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Benjamin Francis
(Ph. D. thesis)

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THE FRACTURE TOUGHNESS OF HIGH STRENGTH Fe-Ni-Ti ALLOYS

Benjamin Francis

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

ABSTRACT

The fracture toughness of a series of Fe-Ni-Ti alloys was investigated. The yield strengths of these alloys were in the range of 180-200 ksi. The alloys investigated were Fe-12Ni-1Ti, 12Ni-2Ti, 16Ni-1Ti, 16Ni-1.5Ti, and 20Ni-1.5Ti. For the 16Ni alloys, which had the best properties it was found that excellent fracture toughness \( K_{IC} > 100 \text{ ksi}\sqrt{\text{in}} \) could be obtained if the age hardening temperature (550-600°C) was higher than that normally used for maraging steels (450-480°C). If aging temperatures in the range 450-500°C were used severe grain boundary precipitation occurred and the alloys were brittle. Some evidence is presented which suggests that at the higher aging temperatures austenite forms very rapidly at the grain boundaries and greatly reduces the amount of boundary precipitate, thereby increasing the fracture toughness.
I. INTRODUCTION

It is well known that high strength and high fracture toughness (as measured by $K_{IC}/\sigma_y$ ratio) rarely occur together. This is particularly true of ferritic alloys. The reason for this is of course, that at high yield strength levels the tensile stresses at the tip of a sharp crack become very high. At some point these tensile stresses become sufficiently high that fracture occurs by cleavage rather than by ductile tearing. Thus, high fracture toughness in high strength ferritic alloys may be obtained only by eliminating cleavage initiation sites which can be "activated" at the crack tip stress levels encountered at or near general yield and to thus ensure that fracture occurs by a ductile rather than a brittle mode. At the same time one must of course ensure that the ductile fracture mode is itself conducive to high fracture toughness.

The $18Ni200$ ($18Ni$, $8Co$, $3Mo$, $0.2Ti$, bal Fe) maraging steel is an example of an alloy in which these failure modes have very successfully been controlled. This is clearly demonstrated by the fact that the NDT for this alloy is below $-190^\circ C$ at a yield strength level of $\sim 200$ ksi and that a $K_{IC} > 100$ ksi$/\sqrt{in}$ is routinely obtained. Other maraging steel systems have been markedly less successful. Fe-Ni-Ti is one such system. It has been shown that maraging steels, containing for example $\sim18\%Ni$ and $0.1\%$ to $2\%Ti$, in this alloy system exhibit low toughness because of grain boundary precipitation.

If it was possible to eliminate this grain boundary precipitation by a simple heat treatment and/or a variation in the chemical composition
of the alloy (i.e. varying the Ni and Ti contents) this alloy would be quite attractive. In the first place it would have an economic advantage over the standard maraging steel because of the elimination of Co and Mo. Secondly, and perhaps more importantly, it would provide a simple system from which it might be possible to learn more about the factors controlling fracture toughness in maraging type steels, particularly those factors which influence the critical tensile stress for unstable cleavage failure.1,2,7

There was, in addition, some good evidence that it would be possible to greatly reduce the grain boundary precipitation in the Fe-Ni-Ti system by the proper choice of heat treatment and composition. The evidence for this was that the precipitate formed in this system ($\eta$ Ni$_3$Ti) did not form in austenite, but only in ferrite.8 Also, it was known that austenite forms in all the maraging steels in times comparable to the aging times. It therefore seemed that a judicious choice of composition and heat treatment might produce a sufficient amount of austenite at grain boundaries to inhibit or prevent the formation of grain boundary precipitates (since precipitation does not take place in austenite), and thereby greatly improve the toughness of these alloys. The purpose of this investigation was then to determine whether or not the Fe-Ni-Ti maraging type steels could in fact, when properly processed, attain good fracture toughness at high strength levels.
The properties of 5 alloys were investigated: Fe-12Ni-1Ti, Fe-12Ni-2Ti, Fe-16Ni-1Ti, Fe-16Ni-1.5Ti, Fe-20Ni-1.5Ti. These alloys were chosen to determine the effect of variations in both the Ni and the Ti contents and to give yield strengths in the range 180-200 ksi, the range of interest for this investigation. Maraging of these alloys was carried out at temperatures ranging from 450°C to 600°C in order to determine the effects of aging temperature on the toughness properties.
II. EXPERIMENTAL PROCEDURE

A. Materials Preparation

1. Alloy Preparation

Heats of approximately 20 lbs were melted in a vacuum induction furnace. The raw materials were electrolytic iron (99.9%), high purity nickel (99.9%), and rod titanium (99.9%). The ingots were cast in heavy copper molds and then homogenized in vacuum for 24 hrs at 1175°C. Chemical compositions of the alloys are listed in Table 1. After homogenizing the ingots were upset forged and cross forged at 1100°C into 1 1/4 in. thick plates. Figure 1a illustrates the forging process. The purpose of this forging process was to minimize the texture produced during forging.

The preparation of these alloys may have influenced their subsequent fracture toughness in three major ways: (1) through the introduction of inclusions, (2) through inhomogeneities, particularly segregation, and (3) through the introduction of impurities such as C, N, and O. No attempt was made in this study to determine the effect of any of these three variables on the final toughness levels obtained. However, their possible importance was recognized at the outset, and so the alloy preparation techniques were designed either to minimize their influence or to maintain their influence at a constant level. Thus, rod titanium was used instead of sponge titanium because it has been found to reduce the inclusion count in similar alloys. (For these same alloys the mean center-to-center spacing for the inclusion particles visible in the optical microscope was about 50 μm.) Likewise,
Table 1. Compositions of Alloys

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Fe</th>
<th>Ni</th>
<th>Ti</th>
<th>Al</th>
<th>P</th>
<th>S</th>
<th>N</th>
<th>O</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-12Ni-1Ti</td>
<td>bal</td>
<td>12</td>
<td>1</td>
<td>0.1</td>
<td>N.D.</td>
<td>N.D.</td>
<td>N.D.</td>
<td>N.D.</td>
<td>N.D.</td>
</tr>
<tr>
<td>Fe-12Ni-2Ti</td>
<td>&quot;</td>
<td>12</td>
<td>2</td>
<td>0.1</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
</tr>
<tr>
<td>Fe-16Ni-1Ti</td>
<td>&quot;</td>
<td>16</td>
<td>1</td>
<td>0.1</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
</tr>
<tr>
<td>Fe-16Ni-1Ti</td>
<td>&quot;</td>
<td>(15.96)</td>
<td>(1.07)</td>
<td>(0.07)</td>
<td>(0.002)</td>
<td>(0.005)</td>
<td>(0.003)</td>
<td>(0.003)</td>
<td>(&lt; 0.001)</td>
</tr>
<tr>
<td>Fe-16Ni-1.5Ti</td>
<td>&quot;</td>
<td>16</td>
<td>1.5</td>
<td>0.1</td>
<td>N.D.</td>
<td>N.D.</td>
<td>N.D.</td>
<td>N.D.</td>
<td>N.D.</td>
</tr>
<tr>
<td>Fe-20Ni-1.5Ti</td>
<td>&quot;</td>
<td>20</td>
<td>1.5</td>
<td>0.1</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
<td>&quot;</td>
</tr>
</tbody>
</table>

*These compositions are wt%. The nominal compositions are indicated by open figures; actual compositions are enclosed in parentheses.

+ N.D. = not determined
Fig. 1. (a) Illustration of the forging process.  
(b) Orientation of specimens with respect to the forged bar.
the effectiveness of the homogenization procedure was examined both by direct measurement using the microprobe and by calculation using a diffusion model. The details of this examination are reported elsewhere. The results showed that the homogenization procedure did effectively reduce, but did not eliminate completely, the segregation of Ni and Ti in the ingots used for this study. The effect of the impurities C, N and O on the subsequent fracture toughness was minimized by the presence of Ti, which in addition to being present in the alloy for the purpose of precipitation hardening acted as a getter for these elements, and by the addition of 0.1%Al to help scavenge O₂.

2. Heat Treatment and Specimen Preparation

After forging specimen blanks were cut from the forged stock and heat treated. At this stage all specimen blanks whether for charpy, fracture toughness or tensile tests were approximately the same size. This precaution minimized the possibility of any unanticipated variations in heat treatment due to section size, although it is not likely that the structure of these alloys would be very sensitive to section size since martensite is the only transformation product for normal quenching rates and since the Ms's are sufficiently low that only a slight amount of precipitation might occur upon slow cooling below the Ms. (It should be remembered that these alloys are essentially carbon-free.) Agitated brine quenches were employed for all alloys and treatments including aging.

The heat treating cycle consisted always of a 900°C anneal for 2 hours (hereafter to be written, for example, as 900°C(2hr.)) at temperature to remove the forged structure, usually followed by grain refining treatments.
The standard grain refining treatment was a low temperature anneal at 750°C for 2 hours at temperature. However, in a few instances other grain refining treatments were employed to further reduce the grain size. The rational behind these treatments is discussed by Jin. In these treatments samples were cycled between room temperature and the γ phase field and room temperature and the three phase field α + γ + Ni₃Ti. For example, the alloy Fe-16Ni-1Ti was cycled as follows: 900°C(2h)BQ + 750°C(2h)BQ + 600°C(2h)BQ + 750°C(2h)BQ + 600°C(2h)BQ + 750°C(2h)BQ. For the alloy Fe-16Ni-1.5Ti this cycling was done between 800°C and 600°C because at 750°C for the upper cycling temperature optical examination of the structure indicated the specimen had been in the three phase field. This now seems to have been a misinterpretation of the structure. The treatment used, however, gave a grain size comparable to the treatments used for the other alloys, and hence was deemed satisfactory. These treatments yielded average grain boundary linear intercept distances of 5-10 μm for all alloys.

Following heat treatment, specimens were rough machined from the specimen blanks, age hardened, and then finish machined. The purpose of doing the final machining after age hardening was to eliminate the introduction of residual stresses around notches, etc., during the quench from the aging temperature. In the case of the compact tension specimens final machining encompassed grinding, drilling of pin holes, and cutting of the notch; for charpy bars it encompassed grinding and notching; for tensile specimens it encompassed cutting of the
gauge section and threading. It should be noted in the case of the charpy bars that close attention was paid to machining of the notch in order to minimize variations in its geometry. It should also be noted that machining removed a minimum of 1/8 in of material from the outside edges of all specimen blanks, so that surface defects were removed.

The orientation of specimens in relation to the forged ingot is shown in Fig. 1b.

B. Mechanical Testing

1. Aging Curves

The aging response of each alloy and heat treatment was measured on approximately 1/4 in. sq. blanks which had previously been heat treated in large pieces of the same size as the specimen blanks. Each aging curve was obtained from one 1/4 sq. blank, as this method gave identical results to using one blank for each point on the aging curve. In all cases salt was used for the aging temperature quenching medium to minimize heat-up time. The specimens were quenched in agitated brine at the end of each aging period. All hardness measurements in this study were on the Rockwell "C" scale. Each point on an aging curve is the average of at least three measurements. The same machine was used for all measurements and values were periodically checked with a calibration block.
2. **Charpy Tests**

Charpy tests were performed according to ASTM E-23-72 specifications. Specimens were tested on a 223 ft-lbs capacity machine. The specimen dimensions are shown in Fig. 2a.

Test temperatures other than room temperature were obtained with baths of either used silicone diffusion pump oil (for higher temperatures), methyl alcohol cooled by liquid nitrogen (for lower temperatures), or liquid nitrogen.

3. **Tensile Tests**

ASTM standard round tensile specimens with a 1 in. gage length and .250 in. gage section were used for all tensile testing. Figure 2b gives the complete dimensions of these specimens.

All tests were performed on a 300 kip MTS Universal Tensile Testing machine at a crosshead speed of .05 cm/min (.02 in/min) which gives a strain rate of $3 \times 10^{-4}$/sec for a 1 in. gage length.

