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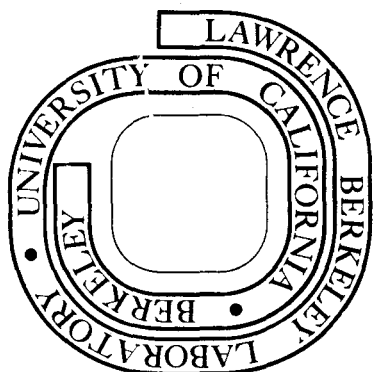
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PRECIPITATION-HARDENED Fe-Ni AUSTENITE

S. Jin, J. W. Morris, Jr., Y. L. Chen,
G. Thomas, and R. I. Jaffee

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AN INVESTIGATION OF TRANSFORMATION STRENGTHENING
IN PRECIPITATION-HARDENED Fe-Ni AUSTENITE

by

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ABSTRACT

Ausaged Fe-33Ni-3Ti and Fe-34Ni-3Ti-0.5Al austenitic alloys were transformation strengthened through sequential martensite ($\alpha' \rightarrow \gamma$) martensitic transformations. An increase in yield strength of 20-30 ksi (130-210 MPa) was obtained, leading to alloy yield strengths near 180 ksi (1250 MPa) in the ausaged and transformation strengthened condition. The presence of the γ' precipitates formed on ausaging did not radically change the nature of the shear transformation on the morphology and substructure of the transformation products. The presence of the γ' precipitates did, however, have the beneficial consequence that the $\alpha' \rightarrow \gamma$ reversion could be accomplished with slow heating rates without loss of transformation strengthening. The yield strength actually increased when a slow heating rate was used, a result attributed to the successful pinning of transformation-induced defects by the γ' precipitates and to the occurrence of additional precipitation during heating. The efficient pinning of transformation-induced defects had the further consequence that the alloys could be annealed for long periods after reversion to austenite without significant loss of strength.

I. INTRODUCTION

A reverse martensitic phase transformation influences the properties of austenite in a number of ferrous alloys. It has been established that a reverted austenite is appreciably stronger than an annealed austenite due principally to an increased dislocation density and to the formation of a fine substructure¹⁻⁸ resembling the structure of martensite. The strengthening achieved in an austenite through reverse martensitic transformation has been referred to as "transformation strengthening."

To transformation strengthen an austenitic alloy one must begin with an alloy composition such that the martensite start temperature (M_s) is between room temperature and liquid nitrogen temperature (-196°C). The alloy may then be transformed to martensite (at least in part) by cooling in liquid nitrogen. The martensitic alloy is then reverted to austenite by heating to above the austenite finish temperature (A_f) at a rate rapid enough to ensure that the reversion reaction proceeds through a reverse shear mechanism rather than through a diffusional nucleation and growth process. When the reversion reaction is complete the alloy is cooled to room temperature. For the transformation strengthening to have a beneficial effect on room temperature properties the reverted austenite must, of course, be mechanically stable with respect to stress-induced transformation to martensite at loads below the matrix yield strength.

Using this procedure Krauss and Cohen³ demonstrated the transformation strengthening of Fe-(30-34)Ni alloys, obtaining an increase in yield strength from ~ 30 ksi (210 MPa) to ~ 60 ksi (420 MPa) after a $\gamma \rightarrow \alpha' \rightarrow \gamma$

reversion cycle. In further work Koppenaal⁷ obtained a yield strength increase from ~40 ksi (270 MPa) to ~105 ksi (720 MPa) by reverting an Fe-24Ni-4Mo-0.3C alloy, and showed that the yield strength could be further increased by repeated reversion treatments. After five reversion cycles the strength increased to ~160 ksi (1100 MPa). In this case the strengthening is presumed due to the simultaneous influence of transformation-induced defects and carbide precipitates formed during heating.^{7,9}

The evidence cited above suggests that transformation strengthening may provide a useful alternative to thermomechanical treatment in processing austenitic steels to high strength and may offer a means for strengthening austenitic steels for applications which preclude severe thermomechanical treatment. However, the alloys and processes used in prior research have several features which limit their engineering potential. These include in particular the thermal instability of the transformation-induced defect structure and the relatively low strengths obtained.

As noted by Krauss and Cohen³ the transformation-induced defect structure is thermally unstable. On exposure to high temperature the reverted austenite softens rapidly through recovery and recrystallization processes. In laboratory studies the softening is avoided through the use of rapid heating to the reversion temperature, usually in a salt bath, followed by quenching to low temperature. Such a rapid thermal cycle is impractical in sections of reasonable thickness.

Moreover, the strengths obtained after initial reversion in the alloys studied to date are relatively low compared to those attainable

through simple ausaging in available austenitic steels. While higher strengths have been established through multiple cycling treatments elaborate processing is required, and the final strengths do not exceed those achieved in the most promising precipitation-hardened austenitic alloys.

Given this background we have been investigating the use of transformation strengthening in precipitation-hardenable austenitic alloys in the belief that the precipitation and transformation hardening mechanisms may be made to interact constructively in two ways: first, to strengthen the austenitic alloy to levels unattainable by either mechanism alone, and second, to stabilize the transformation-induced defect structure so that an efficient transformation strengthening may be accomplished with the relatively slow heating and cooling rates practical in thick sections. While the alloys studied to date are by no means optimal the evidence presented below documents the promise of transformation strengthening as a technique for achieving very high strength austenite through thermal processing alone.

II. TECHNICAL APPROACH

The choice of an austenite composition and thermal treatment to achieve a beneficial combination of precipitation and transformation strengthening poses several interrelated problems. These center around the need to maintain close control over the martensite transformation temperatures. To achieve an efficient transformation strengthening the alloy must have an M_s temperature well above liquid nitrogen temperature (-196°C) at the point in its processing at which the martensite

transformation is induced. To establish good room temperature mechanical properties in the fully processed condition the austenitic alloy must be mechanically stable with respect to stress-induced transformation to martensite, which requires that the M_s temperature be ultimately well below room temperature.

The opposing constraints on the M_s temperature limit the practical choice of precipitating species and precipitation conditions. The usual hardening precipitates, carbides, nitrides, and γ' intermetallic compounds, cause the M_s temperature to rise during precipitation hardening due to the depletion of γ stabilizing species from the lattice. While we showed in earlier work⁸ that the austenite stability lost on precipitation may be partly restored through suitable post-aging treatments (at least in the case of γ' Ni_3Ti), the restabilizing treatment cannot offset the substantial rise in M_s which occurs on ausaging to high strength. The opposing constraints on the M_s therefore dictate that, if conventional precipitation processes are to be used, the precipitation reaction must be essentially completed before transformation strengthening is carried out, and must not proceed significantly during a slow reversion treatment. The latter requirement appears to rule out precipitation processes based on rapidly diffusing interstitial solutes such as carbon and nitrogen.

