Tensile Properties of an Age-hardenable Fe-16Ni-4Si Alloy with Duplex Microstructures*

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Synopsis

An Fe-16%Ni-4%Si alloy having a large hardenability due to a maraging mechanism was used to study the effect of microstructures present prior to aging upon the properties of tensile strength. In an attempt to obtain microduplex structures showing a good combination of strength and toughness, the present alloy was cold rolled by 80% at a martensitic state (i.e., marforming) after conventional solution treatment in the γ field, and then annealed at the temperatures in the $\alpha + \gamma$ two-phase range followed by air cooling, and finally aged.

The marforming was found to increase both the tensile strength (σ_u) and the reduction of area (φ) due to the formation of proper duplex structures. Large uniform elongation (ε_u) was observed after annealing at the temperatures around 570°C, and this was attributable to a transformation plasticity of precipitated austenite which was stabilized thermally by partitioning of Ni. The strength, however, was rather low and $\sigma_u = 120 \text{ kg/mm}^2$. When annealed at a temperature range between 600°C and 650°C (still in the two-phase range), the reverted austenite was transformed on cooling into martensite which was strengthened by aging proportionally to the martensite fraction; the resultant duplex (martensite + ferrite) structure had a strength of $\sigma_u = 158 \text{ kg/mm}^2$, and a plasticity of $\varphi = 27\%$ and $\varepsilon_u = 1\%$, which were better than the combination, $\sigma_u = 132 \text{ kg/mm}^2$, $\varphi = 25\%$ and $\varepsilon_u = 1\%$ in the same alloy that was age-hardened conventionally without marforming and annealing.

The marforming had a dominant effect of reducing transgranular brittle fracture as well as intergranular fracture associated with preexisting coarse austenite grains.

I. Introduction

Plastic working of unaged martensite is known as marforming,¹⁾ and is an effective strengthening means of age-hardenable ultrahigh strength steels such as maraging steels. One of the merits of marforming is to easily increase the strength of unaged soft martensite. This fact has been described by Decker *et al.*^{1,2)} in the earliest reports of the maraging steels, but the increase in the strength *per se* has been assessed as not so large as in ausforming of ultrahigh strength low alloy steels containing carbon.^{3,4)} Recent studies have revealed another merit of marforming in the light of toughness improvement. Soeno *et al.* have shown that notch tensile strength of maraging steels, which were strengthened by marforming and aging, was larger than other steels strengthened by aging alone, even though their tensile strength was the same.⁵⁾ Marforming is also described as it extends greatly the life of sustained load tests for stress corrosion cracking or hydrogen embrittlement.⁶⁻⁸⁾

These beneficial properties of marforming will still remain when some annealings are made following the marforming. It is well-known that recrystallization of deformed martensite takes place at temperatures higher than A_f , leading to fine austenite grains.⁵⁾

This method is in effect attractive since the prior austenite grains need to be refined within a critical size depending on the strength level in order to assure an acceptable level of toughness.^{9,10)} One of the purposes of the present study is to reduce the grain size further by annealing a marformed steel in the two-phase ($\alpha + \gamma$) range (i.e., below A_{c3}) as well as in the γ field, and to examine the tensile properties of so-called microduplex¹¹⁾ age-hardenable structure. Reverse martensitic transformation behavior in the dual phase field has been described in a recent paper by Hosomi *et al.*,¹²⁾ and the comparison with the present study will be made in a later section.

The material used in this work was an Fe-16%Ni-4%Si alloy, free of carbon. One of the reasons to use this alloy was that it not only has strong age hardenability after solution treatment in the γ range,¹³⁻¹⁵⁾ but still has considerable hardenability after cooling from dual phase range.¹⁶⁾ Another reason was that the enrichment of silicon in a bcc phase may occur due to partitioning during the dual phase annealing, thereby strengthening the otherwise weak phase.

II. Experimental Procedure

Raw materials were electrolytic iron and nickel, and metallic silicon, which were vacuum induction melted, hot forged and hot rolled at 1200°C into sheet 4 mm thick and air cooled. Chemical analysis was made at the hot-rolled state, together with transformation temperature measurement by a dilatometric method, and the results are shown in Tables 1 and 2, respectively. The structure was dislocated lath martensite, and no retained austenite phase was detected with X-ray diffraction.

