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RELATIONSHIPS BETWEEN MICROSTRUCTURE AND FRACTURE TOUGHNESS IN A SECONDARY HARDENING STEEL

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RELATIONSHIPS BETWEEN MICROSTRUCTURE AND FRACTURE TOUGHNESS IN A SECONDARY HARDENING STEEL

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ABSTRACT

The relationships between microstructure and fracture toughness in a 0.3% C, 5% Mo steel have been studied. The techniques of transmission electron microscopy, scanning electron microscopy, fracture toughness testing, and stress wave emission detection were used to characterize the structure. The microstructure of quenched and tempered structures consisted of a ferritic matrix containing a dispersed precipitate of Fe₃C or Mo₂C, depending on the tempering temperature. Fracture tests were conducted both at room temperature and -78°C. The plane strain fracture toughness showed a marked dependence on microstructure. It was found that sharp decreases in toughness were due to preferential precipitation of carbides on martensite laths boundaries. Because the steel studied was a secondary hardening type steel, correlations could be made of microstructure and fracture toughness at similar yield strengths.

Of particular interest were the high toughness values observed for specimens tempered at low temperatures (< 225°C). The fracture toughness of as-quenched specimens tested at room temperature was 100 ksi-in.¹/² at a yield strength of about 220 ksi. High austenitizing temperatures favored high toughness.
I. INTRODUCTION

In the past decade there have been many new applications for high strength steels. Concomitant with the need for high strength has been the requirement of high toughness in these steels. Thus, there has been great interest in reaching a fundamental understanding of fracture toughness and how this property is affected by microstructural features. Unfortunately, only a few transmission electron microscopy studies relating microstructure to fracture toughness have been attempted.\(^1\)\(^-\)\(^6\) One possible reason for this is that both transmission electron microscopy and fracture toughness testing techniques are both relatively new fields of study. The investigations that have been completed have either neglected certain aspects of sound transmission microscopy practice or have not determined fracture toughness values in accordance with current recommended procedure.

The purpose of the present investigation was to examine the relationships between plane strain fracture toughness and microstructure of a 0.3% C, 5% Mo steel subjected to various heat treatments. This was to be accomplished by full use of transmission electron microscopy, scanning electron microscopy, and fracture toughness testing techniques. Stress wave emission was also to be used as an aid in interpretation of the fracture tests.

The particular steel used in this study is classified as a secondary hardening steel, and was chosen because of its unique hardening response on tempering after quenching from the austenitic state. Unlike low alloy and plain carbon steels whose hardness continuously decreases with increasing tempering temperature, secondary hardening steels possess a
peak in hardness at 500-600°C before softening occurs at higher temperatures. Raynor et al. 7 have studied a very similar steel (0.2% C, 4% Mo) and have established the hardness/microstructure relationships in this system. They have observed that the peak hardness value is almost identical to that for the as-quenched specimen. They have also observed that in the overaged condition, Mo₂C is the only precipitate present in the microstructure. Both of these factors were desirable for the present investigation.

The secondary hardening behavior of the steel used in this study was particularly advantageous, because it allowed toughness/microstructure correlations to be made at approximately the same strength level. (This is always desirable in fracture toughness studies.) Thus, the toughness of a peak hardness structure (containing a Mo₂C precipitation strengthened matrix) could be compared with that of an as-quenched martensitic structure. Likewise, toughness comparisons could be made between tempered structures containing only Fe₃C precipitates with those containing only Mo₂C precipitates.

One additional comment should be made and that relates to the choice of the particular steel composition of 0.3% C, 5% Mo. There were two reasons for selection of this particular composition. (1) The 0.3% carbon level was chosen so that yield strengths in the neighborhood of 200,000 psi could be attained on quenching and tempering. (High strength levels are essential for practical plane strain fracture toughness determinations.) (2) The 5% Mo concentration was chosen so that on overaging probably only a single carbide (Mo₂C) would exist in the microstructure.
II. EXPERIMENTAL PROCEDURE

A. Material Preparation

The steel used for this investigation was made by vacuum induction melting. The chemical analysis of the four 20 lb. ingots studied is given in Table I. The as-cast ingots were homogenized for 72 hr at 1100°C and forged at 1100°C into 3/4 in. by 2-1/2 in. bars. The bars were then hot rolled at 1100°C to a 5/8 in. by 2-1/2 in. cross-section. Specimens for mechanical testing were cut from the rolled bars and austenitized at 1220°C in an argon atmosphere for 1 hr. Following this treatment they were ice brine quenched and refrigerated in liquid nitrogen. The tempered specimens were held in a salt bath for 1 hr.

B. Mechanical Testing

1. Hardness Tests

Vickers microhardness values of heat treated specimens were determined using a Leitz Wetzlar microhardness testing unit with an applied load of 500 g. Hardness specimens of 5/16 in. by 5/16 in. by 3/8 in. were heat treated, sectioned, and mounted in 1 in. diameter Koldmount molds. Standard metallographic wet grinding and polishing were done, and then hardness indentations were made on the polished surface. Both diagonals of the indentation were measured and the average value of hardness recorded. At least six indentations were made on each specimen and the hardness values reported for each specimen are average values.

2. Tensile Tests

Tensile properties were determined using the 1 in. gage length, 1/4 in. diameter round specimen shown in Fig. 1. The specimens were
originally machined oversize, heat treated, and subsequently ground to final dimensions. A 300 kip capacity MTS machine was used to test specimens at a loading rate of 0.04 in./min. Tests were conducted at both room temperature and -78°C (in a mixture of ethyl alcohol and dry ice).

3. Fracture Tests

Both Charpy impact testing and plane strain fracture toughness testing were employed to determine fracture properties. Figure 2 illustrates how both specimen types were cut from the as-rolled bar stock. The standard Charpy V-notch specimen and a 120-ft-lb capacity machine were used in determining room temperature impact properties. The specimens were machined to final dimensions (with the exception that the V-notch was not cut) and then heat treated. Each heat treated specimen was then V-notched to standard notch dimensions and subsequently tested. At least two tests were run for each heat treatment and the impact values reported are average values.

Plane strain fracture toughness testing was performed using the 2-1/2 in. square, 9/16 in. thick crackline loaded specimen shown in Fig. 3. These specimens were machined oversize, heat treated, and then machined to final dimensions. The critical stress intensities for this specimen geometry were determined from the boundary collocation solution given by Srawley and Gross. These authors determined the stress intensity as a function of specimen geometry and loading and expressed their solution in the following form:

\[ K = \frac{P}{BW^{1/2}} f\left(\frac{W}{W^*}\right) \]
where $K$ is the stress intensity, $P$ the load, $B$ the thickness, $W$ the specimen width, and $a$ the crack length. In Fig. 4 is given the $\frac{KBw^{1/2}}{P}$ vs $\frac{a}{W}$ curve determined for the $W$ and $H$ values of this specimen ($W = 1.875$ in. and $H = 1.25$ in.), where $H$ is the half specimen height.

A 300 kip capacity MTS machine was used to fatigue precrack the specimens at a cycling rate of 6 cycles/sec. The cracks were fatigued to a minimum length of 0.10 in., requiring approximately 20,000 cycles for this growth. Maximum loads were kept high enough to provide reasonable fatigue crack growth rates (approximately 10,000 cycles per 0.05 in. crack growth), and yet not so high that loading exceeded the recommendations outlined in ASTM publications.\(^9\)\(^{-10}\) These recommendations were:

\[
\frac{K_{\text{max}}}{E} < \approx 0.0012 \text{ in.}^{1/2}
\]

\[
\left(\frac{K_{\text{max}}}{\sigma_y}\right)^2 < \approx 0.02 \text{ in.}
\]

\[
K_{\text{max}} < \left(\frac{K_Q}{2}\right)
\]

Where $K_{\text{max}}$ is the maximum stress intensity used during fatiguing, $E$ is Young's modulus, $\sigma_y$ is the yield strength, and $K_Q$ is the conditional critical stress intensity determined in the subsequent fracture test.

The fracture toughness specimens were tested on both the MTS machine and a 5000 kg capacity Instron. Tests were conducted at room temperature and at \(-78^\circ\text{C}\) (in a mixture of ethyl alcohol and dry ice).
A crack-opening-displacement gage similar to that described in the ASTM standards was used to monitor crack length during testing. The calibration curve of crack-opening-displacement \((v)\) vs ratio of crack length to specimen width \(\left(\frac{a}{W}\right)\) is given in Fig. 5. During testing load and crack-opening-displacement were recorded on an X-Y recorder. Determination of critical stress intensity values \((K_{IC} \text{ and } K_Q)\) values) was made in accordance with the procedure outlined by the ASTM.

4. Stress Wave Emission

The detection of slow crack growth prior to catastrophic failure in all fracture toughness tests was made using the stress wave emission (SWE) technique developed by Gerberich et al.\textsuperscript{11-13} This technique utilizes a piezoelectric transducer attached to the specimen for detection of elastic waves (stress waves) associated with the energy release of crack growth.