Elongation to fracture was obtained by measuring the length change of a 1 in. line scribed on the specimen prior to testing. Yield strength and uniform elongation were measured from the load-ram displacement curve. Generally, a small correction of less than 5% was uniformly applied to the displacements measured from the curve in order to bring the elongation to fracture into agreement with that measured on the specimen.

4. **Fracture Toughness Tests**

All fracture toughness tests were performed according to ASTM specification E 399-72. The specimens were of the compact tension
Fig. 2. Dimensions at (a) Charpy V-notch specimens and (b) round tensile specimens.
type and were 1 in. thick (IT) for all alloys and conditions. The specimen dimensions are shown in Fig. 3.

The specimens were fatigued and fractured on a MTS 300 Kip Universal Tensile Test machine. The ram speed was 0.00125 cm/sec (.0005 in/sec) which gave a stress intensity loading rate generally in the middle of the ASTM specified range. The COD was measured with a clip gage per the above ASTM specification.

C. Measurement of Phase Transformation Temperatures and Characteristics

1. Dilatometry and Magnetometric Technique

The $M_s$, $M_f$, $A_s$, and $A_f$ of the alloys studied were measured by dilatometry at a heating and cooling rate of $20^\circ$C/min. The $M_s$ and $M_f$ were also measured using a magnetometric technique. The details of this technique may be found in the works of Babu and Yuen. Results of the two techniques agreed closely.

The magnetometric technique was also used to determine the isothermal rate of formation of austenite in the Fe-16Ni-1Ti and Fe-16Ni-1.5Ti alloys. It was not possible to check these results explicitly with a more conventional technique: a high temperature x-ray camera was not available, and attempts to observe the formation of austenite using hot stage TEM were foiled by surface precipitation. However, isothermal holding of a specimen of Fe-16Ni-1Ti in the dilatometer and observation of the subsequent length change with time did show qualitative agreement with the magnetometric technique. This fact, and the above mentioned agreement in the measurement of $M_s$ and
Fig. 3. Dimensions of compact tension specimens.
Mf suggest strongly that the magnetometric technique was accurate. In addition, Jin\textsuperscript{14} has used the dilatometer to quantitatively determine the rate of formation of austenite in a Fe-12Ni-.25Ti alloy, and his results also support the accuracy of the magnetic technique.

2. X-ray

A Picker biplane diffractometer was used for all retained austenite measurements. The X-ray source was a Cu tube operated at 40 KV and 14 ma. A LiF monochromator was used between the sample and detector to filter out the fluorescent radiation.

The integrated intensities of the X-ray peaks of interest were measured either by weighing traced cut-outs of the peaks or by using a planimeter. For some of the smaller peaks the integrated intensity was determined by standard counting techniques. The normal scan rate was 2°/min. However, for very small peaks corresponding to \( \leq 1\% \) austenite scans were made at 1/4°/min.

The procedure for determining the percent retained austenite from the integrated intensities was that followed by Cullity,\textsuperscript{17} the direct comparison method. The necessary calculations are shown in Appendix 2. The 311\( \gamma \) and 211\( \alpha \) peaks were used for these calculations. It is well known that texture can cause considerable error in the determination of retained austenite content, because texture influences the relative intensities of the diffraction peaks. The texture of a Fe-20Ni-1.5Ti sample, taken from a broken charpy bar, was examined by Back-Reflection Laue and by diffractometer, using a suitable goniometer. Although no attempt was made to plot a pole-figure sufficient information was obtained to determine that the texture was not severe enough to warrant any special attempts to counteract its influence on the retained austenite measurements.
A Norelco 114.59 mm diameter Debye-Sherrer powder camera was used to obtain precipitate powder patterns. The X-ray source was an Fe tube with a Mn filter. The precipitates were extracted from samples using 5% nital.

D. Microscopy

1. Optical Microscopy

Specimens for optical microscopy were prepared by standard mechanical polishing techniques followed by electropolishing with a solution of 75 gms CrO$_3$ + 400 ml Acetic acid + 25 ml H$_2$O. The specimens were then chemically etched in two steps: first with a 1:1 mixture of Kallings reagent and 5% nital and then with a 5% nital etch. The two step etching procedure was necessary for the later extraction of the precipitates.

2. Transmission Electron Microscopy (TEM)

Foils for TEM were taken from previously broken specimens. The foils were prepared by first abrasive cutting a 0.015-0.020 in. thick slice from the specimens, then chemically thinning this slice in an HF + H$_2$O$_2$ mixture to ~ 0.002 in., and finally electropolishing in a jetpolisher with a solution of 100 gms Na$_2$CrO$_4$ + 400 ml H$_2$O. The polishing voltage varied from 30 to 50 volts.

Carbon replicas were of the single stage type. They were taken from the same surfaces that were used for optical photomicrography, without any additional preparation.

Carbon replicas and foils were examined on either a Siemens Elmskop 1A or a Phillips EM 301 microscope operated at 100 kv.
3. Scanning Electron Microscopy

The fracture surfaces of selected compact tension and charpy specimens were examined with a JEOLCO JSM-U3 SEM in the secondary emission mode and with an accelerating voltage of 25 KV.
II. RESULTS AND DISCUSSION

A. Phase Transformation Structures and Characteristics

All of the alloys used in this study transformed to lath martensite when cooled from the austenite region to room temperature. This is in complete agreement with the data in the literature, where martensite is found to be the only transformation product for normal quenching rates (e.g. AQ). A typical example of this structure is shown in Fig. 4.

The $M_s$, $M_f$, $A_s$ and $A_f$ for these alloys are shown in Fig. 5, along with data for alloys containing 0.5 wt% Ti. The $M_f$, $A_s$, and $A_f$ are increased by the addition of Ti but the $M_s$ is slightly decreased. (The $M_s$ is probably decreased because of solid solution hardening of the austenite by titanium). There was no observed variation in substructure of the as quenched alloys with changes in the titanium content.

It should be noted that Fig. 5 is not thermodynamically correct since there should be a two phase field separating the γ phase field from the α+γ+γTT phase field. However, it appears that this two phase field occurs over a very small temperature range and hence for all practical purposes can be ignored. No evidence of such a field was ever encountered in this study.
Fig. 4. Typical as quenched microstructure of lath martensite.
Fig. 5. The $M_s$, $M_f$, $A_s$ and $A_f$ temperatures for the alloys used in this investigation and for alloys containing 0.5 wt% Ti. 12,13
B. Precipitation Hardening and Tensile Properties

There have been many investigations\textsuperscript{18} of precipitation hardening in systems related to Fe-Ni-Ti because of the interest in maraging steels. Precipitation in these alloy systems generally produces very high yield strengths. The 300 grade maraging steels (\( \sim 300 \) ksi yield strength) are a prime example. It is of some interest, even though the higher yield strengths were not evaluated in this investigation, to determine the maximum yield strengths that may be obtained in the alloys of the present study. This may very simply be estimated by using the theoretical relationship between the \( R_c \) hardness and the Brinell hardness,\textsuperscript{19}

\[
R_c = A - \frac{D}{\sqrt{B}} 
\]  
where \( A \) and \( D \) are constants and \( B \) is the Brinell hardness. For a material which exhibits very low strain hardening, as the Fe-Ni-Ti alloys do,

\[
B \propto \sigma_y 
\]  
where \( \sigma_y \) is the uniaxial tensile yield strength.\textsuperscript{19} Substituting (2) into (1) and evaluating the constants for measured values of \( \sigma_y \) and \( R_c \) gives, after some rearranging,

\[
\sigma_y = \left[ \frac{790}{99-R_c} \right]^2 \text{ (ksi)} .
\]
Thus, since the maximum hardness for the present alloys is \( R_c \approx 49 \), the maximum attainable yield strengths are \( \approx 250 \) ksi. A graph of (3) is shown in Fig. 6.

The aging response of the various alloys are shown in Figs. 7 through 11. There are several generalizations that should be made from these curves. First, the effectiveness of the precipitation hardening is increased, at a given Ti content, by higher Ni contents up to about 16\%Ni. Second, as can be seen by comparing Figs. 9 and 10 with Figs. 25 and 26 respectively, the onset of softening in the aging curves is simultaneous with the onset of austenite formation. This has also been observed in maraging steels. The rapid softening in these alloys apparently occurs because of this austenite formation rather than because of precipitate overaging. The formation of austenite during the aging process is discussed in another section.

It should be noted that Thomas et.al. studied precipitation in a Fe-20Ni-23Co-0.17Ti alloy. Their work suggested that \( \text{Ni}_3\text{Ti} \) precipitated in austenite—so that it was necessary for austenite to form prior to precipitation of \( \text{Ni}_3\text{Ti} \). However, this is clearly not the case for the present alloys. The Fe-16Ni-1.5Ti alloy, for example, has a hardness of 42 \( R_c \) after aging for 1 hr. at 450\(^\circ\)C yet no austenite has been detected after aging for almost 3 hrs. at the same temperature. Thus the kinetics of austenite formation and of precipitation of \( \text{Ni}_3\text{Ti} \) are clearly very different for the present alloys than for the Fe-20Ni-23Co-.17Ti alloy studied by Thomas, et al.

The tensile properties of the alloys are shown in Table 2. There are two prominent tensile characteristics shown by these alloys:
Fig. 6. The approximate relationship between yield strength and $R_c$ hardness, according to Eq. 3.
Fig. 7. Aging curves for the 12Ni-1Ti alloy.
Fig. 8. Aging curves for the 12Ni-2Ti alloy.
Fig. 9. Aging curves for 16Ni-1Ti alloy.
Fig. 10. Aging curves for 16Ni-1.5Ti alloy.
Fig. 11. Aging curves for 20Ni-1.5Ti alloy.
<table>
<thead>
<tr>
<th>Alloy</th>
<th>Rockwell C Hardness and Heat Treatment</th>
<th>Temperature</th>
<th>0.2% offset Yield Strength (ksi)</th>
<th>Ultimate tensile strength (ksi)</th>
<th>Fracture Elongation (pct)</th>
<th>Uniform Elongation (pct)</th>
<th>Reduction in Area (pct)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-12Ni-1Ti</td>
<td>900°C(2h)+500°C Age R41</td>
<td>23°C</td>
<td>174</td>
<td>182</td>
<td>11</td>
<td>1.2</td>
<td>40</td>
</tr>
<tr>
<td></td>
<td>900°C(2h)+750°C(2h) +600°C(1h)+750°C(2h) +500°C Age R38</td>
<td>23°C</td>
<td>182</td>
<td>186</td>
<td>14</td>
<td>0.8</td>
<td>56</td>
</tr>
<tr>
<td></td>
<td>900°C(2h)+750°C(2h) +550°C Age R35</td>
<td>23°C</td>
<td>155</td>
<td>165</td>
<td>21</td>
<td>-</td>
<td>70</td>
</tr>
<tr>
<td>Fe-12Ni-2Ti</td>
<td>900°C(2h)+750°C(2h) +550°C Age R39</td>
<td>23°C</td>
<td>165</td>
<td>190</td>
<td>16</td>
<td>-</td>
<td>47</td>
</tr>
<tr>
<td>Fe-16Ni-1Ti</td>
<td>900°C(2h)+750°C(2h) +550°C Age R41</td>
<td>23°C</td>
<td>178</td>
<td>183</td>
<td>15</td>
<td>.9</td>
<td>69</td>
</tr>
<tr>
<td></td>
<td>underaged Age R41</td>
<td>-196°C</td>
<td>241</td>
<td>247</td>
<td>12</td>
<td>1.2</td>
<td>40</td>
</tr>
<tr>
<td></td>
<td>900°C(2h)+750°C(2h) +500°C Age R43</td>
<td>23°C</td>
<td>189</td>
<td>191</td>
<td>15</td>
<td>.8</td>
<td>69</td>
</tr>
</tbody>
</table>
Table 2. Tensile Properties of Fe-Ni-Ti Alloys (Cont.)

