorder.

Charpy tests were done on 3/4 subsize V-notch specimens in both the longitudinal and transverse directions. Low temperature tests were performed following the ASTM-23-72 specifications⁶. Subzero temperatures were obtained using a mixture of isopentane and liquid nitrogen. The ductile-brittle transition temperature was taken as the temperature corresponding to the midpoint of the upper and lower shelf energies. The impact energy curves were obtained by taking an average of at least 3 tests. Specimens for transmission electron microscopy were prepared and observed in the usual way¹⁵.

RESULTS

A. Microstructure

The structure just prior to rolling, obtained by quenching after solution treatment (1100°C, 45 min), is shown in Fig. 1(a). The prior austenite grain size of this structure was about 250 μm . After the first rolling of 35% reduction at 1000°C, the austenite grain size was decreased to 80 μm (Fig. 1(b)). Complete recrystallization of austenite is evident. After the second pass, complete recrystallization of austenite occurred down to a finish rolling temperature of 850°C. Deformation below this temperature produced mixed austenite structure as shown in Fig. 2. In these lower temperature ranges, recrystallization of austenite became retarded, thus promoting the elongation of most of the austenite grains into pancaked shapes with only some of the austenite grains being recrystallized. However, complete non-recrystallization of austenite was not observed even when the finish rolling was performed just above the A_{r3} temperature (750°C). Figure 3 shows the microstructures of the steel directly quenched from the finish rolling temperature. The general microstructural features are coarse second phase particles surrounded by fine ferrite grains. There was a refinement of ferrite grain size with decrease in the finish rolling temperature as shown in Table 2.

Changes in the finish rolling temperature also affected the morphology of the second phase. The volume fraction and size of the second phase decreased with the reduced finish rolling temperature (Table 2). Also, the distribution of the second phase became more uniform. A transmission electron micrograph of the second phase region is shown in Fig. 4. It is noted from this micrograph that the second phase has the char-

acteristicstypical of classical high carbon upper bainite⁷. When the finish rolling temperature is changed, no significant change occurred in the nature of the second phase except that a small amount of acicular ferrite was found in specimens finish rolled above 950°C (Fig. 5). The substructure of the ferrite was similar in all cases; its significant feature was the occurrence of the aligned, discontinuous carbide precipitates which were located only in the immediate vicinity of the ferrite/bainite boundaries (Fig. 6). Precise identification of the particles was not attempted in this study, but they were probably cementite. The morphology of the carbide precipitation was similar to that of the interphase precipitation which was observed in many steel systems containing strong carbide forming elements⁸⁻¹⁰. This type of precipitation is also observed in several plain carbon steel systems^{11,12}. The ferrite matrix, which was located far away from the bainite region, showed a fairly extensive precipitation of fine particles as shown in Fig. 7. These particles were identified as cementite. The association of the cementite with dislocations suggests that this precipitation occurred from supersaturated ferrite.

B. Mechanical Properties

The mechanical properties determined for these duplex ferrite-bainite steels are summarized in Table 3, and are plotted in Figure 8 as a function of the finish rolling temperature. The general trends are, that as the finish rolling temperature was reduced, the strength increased and the ductile-to-brittle transition temperature (DBTT) decreased. Reducing the finish rolling temperature caused no appreciable change in elongation ductility. Correlation of the changes in

mechanical properties with microstructure indicates that the amount of bainite seems to be minor, and that the important factor is the refinement of ferrite grain size. However, as the finish rolling temperature was **lowered**, the opposing effects of ferrite grain size refinement and decreased volume fraction of bainite became offset so that the rate of change in mechanical properties (specifically, strength) became small.

While the ductile-to-brittle transition temperature is mainly determined by the grain size of the material, the impact energy is influenced by other factors such as the amount and shape of the second phase, cleanliness of the steel and the absence of interstitials. Figure 9 shows the variation of impact energy with testing temperature. The upper shelf energy increased consistently with decreasing deformation temperature. This behavior is presumably due to the decreased volume fraction and size of the second phase. A limited number of Charpy impact test results for transverse specimens indicated that, although the direction of impact testing had an effect on the impact energy values, it had no effect on the ductile-to-brittle transition temperature. The decrease in upper shelf energy for transverse specimens might have been due to the influence of inclusions and/or texture, but no attempt has been made here to analyze these possible effects quantitatively.

