



Redesign of maraging steel: Realization of lightweightness and cost-efficiency without sacrificing mechanical properties

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ABSTRACT

Maraging steels are ultrahigh-strength materials with a robust α' martensitic matrix hardened by nanoscale intermetallic precipitates and are used in demanding structural applications. However, conventional maraging steels contain substantial amounts of alloying elements such as Ni, Co, and Mo (>20 wt%), which limit mass production and broader adoption due to high material costs and high density. Here, we introduce a new maraging steel with a low-Ni, Co-free, and Mo-free composition (Fe–5Mn–9Ni–4Al–0.04C) designed to overcome these limitations. This steel offers improved economic feasibility and reduced density through lean Ni content, cost-effective Mn substitution, and the addition of lightweight Al. Moreover, it exhibits an excellent strength–ductility balance exceeding 25,000 MPa% while maintaining high strength above ~ 1.5 GPa, enabled by the formation of nanoscale Ni(Al, Mn) B2 precipitates and a high fraction of retained austenite. This alloy therefore provides a promising route to expand the practical use of maraging steels in broader structural applications.

1. Introduction

With the continued development of modern industrial structures, high-strength structural materials that combine low density with high ductility are increasingly important [1–3]. Among such materials, maraging steels—characterized by a martensitic matrix strengthened by precipitate formation during the aging process—are widely used as ultrahigh-strength alloys [4,5], particularly in demanding applications in the aerospace and defense industries (e.g., landing gear, missile casings, and rocket motor cases) [1,6]. Commercial maraging steels generally contain high levels of alloying elements, including Ni (17%–19%, by weight), Co (8%–12%), Mo (3%–5%), Ti (0.2%–1.8%), Al (0.1%–0.15%), and C (<0.03%), to promote martensitic transformation upon cooling and to form intermetallic precipitates during aging [4,7]. These alloys typically exhibit a fully α' martensitic microstructure and exhibit marginal tensile strength (TS) values of 1000–1150 MPa with total elongations (TEs) of 17–19% prior to aging (e.g., in the as-hot-rolled, solution-treated, or austenitized condition) [8]. During aging, the fine martensitic hierarchical structure, high dislocation density, and supersaturated solid solution promote a homogeneous

distribution of nanoscale intermetallic precipitates (e.g., Ni₃Mo, Ni₃Ti, and Fe₂Mo) within the α' matrix [4,9], thereby increasing TS to 1300–2400 MPa, albeit with reduced TEs of 5–12% [8,10].

Despite their exceptional strength, maraging steels are not well suited for mass production or widespread use as common structural materials due to their high cost and density, which stem from the substantial additions of expensive and heavy alloying elements such as Ni and Co. To overcome these limitations, low-Ni and/or Co-free maraging steels have been proposed as alternative materials [2,10–13]. For example, Tharian et al. [12] investigated low-Ni, Co-free maraging steels with compositions of Fe–(11.5–13.5)Ni–(3.0–5.1)Mo–(3.2–5.0)Cr–0.1Al–(0.75–1.0)Ti (wt.%) and reported TS values of 1576–1700 MPa, comparable to those of conventional Co-bearing maraging steels, together with TE values of 10–12% after aging at 480 °C for 3 h. The high strength was attributed to precipitation hardening associated with Mo and Ti additions; however, direct microstructural evidence supporting this mechanism was not provided. He et al. [11] reported that a Co-free maraging steel with a composition of Fe–18.9Ni–4.1Mo–1.9Ti exhibited TS comparable to Co-bearing maraging steels (>2100 MPa) and a TE of $\sim 10\%$ after aging at 480 °C for 12 h, owing to a

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homogeneous distribution of needle-shaped η -Ni₃Ti precipitates. Sha et al. [13] developed a low-Ni and Co-free maraging steel with a composition of Fe-12.94Ni-1.61Al-1.01Mo-0.23Nb-0.2Cr-0.05C and evaluated its tensile properties after aging at 600 °C for 4 h. The aged steel exhibited a TS of ~1177 MPa and a TE of ~14.5%, indicating lower strength but higher ductility than conventional Co-bearing maraging steels. More recently, Jiang et al. [2] introduced a Co-free maraging steel with a composition of Fe-18Ni-3Al-4Mo-0.8Nb-0.08C-0.01B. After aging, the steel reached a TS of 2197 MPa with a TE of 8.2%, attributed to highly dispersed coherent B2-Ni(Al, Fe) precipitates. Collectively, these studies indicate that low-Ni, Co-free maraging steels can be cost-competitive alternatives to conventional Co-bearing maraging steels. However, their absolute alloy contents still include substantial fractions of expensive elements, notably Ni, Mo, and Ti. Moreover, although strength levels comparable to conventional Co-bearing maraging steels can be achieved, ductility generally remains limited. These limitations highlight the need for new alloy design strategies that simultaneously improve economic feasibility and the strength–ductility balance of maraging steels.

Here, we propose a novel alloy design strategy for a new maraging steel that can serve as an alternative to conventional maraging steels by simultaneously improving economic feasibility, enhancing the strength–ductility balance, and reducing density. The designed steel has a chemical composition of Fe-5Mn-9Ni-4Al-0.04C and contains less Ni and no Co or Mo, resulting in an approximately 80% lower material cost (2.19 USD/kg) than conventional Co-bearing maraging steels (10.79 USD/kg). Despite its relatively low alloying content, this steel exhibits an excellent strength–ductility balance exceeding 25,000 MPa% after aging while maintaining high strength above ~1.5 GPa. The high strength is primarily attributed to homogeneously distributed spherical B2 Ni(Al, Mn) precipitates within the α' matrix, together with rod-like B2 precipitates formed in the retained austenite (γ_R) phase via a discontinuous precipitation-like mechanism during γ reversion and growth. Discontinuous precipitation-like morphologies generally degrade ductility in steels and alloys due to their brittle nature. However, the present steel maintains excellent ductility primarily owing to a high fraction of ductile γ_R stabilized by Mn addition and an active twinning-induced plasticity (TWIP) deformation, which effectively mitigates the detrimental influence of the discontinuous precipitation-like morphology. Moreover, the addition of the lightweight element Al reduces the density of the steel to 7.73 g/cm³, corresponding to a reduction of approximately 6.5% relative to conventional Co-bearing maraging steels (8.27 g/cm³). Collectively, these advantages highlight the potential of the present alloy as a cost-efficient, lightweight, and high-performance alternative to conventional maraging steels.

2. Methods

2.1. Materials preparation

An ingot with dimensions of 12 × 7 × 2.4 cm³ was fabricated using a vacuum induction melting furnace and subsequently homogenized at 1100 °C for 2 h under an Ar atmosphere. The actual chemical composition of the ingot was determined to be approximately Fe-4.83Mn-9.25Ni-4.28Al-0.04C (wt.%) using a spark optical emission spectrometer (Spark-OES, OBLF Spektrometrie QSN750) for Mn, Ni, and Al, and a carbon/sulfur analyzer (CS analyzer, Leco CS600) for C. The ingot was hot-rolled between 1100 and 900 °C to a final thickness of ~3.0 mm and then water-quenched to ambient temperature. In addition, selected sections of the hot-rolled plate were austenitized at 900 °C for 10 min and water-quenched to ambient temperature. Both the hot-rolled and austenitized samples were aged at 600, 620, and 640 °C for 30 min and subsequently water-quenched to ambient temperature. The temperatures used for hot rolling, austenitizing, and aging were selected based on thermodynamic calculations performed with Thermo-Calc 2023b software using the TCFe12 database.

2.2. Materials characterization

The morphology, grain size, and phase constituents of the steels were examined using scanning electron microscopy (SEM) equipped with an electron backscatter diffraction (EBSD) detector. Martensitic laths and precipitates were visualized by electron channeling contrast (ECC) imaging using a backscattered electron detector in the SEM. General SEM observations (Supplementary Fig. 3 and Fig. 7b–e) were conducted using an FEI Nova NanoSEM 450. Combined EBSD–ECC analyses of undeformed samples (Fig. 2f–i) were performed using a ZEISS Merlin Compact SEM equipped with an Oxford NordlysNano EBSD. ECC imaging of undeformed samples (Fig. 2e and Supplementary Fig. 5b) and EBSD analyses of deformed samples (Fig. 7g–l) were performed using a ZEISS GeminiSEM 560 SEM equipped with an Oxford Symmetry S2 EBSD. ECC imaging was performed at accelerating voltages of 10–15 kV and working distances of 5–6 mm. EBSD analyses of undeformed samples were conducted at an accelerating voltage of 20 kV and a working distance of 15 mm, using step sizes of 0.2 μ m for low-magnification (1000 $\times$) images (Fig. 2a and f) and 0.04 μ m for high-magnification (5000 $\times$) images (Fig. 2b and g). For deformed samples, EBSD was performed at an accelerating voltage of 15 kV and a working distance of 15 mm, with step sizes of 0.36 μ m for low-magnification (500 $\times$) images (Fig. 7g and j) and 0.045 μ m for high-magnification (4000 $\times$) images (Fig. 7h, i, k, and l). Samples for EBSD and ECC were mechanically ground, electropolished in a solution of 90% glacial acetic acid and 10% perchloric acid, and then chemically polished using a 0.04- μ m colloidal SiO₂ suspension. SEM samples for general morphology observations (Supplementary Fig. 3) were etched in a mixed solution of 10 mL HNO₃, 30 mL HCl, and 30 mL glycerol after mechanical grinding and electropolishing, whereas samples for fractography (Fig. 7b–e) were prepared without this step.

Phase fractions and dislocation densities were analyzed using an X-ray diffractometer (XRD, Rigaku MiniFlex600) with Cu-K α radiation (λ = 0.15406 nm), a scanning range of 40°–100°, and a scanning rate of 2° min^{−1}. XRD samples were mechanically ground and electropolished using the same solution employed for EBSD and ECC sample preparation. Phase fractions were determined from the integrated intensities of all relevant diffraction peaks. The dislocation density (ρ_{dis}) of the α' and γ_R phases was calculated using Eq. (1) [14].

$$\rho_{dis} = 3\sqrt{2\pi}\epsilon_M / db \quad (1)$$

where ϵ_M is the microstrain, d is the average crystallite size, and b is the Burgers vector for α' (0.248 nm) and γ_R (0.250 nm) [15]. The ϵ_M and d values were obtained by Williamson–Hall analysis (Fig. 8b and d) using Eq. (2) [16].

$$B\cos\theta = K\lambda / d + 4\epsilon_M \sin\theta \quad (2)$$

where B is the full width at half maximum of each diffraction peak of the α' and γ_R phases, θ is the diffraction angle, K is the Scherrer constant (0.89) [17], and λ is the wavelength of the Cu-K α radiation (0.15406 nm).

For detailed microstructural analysis of precipitates and chemical composition, the aged samples were examined by transmission electron microscopy (TEM, JEOL NEOARM) equipped with an energy-dispersive X-ray spectrometer (EDXS, JEOL JED-2300T) and further characterized by atom probe tomography (APT, CAMECA LEAPTM 4000X HR). TEM samples were prepared using a focused ion beam (FIB, FEI Helios G5). APT specimens were also fabricated by the FIB lift-out technique, followed by final cleaning at an accelerating voltage of 5 kV. APT was performed at a base temperature of 40 K, with a laser pulse frequency of 250 kHz and a pulse energy of 50 pJ. The collected data were reconstructed using IVAS software (version 3.8.1).

