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SUMMARY

An investigation was undertaken to evaluate thermomechanical processing (TMP) as a method of strengthening the normally tough iron - 12-percent-nickel - reactive metal alloys at cryogenic temperatures. Laboratory size heats were thermomechanically processed by rolling at either 25° , 650° , or 1100° C followed by annealing over the temperature range of 450° to 820° C. Five iron-12 nickel alloys with reactive metal additions of aluminum, niobium, titanium, vanadium, and a combination of aluminum plus niobium were investigated. Primary evaluation was based on the -196° C yield strength and K_{Icd} fracture toughness of the thermomechanically processed alloys. Metallographic and fractographic examinations served as supplementary evaluation of the effects of the various TMP treatments on the five alloy systems.

Results from this study showed that TMP can be used as a method of strengthening these tough iron-12 nickel-reactive metal alloys. A -196° C yield strength of 1.3 GPa (187 ksi) with a corresponding K_{Icd} toughness of 243 MPa$\sqrt{m}$ (221 ksi$\sqrt{in.}$) was achieved with an iron-12 nickel-0.5 aluminum alloy which had undergone a TMP treatment consisting of rolling at 25° C plus subsequent annealing in the temperature range of 525° to 625° C. This represents the highest combination of cryogenic strength and toughness observed in iron-nickel alloys. The high strength with a corresponding high toughness is unique to this particular alloy and TMP treatment and is attributed to the fine, equiaxed substructure that resulted from TMP.

Thermomechanical processing (TMP) was effective in strengthening the other alloys of this study with the as-rolled 25° C material exhibiting superior strength, but at a corresponding reduction in toughness. For example, a yield strength equal to 1.5 GPa with a corresponding toughness equal to 167 MPa$\sqrt{m}$ was achieved in an iron-12 nickel-2 vanadium TMP alloy rolled at 650° C. In other cases, TMP was shown to be effective in producing a good combination of toughness and strength at corresponding high levels, but not as high as for the Fe-12Ni-0.5Al alloy. For example, a TMP treatment consisting of rolling an iron-12 nickel-0.25 titanium alloy at 25° C and annealing it at 550° C produced an alloy whose strength was 1.1 GPa and whose toughness was 171 MPa$\sqrt{m}$ at -196° C.

Scanning electron microscopy (SEM) of fractured surfaces revealed that dimpled rupture characterized the fracture behavior of the tougher alloys. Metallographic examination revealed that a heavily worked, fine grain structure characterized the stronger alloys.

INTRODUCTION

Nickel-containing steels have found wide acceptance as structural members in cryogenic service applications. For example, in the transporting and storage of liquified natural gas (which boils at -160° C), type 304 stainless steel, 9 nickel steel, and 5 nickel steel are currently being used (ref. 1). Type 304 stainless steel (which contains 8 percent nickel[1]) is a high-toughness, low-strength alloy while the strengths of the 5 and 9 nickel steels are somewhat higher, but at a sacrifice in toughness. Similarly a 9 nickel-4 cobalt steel and an 18 nickel, 200 grade maraging steel have even higher strengths, but also at a further sacrifice in toughness.

Previous research on iron-base alloys indicated that high toughness at cryogenic temperatures could be achieved in an iron-12 nickel-0.2 titanium (Fe-12Ni-0.2Ti) alloy at strength levels slightly lower than the 9 nickel steel (ref. 2). Toughness could be increased further in this alloy at the same strength level by employing a thermal cycling heat treatment which was shown to be effective as a result of grain refinement (ref. 3). More recently, an experimental program was conducted at the NASA Lewis Research Center to identify a single iron-base alloy that would combine the apparent divergent properties of high toughness and high strength. Specific goals of the program were to achieve a yield strength of 1.4 GPa (200 ksi) with a corresponding K_{Icd} fracture toughness of 220 $MPa\sqrt{m}$ (200 $ksi\sqrt{in.}$) at -196° C. The initial results of this work (ref. 4) identified several reactive metal additions that improve the -196° C toughness of iron-12 nickel (Fe-12Ni) alloy without the need for complex grain-refining heat treatments. Furthermore, it was shown for Fe-12Ni alloys with reactive metal additions of aluminum or titanium (ref. 5) that the cryogenic toughness of weld metal and heat affected zone (HAZ) metal was comparable to the base metal. Although exceptional cryogenic toughness was achieved, yield strengths typically ranged from 0.9 to 1.1 GPa, or about 65 to 70 percent of the 1.4 GPa strength goal (ref. 6).

The purpose of the program reported herein was to investigate strengthening of the Fe-12Ni-reactive metal alloys by thermomechanical processing (TMP) to achieve the strength goal of 1.4 GPa and still maintain high toughness at cryogenic temperatures. Five of the Fe-12Ni-reactive metal alloys investigated in the initial program (ref. 4) were chosen for this investigation. Reactive metals included aluminum (Al), niobium (Nb), titanium (Ti), vanadium (V), and a combination of aluminum plus niobium. Thermomechanical processing (TMP) included three rolling temperatures followed by several annealing treatments. Rolling temperatures were selected within the single-phase austenite and ferrite regions and within the two-phase austenite plus ferrite region of the iron-nickel phase diagram. Primary evaluation consisted of determining the effects of

[1]Commercial alloy compositions are expressed in weight percent while experimental alloys in this and previous reports are expressed in atomic percent.

TMP on strength and toughness of the alloys at -196° C. Supplemental evaluation included metallography, scanning electron microscopy (SEM), and transmission electron microscopy (TEM).

EXPERIMENTAL PROCEDURE

Materials

The Fe-12Ni alloys used in this investigation were prepared from vacuum-processed iron rods of 99.95 weight percent purity, and electrolytic nickel chips containing less than 100 ppm (by weight) total interstitial impurities. The reactive metals aluminum and niobium contained less than 100 ppm (by weight) total oxygen (O), nitrogen (N), and carbon (C), while the reactive metals titanium and vanadium contained less than 5000 ppm interstitial impurities. Table I lists the nominal and analyzed compositions of the five alloy systems investigated including the interstitial analyses.

Ingots were prepared by nonconsumable arc melting of 1 kilogram charges in a 7.5- by 7.5- by 3-centimeter-deep water-cooled copper mold after evacuating and backfilling with argon to one-half atmospheric pressure. To adequately homogenize the ingots, each was given a minimum of four melts.

Thermomechanical Processing

Ingots were rolled at three different temperatures to study the effects of thermomechanical processing (TMP) on strength and toughness. Hot rolling at 1100° C which is in the austenite region for an Fe-12Ni alloy was used as a standard rolling treatment for the initial studies (refs. 4 and 5) and was selected as one of the rolling temperatures in this program. The two additional rolling temperatures were 650° C which is in the two-phase austenite plus ferrite region for the Fe-12Ni alloy and cold rolling at 25° C which is in the ferrite region. All of the initial 25.4-millimeter thick ingots were heated for 1/2 hour at 1100° C and rolled at that temperature to a 20 percent reduction to break up the cast structure. Final rolling at 1100° and 650° C was accomplished by 15 to 20 percent reductions per pass and two rolling passes per 5-minute reheat until a thickness of about 7 millimeters was obtained. The cold rolled ingot was initially rolled at 20 percent reductions per pass, but due to work hardening final rolling passes were decreased to about 10 percent reduction per pass. Test specimens were machined from the rolled plate and annealed in an argon atmosphere for 2 hours at temperatures ranging from 450° to 820° C followed by water quenching.

Evaluation

Mechanical tests. - Tensile specimens were machined from the rolled plate with an overall length (parallel to the rolling direction) of 50 millimeters, an overall diameter of 6 millimeters, a reduced gage diameter of 3.7 millimeters, and a gage length of 25 millimeters. Specimens were tested in a liquid nitrogen bath at a constant crosshead speed of 1.3 millimeters per minute. The 0.2 percent offset yield strength was determined from the load-deflection curves.

Slow-bend test specimens were used to determine the K_{Icd} fracture toughness of the thermomechanical processed material. Test specimens were 50- by 10- by 6.4-millimeter notched Charpy bars oriented longitudinally in the sheet bar with a 45^{o} notch across the thickness (L-T crack plane orientation (ref. 7)). Specimens were fatigue cracked at room temperature (stress intensity increment ΔK less than 20 $MPa\sqrt{m}$) to an initial ratio of crack length to specimen width a/W of approximately 0.4. Testing was conducted in a three-point bending apparatus in a liquid nitrogen bath at a constant crosshead speed of 1.3 millimeters per minute. Details of the test apparatus have been described previously (refs. 4 and 5). The fracture toughness parameter K_{Icd} was determined from load-displacement curves by using the K_{Ic} equation for a slow-bend test specimen given in ASTM Standard E-399 (ref. 7) as modified by the empirical equivalent energy method (ref. 8). The relation used for calculating the fracture toughness of a slow-bend test specimen was

$$K_{Icd} = \frac{SP_2\left(\frac{A_1}{A_2}\right)^{1/2} f\left(\frac{a}{W}\right)}{BW^{3/2}}$$

where

S	span
P_2	any load on linear portion of load-displacement curve
A_1	area under curve to maximum load
A_2	area under curve to P_2
$f(a/W)$	value of power series for a/W (given in ref. 7)
B	specimen thickness

Microstructural studies. - Longitudinal sections of annealed bend bars were examined by light microscopy. An etchant consisting of 1 part by volume hydrofluoric acid, 33 parts nitric acid, and 33 parts acetic acid in 933 water was used to reveal the microstructure of the alloys. Fractured surfaces of the bend test specimens were examined by SEM. In addition, disks were sectioned from the longitudinal cross section of the bend test specimens, thinned to foils, and examined by TEM. Thinning was accomplished using a solution of 20 parts sodium dichromate and 80 parts acetic acid at an operating potential of about 25 volts and an operating temperature of 10^{o} C.

RESULTS

Iron-12 Nickel-0.5 Aluminum Alloys

The effects of TMP on strength and toughness of the iron-12 nickel-0.5 aluminum (Fe-12Ni-0.5Al) alloys are summarized in table II. Yield strength and K_{Icd} fracture toughness are plotted in figure 1 for each of the three rolling treatments for specimens in the as-rolled condition and subsequently annealed over the 450^{o} to 820^{o} C temperature range. The 25^{o} C rolled material exhibits an as-rolled yield strength of 1.72 GPa compared to 1.13 GPa for the 1100^{o} C rolled material and only 0.903 GPa for the 650^{o} C rolled material. However, the as-rolled toughness for the 650^{o} C rolled material was 248 MPa$\sqrt{m}$ compared to 119 MPa$\sqrt{m}$ for the cold rolled material and only 80 mPa$\sqrt{m}$ for the 1100^{o} C rolled alloy. It is seen from these results that in the as-rolled condition high strength is accompanied by low toughness for the 25^{o} C TMP technique while the converse was true for the 650^{o} C TMP technique.

