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### Title

USE OF THE REVERSE MARTENSITIC TRANSFORMATION and PRECIPITATION TO ENHANCE THE STRENGTH and STABILITY OF AUSTENITE

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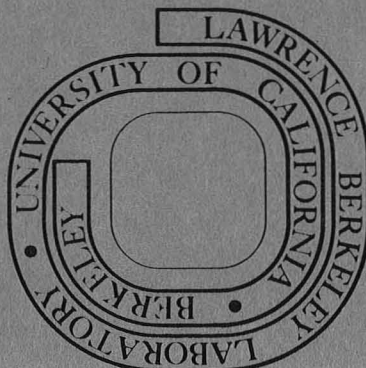
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USE OF THE REVERSE MARTENSITIC TRANSFORMATION AND PRECIPITATION  
TO ENHANCE THE STRENGTH AND STABILITY OF AUSTENITE

by

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ABSTRACT

The addition of martensitic and reverse martensitic phase transformations to precipitation hardening results in significant strengthening of Fe-Ni-Ti austenitic alloys due to the transformation-induced defects. Multiple cycles of  $\gamma$ - $\alpha'$ - $\gamma$  transformations led to a further strengthening. The stability of ausaged austenite as well as ausaged and transformation strengthened austenite is improved significantly through a final isothermal treatment at 500°C. This stabilization results in an increase of both the strength and the ductility in less stable (mechanically) austenites while only the increase of ductility was observed in relatively stable austenites.

USE OF THE REVERSE MARTENSITIC TRANSFORMATION AND PRECIPITATION  
TO IMPROVE THE STRENGTH AND STABILITY OF AUSTENITE

by

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ABSTRACT

The addition of carbonitic and reverse martensitic phase transfor-  
mations to precipitation hardening results in significant strengthening  
of Fe-Ni-Ti austenitic alloys due to the transformation-induced defects.  
Multiple cycles of 1 yr.  $\gamma$  transformations led to a further strengthening.  
The stability of aged austenite as well as aged  $\gamma$  transformation  
strengthened austenite is improved significantly through a final  
thermal treatment at 500°C. This stabilization results in an increase  
of both the strength and the ductility in test samples (mechanically)  
austenitized while only the increase of ductility was observed in  
relatively stable austenites.

## I. INTRODUCTION

The need for a high strength nonmagnetic steel for use in the retaining rings of a large electrical generator rotor instigated this research on the strengthening of austenitic alloys through the combination of precipitation and phase transformations. Previous research<sup>1-6</sup> showed that a metastable austenite could be strengthened considerably by introducing a high defect density and a fine substructure through martensitic ( $\gamma \rightarrow \alpha'$ ) and reverse martensitic ( $\alpha' \rightarrow \gamma$ ) phase transformations. The effect of such phase transformations on the properties of austenite containing precipitate particles, however, has not been studied in detail. The purposes of this investigation are: (1) to study the martensitic and reverse martensitic phase transformations in the presence of ausaged precipitates, and their effects on the properties of high strength austenite, and (2) to find out if the stabilization effect of fine precipitates could be incorporated to the development of high strength austenite.

## II. EXPERIMENTAL

Twenty-pound ingots of Fe-Ni-Ti alloys were prepared by induction melting under inert gas atmosphere. The chemical compositions are given in Table I. The ingots were homogenized at 1200°C for 24 hours under vacuum, forged to 13 mm thick plate, solution annealed at 1100°C for 2 hours under argon gas atmosphere, and water quenched. The  $M_s$  temperatures were measured in a dilatometer which allows the measurement of  $M_s$  down to liquid nitrogen temperature (-196°C). The tensile properties were measured in an Instron machine with subsize flat



tensile specimens. A Philips 301 electron microscope was operated at 100 kV for the transmission electron microscopy.

### III. RESULTS AND DISCUSSIONS

The microstructures, phase transformations, and the stabilization behavior are quite similar in the three alloys studied here, and will be described only for the Fe-29Ni-4.3Ti alloy. The mechanical properties will be discussed for all three alloys.

