Lawrence Berkeley National Laboratory

Recent Work

Title

THE STRUCTURE AND MECHANICAL PROPERTIES OF LOWER BAIHITE IN Fe/4Cr/O.4c ALLOY

Permalink https://escholarship.org/uc/item/0bm165qv

Author Miller, Robert Walter.

Publication Date 1974-09-01

LBL-3104 c.0

LBL-3104

THE STRUCTURE AND MECHANICAL PROPERTIES OF LOWER BAINITE IN Fe /4Cr/0.4C ALLOY

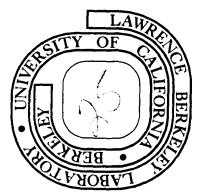
Robert Walter Miller (M.S. thesis)

September, 1974

Prepared for the U. S. Atomic Energy Commission under Contract W-7405-ENG-48

TWO-WEEK LOAN COPY

This is a Library Circulating Copy which may be borrowed for two weeks. For a personal retention copy, call Tech. Info. Division, Ext. 5545



DISCLAIMER

This document was prepared as an account of work sponsored by the United States Government. While this document is believed to contain correct information, neither the United States Government nor any agency thereof, nor the Regents of the University of California, nor any of their employees, makes any warranty, express or implied, or assumes any legal responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by its trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or the Regents of the University of California. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof or the Regents of the University of California.

THE STRUCTURE AND MECHANICAL PROPERTIES OF LOWER BAINITE IN Fe/4Cr/0.4C ALLOY

	contents	
ABSTR	ACT	
Ι.	INTRODUCTION	
	A. Morphology of Martensite	
	B. Morphology of Bainite	
II.	EXPERIMENTAL PROCEDURE	
III.	RESULTS	
	A. Time-Temperature-Transformation Diagram	
•	B. Mechanical Properties	
	C. Optical Microstructure	
•	D. Electron Microscopy	
	E. X-Ray Analysis	
	F. Electron Fractography	
IV.	DISCUSSION	
·	A. Mechanical Properties	
۷.	CONCLUSIONS	
REFERI	ENCES	
ACKNO	WLEDGEMENTS	
TABLE		
FIGUR	E CAPTIONS	
FIGUR	ES	

THE STRUCTURE AND MECHANICAL PROPERTIES OF LOWER BAINITE IN Fe/4Cr/0.4C ALLOY

Robert Walter Miller

Inorganic Materials Research Division, Lawrence Berkeley Laboratory, and Department of Materials Science and Engineering, College of Engineering; University of California, Berkeley, California

ABSTRACT

The structure and mechanical properties of lower bainite were investigated in an Fe/4Cr/0.4C alloy. An earlier study found that an Fe/4Cr/0.35C alloy had very favorable mechanical properties in the untempered martensite state. However, in a similar alloy with slightly more carbon, Fe/4Cr/0.4C, the martensite structure was very brittle. It was hoped that a lower bainite structure of the Fe/4Cr/0.4C alloy would have greater toughness. Lower bainite was obtained by isothermally transforming austenite just above the M_S temperature. The cooling rate from the isothermal transformation temperature was varied to investigate this effect on the mechanical properties of the nearly 100% bainite structure. Tempering experiments, following bainitic treatments, were also performed in an attempt to improve the toughness of the material.

Strength and toughness properties were comparable with a 0.4C quenched and tempered plain steel. The properties of the nearly 100% bainite structure were unaffected by the cooling rate from the transformation temperature. The chromium appeared to have a strong stabilizing effect on the carbides as the bainitic alloy showed greater temper resistance. The small martensite regions present in the bainitic structure had a relatively higher carbon content than the bainite regions. The effect of the martensite regions on the mechanical properties was unclear.

I. INTRODUCTION

-1-

In recent years, the application of transmission electron microscopy to steel research has contributed significantly to our understanding of the factors controlling the mechanical properties in steels. The prime interest of microscopists has been to relate the details of substructure in iron alloys to the properties of the bulk specimen. Accurate understanding of the relation of the substructure to the properties of the bulk specimen requires long and tedious study of the variables that are responsible for the substructure in steels.

A. Morphology of Martensite

Of prime interest to researchers aiming for high strength and toughness in steels are the martensite and bainite structure. Both of these morphologies offer structures conducive to high strength and toughness, namely, subgrains of small transformation units with a uniform distribution of fine carbide precipitates within the matrix. Two major types of martensite form in iron-base alloys. In the martensite that forms in dilute alloys of iron, the basic transformation unit has a narrow lath shape and is commonly known as lath martensite.¹ Each lath is the result of a homogeneous shear and successive shears produce a "packet" of parallel laths containing a high density of tangled dislocations. The other type, plate martensite, differs in the shape of the transformation unit. Plate martensite is long and "needle like" as viewed with an optical microscope. This structure differs from that of lath martensite also in the fact that adjacent plates usually do not form parallel to one another. Twinned formation is

associated with this type leading many researchers to refer to plate martensite as twin martensite.

Investigations of a large number of binary ferrous systems have shown that alloy composition and the transformation temperature influence the transition from lath to plate martensite.² Thomas ³ has suggested that the strength of martensite and the austenite from which it forms are, probably the most important factor that determine whether martensite is dislocated or twinned or both. Since most alloying elements act as solid solution strengtheners in austenite, they increase the thermodynamic driving force necessary to initiate the martensite shear transformation and consequently lower the Ms temperature. The yield strength of the martensite at the temperature of transformation then becomes the controlling factor. Whether the austenite slips or twins depends on the critical resolved shear stress for slip vs twinning at the temperature of formation. Since martensite forms over a range of temperatures, this range may include the slip=twin transformation, and mixed substructures are expected.

Carbon is the most potent of the alloying elements in promoting twinning. Carbon is also the most potent strengthener of steel. Thus, the stronger the steel the more difficult it is for slip to occur and then twinning is preferred. At low carbon levels in plain carbon steels, <0.15 wt% C, the martensite consists of bundles of heavily dislocated laths. Adjacent laths are sometimes twin related or separated by small angle boundaries. At high carbon levels, >0.8%, the substructure is mainly plates containing transformation twins.

-2-

B. Morphology of Bainite

-3-

It has long been known that there are two major forms of bainite, upper and lower bainite. In many steels, there has been shown to be a distinct microstructural difference between these two forms. In upper bainite the carbides frequently form as elongated particles between the bainitic ferrite grains. In lower bainite, the carbides tend to precipitate at an angle to the major growth direction, or longitudinal axis, of the bainitic ferrite grain.⁴

There is also evidence of a difference in the reaction kinetics of the upper and lower bainite transformations, particularly in high carbon steels. At high temperatures in the upper bainite range, ferrite laths nucleate and grow, the reaction being a shear transformation. The bainitic ferrite contains a low carbon content, but carbon has a sufficiently rapid rate of diffusion in austenite to be able to diffuse away ahead of the moving ferrite-austenite interface. The strains accompanying the shear transformation appear to cause cooperative nucleation of many bainitic ferrite laths, side-by-side (Pickering⁵).

It is Pickering's contention that as the ferrite laths grow together, the carbon content in the austenite trapped between them is increased until it exceeds the solubility of carbon in the austenite. Pickering believes that when this occurs, cementite is precipitated directly from the austenite. The extent to which carbon enrichment occurs before cementite forms directly from the austenite will depend upon the transformation temperature and the carbon content of the steel.⁶ The lower the transformation temperature, or the higher the carbon content of the steel, the less will be the enrichment in carbon required for this to occur. Also, the higher the carbon content or the lower the transformation temperature, the larger will be the regions of austenite enriched in carbon.

With decreasing transformation temperature, the rate at which carbon can diffuse in front of the ferrite-austenite interface decreases. The sideways growth of ferrite laths is slowed down, resulting in narrower ferrite laths. Also the increased strain associated with the reaction probably restricts the edgewise growth of the ferrite plates in the initial part of the shear reaction. Both of these effects lead to a smaller bainitic ferrite grain size.

Eventually, Pickering speculates, a temperature is reached at which the carbon diffuses so slowly that it can no longer diffuse away adequately in front of the bainitic ferrite. When this occurs the carbon content of the ferrite is too high for the reaction to continue. At this point, upper bainite is replaced by lower bainite. In the growth of lower bainite, the initial bainitic ferrite forms by shear with the same orientation relationship with the austenite as in upper bainitic ferrite. However, the laths are very thin and precipitation of carbides is required before the ferrite can grow laterally. The orientation relationship between the ferrite and carbide is similar to that in tempered, twinned martensite. Lowering the carbon content of the ferrite by precipitation restores the driving force to a level which allows the bainitic ferrite to grow. The new growth again produces supersaturated ferrite, and again carbide precipitation is required within the ferrite for continued growth. This process of carbide precipitation and growth is continuous until the growing plate impinges on an adjacent plate.⁷

-4-

Depending on the position and extent of the bainitic transformation region on a non-equilibrium phase diagram (such as a TTT diagram), some steels may have bainitic regions that are dominated by one or the other type of bainitic structure. If the bainitic start temperature exists at relatively high transformation temperatures (550-600°C), upper bainite should be easy to obtain. However, if the transformation curve is surpressed downward by alloying elements, then the structure most likely to form is that resembling lower bainite.

