

Effect of Thermal Cycling on the Mechanical Properties of 350-Grade Maraging Steel

U.K. VISWANATHAN, R. KISHORE, and M.K. ASUNDI

The effects of retained austenite produced by thermal cycling on the mechanical properties of a precipitation-hardened 350-grade commercial maraging steel were examined. The presence of retained austenite caused decreases in the yield strength (YS) and ultimate tensile strength (UTS) and effected a significant increase in the tensile ductility. Increased impact toughness was also produced by this treatment. The mechanical stability of retained austenite was evaluated by tension and impact tests at subambient temperatures. A deformation-induced transformation of the austenite was manifested as load drops on the load-elongation plots at subzero temperatures. This transformation imparts excellent low-temperature ductility to the material. A wide range of strength, ductility, and toughness can be obtained by subjecting the steel to thermal cycling before the precipitation-hardening treatment.

I. INTRODUCTION

AUSTENITE can be introduced in the microstructure through two different methods of heat treatment. When the alloy is isothermally heated at the temperature at which the dissolution of the intermetallic precipitates occurs, austenite starts nucleating at regions where the nickel content is high.^[1] Prolonged aging at this temperature leads to the formation of a substantial amount of austenite, and the austenite so formed is known as "reverted austenite." In the second method, the steel is subjected to thermal cycling between room temperature (RT) and the temperature (A_f) at which the austenitic transformation is completed. When rapidly cooled from this temperature, without allowing the austenite to attain equilibrium composition, the solute-rich austenite remains untransformed and the solute-depleted austenite transforms to martensite.^[2] The austenite formed by this treatment is known as "retained austenite."

The effect of austenite on the mechanical properties has received much attention in the past,^[3,4] and it has recently been demonstrated that the effect depends upon the morphology of the austenite.^[5] In this investigation, austenite was retained in a 350-grade commercial maraging steel by thermal cycling, and its effects on RT mechanical properties were evaluated by carrying out tension and Charpy impact tests on samples with different fractions of austenite. The influence of the stability of retained austenite on the deformation behavior was also examined at subambient temperatures.

II. EXPERIMENTAL PROCEDURE

The material used in this investigation was a 350-grade commercial quality maraging steel. The chemical composition of the alloy is given in Table I. The material was

received in the form of a forged bar 70 mm in diameter in a double solution-annealed condition. The solution annealing consisted of a first anneal at 950 °C for 2 hours followed by air cooling and a second anneal at 820 °C for 3.5 hours followed by air cooling. Tension specimens of 4-mm diameter and 20-mm-gage length and standard Charpy V-notch specimens of 10 × 10 × 55 mm were fabricated from the forged bar in such a way that the major axes of the specimens were parallel to the length of the bar.

The investigation was conducted on the samples given one of two specific heat treatments: (1) direct aging of the solution-annealed material at 510 °C for 3 hours (hereafter called "conventionally aged") or (2) thermal cycling of the solution-annealed material, followed by aging at 510 °C for 3 hours. The thermal cycling treatment consisted of heating the material at a specific heating rate between RT and the predetermined A_f temperature of 750 °C. After thermal cycling, the samples were quenched in water as soon as the A_f temperature was reached, thus preserving the chemical inhomogeneity of the austenite. Thermal cycling of the tension specimens was carried out at either 6 °C/min or 9.5 °C/min, and the Charpy specimens were cycled at a heating rate of 11.5 °C/min. Repeat thermal cycling up to five cycles was given to vary the fraction of austenite in the samples. After the desired number of cycles, all specimens were given a typical aging treatment at 510 °C for 3 hours.

Tension tests were conducted using a floor model screw-driven machine at a constant crosshead speed of 0.5 mm/min, which corresponded to a nominal strain rate of $4.2 \times 10^{-4} \text{ s}^{-1}$. The load elongation plots were recorded on a strip chart recorder. A specially designed cage was used to carry out tests at subambient temperatures. The test specimens and the cage were cooled for 30 minutes in a cryogenic bath of methyl alcohol and dry ice before the test. A calibrated chromel-alumel thermocouple (accurate to ± 2 °C) was used for temperature measurements.

Impact tests were carried out using an instrumented impact tester. Tests were carried out over a range of temperature from -196 °C to 175 °C, and the absorbed energy was measured from the load-time traces.