#### (1) Fe-29Ni-4.3Ti

The annealed Fe-29Ni-4.3Ti austenite has an  $M_s$  temperature of  $-89^\circ\text{C}$ , and is extremely soft. After ausaging at  $750^\circ\text{C}$  for 30 minutes, fine spherical  $\gamma'$  ( $\text{Ni}_3\text{Ti}$ ) precipitates of  $40\sim 60\text{\AA}$  in diameter are uniformly distributed throughout the austenite matrix as shown in Fig. 1. The specimen ausaged at  $750^\circ\text{C}$  for 30 minutes ( $M_s = -5^\circ\text{C}$ ) was cooled to liquid nitrogen temperature ( $-196^\circ\text{C}$ ) in order to induce a martensitic phase transformation. Approximately 70 pct of the matrix transformed to martensite as measured by X-ray diffractometry. The hardness of the alloy increased from  $R_C$  35.3 in the ausaged condition to  $R_C$  49.2 due to the transformation substructure. The  $\gamma'$  precipitates are apparently retained in the product martensite as well as in the untransformed austenite region. The substructure of martensite appeared to be a mixture of dislocated and twinned martensite.

The reverse martensitic transformation of the ausaged and  $\text{LN}_2$ -cooled specimen was achieved by rapid heating to  $800^\circ\text{C}$  in a salt bath and holding for 30 seconds. The reversion to austenite was complete.

The alloy remained fully austenitic on cooling to room temperature. The microstructures of reverted austenite are shown in Fig. 2(a) and (b). It is well known that the reverse martensitic transformation influences the properties of austenite. The reverted austenite shows, among other things, a complicated substructure with high defect density,<sup>4</sup> a high strength,<sup>1-3</sup> and a higher self-diffusion rate<sup>7</sup> than the annealed austenite. Similar results were obtained in the present investigation although the alloy subjected to reversion in this study contained  $\gamma'$  precipitate particles. In Fig. 2(a), the high density of dislocations (compare with Fig. 2(c)) and the lath-like substructure are illustrated. The  $\gamma'$  precipitate particles are retained during the reverse martensitic transformation, as shown in a high magnification micrograph, Fig. 2(b). The hardness of the reverted austenite increased to  $R_C$  41 compared with that of the ausaged austenite,  $R_C$  35.3.

The stabilization of annealed austenite through the formation of fine coherent precipitate has been studied by several investigators.<sup>8,9</sup> The advantage of the marked decrease of  $M_s$  temperature obtainable at the earlier stage of precipitation hardening disappears when the austenite is further aged to obtain a high strength. From the view point of alloy development, it would be desirable to find a method of controlling the austenite stability after the high strength has been obtained, especially when the "transformation-strengthening" mechanism is utilized. In order to allow phase transformations, the austenite stability should be low to start with, but needs to be restored after transformation hardening. Initial success in enhancing the stability of strengthened austenite has been obtained in the present study. The



stabilization of high strength austenite was achieved by isothermal holding at 500°C up to 12 hrs. The hardness of the high strength austenite remained virtually unchanged during the stabilization treatment. The results are shown in Fig. 3. The details of the austenite stabilization and its interpretation are reported elsewhere.<sup>10</sup> The suppression of  $M_s$  to -72°C (which is close to that of annealed austenite,  $M_s$  -89°C) while strengthening the austenite to  $R_C$  hardness 41 through precipitation and phase transformations is a significant restoration of austenite stability considering the solute depletion due to the formation of  $\gamma'$  precipitate. The decrease of  $M_s$  obtained by isothermal holding at 500°C in high strength austenite as well as in an annealed austenite is attributed to the formation of either G.P. zone or short-range order of smaller than  $\sim 10\text{\AA}$  in diameter based on the isothermal resistivity measurements. The details of the analysis are described elsewhere.<sup>10</sup>

## (2) Fe-31Ni-3Ti Austenite

The Fe-31Ni-3Ti alloy has an  $M_s$  temperature of -113°C in the annealed condition. The  $M_s$  temperature increases to -21°C after ausaging at 720°C for 4 hours. This deterioration of stability on aging again has the consequence that the mechanical properties of the alloy are strongly affected by deformation-induced transformation of the austenite. Typical mechanical properties of Fe-31Ni-3Ti austenite after various thermal treatments are listed in Table II. The yield strengths are low and are affected by the deformation-induced martensitic transformation. The yield strength of the sample ausaged for 4

hours is actually lower than that of the sample ausaged for 100 minutes, although the peak age hardening has not been reached. This is attributed to the lowered austenite stability caused by further decrease of matrix solute content on longer aging; the reverse trend is observed in a more stable alloy, Fe-33Ni-3Ti, as shown in Table II. A high work hardening coefficient and strong ferromagnetism are exhibited by the deformed specimens after testing. The  $\gamma$ - $\alpha'$ - $\gamma$  transformation sequence does lead to an increase of  $\sim 20$  ksi in the yield strength of these alloys as can be seen from Table II, and both the strength and ductility are slightly improved by an additional stabilizing anneal at  $500^{\circ}\text{C}$  for 12 hours.