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Rockwell C Hardness and Heat Treatment</th>
<th>Temperature</th>
<th>0.2% offset Yield Strength ksi</th>
<th>Ultimate tensile strength</th>
<th>Fracture Elongation pct</th>
<th>Uniform Elongation pct</th>
<th>Reduction in Area pct</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-16Ni-1.5Ti</td>
<td>900°C(2h)+800°C(2h) +600°C(2h)+800°C(2h) +600°C age</td>
<td>-196°C</td>
<td>248</td>
<td>255</td>
<td>10</td>
<td>.9</td>
<td>.40</td>
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<td></td>
<td></td>
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<td>199</td>
<td>204</td>
<td>14</td>
<td>1</td>
<td>65</td>
</tr>
<tr>
<td></td>
<td>900°C(2h)+800°C(2h) +600°C(2h)+800°C(2h) +600°C age</td>
<td>-196°C</td>
<td>236</td>
<td>246</td>
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<td></td>
<td></td>
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<td>185</td>
<td>195</td>
<td>16</td>
<td>4</td>
<td>65</td>
</tr>
<tr>
<td>Fe-20Ni-1.5Ti</td>
<td>900°C(2h)+750°C(2h) +550°C age average</td>
<td>-196°C</td>
<td>196</td>
<td>257</td>
<td>20</td>
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<tr>
<td></td>
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<td>196</td>
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<td>7</td>
<td>60</td>
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<tr>
<td></td>
<td></td>
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<td>196</td>
<td>202</td>
<td>14</td>
<td>.9</td>
<td>55</td>
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<tr>
<td></td>
<td></td>
<td>23°C</td>
<td>184</td>
<td>195</td>
<td>17</td>
<td>5</td>
<td>59</td>
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</tbody>
</table>
(1) work hardening is very low so that the yield strength and the ultimate strength are nearly equal. (2) The reduction in area is quite high, even at \(-196^\circ C\). Some typical load elongation curves are shown in Fig. 12. The Fe-20Ni-1.5Ti alloy is an exception in that it shows considerable work hardening in the overaged condition. This occurs because there is considerable retained austenite in this alloy (15-20 vol%).

C. Precipitate Identification

The main precipitate in this alloy system, identified by X-ray and electron diffraction, is \(\eta\) (hexagonal) \(\text{Ni}_3\text{Ti}\). Both the Debye Scherrer X-ray pattern and the electron diffraction pattern are shown in Fig. 13, and an analysis of the Debye Scherrer lines appears in Table 3. Two Fe-12Ni-1Ti specimens were analyzed by X-ray diffraction; one was aged for 24 hrs at 650°C and the other was aged for 29 days at 630°C. The specimens produced identical powder patterns. This is significant because it demonstrates that the \(\eta\) phase is very stable, even though the ternary phase diagram\(^{12}\) indicates \(\text{Fe}_2\text{Ti}\) to be the equilibrium phase. A metastable ternary diagram, with \(\eta\) as the intermetallic phase, would clearly be more appropriate. The identification of the \(\eta\) phase as the precipitate agrees with many other investigators\(^{22-24}\) who have studied maraging steels.

Among the X-ray diffraction lines for the \(\eta\) precipitate are some which have not been identified. These lines are indicated in Table 3. The possibility that these lines are from an oxide or carbide has been ruled out by examination of the d-spacings and intensities
Fig. 12. Load elongation curves for the 20Ni-1.5Ti alloy in various conditions. The curve for the underaged condition is typical of all the other alloys in both the underaged and overaged conditions, and clearly illustrates the low work hardening characteristic of these alloys.
Fig. 13. (a) Diffraction pattern from the $\text{L}_2\text{Ni-1Ti}$ alloy in the (011) orientation.

(b) Debye Scherer x-ray pattern of precipitate using Fe K$_\alpha$ radiation with a Mn filter.
Fig. 13. (c) Analysis of electron diffraction pattern in (a),
assuming precipitate is Ni$_3$Ti (hexagonal) and
$a_o = 5.06\text{Å}, c_o = 8.28\text{Å}$. 

XBL 753-5974
Table 3. X-Ray Diffraction Data on the Precipitate in Fe-Ni-Ti Alloys

<table>
<thead>
<tr>
<th>X-Ray Data from Present Investigation</th>
<th>X-Ray Data from ASTM Powder Diffraction File</th>
</tr>
</thead>
<tbody>
<tr>
<td>d, Å</td>
<td>Ni$_3$Ti</td>
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<tr>
<td>I/I$_1$</td>
<td>d, Å</td>
</tr>
<tr>
<td></td>
<td>I/I$_1$</td>
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<tr>
<td></td>
<td>Fe$_2$Ti</td>
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<tr>
<td></td>
<td>d, Å</td>
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<tr>
<td></td>
<td>I/I$_1$</td>
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<tr>
<td>4.132*</td>
<td>4.39</td>
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<td>3.90</td>
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<td>3.728*</td>
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<td>2.536</td>
<td>2.56</td>
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<td>2.359*</td>
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<tr>
<td>2.297*</td>
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<td>1.951</td>
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<td>1.729</td>
<td>1.760</td>
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<td>1.620</td>
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<td>1.515</td>
<td>1.382</td>
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<tr>
<td>1.409*</td>
<td>1.341</td>
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<tr>
<td>1.295*</td>
<td>1.302</td>
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<td>1.277</td>
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<tr>
<td>1.028</td>
<td>1.027</td>
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</table>

*These x-ray lines have not been identified.
of the various possible oxides. They also are not from Fe₄Ti. There is the additional possibility that "mistakes" in crystal packing, such as occasional substitution of Fe for Ni, would "relax" the requirements of hexagonal symmetry and otherwise forbidden lines (for which the structure factor = 0) would appear. This possibility has also been checked and did not account for the extra lines. The origin of these lines is thus unknown.

D. Fracture Toughness

Fracture toughness is well known to be a strong function of microstructural features. In carbon steels, for example, grain boundary carbides have a major effect on $K_{IC}$. It is also well known that chemistry can influence toughness. For example, Ni has a powerful (but not completely understood) effect on the toughness-temperature transition of $\alpha$ iron. As pointed out in the introduction, the present investigation was designed to investigate the effect of both chemical and microstructural variations on the fracture toughness of Fe-Ni-Ti alloys. The chemical variations centered primarily around altering the Ni content of the alloy and consequently the final Ni content of the matrix (after precipitation). The microstructural variations were accomplished by using different aging temperatures and times (i.e. underaging and overaging) to alter the precipitation kinetics and thus to exert some control over preferential precipitation on grain and lath boundaries. Both these factors will be discussed in depth in later sections, after the presentation of the fracture toughness testing results.
Fracture toughness was measured using charpy V-notch (CVN) and compact tension specimens (CTS). These results will be discussed for each alloy separately.

12Ni-1Ti and 12Ni-2Ti

The charpy V-notch properties of the Fe-12Ni-1Ti alloy are given in Fig. 14, for a hardness of $R_c$ 36, and in Fig. 15, for $R_c$ 40. These hardness levels correspond to $\sigma_y$'s of $\sim$ 155 ksi and $\sim$ 180 ksi respectively. In terms of the transition temperature, $T_N$, the alloy does not have good toughness at either strength level. However, it does demonstrate an important feature; namely, that the toughness is a strong function of aging temperature. This is also true of the 16Ni alloys and to a lesser extent the 20 Ni alloys. Thus the higher aging temperature (550°C) gives both a lower $T_N$ and a higher upper shelf energy. As shall be discussed in a later section, the reason for this is that at the higher aging temperatures there is less boundary precipitation.

The results of CTS tests on the 12Ni-1Ti alloy are given in Table 4 and also indicate low toughness.

Charpy results for the 12Ni-2Ti alloy are given in Fig. 16 for a hardness level of $R_c$ 43 ($\sigma_y$ $\sim$ 200 ksi). Here $T_N$ is above room temperature and very low toughness is indicated. The fact that the toughness of this alloy is not affected by the aging temperature is probably due to the higher supersaturation of Ti, which promotes very rapid precipitation (see Fig. 8) and large amounts of boundary precipitate irrespective of the aging temperature. The extensive
Table 4. Fracture Toughness Test Data on Fe-Ni-Ti Alloys

<table>
<thead>
<tr>
<th>Alloy &amp; Test Temperature</th>
<th>Treatment</th>
<th>( K_Q ) ksi-in( \frac{1}{2} )</th>
<th>( K_{IC} ) ksi-in( \frac{1}{2} )</th>
<th>Hardness</th>
<th>( P_{Q'} ) 1000 lb</th>
<th>( P_{max} ) 1000 lb</th>
<th>Charpy Energy Ft lb</th>
<th>( K_{IC} ) ksi-in( \frac{1}{2} ) **</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-12Ni-1Ti 23°C</td>
<td>900°C(2h) +500°C Age UA</td>
<td>44</td>
<td>40</td>
<td>7.2</td>
<td>8.3</td>
<td>7</td>
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<tr>
<td></td>
<td>23°C</td>
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<td>44</td>
<td>6.7</td>
<td>6.9</td>
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<tr>
<td>Fe-16Ni-1Ti 23°C</td>
<td>900°C(2h) +750°C(2h) +550°C Age UA</td>
<td>149</td>
<td>40</td>
<td>22.5</td>
<td>27.7</td>
<td>65</td>
<td>222/247</td>
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<td>40</td>
<td>24.7</td>
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<td>18.1</td>
<td>18.1</td>
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<tr>
<td>Fe-16Ni-1.5Ti 23°C</td>
<td>900°C(2h) +800°C(2h) +600°C(2h) +800°C(2h) +550°C Age UA</td>
<td>50</td>
<td>40-45</td>
<td>8.6</td>
<td>8.6</td>
<td>14</td>
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</tbody>
</table>
Table 4. Fracture Toughness Test Data on Fe-Ni-Ti Alloys

<table>
<thead>
<tr>
<th>Alloy &amp; Test Temperature</th>
<th>Treatment</th>
<th>( K_Q ) ksi-in ( \frac{1}{2} )</th>
<th>( K_{IC} ) ksi-in ( \frac{1}{2} )</th>
<th>Hardness ( \text{E}_c )</th>
<th>( P_Q ) 1000 lb</th>
<th>( P_{max} ) 1000 lb</th>
<th>Charpy Energy Ft lb</th>
<th>( K_{ICL} ) ksi-in ( \frac{1}{2} )</th>
<th>( K_{ICJ} )</th>
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<td>43</td>
<td>16.1</td>
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<tr>
<td>-20°C</td>
<td>OA</td>
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<td>17.4</td>
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<tr>
<td>Fe-20Ni-1.5Ti</td>
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<tr>
<td>23°C</td>
<td>OA</td>
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<td>16.6</td>
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<td>22</td>
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<td>+500°C(2h) Age</td>
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<td>23°C</td>
<td>OA</td>
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<td>44</td>
<td>16.4</td>
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<td>+750°C(2h)</td>
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</tr>
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<td>10</td>
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</tr>
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</table>

* UA = underaged, OA = overaged

** \( K_{ICL} \) is calculated \( K_{IC} \) using the Equivalent Energy theory. \( K_{ICJ} \) is calculated \( K_{IC} \) using the J-integral theory.
Fig. 14. The charpy transition curves for the 12Ni-1Ti alloy at a hardness of Rc 36.
Fig. 15. The charpy transition curves for the 12Ni-1Ti alloy at a hardness of \( R_c 40 \).
Fig. 16. The charpy transition curves for the 12Ni-2Ti alloy at a hardness of $R_c 43$. 

ALLOY Fe-12Ni-2Ti
HARDNESS 43 $R_c$

AGING TEMP.

- $550^\circ C$
- $600^\circ C$

CHARPY ENERGY, FT-LB

TEST TEMPERATURE, $^\circ C$

$XBL744-5978$
grain boundary cracking in this alloy, indicative of the large amount of boundary precipitation, is shown in Fig. 40 (in the section "Precipitation on Boundaries"). Because of the obviously low toughness of the 12Ni-2Ti alloy, as indicated by the charpy results, no CTS tests were performed.