DISCUSSION

The main purpose of controlled rolling is to develop a fine, uniform ferrite grain size in the final structure. It is well known that the austenite grain boundaries are the preferred nucleation sites for austenite-to-ferrite transformation. Deformation bands also provide nucleation sites. Hence most emphasis in controlled rolling has been put on obtaining fine recrystallized austenite grains or very thin "pancaked" non-recrystallized austenite grains during controlled rolling. Controlled rolling is often divided into two stages, i.e., rolling above the recrystallization temperature and rolling below the recrystallization temperature.

The response of plain carbon steel to controlled rolling is quite different from that of microalloyed steels, and has a strong effect on the structure and properties of its final products. In microalloyed steels, elements such as Nb and V retard the recrystallization of austenite, thus deformation of austenite can be performed without the occurrence of any recrystallization. On the other hand, in plain carbon steels, formation of fully non-recrystallized austenite grains is not possible because recrystallization of austenite occurs at temperatures down to the A_{r3} temperature; deformation of austenite just above A_{r3} produces a mixed structure of elongated austenite and recrystallized austenite. During high temperature rolling of plain carbon steel, recrystallization of austenite occurs rapidly and the recrystallized austenite grains become coarser with time due to the absence of fine precipitates to inhibit grain growth in the stable austenite range. As the finish rolling temperature is decreased, the recrystallized austenite grains become finer and the volume fraction of elongated

austenite increases, thus producing more nucleation sites. These structural changes in austenite are responsible for the formation of coarse second phase particles within fine ferrite grains, as explained below. It is well known that coarse recrystallized austenite grains promote the formation of coarse low temperature products, such as bainite and martensite. Even when the austenite grains are fully non-recrystallized, a coarse transformation product can result due to non-uniform distribution of nucleation sites. Kozasu et al.¹³ claimed that not all deformation bands have the same ferrite nucleation potential and some bands have a poor nucleation capability. Also, Fukuda et al.¹⁴ found that even after a very large deformation it was impossible to obtain a uniform distribution of deformation bands, and there existed regions which had "poor-nucleation capabilities". Upon cooling, ferrite forms first at the easiest nucleation sites such as prior austenite grain boundaries and/or where the deformation is highest.

The results of the present work show that the duplex ferrite-bainite steel attains attractive mechanical properties when the second phase microstructure is of upper bainite. It has been generally believed that upper bainite should be avoided in the final structure to obtain optimum strength-toughness combinations. This is because in upper bainite, large interlath carbides crack to form super-critical defects. These cleavage cracks, once initiated, are not obstructed by the low-angle bainitic ferrite boundaries but only by bainite packet

boundaries or prior austenite grain boundaries. Thus, cracks propagate rapidly. However, this effect is certainly not an important factor in this duplex system, in which cracks were nucleated in the polygonal ferrite matrix before fracture occurred in the upper bainite (Fig. 10). This is in agreement with other recent studies of a number of dual-phase steels¹⁵, in which cracking was found to initiate always in the ferrite.

The advantages of the duplex ferrite-bainite structure are its continuous stress-strain behavior and high initial work hardening rate. These effects will result in an additional increase in yield strength during pipe forming operations, whereas in pipe made from conventional ferrite - pearlite structures¹⁶⁻¹⁸, such operations cause a 10 to 15% loss in yield strength due to yield point elongation and the Baushinger effect. Fig. 11 shows the changes in mechanical properties resulting from cold reduction. When pipe is formed by the UOE process, the yield strength cannot be estimated directly from the stress-strain curve obtained from uniaxial tensile tests because the deformation of material by bending and expansion is mostly under plane strain conditions. Thus, the above results (Fig. 11) can be used to give only an approximation of the mechanical properties possible in the Fe/Mn/C duplex pipe formed by the UOE process. However, it is noted that the ductile-brittle transition temperature increases only slightly while there is an abrupt increase in yield strength after small amounts of cold reduction. This clearly demonstrates the potential of the as-hot-rolled ferrite-bainite structure for line pipe applications.

SUMMARY

An investigation has been made to develop a simple duplex steel for line pipe application. The results obtained are summarized as follows:

1) The Fe/1.5Mn/0.06C alloy is controlled rolled followed by direct quenching, to obtain a duplex ferrite-bainite structure in which coarse upper bainite regions are uniformly distributed within a fine grained ferrite matrix.

2) Decreasing the finish rolling temperature resulted in a finer ferrite grain size with a decrease in the volume fraction and size of the second phase. These structural changes were accompanied by an improvement in tensile and impact properties.

3) The mechanical properties of this duplex ferrite-bainite steel are shown to be attractive for line pipe application. Subsequent cold working causes an abrupt increase in yield strength with a small increase in ductile-brittle transition temperature. This is due to its continuous yielding behavior and high initial work hardening rate.