The tensile properties of the present steels were evaluated using a universal testing machine (MTDI, MINOS-100). Dogbone-shaped

specimens with gauge dimensions of 15 mm (length) $\times$ 4.0 mm (width) $\times$ 1.5 mm (thickness) were machined from the mid-thickness of the hot-rolled or austenitized plates along the rolling direction. Tensile tests were conducted at an initial strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ and repeated at least twice to ensure reproducibility.

2.3. DFT calculations

First-principles density functional theory (DFT) calculations were performed using the Vienna Ab initio Simulation Package (VASP) [18]. The projector-augmented wave (PAW) method [19], within the Perdew–Burke–Ernzerhof (PBE) [20] generalized gradient approximation, was employed to describe the exchange–correlation energy. A plane-wave cutoff energy of 450 eV and the Methfessel–Paxton smearing method with a width of 0.1 eV were applied. The Brillouin zone was sampled using Γ -centered k -point meshes of $16 \times 16 \times 16$ for the equiatomic B2 compound structure, which contains two atoms per cell. Comparable k -point densities were used for the unit cells of the pure elements (i.e., fcc Al, fcc Ni, bcc Fe, and α -Mn) as reference states for calculating the formation energies of the B2 compounds. Ionic

relaxations were performed using the conjugate gradient algorithm, with convergence criteria of 10^{-6} eV for the total energy and 10^{-2} eV/Å for the forces.

3. Results

3.1. Alloy design using thermodynamic approaches

Because the primary objective of the present alloy design was to develop a cost-effective maraging steel, expensive alloying elements such as Co, Mo, and Ti were entirely excluded. In addition, the Ni content was substantially reduced relative to conventional maraging steels (17%–19%) to approximately 9%. This Ni level was determined based on two key considerations. (i) The Fe–9Ni steel, which is commercially used for cryogenic applications (see, e.g., American Society for Testing and Materials (ASTM) A553/A353), forms a fully α' structure even in the absence of sufficient C content [21,22]. (ii) Ni is a major constituent in the formation of intermetallic precipitates in maraging steels [9]. Another important aspect of the alloy design was to enable weight reduction. To this end, 4% Al was added because the steel density

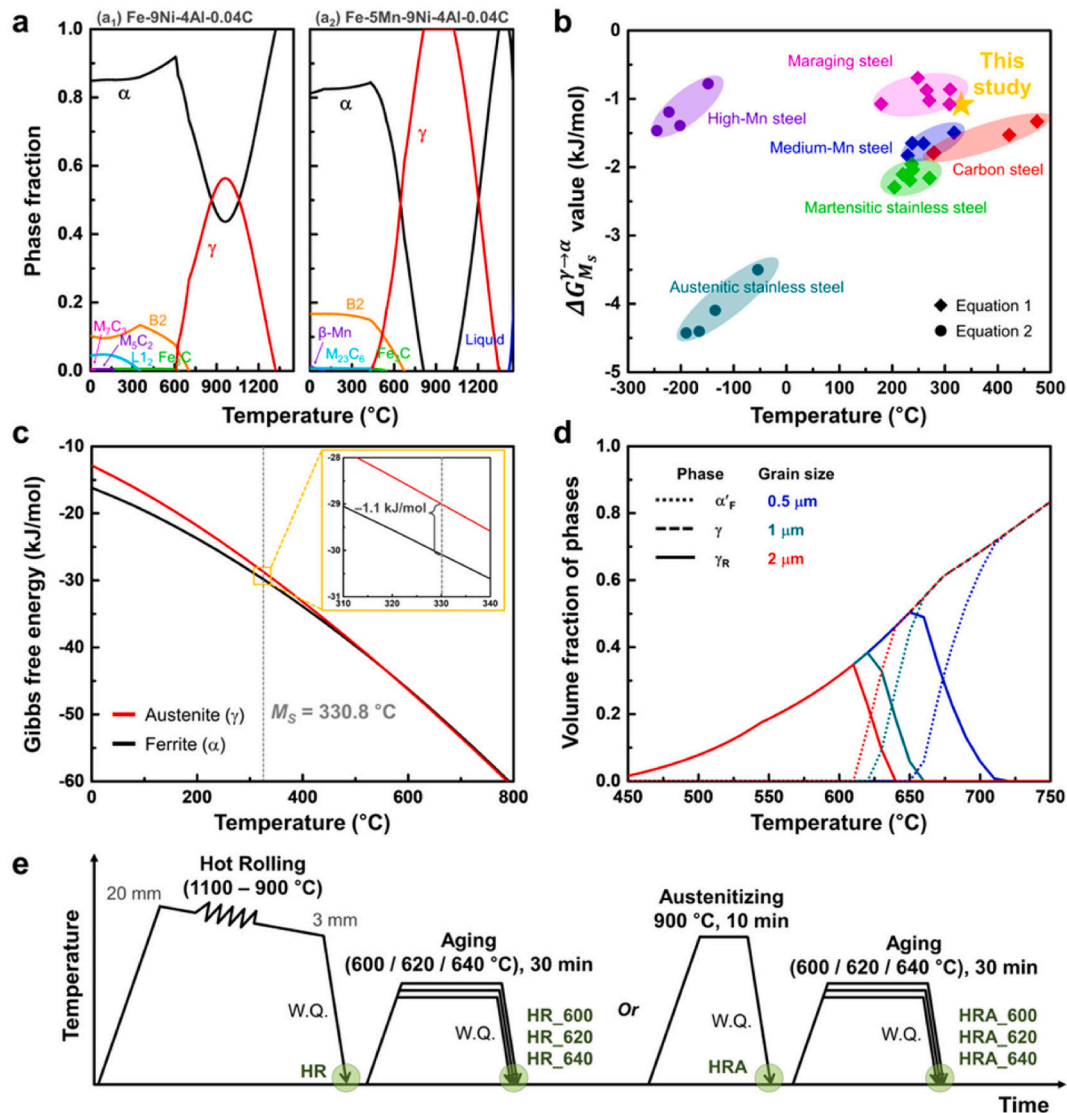


Fig. 1. Alloy design based on thermodynamic approaches: (a) Equilibrium phase diagrams of Fe–9Ni–4Al–0.04C steel and Fe–5Mn–9Ni–4Al–0.04C steel; (b) $\Delta G_{M_s}^{\gamma \rightarrow \alpha}$ value–temperature maps of the martensitic and austenitic steels; (c) Gibbs free energy of γ and α phases according to the temperature of the Fe–5Mn–9Ni–4Al–0.04C steel; (d) simulation of the γ_R and α'_F volume fractions in Fe–5Mn–9Ni–4Al–0.04C steel as functions of aging temperature, using average grain sizes of 0.5, 1 and 2 μm ; (e) thermo-mechanical process of the Fe–5Mn–9Ni–4Al–0.04C steel.

decreases by approximately 1.3% per 1% Al addition, and lightweight steels with a ferritic phase typically contain more than 3% Al [3]. In addition, the C content was limited to 0.04% to suppress carbide precipitation and the associated tempering of α' , which could reduce the steel strength.

Based on the aforementioned hypothesis, the chemical composition of the steel was initially determined to be Fe–9Ni–4Al–0.04C. However, this composition could not produce a fully α' microstructure after hot rolling because it lacks a single-phase austenite (γ) region at high temperatures, as shown in the equilibrium phase diagram simulated using Thermo-Calc (Fig. 1a₁), owing to the relatively high addition of the ferrite (α) stabilizer (Al) [23]. Therefore, 5% Mn, a strong γ stabilizer [23] and an inexpensive alloying element, was added to obtain a single-phase γ region within the hot-rolling temperature range and to promote transformation to a fully α' phase after cooling (Fig. 1a₂). Additionally, Mn was expected to modify the chemical composition of the intermetallic (B2) precipitates, as indicated by the thermodynamic calculations in Fig. 1a. Whereas the B2 phase in the Mn-free steel is primarily composed of Ni and Al, the B2 phase in the Mn-containing steel shows a lower Al content and substantial Mn enrichment (Supplementary Fig. 1). Moreover, because of the strong γ -stabilizing

effect of Mn, γ reversion during aging is expected to be more pronounced in the Mn-containing steel than in the Mn-free steel.

To evaluate the likelihood of martensitic transformation in the present steel, the martensite start temperature (M_s) and the Gibbs free energy change for the $\gamma \rightarrow \alpha$ transformation at M_s ($\Delta G_{M_s}^{\gamma \rightarrow \alpha}$) were compared with those of conventional martensitic steels (e.g., maraging, carbon, medium-Mn, and martensitic stainless steels) and austenitic steels (e.g., high-Mn and austenitic stainless steels) (Fig. 1b; Supplementary Table 1). The $\Delta G_{M_s}^{\gamma \rightarrow \alpha}$ values were calculated using Thermo-Calc software (Fig. 1c), while the M_s were calculated using the following empirical equations:

$$M_s (^{\circ}\text{C}) \text{ for martensitic steel} = 539 - 423\text{C}\% - 30.4\text{Mn}\% - 7.5\text{Si}\% + 30\text{Al}\% - 12.1\text{Cr}\% - 17.7\text{Ni}\% - 7.5\text{Mo}\% + 10\text{Co}\% \quad (3)$$

$$M_s (^{\circ}\text{C}) \text{ for austenitic steel} = 502 - 810\text{C}\% - 1230\text{N}\% - 13\text{Mn}\% - 12\text{Cr}\% - 30\text{Ni}\% - 54\text{Cu}\% - 46\text{Mo}\% \quad (4)$$