16Ni-1Ti

The charpy properties of the 16Ni-1Ti alloy are given in Fig. 17, for two hardness levels: $R_{c40}$ ($\sigma_y \sim 180$ ksi) and $R_{c43}$ ($\sigma_y \sim 190$ ksi). This alloy has excellent toughness with room temperature charpy energies of 65 ft-lbs for 550°C aging and 47 ft-lbs for 500°C aging. The CTS specimens also indicate excellent toughness, as shown in Table 4. There is in fact a strong correlation, observed for all of the alloys, between charpy and CTS toughness. This is discussed later.

Again, it is observed that the higher aging temperatures give higher toughness, as measured both by charpy and CTS tests. As mentioned earlier, microstructural analysis of the effects of the increasing aging temperature will be presented in another section.

It should be noted that the $R_{c40}$ CTS were not nearly valid for $K_{IC}$, as may be seen from Fig. 18 which shows a typical load-displacement curve. In these cases there is not a satisfactory method of quantitatively determining the fracture toughness unless one resorts to very large specimens to produce the necessary constraint at the crack-tip. One may, however, estimate the toughness by using either the J-integral or the Equivalent Energy concept. Both techniques use the COD curve obtained from a CTS test to obtain the necessary data for this estimate.
Fig. 17. The charpy transition curves for the 16Ni-1Ti alloy at a hardness of $R_c$ 40 and 43.
Fig. 18. The load-displacement curve for CTS specimen FDA, 16Ni-1Ti. This curve is typical of those for the tougher specimens.
The details associated with using these techniques are given in Appendix 1.

Using the J-integral and Equivalent Energy techniques the upper limit of $K_{IC}$ for the 16Ni-1Ti alloy ($\sigma_y=180$) is estimated to be $\sim 220 \text{ ksi/} \sqrt{\text{in}}$. The lower limit may be taken as the $K_Q$ calculated according to ASTM E399-72, or $150 \text{ ksi/} \sqrt{\text{in}}$. From the charpy correlation $K_{IC}$ is estimated to be $177 \text{ ksi/} \sqrt{\text{in}}$ (for CVN = 65 ft.-lbs.). Thus it is strongly indicated that $K_{IC}$ for the 16Ni-1Ti alloy aged at 550°C is $\sim 170 \text{ ksi/} \sqrt{\text{in}}$ and that at $\sigma_y=180$ the $K_{IC}/\sigma_y$ ratio approximates unity.

The CTS results for 16Ni-1Ti aged at 500°C are somewhat uncertain and are so indicated in Table 4. However on the basis of the charpy results it would appear that the actual $K_{IC}$ is somewhat lower than for the 550°C age and is approximately $150 \text{ ksi/} \sqrt{\text{in}}$.

16Ni-1.5Ti

The charpy properties of the 16Ni-1.5Ti alloy aged at 600°C, 550°C, and 450°C are shown in Fig. 19 (all three in the underaged condition). Again, it is clear that the higher aging temperatures yield higher toughness. This conclusion is also verified by the CTS results listed in Table 4; for the 600°C age $K_{IC} = 114 \text{ ksi/} \sqrt{\text{in}}$ and for the 550°C age $K_{IC} = 50 \text{ ksi/} \sqrt{\text{in}}$. Note that this CTS results agrees with the charpy results. In the room temperature charpy tests specimen COA, aged at 550°C, absorbed 13 ft-lbs and specimen CPA, aged at 600°C, absorbed 27 ft-lbs. SEM photomicrographs of these two specimens are shown in Fig. 20. CPA failed in a fully ductile manner whereas COA failed in a partially brittle manner. For COA note particularly the grain boundary separation in the center of the micrograph.
Fig. 19. The charpy transition curves for the 16Ni-1.5Ti alloy at a hardness of $R_c$ 43.
Fig. 20. SEM fractographs of specimens COA (a), aged at 550°C, and CPA (b), aged at 600°C. These specimens were tested at 23°C.
Additional charpy properties were measured for the 16Ni-1.5Ti alloy. Figure 21 illustrates the effect of hardness (or yield strength) on the charpy transition. \( R_{y}^{C} \) 43 corresponds to \( \sigma_y = 200 \text{ ksi} \) and \( R_{y}^{C} \) 46 corresponds to \( \sigma_y = 225 \text{ ksi} \). It is apparent that the yield strength has a large effect on the fracture toughness in this yield strength range. This will be discussed further in a later section.

Figures 22 and 23 illustrate the effect of aging time on the charpy properties (at a constant strength level). These figures show the effect of underaging and overaging at 450°C and 550°C, respectively. The overaging treatment has two effects on the charpy properties: (1) the upper shelf energy is lowered and (2) the lower shelf energy is increased. In both cases these effects are probably due to the presence of retained austenite. As shown in Table 5 (austenite formation section) overaging at 450°C produced about 3% and overaging at 550°C produced about 1% retained austenite. Lower shelf fracture surfaces of both specimens are shown in Fig. 24. Both specimens show a considerable amount of low energy tearing, as for example marked at region A, and also cleavage fracture. The low energy tearing is probably due to thin regions of retained austenite which formed at grain and lath boundaries during aging. There is also some evidence from TEM which supports the above. Figure 25a shows a transmission micrograph of the 16Ni-1.5Ti alloy overaged at 450°C. Measured d-spacings indicate the region marked B in this figure to be \( \gamma \). The similarity of this structure with that observed by Speich\textsuperscript{27} in an Fe-30Ni-6Ti alloy also supports the conclusion that this region is \( \gamma \).
Fig. 21. The effect of yield strength, as represented by the hardness, on the charpy transition of the 16Ni-1.5Ti alloy.
Fig. 22. Plots of Charpy V-notch impact energy vs test temperature for overaged and underaged specimens of Fe-16Ni-1.5Ti alloy, aged at 450°C to attain a hardness of $R_c$ 43.
Fig. 23. Plots of Charpy V-notch impact energy vs test temperature for overaged and underaged specimens of Fe-16Ni-1.5Ti alloy, aged at 550°C to attain a hardness of $R_c 43$. 

ALLOY Fe-16Ni-1.5Ti
HARDNESS 43 $R_c$
AGING TEMP. 550°C

OVERAGED
UNDERAGED
Fig. 24. Lower shelf fracture surfaces of charpy specimens of the 16Ni-1.5Ti alloy. (a) Overaged at 450°C to produce ~3 percent retained austenite. (b) Overaged at 550°C to produce ~1 percent retained austenite. The areas which show low energy tearing, as indicated at A, are probably due to the crack passing thru regions retained austenite.
Fig. 25. TEM micrograph (a) and carbon replica micrograph (b) of the 16Ni-1.5Ti alloy overaged at 450°C. These micrographs show the cellular precipitation reaction which occurs in this alloy upon overaging at 450°C. The constituent marked B in (a) is austenite. The light regions in (b) correspond to austenite also and this micrograph suggests the cellular reaction occurs at internal boundaries.
Figure 25b shows a carbon replica extraction micrograph of the same specimen and clearly illustrates the presence of what is most probably $\gamma$ at the grain boundaries, as well as precipitates particles.

20Ni-1.5Ti

The charpy properties of the 20Ni-1.5Ti alloy aged at 550°C, 500°C, and 450°C are shown in Fig. 26. These properties are quite different from those of the other alloys in two respects: (1) the upper shelf energy is very low in comparison (~ 30 ft-lbs vs. ~ 100 ft-lbs) and (2) the properties do not show much sensitivity to changes in the aging time (underaging vs. overaging). However, despite the rather low charpy properties the $K_{IC}$ is still rather good, as indicated in Table 4. For example, underaging at 450°C gives a $K_{IC} = 111$ ksi$\sqrt{in}$, which compares very favorably with the 16Ni-1.5Ti alloy at 114 ksi$\sqrt{in}$.

The low charpy properties can not be caused solely by retained austenite since, as shown in Table 5, not all of the specimens contained retained austenite. However, examination of the fracture surfaces does indicate a possible cause. SEM micrographs of the fracture surfaces of charpy specimens underaged at 450°C, 500°C, and 550°C, and overaged at 500°C and 550°C are shown in Fig. 27. These micrographs are all of upper shelf fractures. The fractures are all very similar, indicating that the fracture mechanism is the same for specimens underaged or overaged and for specimens with or without retained austenite. A distinctive feature of the fracture surfaces is that they all exhibit large amounts of low energy tearing, characterized in these micrographs by a very fine dimpled rupture. Moreover, it appears that fractures
Fig. 26. The charpy transition curves for the 20Ni-1.5Ti alloy.
Fig. 27. SEM fractographs of the fracture surfaces of charpy specimens underaged at (a) 450°C, (b) 500°C and (c) 550°C and overaged at (d) 500°C and (e) 550°C. These fractographs show the low energy tearing which occurs in these alloys and which limits the toughness. These fractographs are of specimens broken at or above 23°C.
Fig. 27. (Cont'd)
on the lower shelf occur by the same low energy tearing mechanism. SEM micrographs of two lower shelf fractures are shown in Fig. 28. In fact the difference in energy absorbed between the upper and lower shelf seems to be due to the presence in upper shelf fractures of some regions of relatively large dimples, which are characteristic of a fracture that absorbs more energy.

Thus it is evident that the low charpy energy absorption of the 20Ni-1.5Ti alloy is caused by the presence of a low energy tearing mechanism. However, the microstructural feature responsible for this mechanism has not been positively identified. A possible explanation is that there may be regions in the alloy which are not austenite at room temperature but are nonetheless devoid of precipitates and consequently have a low local yield stress. Such regions, if sufficiently numerous and dispersed, could cause a low energy tearing fracture, even at -196°C in view of the work of Floreen and Speich, Horwood and Sasaki. Attempts to identify such areas by TEM were, however, not successful.

In the 20Ni-1.5Ti alloy retained austenite occurs in relatively large amounts, as indicated in Table 5, and is very stable as evidenced by the fact that quenching into LN did not appreciably transform it into martensite. In order to determine the stability of this austenite tensile specimens were tested at -196°C. These specimens were then x-rayed in the neck and thread sections to determine the amount of austenite transformed by deformation. In all cases the austenite in the neck section was completely transformed to martensite. The
Fig. 28. SEM fractographs of charpy specimens at the 20Ni-1.5Ti alloy. These specimens were broken at -196°C. It is clear that these specimens failed by a low energy tearing mechanism.
tensile data for these specimens is shown in Table 2. This data clearly shows the effect of the austenite "tripping" — that is the uniform elongation is considerably increased. The large increase in uniform elongation also suggests that the austenite transformation is strain-induced.

It seemed that this "tripping" might have a favorable effect on the fracture toughness. Therefore CTS specimens were tested at -196°C. The results are given in Table 4. The $K_{IC}$ at -196°C was 65 ksi$\sqrt{\text{in}}$ at a yield strength (-196°C) of 230 ksi. This is reasonably good toughness at this yield strength level and temperature. The fracture mechanism was again low energy tearing, as shown Fig. 29. However, no direct assessment of the "tripping" effect can be made because specimens without retained austenite were not tested.

E. Austenite Formation

As mentioned earlier, in all the alloys studied in this investigation austenite is formed at some time during the aging process. The formation of austenite during aging has been studied in some detail for the 16Ni-1.0Ti and 16Ni-1.5Ti alloys. Using a magnetometric technique developed by Babu and Parker$^{15}$ and Yuen and Parker$^{16}$, the isothermal rate of formation of austenite at various aging temperatures was measured. The results are shown in Fig. 30 for the 16Ni-1Ti alloy and in Fig. 31 for the 16Ni-1.5Ti alloy.