Since 1963, systematic studies of the effect of principle alloying elements on the structure and properties of steels have been conducted in Professor Thomas' group in the Materials Science Department, U. C. Berkeley. These studies are part of a program designed to investigate the effects of carbon and substitutional elements on the microstructure and mechanical properties of martensitic and bainitic steels of relatively high purity. Such studies provide the basis for the design of simple steels where the known effects caused by each element and heat treatment can be incorporated into alloys of future investigations to give a predetermined set of properties.

The present study was one such investigation fathered by the work of researchers in this program. One such researcher, Das, studied a series of Fe-Ni-Co-C alloys with the carbon level ranging from 0.2 to 0.4%.⁸ He noted that while increasing carbon content increases the strength and hardeness of martensite, the toughness drops dramatically. The important result from this was that at the same strength level the twinned martensite has a lower fracture toughness than the dislocated martensite.

-5-

To prove that twinning and not carbon content was responsible for this phenomenon, recent work was done on Fe/Cr/C steels by McMahon.⁹ The result of this work clearly showed that in as-quenched steels, for the same carbon content, fracture toughness decreases with increasing twinning. In this study, McMahon found that the volume fraction of martensite containing transformation twins increased with chromium content.

Similarly, toughness was found to decrease with increasing mangenese content at the same carbon and strength level in 0.24% C steel. Huang and Thomas 10 in a study of Fe/Ni/Mn/C martensite showed that increasing manganese above 2% in 5 Ni/0.25 C or 3% in 0.25 C plain steel was deleterous to the toughness of these tempered martensite steels. Manganese additions lowered the Ms temperature and hence promoted twinning by lowering the martensite transformation temperature into the region where twinning has the lowest critical resolve shear stress.

It is largely from McMahon's work that the idea for this project took root. One of the Cr alloys he studied, 0.35 C/4 Cr steel, proved to have just the right quantity of alloy additions for optimum toughness and strength in the untempered condition. An increase in carbon over 0.35 wt% produced a martensite structure that was severly embrittled by twinning while a carbon level of 0.35% C did not cause serious reduction in the toughness. Four percent chromium and 0.35% C also seemed to be the optimum Cr level from the point of view of twin content and toughness since exceeding 4% caused poor toughness due to increased twin density. It was hoped that since the bainite transformation

-6-

should produce a twin free matrix of uniformly distributed carbide particles similar to that of the favorable dislocated tempered (or autotempered) martensite structure, a strong, tough alloy may be developed by obtaining a bainite morphology in a steel with the same level of chromium (4%) and with a slight increase in the carbon to 0.4 wt%.

An alloy with 0.4 C, it was hoped, would also be ideal for a study comparing the martensite and bainite structures for toughness at comparable strength levels. Studies comparing the toughness of bainite and martensite structures have found mixed results. In one such study, Das¹¹ investigated the structure and properties of tempered martensite and bainite in a series of steels with varying C, Ni and Co levels. At similar strength levels, the toughness of lower bainite was found to be superior to martensite in two of the alloys tested. The alloy content of these were Fe/8Ni/7Co/0.4C and Fe/8Ni/4Co/0.4C. The microstructural explanation for the tougher bainite structure was that the relatively high carbon content of 0.4% C promoted a high twin density in the martensite. Upon tempering, preferential carbide precipitation occurred on twin and grain boundaries. The lower bainite structure, on the other hand, consisted of ferrite laths or plates with carbides distributed uniformly within the matrix. This morphology proved more resistant to fracture.

In the lower carbon steels, 0.24% C, Das found that the density of twins was much reduced, as would be expected, and the martensite consisted mostly of dislocated laths. For these steels, the embrittlement associated with twins was avoided. Hence, the tempered martensite

-7-

structure, with the cementite in a predominantly Widmanstatten pattern, had superior toughness properties at the same strength level than bainite.

Huang,² in his study of Fe/Ni/Mn/C steels investigated the impact properties of the martensite and bainite structures. Since only a complete bainite structure was attained in the lower manganese steels, structural comparisons made only made for these alloys. He found that the tempered martensite of the lower manganese steels possessed better impact toughness than the bainite structure. As mentioned before, these lower manganese alloys had a relatively low density of martensite twins. Huang was thus able to show that alloys of dislocated lath martensites have superior toughness compared to a lower bainite structure.

It appears then that steels of high or medium carbon contents (above 0.4 wt% C) could have superior properties in the form of bainite due to a twinned, embrittled substructure if in the form of martensite while steels of lower carbon content with dislocated martensite substructures usually have superior properties compared to the bainite structure.

The present study hoped to show whether or not a strong tough bainite structure, isothermally produced, could be produced in a steel alloy with 0.4% C. If successful, this study could pave the way for a simple, one step, continuously cooled transformation that would yield a bainite microstructure with favorable mechanical properties. Also, it was hoped that the martensite properties of this alloy could be compared to the bainite properties, particularly the toughness, at similar strength levels.

-8-

II. EXPERIMENTAL PROCEDURE

The steel alloy investigated, with 0.4 C and 4 Cr, herein referred to as 0440 steel, was prepared by Republic Steel Corporation. It was received in the form of 1/8 in. rolled sheet and a 30 lb piece of 1 1/2 in. plate. The material was homogenized at 1200°C for 24 hr in a vacuum furnace. Tests were run after homogenization to ensure that the alloying elements were evenly distributed and of the right level of concentration. Microprobe analysis of the specimens was used to test the chromium level and distribution. Samples were taken from the top, bottom and middle of the plate, perpendicular and parallel to the rolling direction to be tested for the correct chromium composition. Optical specimens were examined to ensure that no alloy segregation was present.

In order to determine the heat treatment necessary for obtaining the desired lower bainite structure, a time-temperature-transformation (T-T-T) diagram was mapped out for the 0440 alloy. This was done by optical and electron metallographic studies. Specimens for this study were prepared by cutting the steels into pieces 1 in. square and 20-30 mils thick to insure uniform and rapid cooling. The thin specimens were austenitized for 8 min at 1200°C in a vertical tube furnace. Argon gas was constantly flowing through the furnace to displace the air and prevent decarburization. At the end of the austenitizing period, the specimens were quenched directly into the bainite region temperature and held. After transforming isothermally for the designated length of time, the specimens were quenched to room temperature. The extent of the transformation was then found by polishing and etching the specimen in a 5% nital solution and examining the microstructure in an optical microscope. For specimens where the limits of the optical microscope made characterization of the microstructure difficult, electron microscopy was utilized. Thin foils for electron work were prepared by the following steps:

- Wet cut 20 mil slices from the bulk specimens using a 1/32 in. cut off wheel.
- 2. Chemically thin the slices to 5-7 mills in a solution of 100 ml 30% H_2O_2 and 5 ml of HF.
- 3. Spark erosion cut 2.3 mm diameter discs out of the foils.
- 4. Mechanically sand discs to 1-2 mills.
- 5. Final thinning by jet polishing in a solution of 400 ml

 CH_2OOH , 75 gr CrO_3 , 21 ml H_2O with a voltage of 20-25 millivolts. Foils were examined in a Siemens Elmiskop 1A microscope operated at 100 kV.

The martensite start temperature was measured by recording the change in the magnetic permeability during rapid quench from an austenitizing temperature. This was performed by preparing 30 mil wire of the 0440 alloy. The wire was heated to the austenite region by resistance heating inside a closed quartz cylinder with a high vacuum. After austenitizing for 10 min, the wire specimen was gas quenched to room temperature. As the austenite transformed to martensite there was an accompanying increase in permeability. An induction coil which was located coaxially outside of the quartz cylinder measured the change in the permeability as the specimen decomposed from austenite. The permeability change was recorded on paper tape as a function of time. Chromel-alumel thermocouples were used to also record the temperature of the wire specimen from a junction spot welded on the wire. By correlating the paper tape output with the temperature chart, the Ms temperature could be estimated.

From the study of the T-T-T curve for this alloy, bainite transformation temperatures were chosen on the basis of obtaining complete transformation at as low a temperature as possible. This was to ensure that the desirable lower bainite structure was obtained.

All the specimens for mechanical property studies were austenitized for 1 hr at 1200°C in an argon atmosphere. The specimens were then quenched into a molten salt bath at the desired bainite transformation temperature. After holding isothermally for the designated length of time, the specimens were water quenched or air cooled to room temperature. Tempering experiments were performed on some of the specimens after the room temperature quench. Tempering was done in salt pots for 1 hr at temperatures of 500°C, 400°C, 350°C and 300°C. Before heat treating the specimens were machined to within 10 mil of the final dimensions. After heat treating, this extra layer was ground off to remove any decarburized material.