A schematic diagram of the SWE experimental apparatus is shown in Fig. 6. A model 2234E Endevco accelerometer was attached to one of the specimen grips during testing. This piezoelectric transducer had a charge sensitivity of 59.4 picocoulomb/g and a major resonant frequency of 34 kHz. The signal from the accelerometer was amplified by charge and voltage amplifiers for a total gain of approximately twelve. The amplified signal was then passed through a band pass filter to filter out extraneous noise. The band filter was set at a low pass of 40 kHz and a high pass of 25 kHz for tests run on the MTS machine, and the high pass was changed to 10 kHz for tests run on the Instron. The signal was then fed in parallel to an oscilloscope for observation during testing, and also to an Ampex tape recorder which had a maximum
frequency response of 40 kHz at the recording speed used (15 in./sec).

After the completion of a test, the tape was played back at a reduced speed (1/8 of the recorded speed) onto a model 1108 Visicorder oscillograph of appropriate frequency response (5 kHz) for detailed analysis of the stress wave history.

C. Microscopy

1. Optical Metallography

The hardness specimens discussed previously were used for optical metallographic observation. These specimens were sectioned, mounted in Koldmount, abraded on silicon carbide papers down to 600 grit, and polished on a 1μ diamond abrasive wheel. Specimens were etched with either 1%, 2%, or 5% nital for 5 to 30 sec.

2. Transmission Electron Microscopy

Thin foils were observed with a Siemens Elmiskop IA microscope operated at 100 kV and a Hitachi HU625 microscope operated at 625 kV. Foil preparation consisted of chemically thinning 75 mil thick sections to 4 mils, followed by jet polishing. The sections were taken from bulk fracture toughness specimens. The chemical thinning solution was 5% hydrofluoric acid in hydrogen peroxide. A twin jet electropolishing unit was used at 45-53 V and 20-25 mA, with a solution of 100g of Na₂CrO₄ in 500 ml of acetic acid. Spark cutting was used to obtain the sections from the as-quenched fracture toughness specimens, while friction cutting was used for the other specimens.

3. Scanning Electron Microscopy

The fracture surface of each fracture toughness specimen was examined using a Jeolco JSM-U3 scanning electron microscope (SEM) with
secondary emission at 25 kV. The fracture specimens were sectioned to dimensions suitable for insertion of the fracture pieces into the SEM. The fracture surfaces were covered with acetate tape during the cutting operation. This tape was then stripped and the fracture surfaces ultrasonically cleaned with acetone.
III. RESULTS AND DISCUSSION

A. Mechanical Properties

1. Hardness and Tensile Properties

The 0.3% C, 5% Mo steel used in this investigation is classified as a secondary hardening type steel. Secondary hardening steels have been the subject of many studies over the years. The principal characteristic of these steels is the unique retention of hardness up to high tempering temperatures in the quenched and tempered state. Unlike low alloy and plain carbon steels whose hardness continuously decreases with increasing tempering temperature, these steels often possess a peak in hardness which occurs in the 500 to 600°C tempering range. This behavior is due to the large amount of carbide forming elements present in the composition. The most common elements which cause secondary hardening are molybdenum, vanadium, chromium, and tungsten. The investigators who have studied secondary hardening steels attribute the hardness peak to the precipitation of an alloy carbide at the high tempering temperatures. The most extensive and systematic studies of the microstructural changes which occur in these steels have been conducted by Honeycombe and his co-workers. They have studied both simple and complex alloy steels and have shown that the hardness peak is always associated with the precipitation of an alloy carbide.

The hardness vs. tempering temperature curve for the steel used in this study is given in Fig. 7. The data for this curve were obtained from hardness specimens that were austenitized, quenched, and tempered as described previously. Since the purpose of this investigation was to
compare the fracture behavior of specimens having similar strength levels (and yet having different microstructures), it was decided that specimens tempered at 300, 500, 600, and 650°C, as well as an as-quenched specimen, should be examined. The hardnesses (and therefore the strength levels) of these specimens allowed comparison of the as-quenched specimen with the specimen tempered at 600°C (both having a Vickers hardness of approximately 675). Comparison was also possible between the specimens tempered at 300, 500, and 650°C (all having Vickers hardness values of approximately 510). It was anticipated from existing published reports that each of these specimens selected should possess a significantly different microstructure. After fracture tests were completed on these specimens, it was decided that specimens tempered at 150 and 225°C would also be of interest and so tests were also run on these specimens.

The room temperature tensile properties for both longitudinal and transverse tests are given in Tables II and III and also in curves in Figs. 8 and 9. The longitudinal -78°C tensile properties are given in Table IV and in Fig. 10. The values of yield strength ($\sigma_y$), ultimate strength ($\sigma_u$), elongation, and reduction of area were determined by conventional means. The values of the strain hardening coefficient ($n$) and true strain at fracture ($\varepsilon_f$) were determined from the following equations:

$$n = \varepsilon_u = kn(1 + \varepsilon_u)$$

$$\varepsilon_f = \ln \left( \frac{A_0}{A_f} \right)$$

where $\varepsilon_u$ is the true ultimate strain, $\varepsilon_u$ the true engineering strain,
the original cross-sectional area, and \( A_f \) the cross-sectional area at fracture.

It can be seen that the \( \sigma_y \) and the \( \sigma_u \) vs. tempering temperature curves of Figs. 8(b), 9(b), and 10(b) showed the same trend observed in the hardness vs. tempering temperature curve of Fig. 7. The strength levels are initially high at low tempering temperatures, then decrease to a somewhat lower level in the intermediate tempering range (300-500°C), reach a peak for specimens tempered at 600°C, and finally drop off rather markedly at tempering temperatures greater than 600°C. The specimens tempered at 300, 500, and 650°C had similar \( \sigma_y \) and \( \sigma_u \) values; however, the as-quenched and 600°C specimens had significantly different \( \sigma_y \) values but similar \( \sigma_u \) values. Thus, comparison of fracture behavior and microstructure between these sets of specimens was made at either constant \( \sigma_y \) and/or \( \sigma_u \) levels.

As shown in Figs. 8(a), 9(a), and 10(a), the percent elongation and reduction of area varied inversely with the strength levels measured. However, there was a significant decrease in these properties at the high tempering temperature. Thus, a specimen tempered at 600°C and tested at room temperature had no appreciable elongation or reduction of area while an as-quenched specimen, having approximately the same ultimate strength, had an elongation of 10% and a reduction of area of 30%. Similarly, a specimen tempered at 650°C had much lower values of percent elongation and reduction of area than specimens tempered at 300 and 500°C which had yield and ultimate strengths similar to that of the 650°C specimen.
2. Fracture Properties

Fracture mechanics testing techniques were used almost exclusively for fracture testing. A few Charpy impact tests were conducted, but the primary interest was in measuring fracture toughness. The main advantage of measuring fracture toughness over Charpy impact behavior is that plane strain fracture toughness \( K_{IC} \) is a material property while the Charpy impact value gives only a relative indication of toughness under extreme loading conditions. This means that significant correlations between \( K_{IC} \) and other material properties or between \( K_{IC} \) and microstructural features should be possible.

When studying quenched and tempered secondary hardening steels, it is extremely important that the initial quenched product be 100% martensite. Otherwise, more complicated transformations occur on tempering and the hardness results which are obtained are ambiguous. This problem was encountered by Irani and Honeycombe,\(^{27,28}\) who studied the microstructure and hardness behavior of several secondary hardening steels which contained selected trace amounts of various elements. They were unaware, however, that the as-quenched material was not martensitic. As a consequence, all the hardness results they obtained and the conclusions they made regarding the effect of trace elements were unreliable, because these could not be duplicated by subsequent workers studying rapidly quenched 100% martensitic material. The fracture toughness specimen used in this investigation had a 9/16 in. thickness, and before experimental tests were begun this specimen size was checked for through-thickness hardenability to martensite by using both optical metallography and microhardness measurements. It was found that water
quenching was probably sufficient to produce thorough hardening, but as added insurance the more rapid ice brine quenching was subsequently used for all heat treatments. The observation of through-thickness transformation to 100% martensite was later confirmed by transmission electron microscopy.

The fracture toughness values for both longitudinal and transverse specimens tested at room temperature are given in Tables II and III. Line drawings of this data, showing $K_{IC}$, $\sigma_y$, and $\sigma_u$ vs. tempering temperature and $K_{IC}/\sigma_y$ vs. tempering temperature are shown in Figs. 11-14. The fracture properties of longitudinal specimens tested at -78°C are given in Table IV and Figs. 15 and 16. Valid plane strain fracture toughness values were obtained for all specimens tested except for the 150 and 225°C tempered specimens tested at room temperature. The values given for these specimens are apparent toughness values ($K_Q$) and were not valid $K_{IC}$ values because of two reasons:

1. the value of $2.5 (K_{IC}/\sigma_y)^2$ for each specimen was greater than the specimen thickness (9/16 in.) and
2. the deviation from linearity of the load (P) vs. crack-opening-displacement (v) curve was greater than that recommended in ASTM publications.\textsuperscript{9,10} The fracture tests for these specimens are very close to meeting plane strain requirements, however, and their apparent toughness values are indicative of relative toughness in this investigation.