ACKNOWLEDGEMENTS

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FIGURE CAPTIONS

1. Optical micrographs showing prior austenite grain size; a) before rolling, b) after 35% deformation at 1000°C. 5% Nital etch.
2. Optical micrograph of mixed austenite structure (finish rolled at 800°C) showing elongated austenite grains and recrystallized austenite grains. Etchant: 4% picric acid + $K_2S_2O_5$ (supersaturated).
3. Optical micrographs of the duplex ferrite-bainite structure for various finish rolling temperatures; a) 750°C, b) 900°C. 5% Nital etch.
4. TEM of upper bainite region; a) bright field, b) dark field of interlath carbide. Finish rolled at 800°C.
5. Acicular ferrite developed in the specimen finish rolled at 950°C.
6. Morphology of interphase carbide precipitation in the vicinity of the ferrite/bainite interface. Finish rolled at 800°C.
7. Bright field (a) and dark field (b) of cementite precipitation in the ferrite matrix. Finish rolled at 800°C.
8. Variation of the mechanical properties of duplex steel with finish rolling temperature.
9. Ductile-to-brittle transition curves of duplex steel with various finish rolling temperature.
10. SEM showing microcrack formation in polygonal ferrite matrix.
11. Variation of the mechanical properties of duplex steel (finish rolled at 850°C) with cold working.

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Table I. Alloy Composition (wt. pct.)

Alloy	C	Mn	Si	Al	N	P	S	Fe
Fe/1.5Mn/.06C	0.06	1.51	0.05	0.041	0.005	0.006	0.005	Bal.

Table II. Metallographic Data of As-Hot Rolled Fe/1.5Mn/.06C Steel

Finish Rolling Temp (°C)	Ferrite Grain Size (μm)	Vol.%	Bainite Size (μm)
750	5.2	22	15.5
800	6.8	25	25.1
850	8.0	27	31.2
900	10.2	29	37.5
950	14.5	30	38.0

Table III. Mechanical Properties of Duplex Ferrite
- Bainite Steel With Finish Rolling Temperature

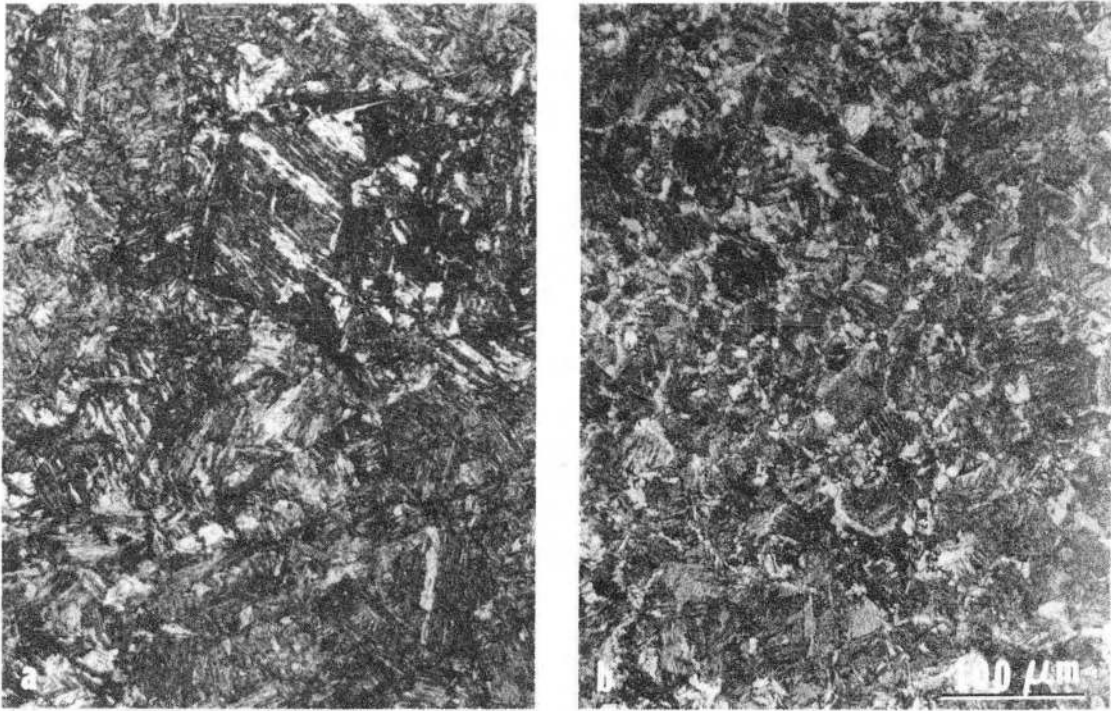
Finish Rolling Temp (°C)	DBTT (°C)	YS (.2% Offset)		UTS		e [*]	e [*]
		(ksi)	(Mpa)	(ksi)	(Mpa)	(%) ^u	(%) ^t
750	-122	65.0	448.2	93.2	642.6	17.2	30.9
800	-110	65.1	448.9	92.8	639.9	17.5	31.2
850	-109	64.4	444.0	91.0	627.4	17.0	30.8
900	- 98	61.6	424.7	86.6	597.0	16.5	30.1
950	- 90	55.6	383.4	80.0	551.5	17.8	31.5