Specimens for optical microscopy were electrolytically

U.K. VISWANATHAN and R. KISHORE, Radiometallurgy and Metallurgy Divisions, respectively, are with the Bhabha Atomic Research Centre, Bombay 400 085, India. M.K. ASUNDI, Metallurgical Consultant, is with Vibha, Bandra (East), Bombay 400 051, India.

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Table I. Chemical Composition of the 350-Grade Maraging Steel (Weight Percent)

Element	C	Ni	Mo	Co	Ti	Al	Mn	S	P	Fe
Wt pct	0.0046	18.39	3.99	12.32	1.63	0.12	0.021	0.0028	0.005	bal

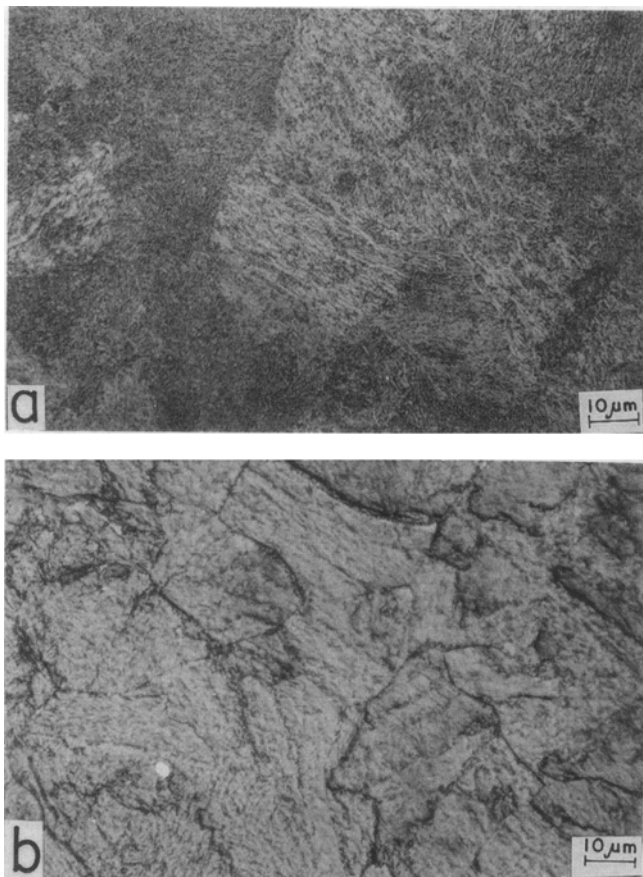


Fig. 1—Optical micrograph of the alloy: (a) cycled five times and aged at 510 °C for 3 hours and (b) directly aged at 510 °C for 3 hours after solution annealing.

etched in 5 pct chromic acid using a voltage of 6 V dc. Specimens for transmission electron microscopy (TEM) were prepared by the “window” technique using a solution of 6 pct perchloric acid, 34 pct *n*-butanol, and 60 pct methanol at an operating voltage of 20 V dc and a temperature of -30 °C.

The volume fraction of austenite was estimated by X-ray diffraction (XRD) using the samples prepared for optical metallography. Graphite monochromated Cu K_α radiation was used, and the volume fraction of austenite was estimated from the (110)_α and the (111) peaks of martensite and austenite, respectively, using the methods described by Cullity.^[6]

III. RESULTS AND DISCUSSION

Figure 1(a), shows the optical microstructure of the alloy subjected to five thermal cycles at a heating rate of 6 °C/min and aged. Figure 1(b) shows the optical microstructure of the conventionally aged alloy. Figure 1(a) shows a fine lamellar morphology. The prior austenite grain boundaries, which are readily apparent in Figure 1(b), are not seen in Figure 1(a). The corresponding bright-field mi-