### (3) Fe-33Ni-3Ti Austenite

In the annealed condition the Fe-33Ni-3Ti alloy has an  $M_s$  temperature below liquid nitrogen temperature. However, after ausaging at  $720^{\circ}\text{C}$  for four hours the  $M_s$  temperature increases to  $-71^{\circ}\text{C}$ . This value of the  $M_s$  temperature appears to be near the optimum for Fe-Ni-Ti alloys with respect to the objectives of this research. At room temperature the aged austenite has sufficient stability to retain high yield strength, though there is some degree of mechanically induced martensite on deformation. The  $M_s$  temperature is, however, high enough to obtain a significant transformation (50-60%  $\alpha'$ ) on cooling to liquid nitrogen temperature, hence allowing  $\gamma$ - $\alpha'$ - $\gamma$  transformation processing.

An ausaging treatment at  $720^{\circ}\text{C}$  for four hours (to  $R_C$  hardness  $\sim 36$ ) was chosen for initial processing studies. Studies of martensite transformation and its reversion in the ausaged alloy showed that

heating to 650~750°C for thirty seconds in a salt bath sufficed to completely revert martensite formed on cooling to liquid nitrogen. The tensile properties resulting from ausaging, phase transformations, and stabilization processes are given in Table II. The alloy as ausaged at 720°C for 4 hours has a yield strength ~135 ksi which increases to ~150 ksi after cooling in liquid nitrogen and reversion at 750°C. A subsequent stabilization anneal at 500°C for 12 hours yields only a slight change in strength but improves the ductility as measured by tensile elongation. Longer final anneals cause a deterioration in both strength and toughness. The evolution of microstructure during these processes is now under investigation through transmission electron microscopy. The results of this study should provide fundamental information on the sources of the mechanical property variations observed.

The effect of multiple transformation cycles was tested by subjecting an alloy which had been aged at 720°C for four hours to repeated  $\gamma$ - $\alpha'$ - $\gamma$  cycles. The reversion treatment for these cycles was fixed at 750°C/30 sec. Repetition of the transformation is likely to increase the dislocation density, hence improving final strength. The results are plotted in Fig. 4. As is apparent from the figure multiple  $\gamma$ - $\alpha'$ - $\gamma$  cycles lead to an increase in strength. The yield strength rises from ~135 ksi in the ausaged condition to ~150 ksi after the first cycle, to ~162 ksi after two cycles, and to an asymptote at ~166 ksi after five cycles. The achievement of this strength level is encouraging in several respects. A yield strength of 166 ksi exceeds the best values obtained by Kopenaal<sup>6</sup> in transformation strengthening a carbon-containing alloy and is, to our knowledge, the highest



strength ever obtained in an austenitic steel by thermal processing alone. These strength levels are comparable to those of the alloys now in use for retaining rings (for example, work-hardened Fe-18Mn-18Cr-1N austenitic alloy).

#### IV. SUMMARY

The reverse martensitic transformation of the precipitation hardened matrix resulted in a further strengthening of the Fe-Ni-Ti austenitic alloys. The stability of high strength austenite was improved significantly through an additional low temperature precipitation process.

#### ACKNOWLEDGMENT

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Table I. Chemical Compositions (wt. pct.)

| Alloy         | Fe   | Ni    | Ti   | C     | N     | S     | P     |
|---------------|------|-------|------|-------|-------|-------|-------|
| Fe-29Ni-4.3Ti | Bal. | 29.08 | 4.26 | 0.012 | 0.005 | 0.005 | 0.003 |
| Fe-31Ni-3Ti   | Bal. | 31.3  | 3.03 | ---   | ---   | ---   | ---   |
| Fe-33Ni-3Ti   | Bal. | 33.2  | 2.99 | ---   | ---   | ---   | ---   |

Table II. Tensile Properties of Austenitic Alloys

|   | Heat Treatment             | Fe-31Ni-3Ti |             |               | Fe-33Ni-3Ti |             |               |
|---|----------------------------|-------------|-------------|---------------|-------------|-------------|---------------|
|   |                            | YS<br>(ksi) | TS<br>(ksi) | Elong.<br>(%) | YS<br>(ksi) | TS<br>(ksi) | Elong.<br>(%) |
| A | 720°C/100 min.             | 94          | 174         | 12.0          | 123         | 183         | 19.0          |
| B | A + LN <sub>2</sub> + Rev. | 118         | 173         | 8.9           | 138         | 177         | 13.8          |
| C | B + Stab.                  | 121         | 215         | 13.0          | 133         | 186         | 21.4          |
| D | 720°C/4 hr.                | 78          | 180         | 10.4          | 135         | 174         | 9.9           |
| E | D + LN <sub>2</sub> + Rev. | 95          | 179         | 8.8           | 152         | 178         | 10.3          |
| F | E + Stab.                  | 104         | 213         | 11.9          | 151         | 197         | 15.9          |

LN<sub>2</sub>: Cooled to -196°C; Rev.: Reverted to austenite (750°C/30 sec.); Stab.: Stabilization (500°C/12 hr.).