Round, 1/4 in. tensile specimens were tested on a 300 kip capacity M.T.S. machine, Fig. 1(B). Standard size Charpy V-notch specimens were used for measuring the impact toughness, Fig. 1(A). Compact, fracture toughness specimens were also used in order to determine plane strain fracture toughness, Fig. 1(C). All the tensile, impact

-11-

and fracture toughness specimens were tested at room temperature. Several impact specimens were also tested above room temperature in the case of specimen 1-B, in order to determine the Charpy V-notch (CVN) transition temperature.

III. RESULTS

A. Time-Temperature-Transformation Diagram

From the examination of more than 30 specimens, transformed for varying lengths of time at different temperatures in the bainite regions, part of the TTT curve for the 0440 steel was plotted, Fig. 2.

The martensite start temperature experiment estimated the Ms temperature from specimens austenitized at 1000°C, 920°C and 870°C. Unfortunately, due to the operating temperature limit of the quartz tube used in the experiment, higher austenitizing temperatures could not be used. Thus, it is doubtful that all the chromium carbides were in solution before the gas quench. In all three specimens, the Ms temperature was estimated at 308°C. This agreed closely with the calculated Ms of 322°C using Bain and Paxton's equation. By isothermally transforming 30 ml specimens at 5°C intervals and then examining the structures in an electron microscope, the start of the martensite transformation was also detected. Electron microscopic examination of specimens transformed in this way supported the belief that the Ms was approximately 320°C.

B. Mechanical Properties

Table I shows the results of the study of the room temperature mechanical properties of the 0440 steel alloy with a bainitic structure. Two bainite transformation temperatures were tested, 360° C and 330° C. These temperatures were chosen near the Ms because it is well documented that the lower the bainitic transformation temperature, the more favorable are the properties. ^{13,14} This fact was confirmed in the present investigation. At 330°C the yield and ultimate strengths increased as much as 10% over the specimens transformed for the same length of time

-13-

at 360°C. Toughness and elongation results were generally low with no significant difference between the two transformation temperatures.

Modifications in the basic isothermal heat treatment were studied to investigate possible means of improving the properties. These modifications were length of the isothermal holding time, transformation temperature, rate of cooling after isothermal transformation, grain size refinement and tempering of the final bainitic structures.

These modifications were tried for several reasons with the prime objective being to improve toughness. After 15 min of isothermal transformation the period of rapid bainitic growth was over before the structure was completely transformed. Longer times were studied to see if the remaining austenite would transform in a reasonable length of time. Two different transformation temperatures were investigated in the lower bainite range of the T-T-T curve. The first transformation temperature studied was 360°C. However, the appearance of interlath carbides in specimens transformed at this temperature prompted the use of a lower transformation temperature to improve the carbide morphology.

Since the complete bainite transformation could not be attained, it was hoped that the remaining structure, which was martensitic at room temperature, could be autotempered. This was tried by air cooling the specimen from the isothermal transformation temperature. It was speculated that if the martensite in the structure was partially responsible for the low toughness of the 0440 steel, then this might be a means of "softening" the martensite without requiring a post heat treatment temper.

-14-

Since both the toughness and strength of a steel can improve as the grain size decreases, the modification of grain size refinement was performed to reduce the relatively large grain size caused by the high austenitizing temperature of 1200°C.

Tempering the steel after the bainitic transformation was tried as an attempt to improve the toughness without seriously lowering the strength of the alloy.

The results of varying the transformation time have already been mentioned. After transforming for 15 min (specimen 1A and 1-B) the period of rapid transformation had ended and there was no detectable increase in the amount of bainite in specimens held for longer times. The bainite transformation was nearly complete after 15 min with the balance (3-4%) transformed to martensite. Therefore, it was not surprising that transforming for longer times of up to 60 min resulted in no significant change in the strength. To see the effect of a very long holding time, specimen 8-B was transformed for 5 days at 330°C, [Fig. 21]. This resulted in a significant decrease in the strength and an increase in the elongation. It is suspected that the long transformation time allowed some homogenizing of the carbon. This could have resulted in a more complete bainite transformation and a reduction of the localized regions of high carbon concentration formed during the initial 15 min of the transformation.

The percent of uniform elongation was measured before necking occurred in most of the specimens due to early fracture. Therefore, valid comparisons of different tests involving those early failures was not possible. In the specimens that did not fail until after

-15-

necking had started, the elongation results were still low indicating a lack of ductility in this structure.

Slower cooling rates from the isothermal temperature were studied by air cooling specimens from the salt pot. It was hoped that this would have some effect on the stabilized austenite regions which were not transformed till after cooling below the Ms temperature. These regions are known to be high in carbon. Air cooling after the isothermal treatment resulted in no change in the strength or impact toughness of the material (4-A, 3-B).

Tempering experiments were also tried to improve the toughness of the bainitic structure. Tempering temperatures of 300° C, 350° C and 400° C seemed to have no effect on the strength while toughness seemed to decrease, dropping from a K_{IC} value of 37 to 28. At 500° C, tempering a substantial decrease in tensile strength for both transformation temperatures. The Charpy impact toughness was unchanged by the 500° C tempering temperature.

The attempt to improve the toughness by grain size refinement was inconclusive. In the one experiment performed, a double austenitizing treatment was used (specimen 1-C). After austenitizing at 1200°C for 1 hr the specimen was transformed in the bainite region to get a ferrite structure with a fine uniform dispersion of cementite. Heating again to a low austenitizing temperature for a short period of time was known to produce a fine grained austenite due to the nucleation process from the bainite structure.¹⁵ However, holding for 10 min at the second austenitizing temperature failed to redissolve the carbides

-16-

that had precipitated out of solution during the bainite reaction. Instead of dissolving, the carbides spheriodized, Fig. 20. In this condition the carbides were rendered useless as a strengthening mechanism. Although CVN toughness increased dramatically to 55 ft-lb the strength was cut by half. Later studies of this treatment showed that due to the stability of chromium carbides at high temperatures, it is difficult getting all the carbides back in solution within a resonable time below an austenitizing temperature of 1100°C. The long times and high temperatures required to redisolve the carbides resulted in almost complete grain growth to original grain size.

Figure 3 shows the Charpy impact transition curve for specimen 1-B. Impact toughness specimens were tested above room temperature by heating to the designated temperature in water and then transferring them from the hot bath to the impact testing machine within 5 sec. It appears that at 100°C the upper shelf of the transition curve had been reached. Even at this temperature, the structure showed very poor impact toughness.

C. Optical Microstructure

The T-T-T diagram was defined by optical and electron observation of specimens transformed for various lengths of time through a wide range of temperatures that encompassed the complete bainite region. One such series of specimens, Fig. 4, shows the variation of the structure of the bainite plates or needles from the low to high temperature bainite. At a transformation temperature of 325°C the bainite structure is the classic lower bainite with very fine, parallel needles,

-17-

Figure 4(A). Increasing the temperature to 350° C, Fig. 4(B), seems to give a similar microstructure except for a noticeable broadening of the needles. Starting at 380° C, Fig. 4(C), the needles become broader and longer since there is less chance of a growing needle being intercepted with fewer needles being nucleated. Also, with increasing transformation temperature there is an increase in the size of the bainitic "packets" or groups of parallel ferritic plates. Figures 4(C) and 4(D) show mixed structures of fine and broad needles. In the high temperature transformations of Figs. 4(E) and 4(F), the bainite reaction had barely begun. The driving force for the transformation provided by the supercooling of the alloy was sufficient only for heterogeneous nucleation of bainite plates at such nucleation sites as the grain boundaries in 24 hr (Fig. 4(f)).

In order to further characterize the region of the most rapid bainite transformation, specimens were transformed at 350°C for times of from 1 min to 1 hr and then quenched. The progress of the transformation at this temperature can be observed in the series of micrographs in Fig. 5. Figures 5(A) and 5(B) again illustrate that the bainite needles are nucleated first at the grain boundaries. Growth seems to proceed to the interior of the grain with needles branching off of parent needles not unlike primary and secondary dendrite formations. After 60 min the reaction appears to be nearly complete, Fig. 5(E). In Fig. 5(E) only the white irregularly shaped "holes" between some of the bainite plates indicate that the transformation may not have been complete. Since the orientation of the plates to the polished surface affects how the plates are attacked by the etchant, some

-18-

light gray or white regions, which are actually bainite plates not oriented favorably, may be mistaken for regions that were untransformed during the isothermal treatment. Closer study of the structure using electron microscopy revealed that the long narrow light colored regions on optical micrographs were indeed bainite needles unetched. The small, irregular shaped white regions of Fig. 5(E) are areas of untempered martensite.

Another means of distinguishing the martensite regions from the unetched bainite needles was discovered in the specimens tempered at 500°C (5-A, 4-B). During the austenite to ferrite plus cementite transformation, carbon was able to diffuse away from the growing bainite needles and collect in the untransformed austenite regions. Upon quenching, a relatively high carbon concentration was trapped in these localized regions as the austenite transformed to martensite with a supersaturated solution of carbon. After tempering at 500°C the cementite in these regions was able to precipitate out of solution. Upon polishing and etching specimens 5-A and 4-B revealed dark irregular shaped regions, high in carbon and believed to be tempered martensite (Fig. 6) wedged between plates of bainite.