Subsequent annealing of the 25^{o} C thermomechanically processed alloy resulted in a yield strength that was essentially independent of annealing temperature with the yield strength averaging 1.36 GPa or 97 percent of the goal set for this program. Toughness for the 25^{o} C thermomechanically processed alloy reached a maximum of 243 MPa$\sqrt{m}$ after annealing at 550^{o} C. Toughness exceeded the goal of 220 MPa$\sqrt{m}$ over an annealing temperature range of about 525^{o} to 625^{o} C. The combination of high strength and high toughness for the 25^{o} C thermomechanically processed alloy illustrates the ability of this TMP technique to simultaneously improve these normally divergent properties.

Maximum toughness for the thermomechanically processed Fe-12Ni-0.5Al alloys was achieved for the 650^{o} C thermomechanically processed alloy annealed at 500^{o} C where a toughness of 265 MPa$\sqrt{m}$ was achieved with a corresponding yield strength of 0.924 GPa. Strength reached a maximum of about 1.25 GPa for the 650^{o} and 1100^{o} C thermomechanically processed alloys annealed at 685^{o} C, but toughness was only about 66 MPa$\sqrt{m}$ for these TMP techniques. Although the 650^{o} and 1100^{o} C thermomechanically processed alloys did not produce a combination of strength and toughness equal to the goals of this program, a combination of good strength and toughness could be achieved in the alloys. For example, a TMP technique of 650^{o} C rolling and 625^{o} C annealing resulted in a strength of 1.09 GPa and a toughness of 199 MPa$\sqrt{m}$ while the

1100° C rolled material annealed at 630° C exhibited a yield strength of 0.965 GPa and a toughness of 235 MPa$\sqrt{m}$.

The high strength achieved in the 25° C thermomechanically processed alloy can be attributed to the microstructure developed in this alloy during the cold rolling process, as illustrated in figure 2. In the as-rolled condition, figure 2(a), the microstructure is seen to be characterized by a very fine grain structure with grains elongated parallel to the rolling direction. After annealing at 550° C, very little reorientation of the structure has occurred (fig. 2(b)), and the microstructure still exhibits a very fine ferritic grain structure. Even after annealing within the two-phase ferrite-austenite region at 685° C, the resulting ferrite-martensite grain structure is extremely fine with an average grain size of about 5 micrometers, as shown in figure 2(c). During annealing at 820° C, grain coarsening occurs and the microstructure is characterized by a martensitic lath structure, as shown in figure 2(d). This thermomechanically processed alloy was also examined by TEM and the results are shown in figure 3. The as-rolled microstructure (fig. 3(a)) is characterized by a very thin martensitic lath structure with the laths parallel to the rolling direction and exhibiting waviness along their length. The laths typically range from 0.2 to 0.02 micrometer in thickness with their lengths extending across the foil. The fine width of the lath martensite is believed to account for the high strength observed in the as-rolled alloy. After annealing at 550° C, a ferrite substructure was developed as shown in figure 3(b). The equiaxed subgrains were typically 0.5 micrometer in diameter, which is believed to account for the high strength that is maintained in this thermomechanically processed alloy. Annealing at 820° C resulted in a martensitic lath structure with a high dislocation density at lath boundaries and within the laths as shown in figure 3(c). Metallographic examination of the 650° C thermomechanically processed alloy, shown in figure 4(a) suggests that the structure is primarily ferrite with grains elongated parallel to the rolling direction, but with a larger grain width than was observed for the 25° C thermomechanically processed alloy. The 1100° C thermomechanically processed alloy was characterized by an equiaxed, martensitic lath structure as shown in figure 4(b).

It should be noted in figure 1(b) that the toughness of the thermomechanically processed alloys converged at an annealing temperature of 550° C and beyond a temperature of 625° C exhibited a dramatic drop. Examination of fracture surfaces by SEM revealed that fracture of alloys exhibiting high toughness occurred by microvoid coalescence leading to a dimpled fracture surface while lower toughness materials exhibited cleavage and grain boundary separation. The fracture surfaces of the 1100° C thermomechanically processed alloy are shown in figure 5 as an example of this behavior. The low toughness, as-rolled alloy is shown in figure 5(a) where fracture occurred primarily by cleavage with some dimpled fracture. After annealing at 550° C which produced maximum toughness in this alloy, fracture was entirely by dimpled failure. Annealing at 685° or 820° C resulted in failure primarily by grain boundary separation as shown in figures 5(c) and (d), respectively. These results for the annealed conditions were typical for all three thermomechanically processed Fe-12Ni-0.5Al alloys. The as-rolled 25° C thermomechanically processed alloy failed by alternate layers of cleavage failure and dimpled rupture as shown in figure 6(a) at a low magnification. Each of the two zones are shown at a higher magnification in figures 6(b) and (c). The fracture surface of the 650° C thermomechanically processed as-rolled alloy shown in figure 7 is caused entirely by dimpled failure corresponding to the high toughness of this thermomechanically processed alloy.

Iron-12 Nickel-0.25 Niobium Alloys

The effects of TMP on strength and toughness of the niobium-bearing alloys are summarized in table III and shown in figure 8. Highest strength is achieved in this alloy series as a result of the 25° C TMP treatment for specimens in both the as-rolled and subsequently annealed conditions. The 650° and 1100° C treatments exhibited similar strengths under all conditions and approached the strength of the 25° C thermomechanically processed alloy after annealing at 685° and 820° C. Exceptionally high toughness was achieved in the 650° C TMP alloy, as shown in figure 8(b), where toughness ranged from 230 $MPa\sqrt{m}$ for the as-rolled condition to a maximum of 303 $MPa\sqrt{m}$ upon annealing at 685° C. The 25° and 1100° C thermomechanically processed alloys exhibited similar toughness properties in all annealed conditions. The optimum TMP treatment for the iron-12 nickel-0.25 niobium (Fe-12Ni-0.25Nb) alloy to combine high strength and high toughness is the 650° C rolled condition annealed at 500° or 550° C. Yield strengths ranged from 71 to 76 percent of the strength goal with a corresponding toughness ranging from 110 to 135 percent of the toughness goal set for this program.

Metallographic examination of the 650° C thermomechanically processed alloy annealed at 550° C revealed a microstructure with grains lying parallel to the rolling direction as shown in figure 9(a). Examination of fracture surfaces of bend test specimens by SEM indicated that failure was by microvoid coalescence in this tough alloy leading to dimpled rupture as illustrated in figure 9(b). Typical of the Fe-12Ni-0.25Nb alloy is the TEM micrograph in figure 9(c) where the lath widths range from 0.2 to 1.0 micrometer and their lengths extend across the foil.

Iron-12 Nickel-0.25 Titanium Alloys

Yield strength and toughness results are summarized in table IV and shown in figure 10 for the thermomechanically processed iron-12 nickel-0.25 titanium (Fe-12Ni-0.25Ti) alloys. The yield strength of the as-rolled 25° C thermomechanically processed alloy was in excess of the 1.4 GPa yield strength set for this program; however, the corresponding toughness of this alloy was only 136 MPa$\sqrt{m}$, well below the 220 MPa$\sqrt{m}$ toughness goal. The 25° C rolled and annealed thermomechanically processed alloy exhibited decreasing strength with increasing annealing temperature. The 650° and 1100° C thermomechanically processed alloys had very similar strengths in the as-rolled and annealed conditions and exhibited only a minor variation in strength with increase in annealing temperature. The average strength of these two thermomechanically processed alloys was 0.95 GPa or only about two-thirds of the strength goal. The toughness of all three Fe-12Ni-0.25Ti alloys was very sensitive to the TMP treatments, exhibiting a maximum at a different annealing temperature for each of the three rolling procedures. Toughness in excess of the 220 MPa$\sqrt{m}$ goal of this program was achieved for each of the three rolled conditions after several different annealing treatments.

No single TMP treatment produced high strength with corresponding high toughness in the Fe-12Ni-0.25Ti alloys. The 25° C rolled, 550° C annealed thermomechanically processed alloy exhibited 78 percent of both strength and toughness goals. The effect of rolling temperature combined with a 550° C annealing temperature on microstructure is shown in figure 11 for the thermomechanically processed Fe-12Ni-0.25Ti alloys. The 25° C rolled alloy exhibited a very fine martensitic lath width, averaging around 0.1 to 0.3 micrometer. The lath width of the 650° and 1100° C rolled alloys was about 1.0 micrometer; however, the 650° C TMP treatment produced a more equiaxed structure while the length of the 1100° C thermomechanically processed alloy laths extended for several micrometers. The high toughness of the 650° C thermomechanically processed alloy (301 MPa$\sqrt{m}$) is thought to arise in part because of the equiaxed structure developed in this alloy. The small lath width of the 25° C thermomechanically processed alloy may account for the somewhat higher strength for this TMP treatment.

Iron-12 Nickel-2 Vanadium Alloys

The effects of TMP on the iron-12 nickel-2 vanadium (Fe-12Ni-2V) alloys are shown in figure 12 and summarized in table V. Maximum strength was achieved in the as-rolled condition for all three rolling temperatures with the 25° and 650° C thermomechanically processed alloys exceeding the 1.4 GPa strength goal. A minimum in strength was observed after annealing at 600° C for all three thermomechanically processed alloys. It also should be noted that over the 550° to 820° C temperature range there was only a slight variation in strength with annealing temperature and the 25° and 650° C thermomechanically processed alloys exhibited very similar strength properties, both slightly higher than the 1100° C thermomechanically processed alloy. Corresponding toughnesses for these alloys were generally lower in the as-rolled condition and after annealing over the 500° to 600° C temperature range. All the thermomechanically processed alloys annealed over the 685° to 820° C temperature range exhibited a toughness in excess of the 220 MPa$\sqrt{m}$ goal.