Three types of load vs. crack-opening-displacement curves were observed for the specimens tested. A typical example of each type is shown in Figs. 17, 18, and 19. Most specimens failed in the manner depicted in Fig. 17, which is the room temperature test record for the
transverse specimen tempered at 650°C. Except for one discontinuous increase in the crack-opening-displacement, this type of loading curve exhibits linearity up to instability. The discontinuity in the loading curve is due to crack pop-in, and the $K_{IC}$ values were calculated from the pop-in points on these curves if $v$ exceeded the minimum recommended by ASTM. The second type of loading curve observed was that shown in Fig. 18, which is the room temperature fracture test for the transverse specimen tempered at 300°C. This type of failure was characterized by linearity up to instability with no pop-in present. Thus, $K_{IC}$ values for specimens failing in this manner were determined from the $P$ and $v$ at instability. The third type of $P$ vs. $v$ curve was common to all specimens tempered at 225°C and lower and tested at room temperature. A typical curve of this type is shown in Fig. 19, which is the $P$ vs. $v$ curve for the transverse as-quenched specimen tested at room temperature. This curve is characterized by deviation from linearity prior to instability and this deviation is indicative of slow crack growth occurring before instability. The deviation from linearity observed in Fig. 19 was within the acceptable limits prescribed by ASTM; however, specimens tempered at 150 and 225°C possessed deviations too large for valid plane strain toughness determinations.

All three sets of fracture toughness data illustrated in Figs. 11 through 16 show the same general behavior. This consists of very high toughnesses at low tempering temperatures ($<225°C$), followed by a sharp drop in toughness at 300°C and another drop at 600°C. The $K_{IC}$ values obtained for specimens tempered above 225°C were as anticipated and were consistent with $K_{IC}$ values determined for other steels of the
same strength level. However, the high $K_{IC}$ and $K_Q$ values obtained at low tempering temperatures were not expected and were considerably higher than $K_{IC}$ values of other steels of similar strength. Steigerwald\textsuperscript{32} has prepared a plot of plane strain fracture toughness vs. yield strength for several commercial steels, and this plot is shown in Fig. 20. This information was gathered from steels which had been given standard commercial heat treatments of quenching and tempering. These data give a good indication of the relative toughness of the low tempering temperature specimens of this investigation. For instance, the as-quenched specimens had room temperature yield strengths of approximately 220 ksi and $K_{IC}$ values of about 100 ksi-in.$^{1/2}$. From Steigerwald's plot at 220 yield the only material of comparable toughness is the 18\% Ni maraging steel, and the widely used low alloy steels (4340, D6AC, H-11 and 300M) are significantly lower in toughness. A similar comparison for the room temperature properties of the 150 and 225\°C tempered specimens reveals that these also possess significantly higher toughness values when compared with the other materials at the same strength level. Steigerwald's plot does not contain, however, the information necessary for a direct correlation of toughness for similar heat treatments and carbon levels. No such data of $K_{IC}$ at low tempering temperatures for 0.3\% C steels have been found in the literature. There does exist some unpublished work done at Lawrence Berkeley Laboratory\textsuperscript{33} on 4130 which was heat treated as outlined in this investigation. These workers found that the $K_{IC}$ values for the as-quenched 4130 were almost identical to those obtained for the 0.3\%C, 5\% Mo steel of the present work. Further work by these researches on the 0.3\% C, 5\% Mo steel showed that for any
heat treated specimen $K_{IC}$ was a function of austenitizing temperature, and that there existed a small range of austenitizing temperatures (1840-2040°F) over which a large change in toughness occurred. The significance of this observation will be discussed later when microstructural effects on fracture toughness are considered.

Room temperature Charpy V-notch impact tests of the 0.3% C, 5% Mo steel were also conducted to see if similar toughness behavior of tempered specimens existed for impact testing. The results are listed in Table V and are shown in Fig. 21. As is evident the CVN values show similar behavior to that observed in plane strain fracture toughness testing.

3. Stress Wave Emission

Stress wave emissions were monitored during each fracture toughness test using the apparatus described earlier. By proper selection of transducer, pass band filter settings, and system sensitivity, it was possible to detect stress wave activity in all specimens at loads considerably below the critical loads (approximately $3/4$ of $P_{CRIT}$). Each stress wave emitted corresponded to a mechanical crack movement within the specimen, either at the macroscopic moving crack interface or as a microcrack away from the main crack interface. It was possible to make this distinction because of the choice of the monitoring system. This has been fully discussed by Gerberich et al. and the equipment used here is identical to that described in that reference.

As shown in Fig. 6 the stress wave activity for each test was recorded on magnetic tape and then played back onto oscillograph paper for interpretation. The recorded signal was played back through the
pass band filter as a matter of convenience and the filter settings were such that actually no further filtering occurred. Representative stress wave emission oscillograph records for five transverse specimens tested at room temperature are given in Figs. 22 through 24. These plots represent acceleration vs. time information, with the time lines being 1/8 sec apart. These records were taken at approximately 95% $P_{\text{CRIT}}$ loading and were taken for the as-quenched, 300, 500, 600, and 650°C tempered specimens. These tests were run on the MTS. The peaks occurring in each of these records correspond to individual stress waves associated with crack movement. To determine whether this crack movement is associated with cleavage or ductile failure, scanning electron fractography must be used. As is evident from these figures the as-quenched specimen was subject to extensive slow crack growth prior to instability, while the other specimens were relatively inactive. These data are useful in study of slow crack growth, but this type of growth was only observed on six of the 21 specimens tested. The stress wave emission data were also useful in interpretation of load/crack-opening-displacement records, being extremely helpful in the determination of pop-ins.

B. Microscopy

1. Optical Microscopy

Optical metallography was completed for both longitudinal and transverse sections of all tempered specimens. The terminology of longitudinal and transverse used here is different from that used in describing the fracture toughness specimens. Here longitudinal refers to observation of a surface parallel to the rolling direction and transverse refers to observation of a surface perpendicular to the rolling direction.
However, longitudinal and transverse, as used in the description of fracture toughness testing, referred to the direction in which the specimens were pulled with respect to the rolling direction. Thus, the fracture surface of a specimen pulled in the longitudinal direction was parallel to a transverse metallographic specimen surface. Likewise, the fracture surface of a specimen pulled in the transverse direction was parallel to a longitudinal metallographic specimen surface. This differentiation should be kept in mind when comparison of optical micrographs are made with scanning electron micrographs of the fracture surfaces.

Optical micrographs of as-quenched, 300, 600, and 650°C tempered specimens are given in Figs. 25-31. Figures 25-29 are micrographs of transverse specimens, while Figs. 30 and 31 are of longitudinal specimens. These micrographs show that a dominant feature of all specimens was a large prior austenite grain size. The prior austenite grain size of the transverse specimens was quite large and there was a wide range in grain size, but most grains were about 300-400 μ in size. However, the longitudinal prior austenite grain size was only about half that for the transverse, being about 150 μ in size. The prior austenite grain size is most clearly evident in the low magnification micrographs in Fig. 25 (as-quenched transverse section) and Figs. 30 and 31 (as-quenched and 600°C tempered longitudinal sections, respectively).

The as-quenched structure was uniformly martensitic, with no other transformation products present in the microstructure. This is clearly evident in Figs. 25, 26, and 30. Systematic measurement of microhardness across the section confirmed that a fully martensitic structure was present. X-ray diffraction of electropolished sections of
as-quenched fracture toughness specimens also showed that no detectable retained austenite was present in the microstructure. The martensitic structure was a mixture of laths and plates. The plate size was variable, with some plates being on the order of several microns in width while most were about one to two microns in size. The lath width was less than a micron and was measurable best in transmission electron micrographs.

The basic martensitic configuration was evident in all the tempered specimens and there was no indication of recrystallization even up to 650°C in tempering. This observation was later confirmed by transmission electron microscopy. Although all the tempered specimens etched differently, it was not possible to detect individual carbide precipitates in these specimens, because of the small size (≤ 0.2 μ) of the carbides.

2. Transmission Electron Microscopy

Transmission electron microscopy of thin foils was essential for characterizing the microstructure of each toughness specimen. To insure there would be no ambiguity in correlating microstructure with fracture toughness, the foils were taken directly from the bulk toughness specimens. This precaution is not always adhered to by workers in this field and there are cases where there is reason to question whether the bulk microstructure is the same as that of identically heat treated thin sheets that were examined. After the transmission microscopy of this investigation was completed, it was quite evident that there would have been a discrepancy between heat treated thin sheets and bulk specimen microstructures. This difference would have been due to autotempering which occurred during the quench from austenitization for the bulk
fracture specimens but not for thin sheets. This autotempering changes the tempering behavior of the steel up to fairly high tempering temperatures.