D. Electron Microscopy

1. Bainite 330°C

Electron microscopy of the specimens transformed at 330° C revealed a lower bainite structure with no evidence of interlath carbides or retained austenite, Figs. 7(A), 7(B) and 7(C). The cementite particles are evenly distributed throughout the matrix and are aligned undirectionally at 50°-60° to the main axis of the ferrite plate. After 15 min of isothermal holding at this temperature and then quenching, the bainite transformation was nearly complete with better than 95% of the structure bainitic and the remainder martensite. It was difficult obtaining clear images of the martensite regions due to preferential polishing of the bainite. Also, the high carbon content and the great amount of transformation deformation in the martensite did not help the resolution problem. Figures 8(A), 8(B) and 8(C), shows the martensite regions trapped between the bainite plates. The structure of these regions can be seen to be highly strained due to the transformation of austenite to martensite. This was indicated by the appearance of microtwins as in Fig. 8(C), and a high dislocation density in the martensite.

Figure ⁸(B) shows a wavy carbide structure branching out from the martensite leading to the speculation that these carbides may have precipitated directly from the prior austenite as in upper bainite even at this low temperature. Carbide formations such as in this figure were often observed around martensite areas. An explanation for this is given later.

2. Bainite 360°C

At 360°C the bainitic structure was still largely lower bainite with small carbides aligned across the ferrite plates, Figs. 9(A), 9(B) and 9(C). Figure 10 shows more of the lower bainite structure at this temperature with a dark field of the Fe_3C carbides. At this temperature, some of the bainite formed with long, irregular carbides following the growth direction of the plate (Figs. 11(A) and 11(B)). Evidence of interlath carbides were also detected by dark fielding carbide diffraction spots, Fig. 12.

3. Bainite 400°C

In an electron microscopy specimen transformed isothermally at 400°C for 5 min the upper bainite mode of transformation became more apparent. Figure 13 shows the carbides still aligned at an angle to the main axis of the plate. However, there seems to be a greater tendency for the carbides of some plates to form as interlath carbides or within the ferrite aligned along the growth direction, Fig. 14. Figure 13 also shows an adjacent martensite plate from the prior untransformed austenite. A "vein" of martensite also appears in the center of the ferrite grain marked with an "M". The dark fields of Fig. 13 and Fig. 14 help to highlight the precipitates.

A tip of a growing bainite plate shows why these appear as long, narrow needles at the magnification of an optical microscope, Fig. 16. At the front of the tip is a layer of carbide which was concentrated there as carbon diffused away from the growing ferrite. A diagram of the carbon concentration gradients at the ferrite-austenite interface proposed by Pickering is shown in Fig. 16. The carbon concentration is greatest at the interface in the austenite phase. With increasing carbon content in the austenite, the diffusion gradient will be more shallow. This means that the rate of diffusion of carbon away from the ferriteaustenite interface will be less rapid. Therefore, precipitation of carbide within the supersaturated bainitic ferrite (i.e., the lower bainite morphology) is necessary at higher temperatures than in a lower carbon steel, in order for the reaction to proceed. In a study of the effect of carbon on the temperature change from upper to lower

-21-

bainite, Pickering found that with increasing carbon content the temperature at which lower bainite forms instead of upper bainite is increased, Fig. 17. At a carbon content of about 0.5% he proposed seen that the extrapolated $A_{\rm cm}$ line intersects the curve denoting the upper temperature limit of lower bainite formation. When this occurs, primary cementite becomes capable of nucleating and precipitating directly from the austenite.¹⁶

The long, wavy carbide aggregates often seen around regions of martensite such as appears in Fig. 8(B), may be explained on the basis of carbon enrichment in these localized regions. The level of carbon concentration in these areas may have been greater than 0.5% C due to the diffusion of carbon to austenite during the bainite transformation. At this high carbon level, the cementite was able to precipitate directly from the austenite.

The electron micrographs of tempered specimens 4B and 6B are presented in Fig. 18. There was no detectable morphological difference between the tempered specimens and the untempered specimens from the same transformation temperature.

E. X-Ray Analysis

An investigation was made to determine whether or not retained austenite was present in any of the specimens of Table I. A Picker X-ray diffractometer was used for this purpose. The mounted specimens were scanned for austenite through a large angle so that several possible austenite orientations were included. Down to the limiting amount of austenite that can be detected by the diffractometer, which is 1-2% austenite, the results were negative in all of the specimens. Electron microscopic analysis, which is capable of detecting even lower amounts of austenite, substantiated this result.

F. Electron Fractography

Fractographs from specimens 1-A, 1-B and 5-A, presented in Fig. 19, were taken from Charpy impact surfaces. Parallel ridges on the fracture surfaces are believed to be due to the crack paths following the bainite plates. Flat, cleavage fracture surfaces can be seen where the ferrite plates were oriented at an angle out of the plane of the crack. Straight, flat fracture surfaces were observed in all the bainite specimens indicating very brittle failures.

IV. DISCUSSIONA. Mechanical Properties

-24-

The bainitic properties of this 0440 steel had, at best, yield strengths of 190 ksi and 250 ksi U.T.S. This strength level conforms well with the findings of other researchers for a fully transformed bainitic steel with 0.4% C.¹⁷ The lower transformation temperature of 330°C produced a finer needled structure than the 360°C structure. A smaller packet size in the lower temperature specimen may also have accounted for the increased strength here as suggested by Naylor and Krahe.²⁰ The absence of interlath carbides in the structure transformed at 330°C also could have contributed to the superior properties.

The toughness of the 0440 steel was about the same for the two transformation temperatures tested. Impact toughness proved to be quite low. A study of the fracture transition curve for this alloy was conducted from specimens transformed as in 1-B. The upper shelf of the curve did not begin until a test temperature of 100°C was used. Impact toughness only increased from about 6 ft-1bs at room temperature to 12 at 100°C. This proved that the upper shelf of the supposedly ductile fracture region is low and the transition temperature is well above room temperature.

Tempering the specimens failed to improve the toughness of the bainite structure. The alloy showed greater temper resistance than a plain carbon steel. The strength of tempered 0440 did not decrease significantly from the untempered specimen until a tempering temperature of 500° C was reached. Rather than improving the toughness, the tempering treatment seemed to have an embrittling effect on the properties. Fracture toughness decreased by 7-8 ksi-in.^{1/2} over the untempered

specimens while the impact toughness remained about the same. The reason for this embrittlement is not known but the temperature range over which it occurred is similar to the occurrence of temper embrittlement in martensite and so it could be related to the tempering of the martensite in the structure. Also, diffusion of carbon from the supersaturated ferrite to grain and plate boundaries may be one cause of this lower toughness although no evidence of this has been detected using either optical or electron microscopy.

The appearance of parallel ridges on some areas of the fractured surfaces led to the speculation that these corresponded to the bainite plates. Measurement of the spacing and length of these ridges showed that their size agreed quite well with the size and spacing of the bainite plates in optical micrographs. If the crack path follows the plate boundary, as this infers, then the size and distribution of these bainitic transformation units would have a great influence on the strength and toughness of the bulk specimen. Grain size (which in turn affects packet size) is already known to greatly influence the properties in a steel. It is believed in this present study that the large grain size caused by the high austenitizing temperature was detrimental to the toughness of this alloy. A smaller grain size could have reduced the ease of crack propagation by forming more grain boundaries to block the path of a crack. In a similar fashion smaller packet size could have provided an additional obstruction to crack propagation.

Comparison of the toughness of the bainite structure with the martensite in 0440 was impossible owing to the appearance of quench cracking in the specimens quenched to room temperature. The combination

-25-

of the 0.4% C and the 4% Cr seems to have a severe embrittling effect on the martensite structure. Surface cracking has also been observed in all mixed martensite and bainite structures that are less than 50% bainite. On a relative toughness scale, the bainitic structure is superior to the martensite structure in this 0440 alloy. Poor as the toughness properties are in the lower bainite condition, they are still an improvement over the toughness in the crack riddled martensite structure.

Raghavan,¹⁹ in a study of a 4 Cr steel with 0.34% C attempted to compare the mechanical properties of the martensite and bainite structures. However, the Ms temperature of this steel is relatively high (350°C) and his isothermal transformation experiments yielded mixtures of bainites and tempered martensite depending on the temperature of transformation. The mechanical properties of the isothermally transformed steels were inferior to those of the tempered martensite steels due to the influence of upper bainite or martensite during the isothermal transformation. In the 0440 steel the Ms temperature of 320°C was sufficiently low to attain a lower bainite structure with only a small amount of martensite. Charpy impact valves of 6-7 ft-lbs were attained with this alloy compared with <2 ft-lbs for Raghavan's substandard Charpy specimen of the 0.34C-4Cr steel isothermally transformed.