Although no single TMP treatment produced an alloy that met both strength and toughness goals, several of the thermomechanically processed alloys did have an attractive combination of strength and toughness properties. For example, the 650° C thermomechanically processed alloy in the as-rolled condition had a strength equal to 105 percent of the strength goal with a corresponding toughness equal to 76 percent of the 220 MPa$\sqrt{m}$ goal. This alloy annealed at 820° C possessed a strength equal to 80 percent of the 1.4 GPa goal with a corresponding toughness of 128 percent of the toughness goal. All three thermomechanically processed alloys annealed at 820° C possessed similar strength and toughness characteristics. The highest toughness for the iron-12 nickel-2 vanadium alloy occurred in the 1100° C rolled-685° C annealed condition where toughness was 131 percent of goal and strength 74 percent of goal.

Metallographic examination of the high-strength, as-rolled 25° and 650° C thermomechanically processed alloys revealed a typical worked structure, as shown in figure 13(a) for the 25° C thermomechanically processed alloy. After annealing at 820° C, a martensitic lath structure was produced as shown in figure 13(b). The relatively fine grain size of this alloy (about 10 micrometers) may account in part for the combination of good strength and toughness for this TMP treatment. The 25° and 650° C rolled alloys, annealed at 685° C exhibited a two-phase structure with the martensite laths being very small, as shown in figures 13(c) and (d). The 1100° C rolled, 685° C annealed alloy developed a more typical martensite-ferrite structure (fig. 13(e)) characteristic of annealing within the two-phase austenite-ferrite region.

Iron-12 Nickel-0.5 Aluminum-0.25 Niobium Alloys

A summary of the strength and toughness data for the iron-12 nickel with two reactive metal additions is given in table VI. The yield strength data shown in figure 14 indicates that strength of this alloy was not strongly dependent on rolling temperature nor annealing temperature. Toughness tended to increase with increasing annealing temperature for the 25° and 650° C thermomechanically processed alloys. For the 1100° C alloy, toughness reached a maximum after annealing at 685° C. Comparison of these results for the combined reactive metals with the individual results shown in figure 1 for the aluminum addition and in figure 8 for the niobium addition indicates that the effects of TMP are very similar to those observed for the Fe-12Ni-0.25Nb alloy. The high strength achieved in the 25° C thermomechanically processed Fe-12Ni-0.5Al alloy was not achieved in the alloy with aluminum combined with niobium, but rather was at a level and possessed a temperature dependence similar to the results obtained for the Fe-12Ni-0.25Nb alloy. The high toughness achieved upon annealing the alloy with aluminum plus niobium above 650° C was also observed in the alloy containing only niobium and in contrast to the alloy with only aluminum as the reactive metal where a severe drop in toughness was noted.

Metallographic examination of the iron-12 nickel-0.5 aluminum-0.25 niobium (Fe-12Ni-0.5Al-0.25Nb) alloys revealed microstructures similar to those observed for other alloys in this study. For example, the 25° and 650° C thermomechanically processed alloys in the as-rolled and 550° C annealed conditions exhibited a worked microstructure. After annealing at 685° C, the microstructures shown in figure 15 are noted to be quite similar to those observed in the Fe-12Ni-2V alloy (fig. 13) and the Fe-12Ni-0.25Nb alloys.

DISCUSSION

Strength and Toughness

Thermomechanical processing was employed in this study as a means of increasing the strength of Fe-12Ni-reactive metal alloys without sacrificing toughness. A specific goal of the program was to achieve a -196° C yield strength of 1.4 GPa (200 ksi) with a corresponding K_{Icd} toughness of 220 MPa$\sqrt{m}$ (200 ksi$\sqrt{in.}$). Results showed that this goal was essentially reached in the Fe-12Ni-0.5Al alloy which had undergone a TMP treatment involving rolling at 25° C followed by annealing over the temperature range 525° to 625° C. Results further showed that a thermomechanical process involving rolling at 25° C produced a yield strength in excess of the 1.4 GPa goal for the Fe-12Ni-0.5Al, Fe-12Ni-0.25Ti, and Fe-12Ni-2.0V alloys, but at the expense of a lower toughness (see figs. 1, 10, and 12).

The general effects of TMP on the Fe-12Ni alloys was to produce a high yield strength in the as-rolled condition which decreased gradually with increase in subsequent annealing temperatures. The effects of TMP on toughness were the opposite, where as-rolled material in general exhibited lower toughness values which increased with subsequent annealing temperatures up to 820° C. The exception to these general observations, was the Fe-12Ni-0.5Al alloy system, where toughness increased up to an annealing temperature of 550° C which resulted in a combination of high yield strength and high toughness which served as the original impetus for this program.

Microstructure and Fracture

The general effects of TMP on microstructure were to produce a heavily worked, fine grain structure for the 25° C rolling which contributed to the high yield strengths for this condition. Rolling at 650° C produced a similar worked structure, but with grain widths larger than for the 25° C rolled material, while rolling at 1100° C resulted in an equiaxed grain structure. Subsequent annealing within the single-phase α region had only a minor effect on the structure of all three rolled conditions, notably, grain coarsening. Annealing of the alloys within the two-phase $\alpha + \gamma$ region resulted in a rearrangement of the microstructure. For the 25° and 650° C thermomechanically processed alloys, a very fine distribution of martensite within the ferrite matrix resulted while for the 1100° C thermomechanically processed alloys martensitic laths extended across prior austenite grains. Annealing of the 25°, 650°, and 1100° C thermomechanically processed alloys within the single-phase γ region produced a microstructure of large martensite laths bounded by prior austenite grain boundaries.

Fractures of the TMP alloys were characterized primarily by grain boundary separation for those alloys with a toughness less than around 60 $MPa\sqrt{m}$. As toughness increased the fracture mode changed to a mixture of cleavage and dimpled rupture, typically characterizing alloys with toughnesses ranging from 80 to 200 $MPa\sqrt{m}$. A third type of fracture was almost total dimpled rupture for alloys exhibiting high toughness in excess of 200 $MPa\sqrt{m}$.

Controlling Mechanisms

Metallographic and fractographic examinations have helped to elucidate the response of strength and toughness of the Fe-12Ni alloys to thermomechanical processing. High strength was achieved in all alloys containing a worked, fine grained structure such as existed for the 25° and 650° C rolled thermomechanically processed alloys. However, the long, straight sub-boundaries such as those shown by TEM (fig. 3(a)) for the Fe-12Ni-0.5Al alloy may provide a path for easy crack propagation leading to a corresponding low toughness. A more equiaxed structure which developed upon annealing the Fe-12Ni alloys with reactive metal additions of Nb, Ti, V, or Al plus Nb in the γ region or a fine dispersion of the martensite phase upon annealing in the $\alpha + \gamma$ region was shown to improve toughness; however, overall coarsening of the structure led to a reduction in strength from the as-rolled condition. In contrast to this annealing response of high toughness and lower strength, the combination of good toughness and strength achieved in the 25° C thermomechanically processed Fe-12Ni-0.5Al alloy upon annealing over the temperature range 525° to 625° C is attributed to the fine-equiaxed substructure developed in this alloy, for example, figure 3(b) for a 550° C anneal TMP treatment. The fine equiaxed structure appears to be unique to this TMP treatment and is expected to result in transverse properties similar to the properties obtained herein upon testing parallel to the rolling direction. In contrast, the worked structure with elongated grains parallel to the rolling direction is expected to exhibit lower mechanical properties in the transverse direction.

The reduction in toughness for the Fe-12Ni-0.5Al alloy at the higher annealing temperatures for all three thermomechanically processed rolling conditions cannot be explained directly by the structures developed in this alloy system since the structures are similar to those developed in the other four alloy systems. Rather, these results are thought to be due to the interstitial content of the Fe-12Ni-0.5Al alloys, especially carbon content. Previous results by the authors (ref. 4) on Fe-12Ni-0.5Al alloy containing only 158 ppm carbon showed that toughness was in excess of 200 MPa$\sqrt{m}$ upon annealing a 1100° C rolled alloy at 685° or 820° C. In contrast, the current 1100° C rolled alloy (as well as the 25° and 650° C rolled alloy) with 286 ppm carbon or higher exhibited toughnesses less than 100 MPa$\sqrt{m}$ after similar annealing treatments. The lower toughness in this study may be due to the higher carbon content of these alloys. Due to the low free energy of formation of aluminum carbide (Al_4C_3), which is less than -15 kcal/g atom (ref. 9), the reactive metal may not react with the carbon in these alloys. Carbon or iron carbides may be present at prior austenite grain boundaries upon annealing in the $\alpha + \gamma$ region or γ region leading to reduced toughness and brittle grain boundary failure as was observed in this study. Lowering the carbon content as was done in the previous program should help improve toughness at the higher annealing temperatures. The other reactive metals, Nb, Ti, and V have more than double the heats of formation with carbon, ranging from -28 kcal/g atom for VC to -43 kcal/g atom for TiC (refs. 9 and 10), which may explain the higher toughnesses observed for these alloy additions.

CONCLUSIONS

The following conclusions are drawn from this study on thermomechanical processing of Fe-12Ni-reactive metal alloys:

1. The normally divergent properties of high strength and high toughness at cryogenic temperatures can be combined in a single alloy by proper thermomechanical processing.

2. A 1.3 GPa (187 ksi) yield strength with a corresponding 243 MPa$\sqrt{m}$ (221 ksi$\sqrt{in.}$) K_{Icd} toughness at -196° C can be achieved in an Fe-12Ni-0.5Al alloy by thermomechanical processing involving rolling at 25° C followed by annealing over the temperature range 525° to 625° C. This represents the highest combination of cryogenic strength and toughness observed in Fe-Ni alloys.

3. The Fe-12Ni-0.5Al TMP alloy possesses unique cryogenic strength and toughness properties which are thought to be controlled primarily by the fine equiaxed substructure developed during the 25° C rolling - 525° to 625° C annealing treatments.

4. Thermomechanical processing can be used as a mechanism for increasing the

cryogenic strength of Fe-12Ni alloys with reactive metal additions of Nb, Ti, V, or Al plus Nb, but at a correspondingly lower toughness level.