The considerations above make the Fe-Ni-Ti (or Fe-Ni-Ti-Al) system an attractive candidate. The ternary alloys of this system may be ausaged to high strength through fine-scale precipitation of the coherent γ' (cubic) Ni_3Ti ($Ni_3(Ti,Al)$) phase. The M_s temperature after ausaging can be controlled by varying the nickel content so that the precipitation-

hardened alloy can be transformation strengthened. Since precipitation kinetics are slow in this system the precipitates should not coarsen dramatically during slow heating or cooling. Moreover, the ultimate stability of aged and reverted Fe-Ni-Ti austenite can be "fine-tuned" through low-temperature aging treatments.⁸

The approach taken in this research was, therefore, to select Fe-Ni-Ti and Fe-Ni-(Ti,Al) alloys with compositions chosen such that after ausaging to near peak strength the M_s temperature would lie in the range -50 to -100°C. The alloys could then be transformation strengthened by cooling to -196°C and heating to above the austenite finish (A_f) temperature. The interaction between the embedded precipitates and the transformation-induced defects was then expected to inhibit recovery so that useful transformation strengthening could be obtained with slow heating and cooling rates.

III. EXPERIMENTAL PROCEDURE

Two twenty-pound ingots of nominal composition Fe-33Ni-3Ti and Fe-34Ni-3Ti-0.5Al in wt. pct., were prepared by vacuum induction melting. The precise chemical compositions are given in Table I. The ingots were homogenized under vacuum at 1200°C for 24 hours, forged to 13 mm thick plates, solution annealed at 1050°C for 2 hours under argon gas atmosphere, and water cooled. Heat treatment for rapid reversion was carried out in a furnace under argon gas atmosphere.

The martensitic and reverse martensitic phase transformation temperatures were measured in a dilatometer equipped with both a cryostat capable of cooling to -196°C and a high temperature furnace under argon

Table I. Chemical Compositions (Wt. Pct.)

	Fe	Ni	Ti	Al	C	N
Fe-33Ni-3Ti	Bal.	33.2	2.99	---	---	---
Fe-34Ni-3Ti-0.5Al	Bal.	34.0	3.08	0.47	0.001	0.001

gas atmosphere (with a heating rate control). An alumel-chromel thermocouple was spot welded to the center of the tube shaped dilatometry specimen to monitor the temperature change continuously. At least two specimens were tested for each data point.

Optical metallography was performed according to standard laboratory practice. An etching solution of FeCl_3 (1 gm) + HCl (10 ml) + H_2O (25 ml) was used. Thin foils for transmission electron microscopy were prepared by chemical thinning in a solution of 100 ml H_2O_2 + 5 ml HF followed by electropolishing in a conventional chromic-acetic electrolyte using the window technique. The foils were examined in a Siemens Elmiskope I electron microscope, and in a Philips 301 electron microscope with a high resolution stage operated at 100 kV. Samples for X-ray diffraction analysis were chemically cleaned and polished on a metallographic polishing cloth.

Tensile tests were conducted at room temperature in an Instron machine using subsize round specimens of 12.7 mm gauge length and 3 mm gauge diameter at a crosshead speed of 0.05 cm/min. Two specimens were tested for each data point.

IV. RESULTS AND DISCUSSION

A. Microstructure and Properties of Ausaged Austenite

The microstructure of Fe-33Ni-3Ti austenite in the solution-annealed condition (1050°C, 2 hours) is shown in Fig. 1. This alloy has a large grain size, 150~200 μm mean grain diameter, and a low density of dislocations.

Ausaging at 720°C for 4 hours introduces a uniform dispersion of fine spherical γ' precipitates (Ni_3Ti or $\text{Ni}_3(\text{Ti},\text{Al})$ in the aluminum-bearing alloy^{10,11}). The precipitates are 50~80Å in diameter, and are shown in Fig. 2. The density of dislocations remains low in the ausaged austenite. Previous research^{8,10-12} indicates that the γ' precipitates are ordered and coherent with the austenite matrix. Similar results were obtained in the present study. Occasional grain boundary precipitates and cellular precipitates were observed in thin foil examination and optical metallography (Fig. 5(a)).

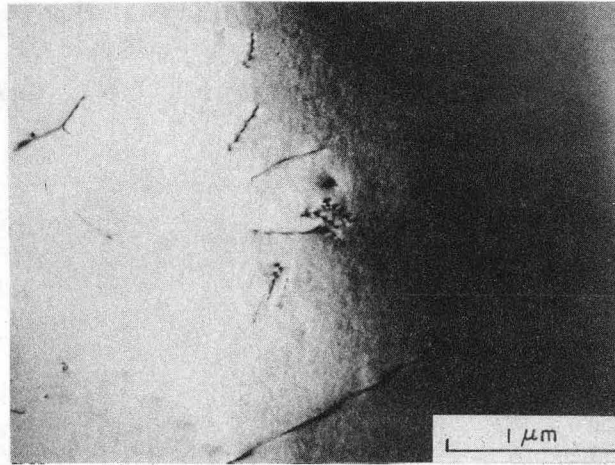
The aging response is similar in the Fe-33Ni-3Ti and Fe-34Ni-3Ti-0.5Al alloys. The strengths of both alloys increased significantly on precipitation of γ' as shown in Fig. 3, Fig. 4, and Table II. However, a severe loss of ductility occurs on ausaging. The soft annealed austenite exhibited a tensile elongation of 45~50 pct; after ausaging the elongation decreased to ~5-8 pct. The decrease in ductility is presumed to be caused by the reduced dislocation mobility due to the γ' precipitates and aggravated by the observed grain boundary precipitation. The alloy containing aluminum (Fe-35Ni-3Ti-0.5Al) was more ductile after ausaging than the aluminum-free Fe-33Ni-3Ti austenite. This observation agrees with the results of Wilson and Pickering.¹³ They suggested that

Table II. Yield Strengths (0.2 pct Offset)

Unit = ksi

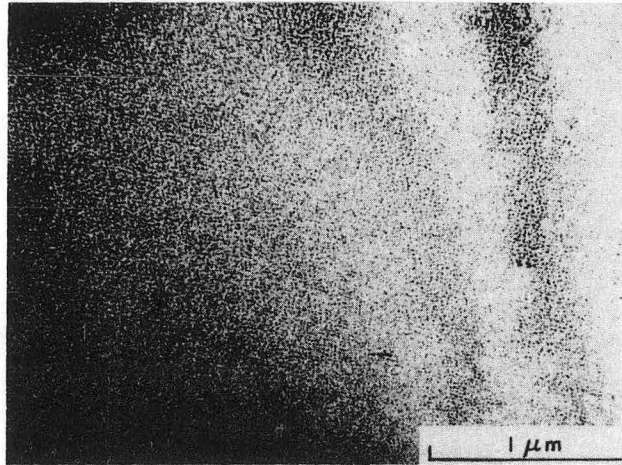
	Annealed	Ausaged*	Ausaged + LN ₂ cool	Fast Rev. 650°C/2 min	Fast Rev. 700°C/2 min	Slow Rev. 700°C/5 min
Fe-33Ni-3Ti	50	144	151	168	170	173
Fe-34Ni-3Ti-0.5Al	56	158	---	169	171	178

*720°C/4 hr. for Fe-33Ni-3Ti alloy and 720°C/12 hr. for Fe-34Ni-3Ti-0.5Al alloy.
 To convert to SI units, 1 ksi = 6.89 MPa.