Comparison of Figs. 30 and 31 with Figs. 9 and 10 suggests that the formation of austenite is probably responsible for overaging in
Fig. 29. Fracture surface of CTS specimen of 20Ni-1.5Ti alloy. This specimen was broken at -196°C.
Fig. 30. The isothermal formation of austenite, as measured by the magnetometric technique, for the 16Ni-1Ti alloy.
Fig. 31. The isothermal formation of austenite, as measured by the magnetometric technique, for the Fe-16Ni-1.5Ti.
these alloys since the beginning of austenite formation is generally coincident with peak hardness. It is possible to check this conclusion using activation energy analysis. For this purpose it has been assumed that a general Zener-Wert-Avrami type Eq. (28) adequately represents the rate of formation of austenite:

\[ 1 - x = \exp\left[-\left(\frac{t}{\tau}\right)^m\right], \quad (4) \]

where \( x \) = fraction of volume that is austenite at time \( t \), and \( \tau \) = time constant which includes among other factors the diffusion coefficient \( D \). If this relationship is indeed adequate a plot of \( \ln \ln \frac{1}{1-x} \) vs. \( \ln t \) should yield a straight line. These quantities are shown plotted in Figs. 32 and 33 for 16Ni-1Ti and 16Ni-1.5Ti respectively, and they do yield straight lines with approximately equal slopes.

The apparent activation energy \( Q \) for the formation of austenite can then be determined from the relationship.

\[ \frac{Q}{RT} = \ln t + \text{constant}, \quad (5) \]

where it has been assumed that

\[ \ln \tau = \ln y + \ln D = \ln y + \ln D_0 - \frac{Q}{RT}, \quad (6) \]

where \( y \) is a constant. A plot of this relationship is shown in Fig. 34 for both alloys. It is apparent that there is considerable scatter in the value obtained for \( Q \). This may be due to complicating effects of precipitation and austenite formation occurring simultaneously.
Fig. 32. Plot of $\ln \ln \frac{1}{1-x}$ vs. $\ln t$ for the 16Ni-1Ti alloy. This plot shows that equation 4 approximately describes the kinetics of austenite formation. The experimental uncertainty is not known so error bars are not shown.
Fig. 33. Plot of $\ln \ln \frac{1}{1-x}$ vs. $\ln t$ for the $16\text{Ni}-1.5\text{Ti}$ alloy. This plot shows that equation 4 approximately describes the kinetics of austenite formation. The experimental uncertainty is not known so error bars are not shown.
<table>
<thead>
<tr>
<th>ALLOY</th>
<th>Q (KCAL/MOLE)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-16 Ni-1Ti</td>
<td>72</td>
</tr>
<tr>
<td>Fe-16 Ni-1.5Ti</td>
<td>61.5</td>
</tr>
</tbody>
</table>

The reason for the wide scatter in Q is not known.

Fig. 34. Arrhenius plot to determine the activation energy for the formation of austenite. The reason for the wide scatter in Q is not known.
or to inaccuracies in the original data. However, ignoring these problems the average value obtained for Q is 87 kcal/mole. This is too high a value to attribute to a simple diffusion process. A possible explanation for this high Q is discussed later.

In a similar manner we may obtain an apparent activation energy for the overaging of the 16Ni-1.5Ti alloy. Here, we make use of the empirically observed fact that for overaging

$$\frac{\Delta R}{\Delta R_0} \approx k t^n,$$

(7)

where $\Delta R$ is the observed hardness change in time t, $\Delta R_0$ is a constant, and k is independent of time but assumed to be a function of the diffusion coefficient D. This type of relationship has been observed to describe the initial aging kinetics of maraging steels.\textsuperscript{29,30} A plot of Eq. (7) for the overaging data of 16Ni-1.5Ti is shown in Fig. 35. It should be noted that for this plot $t=0$ starts when the austenite begins to form; and that this time was estimated from Fig. 31 to be 20 seconds for aging at 550°C and 1000 seconds for 500°C; the estimated time for the 450°C aging was taken from both Fig. 31 and Fig. 10 and was taken to be 15,000 seconds. In Fig. 36 the data is plotted according to Eq. (5) to yield an apparent activation energy for the overaging process of 82 kcal/mole.
Fig. 35. Plot of Eq. (7) for the overaging of the 16Ni-1.5Ti alloy. Data from this plot is used to find the activation energy for overaging, as shown in Fig. 36.
Fig. 36. Arrhenius plot to determine the activation energy for overaging of the 16Ni-1.5Ti alloy.
The close agreement with the average apparent activation energy for formation of austenite (= 87 kcal/mole) is perhaps fortuitous. However, the general agreement does give strong support to the conclusion that overaging occurs primarily because of the formation of austenite.

It is of interest to compare the apparent activation energy obtained here with that obtained from the data in Fig. 6 of Peters and Cupp\textsuperscript{30} for an Fe-18Ni-8Co-5Mo alloy. An Arrhenius plot of their data is shown in Fig. 37. Note that for this plot \( t = 0 \), the time when austenite first starts to form, was taken to be 0, .8, and 10 hours respectively for the 566°C, 510°C, and 454°C aging temperatures. The apparent activation energy measured from this plot is 64 kcal/mole. This \( Q \) is in the range of the activation energy for diffusion of substitutional elements in austenite,\textsuperscript{31} and it is considerably less than that measured in this investigation. The reason for the difference lies in the different austenite contents measured. In the work of Peters and Cupp what was actually measured was the amount of retained austenite, since the measurements were taken by X-rays after the aging treatment. In this investigation, however, the actual amount of austenite formed at temperature was measured, and as shown in this investigation the two quantities are not the same.

It was not determined why the two measured quantities had a different temperature dependence. However, as discussed by Jin\textsuperscript{14} there is ample evidence that in maraging steels in general and also in many other Fe-Ni base alloys austenite can form either by a shear or a diffusion process. It may be that the high apparent activation energy
Fig. 37. Arrhenius plot of the data of Peters and Cupp\textsuperscript{30} for the formation of retained austenite. The activation energy for this reaction is consistent with the activation energy one would expect for substitutional solid diffusion.
is associated with the shear transformation and that the large scatter in Q values, as seen in Fig. 34, is due to transformation occurring both by shear and by diffusion (hence an apparent activation energy is measured). Further study is needed to clarify this problem.

The amount of retained austenite, as determined by X-ray analysis, for the 16Ni-1Ti, 16Ni-1.5Ti, and 20Ni-1.5Ti alloys with various aging treatments is shown in Table 5. In general the amount of retained austenite increases with increasing Ni content, as one might expect. It is interesting to note that the austenite formed at 600°C in the 16Ni-1.5Ti alloy had an $M_f$ above room temperature. As measured both by the magnetometric technique and by dilatometry the austenite in this case had an $M_S$ of $\sim 295°C$ and an $M_f$ of $\sim 185°C$. These values are only slightly less than those for the fully annealed austenite, as shown in Fig. 5. Thus, it appears that retained austenite will not be found for 600°C aging simply because the austenite formed does not have an $M_f$ below room temperature. In this case there is insufficient partitioning of the Ni during aging to stabilize the austenite; however at the lower aging temperatures there is sufficient partitioning to produce retained austenite.

It was of some interest to determine the distribution of the retained austenite, particularly because of the influence retained austenite has on the fracture properties. Figure 38 shows TEM micrographs of a 20Ni-1.5Ti alloy overaged 5 hrs. at 550°C. The austenite is uniformly distributed, as "laths", throughout the microstructure. This figure also shows a carbon extraction replica
Table 5. Retained Austenite in Fe-Ni-Ti Alloys

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Treatment</th>
<th>% Austenite</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>without LN quench</td>
</tr>
<tr>
<td>Fe-16Ni-Ti</td>
<td>900°C(2h)+750°C(2h)+500°C(21m) age</td>
<td>ND</td>
</tr>
<tr>
<td>Fe-16Ni-1.5Ti</td>
<td>900°C(2h)+800°C(2h)</td>
<td>~1</td>
</tr>
<tr>
<td></td>
<td>+600°C(2h)+800°C(2h)+550°C(3h) age</td>
<td>ND*</td>
</tr>
<tr>
<td></td>
<td>900°C(2h)+800°C(2h)</td>
<td>ND</td>
</tr>
<tr>
<td></td>
<td>+600°C(2h)+800°C(2h)+550°C(4m) age</td>
<td>ND</td>
</tr>
<tr>
<td></td>
<td>900°C(2h)+800°C(2h)</td>
<td>3.0</td>
</tr>
<tr>
<td></td>
<td>+600°C(2h)+800°C(2h)+450°C(29 days) age</td>
<td></td>
</tr>
<tr>
<td></td>
<td>900°C(2h)+800°C(2h)</td>
<td>ND</td>
</tr>
<tr>
<td></td>
<td>+600°C(2h)+800°C(2h)+450°C(1h) age</td>
<td>ND</td>
</tr>
<tr>
<td></td>
<td>900°C(2h)+800°C(2h)</td>
<td>ND</td>
</tr>
<tr>
<td></td>
<td>+600°C(2h)+800°C(2h)+600°C(30m) age</td>
<td>ND</td>
</tr>
<tr>
<td></td>
<td>900°C(2h)+800°C(2h)</td>
<td>ND</td>
</tr>
<tr>
<td></td>
<td>+600°C(2h)+800°C(2h)+600°C(10m) age</td>
<td>ND</td>
</tr>
<tr>
<td>Fe-20Ni-1.5Ti</td>
<td>900°C(2h)+750°C(2h)+550°C(5h) age</td>
<td>23.8</td>
</tr>
<tr>
<td></td>
<td>&quot;</td>
<td>ND</td>
</tr>
<tr>
<td></td>
<td>+550°C(1m) age</td>
<td>ND</td>
</tr>
<tr>
<td></td>
<td>&quot;</td>
<td>18</td>
</tr>
<tr>
<td></td>
<td>+500°C(4m) age</td>
<td>ND</td>
</tr>
<tr>
<td></td>
<td>&quot;</td>
<td>ND</td>
</tr>
</tbody>
</table>

* ND - not detectable
Fig. 38. TEM micrographs of the 20Ni-1.5Ti alloy averaged at 550°C, (a) BF, (b) DF, (c) SAD, and (d) carbon replica. This series of micrographs shows the distribution of retained austenite in this alloy. The "blocky" areas in (d) are probably at a grain boundary. "Austenite" appears in (d) as light regions, void of precipitates.
of the same specimen. The austenite regions may be identified by the absence of precipitates and by comparison with the TEM micrographs. For the 16Ni-1.5Ti alloy overaged at 450°C the retained austenite appears to be located in the grain boundaries, as previously shown in Fig. 25. The distribution of retained austenite in 16Ni-1.5Ti specimens overaged at 550°C was not determined.

F. Precipitation on Boundaries

In the alloys used in this investigation, and in maraging steels in general, Ti is a very effective alloying element for achieving high strengths. However, it usually has a detrimental effect on the fracture toughness because of preferential precipitation on internal boundaries, which promotes intergranular failure. This intergranular failure was manifested most clearly in the 12Ni-2Ti alloy, which had the worst fracture properties of any of the alloys investigated. Figure 39 shows optical, SEM, and carbon extraction replica micrographs of a 12Ni-2Ti specimen underaged at 550°C. The SEM micrograph clearly shows extensive amounts of grain boundary failure, and the carbon replica shows the boundary precipitation. Selected area diffraction (SAD) indicates that these boundary precipitates are also \( \eta \) Ni$_3$Ti. It is interesting to note that the internal boundaries are easily delineated in the optical micrograph of this specimen, whereas they are difficult to observe in a specimen annealed and then quenched. The delineation of internal boundaries in the aged specimen is undoubtably due to the extensive boundary precipitation.
Fig. 39. Optical (a), SEM (b), and carbon replica (d) and (d) micrographs of the 12Ni-2Ti alloy, underaged at 550°C. The brittleness due to grain boundary precipitation is evident in (b). Grain boundary precipitate particles are evident in (c) and (d).
Intergranular failure due to boundary precipitation was also evident in the 16Ni-1.5Ti alloy, as shown in Fig. 40 for underaging at 450°C, and in Fig. 41 for underaging at 550°C. TEM micrographs for this alloy have also shown boundary precipitation, as shown in Fig. 42 and 43. The boundary precipitates were tentatively identified by SAD to be $\eta \text{ Ni}_3\text{Ti}$.