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TABLE I. - CHEMICAL ANALYSIS OF Fe-12Ni ALLOYS

Alloy	Thermomechanically processed rolling temperature, °C	Analyzed composition, at. %		Interstitial content, ppm by weight		
		Nickel	Reactive metal	Carbon	Nitrogen	Oxygen
Fe-12Ni-0.5Al	25	12.0	0.46Al	373	27	10
	650	12.0	.46Al	334	48	10
	1100	12.1	.42Al	286	26	26
Fe-12Ni-0.25Nb	25	12.3	0.22Nb	175	19	8
	650	11.7	.23Nb	293	24	35
	1100	11.9	.22Nb	300	18	11
Fe-12Ni-0.25Ti	25	13.9	0.28Ti	336	11	8
	650	11.7	.25Ti	348	22	16
	1100	11.9	.17Ti	352	7	8
Fe-12Ni-2V	25	11.9	1.97V	191	16	9
	650	12.3	2.08V	181	10	10
	1100	12.0	1.99V	150	44	25
Fe-12Ni-0.5Al-0.25Nb	25	12.0	0.35Al-0.26Nb	23	13	41
	650	11.9	.37Al-0.26Nb	76	12	32
	1100	12.2	.42Al-0.21Nb	74	16	35

TABLE II. - SUMMARY OF MECHANICAL PROPERTY DATA

FOR Fe-12Ni-0.5Al ALLOYS

Thermomechanical processing		Yield strength (0.2% offset)		Ultimate tensile strength		Elongation, percent	Fracture toughness, K_{Icd}	
Rolling temperature, °C	2-Hour annealing temperature, °C	GPa	ksi	GPa	ksi		$MPa\sqrt{m}$	$ksi\sqrt{in.}$
25	As rolled	1.72	249	1.77	256	7	119	108
	500	1.30	188	1.30	188	18	179	163
	550	1.29	187	1.32	191	22	243	221
	625	1.29	187	1.49	216	17	218	198
	650	1.41	205	1.55	225	14	173	157
	685	1.35	196	1.49	216	11	70	64
	700	1.45	210	1.50	217	14	59	54
	750	1.34	194	1.46	212	14	52	47
	820	1.28	186	1.41	205	14	48	44
650	As rolled	0.903	131	1.23	178	22	248	226
	500	.924	134	1.19	173	21	265	241
	550	.855	124	1.19	173	21	235	214
	625	1.09	158	1.30	189	16	199	181
	650	1.24	180	1.37	199	14	159	145
	685	1.26	182	1.39	202	12	68	62
	750	1.20	174	1.35	196	15	53	48
	820	1.15	166	1.30	189	14	50	45
1100	As rolled	1.13	184	1.28	185	6	80	73
	450	1.11	161	1.11	161	13	215	196
	500	1.01	147	1.03	150	3	244	222
	550	.917	133	1.08	156	19	233	212
	630	.965	140	1.22	177	8	235	214
	685	1.21	175	1.32	191	12	64	58
	750	1.05	153	1.18	171	7	51	46
	820	1.02	148	1.17	170	11	43	39

TABLE III. - SUMMARY OF MECHANICAL PROPERTY DATA

FOR Fe-12Ni-0.25Nb ALLOYS

Thermomechanical processing		Yield strength (0.2% off-set)		Ultimate tensile strength		Elongation, percent	Fracture toughness, K_{Icd}	
Rolling temperature, °C	2-Hour annealing temperature, °C	GPa	ksi	GPa	ksi		MPa$\sqrt{m}$	ksi$\sqrt{in.}$
25	As rolled	1.26	183	1.32	191	9	160	146
	500	1.27	184	1.30	189	15	166	151
	550	1.17	169	1.21	176	18	168	153
	600	1.01	147	1.14	165	15	179	163
	685	1.00	145	1.15	166	16	256	233
	820	.929	134	1.03	150	14	266	242
650	As rolled	0.986	143	1.10	159	20	230	209
	500	1.05	152	1.08	157	18	242	220
	550	.986	143	1.08	156	22	296	269
	600	.889	129	1.05	153	21	289	263
	685	.965	140	1.08	157	18	303	276
	820	.924	134	1.04	151	15	297	270
1100	As rolled	1.00	145	1.09	158	11	210	191
	550	.717	104	.752	109	16	188	171
	600	.931	135	1.02	148	18	230	209
	685	.938	136	1.03	150	10	227	207
	820	.896	130	1.00	145	11	257	234

TABLE IV. - SUMMARY OF MECHANICAL PROPERTY DATA

FOR Fe-12Ni-0.25Ti ALLOYS

Thermomechanical processing		Yield strength (0.2% offset)		Ultimate tensile strength		Elongation, percent	Fracture toughness, K_{Icd}	
Rolling temperature, °C	2-Hour annealing temperature, °C	GPa	ksi	GPa	ksi		MPa$\sqrt{m}$	ksi$\sqrt{in.}$
25	As rolled	1.49	216	1.57	228	5	136	124
	500	1.35	196	1.37	198	7	146	133
	550	1.08	156	1.28	186	13	171	156
	600	1.02	148	1.15	167	15	173	157
	685	.965	140	1.08	157	19	266	242
	820	.958	139	1.09	158	17	193	176
650	As rolled	0.972	141	1.13	164	17	218	198
	500	1.01	147	1.08	157	18	233	212
	550	.958	139	1.10	159	18	301	274
	600	.903	131	1.07	155	18	238	217
	685	.917	133	1.04	151	17	251	228
	820	.945	137	1.09	158	15	277	252
1100	As rolled	1.03	149	1.12	162	12	266	242
	550	.965	140	1.01	147	17	193	176
	600	.883	128	.986	143	22	279	254
	685	.917	133	1.03	149	16	224	204
	820	.903	131	1.01	146	10	244	222

TABLE V. - SUMMARY OF MECHANICAL PROPERTY DATA

FOR Fe-12Ni-2V ALLOYS

Thermomechanical processing		Yield strength (0.2% offset)		Ultimate tensile strength		Elongation, percent	Fracture toughness, K_{Icd}	
Rolling temperature, °C	2-Hour annealing temperature, °C	GPa	ksi	GPa	ksi		$MPa\sqrt{m}$	$ksi\sqrt{in.}$
25	As rolled	1.45	210	1.49	216	21	136	124
	500	1.25	181	1.26	183	18	129	117
	550	1.06	154	1.18	171	22	149	136
	600	.927	141	1.13	164	25	174	158
	685	1.05	152	1.15	167	16	249	227
	820	1.10	160	1.19	173	14	263	239
650	As rolled	1.52	221	1.52	221	6	167	152
	500	1.07	155	1.16	168	28	181	165
	550	1.01	146	1.21	175	25	174	158
	600	.945	137	1.10	160	21	159	145
	685	1.03	149	1.14	165	17	241	219
	820	1.09	158	1.20	174	13	280	255
1100	As rolled	1.21	176	1.34	194	5	165	150
	550	1.01	146	1.07	155	21	112	102
	600	.876	127	1.03	149	16	190	173
	650	.924	134	1.03	150	10	210	191
	685	1.01	147	1.10	159	10	288	262
	710	.972	141	1.09	158	12	284	258
	735	1.02	148	1.11	161	10	284	258
	820	1.01	147	1.11	161	14	247	225

TABLE VI. - SUMMARY OF MECHANICAL PROPERTY DATA

FOR Fe-12Ni-0.5Al-0.25Nb ALLOYS

Thermomechanical processing		Yield strength (0.2% offset)		Ultimate tensile strength		Elongation, percent	Fracture toughness, K_{Icd}	
Rolling temperature, °C	2-Hour annealing temperature, °C	GPa	ksi	GPa	ksi		$MPa\sqrt{m}$	$ksi\sqrt{in.}$
25	As rolled	1.16	168	1.19	173	12	143	130
	500	1.16	168	1.17	170	9	163	148
	550	1.16	162	1.17	169	16	155	141
	600	1.00	145	1.10	159	19	182	166
	685	.986	143	1.12	162	16	201	183
	750	.972	141	1.10	159	15	222	202
	820	.938	136	1.04	151	17	240	218
650	As rolled	1.11	161	1.17	169	17	158	144
	500	1.15	167	1.18	171	18	166	151
	550	1.08	156	1.14	165	18	225	205
	600	1.00	145	1.10	160	13	251	228
	685	1.00	145	1.14	165	15	240	218
	750	.986	143	1.09	158	12	269	245
	820	.931	135	1.06	154	16	273	248
1100	As rolled	0.979	142	1.07	155	12	93	85
	550	1.04	151	1.06	154	15	149	136
	600	.896	130	.986	143	18	194	177
	650	.924	134	1.03	150	7	280	255
	685	.945	137	1.01	147	12	287	261
	750	.952	138	1.07	155	10	233	212
	820	.862	125	.924	134	13	254	231

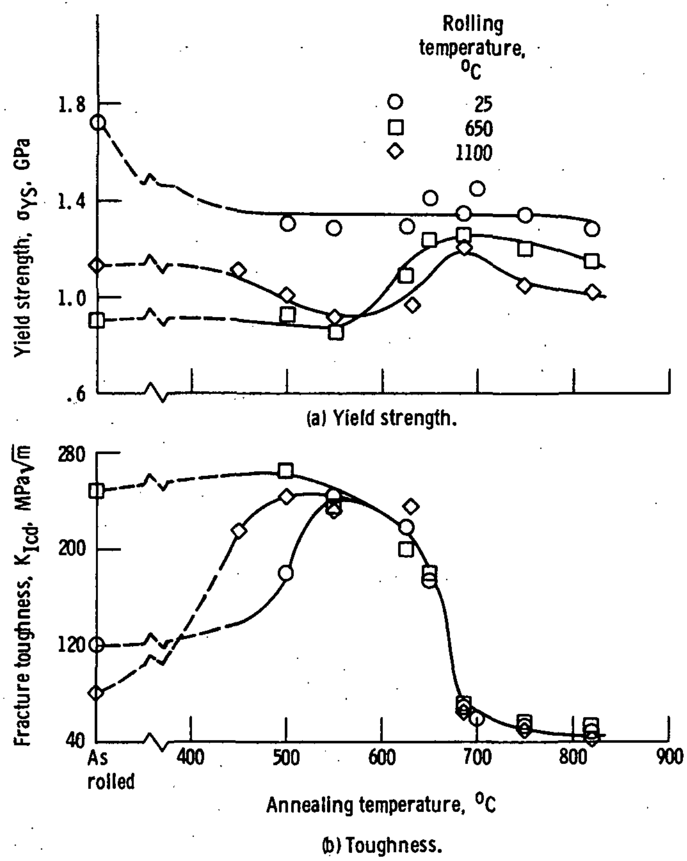
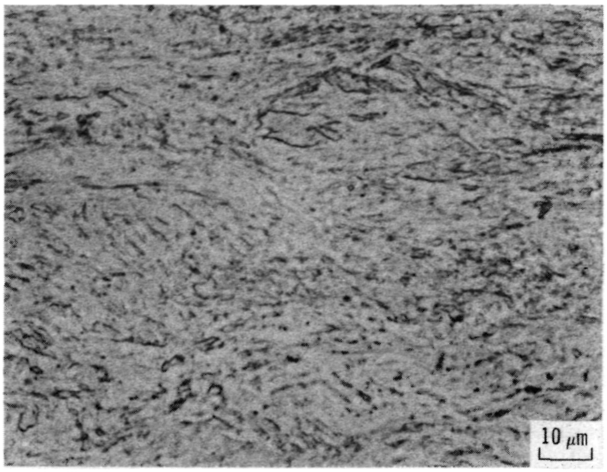
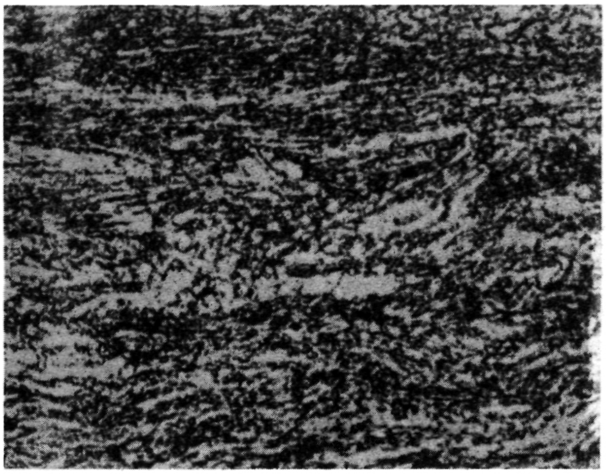