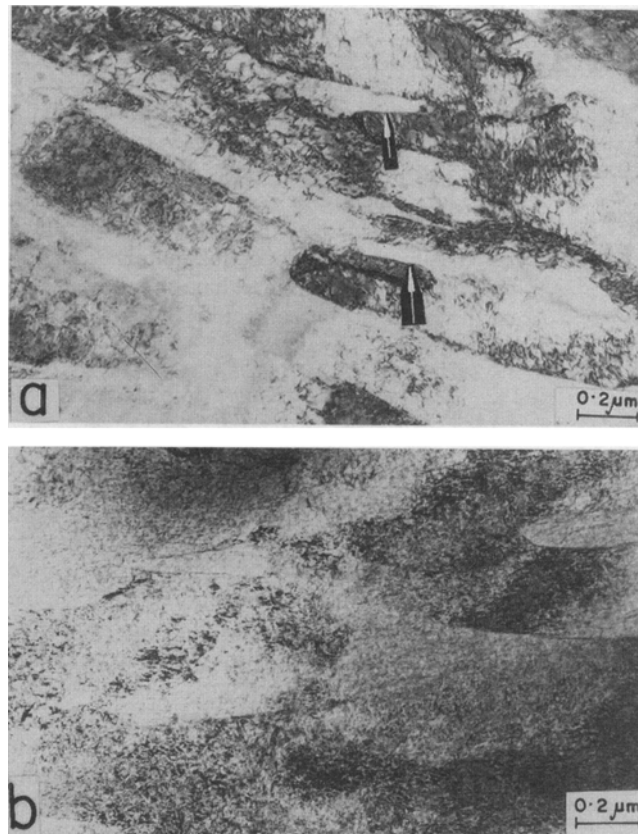


Fig. 2—Bright-field TEM micrograph of the alloy: (a) cycled five times and aged at 510 °C for 3 hours and (b) directly aged at 510 °C for 3 hours after solution annealing.

crographs are shown in Figures 2(a) and (b). The fine lamellae formed along the martensite lath boundaries (Figure 2(a)) were identified, using selected area diffraction (SAD) analysis, as austenite laths. The proportion of the austenite phase was found to increase with increasing numbers of cycles. The microstructure of the conventionally aged alloy (Figure 2(b)) consists essentially of lath martensite of 0.3-μm width. These laths lie parallel within the prior austenite grains. Retained austenite was not seen in the microstructure. The strengthening precipitates formed in this alloy during aging were identified as Ni₃, (Ti, Mo) and Fe₂Mo types. Their microstructural characterizations are reported elsewhere.^[7]

The results of X-ray diffraction analyses show that the maximum change in the volume fraction of austenite occurred during the first cycle and its fraction depended on the heating rate. At a heating rate of 9.5 °C/min, the first cycle was found to have introduced about 40 pct austenite. Thereafter, the austenite content was found to increase slowly with increasing number of cycles; about 53 pct austenite was measured after the fifth cycle. The corresponding amount of austenite at a heating rate of 6 °C/min was estimated to be about 66 pct at the end of the fifth cycle.

The mechanism of formation of austenite in maraging steel during thermal cycling has recently been investi-

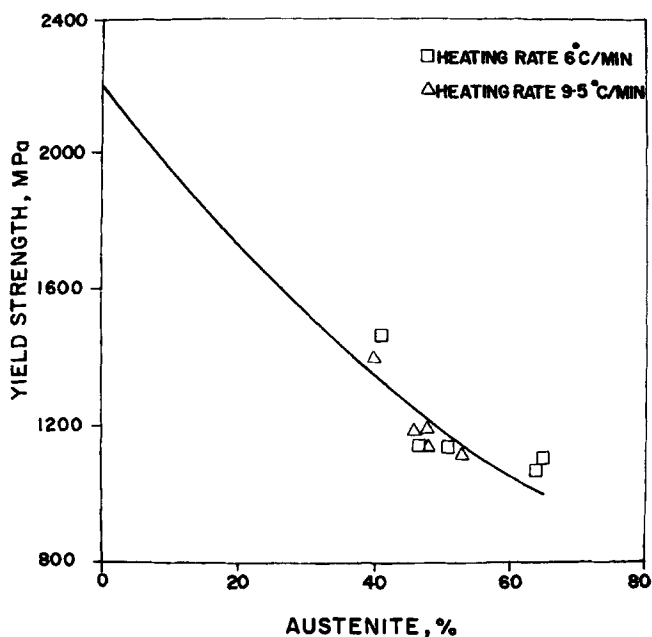


Fig. 3—The YS of cycled alloy as a function of austenite content.