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Fig. 1. Transmission electron micrograph of Fe-33Ni-3Ti austenite in the annealed and quenched condition.



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Fig. 2. Transmission electron micrograph of Fe-33Ni-3Ti alloy aged at 720°C for 4 hours showing a fine dispersion of γ' precipitates.

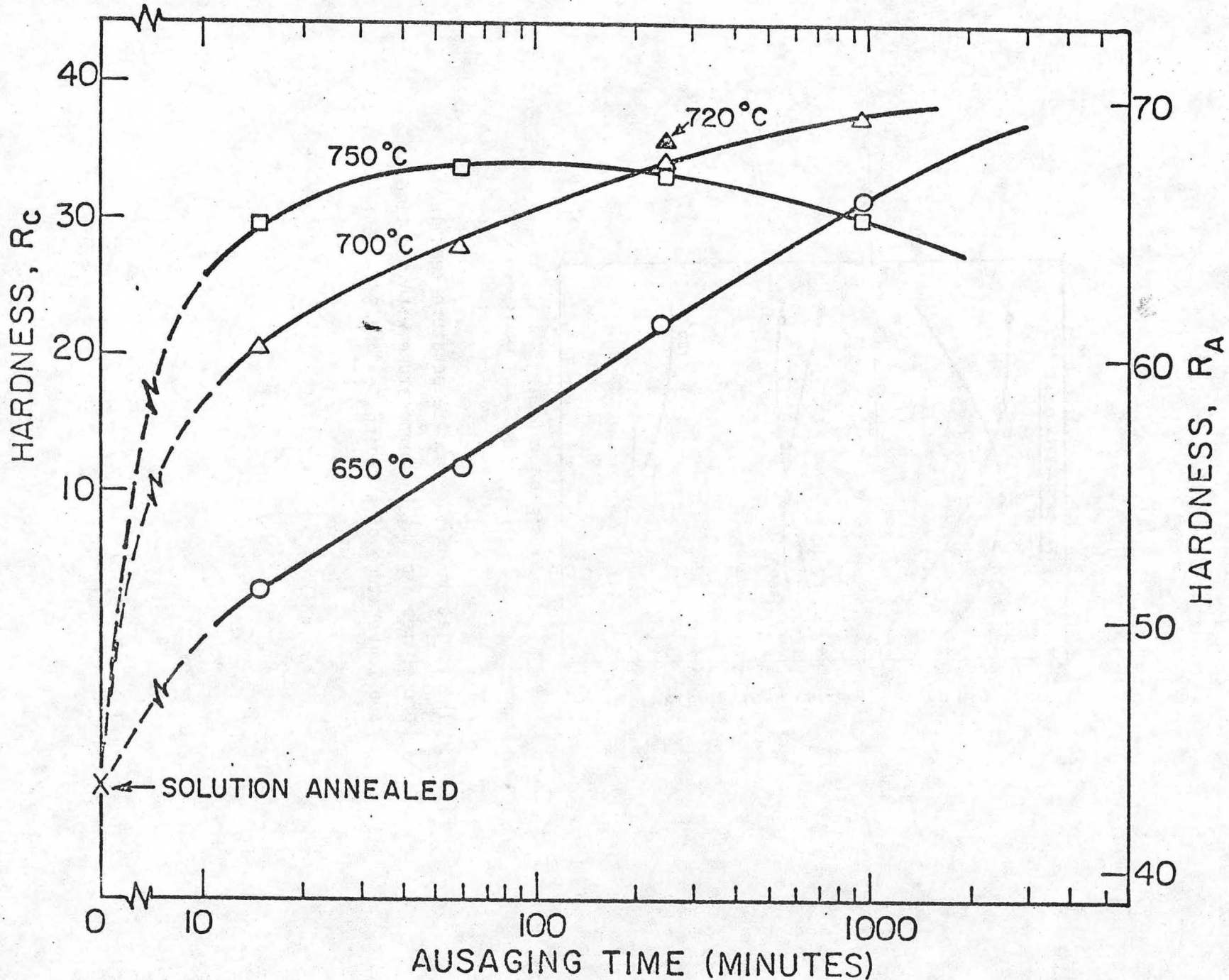
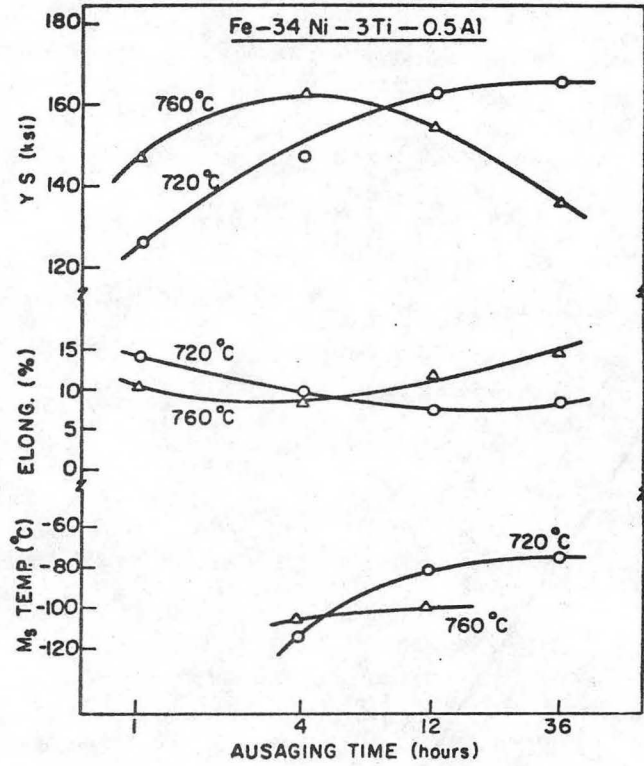


Fig. 3. Ausaging kinetics in Fe-33Ni-3Ti.