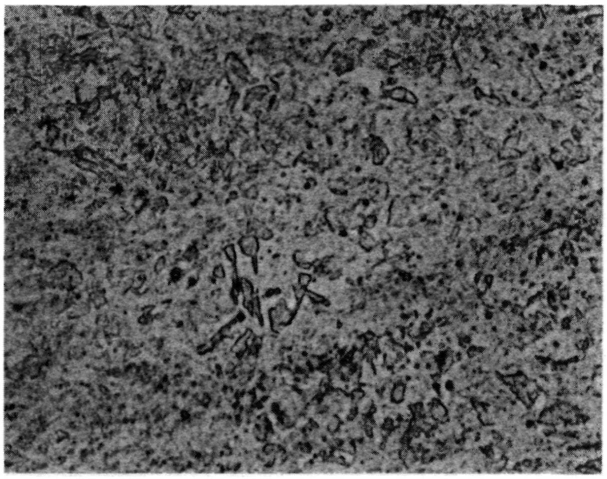
Figure 1. - Effects of thermomechanical processing on strength and toughness of Fe-12Ni-0.5Al alloys at -196° C.



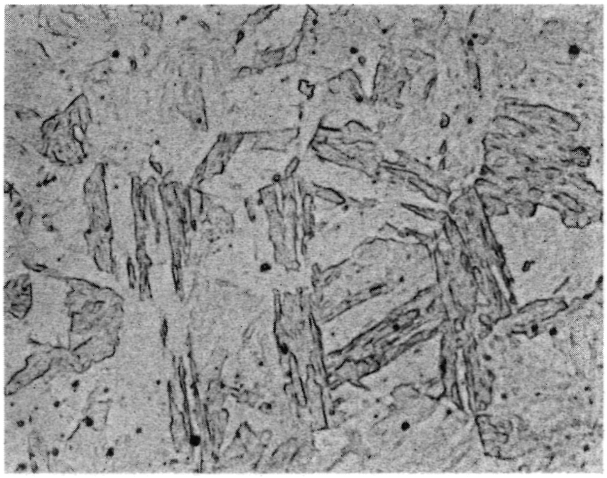
(a) As rolled.



(b) 550° C anneal.



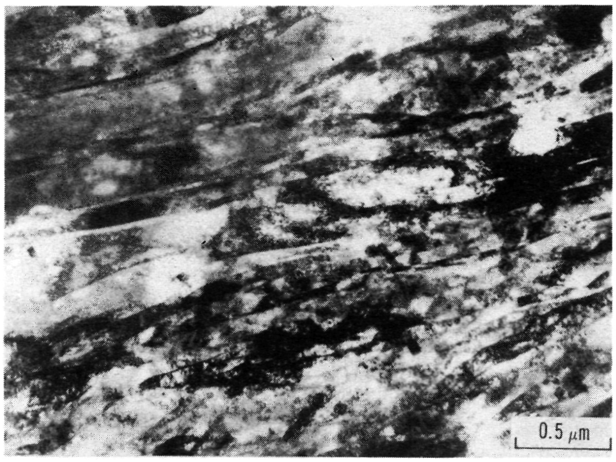
(c) 685° C anneal.



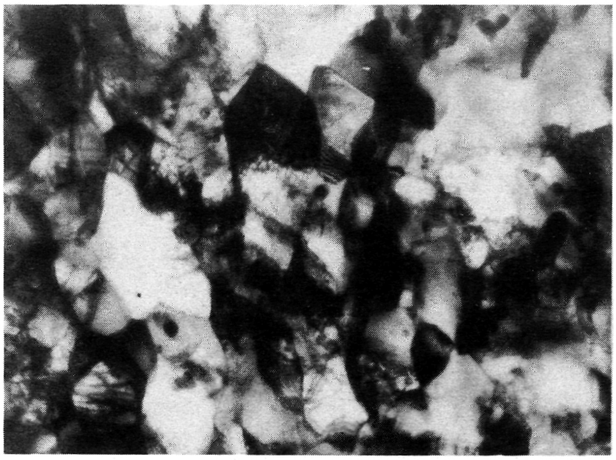
(d) 820° C anneal.

Rolling direction

Figure 2. - Microstructures of 25° C thermomechanically processed Fe-12Ni-0.5Al alloys.



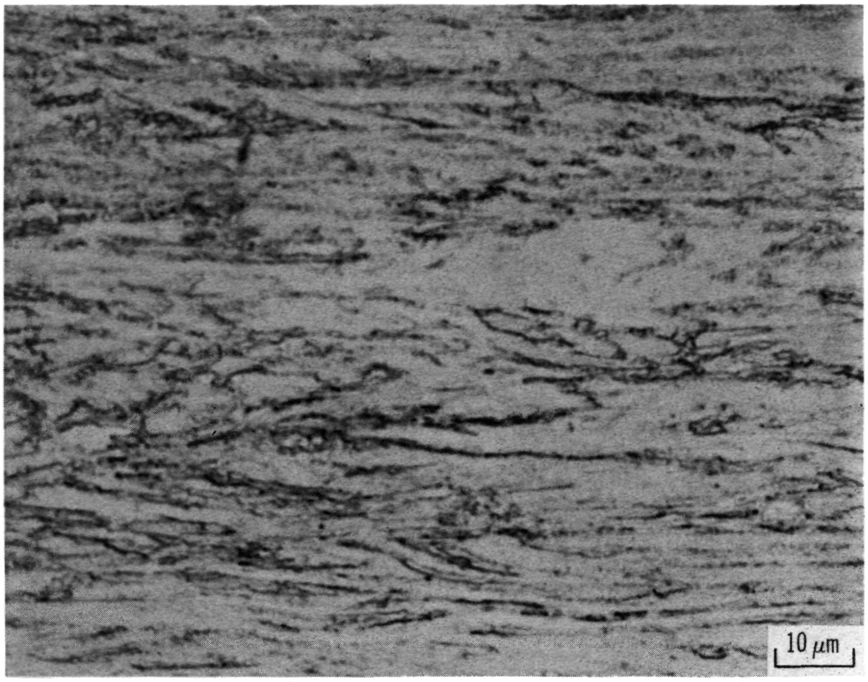
(a) As rolled.



(b) 550° C anneal.

(c) 820° C anneal.

Figure 3. - Substructure developed in 25° C thermomechanically processed alloy as revealed by TEM.