Thermal-mechanical processes are shown in Figs. 1(a) and (b). The hot-rolled sheet was cut into small specimens. In the process (a), the specimens were homogenized at 1200°C for 8 to 24 h in evacuated capsules, and water quenched. The martensite thus formed is known to have intense age hardenability.¹³⁻¹⁵⁾ In the present work, the martensite was cold rolled by 80%, and the "rolled" specimen will be designated as "R". The specimens were then annealed in argon-hydrogen gas mixture for 2 h at several temperatures up to 820°C, air cooled, and then "aged" at 420°C for 2 h. This annealing process is essential in the present work. As-annealed or annealed-and-aged specimens which are annealed at 820°C, for example, will be indicated by R820 or

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Table 1. Chemical composition. (wt%)

Ni	Si	C	Mn	S	Fe
15.6	3.7	0.007	0.001	0.004	Bul.

Table 2. Transformation temperatures. (°C)

<i>As</i>	<i>Af</i>	<i>Ms</i>	<i>Mf</i>
655	675	335	240

(a) "Rolled" specimen

(b) "Homogenized" specimen

Fig. 1. Heat patterns for two kinds of thermal-mechanical processes used in this study; in (a) "rolled" specimens (R) were marformed before annealing and aging, while no deformation was made in "homogenized" specimens (H) in (b).

RA820, where A means the specimens are aged finally.

On the other hand, undeformed case is shown in Fig. 1(b) for comparison. The hot-rolled sheet was cold rolled by 80% before homogenization treatment at 1 200°C, thus having the same final thickness as the R-specimens. The specimens of this series will be indicated by a letter H (Homogenized) as H820, HA820, etc.

In order to observe a general tendency of recovery of marformed structures and the states of phases formed during annealing in R-specimens, the line broadening parameter (seen in Fig. 2) of the 222 X-ray diffraction line of martensite or of other BCC phases was measured with MoK α radiation, similarly to the previous reports.^{17,18)} Diffraction intensity may come from following BCC phases: (1) martensite phase (α') preexisted before annealing, (2) ferrite phase (α) which was formed during annealing and (3) another martensite phase (α'') which was formed from reverted austenite on cooling from the annealing temperatures, as shown in Table 3. During the dual phase annealing, partitioning of alloying elements is considered to take place; Si will be enriched in α phase and Ni in γ and α'' phases, and this will make some contribution to the thermal stability or mechanical properties.

Fig. 2. Variation of line broadening parameter, Δ , and retained austenite fraction, $F(\gamma)$, in rolled specimen (R), after annealing as a function of the annealing temperature, T .

Volume fraction of retained austenite after each annealing was determined by a similar method used by Miller,¹⁹⁾ as described previously.¹⁸⁾ Metallographic observations and Vickers hardness measurement were also made in a conventional manner.

In order to measure room temperature tensile properties, rectangular (tetragonal prism) tensile specimens 4 mm wide, 60 mm long, 0.8 mm thick and 40 mm in effective gauge length, were cut from as-rolled (R) or as-homogenized (H) specimens before annealing. The tensile direction was normally parallel to the rolling direction unless noted otherwise. Tensile tests were made with Instron type machine with a crosshead speed of 0.5 mm/min.

III. Experimental Results and Discussion

1. Change in Structures of Rolled (R-) Specimens during Annealing

In Fig. 2 the change in X-ray line broadening parameter Δ and retained austenite fraction $F(\gamma)$ of the rolled (R-) specimens as a function of annealing temperature are shown. The variation of Vickers hardness Hv is shown in Fig. 3. Transmission electron micrographs of representative annealing structures are exhibited in Photo. 1. From these data, one may conclude the following six annealing stages: As shown in Table 3, Stage I is characterized by the decrease in Hv and in Δ which will mean recovery of deformed martensite α' . In Stage II the Δ still diminishes while the Hv increases to the maximum age hardness $Hv=483$ at around 420°C. Above 450°C softening is remarkable, but no sign of austenite formation is detected upto 510°C. Dislocation density decrease is noted, but subgrain formation is not recognized in the deformed martensite by transmission electron microscopy (TEM).

The maximum hardness value mentioned above is an expectable one from the result by Kanao *et al.*¹⁴⁾ As to the age hardening mechanisms, Yasunaka and Araki¹⁵⁾ have attributed to the clustering or precipita-

tion of compounds such as $(\text{Fe,Ni})_3\text{Si}$ rather than ordering of matrix, though no further investigation

Fig. 3. Change in Vickers hardness, H_v , of rolled specimen (R), with annealing temperature, T . HT or CR means homogenization-treated or cold-rolled state, respectively.