* Gauge length: 13 mm

Table III. Mechanical Properties of Duplex Ferrite
- Bainite Steel With Finish Rolling Temperature

Finish Rolling Temp (°C)	DBTT (°C)	YS (.2% Offset)		UTS		e [*] (%) [†]	e [*] (%) [†]
		(ksi)	(Mpa)	(ksi)	(Mpa)		
750	-122	65.0	448.2	93.2	642.6	17.2	30.9
800	-110	65.1	448.9	92.8	639.9	17.5	31.2
850	-109	64.4	444.0	91.0	627.4	17.0	30.8
900	- 98	61.6	424.7	86.6	597.0	16.5	30.1
950	- 90	55.6	383.4	80.0	551.5	17.8	31.5

* Gauge length: 13 mm



XBB 814-3213

Fig. 1

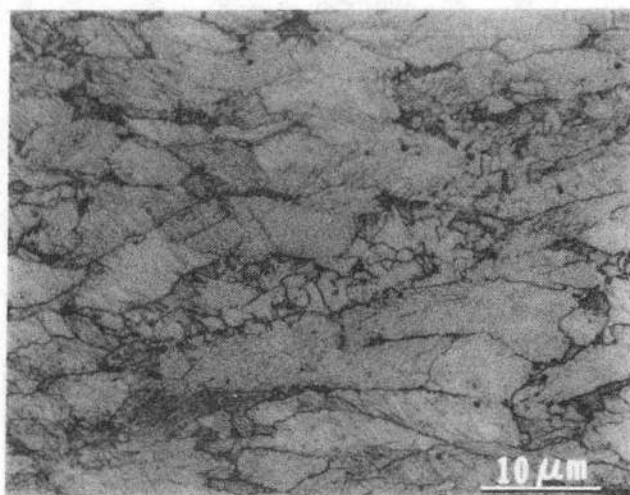
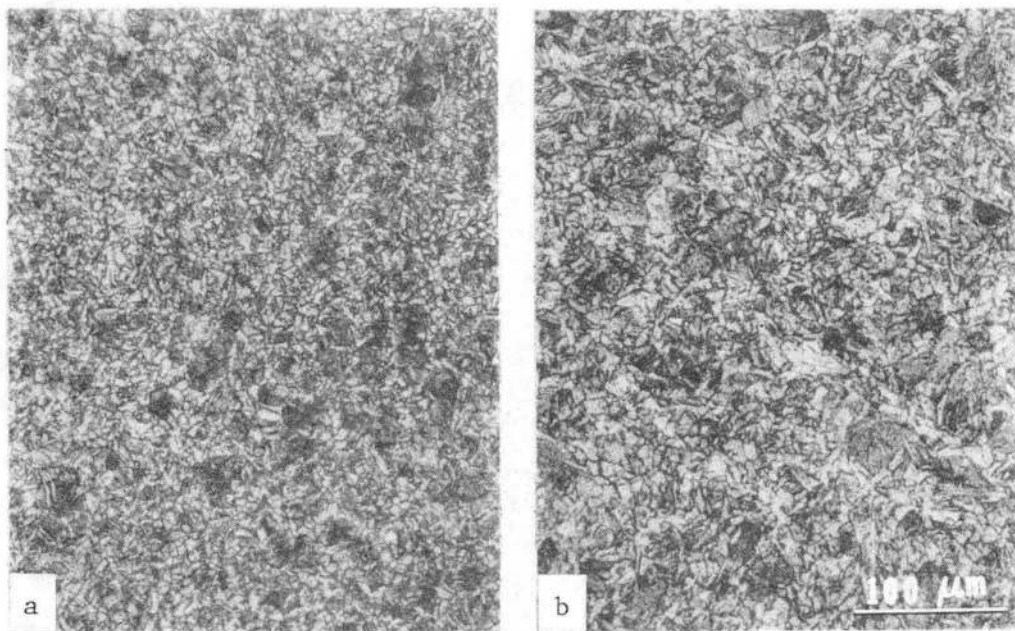