where elemental concentrations are in weight percent. Equation (3) is

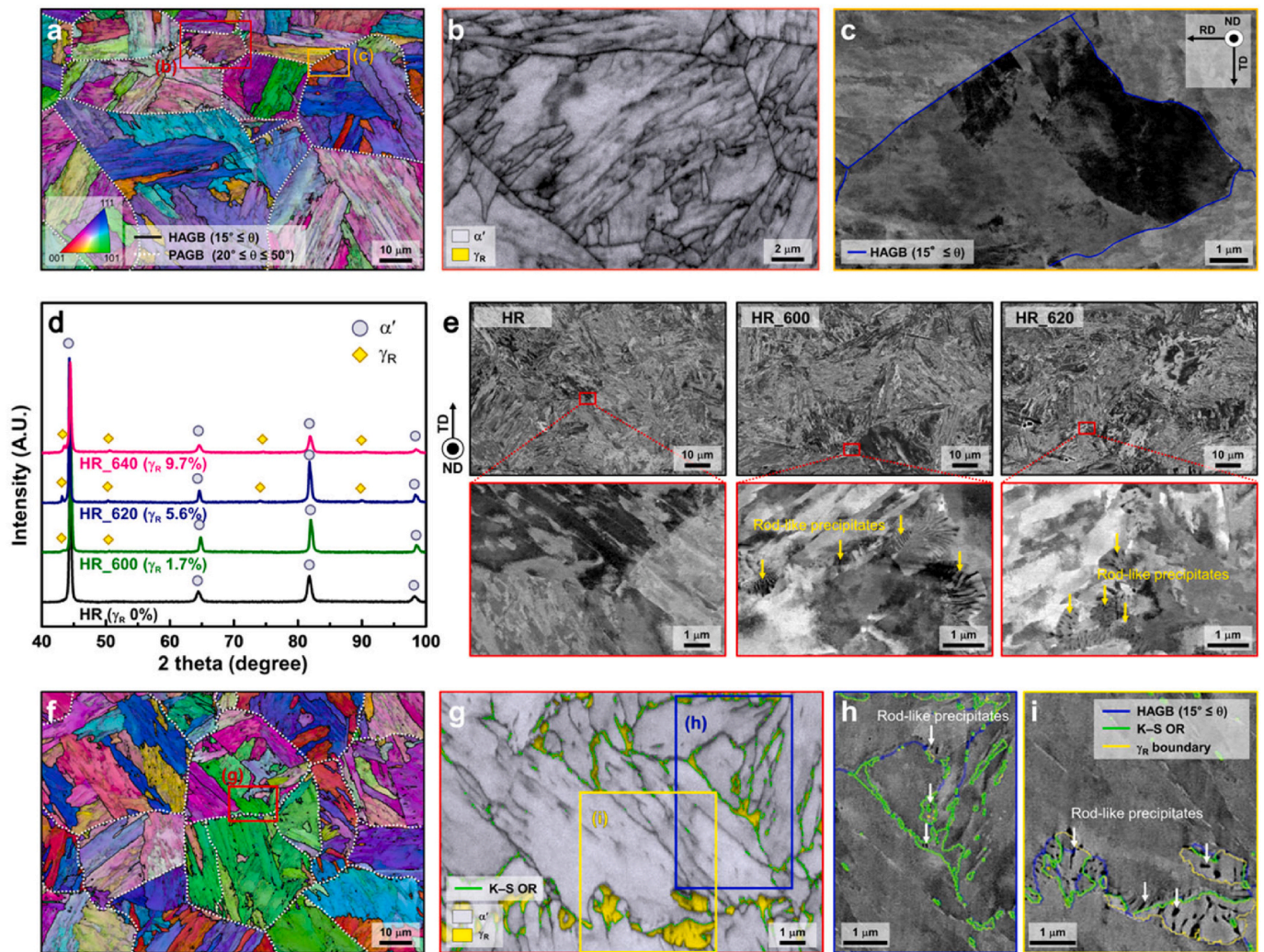


Fig. 2. EBSD (a) IQ-IPF map and (b) IQ-phase map of the HR sample. (c) ECC image taken from the boxed region in Fig. 2a. (d) XRD patterns of the HR samples before and after aging. (e) ECC images of the HR samples before and after aging at 600 and 620 °C. EBSD (f) IQ-IPF map and (g) IQ-phase map of the HR sample after aging at 640 °C. (h, i) ECC images taken from the boxed region in Fig. 2g. In the IQ-IPF maps, the black solid lines indicate high-angle boundaries and the white dashed lines denote PAGBs in the α' matrix. In the IQ-phase maps, the gray phase represents α' , the yellow phase corresponds to γ_R , and the green solid lines denote boundaries that satisfy the K-S relationship. In the ECC images, the blue lines represent high-angle grain boundaries, the yellow lines indicate γ_R grain boundaries, and the green lines denote boundaries satisfying the K-S relationship, as identified by EBSD analysis.

derived from the Kung and Rayment equation [24], with the contribution of the Al term taken from the equation reported by Mahieu et al. [25]; it is widely used to predict M_s in martensitic steels. Equation (4) was reported by Bott et al. [26] for Mn-bearing austenitic steels. As shown in Fig. 1b, the M_s of the martensitic steels, including the present steel ($\sim 330.8^\circ\text{C}$), were calculated to be above ambient temperature, whereas those of the austenitic steels were below ambient temperature. Additionally, the $\Delta G_{M_s}^{\gamma \rightarrow \alpha}$ value for the present steel was approximately -1.1 kJ/mol , which is within the range of other martensitic steels. The $\Delta G_{M_s}^{\gamma \rightarrow \alpha}$ values associated with martensitic transformation typically range from -0.9 to -1.4 kJ/mol [27]. Therefore, based on thermodynamic calculations and previous studies, the present steel is expected to form a fully α' microstructure after hot rolling or austenitizing.

To prevent a decrease in strength, the aging temperature of the present steel was selected to be above the carbide dissolution temperature (545°C ; Fig. 1a₂) and below the dissolution temperature of the intermetallic (B2) precipitates (674°C ; Fig. 1a₂). In addition, to enhance the ductility originated from the formation of the γ_R phase, the γ_R volume fraction as a function of aging temperature was simulated (Fig. 1d) based on the difference between the γ fraction calculated from Fig. 1a₂ and the fresh α' (α'_F) fraction estimated using Eq. (5) [28].

$$V\alpha'_F = 1 - \exp[-\alpha(M_s - 25)^\beta] \quad (5)$$

where α and β are constants calculated using the following equations proposed in a previous study [29].

$$\alpha = 0.0076 - 0.0182C\% + 0.00014Mn\% \quad (6)$$

$$\beta = 1.4609 + 0.4483C\% - 0.0545Mn\% \quad (7)$$

where $C\%$ and $Mn\%$ denote the concentrations of C and Mn in the γ phase (wt.%), as calculated by Thermo-Calc. The M_s was calculated using Eq. (3) and the corresponding γ -phase compositions predicted by Thermo-Calc. To improve simulation fidelity, the effect of γ grain size on M_s was incorporated into Eq. (3) via the term $-60.5 \times (\text{volume of } \gamma \text{ grain})^{-1/3}$ [28,29]. Although the calculations indicated that a fraction of reverted γ could transform into α'_F after aging, a relatively high γ_R fraction was still expected at aging temperatures below 650°C (Fig. 1d). This expectation is supported by the fact that γ_R in martensitic steels typically exhibits an ultrafine grain size, with widths or diameters typically ranging from 0.5 to $2.0\text{ }\mu\text{m}$ [30,31]. Hence, considering the dissolution temperatures of carbide and B2 precipitates and the predicted γ_R fraction, the aging temperatures were selected as 600 , 620 , and 640°C . To determine an appropriate aging time, preliminary hardness screening was performed on samples aged at 640°C for 0 – 60 min (Supplementary Fig. 2). Based on the yield strength estimated from the hardness values, an aging time of 30 min was selected because it was expected to provide a favorable balance between high strength ($\sim 1.5\text{ GPa}$) and a sufficient γ_R fraction to ensure adequate ductility (Supplementary Fig. 2). In addition, prior to aging, some as-hot-rolled plates were austenitized in the γ single-phase region (900°C , Fig. 1a₂) for 10 min to refine the prior γ grains (PAGs) and promote more active γ reversion along the martensitic boundaries during aging. The PAG size was $\sim 50.1\text{ }\mu\text{m}$ in the as-hot-rolled sample and decreased to $\sim 24.5\text{ }\mu\text{m}$ after austenitization (Supplementary Fig. 3). Consequently, the as-hot-rolled and austenitized steels were aged at 600 , 620 , and 640°C for 30 min , followed by water quenching (Fig. 1e). The former samples are designated HR, while the latter are designated HRA, with the aging temperature indicated after the underscore (e.g., HR_640).

3.2. Microstructures of the present maraging steel

Fig. 2a–c shows the microstructures of the HR steel characterized by EBSD and ECC. As predicted by thermodynamic simulations (Fig. 1a–c), the corresponding steel exhibited a fully α' microstructure after hot

rolling, with no detectable γ_R (Fig. 2b–d). The martensitic hierarchy, including PAGs, packets, blocks, and laths, was well developed, as confirmed by the image quality (IQ)–inverse pole figure (IPF) map (Fig. 2a) and ECC images (Fig. 2c and e). In the IQ-IPF maps (Fig. 2a and f), the black lines indicate high-angle grain boundaries with misorientation angles $>15^\circ$. The PAG boundaries (PAGBs) were primarily identified by misorientation angles of 20° – 50° [32,33] and were retraced with white dashed lines for improved visualization; the original results are provided in Supplementary Fig. 4. In the IQ-phase maps (Fig. 2b and g), gray phase represents α' and yellow phase corresponds to γ_R . The HRA steel exhibited a similar α' microstructure to the HR steel, as shown in Supplementary Fig. 5a and b.

Considering the microstructure of the aged steel, the γ_R fraction measured by XRD increased from $\sim 1.7\%$ to $\sim 9.7\%$ as the aging temperature increased from 600°C to 640°C (Fig. 2d). A similar trend was observed for the HRA samples, in which the γ_R fraction increased from $\sim 1.8\%$ to $\sim 12.1\%$ with increasing aging temperature (Supplementary Fig. 5a). Comparison of γ_R fractions between the HR and HRA samples showed that, at a given aging temperature, the HRA samples exhibited higher γ_R fractions, because the finer martensitic hierarchy in the HRA samples promoted more active γ reversion during aging (Supplementary Fig. 3). However, the experimentally measured γ_R fractions in both HR and HRA samples were lower than those predicted by Thermo-Calc (Fig. 1d), most likely due to the relatively short aging duration (30 min) to reach equilibrium and because of inherent limitations of thermodynamics-based calculations, such as database constraints and uncertainties in empirical equations. Nevertheless, the Thermo-Calc predictions and experimental measurements exhibited consistent trends, with γ_R fractions increasing with aging temperature. Additionally, ECC characterization revealed that the HR and HRA specimens before aging consisted predominantly of an α' phase without distinct secondary phases, whereas the aged specimens exhibited rod-like phases within the grains near boundaries (Fig. 2e and Supplementary Fig. 5b).

To elucidate the detailed microstructure of the aged steel, the HR_640 sample was further examined by EBSD and ECC (Fig. 2f–i). In the IQ-phase map (Fig. 2g), green solid lines denote interphase boundaries that satisfy the Kurdjumov–Sachs (K–S) orientation relationship. In the corresponding ECC images (Fig. 2h and i), boundaries identified by EBSD—high-angle boundaries, γ_R boundaries, and K–S-related boundaries—are highlighted by blue solid lines, yellow solid lines, and green solid lines, respectively. The HR_640 steel exhibited an α' matrix similar to that of the HR steel (Fig. 2f). However, as expected from the predicted and measured γ_R fractions (Fig. 1d and 2d), ultrafine γ_R grains ($\sim 400\text{ nm}$ to $1.8\text{ }\mu\text{m}$) formed readily, predominantly at PAGBs and along packet, block, and lath boundaries (Fig. 2g). The γ_R grains formed at PAGBs exhibited interphase boundaries that only partially satisfied the K–S relationship, whereas those formed along packet, block, and lath boundaries showed interphase boundaries that fully satisfied the K–S relationship. Accordingly, γ_R grains nucleated at PAGBs had interphase boundaries comprising both semi-coherent segments (i.e., K–S-related boundaries) and incoherent segments (i.e., general high-angle boundaries that do not follow the K–S relationship), while γ_R grains formed along packet, block, and lath boundaries exhibited predominantly semi-coherent interphase boundaries. ECC analysis of selected regions containing γ_R grains further revealed rod-like phases within the γ_R grains (Fig. 2h and i). Notably, these rod-like phases were more pronounced in γ_R grains whose interphase boundaries did not fully satisfy the K–S relationship, specifically those nucleated at PAGBs. Based on their morphology, these rod-like phases were considered to exhibit features consistent with a discontinuous precipitation-like mechanism [34] during γ reversion and growth, as was further supported by Supplementary Fig. 6. With increasing aging time, the rod-like phase became more pronounced and its area fraction increased. This evolution suggests that growth of the rod-like phases is closely coupled to growth of the reverted γ phase during aging, consistent with a discontinuous precipitation-like mechanism.