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Fig. 4. The variation of the room temperature tensile properties and the M_s temperature with the time and temperature of ausaging Fe-34Ni-3Ti-0.5Al.

the addition of a small amount of aluminum to Fe-Ni-Cr-Ti alloys reduces the misfit between the austenite matrix and the γ' precipitate particles, hence lowering the strain in the matrix and decreasing the driving force for cellular precipitation at grain boundaries.

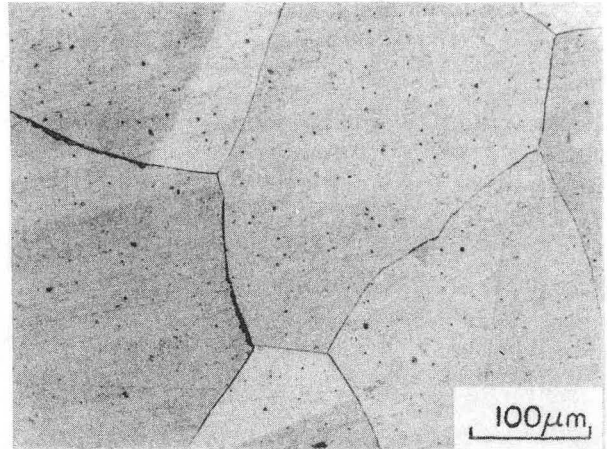
B. Martensitic Transformation of Ausaged Austenite

In the solution annealed condition both alloys have M_s temperatures below -196°C (liquid nitrogen temperature). However, the M_s temperatures increased during ausaging due to solute depletion from the matrix.^{8,14-16} After ausaging at 720°C for four hours the M_s temperature of the Fe-33Ni-3Ti alloy rose to -71°C . That of the Fe-34Ni-3Ti-0.5Al alloy increased to -81°C after twelve hours ausaging at 720°C .

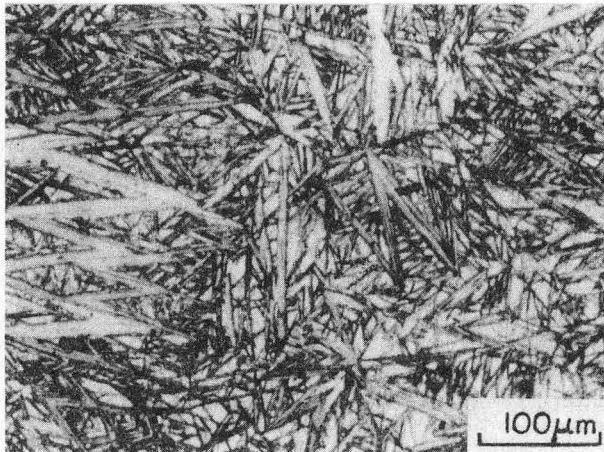
Both alloys could then be transformed to martensite by cooling in liquid nitrogen. After reheating to room temperature both alloys contained 60-70 pct martensite as measured by X-ray diffraction and optical metallography. Relevant microstructures are shown in Figs. 5(b) and 6. The martensite appears to have a mixture of dislocated and twinned substructures.

The behavior of the coherent γ' precipitates during the martensite transformation is of considerable interest. At least three alternatives exist:^{15,17} (1) the cubic precipitates may undergo a structural transformation to conform to the surrounding matrix; (2) the precipitates may retain their structure and orientation, but lose coherence with the surrounding matrix during transformation; (3) the precipitates may retain their structure, but undergo a rotation to accommodate the shear of the surrounding matrix.

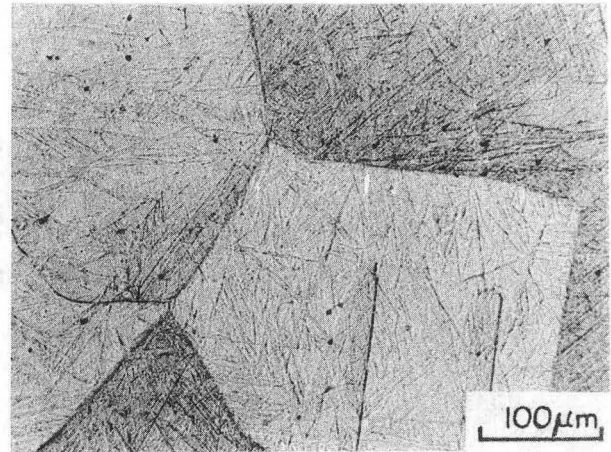
Fig. 5. Optical micrographs showing the change of microstructure on processing Fe-33Ni-3Ti. (a) Ausaged at 720°C for 4 hours; (b) Treatment (a) + cooled in liquid nitrogen, showing martensite laths; (c) treatment (b) + fast heating to 650°C (2 min.); (d) treatment (c) + anneal at 650°C for 7 hours; (e) treatment (b) + slow heating to 700°C.



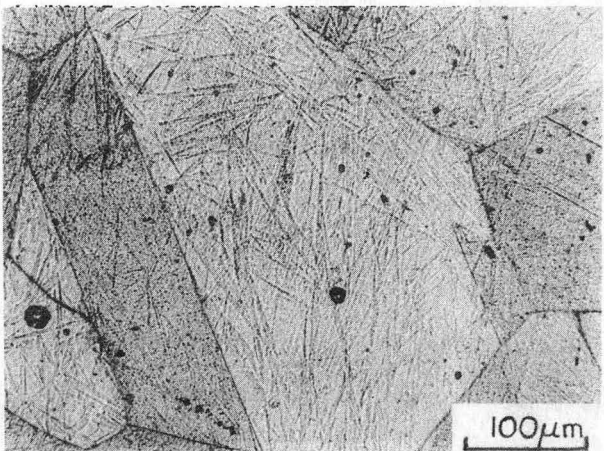
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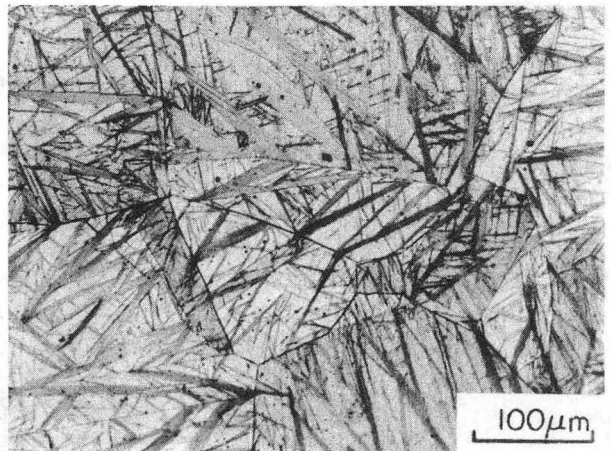
(b) XBB 766-5804