Fig. 2

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Fig. 3

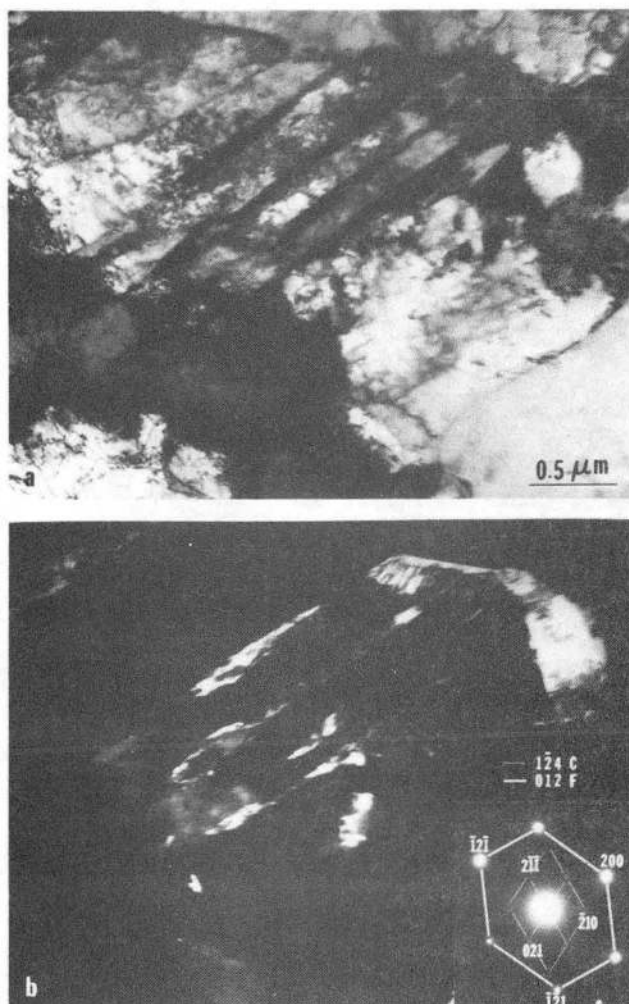


Fig. 4

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Fig. 5

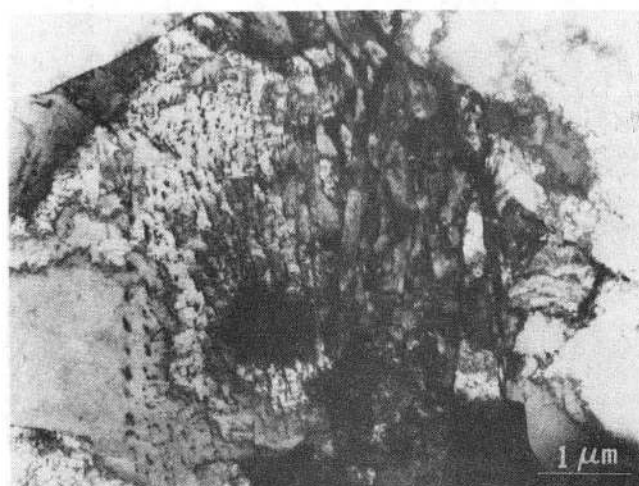


Fig. 6

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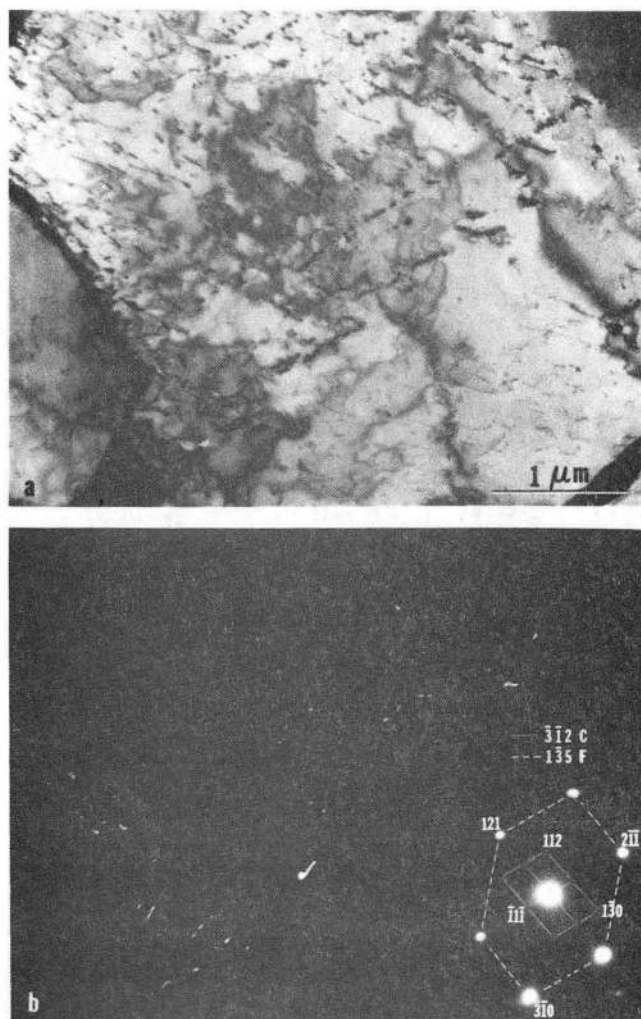