A twin jet polishing technique was used to perform final thinning of foils. In this technique a small disk is punched from the foil, and subsequently placed in the polishing unit and thinned for observation in the microscope. The disks can be punched from any part of the foil and the relative position of the disk noted. In the case of the foils prepared in this investigation, this allowed checking the microstructure for uniformity across the thickness of the fracture toughness specimen. This was possible because the width dimension of the foil was actually the thickness dimension of the fracture toughness specimen from which it was taken.

Thus, two significant points about the transmission microscopy examination were considered extremely important. These were: (1) the fact that foils were taken directly from fracture toughness specimens, and (2) the fact that the microstructure of all toughness specimens could be checked for uniformity. Establishment of these two key points in this investigation makes the conclusions made unambiguous.

Reviewing the toughness results of Figs. 11 through 16, there were three tempering ranges of interest. These were temperatures \(< 225^\circ C\), temperatures in the 300-500\(^\circ\)C range, and temperatures 600\(^\circ\)C and higher. It was particularly important to identify microstructural features that could explain: (1) the high toughness exhibited by specimens tempered at 225\(^\circ\)C and lower, (2) the sharp drop in toughness occurring between 225\(^\circ\)C and 300\(^\circ\)C, and (3) the embrittlement which occurs at high
temperatures (≈ 600°C). It was felt at the outset that one possible reason for the abrupt changes in toughness that were observed for this steel could be the presence of preferential precipitation. This type of precipitation might be at either grain boundaries or martensite lath or plate boundaries. Thus, particular attention was given to this aspect of the microstructural study. It was found that preferential precipitation could best be detected by foil tilting and scanning in dark field illumination. So, in addition to the normal procedure of imaging, tilting, and scanning in bright field, this additional investigation was completed for every specimen studied.

As-quenched structure. The structure of the as-quenched specimen consisted almost entirely of a dislocated lath type martensite. There were also present a substantial number of large martensite plates within the structure; however, there were only isolated instances of internal twinning within these plates. This martensite morphology is quite common and is similar to that first reported by Kelly and Nutting.35,36 These authors showed that in Fe-C and Fe-Ni-C alloys, there exists a transition in martensite morphology. The martensite changes from a twin-free lath type at low carbon levels to a twinned plate type at high carbon levels. At intermediate carbon levels a mixed morphology of laths and twinned plates exists. The 0.3% carbon level of the steel of this investigation is at a level where either a lath structure or a mixed morphology containing both laths and plates would be expected. A somewhat mixed morphology was observed; however, there was a definite lack of twinning within the plates that were present.
Another prominent feature of the as-quenched specimen was the occurrence of autotempering. The precipitates associated with autotempering are shown very clearly in the bright and dark field micrograph pairs of Figs. 32 and 33. The dark field micrographs in each of these figures was taken from a cementite reflection and shows reversal of contrast for the precipitates. Thus, the fine Widmanstatten dispersion of autotempered precipitates was identified as Fe$_3$C, having (110) habits. Identification consisted of selected area electron diffraction and trace analysis. Autotempering occurred because of the comparatively large thickness of the fracture specimen from which the foil was taken (9/16 in.), and because of the relatively high $M_S$ temperature for this alloy (estimated to be approximately 365°C).37

In analyzing the micrographs of this specimen, special care was taken to see if epsilon carbide precipitates were present within the microstructure. It is known that epsilon carbide has (100) habit in contrast to the (110) habit of cementite. Under many circumstances in tempered steels epsilon carbide does not give electron diffraction patterns capable of being easily indexed. Consequently, trace analysis is usually the best way of detecting the presence of this carbide. Also, the apparent waviness of this precipitate in bright field distinguishes it from cementite. In the case of the as-quenched steel of this study, no epsilon carbide precipitates could be identified. Cementite was the only precipitate positively identified. This observation is consistent with what has been reported previously for autotempered as-quenched martensites.39,40
Special care was also taken to foil tilt and scan in dark field illumination to see if preferential precipitation of any kind had taken place on boundaries. No such precipitation was observed.

225°C tempered structure. It was of prime interest to see if any dissimilarity in structure existed between the as-quenched specimen and the 225°C tempered specimen, since no significant difference in fracture toughness had been recorded between these specimens. Examination of the 225°C tempered specimen revealed essentially the same microstructure as noted for the as-quenched specimen. The basic lath type martensite characteristic of all the specimens examined in this investigation is displayed in the bright field micrographs of Figs. 34 and 35. Figure 35 has a rather large martensite plate visible in the micrograph and this size plate was typical of plates that were prevalent throughout the microstructure of all the specimens studied.

Again, as in the as-quenched specimen, Widmanstatten precipitation within highly dislocated laths and plates was seen. This precipitation is shown in the bright and dark field micrograph pair in Fig. 36. The reversal of contrast shows the precipitates are cementite, since the dark field was taken from a cementite reflection. No epsilon carbide precipitates were observed. This is consistent with the same observation that was made for the as-quenched specimen. Epsilon carbide precipitation would certainly not be expected in a 225°C tempered specimen which had contained a large amount of autotempered Fe₃C precipitates even before tempering.

Extensive foil tilting and scanning was conducted in dark field illumination in search of preferential precipitation at lath boundaries.
As was the case for the as-quenched specimen, no such precipitation was detected.

**300°C tempered structure.** The microstructure of the specimen tempered at 300°C differed markedly from the structures observed for the as-quenched and the 225°C tempered specimens in that extensive Fe₃C precipitation at lath boundaries was found. The bright and dark field micrograph pair in Fig. 37 shows very clearly cementite precipitation both within laths and at lath boundaries. Widmanstatten precipitation of Fe₃C occurred within the laths, as noted earlier for the as-quenched and 225°C tempered specimens. The lath boundary precipitation was extensive throughout the microstructure and was not limited to only a few laths. In looking at the bright field micrographs it was not at all obvious that a precipitate was present at the lath boundaries. Dark field micrographs, however, very clearly and unambiguously showed cementite precipitation at lath boundaries. This illustrates how important it is to conduct foil tilting and scanning in dark field illumination. Had this procedure not been employed the preferential precipitation would most likely not have been observed in this specimen.

Lath boundary precipitation of cementite at 300°C tempering temperature is not unexpected. Other workers have noted this type of precipitation occurring in steels of similar carbon level and tempering treatments. Specifically, Huang and Thomas have seen cementite lath boundary precipitation at tempering temperatures as low as 205°C (for 2 + 2 hours of tempering).
500°C tempered specimen. The specimen tempered at 500°C also had the preferential precipitation of Fe₃C that was characteristic of the 300°C tempered specimen. However, at 500°C tempering the lath boundary precipitation was more evident and could be rather clearly seen even in bright field illumination. The micrographs of this specimen are given in Figs. 38 and 39. The two sets of bright and dark field pairs show the cementite precipitates both within laths and at their boundaries. The dark field micrographs were taken from cementite reflections in selected area diffraction.

From the work of Honeycombe and his fellow researchers,⁷ ²⁹ it might be expected that Mo₂C precipitation could also take place at this tempering temperature. (However, Honeycombe and his co-workers never did observe Mo₂C precipitation at tempering temperatures as low as 500°C after only one hour of tempering.) A very exhaustive effort was conducted to see if Mo₂C precipitates were present in the 500°C tempered specimen. Study consisted both of bright and dark field foil tilting and scanning, but these proved unsuccessful in revealing any Mo₂C precipitation. If Mo₂C were present in the microstructure it would be present in the form of very small coherent rods (perhaps less than 15 Å in diameter and 100 Å in length⁷). This small size makes observance of Mo₂C very difficult if only bright and dark field imaging techniques are used. An electron diffraction effect, peculiar to very fine precipitates, makes identification of Mo₂C more probable. This is accomplished by using information available with selected area electron diffraction. The extremely fine size of Mo₂C during the early stages of formation causes streaking to appear in selected area electron
diffraction patterns. Streaking of this type is common to coherent precipitates and a complete discussion of this effect has already been presented.\textsuperscript{42} As a last effort in identifying any Mo\textsubscript{2}C which might be present at this tempering temperature, exhaustive foil tilting and scanning were conducted in the selected area electron diffraction mode. Careful attention was given to observation of streaks, but no streaking was seen. Thus, no Mo\textsubscript{2}C precipitation at 500°C tempering could be established. This is in agreement with the findings of Murphy and Whiteman,\textsuperscript{43} who studied the kinetics of Mo\textsubscript{2}C formation at temperatures between 500 and 600°C. They found that at 500°C after one hour no appreciable formation of Mo\textsubscript{2}C had taken place.