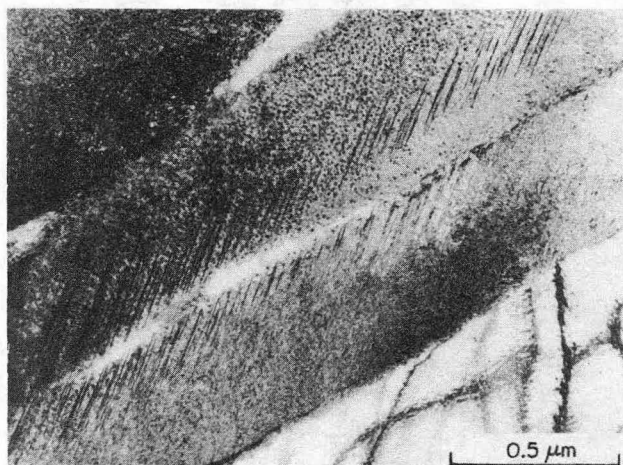
(c) XBB 766-5814



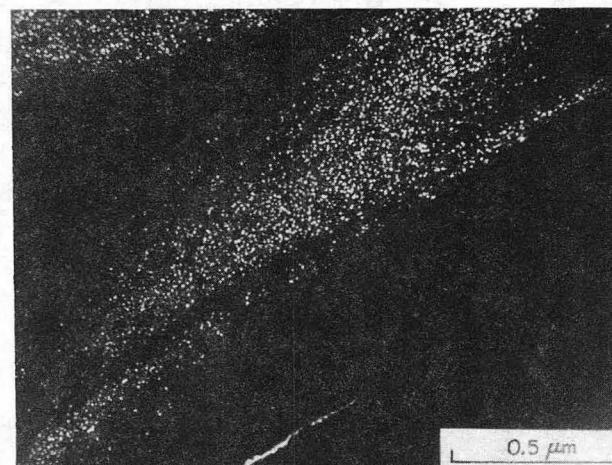
(d) XBB 766-5812



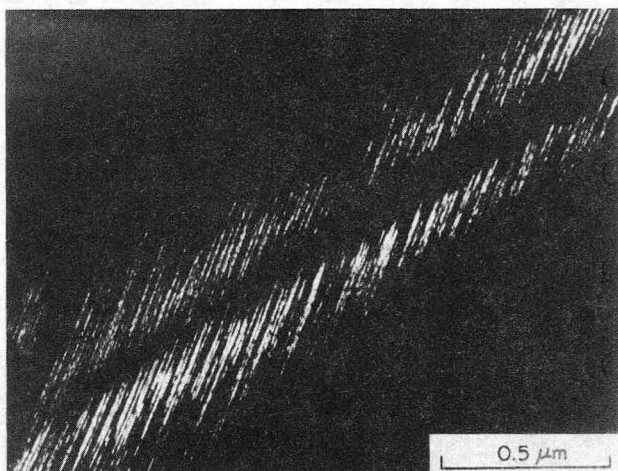
(e) XBB 766-5805



(a) XBB 766-5790



(b) XBB 766-5792



(c) XBB 766-5794

Fig. 6. Transmission electron micrographs of Fe-33Ni-3Ti after ausaging for 4 hours at 720°C and refrigeration in liquid nitrogen. (a) Bright field regions of martensite and untransformed austenite are marked M and A respectively. (b) Dark field image of the $(200)_{\gamma'}$ diffracted beam showing γ' precipitates within martensite plates. (c) Dark field image showing internal twinning within the martensite.

The third alternative was taken in the present case. Electron diffraction analysis shows that the embedded γ' particles remain fcc after the martensite transformation of the surrounding matrix. Fig. 6(b) presents a dark field image of γ' precipitates within the martensite plates; it was taken from a $(200)_{\gamma'}$ spot. The fact that only the γ' precipitates with the martensite appear in the dark field image shows that these precipitates differ in orientation from those in the neighboring untransformed region. Since all γ' precipitates in a given austenite grain have the same initial orientation the γ' particles within the martensite plate must have undergone a reorientation at constant structure during the transformation. It is not clear whether the precipitates have been plastically sheared or simply rotated. Nor is it clear whether the particles have retained interfacial coherence with the matrix, though they appear to have done so.

C. Reverse Martensitic Transformation

Specimens previously ausaged and cooled to -196°C transformed partially to martensite (60-70% by volume). The martensite was then reverted to austenite by heating to temperatures above the austenite finish (A_f) temperature. On subsequent cooling to room temperature the austenite structure is retained.

The effect of heating rate on the reversion reaction and on the resulting strength of the austenite was explored by using two alternate techniques giving radically different heating rates. In the first approach, the reversion was accomplished by immersing the specimen in a

high temperature salt bath (fast reversion). This technique imposes a heating rate greater than $100^{\circ}\text{C}/\text{min.}$ which, following prior work,⁸ is sufficiently rapid to ensure a reverse shear transformation in these alloys. In the second set of experiments the alloys were slowly heated at a controlled rate in an electric furnace (slow reversion). The imposed rate, $\sim 8^{\circ}\text{C}/\text{min.}$, was chosen to simulate the uniform heating rates attainable in the thermal processing of thick sections. The two sets of experiments will be discussed in turn.

1. Fast Reversion

In fast reversion the samples were immersed in a salt bath of fixed temperature (650°C , 700°C , or 720°C) and held for pre-selected times before cooling or quenching to room temperature. Fig. 5(c) presents an optical micrograph of the resulting structure. A substructure resembling that of martensite (Fig. 5(b)) is clearly seen. Phase analysis by X-ray diffraction shows, however, that the reversion to austenite is essentially complete at each of these temperatures. No martensite is detected. Selected area electron diffraction suggests that the reversion reaction is the crystallographic reverse of the original martensite transformation. The reverted laths examined have orientations identical to those of the parent grains from which they formed.

A transmission electron micrograph of the product austenite is presented in Fig. 7. The area marked A in the figure is a plate (or lath) of reverted austenite. The high dislocation density within the plate is apparent, and stands in marked contrast to the very low dislocation density in the adjacent region (B) which did not undergo phase

transformation. A high resolution transmission electron microscopic study showed that the γ' precipitates are retained during the reverse martensite transformation and remain virtually unchanged in size.