Since a 100% lower bainite structure could not be attained in the present investigation, it is not clear what effect the untransformed regions had on the mechanical properties of the 0440 steel. In the regions that were untransformed during the isothermal treatment there was evidence of a high carbon concentration due to diffusion of carbon ahead of the

-26-

growing ferrite-austenite interface. As the transformation temperature was raised, the migration of carbon during the transformation became more obvious. It is believed that in an alloy of similar carbon level but witha lower M_s temperature, the diffusion of carbon during the bainite transformation could be lowered since lower transformation temperatures would then be allowed. These lower transformation temperatures may then result in a more complete bainite transformation. With a decrease in the localized regions of high carbon concentration there would be a corresponding decrease in the amount of martensite in the structure. It is believed that eliminating these brittle martensite-carbon regions could improve the toughness of the lower bainite structure.

-27-

. CONCLUSIONS

1. The strength level of Fe/4Cr/0.4C (0440) steel was generally high and comparible to a high-strength 0.4C plain steel with a lower bainite structure.

2. The toughness of the 0440 was poor but better than that in Fe/4Cr/0.34C with a bainite structure. The 0440 with its lower Ms temperature had a nearly complete lower bainite morphology, while the Fe/4Cr/0.34C alloy had a mixed morphology of upper and lower bainite.

3. The isothermally transformed lower bainite structure avoided the quench cracking present when this alloy is quenched from the austenitized state to a temperature below the Ms. Therefore, the bainite structure exhibited superior toughness over the austenite structure in the 0440 alloy.

4. A 100% lower bainitic structure could not be attained. Besides bainite the structure also contained a few volume percent of small irregularly shaped regions of martensite with relatively high carbon concentration. The effect of these nonbainitic regions on the mechanical properties was unclear.

5. The bainitic structure of this alloy showed high resistance to tempering. Tempering at temperatures up to 500°C for 1 hr caused only slight reductions in the strength. Instead of increasing the toughness, the tempering had an embrittling effect on the alloy similar to the temper embrittlement phenomenon in martensite.

-28-

REFERENCES

-29-

- 1. G. Drauss and Marder, Met. Trans. 2, 2343 (1971).
- 2. P. M. Kelley and J. Nutting, J.I.S.I. <u>197</u>, 199 (1961).
- 3. G. Thomas, Iron and Steel International 46, 451 (1973).
- 4. S. J. Matas, R. F. Heheman, Trans. AIME 221, 179 (1961).
- 5. F. B. Pickering, Symposium: <u>Transformation and Hardenability in</u> Steels (Climax Moly, Ann Arbor, 1967), p. 109.
- 6. F. B. Pickering, ibid, p. 119.
- 7. F. B. Pickering, ibid, p. 119.
- 8. S. K. Das and G. Thomas, Trans. ASM 62, 659 (1969).
- J. McMahon and G. Thomas, <u>The Microstructure and Design of Alloys</u>, Proc. of the Intern. Conf. on the Strength of Metals and Alloys, Cambridge, England, August 20-25, 1973, Vol. 1, pp. 180-183.
- 10. Der-Hung Huang and G. Thomas, Met. Trans. 2, 1587 (1971).
- 11. S. K. Das and G. Thomas, Trans. ASM 62, 661 (1969).
- 12. Der-Hung Huang and G. Thomas, Met. Trans. 2, 1587 (1971).
- 13. K. J. Irvine, F. B. Pickering, J.I.S.I. 34, 518 (1963).
- 14. R. F. Heheman, V. J. Luhan and A. R. Troiano, Trans. ASM 49, 409 (1957).
- 15. B. V. N. Rao (M. S. Thesis), in progress.
- F. B. Pickering, Symposium: <u>Transformation and Hardenability in</u> Steels (Climax Moly, Ann Arbon, 1967), p. 115.
- 17. L. J. Klingler, W. J. Barnett, R. P. Forlumbert and A. R. Troiano, Trans. ASM <u>44</u>, 1557 (1954).
- 18. R. F. Heheman, V. J. Luhan, A. R. Troiano, Trans. ASM <u>49</u>, 419 (1957).
- 19. M. Raghavan and G. Thomas, Met. Trans. 2, 3433 (1971).
- 20. J. P. Naylor and P. R. Krahe, Met. Trans. 5, 1699 (1974).

ACKNOWLEDGEMENTS

-30-

I wish to thank Professor Gareth Thomas for his continued encouragement and support during the course of this work.

This work was done under the auspices of the U. S. Atomic Energy Commission through the Inorganic Materials Research Division of the Lawrence Berkeley Laboratory.

Specimen	Isot	h ermal Heat Time	Treatment Cooling Rate	Tempering Temp °C	YS ×IO ³ psi	UTS ×IO ³ psi	Uniform Elongation %	K _{IC} ksi-in/2	Charpy V-notch ft-lbs
I-A		[15 min.	H ₂ O Quench	_	165	230	5 *	35	6.6
2-A		30 min.	H ₂ O Quench		162	220	4.7 *		6.6
3-A	360 [°] C	60 min,	H ₂ O Quench	·	168	220	2.5 *		7.5
4-A		I 5 min.	Air Cool		178	230	5 *	<u> </u>	6.5
5-A		[15 min.	H ₂ O Quench	500	172	201	3.5 *	. /	7
I - B		(15 min.)	H ₂ O Quench		192	250	3.5 *	37	7
2-B		60 min.	H ₂ O Quench		193	234	4.7	35	5.5
3-B		15 min.	Air Cool	_	190	250	4.3		6
4-B	-	15 min.	H ₂ O Quench	300	186	245	3.0	28	_
5-B	330°C	{ 15 min.	H ₂ O Quench	350	187	240	2.5*	28	
6-B		15 min.	H ₂ O Quench	400	187	235	3.5*	29	
7-8		15 min.	H ₂ O Quench	500	180	210	2.5*		6.5
8-B		120 hrs.	H ₂ OQuench		170	225	7	29	

Table I.

* Failed before onset of necking

XBL747-6686

-31-

FIGURE CAPTIONS

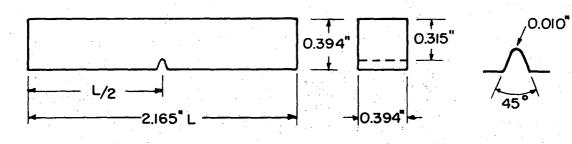
Fig.	1.	(A) and	(B) a	s it	is	written.	(C)	Compact	fracture	toughness
•		specimen	•	4						

- Fig. 2. Isothermal transformation diagram for 0440 steel.
- Fig. 3. Charpy impact transition curve for Specimen 1-B.
- Fig. 4. Variation of the bainite structure with transformation temperature, transformation time 24 hrs, 250X. A) 325°C, B) 350°C, C) 380°C,
 D) 410°C, E) 440°C and F) 470°C.
- Fig. 5. Bainite transformed at 350°C, 100X. A) 1 min, B) 2 min, C) 4 min, D) 8 min and E) 60 min.
- Fig. 6. Tempered martensite wedged between plates of bainite, 350X.
- Fig. 7. Bainite 330°C, transformation time 15 min, 30,000X.
- Fig. 8. Martensite regions traped between bainite plates, 30,000X.
- Fig. 9. Bainite 360°C, transformation time 15 min, 30,000X.
- Fig. 10. Bainite 360°C, 30,000X. A) Bright field, B) dark field of carbide diffraction spot.
- Fig. 11. Bainite 360°C, 30,000X
- Fig. 12. Interlath carbides in bainite transformed at 360°C, 30,000x.

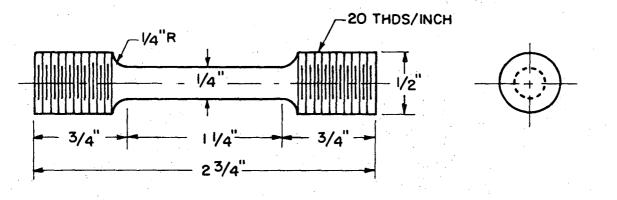
A) Bright field, B) dark field of carbide diffraction spot.

- Fig. 13. Bainite 400°C, transformation time 5 min, 30,000X. A) Dark field of carbide diffraction spot, B) bright field.
- Fig. 14. Bainite 400°C, transformation time 5 min, 30,000X. A) Bright field, B) dark field.
- Fig. 15. Carbon concentration gradient at the ferrite-austenite interface during the bainite transformation.

- Fig. 16. Bainite plate, transformed at 400°C for 5 min, 30,000X.
- Fig. 17. Temperature limites of the formation of upper and lower bainite as a function of carbon content.
- Fig. 18. Tempered bainite structure, 30,000X. A) Specimen 4B--tempered 300°C, 1 hr. B) Specimen 6B--tempered 400°C, 1 hr.
- Fig. 19. Electron fractographs of Charpy impact surfaces. A) Specimen 1-A, 300X, B) Specimen 1-B, 300X and C) Specimen 5-A, 1000X.
- Fig. 20. Spheriodized carbides from grain size refining 30,000X.
- Fig. 21. Specimen 8-B, transformed 5 days at 330°C, 30,000X.