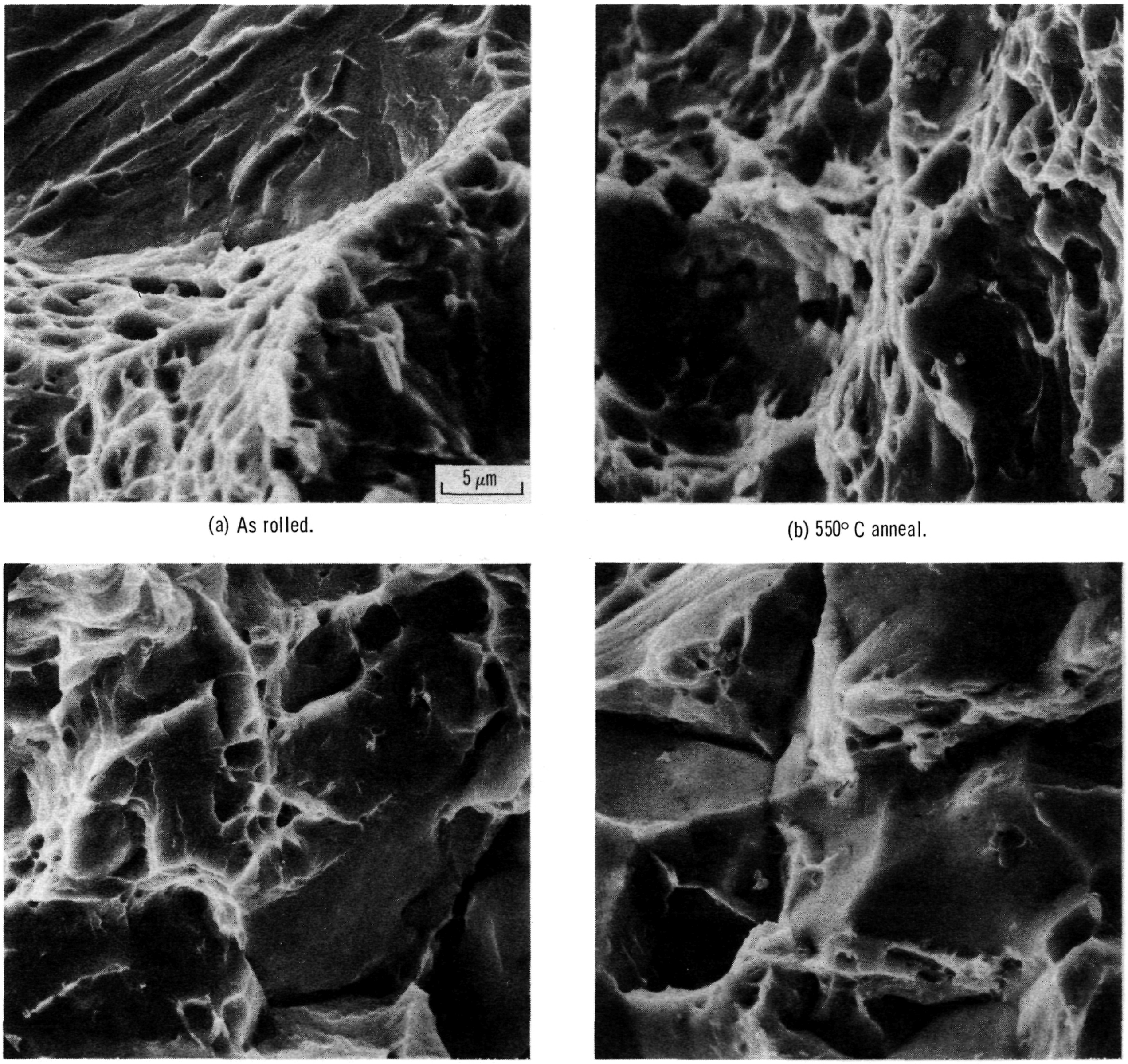


(a) Rolled at 650° C.

(b) Rolled at 1100° C.

Rolling direction

Figure 4. - Microstructures of as-rolled Fe-12Ni-0.5Al alloys.



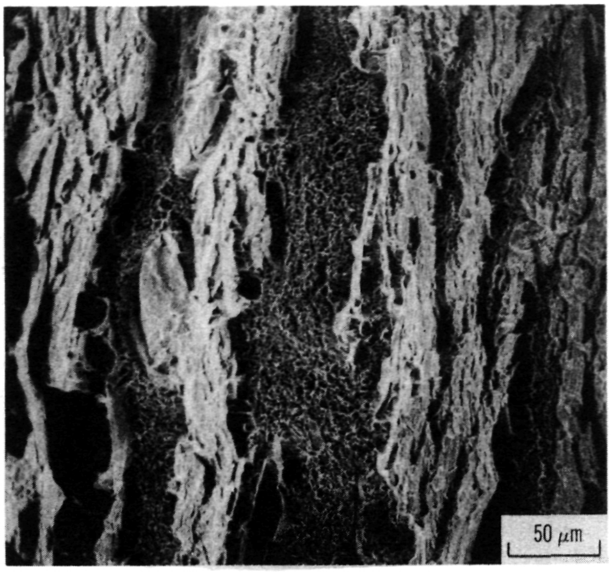
(a) As rolled.

(b) 550° C anneal.

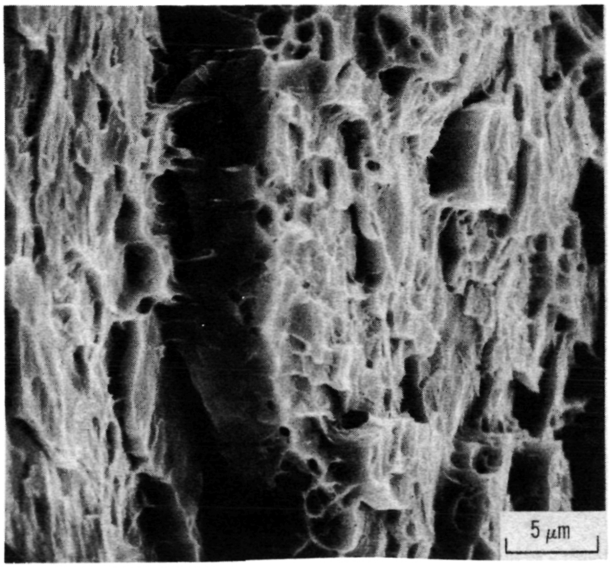
(c) 685° C anneal.

(d) 820° C anneal.

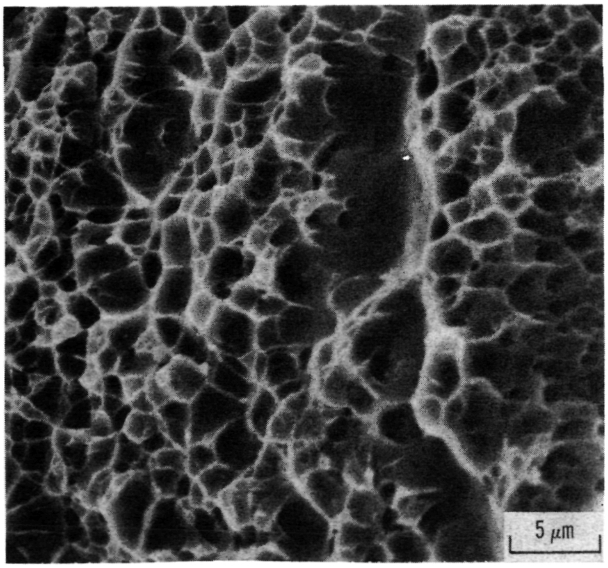
Figure 5. - Fracture surfaces of 1100° C thermomechanically processed Fe-12Ni-0.5Al alloys as revealed by SEM.



(a) Duplex failure mode.



(b) Primarily cleavage failure.



(c) Primarily dimpled failure.

Figure 6. - Fracture surface of as-rolled 25° C thermomechanically processed Fe-12Ni-0.5Al alloy revealing duplex failure mode.

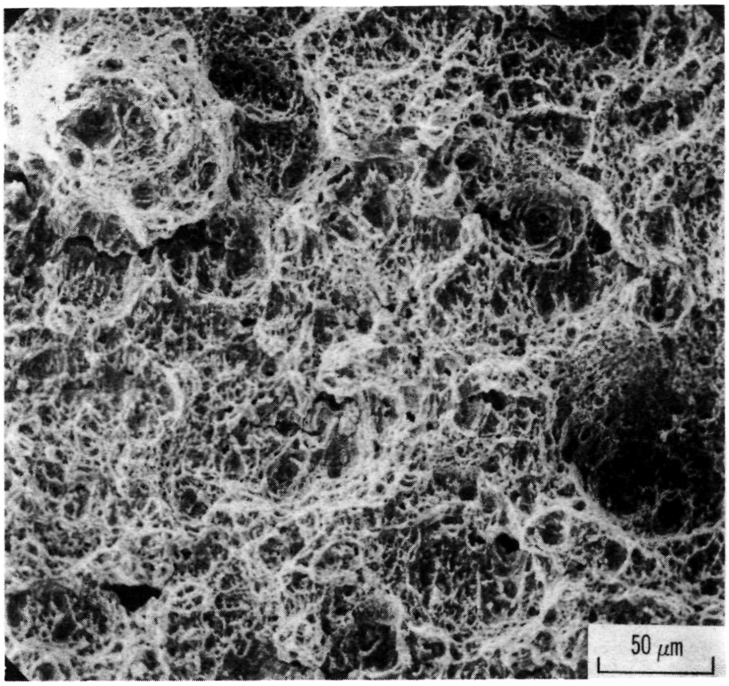


Figure 7. - Dimpled failure of as-rolled 650° C Fe-12Ni-0.5Al alloy.

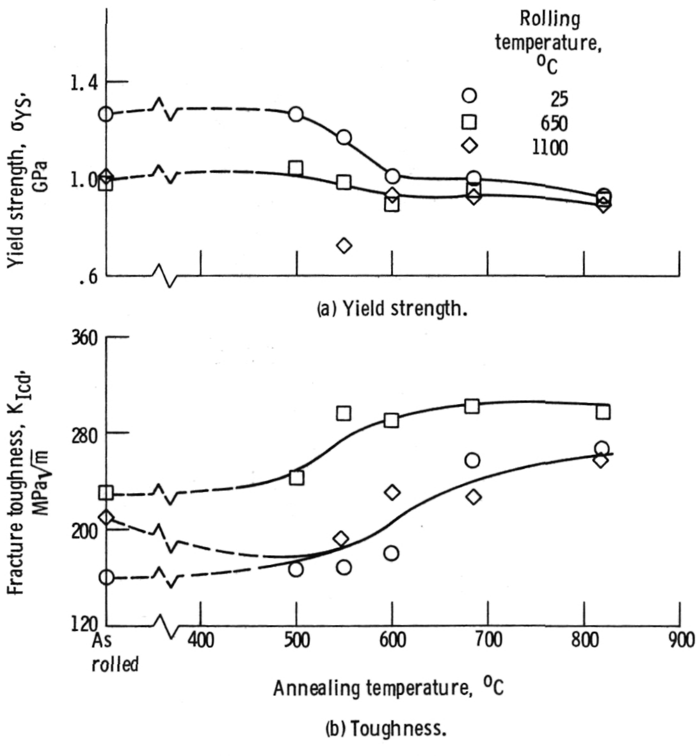
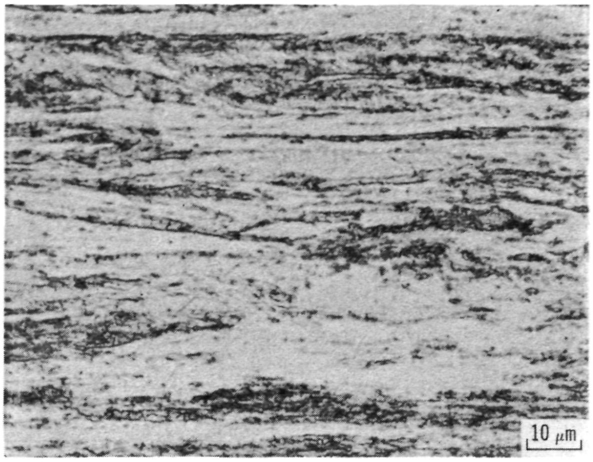
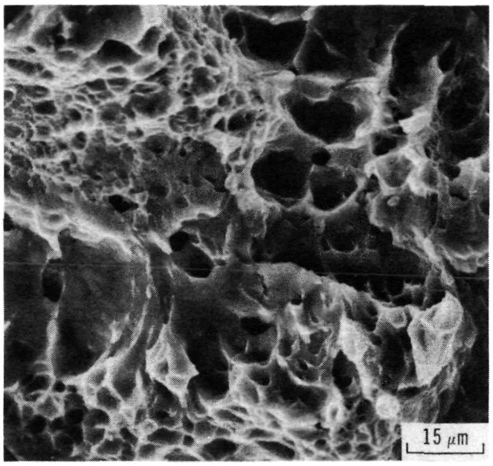