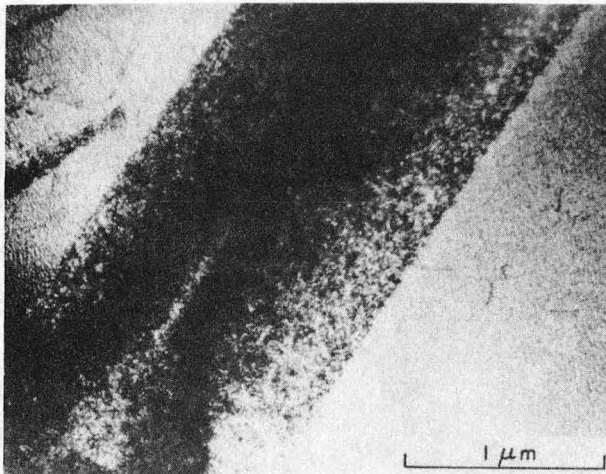
The dislocated lath structure shown in Fig. 7 is typical of the reverted austenite in these alloys. While internally twinned austenite is occasionally observed its occurrence is rare.

The strength increase obtained through the reversion treatment is presented in Table II. The increase in yield strength is significant, particularly when recognized as a supplement to the already high strength of the alloy in the ausaged condition.

2. Slow Reversion

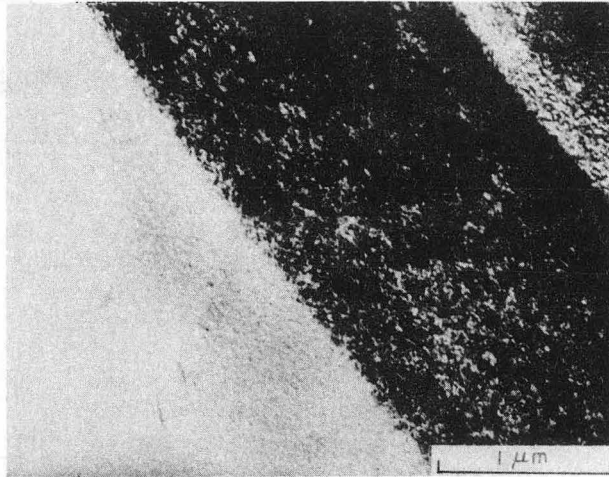
In slow reversion the alloy was reverted to austenite by heating in an electric furnace at a controlled rate of $\sim 8^\circ\text{C}/\text{min.}$ to a pre-selected final temperature of 700°C or 720°C . Optical and transmission electron micrographs of the resulting structure are presented in Figs. 5(c) and 8. The yield strengths obtained are included in Table II.

The results found after slow reversion are encouraging in two respects. First, despite the slow rate of heating the reverse martensitic transformation occurs through a reverse shear mechanism. The product microstructure is virtually identical to that seen after fast reversion. No evidence of the alternative nucleation and growth transformation mechanism was discovered. No apparent recrystallization occurred on slow heating to 720°C . Secondly, transformation strengthening occurs on slow heating. In fact, the yield strengths measured after slow reversion exceed those established by fast reversion by a small but consistent margin of ~ 5 ksi (35 MPa). Transmission electron micrographic studies



XBB 766-5816

Fig. 7. Reverted austenite (marked A) after rapid heating of Fe-33Ni-3Ti. The high density of dislocations in the reverted lath contrasts with the low density in the untransformed austenite (B).



XBB 766-5833

Fig. 8. Reverted austenite formed on slow reversion of Fe-33Ni-3Ti. The structure is similar to that established on fast reversion (Fig. 7).

(Fig. 8) suggest that the transformation strengthening is due to the retention of transformation-induced dislocations which are pinned by the embedded γ' precipitates.

The consistent relative increase in strength after slow reversion was initially surprising. The anticipated result had been a deterioration of strength due to thermal recovery of transformation-induced defects during slow heating. The strength increment actually observed may have several causes, but is tentatively attributed to an additional precipitation of Ni_3Ti (or $\text{Ni}_3(\text{Ti},\text{Al})$) in martensite during slow heating through the maraging temperature range ($\sim 400\text{-}550^\circ\text{C}$).

Since the reverted austenite laths are heavily loaded with tangled dislocations and γ' precipitates it would be difficult to identify precipitates in the final structure and establish that they formed during maraging. The presence of such precipitates is, however, indicated by several indirect pieces of evidence. First, the strength increase on slow reversion (Table II) would be expected if a maraging precipitation occurred. Second, a maraging reaction would be expected to raise the austenite transformation temperatures (A_s , A_f) through further depletion of γ -stabilizing species from the lattice. A dilatometric investigation (Table III) showed that the A_s and A_f temperatures do increase significantly when a low heating rate is used. Third, since a maraging precipitation would be specific to martensite such a reaction would cause the reverted austenite to have a composition slightly different from that of the untransformed austenite. The slowly reverted austenite shows a high contrast effect after etching (Fig. 5(e)) compared with the rapidly reverted austenite (Fig. 5(c)), as would be

expected if a composition difference were established during slow heating. Finally, the alloys are supersaturated during heating through the martensite range so maraging reactions are thermodynamically favored.

Table III. Phase Transformation Temperatures During Reversion of Ausaged Fe-33Ni-3Ti Alloy

Heating Rate	A _s	A _f
Fast Reversion ~100°C/min	431°C	525°C
Slow Reversion ~8°C/min	478°C	607°C

*A_s temperatures were taken at the maxima in dilation-temperature curves.

D. Annealing Response

The reversion of martensite in a section of reasonable thickness will, in practice, necessarily involve some holding period at high temperature. The annealing response of the reverted austenite is hence important. Fig. 9 shows the variation of the room temperature yield strength of the reverted Fe-33Ni-3Ti alloy as a function of annealing time at various temperatures. Equivalent data for the Fe-34Ni-3Ti-0.5Al alloy is given in Fig. 10. While the yield strength decreases with increasing annealing time the rate of softening at 700-720°C is relatively slow. Moreover, the strength advantage of the slowly reverted specimens is maintained during annealing. The softening curves for the fast and slow-reverted specimens are roughly parallel.

The rate of softening on annealing these alloys is very low compared to that in the Fe-Ni binary system studied by Krauss and Cohen.³ The improvement in annealing response is presumed to be due to the high precipitate density in the reverted austenite, which retards the thermal recovery of transformation-induced dislocations. The stability of the defect structure is illustrated in Fig. 11, a transmission electron micrograph of a specimen which was slowly reverted to 720°C and held for 7 hours. The fine substructure is retained during the long anneal. While there is some evidence of thermal recovery in the apparent leakage of dislocations from the edges of the reverted austenite laths, the reverted austenite remains highly dislocated.