Boundary precipitation has also been observed in the 20Ni-1.5Ti alloy, as shown in Fig. 44. The structure of this precipitate has not been determined.

If boundary precipitation is providing an easy fracture path then it should be possible to extract precipitates from the fracture surfaces. Such carbon extraction replica fractographs were made, from the fracture surfaces of charpy bars, for the 12Ni-2Ti, 16Ni-1Ti, and 16Ni-1.5Ti alloys. (The replication of some specimens by this technique proved impossible because the carbon film could not be removed from the fracture surface.) These fractographs are shown in Fig. 45. It is evident that precipitate particles are present on all the fracture surfaces. In all cases where precipitates were present on the surface replicas, SAD showed the crystal structure of these precipitates to be consistent with that of $\eta \text{ Ni}_3\text{Ti}$. Their presence is most prominent on the 12Ni-2Ti alloy, where they are quite large; the poor fracture toughness of this alloy is clearly due to these precipitates. However, in the other alloys they are not very prominent, and their effect on the fracture toughness is not so easily explained.
Fig. 40. Optical (a), SEM fractographs at a charpy specimen (b), and carbon replica (c) micrographs of the 16Ni-1.5Ti alloy. Grain boundary brittleness is evident in (b), as are boundary precipitates in (c). These samples were underaged at 450°C. The charpy specimen in (b) was broken at 23°C.
Fig. 41. Optical (a), SEM fractograph of charpy specimen (b), and carbon replica (c) of the 16Ni-1.5Ti alloy, underaged at 550°C. Grain boundary brittleness is evident in (b). In (c) grain boundary precipitation is evident, as indicated by the arrow. These samples were underaged at 550°C. The charpy specimen was broken at -77°C.
Fig. 42. BF(a) and DF(b) TEM micrographs of the 16Ni-1.5Ti alloy underaged at 450°C. These micrographs show the grain boundary precipitation.
Fig. 43. BF(a) and DF(b) TEM micrographs of the 16Ni-1.5Ti alloy underaged at 550°C. These micrographs show the grain boundary precipitation.
Fig. 44. BF(a), DF(b), and SAD(c) micrographs of the 20Ni-1.5Ti alloy, underaged at 500°C. These micrographs show boundary precipitation. Arrow indicates spot used for DF.
Fig. 45. Carbon replica fractographs of the 16Ni-1Ti alloy (a), the 12Ni-2Ti alloy (b) and the 16Ni0.5Ti alloy (c), all underaged at 550°C, and the 16Ni-1.5Ti alloy (d) underaged at 450°C. These lower shelf fracture surfaces all show evidence of precipitate particles on the fracture surface.
A logical explanation for the observed improvement of fracture properties for the higher aging temperatures would be that the amount and/or size of the boundary precipitate particles was decreased by the higher aging temperatures. It is very difficult if not impossible to directly support this explanation because of the difficulty in examining a sufficient number of grain boundaries to obtain reasonable statistics. Thus for the 16Ni-1.5Ti alloy aged at 600°C, which aging temperature gives the best properties for this alloy, carbon extraction replication shows the presence of boundary precipitates, as shown in Fig. 46a even though the fracture surface does not show any boundary "sensitivity", as may be seen from Fig. 46b.

There is, however, some additional evidence to support the hypothesis that the higher aging temperatures produce less boundary precipitation. This evidence is shown in Fig. 47. These fractographs are from the fatigue cracked region of 16Ni-1.5Ti specimens aged at 550°C and 600°C. The smooth, faceted portions of Fig. 48a show that the fatigue crack path in the 550°C specimen generally followed grain boundaries (not necessarily prior austenite grain boundaries). In the 600°C specimen, however, the fatigue crack path was only occasionally along grain boundaries. This indicates that fewer boundaries were "sensitized" at the higher aging temperature.

Thus it is clear that the aging temperature does affect the amount and/or size of boundary precipitation. Other investigations have found boundary precipitation in maraging steels, but the strong effect of aging temperature on this precipitation appears to be previously unknown.
Fig. 46. TEM carbon replica (a) and SEM fractograph of charpy specimen broken at -77°C (b). Grain boundary precipitation is evident in (a) but there does not appear to be much grain boundary fracture in (b).
Fig. 47. SEM fractographs of fatigue cracked portions of CTS specimens of the 16Ni-1.5Ti alloy: (a) aged at 550°C, (b) aged at 600°C. The greatly increased amount of grain boundary fatigue in (a), compared to (b), is evident. These regions are representative of the average appearance of the fatigued portion of the specimens.
It should be noted that there may be other factors affecting the fracture properties also. Precipitate size and coherency for example would be of considerable importance.\textsuperscript{32} There factors were not isolated in this investigation.

Also, several investigators have found that certain annealing procedures produce severe grain boundary embrittlement in 18Ni maraging steels.\textsuperscript{33,34} This embrittlement appears to be due to segregation of Ti and C to the austenite grain boundaries during annealing. However, in the present investigation annealed charpy specimens of the 12Ni-1Ti alloy were tested at temperatures down to \(-75^\circ\text{C}\). The charpy energy absorbed was 150 ft-lbs. Thus, the austenite boundaries are clearly not embrittled by the annealing treatments in the present investigation.

G. Precipitate-Austenite Interaction

There is some strong indirect evidence which suggests there is a beneficial interaction between austenite formation and boundary precipitation, and that this interaction is responsible for the reduced sensitivity to grain boundary cracking in specimens aged at the higher temperatures. Consider Fig. 48, which shows both the percent austenite and the Rockwell C hardness (as a function of aging time), for the 16Ni-1.5Ti alloy. The hardness is used here as a measure of the "completeness" of the precipitation reaction. A comparison of the two sets of curves reveals that at the lower aging temperatures, for example 500°C, austenite has just begun to form when the peak hardness is reached at \(10^3\) secs. Conversely, at the highest aging temperature,
Fig. 48. Comparison of the rate of formation of austenite with the rate of formation of precipitate for the 16Ni-1.5Ti alloy.
600°C, austenite formation and Ni$_3$Ti precipitation are occurring simultaneously. For example, at 600°C the hardness is $R_c$ 30 at an aging time of 35 secs and at this time there is approximately 10% austenite. Thus there is a correlation between the relative rates of formation of austenite and precipitate with the development of the best fracture toughness—namely that when the formation of austenite and of Ni$_3$Ti occur simultaneously, i.e., at 600°C, the fracture toughness improves. The improvement of fracture toughness with increased aging temperature is shown in Table 6 for the 16Ni-1.5Ti alloy.

It will be noted from Table 6 that the toughness of specimens aged at 600°C is considerably better than for specimens aged at 550°C, even though in both cases austenite and precipitate formation (Fig. 49) are occurring approximately simultaneously. This suggests that there is a critical balance between the rate of formation of austenite and of precipitate at the grain or lath boundaries, and that at 500°C a deleterious amount of boundary precipitation has taken place prior to the onset of austenite formation. Evidence in support of this view has been shown in the previously discussed Fig. 47, which shows SEM fractographs of the fatigue cracked regions of fracture toughness specimens aged at 550°C and 600°C. The 550°C specimen shows considerably more grain boundary cracking than does the 600°C specimen. This indicates that there was more boundary precipitation in the 550°C specimen, and is consistent with the lower fracture toughness. In this regard it should be noted that the fracture toughness of 550°C
Table 6. Fracture toughness for various aging temperatures.

<table>
<thead>
<tr>
<th>Alloy (wt %)</th>
<th>Aging Temp.</th>
<th>Hardness (y 200ksi)</th>
<th>$R_c$</th>
<th>$K_{IC}$ (ksi√in)</th>
<th>CVN (ft-lbs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-16Ni-1.5Ti</td>
<td>600°C</td>
<td>43</td>
<td>~114</td>
<td>27</td>
<td></td>
</tr>
<tr>
<td></td>
<td>550°C</td>
<td>43</td>
<td>50</td>
<td>14</td>
<td></td>
</tr>
<tr>
<td></td>
<td>450°C</td>
<td>43</td>
<td>-</td>
<td>3</td>
<td></td>
</tr>
</tbody>
</table>
aged specimens is greater, as shown by the CVN (Table 6), than that of the 450°C aged specimens; so there is some beneficial interaction occurring during aging at 550°C, as Fig. 48 would suggest there might be.

Attempts to substantiate this interaction between the austenite and the precipitate with direct evidence have proven to be difficult. Hot stage electron microscopy was unproductive due to the formation of a surface rather than a bulk precipitate—so that the structures obtained were very different from those observed on specimens aged in bulk and then examined. Thus it was not possible to observe directly the formation of the austenite on the boundary during aging. Furthermore, electron microscopy of specimens heat treated prior to examination did not clarify the problem for two reasons: (1) Austenite formed at the higher aging temperatures retransforms to ferrite upon cooling to room temperature and does not leave any observable evidence of its existence (the microstructures are highly dislocated so that evidence of the retransformation is obscured). (2) All of the specimens exhibit at least some boundary precipitation, and so it is not possible to discern clearly the difference between specimens aged at the various temperatures.

It has been observed in this investigation that when retained austenite is present it first appears at grain and lath boundaries. It seems clear that even when the austenite is not retained, as in case of the 16Ni-1.5Ti alloy in the present discussion, austenite forms initially at internal boundaries. Jin\textsuperscript{38} and Peters\textsuperscript{11} have also observed that in similar alloys the austenite forms first at internal boundaries.
In addition, the precipitate in this alloy, hexagonal Ni$_3$Ti (DO$_{24}$), does not appear to precipitate in austenite. For example, in specimens aged at 650°C (a temperature at which a considerable amount of austenite is formed very rapidly) no precipitation is observed in the austenite; precipitation occurs only in the ferritic portions of the specimen. Furthermore, specimens which are fully austenitic may be cooled to temperatures anywhere in the range above the $M_s$ (315°C for the Fe-16Ni-1.5Ti alloy) and held there without any precipitation being observed.

Thus, the reason for the beneficial interaction between the austenite and the Ni$_3$Ti at the internal boundaries appears to be that the austenite prevents either the nucleation or the growth of the precipitate at the boundary. The degree of interaction between the austenite and the boundary precipitate is controlled by the relative rates of formation of the austenite and the precipitate.

This discussion has so far been confined to the 16Ni-1.5Ti alloy because it demonstrated the proposed interaction between austenite and Ni$_3$Ti most clearly. The charpy results of the 12Ni-1Ti and 16Ni-1Ti alloys indicate there is also an interaction for these alloys. For the 16Ni-1Ti alloy the interaction does not seem to be as important as for the 16Ni-1.5Ti. This may be seen by comparing Fig. 9 with Fig. 30 (i.e. comparing hardness vs. percent austenite as has been done for the 16Ni-1.5Ti alloy). It is clear that in this case the austenite is not forming as rapidly, relative to the precipitate, as it was for the 16Ni-1.5Ti alloy. The boundary precipitation appears to be less severe.
for the 16Ni-1Ti alloy primarily because of the lower Ti content.
However, as shown by the charpy results there is still an effect due
to variations in the aging temperature. Similarly, the charpy results
for the 12Ni-1Ti alloy show a variation with aging temperature.
However the isothermal formation of austenite has not been measured
for the 12Ni-1Ti alloy so it is not possible to compare the relative
formation rates of austenite and Ni₃Ti for this alloy.