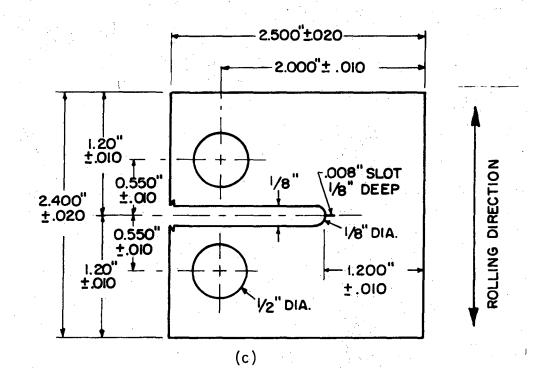




(b) ROUND TENSILE SPECIMEN

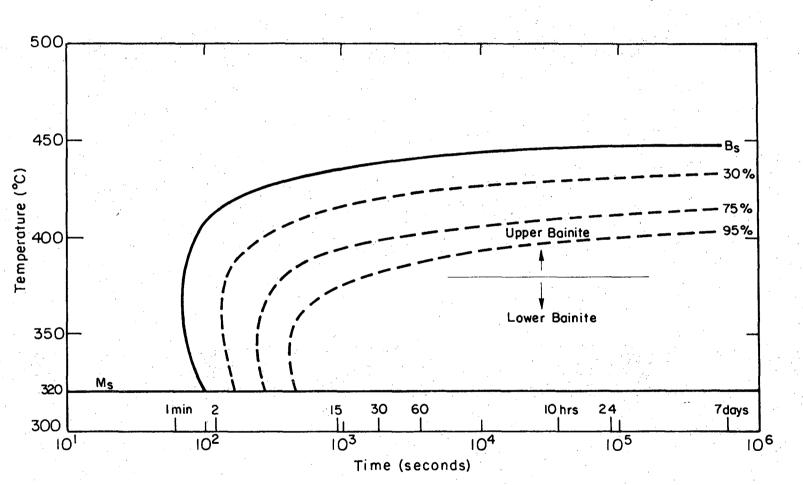
XBL735-6189

Fig. 1



XBL 735-6190

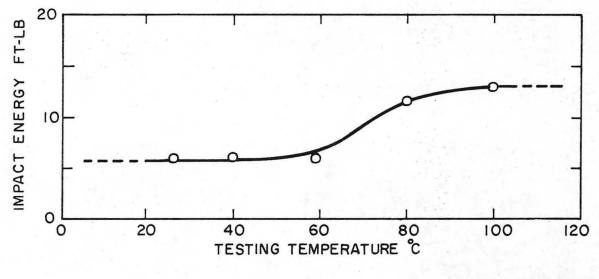
Fig. 1 cont.





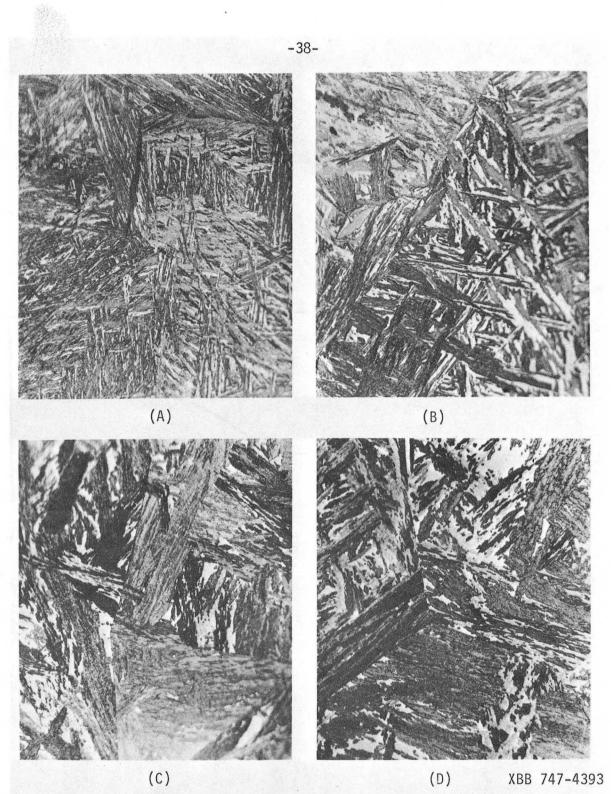
XBL747-6685

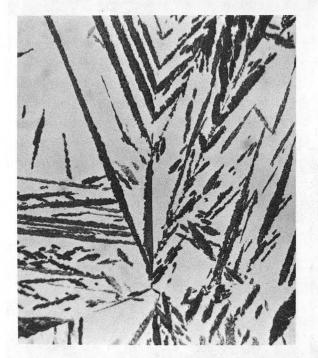
မ္ပ်



XBL 747-6798

Fig. 3





(F)

(E)

XBB 747-4399

Fig. 4 cont.

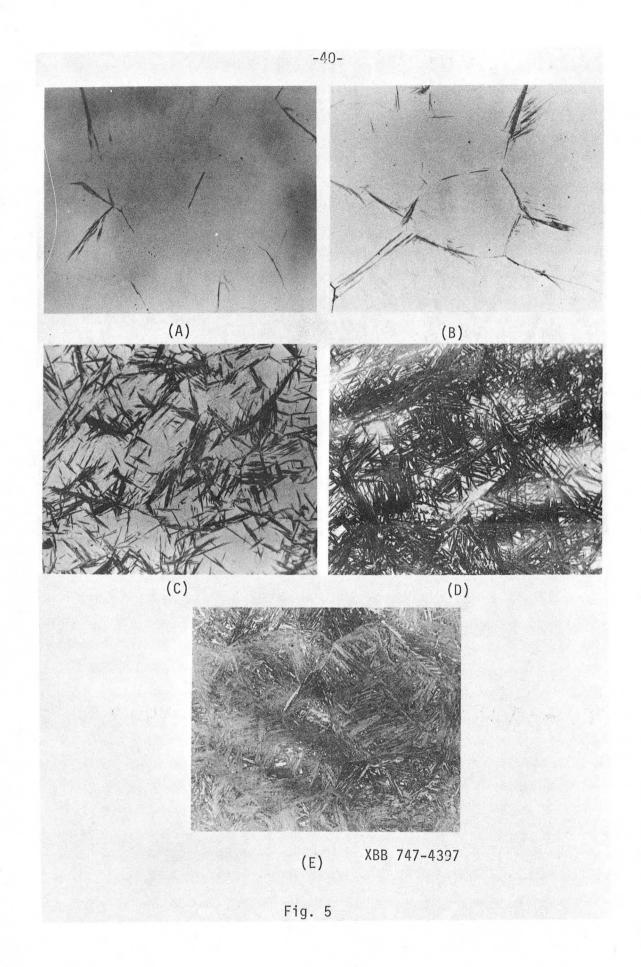
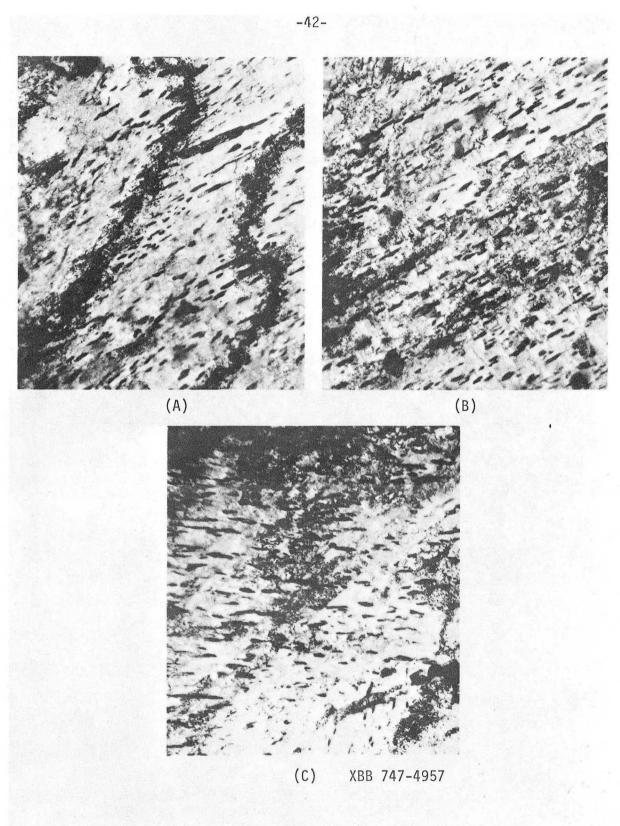
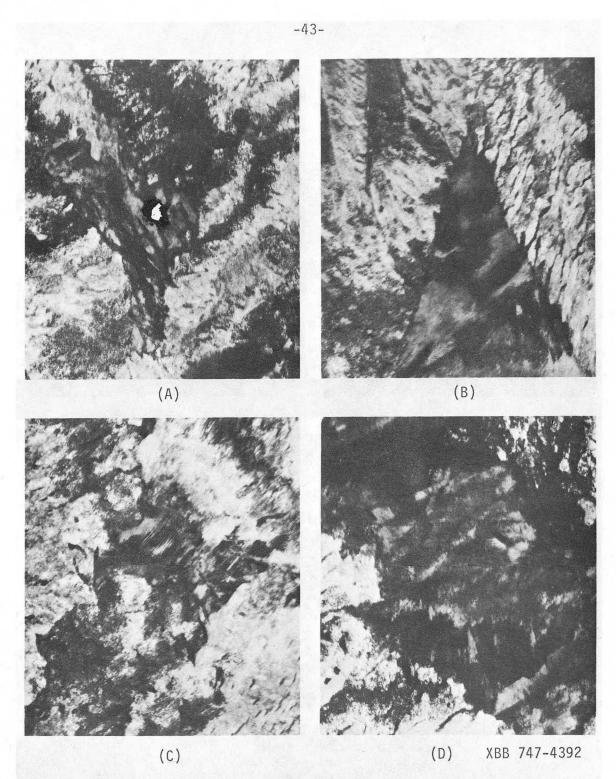