An interesting and unanticipated result of these studies was the inverse temperature dependence of the softening rate. As documented in Figs. 9 and 10 the rate of softening increases when the annealing

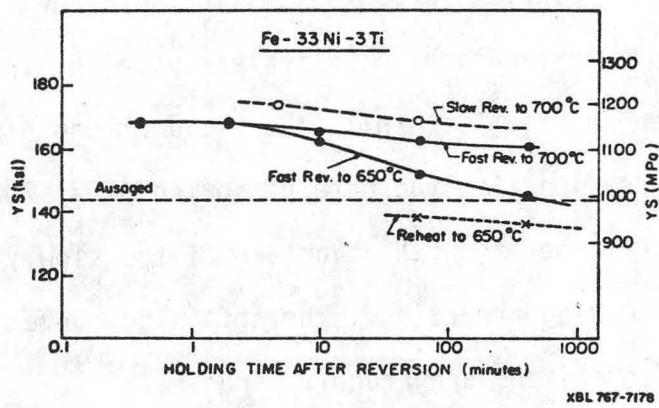


Fig. 9. Room temperature yield strength of ausaged and transformation strengthened Fe-33Ni-3Ti as a function of annealing time after reversion, annealing temperature, and rate of heating during reversion.

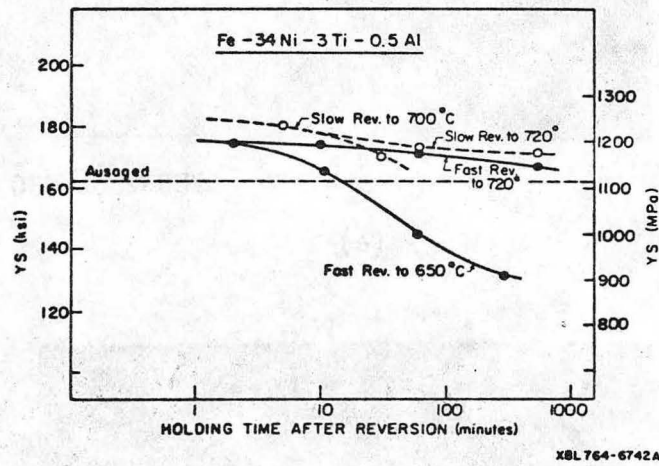
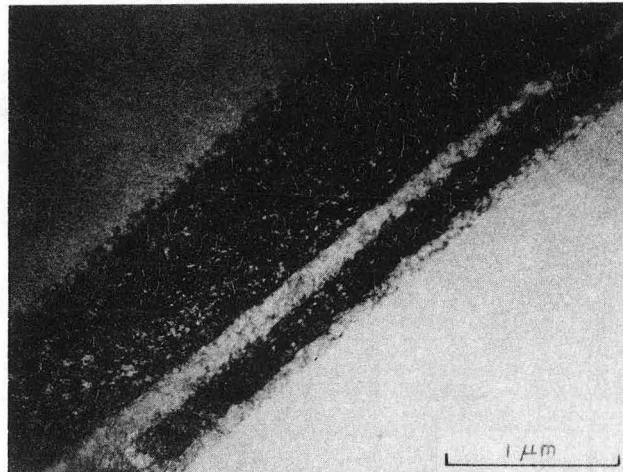
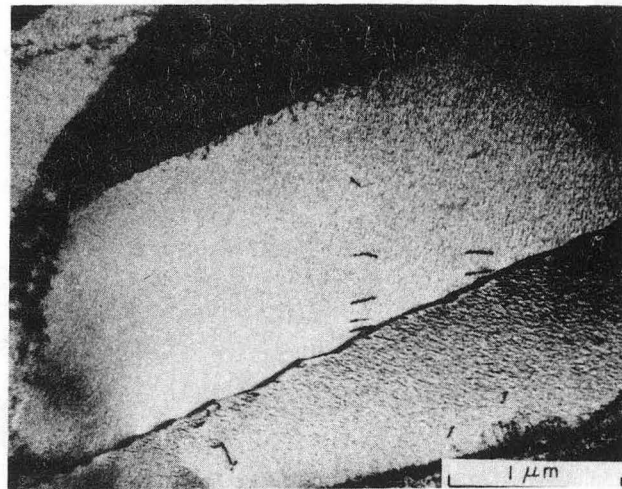


Fig. 10. Room temperature yield strength of ausaged and transformation strengthened Fe-34Ni-3Ti-0.5Al as a function of annealing time after reversion, annealing temperature, and rate of heating during reversion.



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(a)



XBB 766-5825

(b)

Fig. 11. Transmission electron micrographs of Fe-33Ni-3Ti after the treatment: 720°C/4 hours + refrigeration at -196°C + slow reversion + 720°C/7 hours. Two regions of the same specimen show the retention of transformation-induced dislocations within laths along with some evidence of recovery due to leakage of dislocations through lath boundaries.

temperature is lowered to 650°C, and leads to yield strengths which may fall below that of the unreverted austenite. These data suggest that metallurgical reactions other than thermal recovery are occurring.

There are several possible softening mechanisms in the alloys studied here including recovery and recrystallization, precipitate coarsening, and loss of austenite stability. Optical micrography of a specimen held for 7 hours at 650°C, Fig. 5(d), indicates, however, that recrystallization has not taken place. Transmission electron microscopy studies suggest that no substantial recovery occurs, and that the coarsening of pre-existing precipitates is negligible or small. The major source of low-temperature softening appears to be the loss of austenite stability due to additional solute precipitation on annealing.

This hypothesis is supported by the data presented in Figs. 4 and 12. Additional precipitation would decrease the solute content in the matrix causing a rise in the M_s temperature. This is presumably the reason for the higher M_s temperature associated with the lower ausaging temperature in Fig. 4. A decrease in austenite stability increases the likelihood of a stress-induced transformation to martensite with a concomitant decrease in yield strength. Fig. 12 is a plot of yield strength as a function of ausaging time at three aging temperatures. The specimens were initially ausaged at 720°C for 12 hours but were not transformation strengthened. The specimens soften more rapidly as the additional ausaging temperature is lowered from 720°C to 680°C to 640°C. Dilatometric studies indicate an associated increase in M_s . Schematic engineering stress-strain curves are shown in the right-hand side of the figure. As the aging temperature is lowered, the yield strength drops