H. Effect of Composition on Toughness

It has been clearly established by many investigators (see for
example Leslie)¹⁰ that addition of Ni to BCC iron very effectively
lowers the toughness transition temperature. It appears that this
is due to the effect Ni has upon the cleavage stress (i.e. the critical
tensile stress, ahead of the notch or crack, necessary to achieve
fracture) of BCC iron, since it has been shown by Floreen et al.³⁵
that the cleavage stress is increased by the addition of Ni up to at
least 4 wt%. Higher percentages of Ni were not investigated so it
is not known at what %Ni the cleavage stress reaches its maximum value.
Nonetheless, upon the above basis the %Ni in the alloys of the present
investigation may be expected to be influential. In addition the Ni
content will of course effect the aging kinetics (i.e. the relative
rates of formation of austenite and precipitate) which has also been
shown to be influential.
A composition effect was in fact observed. From an examination of charpy toughness of several alloys aged at the same temperature to the same hardness level it was evident that the transition temperature decreased as the nickel content increased and as the titanium content decreased. Since the matrix composition of Ni is the factor of interest here, these two compositional variables can be incorporated into a single parameter called the "effective nickel content." This is the matrix Ni composition after the removal of Ni from substitutional solid solution by precipitation. In calculating this parameter it is assumed that all of the titanium is in the Ni\textsubscript{3}Ti (See Appendix 4). A plot of the 25 ft-lb ductile to brittle transition temperature as a function of "effective nickel content" is shown in Fig. 49, for various hardness levels. It is clear from this figure that the Ni and Ti contents do have a strong effect on the fracture toughness.

I. Comparison of Properties

The mechanical properties and toughness of Fe-Ni-Co-Ti alloys were examined by Floreen and Speich.\textsuperscript{6} They found that these alloys had inferior toughness because of the presence of grain boundary precipitation. A summary of their results is shown in Fig. 50. It is apparent that their results were much inferior to the present investigation. Even for their alloys which used Mo rather than Ti as the principal hardening agent, their room temperature charpy values were considerably less than in the present investigation. However,
Fig. 49. The effect of the effective nickel content upon the ductile-brittle transition temperature of charpy specimens.
Fig. 50. Graphical summary of the results of Floreen and Speich,\textsuperscript{6} for Fe-Ni-Co base alloys.
their aging temperature was 425°C, which, at least in the case of the alloys using Ti, probably accounts for the much lower charpy values.

It is perhaps more interesting and informative to compare with the properties of commercial maraging steels. The charpy properties of commercial maraging steels are shown in Fig. 51.\(^5\) For comparison the best charpy properties for the 16Ni-1Ti alloy (aged at 550°C to \(\sigma_y \sim 200\) ksi) are shown on the same figure. It is apparent that the low temperature charpy properties of the commercial alloy are superior but that at high temperatures the properties of the present alloys are superior. The fact that these alloys absorb more energy in fully ductile rupture than do the commercial steels may almost certainly be explained by variations in the inclusion contents or by variations in some other microstructural feature which affects void nucleation and/or growth (see, for example, Cox and Low\(^4\)). The fact that the commercial alloys are superior at low temperature probably means that cleavage fracture is initiated more readily in the present alloys. Essentially, this would mean the NDT (nil-ductility temperature) is lower for the commercial alloys than for the present alloys.\(^3\)\(^1\) The NDT has been determined for the 16Ni-1.5Ti alloy (averaged at 600°C to a \(\sigma_y = 180\) ksi); the NDT \(= 0°C\). This NDT was determined from the curve of \(K_{IC}\) versus test temperature shown in Fig. 52.\(^2\),\(^3\),\(^3\)\(^6\) This is much higher than the NDT of 18Ni 200 which is listed in Ref. (30) as \(< -196°C\).

In terms of \(K_{IC}\) the present alloys compare very favorably at room temperature with the commercial alloys. For example, at room temperature
Fig. 51. Comparison of charpy properties of 16Ni-1Ti, and 16Ni-1.5Ti alloys with those of commercial maraging steel grades.
Fig. 52. Plot of $K_{IC}$ vs. test temperature for the 16Ni-1.5Ti alloy, for determining the NDT.
Ref. (30) gives a $K_{IC}$ for 18Ni200 of ~ 120 ksi, and Ref. (34) gives a $K_{IC}$ of ~ 100 ksi. The value for the 16Ni-1.5Ti alloy ($\sigma_y$ ~ 200 ksi) was 114 ksi. It is not known how the $K_{IC}$ values compare at lower temperatures, but presumably (on the basis of the NDT) the 16-1.5 alloy would be inferior at lower temperatures.

It would be very useful to know why cleavage initiates more readily in the present alloys than in commercial ones. Unfortunately, it is very difficult to determine the source of cleavage initiation, and it was not possible to do so in this research. However, some speculation as to the source of initiation would still be useful.

It is generally accepted that in mild steel cleavage fracture is usually associated with cracking of grain boundary carbides.\textsuperscript{1,2,7} In the present alloys grain boundary precipitates are also clearly associated with cleavage fracture, as demonstrated by their effect on the charpy transition temperature. It has been shown that the higher aging temperatures reduced this grain boundary precipitation. However, there may still be some grain boundary precipitates scattered throughout the microstructure on higher energy boundaries. These precipitates could easily be sufficiently potent cleavage initiation sites that the NDT would occur at a higher temperature than might otherwise be expected. On this basis the superior NDT of the commercial alloys could be explained by a significantly reduced amount of boundary precipitation. It should be noted that this has been found to be one of the major benefits of using Mo as an alloying element in maraging steels.\textsuperscript{6,18} It appears that Mo additions may be more effective in reducing boundary precipitation.
than is raising the aging temperature. However, this is far from certain since in the commercial maraging steels the Ti contents are considerably lower than in the present alloys.

It should also be noted that increasing the yield strength has the same effect as lowering the test temperature as far as fracture toughness is concerned. This is because the stress intensification at the crack tip is increased in either case. Thus the amount of boundary precipitation will have a strong effect on the maximum usable yield strength (consistent with good toughness).
IV. CONCLUSIONS

This investigation has shown that good fracture toughness may be achieved in the Fe-Ni-Ti system, in the composition range Fe-\(^{\sim}16\text{Ni} -^{\sim}1.0-1.5\text{Ti}\), if sufficiently high age hardening temperatures \((\sim550^\circ\text{C} - 600^\circ\text{C})\) are used. For example, the \(16\text{Ni}-1.0\text{Ti}\) alloy aged at \(550^\circ\text{C}\) to a yield strength of 180 ksi has a \(K_{IC}(23^\circ\text{C})\) of \(\sim\) 170 ksi\(\sqrt{\text{in}}\), and the \(16\text{Ni}-1.5\text{Ti}\) alloy aged at \(600^\circ\text{C}\) to a yield strength of 200 ksi has a \(K_{IC}(23^\circ\text{C})\) of \(\sim\) 114 ksi. The high aging temperatures produce the high fracture toughness by reducing the amount of grain boundary precipitation. Lower aging temperatures \((\sim450^\circ\text{C})\) produce large amounts of grain boundary precipitation and therefore low fracture toughness.

It appears that the higher aging temperatures reduce the amount of grain boundary precipitation because at these temperatures austenite forms at the boundaries simultaneously with the precipitate. The presence of austenite at the boundaries during precipitation reduces the amount of boundary precipitate that is formed. This austenite is not necessarily retained upon cooling back to room temperature.

In addition to the effect of aging temperature on toughness it was also shown that composition has an important effect on the fracture properties. Thus, the optimum Ni content is \(\sim\) 16 percent. At this Ni level austenite forms sufficiently rapidly at the higher aging temperatures that the austenite/precipitate interaction at the grain boundaries can take place. The optimum Ti content is the minimum
amount necessary to obtain the desired strength level. The higher the Ti content the more rapid is the precipitation reaction and the greater is the amount of boundary precipitation.

Although the higher aging temperatures are beneficial to the toughness the use of these higher temperatures has some disadvantages. First, there is the loss of potential strength. For example for the 16Ni-1.5Ti alloy about 50 ksi potential yield strength is lost by aging at 600°C rather than 450°C. Second, at the high aging temperatures precipitation is very rapid (i.e. ~ 10 minutes to peak hardness). Consequently section sensitivity becomes a problem—i.e. it will not be possible to achieve uniform properties in other than small sections. However, while the commercial usefulness of these alloys may thus be limited the concept of the austenite/precipitate interaction could be of considerable benefit in either understanding or developing other alloy systems.

Finally, in this investigation it was shown that there is a good correlation between charpy energy and $K_{IC}$ values, providing that both types of specimens failed by the same mechanism at the test temperature (i.e. ductile failure).
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APPENDIX 1

Estimating $K_{IC}$

It has been shown by Rice et al.\textsuperscript{38} that the load-displacement curve of a CTS may be used to obtain $J_{IC}$, provided it can be established where on the curve initiation of crack propagation occurred. The appropriate equation is\textsuperscript{38}

$$J_{IC} \approx \frac{2A_1}{B(W-a)}$$

(1a)

where $A_1$ is the area under the load-ram displacement curve for a CTS test and $B$, $W$, and $a$ are as defined in ASTM E 399-72. $K_{IC}$ may then be calculated from $J_{IC}$ using the relation

$$K_{IC} = \left(\frac{E J_{IC}}{(1-\nu)^2}\right)^{1/2}$$

(1b)

where $E$ is Young's modulus and $\nu$ is Poisson's ratio.

The difficulty in this technique is in determining the initiation of fibrous fracture. Although it can be done, no attempt was made to determine initiation. Instead, it has been assumed that fracture initiates at peak load. This is probably not true since peak load in these specimens is most likely coincident with the formation of shear lips. Such coincidence was in fact observed during the testing of these specimens, at least within the accuracy of visual observation. The assumption of peak load initiation does however give an upper limit to the actual value of $K_{IC}$. In Table 4 this upper limit is $K_{ICJ}$. 
Note that in using Eq. (1a) one must measure the area under the load-ram-displacement curve, whereas the data is acquired in the form of a load-clip gage displacement curve. The two curves are related by geometry. Consider the two quantities $\delta_{\text{COD}}$, the displacement measured by the clip gage, and $\delta_p$, the displacement of the load application point. If we assume the center of rotation of the specimen is at the fatigue crack tip (this is not strictly true—the center of rotation is a little beyond the fatigue crack tip) then $\delta_{\text{COD}}$ and $\delta_p$ are related by similar triangles. From Fig. 3,

$$\frac{a}{a + \frac{1}{2}} \approx \frac{\delta_p}{\delta_{\text{COD}}}, \quad (1c)$$

For the CTS used in this investigation $a \approx 1''$. Therefore,

$$\delta_p \approx \frac{2}{3} \delta_{\text{COD}}, \quad (1d)$$

and

$$A_1 \approx \frac{2}{3} A_{\text{COD}}, \quad (1e)$$

where $A_{\text{COD}}$ is the area under the load-COD curve.

In a similar manner the toughness may be estimated using the Equivalent Energy concept. The fracture toughness is determined from the load-COD curve using

$$K_{IC} \approx \frac{P}{Bw} \frac{f(a/w)}{\sqrt{A_1/A_2}} , \quad (1f)$$
where a, B, and w are defined as before and f(a/w) is the shape factor found in the ASTM standards (E399-72). The quantity A₁ is the area under the curve to crack initiation and the quantity A₂ is the area under the curve to any point PQ on the linear portion of the curve. This estimate of K_{IC} is shown in Table 4 as K_{ICE}. This technique is subject to the same problems of determining fracture initiation as the J_{IC} technique. Because of this, K_{ICE} is probably also an upper limit estimate of K_{IC}.