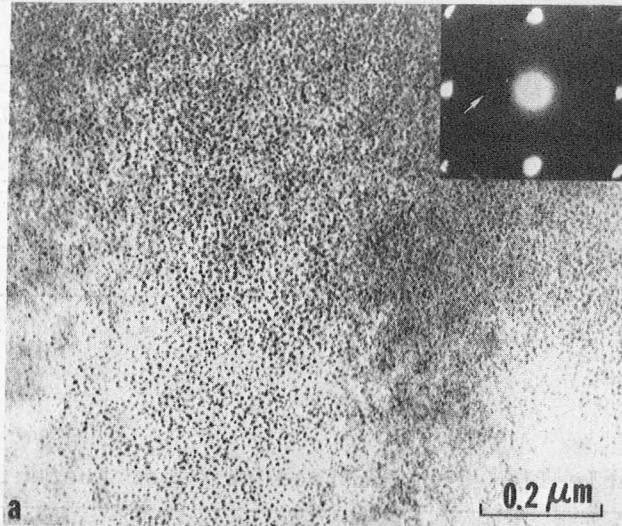


Fig. 1. Precipitation hardened austenite showing the  $\gamma'$  precipitates.

XBB 759-6637

| Heat Treatment               | YS (ksi) | YS (MPa) | TS (ksi) | TS (MPa) |
|------------------------------|----------|----------|----------|----------|
| A 720°C/100 min              | 183      | 129      | 19.0     | 133      |
| B A + 1N <sub>2</sub> + Rev. | 177      | 125      | 13.8     | 121      |
| C B + 2N <sub>2</sub>        | 186      | 132      | 21.4     | 152      |
| D 720°C/1 hr                 | 174      | 124      | 9.9      | 118      |
| E D + 1N <sub>2</sub> + Rev. | 178      | 126      | 10.3     | 121      |
| F E + 2N <sub>2</sub>        | 195      | 137      | 13.9     | 152      |

YS: Cooled to -196°C; Rev.: Reverted to austenite (750°C/30 sec.); N<sub>2</sub>: Stabilization (500°C/12 hr.)