Fig. 7 XBB 809-11370

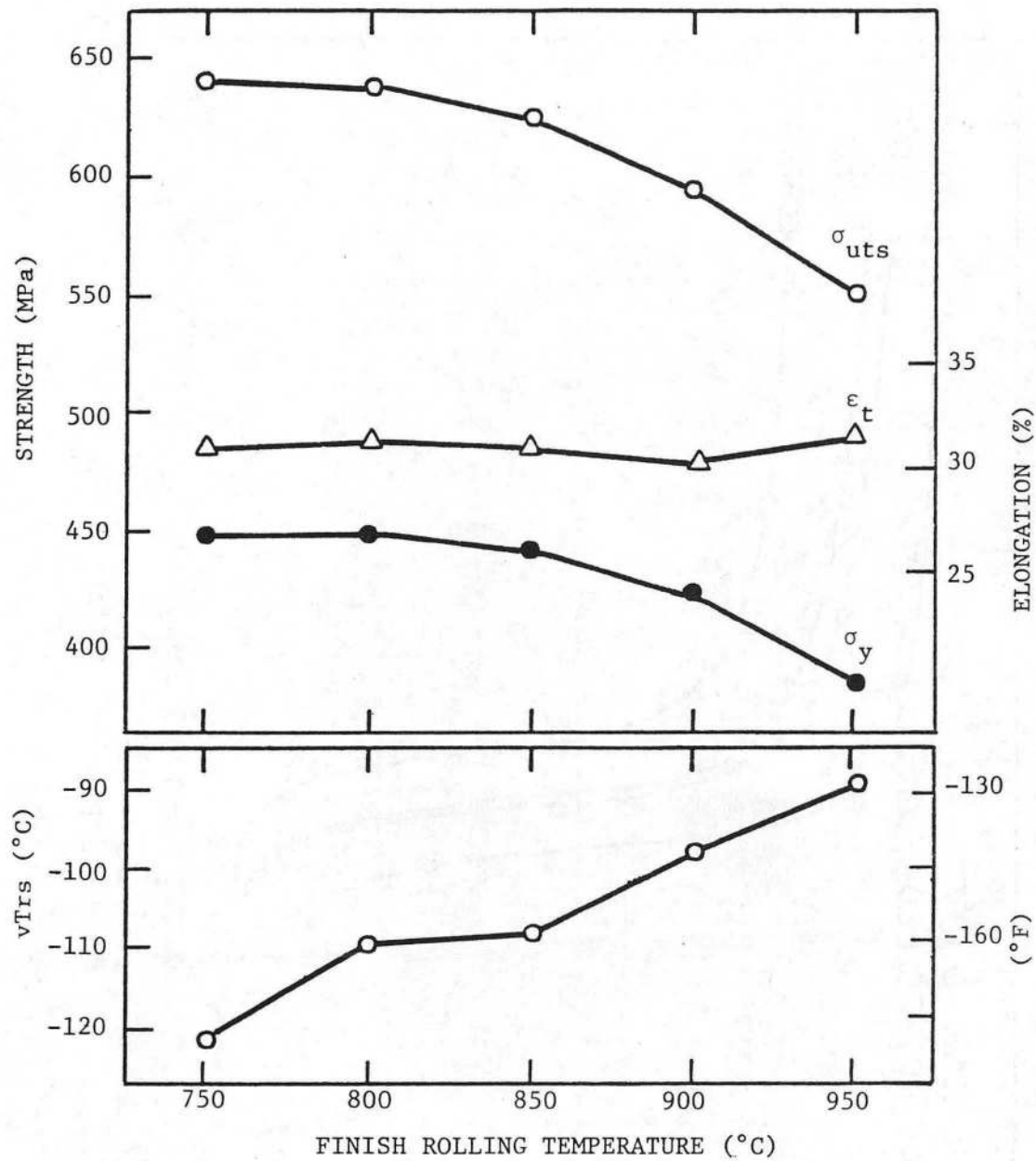


Fig. 8