For a more thorough analysis of the precipitates in the aged steels, the HR_640 sample was examined using transmission electron microscopy (TEM; Figs. 3 and 4). A TEM specimen was extracted from a region containing rod-like precipitates using an FIB. Fig. 3a shows an annular dark-field (ADF) scanning TEM (STEM) image. The region marked by the white box in Fig. 3a was further characterized using dark-field TEM imaging (Fig. 3b) to highlight the γ_R grain and precipitates and was analyzed by EDXS (Fig. 3c) to visualize the corresponding elemental distribution. The γ_R grain boundary was marked by a yellow dashed line, based on the dark-field TEM image highlighting the γ_R grain (Fig. 3b₁). The rod-like precipitates were identified as body-centered cubic (bcc) B2 precipitates based on the selected-area diffraction pattern (Fig. 3b₂), consistent with the thermodynamic calculations (Fig. 1a₂). They were enriched in Ni, Al, and Mn, with a corresponding depletion of Fe in these regions (Fig. 3c). The rod-like B2 precipitates were well developed within the γ_R grain and extended into the adjacent region immediately beneath it, which was identified as an α' grain (outlined by the red dashed line in Fig. 3b). These observations suggest the following sequence of microstructural evolution during aging. First, γ reversion and B2 precipitation occurred simultaneously. As the γ grains grew, the precipitates within them coarsened into rod-like morphologies, consistent with a discontinuous precipitation-like mechanism involving a moving grain-boundary reaction [34,35], as reported previously. The resulting local depletion of Ni and Mn in the γ grain reduced its stability, leading to the partial transformation of γ into fresh α' (α'_F) during

cooling. TEM observations of another region revealed similar features (Fig. 4), including well-developed rod-like B2 precipitates enriched in Ni, Al, and Mn within a grain identified as α'_F rather than γ_R (Fig. 4a–c). Further evidence supporting this mechanism was obtained from parent- γ -grain reconstruction analysis of α'_F -containing regions using EBSD data (Supplementary Fig. 7).

The original α' matrix contained spherical, Ni–Al–Mn-rich B2 precipitates that were homogeneously distributed throughout the matrix (Fig. 3d–f and 4c–d). The diameter of the spherical B2 precipitates in the α' phase (~ 3 – 12 nm) was smaller than the width of the rod-like B2 precipitates in the γ_R or α'_F phase (~ 19 – 38 nm). This morphological difference was attributed to the distinct crystallographic environments in which the bcc B2 phase formed. In the bcc α' matrix, B2 precipitates could nucleate with a low lattice misfit and therefore retain a fine spherical morphology [2]. By contrast, in the face-centered cubic (fcc) γ phase, the larger lattice mismatch could promote precipitate coarsening. As the γ grains grew, these precipitates subsequently elongated to form rod-like morphologies.

The elemental compositions of both the precipitates and the constituent phases in the HR_640 sample were quantified by APT. The APT tip was prepared by FIB lift-out from a region containing the α' phase with spherical B2 precipitates and the γ_R phase with rod-like B2 precipitates. A three-dimensional APT reconstruction of the HR_640 sample is shown in Fig. 5a. To improve visualization of the B2 precipitates, APT reconstructions overlaid with 70 at.% Ni + Mn + Al isosurfaces are

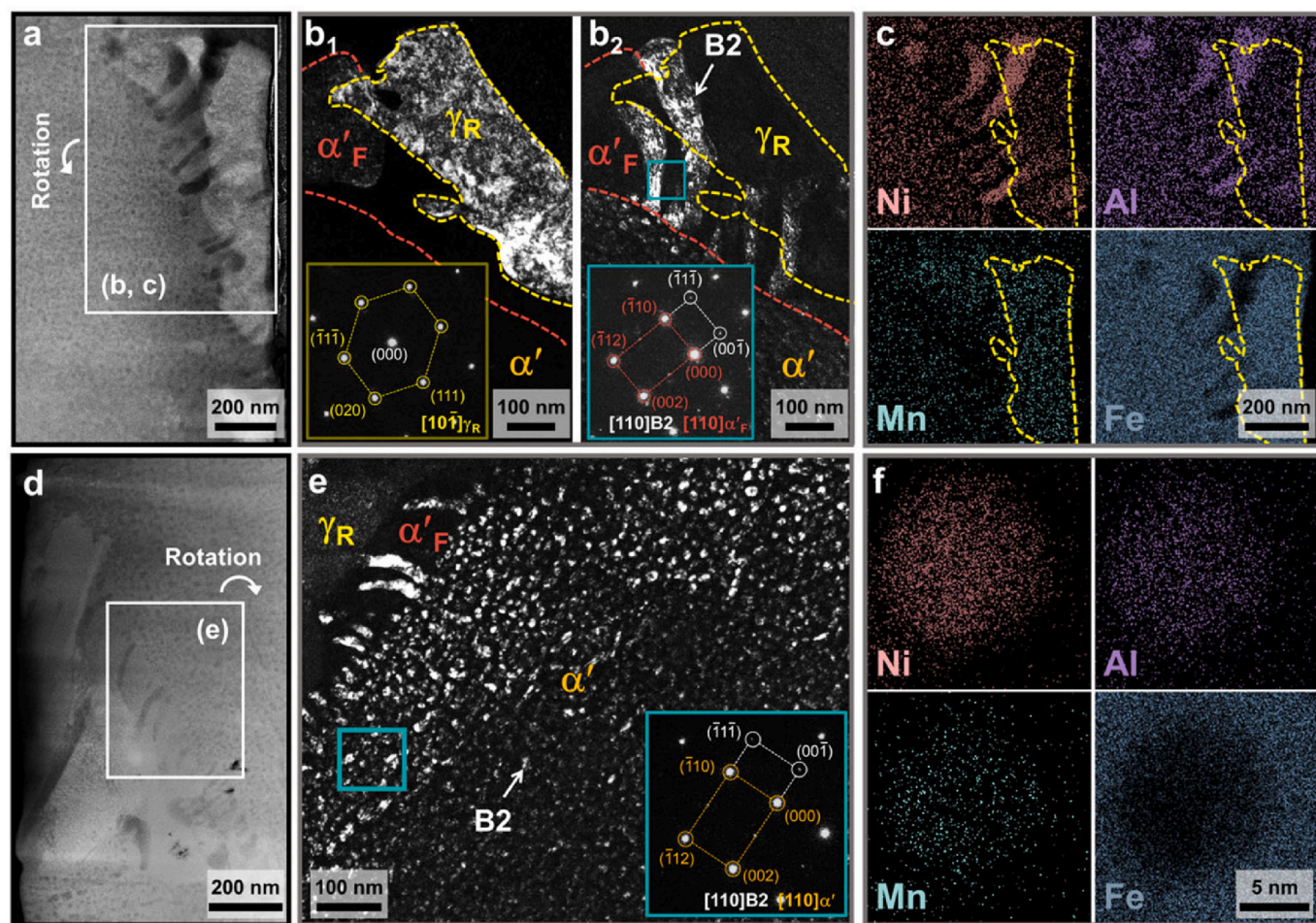


Fig. 3. TEM micrographs of the HR sample aged at 640 °C. (a) ADF-STEM image; (b) dark-field TEM images with selected area electron diffraction patterns highlighting (b₁) γ_R grain and (b₂) B2 precipitates; (c) STEM-EDXS elemental maps (panels (b) and (c) are obtained from the region marked by the white box in (a)); (d) ADF-STEM image of another sample; (e) dark-field TEM image with selected area electron diffraction pattern showing B2 precipitates, taken from the white box region in Fig. 3d; (f) STEM-EDXS elemental maps acquired from a spherical B2 precipitate located in the α' phase, as shown in Fig. 3e.

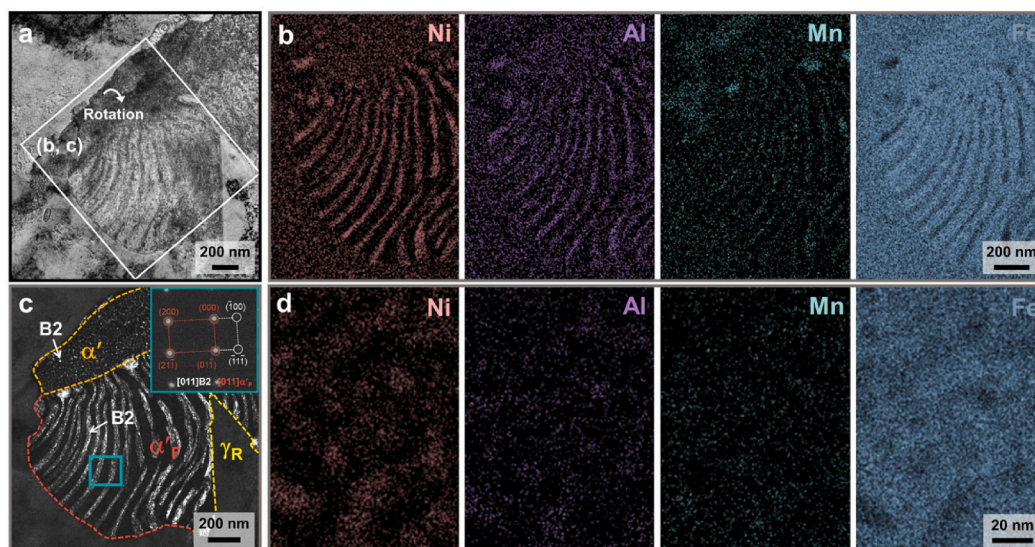


Fig. 4. TEM micrographs of the HR sample aged at 640 °C. (a) Bright-field TEM image; (b) STEM-EDXS elemental maps acquired from a white box region in Fig. 4a; (c) dark-field TEM images with selected area electron diffraction patterns highlighting B2 precipitates, taken from the white box region in Fig. 4a; (d) STEM-EDXS elemental maps acquired from the region containing spherical B2 precipitates within the α' phase, as shown in Fig. 4c.

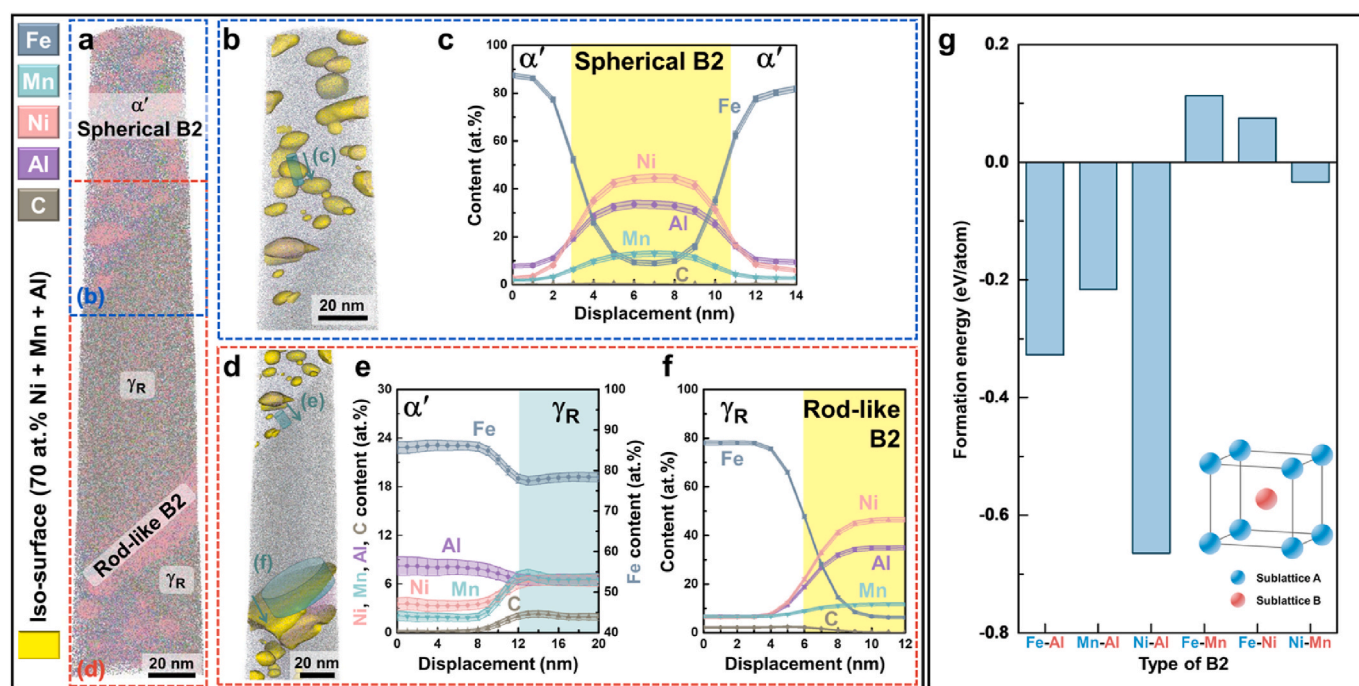


Fig. 5. Analysis of B2 precipitates in the HR sample aged at 640 °C: (a) Three-dimensional APT reconstruction showing elemental distributions in B2 precipitates, γ_R phase, and α' phase. (b, d) Three-dimensional APT reconstructions overlaid with 70 at.% Ni + Mn + Al isosurfaces, corresponding to the regions marked by blue and red boxes in Fig. 5a. (c, e, f) One-dimensional elemental profiles measured along the markers in Fig. 5b–d. (g) Formation energies of candidate B2 precipitates predicted by DFT calculations.