was performed in this work. Retained austenite is formed and its amount increases with temperature in Stage III. TEM observation has shown corresponding increase in the tendency of formation of ultrafine grain duplex ($\gamma + \alpha$) structure with the increase of temperature. Most of the grains are free of dislocations below 600°C. However, in Stage IV (above 600°C), some of the grains are dislocated densely, and the fraction of such grains increases with temperature until 650°C where most grains have a high dislocation density. Corresponding to the decrease in $F(\gamma)$ in Stage IV, the Δ increases abruptly as the temperature exceeds 600°C, as seen in Fig. 2. This will provide the following explanation;^{17,20)} the annealing in the two-phase range is expected to form a duplex structure composed of a Ni enriched austenite phase and Si-rich Ni-poor ferrite phase from a thermodynamic consideration. The Ni concentration in the γ phase decreases monotonically

Photo. 1. Transmission electron micrographs of rolled specimens (R); (a) as 80% rolled (RO), (b) annealed at 420°C (R420 or RA0), (c) at 500°C (R500), (d) at 550°C (R550), (e) at 600°C (R600), and (f) at 650°C (R650).

Table 3. Classification of alloy structures at various annealing temperatures in marformed specimen.

Stage	Temperature range (°C)	Characteristic phenomena	Structure	
			At annealing temp.	After cooling
I	r.t.~360	Recovery of defects in martensite (α')*	α'	α'
II	360~510	Hardening and softening due to maraging	α'	α'
III	510~600	Formation of stable austenite (γ) & ferrite (α)	$\gamma + \alpha$	$\gamma + \alpha$
IV	600~650	Formation of stable and unstable austenite and ferrite	$\gamma + \alpha$	$(\gamma + \alpha'')^{**} + \alpha$
V	650~675 (A_f)	Formation of unstable austenite and ferrite	$\gamma + \alpha$	$\alpha'' + \alpha$
VI	675~ (A_f)	Recrystallization of γ which reverted from α'	γ	α''

* α' : Primary martensite, deformed or undeformed.
** α'' : Martensite formed on cooling from unstable austenite.

cally with annealing temperature, thereby the stability of the γ decreases with the temperature. Since the $F(\gamma)$ is measured at room temperature after each annealing, the onset of instability is detected by TEM technique at the annealing temperature of 600°C, and the γ becomes completely unstable and transformed into dislocated martensite (α'') at or above 650°C (Stage V) accompanied with marked increase in Δ . This explanation is based essentially on the same standpoint as the chemical stabilization effect by Hosomi *et al.*¹²⁾ who came to the conclusion from the measurement of reverse transformation temperatures after the dual phase range annealing of maraging steels. In Stage VI (above A_f), no elemental partitioning will take place since reverse transformation occurs before the phase separation. Figure 4 shows the variation of grain size below and above the A_f . Above A_f the reverted austenite from deformed martensite is subject to conventional recrystallization in the γ range. Ultrafine grain size below A_f is an indication of microduplex structure.

2. Tensile Deformation Behavior

1. Strength and Ductility of Rolled (R-) Specimens

Typical stress strain curves in the rolled (R-)specimens are shown in Fig. 5 for several annealing temperatures. The elongation to fracture is generally low for high strength specimens. RA-specimens are generally stronger than R-specimens as a result of age hardening. The elongation as well as the strength are increased by aging in some cases, (for example, annealing at 820°C or 570°C).

Annealing temperature dependence of the 0.2% proof stress $\sigma_{0.2}$, tensile strength σ_u and uniform elongation ϵ_u is shown in Fig. 6. Softening is observed at temperatures above 450°C but still in Stage II, and this is primarily due to overaging or coarsening of precipitates, since no austenite is formed at this temperature range. The lowest value of $\sigma_{0.2}$ or σ_u was observed at the annealing temperature of 590°C or 570°C, respectively. These temperatures are slightly

lower than 600°C where the retained austenite content is maximum. It has been concluded to be the TRIP effect²¹⁻²⁵⁾ that the ϵ_u exhibits abnormal increase at around 570°C. This is because the annealing-induced austenite, which composes the microduplex structure, is thermally stable at room temperature, but is mechanically unstable, resulting in martensitic transformation during tensile deformation. Measurement of $F(\gamma)$ after the tensile deformation also supported this view. The stress strain curves obtained in this temperature range have revealed inhomogeneous (Lüders) deformation as seen in Fig. 5, which has been ascribed as an indication of TRIP.²¹⁾

Difference in the strength of R- and RA-specimens (*i.e.*, hardenability due to aging) is not large below 600°C, but it increases as the annealing temperature rises from 600°C to 650°C (Fig. 6), where the fraction

Fig. 5. Typical stress-strain curves of rolled and annealed specimens with or without final aging. R and RA mean as-annealed and annealed-and-aged specimens, respectively; numerals show the annealing temperature. Cross marks indicate fast fracture.