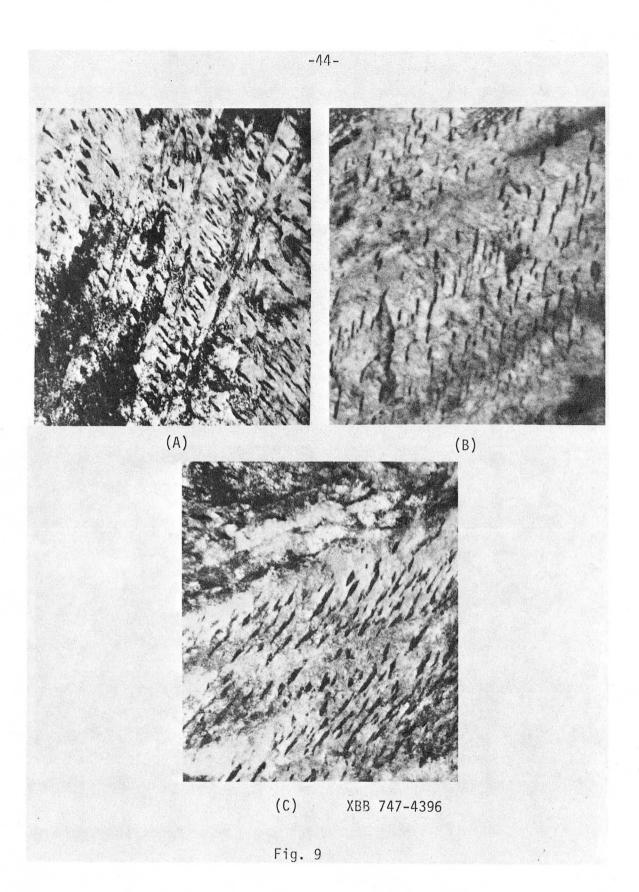
Fig. 6

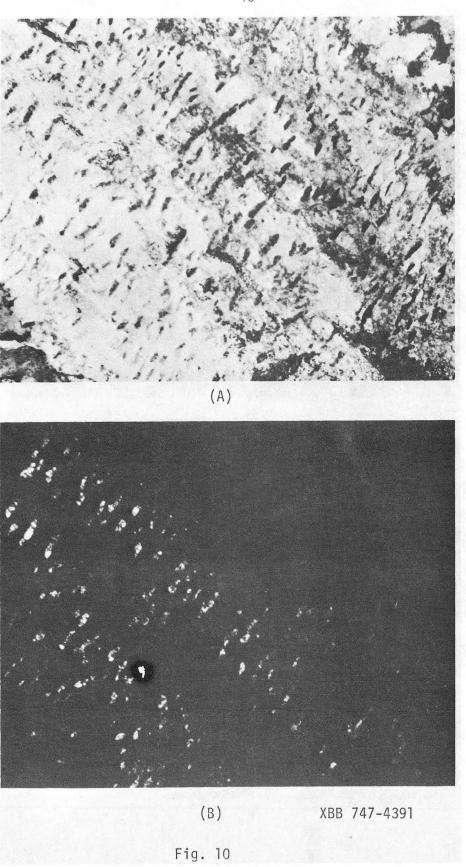


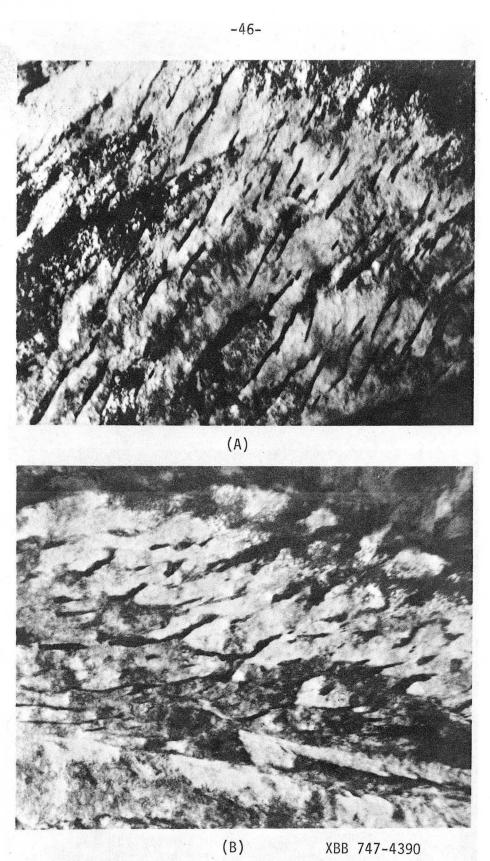


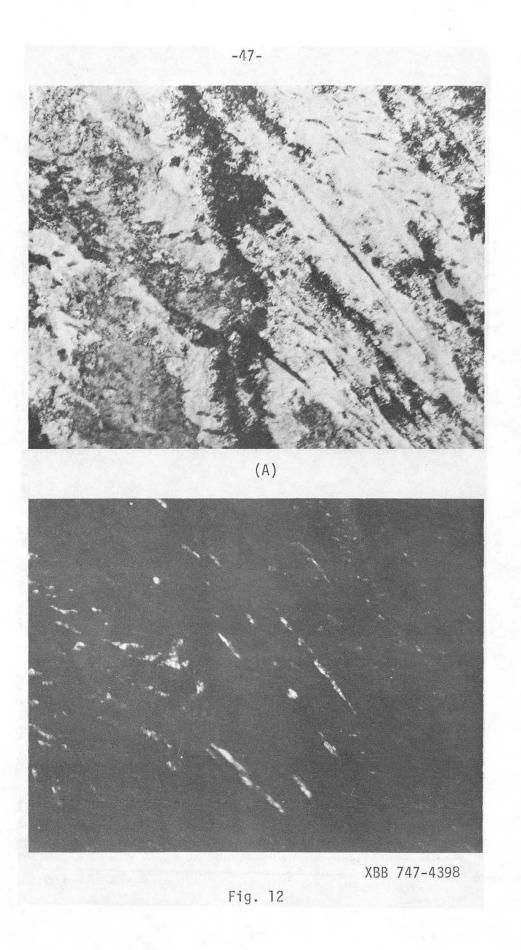


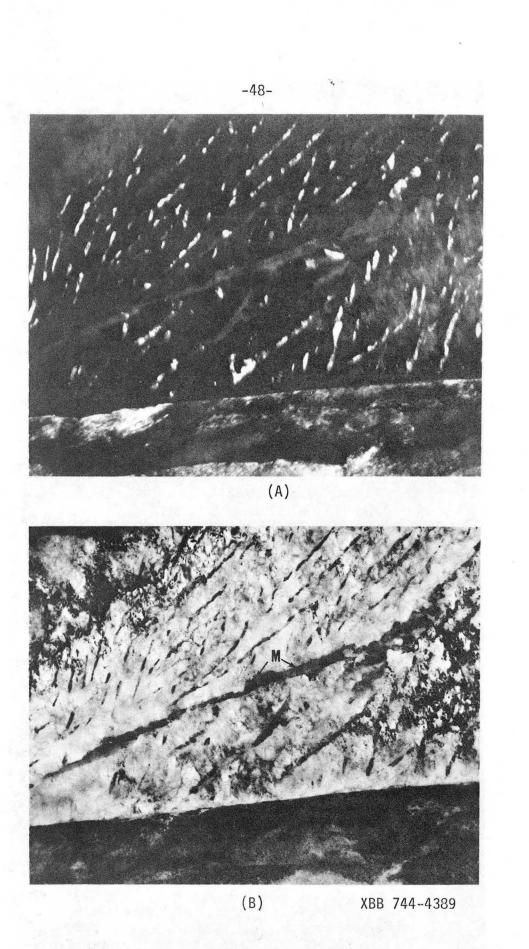


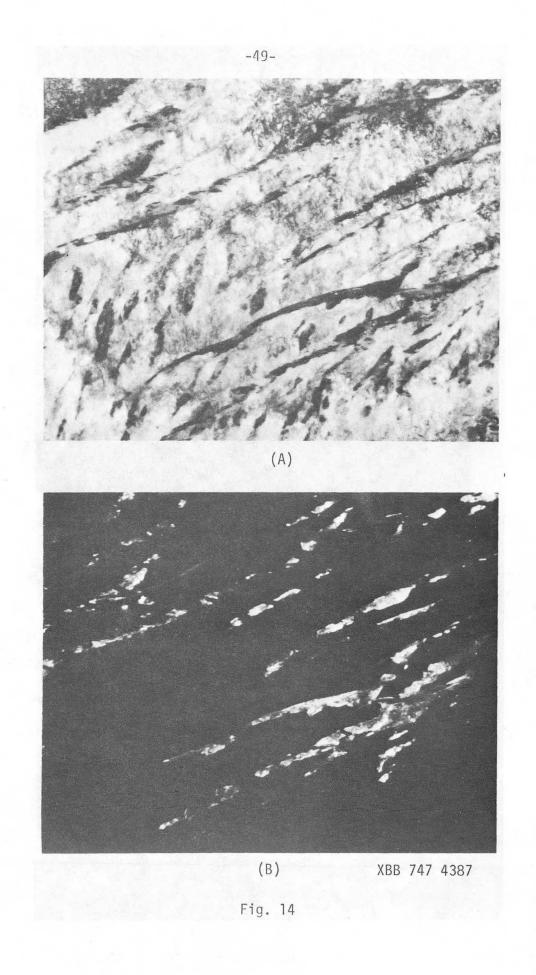


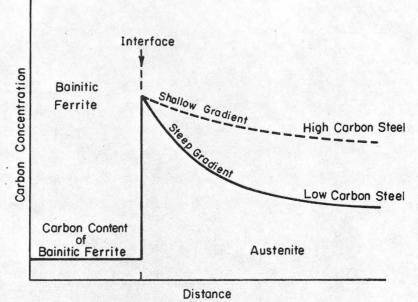












Distance



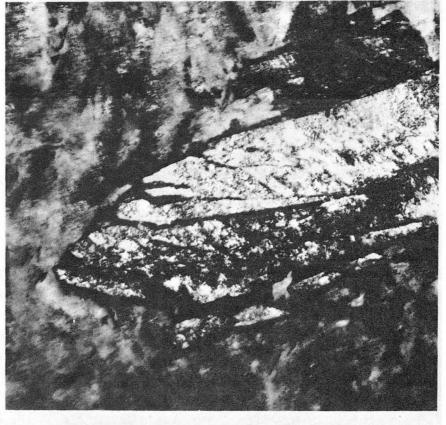
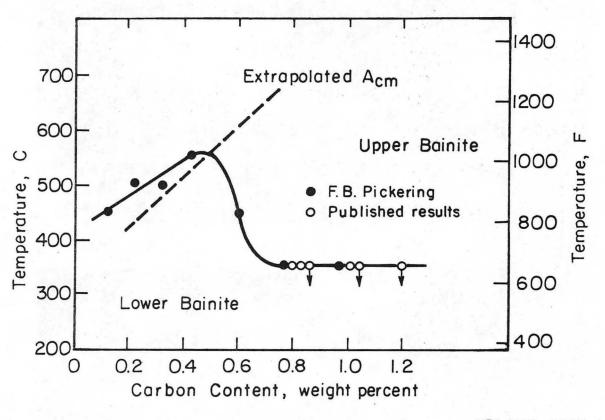


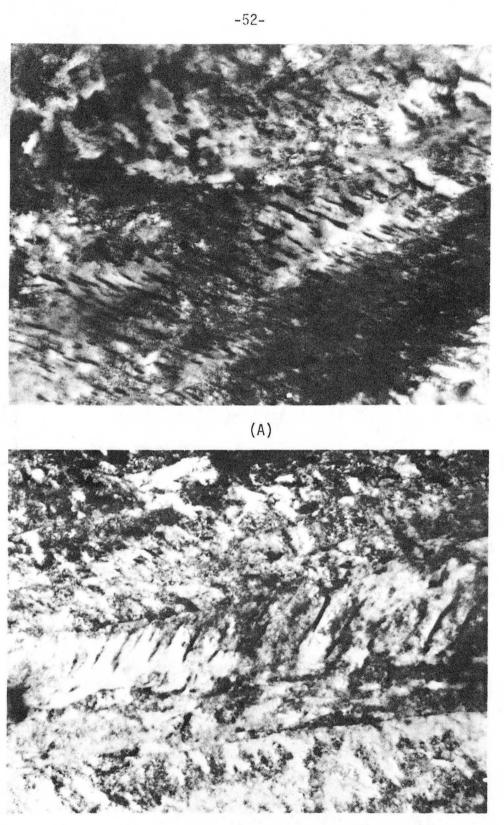