presented in Fig. 5b and d, and one-dimensional elemental profiles corresponding to the markers in the reconstruction images are plotted in Fig. 5c, e, and 5f. Consistent with the TEM observations, the B2 precipitates formed primarily as spherical particles in the α' matrix and as rod-like precipitates in the γ_R phase (Fig. 5a, b, and 5d). Despite these morphological differences, their average chemical composition was approximately 44.75 ± 0.65 at.% Ni, 34.20 ± 0.80 at.% Al, and 11.55 ± 0.25 at.% Mn (balance Fe), yielding a Ni:(Al + Mn) ratio close to 1:1. This composition indicates that the precipitates correspond to the B2 Ni (Al, Mn) phase (Fig. 5c and f). To further rationalize why the B2

precipitates in the present steel preferentially form with Ni and Al among the possible elemental combinations, the formation energies of equiatomic B2 compounds were calculated using DFT (Fig. 5g). These calculations considered all possible combinations among the major alloying elements (Fe, Al, Ni, and Mn) in the present steel. The NiAl compound exhibited the most negative formation energy, indicating the highest thermodynamic stability. This result suggests preferential occupation of different sublattices by Ni and Al in the B2 structure, consistent with the APT results.

Considering the chemical compositions of the α' and γ_R phases, γ_R

exhibited higher concentrations of Ni, Mn, and C and a lower concentration of Al than α' , consistent with the roles of Ni, Mn, and C as γ stabilizers and Al as an α stabilizer (Fig. 5e). The average concentrations of Ni, Mn, Al, and C in γ_R were 6.29 ± 0.08 , 6.59 ± 0.03 , 6.69 ± 0.01 , and 1.92 ± 0.19 at.%, respectively, whereas those in α' were 3.14 ± 0.33 , 2.04 ± 0.09 , 7.78 ± 0.22 , and 0.11 ± 0.03 at.%. These results indicate substantial elemental redistribution between the α' and γ phases during aging. Partitioning of Ni, Mn, and C into the reverted γ phase enhanced its stability, contributing to the higher γ_R fraction in the present steel compared to conventional maraging steels. After heat treatment transformed the fully α' martensitic steel into a two-phase microstructure ($\alpha' + \gamma_R$), the γ_R phase generally tended to be enriched in γ stabilizers relative to the nominal alloy composition. However, in the present steel, γ_R exhibited a lower Ni concentration than the nominal value (8.23 at.%), which is attributed to Ni depletion associated with the formation of the B2 Ni(Al, Mn) phase.

The results of the multiscale microstructural characterization of the present steel are shown in Fig. 6 and can be summarized as follows. (i) The HR and HRA steels exhibited a fully α' microstructure without precipitates. By contrast, the aged steel showed substantial formation of γ_R grains, predominantly at the PAGBs and along the packet, block, and lath boundaries within the α' matrix, driven by partitioning of Ni, Mn, and C. In addition, B2 Ni(Al, Mn) intermetallic precipitates formed within the constituent phases. (ii) In the γ_R phase, the B2 precipitates exhibited a rod-like morphology, attributed to the concurrent occurrence of γ reversion and precipitate formation, followed by coarsening along the γ grain-growth direction. Notably, γ_R grains nucleated at PAGBs exhibited pronounced rod-like precipitate formation because their interphase boundaries only partially satisfied the K–S orientation relationship. Consequently, boundary segments that did not follow the K–S relationship were incoherent, promoting grain-boundary migration and accelerating the growth of rod-like precipitates. In addition, during cooling, a portion of the γ grains transformed into α'_F owing to local depletion of Ni and Mn caused by precipitate formation. (iii) In the α' matrix, the B2 precipitates were nanoscale and spherical, consistent with the low lattice misfit between the precipitates and matrix arising from their similar crystallographic structures.

3.3. Tensile properties of the present maraging steel

Fig. 7a shows the engineering stress–strain curves of the HR samples before and after aging at 600, 620, and 640 °C. The unaged sample (i.e., the HR sample) exhibited a yield strength (YS) of ~ 1020 MPa, TS of ~ 1155 MPa, and TE of $\sim 17.4\%$. After aging, the strength increased significantly due to the formation of B2 precipitates, but gradually decreased with increasing aging temperature as the fraction of B2 precipitates decreased (Fig. 1a₂). The YS values of the HR_600, HR_620, and HR_640 samples were ~ 1650 , ~ 1581 , and ~ 1459 MPa, respectively, and the corresponding TS values were ~ 1711 , ~ 1671 , and ~ 1526 MPa, respectively. TE increased with aging temperature; the HR_600 ($\sim 1.8\%$) and HR_620 ($\sim 8.2\%$) samples exhibited lower TE than the HR sample, whereas the HR_640 sample ($\sim 17.9\%$) exhibited a TE comparable to that of the HR sample. These results indicate that the initial strength increase associated with B2 precipitation reduced TE in the aged samples. With increasing aging temperature, however, the reduced fraction of B2 precipitates (Fig. 1a₂), enhanced recovery of the α' matrix, and increasing fraction of γ_R (Fig. 2d) contributed to the recovery of TE, leading to improved ductility at higher aging temperatures. The aged HRA samples exhibited a similar trend. Strength increased substantially after aging; however, with increasing aging temperature, strength decreased while ductility improved (Supplementary Fig. 8). Comparison of the HR and HRA samples aged at the same temperature showed that the HRA samples exhibited slightly higher TE than the HR samples, which was attributed to their higher fraction of γ_R (Supplementary Fig. 5a).

Fracture-mode analysis can provide additional insight into the variation in TE among aged samples. Fig. 7b–e presents SEM fractographs of the HR samples after tensile testing, before and after aging. A quantitative comparison of the area fractions of ductile and brittle fracture modes is shown in Fig. 7f; these fractions were determined from the fractographs using ImageJ software. The HR sample exhibited predominantly ductile fracture characterized by dimpled features, with an area fraction of $\sim 98.7\%$, and only a minor fraction of quasi-cleavage fracture ($\sim 1.3\%$) (Fig. 7b and f). This behavior suggests that plastic deformation was readily accommodated by the relatively soft α' matrix

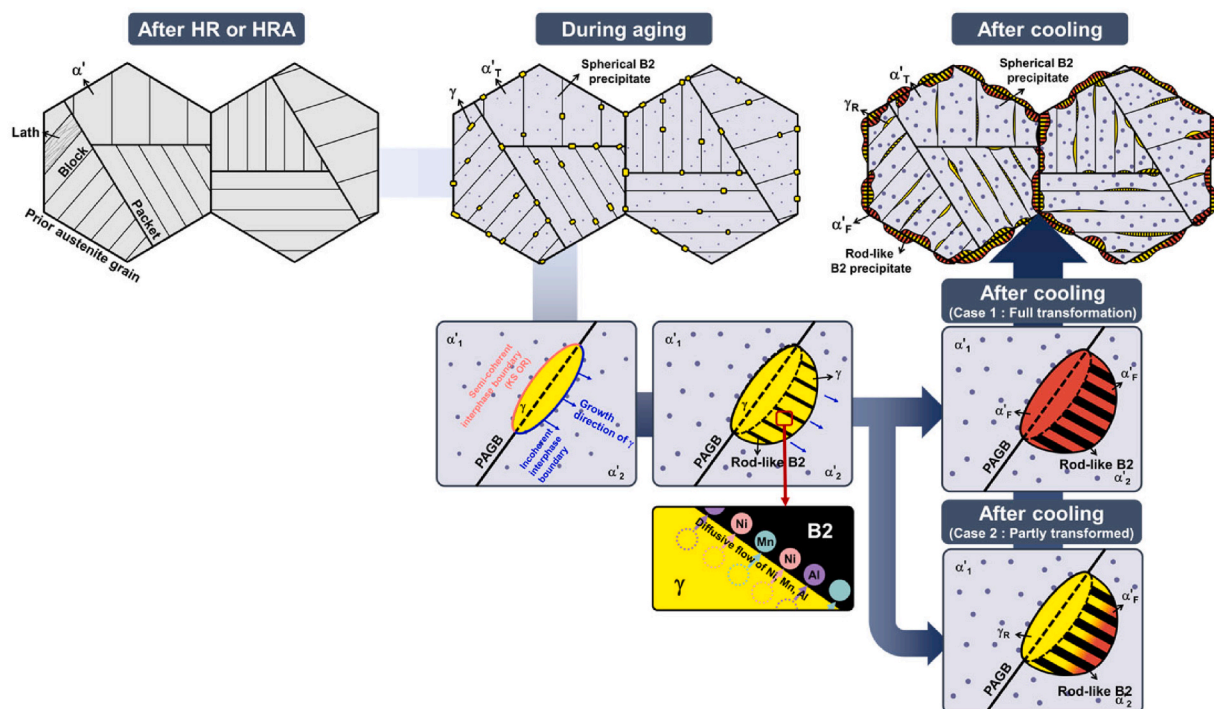


Fig. 6. Schematic of the microstructural evolution of the present steel during aging.