\textbf{600°C tempered specimen.} A significant difference in structure existed between the 600°C tempered specimen and those tempered at 500°C and lower. No cementite was detected for this heat treatment and instead extensive Mo\textsubscript{2}C precipitation was evident. The Mo\textsubscript{2}C precipitates were hard to image in bright field, and dark field illumination proved more fruitful for observation. This precipitation was detected by both dark field imaging and the presence of streaking in selected area electron diffraction. The dark field micrograph of Fig. 40 shows the very fine Mo\textsubscript{2}C precipitates. This dark field micrograph was taken from an Mo\textsubscript{2}C streak in the diffraction pattern.

An example of the streaking that was observed in selected area electron diffraction is given in Fig. 41. Here the foil orientation is (100) and the streaks indicated by the arrows lie in the (200) directions in reciprocal space. This corresponds exactly with what has been observed before for precipitation of Mo\textsubscript{2}C in steel\textsuperscript{7,30} and is consistent with
the anticipated streaking directions for the precipitate/matrix orientation known for this system. The precipitation/matrix orientation relationship of $\text{Mo}_2\text{C}$ in $\alpha$-Fe has been established, and it has been found that $\text{Mo}_2\text{C}$ first forms as fine coherent needles with the following orientation:

$$(011)_{\alpha-\text{Fe}} \parallel (0001)_{\text{Mo}_2\text{C}}$$

$$(0\overline{1}1)_{\alpha-\text{Fe}} \parallel (01\overline{1}0)_{\text{Mo}_2\text{C}}$$

$$(100)_{\alpha-\text{Fe}} \parallel (2\overline{1}0)_{\text{Mo}_2\text{C}}$$

$$[100]_{\alpha-\text{Fe}} \parallel [\overline{1}0\overline{1}0]_{\text{Mo}_2\text{C}}$$

This is identical to what Pitsch and Schrader found for epsilon carbide in $\alpha$-Fe. The growth direction of the $\text{Mo}_2\text{C}$ needles is along the cube directions of the ferrite.

No preferential $\text{Mo}_2\text{C}$ precipitation at lath boundaries was observed in this specimen, but it was felt that this type of precipitation most likely was present yet simply wasn't detected. Extensive preferential $\text{Mo}_2\text{C}$ precipitation was observed for the specimen tempered only $50^\circ\text{C}$ higher in temperature, and it is likely similar behavior exists for the $600^\circ\text{C}$ temper. The detection of $\text{Mo}_2\text{C}$ lath boundary precipitation is extremely difficult because of the very small $\text{Mo}_2\text{C}$ precipitate size and because of the critical dependence for observation of this type of precipitation on foil orientation.
650°C tempered specimen. Observation of the specimen tempered at 650°C revealed the preferential Mo₂C precipitation at lath boundaries that could not be detected for the 600°C temper. This is shown very dramatically in the dark field micrograph in Fig. 42. Here the Mo₂C carbides are reversed in contrast and are clearly evident both at lath boundaries and within the laths. The dark field micrograph of Fig. 43 shows similar behavior, but here the Mo₂C precipitates within the laths are more evident and their fine size illustrated. The Mo₂C precipitates are harder to distinguish in bright field and the precipitates are difficult to see in bright field micrograph in Fig. 44. The dark field micrograph of this figure again shows the preferential precipitation peculiar to this heat treatment. The observation of Mo₂C precipitates at this tempering temperature is consistent with what would be expected for the Mo to C ratio of this composition.

3. Fractography

The fracture morphology of each fracture toughness specimen was thoroughly documented by means of both optical macrophotography and scanning electron microfractography. In Figs. 45, 46, and 47 are shown macrofractographs of each fracture toughness specimen tested. Two points can be made from these photographs. First, almost all specimens possessed a completely flat fracture surface characteristic of plane strain failure. Only the specimens tempered at 225°C and lower and tested at room temperature had any shear lip at all. Even these specimens had only a small shear lip (the total percentage of shear area was approximately 20% of the total fracture area). A second significant point that is evident from these optical fractographs is the difference in
surface appearance caused by a difference in grain size between the longitudinal and transverse specimens. As discussed earlier the grain size of the section parallel to the fracture surface was approximately 300-400 μ for the longitudinal fracture toughness specimens, while it was only about 150 μ for the transverse specimens. The fracture surfaces of the longitudinal specimens (of larger grain size) are shown in Figs. 45 and 47, while the transverse specimen fracture surfaces are shown in Fig. 46. Even though the fracture surfaces appeared quite different, there was no appreciable difference in the room temperature \( K_{IC} \) values measured for the transverse and longitudinal specimens.

Each fracture toughness specimen fracture surface was thoroughly examined using a scanning electron microscope. The fracture mechanisms that were noted in this study included: (1) cleavage, (2) dimpled rupture (micro-void coalescence), (3) quasi-cleavage, and (4) intergranular separation. This is the same classification used by Beachem and Pelloux and a full description of each mechanism has been given by these authors.

All scanning electron fractographs are shown in Figs. 48 through 70. The first two fractographs of this series, Figs. 48 and 49, show the intergranular separation typical of quench cracks which formed at the fracture specimen notch. These quench cracks were present in many of the specimens and can also be seen in the macrophotographs of the fracture surfaces in Figs. 45 through 47. These cracks always were at the machined notch in the toughness specimen, and since the fatigue crack was introduced at this point, these quench cracks were never a source of difficulty in testing. These cracks do point out that during
the severe ice brine quench in the heat treatment the prior austenite grain boundaries were weaker than the grains and failure occurred by grain boundary separation. Also, these first two fractographs illustrate again the difference in prior austenite grain size that existed between longitudinal and transverse specimen fracture surfaces. The quench crack in Fig. 48 shows a large grain size and the fractograph is of a longitudinal specimen, while the quench crack in Fig. 49 shows a much smaller grain size and this is of a transverse specimen.

The fractographs of Figs. 50 through 57 illustrate the fracture morphology for each longitudinal toughness specimen tested at room temperature. This series of micrographs shows very clearly that a significant change in fracture mode from primarily dimpled rupture to mostly quasi-cleavage occurred as the tempering temperature increased. For specimens tempered at 225°C and lower the fracture mode was a mixed one of dimpled rupture and quasi-cleavage. This is shown in Figs. 50, 51, and 53. As is quite evident from these fractographs, there was a large percentage of dimpled rupture associated with the failures of each of these specimens. The percent of dimpled rupture increased slightly from approximately 50% for the as-quenched specimen up to about 65% for the 225°C tempered specimen. A higher magnification fractograph of one of these dimpled rupture areas is given in Fig. 52. For specimens tempered 300°C and higher the fracture mode was entirely quasi-cleavage and cleavage, and the fractographs of these specimens are given in Figs. 54 through 57. The fracture mode of these specimens was primarily quasi-cleavage, but as the tempering temperature increased the amount of cleavage also increased. The specimens tempered at 600 and 650°C
had much more cleaved areas than did the specimen tempered at 300°C. Quasi-cleavage was marked by the presence of tear ridges and such areas are present in all low magnification fractographs of these specimens. With the exception of Fig. 55, these fractographs also show regions of cleavage present. Higher magnification photographs of the quasi-cleavage areas are given in Figs. 54 and 57.

The transverse fracture toughness specimens (which were tested only at room temperature) exhibited the same failure behavior as the longitudinal specimens tested at room temperature. Representative fractographs of selected transverse specimens are given in Figs. 58 through 63. The remarks made above concerning the longitudinal specimens apply equally as well for the transverse specimens; however, there are certain transverse specimen fractographs of particular interest. These include high magnification photographs of: a mixed area of quasi-cleavage and dimpled rupture of the as-quenched specimen (Fig. 58), a tear ridge in a quasi-cleavage area of the 300°C specimen (Fig. 60), and a quasi-cleavage area of the 300°C specimen (Fig. 61).

All longitudinal and transverse specimens tempered at 225°C and lower and tested at room temperature had areas of slow crack growth between the fatigue interface and the rapid growth area. This was known from stress wave emission data and from the load/crack-opening-displacement records. Typically the slow crack growth region would have extended several hundredths of an inch before instability. These specimens were carefully examined with the scanning electron microscope to see if any change in morphology could be detected in going from the fatigue interface into the known rapid growth area. No noticeable difference in
fracture mode was observed in any of the specimens that exhibited slow crack growth.

Fractographs for each longitudinal toughness specimen tested at 
-78°C are shown in Figs. 64 through 70. At this test temperature, no significant change in fracture morphology occurred with increasing tempering temperature. Each specimen failed by quasi-cleavage or a combination of quasi-cleavage and cleavage. There was a large percentage of cleavage present at the higher tempering temperatures. Typical quasi-cleavage areas at higher magnification are shown in Figs. 64, 66, 69, and 70.