Fig. 4. Variation of grain size in rolled and annealed specimen as a function of annealing temperature near A_f .

Fig. 6. Variation of 0.2% proof stress, $\sigma_{0.2}$, ultimate tensile strength, σ_u , and uniform elongation, ϵ_u of rolled specimen as a function of annealing temperature, T .

of α'' martensite phase increases (Table 3). Figure 7 shows the strength difference $\Delta\sigma_u$ between R and RA as related with the fraction of α'' martensite. This figure strongly suggests that the cause of age hardening in this temperature range (Stage IV) is primarily due to the maraging phenomenon of α'' martensite which has been transformed on cooling from annealing-induced Ni-rich austenite.

2. Tensile Fracture Modes and Comparisons with Undeformed (H-) Specimens

Figure 8 shows the change in the reduction of area with annealing temperature, together with $\sigma_{0.2}$ and σ_u in R-, RA-, H- and HA-specimens. The ϕ is used for a measure of toughness among some quantities available from smooth tensile specimens. The strength of deformed specimens (*i.e.*, R and RA) is higher in general than that of undeformed (*i.e.*, H and HA) specimens, particularly below 500°C where no structural change due to annealing was found by TEM observations. The ϕ of H-specimens is much higher than that of R-specimens at least below 300°C, owing to the lower strength of the former. Stress-strain curves of H-specimen did not show clear yield point before annealing. This, together with the low $\sigma_{0.2}$ value of 85 kg/mm², indicates that dislocations in undeformed martensite have high mobility. Microvoid coalescence (MVC) was the fracture mode in this case.

The MVC was also a general mode of fracture in as rolled R-specimens, and dimple pattern was always observed on the fractured surface perpendicular to the tensile axis. Vertical separation or delamination was also observed along planes nearly parallel the rolled surfaces. Such fracture mode, *i.e.*, MVC with separation, was characteristic to the R-specimen unless the annealing temperature exceeded 500°C. Some examples are shown in Photos. 2(a) and (b). In H-specimens, on the other hand, characteristic intergranular brittle fracture was observed when annealed between 420°C and 550°C. The fracture took place along boundaries of prior austenite grains formed in homogenization treatment, and the grain size was as large as 150 μ m.

Observed increase in ϕ in $T \geq 500^\circ\text{C}$ is associated

Fig. 7. Relation between the strength increase, $\Delta\sigma_u$, due to aging and the martensite fraction, $F(\alpha'')$, before aging the specimens rolled and annealed at the temperatures in Stages IV and V.

with the decrease in strength due to the recovery of defects and the formation of duplex structure. This increase in ϕ in R-specimens was seen to occur in a temperature range about 50°C lower than that for H-specimens. This may be ascribed simply to the enhanced annealing process due to marforming. Essential difference, however, between the fracture surfaces of R-specimens (Photo. 2) and H-specimens (Photo. 3) is observed; in R-specimens the fracture mode of the duplex structure is of MVC, whereas in H-specimens intergranular and quasi-cleavage brittle fracture associated with prior austenite grains were present together with partial dimple patterns (Photo. 3(b)). The same is true for Stage VI. These observations imply that the undeformed H-specimens have certain kinds of weakness associated with original coarse grain structure, and the weakness is never destroyed during the annealing process. This may partly be due to a reversible nature of $\alpha' \leftrightarrow \gamma$ transformation and also due to impurity segregation or precipitation along prior austenite grain boundaries.

Figure 9 shows the relations between σ_u and ϕ obtained in this study. RA-specimens indicated by solid circles have better combinations of strength and reduction of area than HA-specimens, and microduplex ($\alpha'' + \alpha$) structure has higher strength than single α'' structure at a same σ_u value.

Anisotropy in the tensile properties was also measured in R- and RA-specimens. Plastic anisotropy (*r*-value) characterizing the texture development was found to be different by 50~100% between longitudinal (L) and transverse (T) directions, but anisotropy of $\sigma_{0.2}$ or σ_u was negligibly small. As for ϕ the value for L direction was twice as large as that for T direction when $T < Af$, whereas the former was smaller than the latter when $T > Af$. In spite of such complication, various merits of marforming mentioned

Fig. 8. Change in the reduction of area, ϕ , σ_u and $\sigma_{0.2}$ as a function of annealing temperature, T , in rolled R- or RA-specimens and in homogenized H- or HA-specimens.

Photo. 2. Scanning electron micrographs of fracture surfaces in rolled-annealed-aged specimens with different annealing temperatures T ; (a) and (b) no annealing (RA0), (c) $T=590^{\circ}\text{C}$ (RA590), (d) $T=630^{\circ}\text{C}$ (RA630), (e) $T=720^{\circ}\text{C}$ (RA720), and (f) $T=820^{\circ}\text{C}$ (RA820).