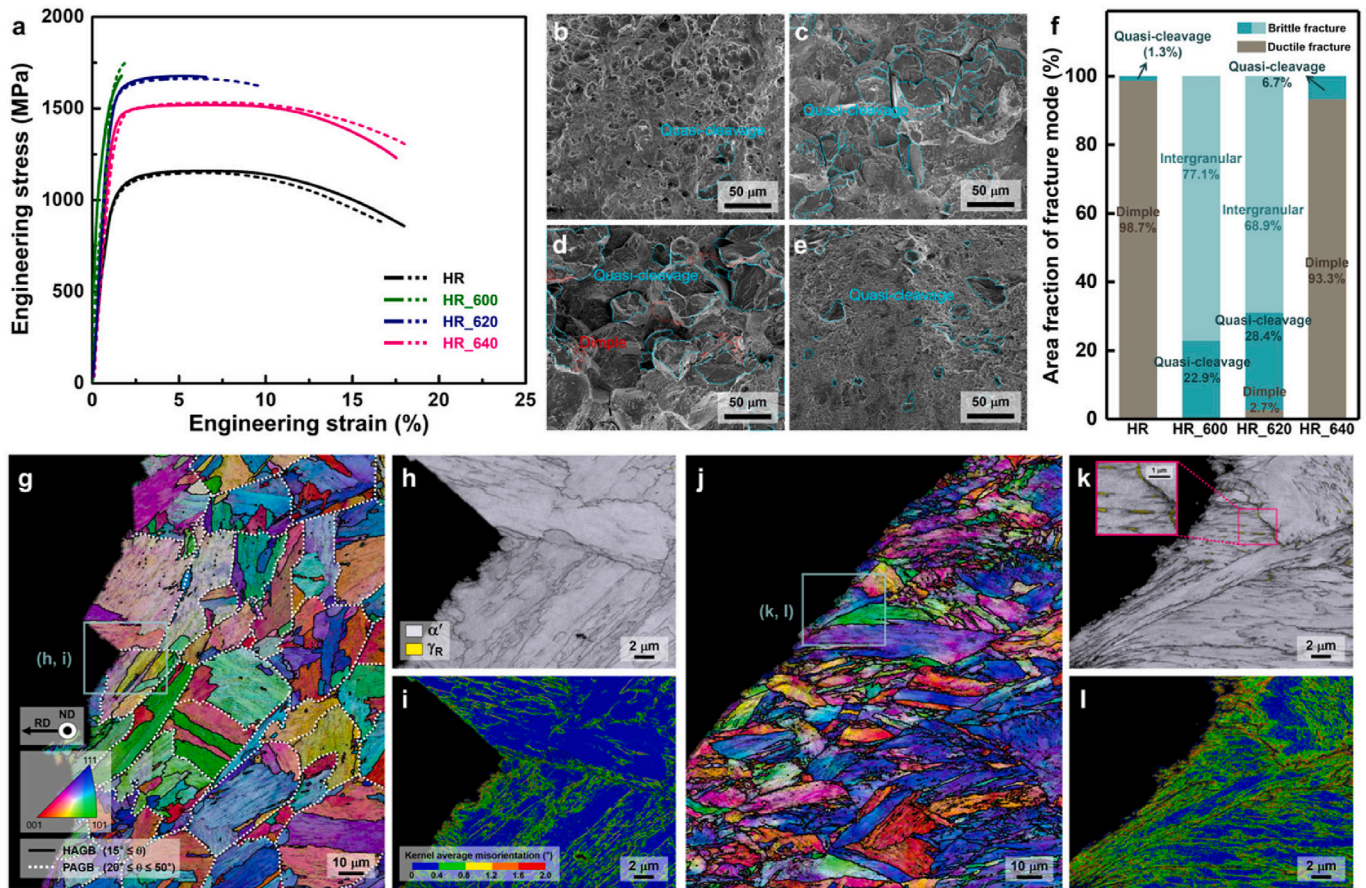


Fig. 7. (a) Engineering stress–strain curves of the HR, HR_600, HR_620, and HR_640 samples. SEM fractographs of the (b) HR, (c) HR_600, (d) HR_620, and (e) HR_640 samples after tensile failure. (f) Comparison of the area fractions of ductile and brittle fracture modes among the samples. EBSD (g, j) IQ–IPF maps, (h, k) IQ–phase maps, and (i, l) KAM maps of the (g, h, i) HR_600 sample and (j, k, l) HR_640 sample.

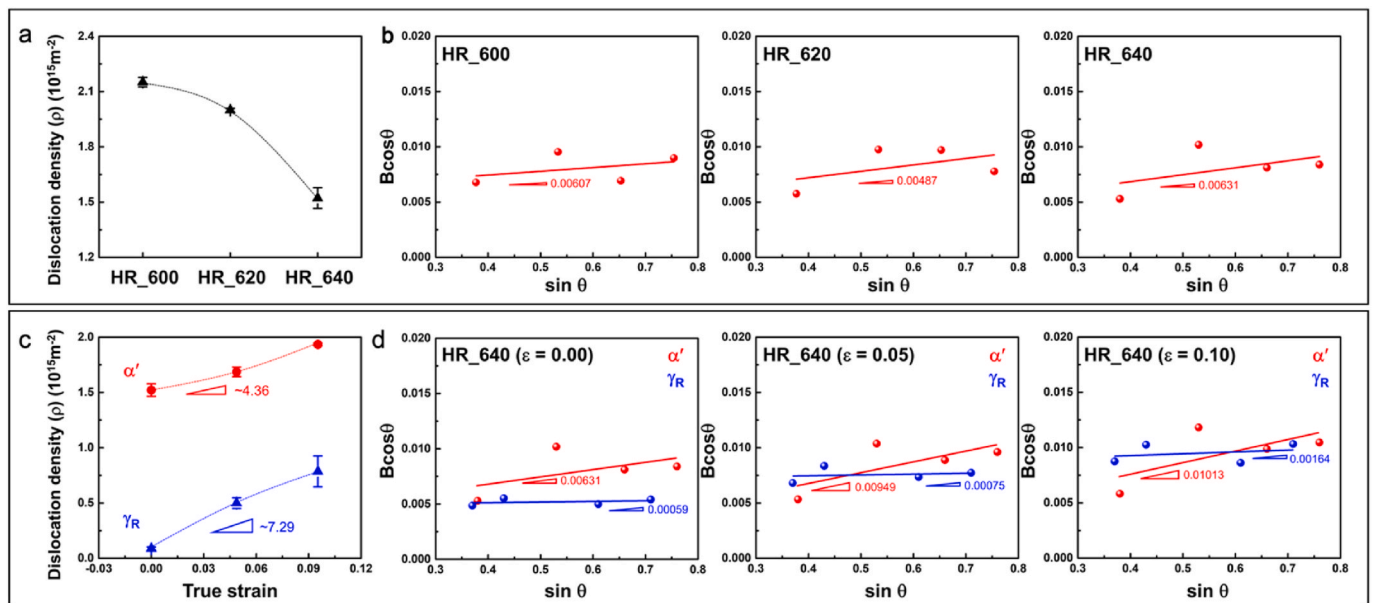


Fig. 8. (a) Calculated dislocation densities and (b) Williamson–Hall plots of the α' phase in aged HR samples as a function of aging temperature. (c) Calculated dislocation densities and (d) Williamson–Hall plots of the α' and γ_R phases in the HR_640 sample according to strain levels.

due to the absence of B2 precipitates. In the aged samples, although the HR_620 sample exhibited a lower area fraction of intergranular fracture ($\sim 68.9\%$) than the HR_600 sample ($\sim 77.1\%$), intergranular cracking

remained the dominant fracture mode in both samples, accompanied by a substantial fraction of quasi-cleavage fracture ($\sim 22.9\%$ for HR_600 and $\sim 28.4\%$ for HR_620) (Fig. 7c, d, and 7f). The intergranular fracture

in HR_600 and HR_620 was attributed to crack propagation along the PAGBs, based on their size. These results indicate that B2 precipitation increased the stress required to activate plastic deformation; as described by traditional Yoffe's theory, intergranular fracture accompanied by quasi-cleavage fracture occurred when this stress exceeded the stress required for brittle fracture. By contrast, when the aging temperature was increased to 640 °C, the fracture mode reverted predominantly to ductile fracture, with dimpled features accounting for ~93.3% of the fracture surface and only a minor fraction of quasi-cleavage fracture (~6.7%) (Fig. 7e and f). This transition is primarily attributed to the increased fraction of ductile γ_R formed along PAGBs (Fig. 2f and g), which effectively suppressed intergranular crack propagation along these boundaries. In addition, the reduced fraction of B2 precipitates (Fig. 1a₂) and the enhanced recovery of the α' matrix likely decreased the resistance to plastic deformation, further promoting ductile fracture. Collectively, these effects suppressed brittle fracture modes and ultimately increased TE.

To further elucidate the fracture mechanisms, the tensile-fractured HR_600 and HR_640 samples were examined along the normal direction using EBSD (Fig. 7g–l). Regarding the HR_600 sample, the crack propagated predominantly along the PAGBs of the α' matrix (Fig. 7g), and γ_R grains were scarcely observed in the vicinity of the crack propagation path because of the relatively low aging temperature (Fig. 7h). PAGBs were identified using the same method as in Fig. 2. The kernel average misorientation (KAM) value near the cracking sites was relatively low (~0.39; Fig. 7i), indicating limited plastic deformation of the α' matrix in HR_600, because KAM is proportional to dislocation density [36]. Consistent with the fractography (Fig. 7e), HR_640 did not exhibit crack propagation along specific boundaries in the α' matrix (Fig. 7j). Moreover, HR_640 exhibited a higher fraction of γ_R grains (Fig. 7k) and a higher KAM value (~0.61; Fig. 7l) near the cracking site than HR_600, indicating more extensive plastic deformation, which contributed to ductile fracture and the recovery of TE.

To better elucidate how the enhanced recovery of the α' matrix affects the ductility of aged samples, the change in dislocation density (ρ_{dis}) in the α' phase was quantified as a function of aging temperature (Fig. 8a) using XRD patterns (Fig. 2d, Supplementary Fig. 9) and Williamson–Hall analysis (Fig. 8b). As the aging temperature increased from 600 to 640 °C, the dislocation density in the α' matrix decreased from $\sim 2.15 \times 10^{15} \text{ m}^{-2}$ to $\sim 1.52 \times 10^{15} \text{ m}^{-2}$, indicating that higher-temperature aging produced a softer α' matrix that enabled more active plastic deformation. To further assess the contribution of γ_R phases to the ductility of aged samples, XRD measurements were performed on HR_640 samples strained to 0.05 and 0.10; the results are shown relative to an unstrained sample (Supplementary Fig. 9). The strain-dependent dislocation densities of the α' and γ_R phases (Fig. 8c) were then calculated using Williamson–Hall analysis (Fig. 8d). With increasing tensile strain, the γ_R phase exhibited a more pronounced increase in dislocation density than the α' matrix (Fig. 8c), suggesting that active plastic deformation of the γ_R phase contributed significantly to the ductility of the aged steel.

Beyond the established role of the ductile γ_R phase in enhancing ductility, the potential contribution of dynamic strengthening mechanisms, such as transformation-induced plasticity (TRIP) and TWIP upon deformation, should also be considered. These mechanisms are typically governed by the stacking fault energy (SFE), a key criterion for activation of TRIP and TWIP effects. For example, TRIP is typically activated when SFE is below 20 mJ/m², whereas the TWIP effect becomes dominant when SFE ranges from 20 to 50 mJ/m² [37]. Accordingly, SFE (mJ/m²) was calculated using the chemical composition obtained by APT (Fig. 5e), following Eq. (8) [38] and thermodynamic parameters reported by Lee et al. [39] (Fe, Mn, and C contributions), Saeed-Akbari et al. [40] (Al), and Curtze et al. [41] (Ni).

$$SFE = 2\rho(\Delta G_{ch}^{\gamma-\epsilon} + \Delta G_{mg}^{\gamma-\epsilon}) + 2\sigma \quad (8)$$

where ρ (mol/m²) is the molar surface density of the (111) plane of the γ phase (0.000294 mol/m²) [42], σ (mJ/m²) is the γ/ϵ interfacial energy (10 mJ m⁻²) [39], and $\Delta G_{ch}^{\gamma-\epsilon}$ and $\Delta G_{mg}^{\gamma-\epsilon}$ (J/mol) represent the chemical and magnetic Gibbs free energy differences, respectively, between the γ and ϵ phases, calculated based on the regular solution model. The SFE of the γ_R phase in HR_640 was calculated to be $\sim 31.3 \text{ mJ/m}^2$, suggesting that although localized TRIP may occur in highly strained regions, the overall deformation behavior is predominantly governed by the TWIP effect. This interpretation is supported by the observation that the volume fraction of the γ_R phase changed little with increasing strain (Supplementary Fig. 9) and that γ_R grains remained readily observable even near the fracture surface, as shown in the IQ-phase map (Fig. 7k). In addition, TEM analysis of a γ_R grain in the deformed HR_640 sample revealed frequent needle-shaped features with widths of $\sim 2\text{--}5 \text{ nm}$ (Fig. 9a). High-resolution fast Fourier transform (FFT)-filtered high-angle annular dark-field (HAADF)-STEM analysis of their interfaces confirmed that these features correspond to mechanical twins (Fig. 9b), and no evidence of strain-induced α' martensite was detected. Collectively, these results indicate that deformation in the present steel is primarily accommodated by the TWIP effect rather than the TRIP effect.