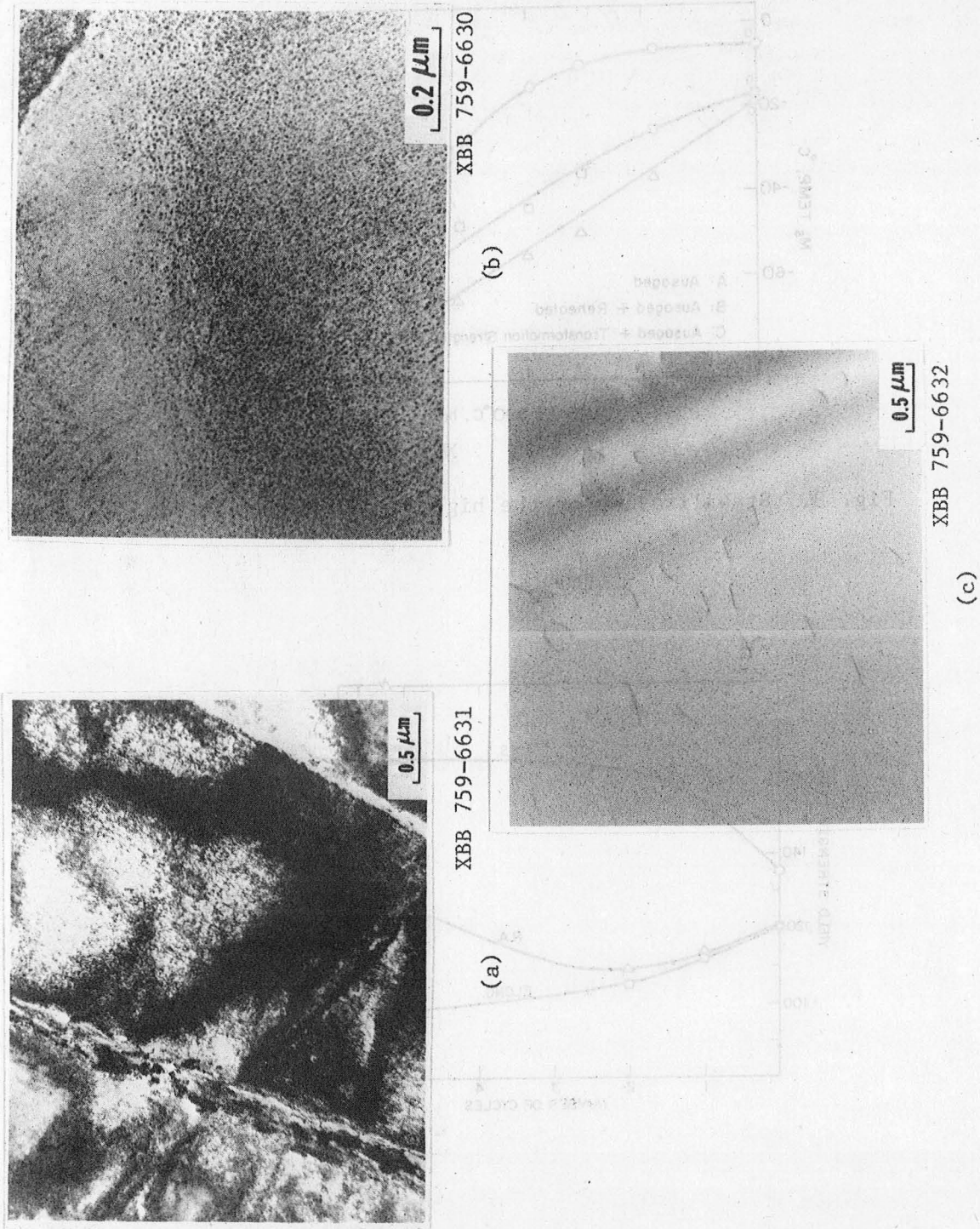


Fig. 2. TEM micrographs. (a) Reverted austenite showing the lath-like substructure and the high density of dislocations. (b)  $\gamma'$  precipitates in the reverted austenite. (c) Annealed austenite.

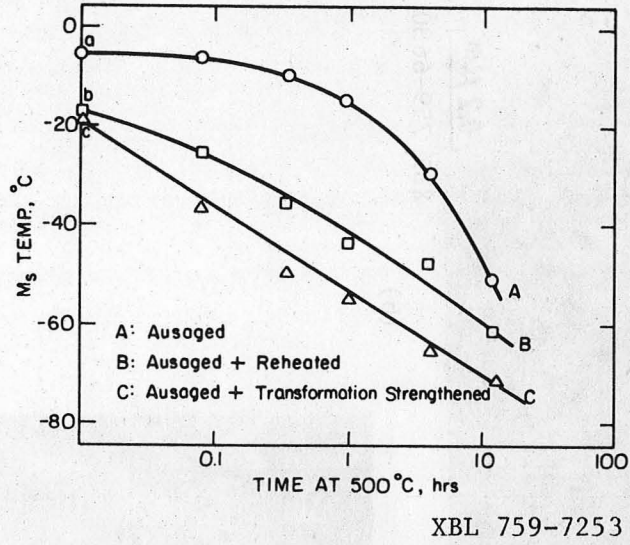


Fig. 3. Stabilization of the high strength austenites.

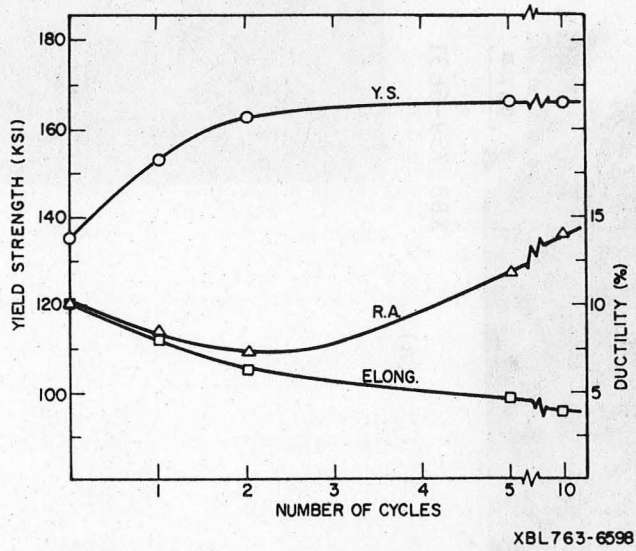


Fig. 4. Tensile properties vs. number of cycles of  $\gamma$ - $\alpha'$ - $\gamma$  transformations.



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