



Microstructure and Cu-precipitation Engineering in Warm-rolled Medium-Mn Steel Via Short-time Intercritical Annealing and Aging

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Abstract

Warm-rolled medium-Mn steels (MMnS) are attractive for automotive industry and energy applications, due to their balanced mechanical and corrosion properties. The present study investigated how newly-designed process routes with short post-warm-rolling heat treatments can further tune the microstructure and mechanical properties of an Fe-7Mn-0.05 C-1.5Al-1.5Si-1.5Ni-1.5Cu (wt%) steel. Warm-rolled MMnS (WarmR) was subjected to three heat treatment routes: intercritical annealing (IA) at 700 °C for 1 min (WRA), aging at 500 °C for 5 min (WRAg), and a combined IA+aging process (WRAA). The material under WarmR condition is characterized by a heavily deformed ferrite matrix and refined, mechanically stabilized austenite, providing high yield strength (YS: 926.2 ± 17.8 MPa) but low ductility (TE of $6 \pm 1.2\%$) due to a suppressed transformation induced plasticity (TRIP) effect. In the material under WRAg condition, high density of nano-sized Cu precipitates form within the ferrite, further increasing the yield strength (YS: 1179 ± 17.6 MPa) but not ductility (TE $5.7 \pm 1.5\%$). The WRA process promotes the reactivation of the TRIP effect and enhances ductility (TE of $14.5 \pm 2.6\%$), albeit at the expense of strength (YS: 743.7 ± 25.1 MPa). In contrast, the combined route (WRAA) allows to form nano-sized Cu precipitates with a moderate volume fraction while maintaining an active TRIP effect, thereby resulting in superior strength-ductility balance (YS: 938 ± 11.2 MPa and TE of $16.7 \pm 0.9\%$). The present study provides a mechanistic understanding, connecting processing condition, micro/nanostructure evolution and mechanical properties, thereby, offering an effective pathway for tuning strength-ductility synergy in warm-rolled medium-Mn steels.

Keywords Medium-Mn steels · Warm rolling · Elemental partitioning · Nano-sized Cu-rich precipitates · 3D-atom probe tomography

1 Introduction

Medium-Mn steels (MMnSs), containing 3–12 wt% Mn, represent an important class of third-generation advanced high-strength steels. They are key candidate materials for lightweight structural applications in the automotive and

off-shore sectors due to their tunable mechanical properties and cost-efficiency in direct comparison to common austenitic steels [1–6]. However, MMnS faces the major challenge not being able to simultaneously achieving high yield strength (YS) and high ductility [7, 8], which

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continues to limit their industrial applications compared to other steel classes.

The mechanical properties of MMnS are primarily influenced by the volume fraction of the austenite phase and its stability, as these factors govern the distinctive strain hardening response of this two-phase (ferrite + austenite) alloy system [9]. The volume fraction of the austenite phase is predominantly affected by the amount of austenite stabilizers, *e.g.* Mn and C, and the employed processing routes. Meanwhile, the stability of the austenite phase is influenced by the local chemical enrichment of austenite stabilizing elements eventually shaping the appearance of the austenite phase, *i.e.*, grain size and morphology [10–13]. The enrichment of austenite-stabilizing elements in the austenite phase is generally accompanied by their depletion from the ferrite phase, and this leads to softening the ferrite phase [8, 14]. In dual-phase MMnS, microscopic yielding is commonly initiated in the mechanically softer phase. At the same time, macroscopic yield strength is also determined by additional factors such as load partitioning between phases, phase connectivity, and load constraint effects from the harder phase [15–17]. Furthermore, the yield strength may also be influenced by solid-solution strengthening in the austenite phase [1, 18]. Accordingly, achieving a favorable combination of yield strength, ultimate tensile strength, and ductility requires tailoring fraction, stability, and strength of both ferrite and austenite phases.

Among various hardening and strengthening approaches, the utilization of nano-sized precipitation has emerged as a notable approach in recent past [19–23]. Specifically, the co-precipitation of Cu-rich and B2-type Ni-(Al or Mn)-rich precipitates has been proven to be an efficient strengthening strategy for low-carbon steels [22–25], providing a favorable synergy of high strength, ductility, and weldability at comparatively low cost. The precipitation characteristics, such as chemical composition, size, number density, and coherency with the matrix, critically determine strengthening efficiency. Through tailored heat treatments, these parameters can be optimized to balance mechanical performance [25–28]. According to the Lifshitz-Slyozov-Wagner (LSW) mean-field theory [29, 30], the mean precipitate radius in Fe-Cu systems increases with aging time ($t^{1/3}$), while the number density decreases proportionally to t^{-1} , eventually influencing hardness and YS [29, 31]. Reported studies [25–28, 32–34] have shown that extended aging time promotes additional nucleation events [35] and helps to increase the precipitate number density. This eventually allows to increase the YS at the expense of ductility.