C. Correlation of Toughness with Tensile Properties and Microstructure

1. Relation of Tensile Properties to Fracture Toughness

For many years investigators have sought to link toughness with normal uniaxial tensile properties. Before the development of fracture toughness many workers generally identified a tough high strength material as one which had either good elongation or reduction of area. As fracture toughness testing techniques have come to wide acceptance over the past few years, it has become clear that no simple relationship exists between $K_{IC}$ and elongation and reduction of area. It is expected from the physical aspects of $K_{IC}$ testing that unaxial tensile properties such as yield and ultimate strength, strain hardening coefficient, and fracture strain should be related to $K_{IC}$. Now that a sufficient amount of $K_{IC}$ data is available for a wide variety of materials, there has been greater interest in trying to establish a quantitative relationship between uniaxial tensile properties and fracture toughness. The main reason for such interest is that $K_{IC}$ values could be estimated in cases
where specimen size requirements would make valid $K_{IC}$ tests impractical.

Recently, Hahn and Rosenfield have developed a ductile fracture model which provides a quantitative relationship between plane strain fracture toughness and uniaxial tensile properties. This model is based on relating crack-tip-displacement to peak strain which is then combined with a critical strain criteria for ductile fracture. Hahn and Rosenfield proposed the following relationship:

$$K_{IC} = \left[ \frac{2}{3} E \varepsilon_f n^2 \sigma_y \right]^{1/2}$$

where $K_{IC}$ is the plane strain fracture toughness, $E$ is Young's modulus, $\varepsilon_f$ the true strain at fracture, $n$ the strain hardening coefficient, and $\sigma_y$ the yield strength. By using collected data for steel, titanium, and aluminum alloys, they found reasonable agreement between experimentally determined $K_{IC}$ values and calculated ones. They found that almost all of the experimental data fell within a $\pm 30\%$ band of that predicted by the above equation.

It was of interest then to see if the tensile data of this study could be used to calculate $K_{IC}$ values with the Hahn and Rosenfield equation which would agree with experimentally determined values. A plot of measured $K_{IC}$ values vs. $(\frac{2}{3} E \varepsilon_f n^2 \sigma_y)^{1/2}$ is given in Fig. 71. Indicated in this figure is the $K_{IC}$ (measured) = $K_{IC}$ (calculated) line plus lines representing $+30\%$ and $-30\%$ deviation. This is presented in the same manner as originally presented by Hahn and Rosenfield. Data are included from tests at both room temperature and $-78^\circ C$. As is quite evident the agreement between measured and calculated $K_{IC}$ values is very poor. Most data points fell outside the $30\%$ deviations for which Hahn and Rosenfield
expected their model to be valid. This same observation has been made by Jones and Brown\textsuperscript{50} and more recently by Vishnevsky and Steigerwald\textsuperscript{49}, who worked on a variety of medium carbon low alloy steels. Vishnevsky and Steigerwald believed the discrepancy they found was due to the failure of the Hahn and Rosenfield model to account for the microscopic failure modes which might be operating (i.e. cleavage, quasi-cleavage, or dimpled rupture). They felt that if a portion of the failure involved quasi-cleavage or cleavage, then the observed $K_{IC}$ values would be less than the predicted values. This type of discrepancy was not observed for the specimens of this study. Rather, no general trend regarding effect of microstructural failure mode was noted. The basic conclusion that could be made from the data of the present investigation is that the Hahn-Rosenfield model is incapable of predicting reliable $K_{IC}$ estimates. Furthermore, this model is remarkably insensitive to microstructural changes which in actuality cause large changes in measured $K_{IC}$ values.

Referring back to the curves of tensile data and fracture toughness values vs. tempering temperature (Figs. 8-16), it should be noted that the abrupt changes in fracture toughness are not related to a simultaneous change in ductility. Similar observations have been made by Jones and Brown\textsuperscript{50} and Brown and Srawley\textsuperscript{51}. The article by Brown and Srawley in particular reviews data that show $K_{IC}$ is not simply related to ductility. Thus, it should be emphasized strongly that good ductility does not mean good fracture toughness.
2. **Effect of Microstructure on Fracture Toughness**

There have been relatively few transmission electron microscope investigations of the effect of microstructure on fracture toughness in high strength steels. This is due to the fact that fracture toughness concepts have come into general acceptance only in the past few years and also because transmission electron microscopy has been in use only a few years longer. Almost all work that has been done has been by specialists in either transmission electron microscopy (who either ignored or misused fracture toughness testing) or fracture mechanics (who generally ignored microstructural features). Thus, the present investigation is unique in that special care has been taken to insure that both fracture toughness techniques and transmission electron microscopy were used to their fullest extent. In the case of fracture toughness testing, each test was carefully conducted to conform with current ASTM recommendations. Also, use of scanning electron fractography and stress wave emission helped in interpretation of fracture results. In the case of transmission electron microscopy, each mode of observation, i.e., bright field, dark field, and selected area diffraction, was used to its fullest extent.

In reviewing the fracture toughness results of this study, several key points should be brought out: (1) the toughness at low tempering temperatures was very high, (2) there was a sudden drop in toughness at tempering temperatures between 225 and 300°C, and (3) there was another drop in toughness at about 600°C tempering. Also, when comparing toughness at similar strength levels, the following conclusions can be made: (1) the as-quenched structure was tougher than the specimen tempered to
peak hardness by a factor of about three, and (2) the toughness decreased progressively on tempering to higher temperatures (from 300 to 500 to 650°C). This behavior was observed for both longitudinal and transverse toughness specimens tested at room temperature and for longitudinal specimens tested at -78°C. The room temperature Charpy V-notch data generally showed similar behavior to that of fracture toughness.

The points made in the previous paragraph suggest that there were two separate embrittlement phenomenon observed in this investigation. These are commonly known as tempered martensite embrittlement (400°F embrittlement) and temper brittleness (1000°F embrittlement). These embrittlements have been recognized in high strength steels for some time and many investigations of these phenomena have been made.52-59 A thorough review of these investigations has been prepared by Kula and Anctil59 and only the pertinent studies will be discussed here. There is no generally accepted theory to explain either type of embrittlement, but several factors are considered detrimental to toughness: (1) certain impurity elements60,61 (As, Sn, Sb, S, Si, P, N), (2) preferential precipitation at either prior austenite grain boundaries or martensite lath boundaries,1,55,62 (3) changes in precipitation within martensite laths,3,4 (4) the combined effect of change of precipitation within laths with the presence of an impurity element,59,63 and (5) the existence of internal twinning within martensite plates.6,41

Both embrittlements observed in this study seem to be related to a common cause, i.e. preferential carbide precipitation at martensite lath boundaries. The first drop in toughness (which occurred between 225 and
300°C in tempering) happened concurrently with the first evidence of extensive Fe₃C precipitation on martensite lath boundaries. At 650°C tempering (by which the second drop in toughness had occurred) the preferential precipitation was seen to be even more extensive than that observed at the lower tempering temperatures, and the precipitate was then Mo₂C instead of Fe₃C. The preferential precipitation is detrimental to toughness in two ways: (1) the boundary acts as a barrier to slip across lath boundaries, and (2) the boundaries become preferential crack paths. The association of embrittlement with preferential lath boundary precipitation is not a new one, but the evidence shown here is unique and unambiguous. Lement et al.⁵⁵ suggested that lath boundary precipitation of cementite was responsible for the observed embrittlement, but these workers did no transmission foil studies of the microstructure and had no solid proof of their claim. Similarly Baker et al.¹⁶² and Liu also attributed the drop in toughness to this type of precipitation, but they showed only bright field micrographs which were not at all conclusive in their evidence of preferential precipitation. (No dark field micrographs were presented, nor was there any discussion of how positive identification was made.) Such incomplete microstructural work has typified almost all fracture studies that have been made. In the present study it has been shown that unique identification of preferential precipitation can be made by simple adherence to good transmission electron microscopy practice of analysis by bright field, dark field, and selected area electron diffraction.

Banerjee³,⁴ and Reisdorf⁶³ attributed tempered martensite embrittlement to different phenomena both associated with the simultaneous
dissolution of epsilon carbide and the precipitation of Fe$_3$C. However, no epsilon carbide was detected for any of the specimens in this study. As discussed previously, since the specimens were all autotempered and containing Fe$_3$C initially, this observation is not unanticipated. This means, though, that the use of epsilon carbide dissolution to explain tempered martensite embrittlement is subject to doubt.

Mention should also be made of the toughness/microstructure comparisons made at similar strength levels. It was observed that the toughness was higher for structures containing no preferential lath boundary precipitation. Where all specimens compared had lath boundary precipitation, the toughness was highest for the specimen having the least amount of lath boundary precipitation. Thus, the specimen tempered at 300°C had less extensive preferential precipitation than the ones tempered at 500 and 650°C, and its toughness was higher.