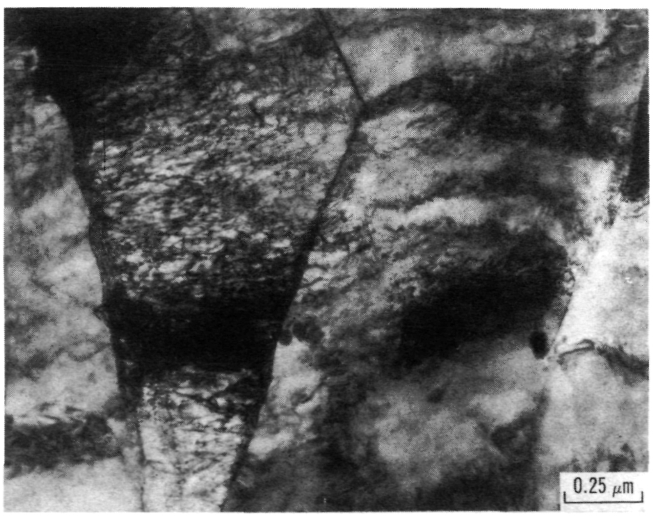
Figure 8. - Effects of thermomechanical processing on strength and toughness of Fe-12Ni-0.25Nb alloys at -196° C.



(a) Light optical microstructure.



(b) Fracture surface.



(c) TEM microstructure.

Figure 9. - Microstructures of thermomechanically processed Fe-12Ni-0.25Nb alloy (650° C rolling temperature, 550° C anneal.)

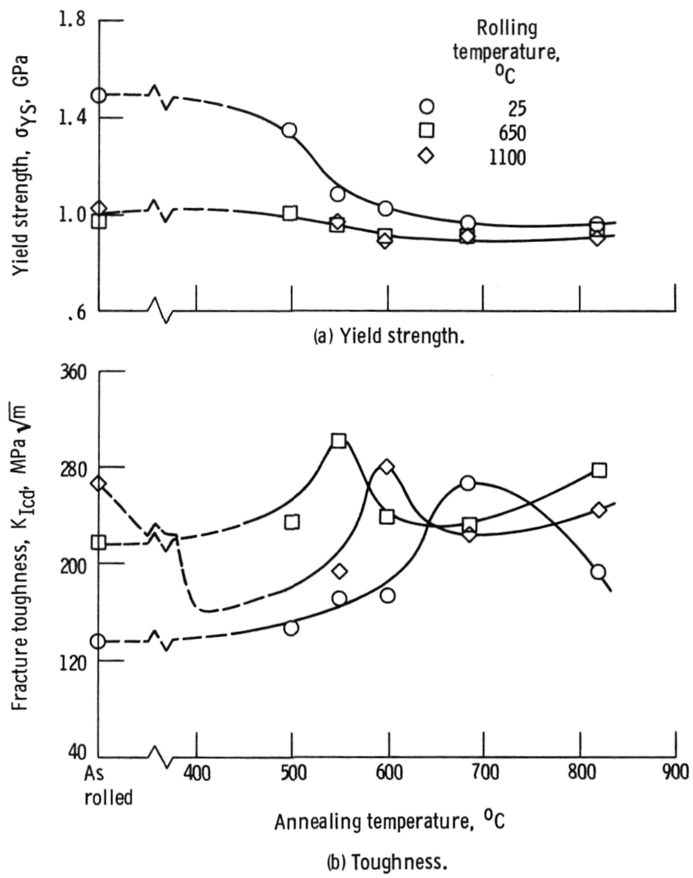
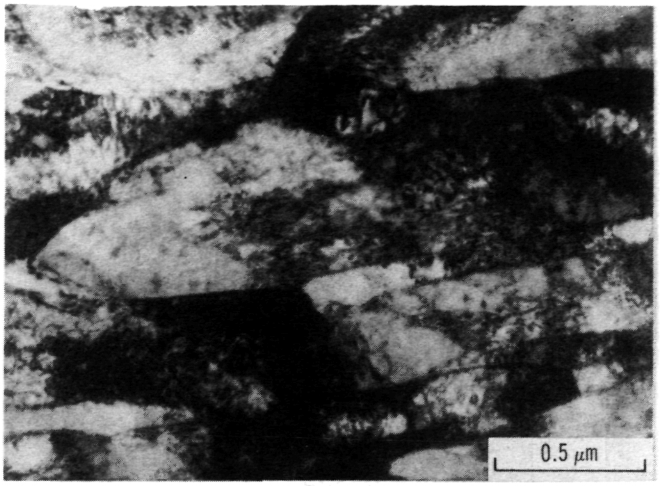
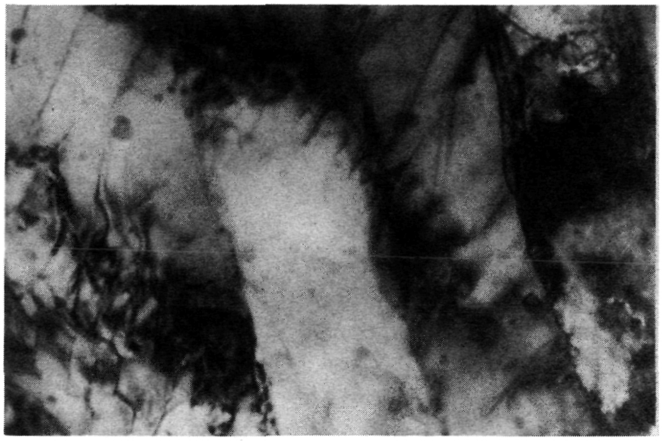


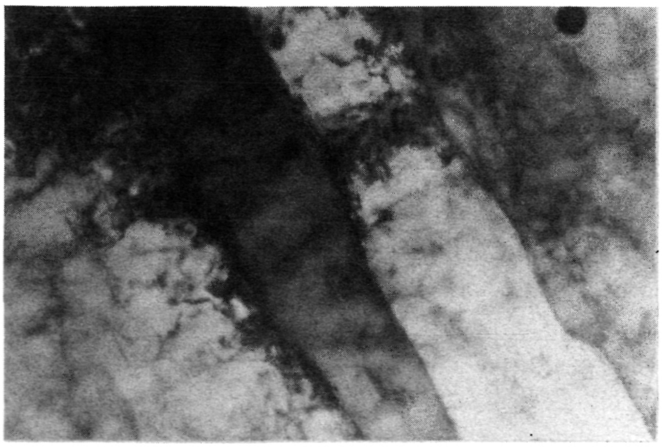
Figure 10. - Effects of thermomechanical processing on strength and toughness of Fe-12Ni-0.25Ti alloys at -196° C.



(a) Rolled at 25° C.



(b) Rolled at 650° C.



(c) Rolled at 1100° C.

Figure 11. - Effect of rolling temperature and subsequent 550° C annealing on substructure of thermomechanically processed Fe-12Ni-0.25Ti alloys as revealed by TEM.

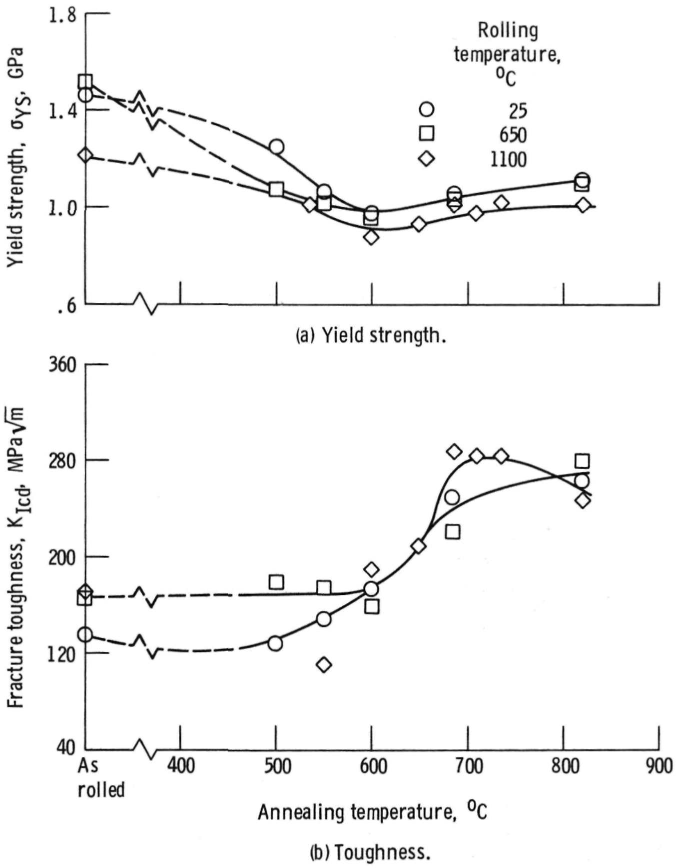
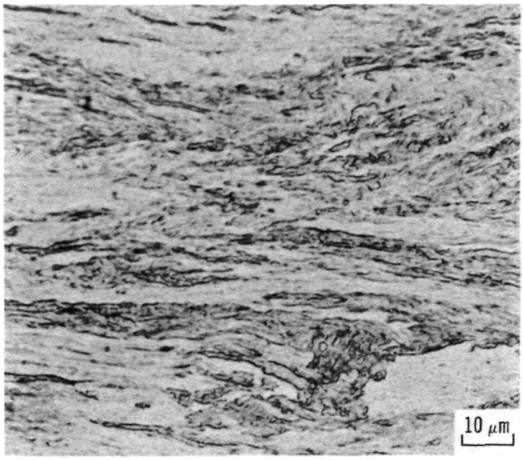
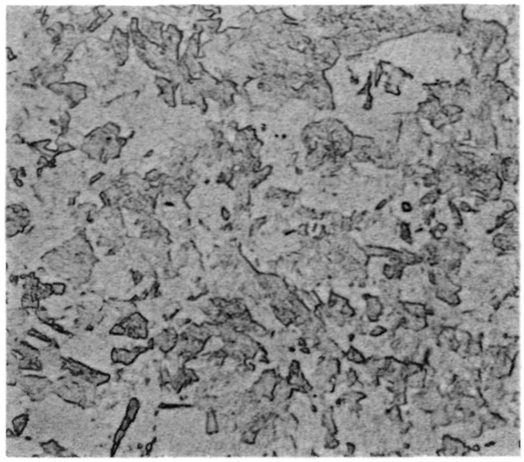


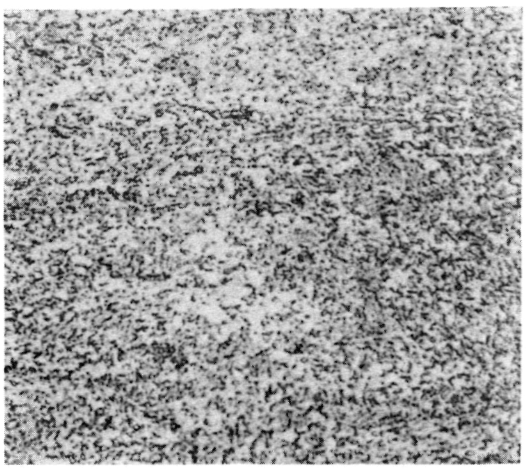
Figure 12. - Effects of thermomechanical processing on strength and toughness of Fe-12Ni-2.0V alloys at -196° C.