Photo. 3. Scanning electron micrographs of fracture surfaces in homogenized-annealed-aged specimens with different annealing temperatures T ; (a) no annealing (HA0), (b) $T=600^{\circ}\text{C}$ (HA600), and (c) $T=750^{\circ}\text{C}$ (HA750).

Fig. 9. Relation between ϕ and σ_u in typical specimens used in this work. Beneficial effects of marforming and annealing in the two-phase field to form microduplex structures are evident at a finally aged condition. Numerals and zeros mean the annealing temperatures in degree C and without annealing, respectively.

above was confirmed never due to an apparent effect of crystalline texture.

3. Discussion on the Effects of Marforming and of Annealing

As described in the previous sections, attractive natures of marforming are found even in annealed specimens as well as in as marformed ones. In case the annealing is conducted below 500°C , strength increase due to marforming still remains sizable, and at the same time premature brittle failure along prior austenite grain boundaries is suppressed effectively. The fractured surfaces have consisted of dimple pattern on surfaces perpendicular to the tensile axis and of separations along planes parallel to the rolling surfaces. These separations are believed to improve the toughness apparently based on a geometrical effect, but this effect is not considered to account for the major part of the increased toughness, since no separa-

tions were observed in specimens annealed above 500°C where intrinsic effect of marforming was still observed. More essential influence will be the macroscopic structural inhomogeneity, such as deformation bands. The rolling reduction of 80% is high enough for the deformation band formation.²⁷⁾ The crystals are fragmented by the bands, and high density of mobile dislocations associated with the fragmentation are responsible for the reduction of stress concentration.

In annealed specimens annealed above 500°C, the marformed structure itself is destroyed by the annealing, and microduplex structure is formed, but the mechanical properties are better than the undeformed H-specimens. In H-specimens, nucleation in recrystallization or phase transformation is much restricted kinetically and the nucleation sites are confined to the prior austenite grain boundaries. Furthermore, transformation tends to occur reversibly, so that after annealing the martensite α'' has the same orientation as the original martensite α' . Thus even though apparent grains were refined, the annealing is not expected to reduce the unit fracture volume where the crystals have a similar orientations.^{28,29)} Fragmented structure has a lot of deformation bands which are responsible for new nucleation sites on annealing.^{27,30,31)} Variety of crystal orientations will be formed after reverse and forward transformations, since the reversibility of orientation will be destroyed. Metallographically, austenite grains formed are spherical in the marformed specimens, in contrast to directional morphology in the undeformed ones.²⁰⁾ In this way the fragmented structure is considered to produce fine grains or dense boundaries which are effective to suppress the brittle fracture. This view is supported by the present observation that the ϕ , as a measure of toughness, as well as the strength was increased (Fig. 8), and brittle fractures along prior austenite boundaries or cleavage planes disappeared (Photos. 2 and 3) even by annealing the marformed specimens either in two-phase or in single γ phase range. The annealing gives a number of beneficial effects on the strength properties without loss of ductility inherent to marforming alone.

IV. Conclusion

An age-hardenable Fe-16Ni-4Si martensitic alloy is used to study the effects of 80% of cold rolling (marforming) followed by annealing in the two-phase ($\alpha+\gamma$) field prior to the final aging. Tensile properties were analyzed with the electron metallographic observations as well as X-ray diffraction analysis. Following conclusions may be drawn:

(1) Large uniform elongation due to the TRIP effect was observed in specimens having ($\gamma+\alpha$) microduplex structure which was formed on annealing between 540°C and 610°C. The temperature range was near or slightly lower than 600°C where the volume fraction of retained austenite had the maximum. The strength, however, was rather low in specimens either with or without aging.

(2) The strength increase due to aging was in-

creased with the volume fraction of unstable austenite formed during annealing in the two-phase range, but was negligibly small in the case of stable austenite formation. This will explain the mechanism of age hardening as entirely due to the maraging phenomenon of martensite (α'') which transformed from unstable γ phase on the way of cooling from the two-phase field.

(3) Marforming has a beneficial effect of increasing the strength and toughness of aged specimens, with or without succeeding annealing. As to the microduplex structure of marformed specimens, remarkable suppression of intergranular or transgranular fracture inherent to conventional microduplex structure was found. This is discussed on the basis of crystal fragmentation in marformed specimens.

(4) From these properties, a new processing method is proposed. Ultra-high strength steels with maraging type age-hardenability can be strengthened by applying marforming and annealing below A_{c3} temperature to form microduplex structure prior to aging.

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