The high toughness associated with the as-quenched and low tempering temperature specimens was attributed to several factors:

(1) The microstructure was uniformly martensitic, with no bainite or retained austenite present.

(2) The martensite was a dislocated lath type rather than an internally twinned plate type.

(3) The martensite was effectively tempered in the as-quenched state due to autotempering of Fe$_3$C.

(4) There was no extensive lath boundary precipitation present.

(5) The steel had a low impurity level.

(6) The prior austenite grain boundaries were free of transformation products other than martensite.
There were no residual carbides remaining because of insufficient austenitizing.

The last two factors listed above are critical, because they both depend on the austenitizing temperature. As was discussed earlier it has been found that a twofold increase in $K_{IC}$ occurred when the austenitizing temperature was changed from 1840°F to 2040°F. This increase in austenitizing temperature caused two microstructural changes: (1) the carbides dissolved to a large extent, and (2) the austenite grain size became much larger. It was not clear which effect was responsible for the increase in toughness. Large retained grain boundary carbides (several microns in size) could be detrimental to toughness, because during loading the individual carbides could fracture, thereby creating microcracks within the structure. The prior austenite grain size could be of importance in the manner in which it affects transformations. The large grain size has low energy boundaries which effectively retard the nucleation of proeutectoid transformation of ferrite or cementite on cooling from the austenitic state. There are many examples of embrittlement caused by grain boundary carbide films. A ferritic grain boundary film could have a similar effect.
IV. CONCLUSIONS

In this study of a quenched and tempered secondary hardening steel, the following conclusions have been made:

1. The structure of as-quenched fracture toughness specimens consisted primarily of Fe₃C autotempered, dislocated martensite laths.

2. Tempered structures contained precipitation of either Fe₃C or Mo₂C both within martensite laths and at their boundaries. It was found that foil tilting and scanning in dark field illumination (in the transmission electron microscope) was essential for observation of lath boundary precipitation.

3. The high fracture toughness values observed for specimens tempered ≤ 225°C was favored by a high austenitizing temperature.

4. The drops in toughness which occurred at about 300 and 600°C tempering were both attributed to preferential carbide precipitation at martensite lath boundaries.

5. When comparing toughnesses and microstructures at similar strength levels, the following conclusions were made: (a) A structure containing lath boundary precipitates was much inferior in toughness to a structure devoid of such precipitation. (b) The more extensive the lath boundary precipitation, the lower was the toughness.

6. A simple relationship between ductility and fracture toughness does not exist.

7. Calculated values of fracture toughness from tensile properties
using Hahn and Rosenfield's model showed very poor correlation to measured values. This model seems extremely insensitive to microstructural changes which in actuality cause significant changes in fracture toughness.

8. Fracture toughness is a complex material property. As such, more fundamental studies of the effect of microstructure, heat treatment, composition, etc. on fracture toughness must be undertaken. A thorough re-examination must be made of old ideas about the effect on fracture toughness of microstructure, heat treatment, and ductility.
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<td>0.010</td>
<td>&lt;0.005</td>
<td>&lt;0.002</td>
<td>&lt;0.002</td>
</tr>
<tr>
<td>D</td>
<td>713-7</td>
<td>0.28</td>
<td>5.03</td>
<td>0.60</td>
<td>0.005</td>
<td>&lt;0.02</td>
<td>0.008</td>
<td>&lt;0.005</td>
<td>&lt;0.002</td>
<td>&lt;0.002</td>
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</tbody>
</table>

Table I. Chemical composition of the alloys used (given in Wt.%)
Table II. Longitudinal mechanical properties of ingot A tested at room temperature.

<table>
<thead>
<tr>
<th>Tempering Temperature, °C</th>
<th>0.2% Yield Strength, ksi, $\sigma_y$</th>
<th>Ultimate Tensile Strength, ksi, $\sigma_u$</th>
<th>Elongation, %</th>
<th>Reduction of Area, %</th>
<th>True Fracture Strain, $\varepsilon_f$</th>
<th>Strain Hardening Exponent, n</th>
<th>Plane Strain Fracture Toughness, ksi(in.)$^{1/2}$</th>
<th>Critical Stress Intensity, ksi(in.)$^{1/2}$</th>
<th>Fracture Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>As-quenched</td>
<td>214</td>
<td>261</td>
<td>8.3</td>
<td>26</td>
<td>0.301</td>
<td>0.056</td>
<td>99</td>
<td>122</td>
<td>0.46</td>
</tr>
<tr>
<td>150</td>
<td>210</td>
<td>255</td>
<td>10.9</td>
<td>36</td>
<td>0.445</td>
<td>0.060</td>
<td>104*</td>
<td>135</td>
<td>0.50</td>
</tr>
<tr>
<td>225</td>
<td>196</td>
<td>234</td>
<td>11.0</td>
<td>40</td>
<td>0.522</td>
<td>0.055</td>
<td>109*</td>
<td>137</td>
<td>0.56</td>
</tr>
<tr>
<td>300</td>
<td>182</td>
<td>217</td>
<td>9.5</td>
<td>40</td>
<td>0.515</td>
<td>0.047</td>
<td>67</td>
<td>88</td>
<td>0.37</td>
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<tr>
<td>500</td>
<td>180</td>
<td>214</td>
<td>13.4</td>
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<td>0.580</td>
<td>0.072</td>
<td>51</td>
<td>82</td>
<td>0.28</td>
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<tr>
<td>600</td>
<td>251</td>
<td>259</td>
<td>1.0</td>
<td>5</td>
<td>0.049</td>
<td>---</td>
<td>36</td>
<td>38</td>
<td>0.14</td>
</tr>
<tr>
<td>650</td>
<td>192</td>
<td>203</td>
<td>5.4</td>
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<td>0.175</td>
<td>0.042</td>
<td>39</td>
<td>49</td>
<td>0.20</td>
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</table>

*These toughness values are apparent ones ($K_q$) and do not meet the plane strain conditions specified in ASTM publications.
Table III. Transverse mechanical properties of ingot B tested at room temperature.

<table>
<thead>
<tr>
<th>Tempering Temperature, °C</th>
<th>Tensile Properties</th>
<th>Fracture Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.2% Yield Strength, ksi, $\sigma_y$</td>
<td>Ultimate Tensile Strength, ksi, $\sigma_u$</td>
</tr>
<tr>
<td>As-quenched</td>
<td>226</td>
<td>268</td>
</tr>
<tr>
<td></td>
<td>150</td>
<td>220</td>
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<tr>
<td></td>
<td>192</td>
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<td>260</td>
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<tr>
<td></td>
<td>192</td>
<td>203</td>
</tr>
</tbody>
</table>

*These toughness values are apparent ones ($K_q$) and do not meet the plane strain conditions specified in ASTM publications.
Table IV. Longitudinal mechanical properties of ingot C tested at -78°C.

<table>
<thead>
<tr>
<th>Tempering Temperature, °C</th>
<th>Tensile Properties</th>
<th>Fracture Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.2% Yield Strength, ksi, σy</td>
<td>Ultimate Tensile Strength, ksi, σu</td>
</tr>
<tr>
<td>As-quenched</td>
<td>237</td>
<td>284</td>
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<tr>
<td>150</td>
<td>234</td>
<td>280</td>
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<td>225</td>
<td>214</td>
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<tr>
<td>300</td>
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<td>600</td>
<td>278</td>
<td>278</td>
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<tr>
<td>650</td>
<td>203</td>
<td>211</td>
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</table>
Table V. Charpy V-notch impact energy values of tempered specimens tested at room temperature (Ingot D).