There is another method of estimating K_{IC} which in some instances has worked rather well. That is to use correlations between charpy and CTS data. There are two correlations which have been used with some success. It has been shown by Rolfe et.al.⁴⁰ that for many materials which fail in a fully ductile manner in the charpy test there is a good correlation between K_{IC} and the charpy energy (CVN). The empirical relationship is

\[ K_{IC}^2 = 5 \sigma_y CVN - \frac{\sigma_y^2}{4}, \]

where K_{IC} and \sigma_y are in ksi, CVN is in ft.-lbs. and \sigma_y is the tensile yield strength. On the other hand Sailors and Corten⁴¹ found that empirically

\[ K_{IC} = 15.5 (CVN)^{1/2}, \]

where again K_{IC} and CVN are in ksi/in and ft.-lbs. respectively. For reasons discussed in Appendix 3 the correlation used for this investigation is
where $K_{IC}$ is in ksi $\sqrt{\text{in}}$ and CVN is in ft.-lbs. This correlation is plotted in Fig. 3A, Appendix 3, along with data from Rolfe et al. and from this investigation. The two points from this investigation which do not correlate well are from 20Ni-1.5Ti specimens. There is approximately 20% retained austenite in these samples and the presence of this austenite undoubtedly causes some alterations in the failure mechanism. Thus it is not unexpected to find they do not correlate. The 16Ni-1.5Ti point represents a fully ductile failure in both the charpy and CTS tests. Specimens which did not fail in a fully ductile manner in both charpy and CTS tests were not plotted.
APPENDIX 2

Calculation of Retained Austenite

The amount of retained austenite was determined from X-ray data using the direct comparison method as described by Cullity. The development of this method begins with the exact expression for the diffracted intensity from a single phase completely random powder specimen in a diffractometer,

\[
I = \left( \frac{I_0 e^4}{m^2 c^4} \right) \left( \frac{\lambda^2 A}{32 \pi r} \right) \left( \frac{1}{v^2} \right) \left[ |F|^2 p \left( \frac{1+\cos^2 2\theta}{\sin^2 \theta \cos \theta} \right) \right] \left( e^{-2M} \right) .
\]

(2a)

where \( I \) = integrated intensity per unit length of diffraction line, \( I_0 \) = intensity of incident beam, \( e, m \) = the charge and mass of the electron respectively, \( c \) = the speed of light, \( \lambda \) = wavelength of incident radiation, \( r \) = radius of diffractometer circle, \( A \) = cross sectional area of incident beam, \( v \) = volume of unit cell, \( F \) = structure factor, \( p \) = multiplicity, \( \theta \) = Bragg angle, \( e^{-2M} \) = temperature factor, and \( u \) = linear absorption coefficient. Equation (2a) may be divided into two factors

\[
K = \left( \frac{I_0 e^4}{m^2 c^4} \right) \left( \frac{\lambda^2 A}{32 \pi r} \right) .
\]

(2b)

and

\[
R = \frac{1}{v^2} \left[ |F|^2 p \left( \frac{1+\cos^2 2\theta}{\sin^2 \theta \cos \theta} \right) \right] e^{-2M} ,
\]

(2c)
where \( K \) is constant for a given diffractometer and \( R \) is a function of the diffraction conditions and substance. Now for a multiphase material the total diffracted intensity will be the sum of the diffracted intensities of each phase, and the diffracted intensity of each phase will be proportional to the amount by volume of each phase. If these are three phases present, as there are in this investigation, we have

\[
C_\gamma + C_\alpha + C_p = 1 ,
\]

(2d)

where \( \gamma = \text{austenite}, \alpha = \text{ferrite or martensite}, p = \text{precipitate}, \) and \( c = \text{fractional amount of phase present}. \) Moreover, we have

\[
\frac{I_\gamma}{I_\gamma} = \frac{R_\gamma C_\gamma}{R_\alpha C_\alpha} .
\]

(2e)

The \( u \)'s cancel because the diffracted beams from each phase see the same average \( u_m = \text{linear absorption coefficient of mixture of phases}. \) Thus, substituting into (2d) we get,

\[
\frac{I_\alpha}{I_\gamma} \frac{R_\gamma}{R_\alpha} C_\gamma + C_\gamma + C_p = 1 .
\]

(2f)

Finally, we have

\[
C_\gamma = (1 - C_p) \frac{1}{1 + \frac{I_\alpha}{I_\gamma} \frac{R_\gamma}{R_\alpha}} .
\]

(2g)
If we know $C_p$ we can determine $C_\gamma$ from the measured intensities and the calculated R's. Since the lattice parameters and the number of atoms per unit cell are known for $\eta$ Ni$_3$Ti($c_o$=8.31\AA, $a_o$=5.1\AA, 12Ni, 4Ti) $C_p$ can be calculated if it is assumed all the Ti goes into the precipitate. Performing the necessary calculations for a 1.5 wt\% Ti alloy

$$C_p = 6.8 \text{ vol}\%.$$ (2h)

Also performing the necessary calculations for R it is found that

$$R_{311\gamma} = 5.388 \times 10^{49} \text{ \AA}^{-6},$$ (2i)

$$R_{211\alpha} = 6.553 \times 10^{49} \text{ \AA}^{-6}$$ (2j)

and

$$\frac{R_{311\gamma}}{R_{211\alpha}} = .822.$$ (2k)

Thus,

$$C_\alpha = .93 \frac{1}{1 + .822 \frac{I\alpha}{I\gamma}}.$$ (21)

This is the equation used to determine the amount of retained austenite from X-ray intensities.
APPENDIX 3

Correlation Between Charpy and CTS Data

The empirical relation obtained by Rolfe et al. 40

\[ K_{IC}^2 = 5 \sigma_y (CVN) - \frac{\sigma_y^2}{4} \]  \hspace{1cm} (3a)

The data used by Rolfe et al. to obtain this relationship is tabulated in Table 3A and plotted in Fig. 3A. However, there is reason to believe the correlation represented by Eq. (3a) is really not correct. Consider Fig. 3A. The three points which do not fall on the line drawn are all points which have very high \( K_{IC} \) values 40 and which have \( K_{IC}/\sigma_y \) ratios considerably greater than unity. The greatest specimen thickness used by Rolfe et al. for obtaining the data of Table 3A was 2 inches. Therefore they did not obtain \( K \) values even close to meeting \( K_{IC} \) requirements on these specimens. Rolfe et al. acknowledge this point but claim the these data satisfy an apparent \( K_{IC} \) criteria (without stating what the criteria is) which "appears to be valid on the basis of the general service experience with these steels." However, it is very likely that these \( K_{IC} \) values are not accurate. Assuming they are not then the correlation which more accurately represents the data of Table 3A is

\[ K_{IC}^2 = 480 \text{ CVN} \]  \hspace{1cm} (3b)
Fig. 3A. Plot of $K_{IC}^2 = 480$ cun, Eq. 3b. Data from Rolfe, et al. $^{40}$ and from this investigation are shown on this figure.
Table 3A. Mechanical properties of steels investigated for room temperature

\( K_{lc} - \text{CVN correlation. Taken from [40].} \)

<table>
<thead>
<tr>
<th>Steel and melting Practice</th>
<th>Yield (0.2% Offset), ksi</th>
<th>Tensile Strength, ksi</th>
<th>Elongation in 1 inch, %</th>
<th>Reduction of Area, %</th>
<th>Charpy V-Notch Energy Absorption at +80 F, ft-lb</th>
<th>( K_{lc} ) ksi/( \sqrt{\text{inch}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>A517-F, AM</td>
<td>110</td>
<td>121</td>
<td>20.0</td>
<td>66.0</td>
<td>62</td>
<td>170</td>
</tr>
<tr>
<td>4147, AM</td>
<td>137</td>
<td>154</td>
<td>15.0</td>
<td>49.0</td>
<td>26</td>
<td>109</td>
</tr>
<tr>
<td>HY-130, AM</td>
<td>149</td>
<td>159</td>
<td>20.0</td>
<td>68.4</td>
<td>89</td>
<td>246</td>
</tr>
<tr>
<td>4130, AM</td>
<td>158</td>
<td>167</td>
<td>14.0</td>
<td>49.2</td>
<td>23</td>
<td>100</td>
</tr>
<tr>
<td>12Ni-5Cr-3Mo, AM</td>
<td>175</td>
<td>181</td>
<td>14.0</td>
<td>62.2</td>
<td>32</td>
<td>130</td>
</tr>
<tr>
<td>12Ni-5Cr-3Mo, VIM</td>
<td>183</td>
<td>191</td>
<td>15.0</td>
<td>61.2</td>
<td>60</td>
<td>220</td>
</tr>
<tr>
<td>12Ni-5Cr-3Mo, VIM</td>
<td>186</td>
<td>192</td>
<td>17.0</td>
<td>67.1</td>
<td>65</td>
<td>226</td>
</tr>
<tr>
<td>18Ni-8Co=3Mo (200 Grade), AM</td>
<td>193</td>
<td>200</td>
<td>12.5</td>
<td>48.4</td>
<td>25</td>
<td>105</td>
</tr>
<tr>
<td>18Ni-8Co-3Mo (200 Grade), AM</td>
<td>190</td>
<td>196</td>
<td>12.0</td>
<td>53.7</td>
<td>25</td>
<td>112</td>
</tr>
<tr>
<td>18Ni-8Co-3Mo (190 Grade), VIM</td>
<td>187</td>
<td>195</td>
<td>15.0</td>
<td>65.7</td>
<td>49</td>
<td>160</td>
</tr>
<tr>
<td>18Ni-8Co-3Mo (250 Grade), VIM</td>
<td>246</td>
<td>257</td>
<td>11.5</td>
<td>53.9</td>
<td>16</td>
<td>87</td>
</tr>
</tbody>
</table>

* AM signifies electric-furnace air-melted; VIM signifies vacuum-induction-melted.
This is the equation of the line drawn in Fig. 3A.

There are also some theoretical reasons for questioning Eq. (3a). Let us adopt the development of Rice et al.\textsuperscript{38} $K_{IC}$ and $J_{IC}$ are related by

$$J_{IC} = \frac{K_{IC}^2}{E} = \frac{2}{b} \int_{0}^{f_{lc}} Pd\delta \ , \quad (3c)$$

where $\delta$ is the displacement of the load point and $\delta_c$ is the displacement of the load point at failure, $P$ is the load per unit thickness, and $b$ is the width of the uncracked material. If we assume CVN includes primarily the energy dissipated up to crack extension

$$CVN = \int_{0}^{\delta_{lc}} Pd\delta \ . \quad (3d)$$

Now Rice et al. also consider the elastic stored energy

$$E = \frac{B\sigma_y^2}{y} \ , \quad (3e)$$

where $B$ is a constant. This energy must be subtracted from the CVN. Thus,

$$K_{IC}^2 = A(CVN) - \frac{B\sigma_y^2}{y} \ . \quad (3f)$$

Figure 3B is a plot of this equation and shows that (i.e. zero intercept) under the assumptions taken $B\sigma_y^2$ is negligibly small and so we are left the task of evaluating $A$. From Eqs. (3c) and (3d)

$$K_{IC}^2 = \frac{2E}{bt} \int_{0}^{\sigma_{lc}} P'd\delta \ , \quad (3g)$$
where $t$ is the thickness at the charpy bar along the crack (or notch), and $P'$ is the actual load on the bar. Then,

$$A = \frac{2E}{bt}.$$  

(3h)

Thus, on this basis the constant $A$ should be independent of the yield stress.

None of the above proves that Eq. (3a) is not the correct way to describe the data of Rolfe et al. It does however point out the possibility of a theoretically more appealing correlation and it does illustrate the possible dangers of developing a correlation without a theoretical foundation. Thus it seemed best to use Eq. (3i) in this investigation to relate $K_{IC}$ and CVN.
APPENDIX 4

Estimation of Effective Nickel Contents of Fe-Ni-Ti Alloys

In the present investigation, effective nickel content of an alloy was defined as the total nickel minus the nickel content removed in the form of Ni$_3$Ti. For a first approximation, all the titanium content in the alloys was assumed to precipitate out as Ni$_3$Ti. The effective nickel contents calculated on the basis of this assumption are as below:

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Effective Nickel (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fe-12Ni-1Ti</td>
<td>8.3</td>
</tr>
<tr>
<td>Fe-12Ni-2Ti</td>
<td>4.6</td>
</tr>
<tr>
<td>Fe-16Ni-1Ti</td>
<td>12.3</td>
</tr>
<tr>
<td>Fe-16Ni-1.5Ti</td>
<td>10.5</td>
</tr>
<tr>
<td>Fe-20Ni-1.5Ti</td>
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