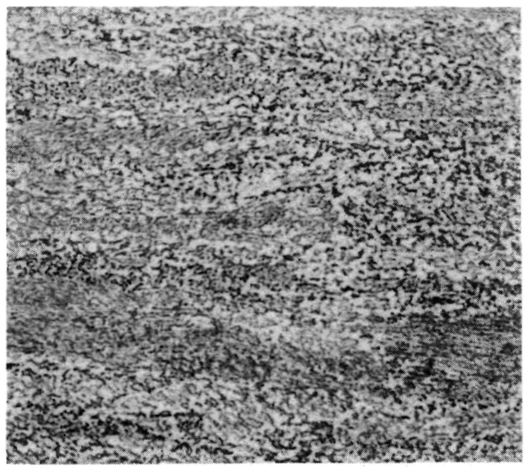
(a) 25° C thermomechanically processed alloy, as rolled.



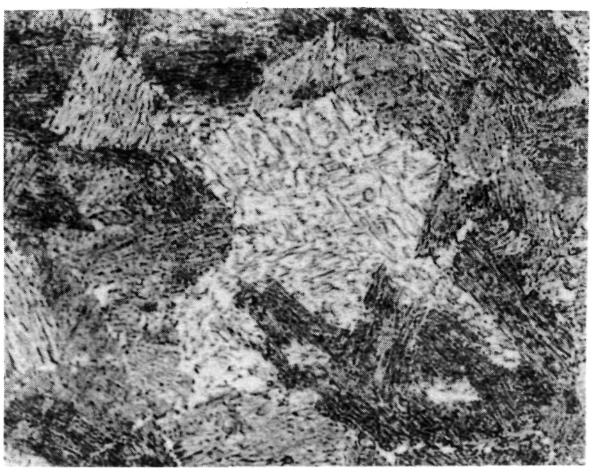
(b) 25° C thermomechanically processed alloy, 820° C anneal.



(c) 25° C thermomechanically processed alloy, 685° C anneal.



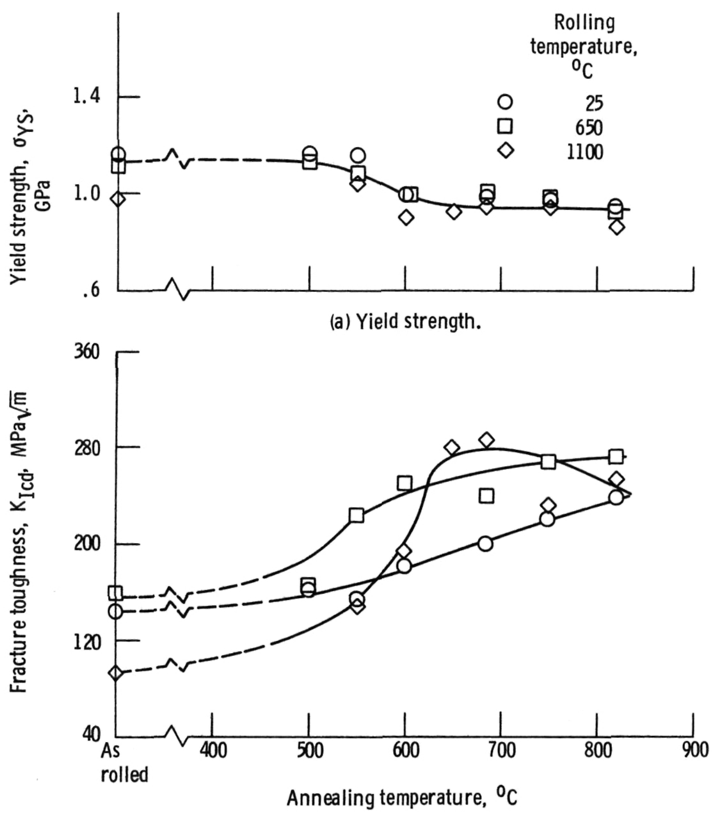
(d) 650° C thermomechanically processed alloy, 685° C anneal.



(e) 1100° C thermomechanically processed alloy, 685° C anneal.

Rolling direction

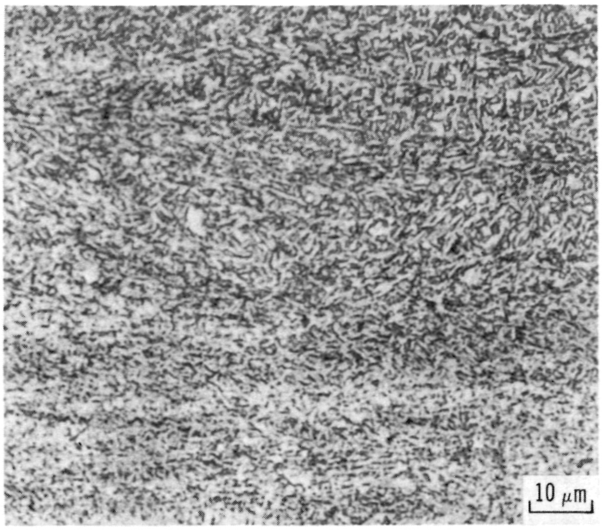
Figure 13. - Effects of TMP treatments on microstructure of Fe-12Ni-2V alloys.



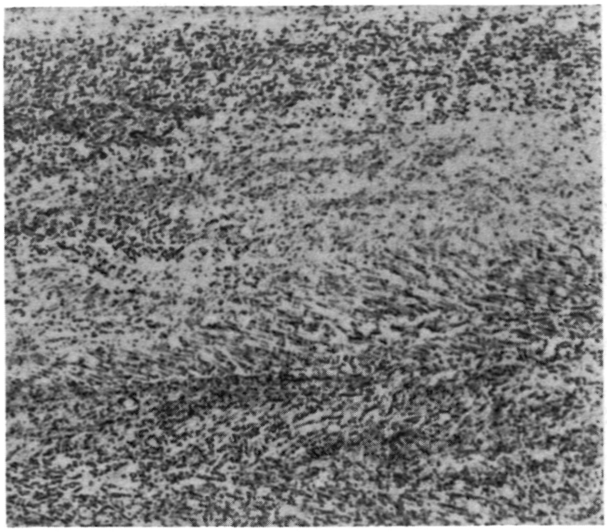
(a) Yield strength.

(b) Toughness.

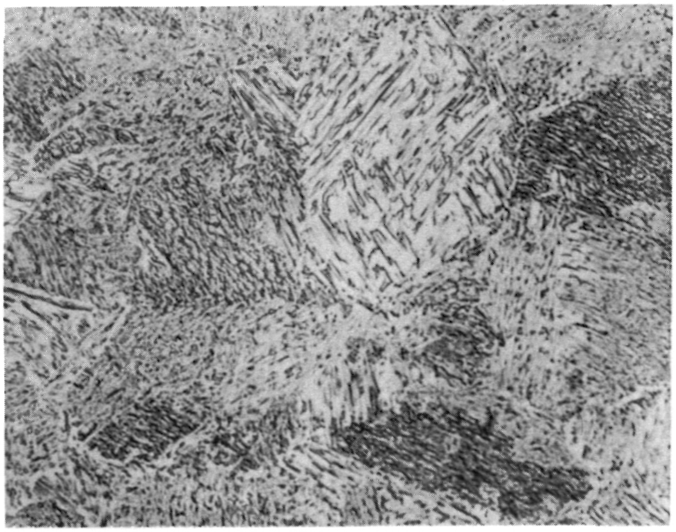
Figure 14. - Effects of thermomechanical processing on strength and toughness of Fe-12Ni-0.5Al-0.25Nb alloys at -196° C.



(a) 25° C thermomechanically processed alloy, 685° C anneal.



(b) 650° C thermomechanically processed alloy, 685° C anneal.



(c) 1100° C thermomechanically processed alloy, 685° C anneal.

Rolling direction

⟷

Figure 15. - Effects of TMP treatments in microstructures of Fe-12Ni-0.5Al-0.25 Nb alloys.

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		6. Performing Organization Code
7. Author(s) Joseph R. Stephens and Walter R. Witzke		8. Performing Organization Report No. E-9583
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16. Abstract

Thermomechanical processing (TMP) was evaluated as a method of strengthening normally tough iron-12 nickel-reactive metal alloys at cryogenic temperatures. Five iron-12 nickel alloys with reactive metal additions of aluminum, niobium, titanium, vanadium, and aluminum plus niobium were investigated. Primary evaluation was based on the yield strength and fracture toughness of the thermomechanically processed alloys at -196° C. A -196° C yield strength of 1.3 GPa (187 ksi) with a corresponding K_{Icd} toughness of 243 MPa$\sqrt{m}$ (221 ksi$\sqrt{in.}$) was achieved with an iron-12 nickel-0.5 aluminum alloy which had undergone a TMP treatment of 25° C rolling followed by annealing at 525° to 625° C. This represents the highest combination of cryogenic strength and toughness observed in iron-nickel alloys.

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