<table>
<thead>
<tr>
<th>CVN Impact Energy Ft-lb</th>
<th>Tempering Temperature, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>As-quenched</td>
</tr>
<tr>
<td></td>
<td>11.8</td>
</tr>
</tbody>
</table>
Fig. 1 Round tensile specimen.
Fig. 2 Schematic of how fracture toughness and Charpy V-notch specimens were cut from as-rolled bars.
Fig. 3 Crackline loaded fracture toughness specimen.
Fig. 4 Stress intensity calibration curve derived from boundary collocation procedure of Srawley and Gross.
Fig. 5 Crack-opening-displacement/crack length calibration curve.
Fig. 6. Schematic of stress wave emission circuitry.
Fig. 7 Microhardness vs tempering temperature curve for 1 hr tempering of 0.3% C, 5% Mo steel.
Fig. 8 Longitudinal room temperature tensile properties as a function of tempering temperature.
Fig. 9 Transverse room temperature tensile properties as a function of tempering temperature.
Fig. 10 Longitudinal -78°C tensile properties as a function of tempering temperature.
Fig. 11 Longitudinal room temperature plane strain fracture toughness and strength properties as a function of tempering temperature.
Fig. 12 Ratio of plane strain fracture toughness to yield strength (longitudinal room temperature properties) as a function of tempering temperature.
Fig. 13 Transverse room temperature plane strain fracture toughness and strength properties as a function of tempering temperature.
Fig. 14  Ratio of plane strain fracture toughness to yield strength (transverse room temperature properties) as a function of tempering temperature.
Fig. 15 Longitudinal -78°C plane strain fracture toughness and strength properties as a function of tempering temperature.
Fig. 16 Ratio of plane strain fracture toughness to yield strength (longitudinal -78°C properties) as a function of tempering temperature.
Fig. 17. Load/crack-opening-displacement fracture toughness test record for transverse specimen tempered at 650°C and tested at room temperature.
Fig. 18. Load/crack-opening-displacement fracture toughness test record for transverse specimen tempered at 300°C and tested at room temperature.
Fig. 19  Load/crack-opening-displacement fracture toughness test record for transverse as-quenched specimen, tested at room temperature.
Fig. 20 Steigerwald's compilation of plane strain fracture toughness vs yield strength data for various steels.\textsuperscript{32}
Fig. 21 Room temperature Charpy V-notch impact energy values as a function of tempering temperature.
Fig. 22 Oscillograms of stress waves recorded at approximately 95% $P_{CRIT}$ during room temperature transverse fracture toughness tests of (a) an as-quenched specimen and (b) a 300°C tempered specimen. The time lines are 1/8 sec apart.
Fig. 23 Oscillograms of stress waves recorded at approximately 95% $P_{CRIT}$ during room temperature transverse fracture toughness tests of (a) a 500°C tempered specimen and (b) a 600°C tempered specimen. The time lines are 1/8 sec apart.
Fig. 24 Oscillogram of stress waves recorded at approximately 95% $P_{\text{CRIT}}$ during room temperature transverse fracture toughness test of a 650°C tempered specimen. The time lines are 1/8 sec apart.
Fig. 25 Martensitic structure of transverse as-quenched specimen, (a) and (b). Note the large prior austenitic grain size shown in (a).
Fig. 26 Martensitic structure of transverse as-quenched specimen.
Fig. 27 Tempered martensitic structure of 300°C tempered transverse specimen.
Fig. 28 Tempered martensitic structure, (a) and (b), of 600°C tempered transverse specimen.
Fig. 29  Tempered martensitic structure, (a) and (b), of 650°C tempered transverse specimen.
Fig. 30 Martensitic structure of longitudinal as-quenched specimen, (a) and (b). Note the prior austenitic grain size in (a).
Fig. 31 Temperd martensitic structure, (a) and (b), of longitudinal 600°C tempered specimen. Note the prior austenitic grain size in (a).
Fig. 32 Structure of as-quenched specimen, showing in bright field (a) autotempered, dislocated martensite laths. (b) Dark field reversal of contrast shows cementite precipitation.
Fig. 33 Structure of as-quenched specimen, showing bright field (a) autotempered, dislocated martensite laths. (b) Dark field reversal of contrast shows cementite precipitation.
Fig. 34 Martensite laths in 225°C tempered specimen.
Fig. 34 Martensitic structure of 225°C tempered specimen. Note the presence of plates and laths within the structure.
Fig. 36 Martensitic structure of 225°C tempered specimen, showing (a) precipitation of cementite within a lath, and (b) dark field reversal of contrast of cementite precipitates.
Fig. 37 Micrographs of 300°C tempered specimen, showing cementite precipitation both at martensite lath boundaries and within laths in bright field (a) and dark field (b) of cementite reflection.
Fig. 38 Micrographs of 500°C tempered specimen, showing extensive cementite precipitation at martensite lath boundaries in bright field (a). Dark field (b) shows reversal of cementite contrast.
Fig. 39 Micrographs of 500°C tempered specimen, showing extensive cementite precipitation at martensite lath boundaries in bright field (a). Dark field (b) shows reversal of cementite contrast.
Fig. 40 Dark field micrograph of 600°C tempered specimen, showing fine Mo₂C precipitates. Dark field was taken from Mo₂C streak in selected area diffraction.
Fig. 41 Selected area electron diffraction pattern of 600°C tempered specimen. Streaking due to Mo₂C precipitation occurs in (200) directions (one streak is marked by arrow). Foil orientation (100).
Fig. 42 Dark field micrograph of 650°C tempered specimen, showing lath boundary decoration by Mo$_2$C. Mo$_2$C diffraction spot used for reversal of contrast.
Fig. 43. Dark field micrograph of 650°C tempered specimen, showing precipitation of Mo$_2$C both within martensite laths and along their boundaries. Reversal of contrast from Mo$_2$C diffraction spot.
Fig. 44 Structure of 650°C tempered specimen. Mo₂C precipitates visible in bright field (a) and shown to be precipitated on boundaries by reversal of contrast in dark field (b).
Fig 45 Fracture surfaces of longitudinal specimens tested at room temperature. Shown from left to right, top row: (300, 225, 150°C tempered specimens, as-quenched specimen), bottom row: (650, 600, 500°C tempered specimens).
Fig. 46 Fracture surfaces of transverse specimens tested at room temperature. Shown from left to right, top row: (300, 225, 150°C tempered specimens, as-quenched specimen), bottom row: (650, 600, 500°C tempered specimens).
Fig. 47 Fracture surfaces of longitudinal specimens tested at -78°C. Shown from left to right, top row: (300, 225, 150°C tempered specimens, as-quenched specimen), bottom row: (650, 600, 500°C tempered specimens).
Fig. 48 Typical quench crack observed for all longitudinal specimens. Note the intergranular failure and the large prior austenitic grain size. Fractograph is of as-quenched longitudinal specimen tested at room temperature.
Fig. 49 Typical quench crack observed for most transverse specimens. Note the intergranular failure and the smaller prior austenitic grain size than observed for longitudinal specimens. Fractograph is of a transverse specimen tempered at 300°C and tested at room temperature.
Fig. 50 Fractographs, (a) and (b), showing mixture of dimpled rupture and quasi-cleavage in as-quenched specimen tested at room temperature.
Fig. 51 Fractographs, (a) and (b), showing primarily dimpled rupture in 150°C tempered longitudinal specimen tested at room temperature.
Fig. 52 High magnification fractograph of dimpled rupture in 150°C tempered specimen tested at room temperature.
Fig. 53 Fractograph showing mixture of dimpled rupture and quasi-cleavage in 225°C tempered specimen tested at room temperature.
Fig. 54 Fractographs of 300°C tempered longitudinal specimen tested at room temperature. (a) Mostly quasi-cleavage with one cleaved area. (b) Quasi-cleavage area at higher magnification.
Fig. 55 Fractograph showing quasi-cleavage in 500°C tempered longitudinal specimen tested at room temperature.
Fig. 56 Fractograph of 600°C tempered longitudinal specimen tested at room temperature, showing mixture of quasi-cleavage and cleavage.
Fig. 57 Fractographs showing (a) mixture of quasi-cleavage and cleavage and (b) higher magnification view of quasi-cleavage. Specimen was longitudinal, tempered at 650°C, and tested at room temperature.
Fig. 58 Fractographs, (a) and (b), showing mixture of dimpled rupture and quasi-cleavage in as-quenched transverse specimen tested at room temperature.
Fig. 59 Fractograph showing mixture of dimpled rupture and quasi-cleavage in 225°C tempered transverse specimen tested at room temperature.
Fig. 60 Fractographs of 300°C tempered transverse specimen tested at room temperature.
(a) Quasi-cleavage area with large tear ridge present. (b) High magnification fractograph of tear ridge of part (a).
Fig. 61 Fractograph of quasi-cleavage in 300°C tempered transverse specimen tested at room temperature.
Fig. 62 Fractograph of 600°C tempered transverse specimen tested at room temperature, showing mixture of quasi-cleavage and cleavage.
Fig. 63 Fractograph showing quasi-cleavage in 650°C tempered transverse specimen tested at room temperature.
Fig. 64 Fractographs, (a) and (b), showing quasi-cleavage in as-quenched longitudinal specimen tested at -78°C.
Fig. 65 Fractograph showing quasi-cleavage in 150°C tempered longitudinal specimen tested at -78°C.
Fig. 66 Fractograph showing quasi-cleavage in 225°C tempered longitudinal specimen tested at -78°C.
Fig. 67 Fractograph showing mixture of quasi-cleavage and cleavage in 300°C tempered longitudinal specimen tested at -78°C.
Fig. 68 Fractograph showing quasi-cleavage in 500°C tempered longitudinal specimen tested at -78°C. Note the large tear ridge present.
Fig. 69 Fractographs, (a) and (b), showing quasi-cleavage in 600°C tempered longitudinal specimen tested at -78°C.
Fig. 70 Fractographs of 650°C tempered longitudinal specimen tested at -78°C. (a) Mostly quasi-cleavage with one area of cleavage. (b) Quasi-cleavage.
Fig. 71. Comparison of measured and calculated values of $K_{IC}$. Calculated values were determined using tensile properties of this study and Hahn and Rosenfield's model.\(^4\)}
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