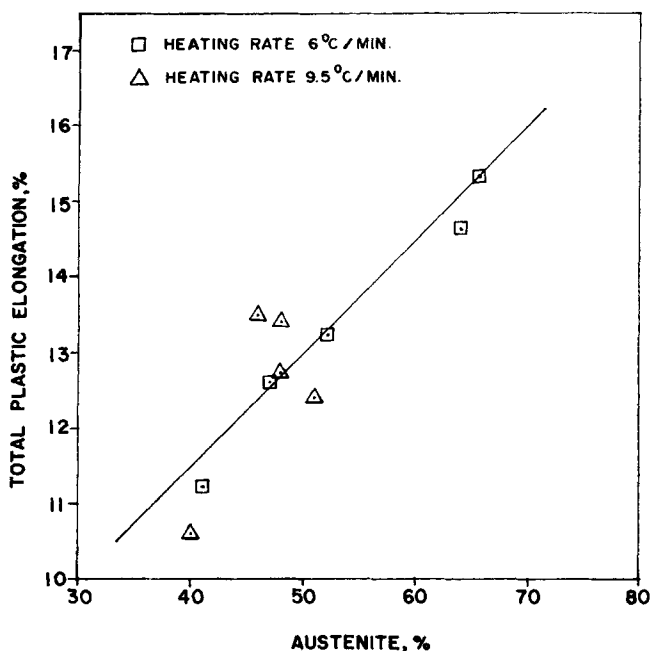


Fig. 4—The ductility of cycled alloy as a function of austenite content.

gated.^[8,9] When maraging steel is heated, a number of solid state reactions occur in different temperature regions, the first being the precipitation of intermetallic phases. On continued heating, these precipitates start dissolving, causing local enrichment of the matrix with respect to the alloying elements. These regions serve as the sites for subsequent nucleation of austenite. By using nonisothermal dilatometry, it has been reported^[10] that at the heating rates used in this work, the alloy is fully within the austenite single-phase region at 750 °C. On cooling, the solute-rich austenite remains untransformed, whereas the solute-lean austenite transforms to martensite.^[2]

The material in the conventionally aged condition possessed an ultimate tensile strength (UTS) of 2227 MPa, a yield strength (YS) of 2195 MPa, and a total elongation of 5.3 pct. With the introduction of retained austenite (treat-

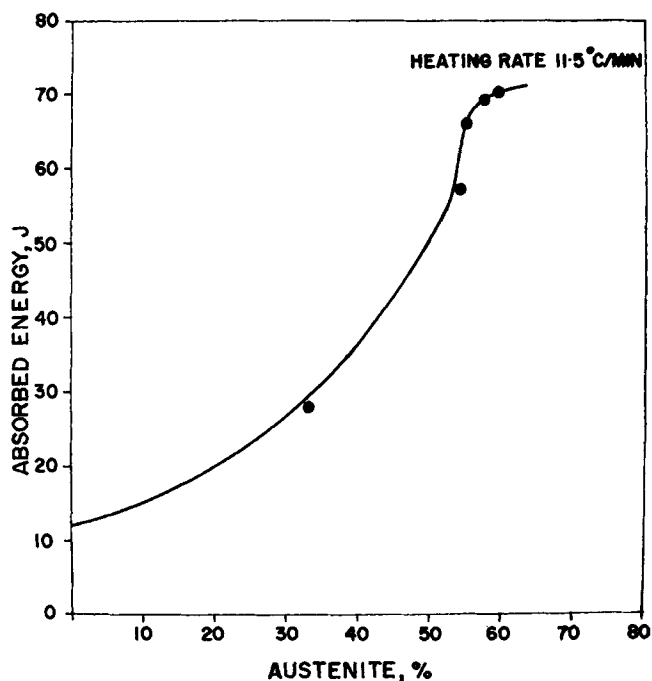


Fig. 5—The absorbed energy of cycled alloy as a function of austenite content.

ment 2), the UTS and YS decreased and the total elongation increased with increasing austenite content. The change in YS as a function of austenite is shown in Figure 3. Data from the specimens cycled at both heating rates (indicated by different symbols) could be fitted into a single exponential curve which could be described by the equation

$$\sigma_{YS} \text{ (MPa)} = A \exp(-1.2078 \times 10^{-2} \times \text{vol pct austenite}) \quad [1]$$

where A is 2195 MPa, the YS of conventionally aged material. An R^2 value of 0.96 was obtained for the fit.

The UTS also varied in a similar way with the austenite content and could be described by the equation

$$\sigma_{UTS} \text{ (MPa)} = B \exp(-1.0755 \times 10^{-2} \times \text{vol pct austenite}) \quad [2]$$

where B is 2227 MPa, the UTS of conventionally aged material. The R^2 value obtained in this case was 0.86.