Additionally, to elucidate the contribution of B2 precipitates to the mechanical response, the deformed HR_640 sample was examined by low-angle annular dark-field (LAADF)-STEM, focusing on regions containing rod-like B2 precipitates (Fig. 9c) and spherical B2 precipitates (Fig. 9e). STEM–EDXS elemental maps were acquired from the same regions to identify the precipitates (Fig. 9d and f). Based on the results in Figs. 3 and 4, Ni-enriched regions were identified as B2 precipitates and were highlighted by yellow dashed lines in the LAADF-STEM images. In LAADF-STEM imaging, lattice defects such as dislocations generally appear with strong contrast relative to the surrounding matrix [43]. In regions containing spherical B2 precipitates within the α' matrix, pronounced dislocation–precipitate interactions were observed, consistent with previous studies on maraging alloys [44], as indicated by enhanced contrast in the vicinity of the precipitates (Fig. 9e). In regions containing rod-like B2 precipitates within γ_R grains, the γ_R phase appeared heavily deformed (i.e., exhibited enhanced contrast), consistent with the strain-dependent increase in dislocation density shown in Fig. 8c. Notably, the rod-like B2 precipitates exhibited stripe-like contrast within the precipitates themselves, which may be associated with deformation-induced lattice defects (Fig. 9c). Although the origin and nature of these features remain to be clarified, the observed contrast suggests that the rod-like B2 precipitates may accommodate a measurable degree of deformation capability, which could contribute to the excellent ductility of the present steel.

4. Discussion

The present Fe–5Mn–9Ni–4Al–0.04C steel exhibits an excellent strength–ductility balance after aging. For example, the strength–ductility combinations reach approximately 27,315 MPa% for the HR_640 sample, 25,653 MPa% for HRA_620, and 26,417 MPa% for HRA_640 (Fig. 7a; Supplementary Fig. 8). The increase in strength after aging is attributed primarily to the formation of intermetallic B2 precipitates (Fig. 3), because soft γ_R formation, matrix solute depletion due to precipitation, and the reduction in dislocation density during aging would otherwise be expected to decrease the strength. Among the precipitates, spherical B2 precipitates are expected to contribute most strongly to strengthening because of their fine size, homogeneous distribution, and high number density within the α' matrix. To quantitatively assess the contributions of the individual strengthening mechanisms to the strength level of the aged steel, the YS (σ_y , MPa) of the HR_640 sample was estimated using Eq. (9) [45].

$$\sigma_y = f^{\alpha'}(\sigma_0^{\alpha'} + \sigma_s^{\alpha'} + \sigma_g^{\alpha'} + \sigma_p^{\alpha'} + \sigma_d^{\alpha'}) + f^{\gamma}(\sigma_0^{\gamma} + \sigma_s^{\gamma} + \sigma_g^{\gamma} + \sigma_p^{\gamma} + \sigma_d^{\gamma}) \quad (9)$$

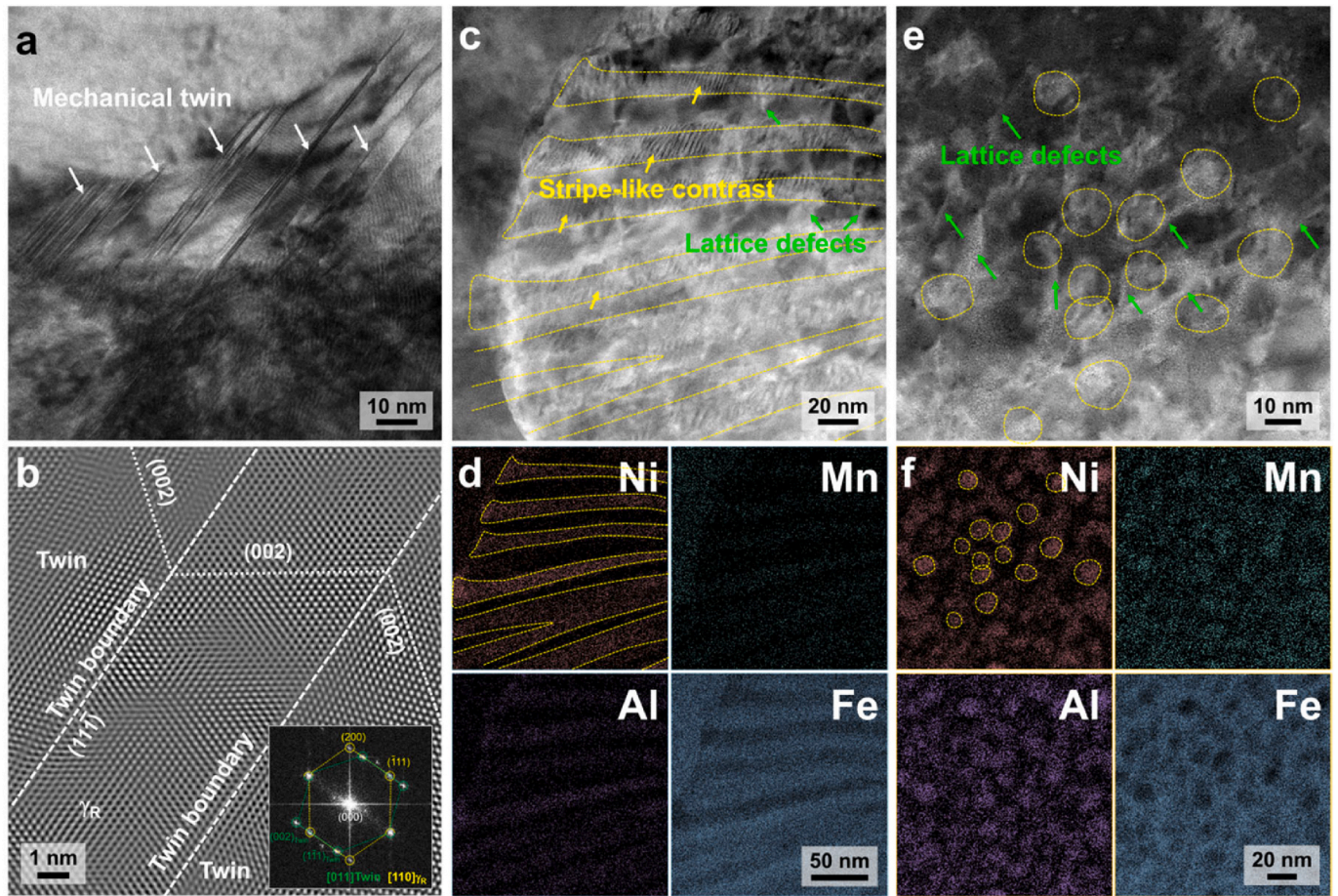


Fig. 9. (a) Bright-field TEM image and (b) FFT-filtered HAADF-STEM image with corresponding FFT pattern of the sample deformed to a true strain of 0.10, showing nanosized mechanical twins. (c) LAADF-STEM image and (d) STEM-EDXS elemental maps acquired from a region containing rod-like B2 precipitates, and (e, f) the corresponding image and maps obtained from a region containing spherical B2 precipitates in the sample deformed to a true strain of 0.10.

where the superscripts denote the corresponding phases, and f represents the volume fraction of each phase, as determined from the XRD measurements (Fig. 2d). The volume fraction of the α'_F phase was not considered separately because it could not be quantitatively distinguished from that of the α' matrix based on the XRD and EBSD results obtained in the present study. σ_θ is the lattice friction stress, with corresponding values adopted from previous studies ($\sigma_\theta^{\alpha'} = 54$ MPa [46], $\sigma_\theta^{\gamma_R} = 97$ MPa [47]). σ_s is the contribution of solid solution strengthening, which was estimated using the following equations for the α' phase (contribution of C [48]; contribution of other alloying elements [49]) and the γ_R phase (contribution of Al [50]; contribution of other alloying elements [51]):

$$\sigma_s^{\alpha'} = 1720(C\%)^{1/2} + 18.3(Ni\%) + 17.2(Mn\%) + 18(Al\%) \quad (10)$$

$$\sigma_s^{\gamma} = 81 + 598(C\%) + 5.7(Ni\%) - 1.4(Mn\%) + 20(Al\%) \quad (11)$$

where $C\%$, $Ni\%$, $Mn\%$, and $Al\%$ are the concentrations of C, Ni, Mn, and Al in weight percent, as measured from the APT results (Fig. 5e). σ_g is the contribution of grain-size strengthening, estimated using the Hall–Petch relationship, irrespective of phase [46]:

$$\sigma_g = k_y \times d^{-0.5} \quad (12)$$

where k_y is the Hall–Petch constant (210 MPa $\mu\text{m}^{1/2}$ for α' [46] and 180 MPa $\mu\text{m}^{1/2}$ for γ_R [47]), and d denotes the average α' block width or the average γ_R grain diameter measured from EBSD analyses (Fig. 2f and g). σ_p is the contribution of precipitation hardening, estimated using the

Ashby–Orowan relationship to calculate the precipitation-induced shear stress (τ_p), followed by conversion to YS through the shear stress–YS relationship [52,53].

$$\tau_p = 0.84 \times (1.2Gb / 2\pi L) \times \ln(d_p / 2b) \quad (13)$$

$$\sigma_p = M \times \tau_p \quad (14)$$

where G is the shear modulus (76 GPa for α' [46] and 75 GPa for γ_R [15]), and b is the Burgers vector (0.248 nm for α' and 0.250 nm for γ_R [15]). L is the interparticle spacing determined from the TEM images (~ 40 nm for the spherical precipitates and ~ 79 nm for the rod-like precipitates; Fig. 4c). d_p denotes the mean diameter of the spherical precipitates (~ 6 nm) and the mean width of the rod-like precipitates (~ 26 nm), also determined from the TEM images. M is the Taylor factor (2.73 for α' [46] and 3.06 for γ_R [15]). σ_d is the contribution from dislocation strengthening, estimated using the Bailey–Hirsch–Taylor equation (Eq. (15)), irrespective of phase [15,46]:

$$\sigma_d = M \times \alpha_d \times G \times b \times \rho_{dis}^{0.5} \quad (15)$$

where α_d is a dimensionless constant (0.25 for α' [46] and 0.4 for γ_R [15]), and ρ_{dis} is the dislocation density (m^{-2}) obtained from Eq. (1).