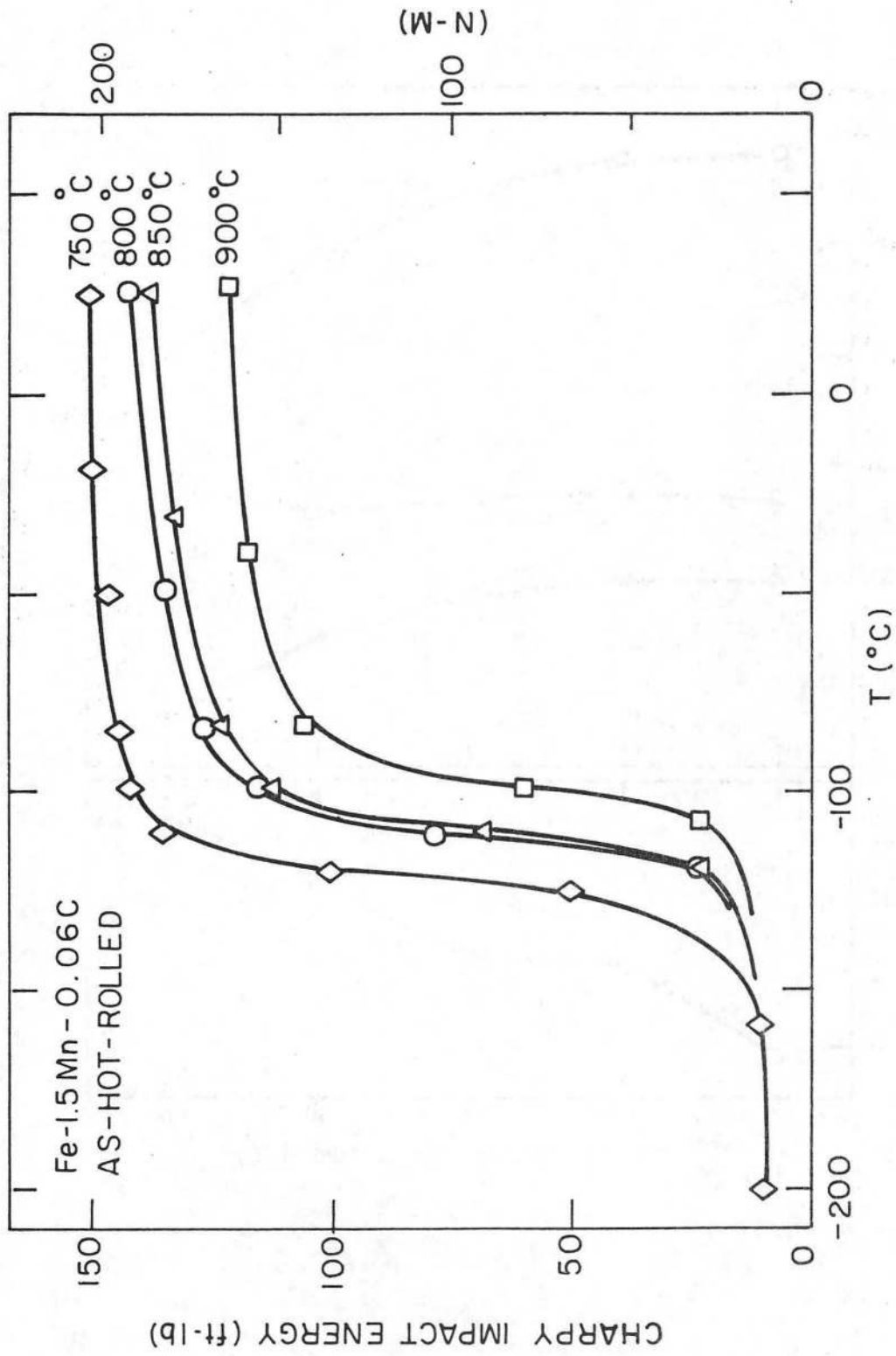
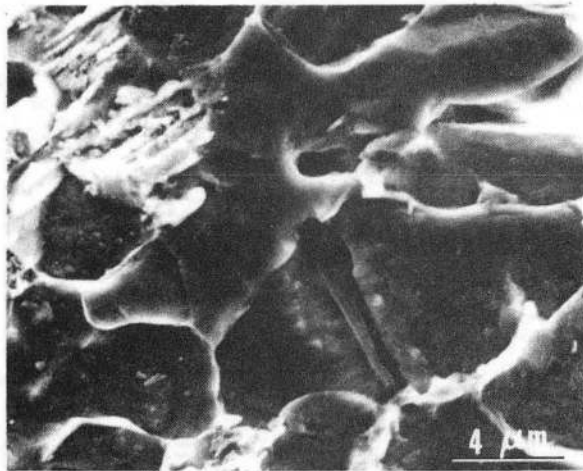