Maraging steels derive their high strengths from the precipitation of intermetallic phases from a supersaturated martensitic matrix.^[1] Because austenite is a soft phase, it decreases the strength.^[2] Due to the higher solubility of alloying elements in austenite, the supersaturation of the martensite is significantly reduced, resulting in decreased precipitation of intermetallic phases during subsequent aging. This decrease in strength with increasing austenite content was accompanied by an appreciable increase in the ductility, as shown in Figure 4. When total plastic elongation is plotted against the austenite content, the ductility of the material is found to increase with austenite content.

Figure 5 shows the change in the RT absorbed energy of cycled specimens as a function of austenite content. It can be noted that the toughness increased significantly with increasing austenite content.

The mechanical stability of the austenite at subambient temperatures was examined by carrying out low-temperature tension and impact tests on specimens containing 55

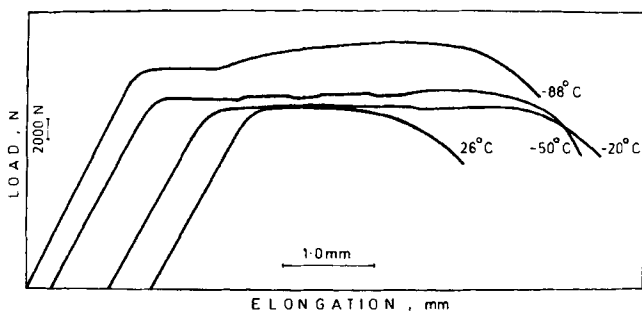


Fig. 6—The load-elongation plot of cycled alloy obtained at different testing temperatures.

pct austenite introduced through thermal cycling. Figure 6 shows the load-elongation plots obtained during tension tests at different temperatures. At RT, the load-elongation plot was a smooth one. As the temperature was decreased, a number of load fluctuations appeared on these plots. At -20°C , the load remained almost steady until a load drop occurred after an elongation of 11 pct. Subsequently, the material exhibited an improved work hardening until the onset of necking. At -50°C , after about 5 pct of elongation, a number of steps appeared on the plot. At -88°C , the load-elongation plot indicated a substantial increase in work hardening.

Deformation-induced martensitic transformation is known to occur in austenitic stainless steels at temperatures well above the martensite transformation temperature.^[11] The former is a complex phenomenon involving factors like degree of deformation, deformation temperature, and chemical composition of austenite. Retained austenite in maraging steel also is found to undergo similar transformation during working.^[12,13] The behavior noticed during the tension tests in this work can be explained in terms of the mechanical instability of the austenite.

The austenite formed during the first cycle is expected to be more stable due to its high alloy content. The load drop noticed on the load-elongation plot at -20°C could be associated with the deformation-induced transformation of the austenite into martensite. The different steps noticed on the load-elongation plot at -50°C could be due to the transformation of austenite grains of varying compositions. However, at -88°C , the load-elongation plot did not show similar steps and the transformation was manifested as a pronounced increase in work hardening.

The changes in the tensile properties as a function of test temperature are shown in Figure 7. The UTS and the YS increased with decreasing temperature. However, the decrease in strength was accompanied by an increase in ductility, as shown by the continuous increase in both the uniform and the total elongation with decreasing temperature. The increase in ductility can be attributed to the delayed onset of necking of the specimen, which occurs because of the increased work hardening of the retained austenite as it transforms to martensite when adequate localized stress and strain levels are achieved. Thus, an alloy with a controlled fraction of retained austenite in a martensitic matrix can be tailored to give increased toughness at low temperatures. The strength of the martensite increases with decreasing temperature, whereas the unstable austenite gives higher ductility.