Fig. 10 summarizes the contributions of the individual strengthening mechanisms to the YS and the resulting calculated YS of the HR 640 sample, accounting for the volume fractions of the constituent phases. As expected, when the volume fractions of the α' and γ_R phases were considered, the contribution from the spherical precipitates (~ 466.9 MPa) was substantially greater than that from the rod-like precipitates

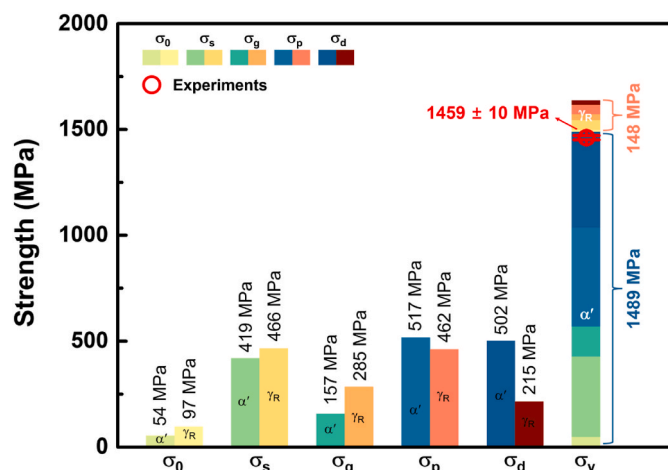


Fig. 10. Contributions of the individual strengthening mechanisms—lattice friction stress (σ_0), solid solution strengthening (σ_s), grain size strengthening (σ_g), precipitation strengthening (σ_p), and dislocation strengthening (σ_d)—to the calculated yield strength (σ_y), together with the experimentally measured yield strength of the HR_640 sample.

(~44.8 MPa), indicating that the aging-induced strength increase was primarily attributed to the formation of spherical precipitates. However, the calculated YS (~1637 MPa) was somewhat higher than the experimentally measured value (~1459 MPa), yielding a difference of ~178 MPa. This discrepancy is likely attributed to the assumption in the strengthening model that the constituent phases yield nearly simultaneously. In steels comprising a hard α' matrix and a relatively soft γ_R phase, prior work has shown that plastic deformation preferentially initiates in the γ_R phase during the early stages of yielding [54]. This interpretation is consistent with previous reports on maraging steels showing that formation of the relatively soft γ_R phase reduces the overall steel strength [55]. Accordingly, the present model likely overestimates YS. Nevertheless, the relative contributions of the individual strengthening mechanisms are expected to remain reliable.

Moreover, the aged steels exhibited excellent ductility, although discontinuous precipitation-like morphologies are generally detrimental because their large size and propensity to act as crack initiation sites reduce ductility [34,35]. In contrast to conventional discontinuous precipitates, the rod-like B2 precipitates in the present steel remain relatively small because they form within ultrafine γ_R grains (Fig. 3) and exhibit a measurable capacity for deformation (Fig. 9c), thereby limiting ductility loss. Furthermore, the surrounding γ_R phase not only deforms readily but also exhibits TWIP behavior during deformation (Fig. 9a and b), which significantly contributes to the improved ductility. Based on these findings, several follow-up strategies to further improve ductility can be proposed, including alloy and thermomechanical modifications to suppress formation of the α'_F phase and rod-like precipitates, which are detrimental to ductility. As discussed above, α'_F forms from reverted γ during cooling because local Ni and Mn depletion—caused by the formation of rod-like B2 Ni(Al, Mn) precipitates—reduces γ stability. Therefore, increasing the stability of reverted γ should suppress α'_F formation and increase the γ_R fraction. Chemical stabilization of γ through alloying additions is a practical approach. In particular, increasing the Mn content beyond 5 wt% is expected to effectively enhance γ stability. APT analyses revealed that B2 precipitates are primarily enriched in Ni and Al (Fig. 5f) because of their low formation energy (Fig. 5g), suggesting that additional Ni would preferentially promote B2 precipitation rather than stabilize γ . In contrast, because Mn is only a minor constituent in the precipitates, increasing Mn should more effectively stabilize γ . This hypothesis was supported by equilibrium phase-diagram simulations and XRD measurements; a steel with higher Mn content (Fe–8Mn–9Ni–4Al–0.04C) exhibited a broader γ

single-phase region in the phase diagram and a higher γ_R fraction after identical hot rolling and aging at 640 °C for 30 min (Supplementary Fig. 10). To suppress rod-like precipitates (i.e., the discontinuous precipitation-like morphology), the addition of a small amount of B is expected to be effective. Boron preferentially segregates to PAGBs in martensitic steels [56], which, in the present study, act as the primary nucleation sites for γ_R grains associated with rod-like precipitate formation (Fig. 2f–i). Accordingly, B segregation is expected to retard the growth of γ_R grains nucleated at PAGBs via solute-drag effects, thereby mitigating rod-like precipitate formation. In addition, because γ reversion and subsequent growth accompanied by rod-like precipitate formation occurred concurrently during single-step aging at 600–640 °C for 30 min, modification of the aging process could provide another viable route to suppress rod-like precipitates. For example, a two-step aging route could be employed: (i) a very short first aging step at an elevated intercritical temperature to induce γ reversion, followed by (ii) a second aging step at a lower temperature to suppress γ -grain coarsening and rod-like precipitate formation while promoting solute partitioning for γ stabilization.

Although the strategies outlined above may be explored in follow-up studies and may yield alloys with superior mechanical properties, the present alloy is already significant in demonstrating that a lean composition combined with a simple processing route can compete with the performance of commercial maraging steels. The tensile properties of the present steels (HR_620, HR_640, HRA_620, and HRA_640) were compared with those of conventional Co-free and Co-bearing maraging steels (Fig. 11a and b, Supplementary Table 2). The average TS of the steel developed in the present study was approximately 1595 MPa, lower than those of Co-free (~1870 MPa) and Co-bearing (~2061 MPa) maraging steels, most likely because the lean alloy composition provides limited solid-solution strengthening (Fig. 11a). However, the present steel exhibited a higher average TE (14.7%) than both Co-free (9.0%) and Co-bearing (6.1%) maraging steels, which is attributed to the large γ_R fraction and activation of the TWIP effect during plastic loading (Fig. 11b).

From an industrial perspective, the present steel offers compelling advantages. Its low-Ni, Co-free, and Mo-free composition provides exceptional cost-efficiency, and Al addition yields a notable reduction in density. As shown in Fig. 11c and d, the present steel exhibited ~4.1% and ~6.5% lower density and ~62% and ~80% lower material cost than the Co-free and Co-bearing maraging steels, respectively. The cost-efficiency discussed in the present study is based solely on the raw material costs associated with the alloy composition and does not account for processing-related costs. Material costs were calculated using the average market prices of the alloying elements during 2021–2024 (<https://www.komis.or.kr/>). Notably, when mechanical properties are considered alongside these advantages, the present steel exhibited higher TS than conventional maraging steels after accounting for both density and cost (i.e., specific TS per cost), while maintaining a favorable balance with TE (Fig. 11e).

5. Conclusion

In this study, the developed steel achieves a rare combination of ultrahigh strength, enhanced ductility, low density, and outstanding cost-efficiency, despite its lean alloy composition of Fe–5Mn–9Ni–4Al–0.04C. These results indicate that the designed steel effectively addresses key limitations of conventional maraging steels, including high material cost associated with high Ni, Co, and Mo contents, a limited strength–ductility balance, and relatively high density, thereby offering strong potential for broader and more practical applications. The principal findings are summarized as follows:

- (1) After aging, the steel exhibited an α' martensitic matrix with a substantial fraction of γ_R , predominantly distributed along PAGBs and martensitic hierarchical boundaries (packets, blocks, and

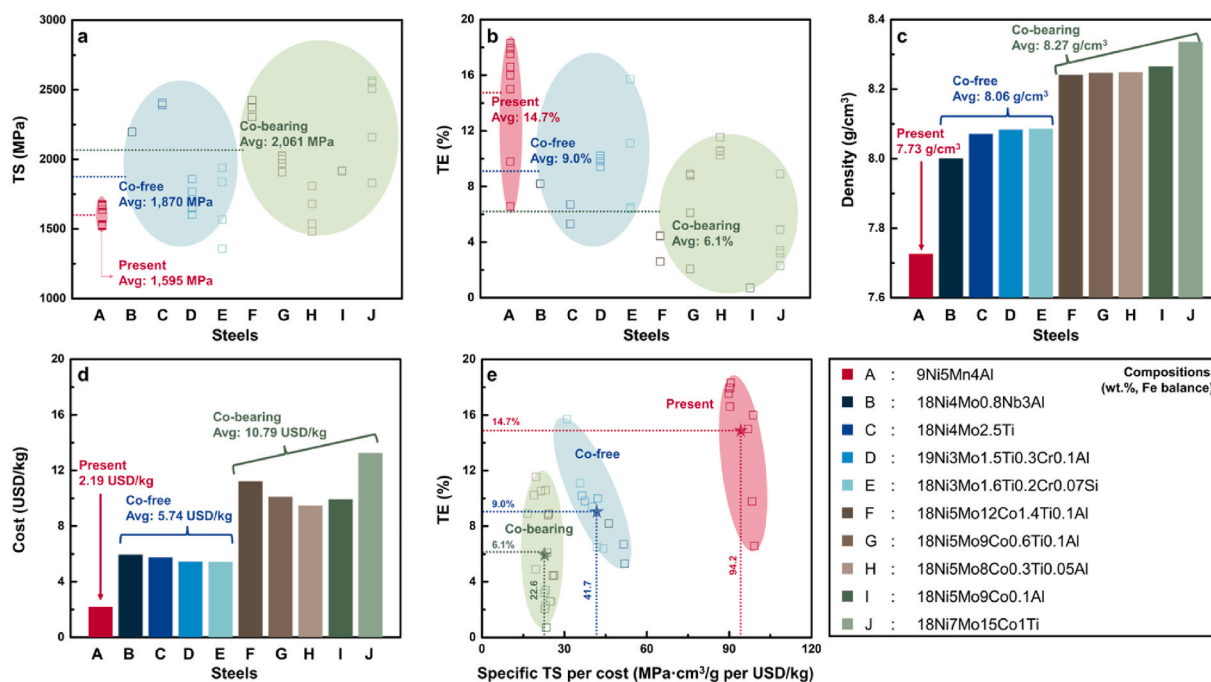


Fig. 11. Comparison of tensile properties, density, and cost among conventional maraging steels (Co-free and Co-bearing steels) and the present steel: (a) Tensile strength (TS), (b) total elongation (TE), (c) density, (d) material cost, and (e) TE as a function of specific TS normalized by cost.

laths), driven by partitioning of Ni, Mn, and C. Concurrently, B2 Ni(Al, Mn) intermetallic precipitates formed in both phases, appearing as nanoscale spherical precipitates in the α' matrix and as rod-like precipitates within the γ_R phase. The former morphology is attributed to the small lattice misfit between the precipitates and matrix, whereas the latter is formed via a discontinuous precipitation-like mechanism associated with γ reversion and subsequent growth.

- (2) The aged steels exhibited strengths exceeding ~ 1.5 GPa, primarily due to the homogeneous distribution of nanoscale spherical B2 precipitates within the α' matrix. Despite the presence of a discontinuous precipitation-like morphology, which typically degrades ductility, the present steel maintained a relatively high average ductility ($\sim 14.7\%$). This behavior is attributed to the fine rod-like B2 precipitates formed within ultrafine γ_R grains and their potential to accommodate deformation. In addition, the surrounding γ_R phase accommodated plastic deformation and exhibited a TWIP effect, further contributing to the improved ductility.

CrediT author statement

Chae Young Kim: Conceptualization, Investigation, Methodology, Formal analysis, Writing – original draft. Mun Sik Jeong: Investigation, Formal analysis, Writing – original draft. Tak Min Park: Investigation, Formal analysis. Jinseok Hong: Investigation, Formal analysis. Seung-Yong Lee: Formal analysis, Writing – review & editing. Seoung-Ho Lim: Investigation, Formal analysis, Writing – review & editing. Pyuck-Pa Choi: Investigation, Formal analysis, Writing – review & editing. Won-Seok Ko: Investigation, Formal analysis, Writing – review & editing. Jeongho Han: Conceptualization, Methodology, Supervision, Formal analysis, Writing – original draft, Writing – review & editing, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence

the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jmrt.2026.07.266>.

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