Fig. 16

XBB 747-4513



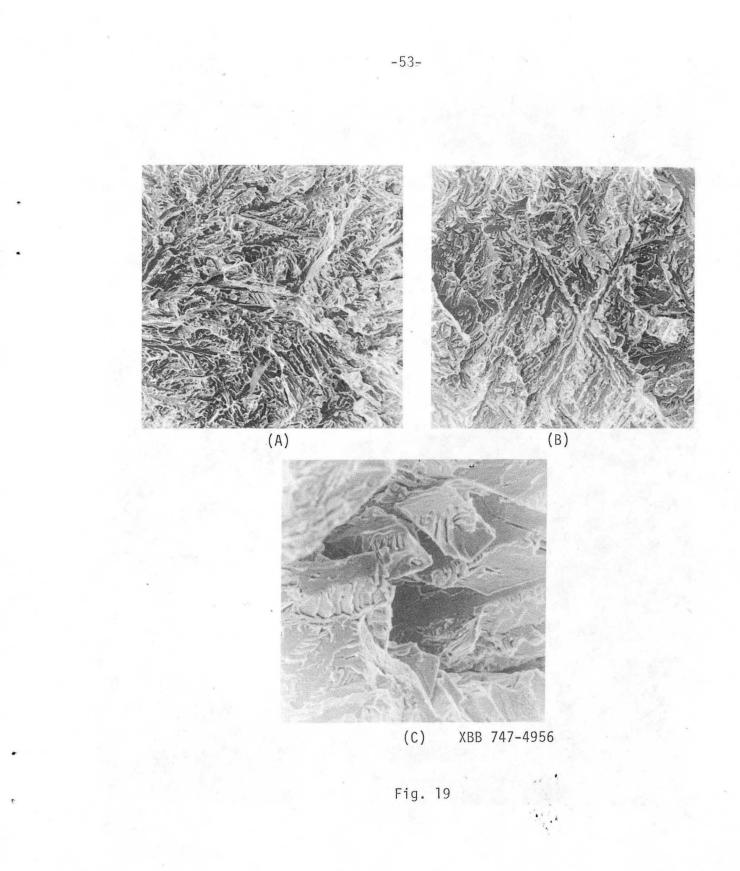
XBL 747-6683

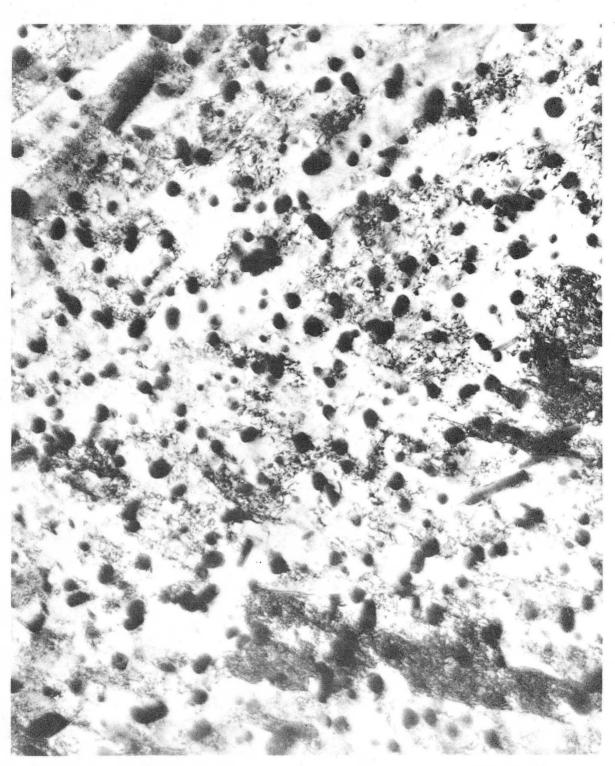
Fig. 17



(B)

XBB 747-4956





XBB 747-4954



XBB 747-4955

LEGAL NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Atomic Energy Commission, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

,

لر

.

.

,

TECHNICAL INFORMATION DIVISION LAWRENCE BERKELEY LABORATORY UNIVERSITY OF CALIFORNIA BERKELEY, CALIFORNIA 94720