Fig. 9



XBB 8311-10287

Fig. 10

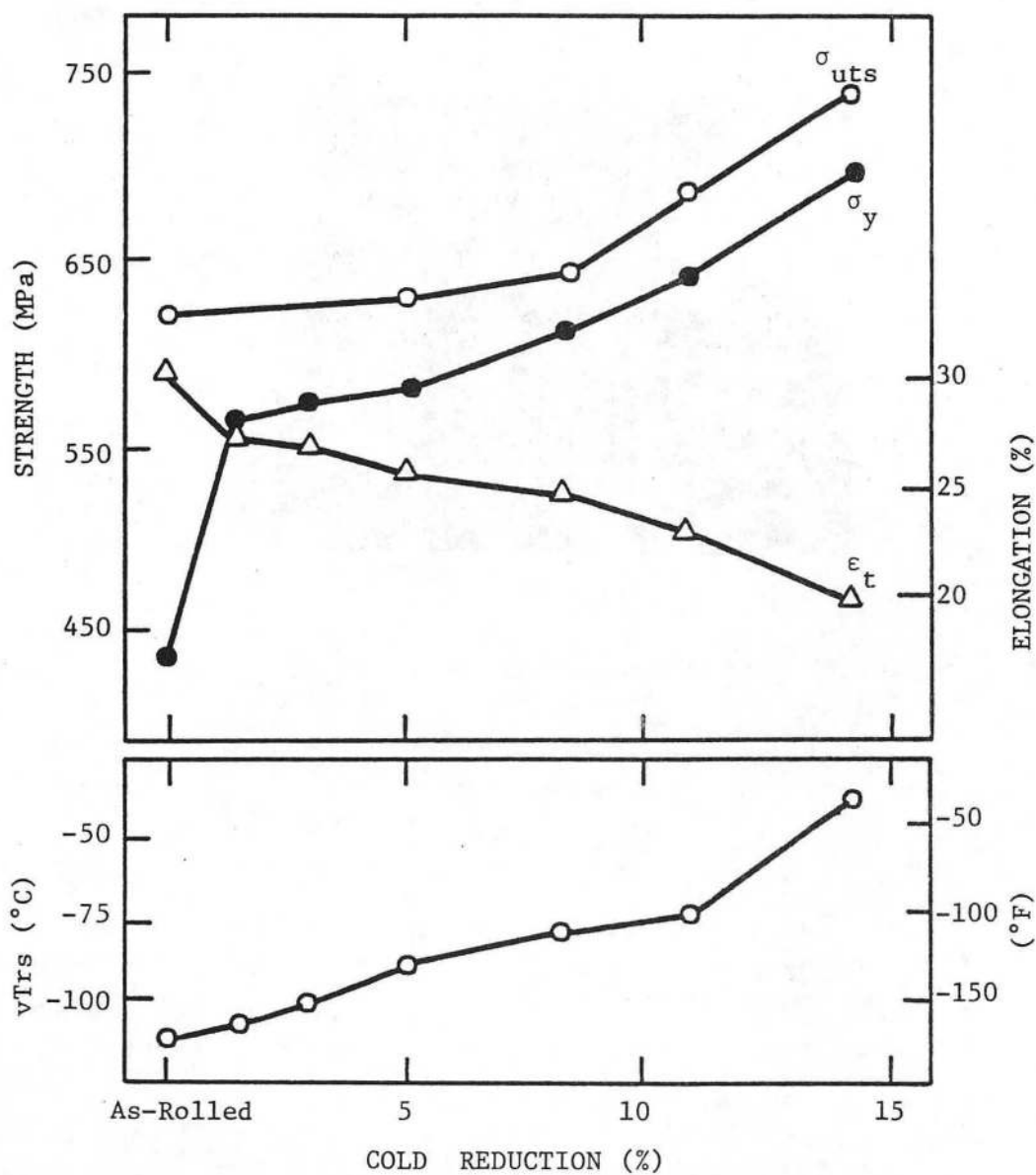


Fig. 11

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