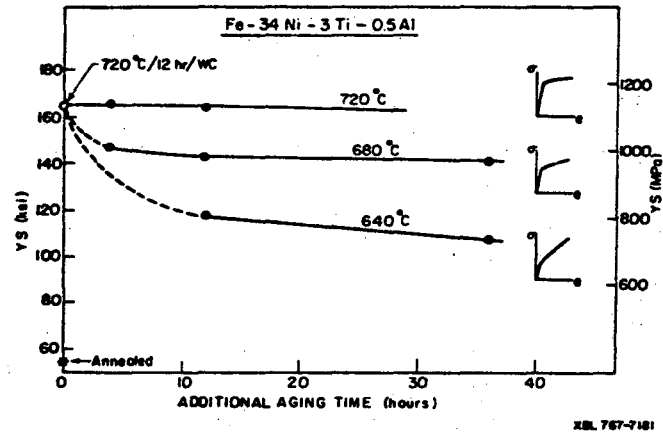
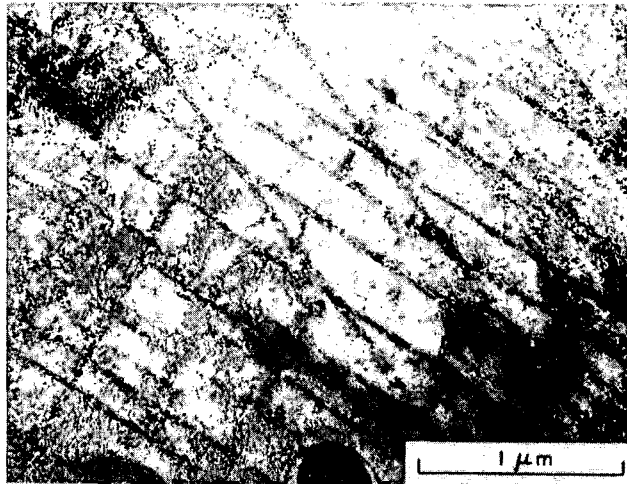


Fig. 12. Variation of the room temperature yield strength of Fe-34Ni-3Ti-0.5Al ausaged for 12 hours at 720°C as a function of the time and temperature of additional ausaging after cooling to room temperature.

and the work hardening rate increases, as would be expected if a stress induced martensitic transformation is responsible for the softening. The deformed regions of the tensile specimens are ferromagnetic after testing, indicating a transformation to martensite.

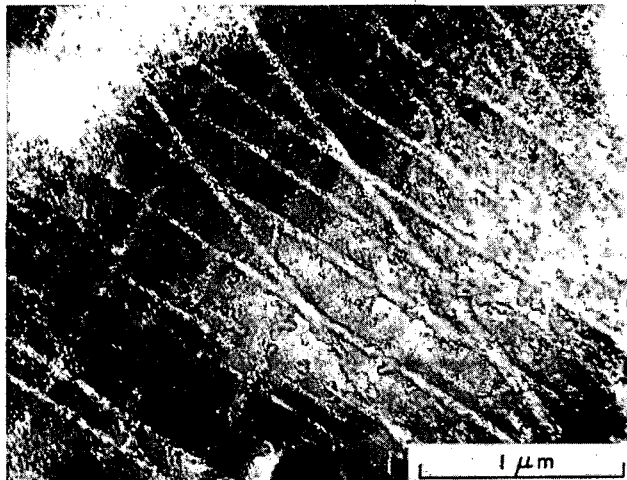
The precise nature of the precipitation reactions which cause solute depletion on low temperature aging is uncertain. However, both the specimens given a two-step aging (ausaging at 720°C, additional aging at 650°C) and those held at relatively low temperature after reversion contained microstructural regions decorated by a grid of band-shaped precipitates, shown in Fig. 13. On close examination it is seen that each band consists of small individual precipitate particles lined up along the band. A similar phenomenon was reported by Clark and Pickering¹³ and by Merrick and Nicholson¹⁷ who noted the alignment and joining of precipitates during the transformation of γ' to hexagonal Widmanstätten Ni_3Ti during aging. They suggested that the compressive stress exerted on the γ' precipitates due to slight lattice mismatch with the austenite matrix induces a transformation to an hexagonal close packed structure such that the $\{111\}$ planes of the γ' become the basal $\{0001\}$ planes of the hcp phase. Relieving the stress field around one particle will affect the stress distribution around neighboring particles. Thus precipitates in which the close-packed direction is collinear will rapidly transform giving rise to a line of hcp precipitates.

The coplanar precipitates observed in the present study were identified as hcp by electron diffraction analysis. Although the hcp particles form bands instead of single rows in this case, the process of precipitate transformation is presumed to be similar to that suggested by



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(a)



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(b)

Fig. 13. Banks of aligned hexagonal precipitates in Fe-33Ni-3Ti after the treatment: 720°C/4 hours + refrigeration at -196°C + fast reversion + 650°C/7 hours. (a) Bright field transmission electron micrograph. (b) Dark field image from an hcp diffracted beam of hexagonal Ni₃Ti.

Clark and Pickering.¹³ The apparent solute depletion found on annealing at 650°C would then be associated with the formation of these planar precipitate structures.

The results of the secondary aging treatments described above contrast with those obtained by Jin and Hwang⁸ in similar research on Fe-29Ni-4.3Ti. In this earlier work specimens were ausaged at 750°C and given a subsequent aging treatment at 500°C. Some of these specimens were transformed through a γ - α' - γ cycle between aging treatments. Others were simply quenched to room temperature. In both sets of samples the M_s temperature decreased significantly during secondary aging. While additional precipitation apparently occurred no planar bands of precipitates were found. The evidence obtained rather indicates the independent precipitation of small clusters on Guinier-Preston zones of $<10\text{\AA}$ size.

Taken together the results of these two investigations suggest that the nature of the precipitation process induced on secondary aging and the resulting changes in strength and austenite stability depend in a rather complicated way on the aging temperature, on the difference between this temperature and the original ausaging temperature, and possibly on other metallurgical variables. As a consequence secondary aging may have a positive, negative, or neutral effect on final alloy properties. These phenomena are under investigation.

V. CONCLUSIONS

(1) Transformation strengthening can be successfully accomplished in ausaged Fe-Ni-Ti and Fe-Ni-Ti-Al alloys through sequential martensite and reverse martensitic transformations. The presence of the γ' precipitates formed on ausaging does not radically change the nature of the shear transformation or the morphology and substructure of the transformation products.

(2) The yield strength of the aged and transformation strengthened alloys increases when a slow heating rate is used to achieve the reversion of martensite. This strength increase is believed to result from maraging reactions which occur during slow heating of the martensite phase.

(3) The annealing response of the transformation strengthened alloys appears to benefit from the presence of ausaged precipitates. The rate of softening during annealing is considerably below that found in Fe-Ni binary alloys where no precipitates are present. The aged and slowly reverted alloys retained their strength even after several hours of annealing at 700-720°C.

(4) The rate of softening of these alloys on annealing increased as the annealing temperature was lowered below the original ausaging temperature. This loss of strength is believed to be due to a decrease in the mechanical stability of the austenite, which is in turn attributable to a depletion of solute due to the formation of bands of hexagonal precipitates.

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