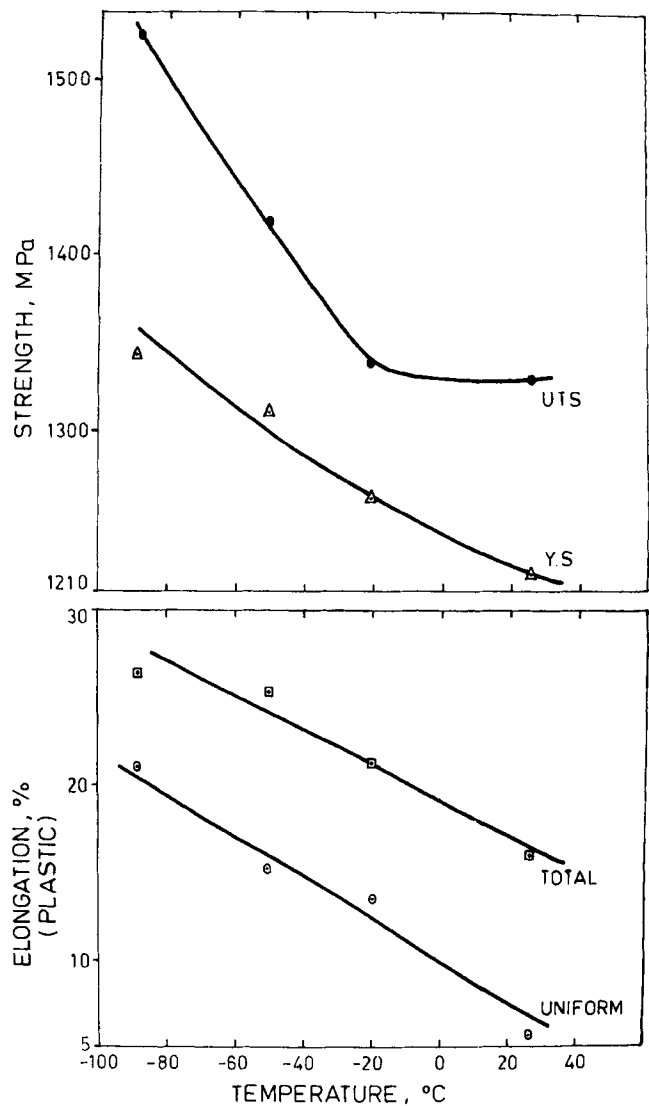


Fig. 7—The UTS, YS, and plastic elongation of cycled alloy as functions of test temperature.

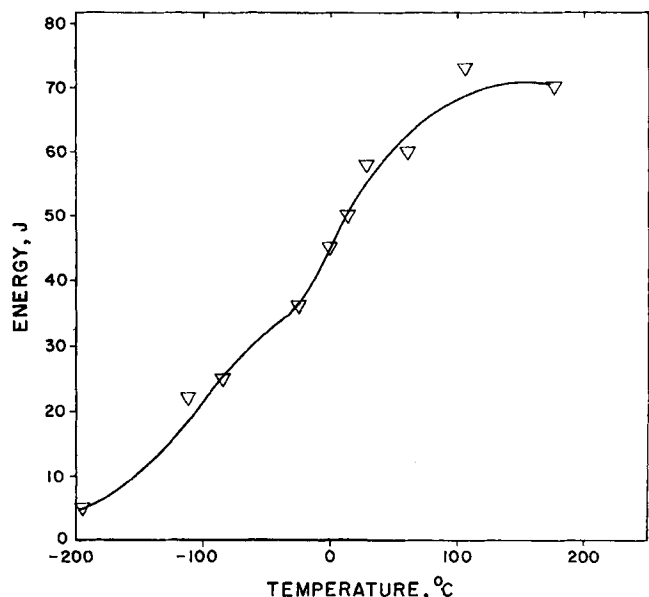


Fig. 8—The absorbed energy of cycled alloy as a function of test temperature.

The absorbed energy of cycled specimens containing 54 pct austenite plotted against the test temperature is shown in Figure 8. The absorbed energy increased with increasing test temperature, and the material showed significant residual toughness even at subambient temperatures.

Present work shows that the retained austenite formed during thermal cycling is beneficial in improving the ductility and toughness of 350-grade maraging steel. Wide variations of strength and ductility over a range of subambient temperatures can be obtained in this material by controlling the fraction of retained austenite through thermal cycling before aging.

IV. CONCLUSIONS

Based on the present investigation, the following conclusions are drawn.

1. Thermal cycling is an effective heat treatment for introducing retained austenite into a 350-grade maraging steel. The volume fraction of the austenite depends on the heating rate; the lower the heating rate, the higher is the fraction.
2. The retained austenite reduces the UTS and YS. The UTS and YS can be correlated to the volume fraction of austenite by exponential equations.
3. The presence of austenite substantially increases ductility and toughness.
4. The retained austenite is unstable during deformation at subambient temperatures, and the instability increases with decreasing temperature.
5. An excellent combination of strength and ductility can be achieved at subambient temperatures in this material by thermal cycling. The low-temperature mechanical properties can be tailored by controlling the fraction of

retained austenite through appropriate selection of heating rates during thermal cycling.

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