Alloying with additional precipitation-inducing elements can further alter the precipitate nucleation mechanisms, size, number density, and morphology, thereby affecting mechanical properties [36, 37]. For example, Jiao et al. [38]

observed a similar trend in Fe-3Mn-2Al-1.5Cu-5Ni alloy (wt%), where high Ni content promotes early nucleation of Ni-rich B2-type precipitates, subsequently followed by Cu-rich precipitation. Conversely, in low-Ni/Al and high-Cu alloy systems (*e.g.* in Fe-1.5Mn-0.5Al-1.5Ni-2.5Cu, wt%), Cu-rich precipitates nucleate directly from the super-saturated solid solution. The same group further observed that increased Mn and Ni content enhances the YS and UTS, primarily through a multi-fold increase in precipitate number density. However, this strategy compromises ductility, as B2-NiAl precipitates transform into NiAlMn (Heusler-type) precipitates, which are known to promote brittle fracture [39, 40].

Previous studies of Cu-alloyed MMnS revealed that combined Cu and Ni additions not only increase the austenite phase fraction, thereby improving strain hardening capacity [41], but also reduce the nucleation energy barrier for Cu- and Ni-rich co-precipitation [19, 25, 42, 43]. Consequently, Cu-Ni alloying has emerged as a promising route for enhancing both ductility and strength, specifically for Cu-Ni alloyed forged MMnS. Elbeltagy et al. [44] demonstrated that a two-step heat treatment involving intercritical annealing (IA) followed by tempering significantly improved YS in hot-forged MMnS. In previous work by our research group, Xu et al. [45] reported that warm-rolling, as compared to conventional hot- or cold-rolling, offers superior tensile properties and compact processing pathway. As widely used hot rolling process is typically accompanied by dynamic recovery and recrystallization, which limits dislocation retention and, consequently, the attainable strength [18]. Subsequent cold rolling introduces severe plastic deformation, leading to high strength but reduced ductility. As a result, post heat treatment is usually required to achieve a good strength-ductility balance after cold rolling. In contrast, warm rolling suppresses full recrystallization while introducing a refined microstructure with deformation induced substructures. This processing route enables dislocation retention and mechanically stabilized austenite, facilitating activation of the TRIP effect, and thereby contributing to a favorable balance between strength-ductility [46, 47].

Building on these findings, the present study aims to achieve a simultaneous enhancement of strength and ductility in warm-rolled MMnS through tailored post-warm-rolling heat treatments. The effects of IA and aging, individually and sequentially, on the microstructural evolution and mechanical response are systematically investigated. Four processing routes were examined: (1) warm-rolled (WarmR), (2) intercritically annealed (WRA), (3) aged (WRAg) and (4) intercritically annealed followed by aging (WRAA). The mechanical properties were assessed through tensile testing, while multi-scale microstructural characterization

was conducted via electron backscatter diffraction (EBSD) and three-dimensional atom probe tomography (3D-APT). The results provide comprehensive insight into the correlation between heat treatment, microstructural evolution, and nano-sized precipitation, thereby offering new guidance for optimizing the mechanical properties of advanced WarmR MMnSs through nano-structural design.

2 Materials and Experiments

2.1 Materials Fabrication

Warm-rolled (WarmR) medium-Mn steel (MMnS) with one-step and two-step post-warm-rolling heat treatment were investigated. Table 1 provides the detailed chemical composition of principle alloying elements, determined through pulse discrimination analysis using optical emission spectrometry (OES, OBLF company, Witten, Germany). Al and Si were alloyed to prevent the formation of carbides, while Mo was alloyed to avoid Mn-segregation at the grain boundaries [45]. The A_{C1} and A_{C3} temperature values listed in Table 1 were determined according to dilatometric experiments, reported in literature [45]. The ingots, with a cross-sectional size of $100 \times 100 \text{ mm}^2$, were produced using a vacuum induction furnace. Subsequently, the ingots were homogenized at 1250°C for 8 h and then hot-forged in multi-steps at 1150°C to achieve 10 mm thick plates. The hot-forged samples were ramp-heated to 750°C in 20 min, followed by 10 min soaking at the same temperature. The samples were subjected to a series of warm-rolling passes, resulting in a final sheet thickness of 2 mm, with each pass achieving a thickness reduction of 15–17%. The WarmR finish temperature was 610°C with a cumulative warm-rolling time of 2–3 min. The samples were subsequently cooled to room temperature in ambient air.

2.2 Heat Treatment

Four distinct heat treatment conditions were examined to establish the structure-property relationships of the investigated WarmR MMnS. The first condition corresponded to an as-WarmR state. The second condition involved an intercritical annealing (IA) treatment at 700°C for 1 min, this condition named WarmR + annealed (denoted as WRA) specimen. The third condition consisted of an aging treatment at 500°C for 5 min applied to the WarmR material, referred to as WRAg. The fourth condition, designated as WRAA, comprised a two-step heat treatment sequence: initial intercritical annealing at 700°C for 1 min followed by aging at 500°C for 5 min. All heat treatment processes were performed in pre-heated electrode salt bath furnaces

Table 1 Chemical composition (in wt%) and transformation temperatures (in $^\circ\text{C}$) of the investigated WarmR MMnS

Elements	Fe	Mn	C	Al	Si	Mo	Cu	Ni	P	S	N	A_{C1}	A_{C3}
WarmR	Bal.	7	0.05	1.5	1.5	0.5	1.5	1.5	0.005	0.004	0.006	588	940
MMnS													

ensuring uniform and rapid heating of samples, followed by water-quenching to room temperature. In the present study, a short IA time of 1 min was selected to prevent the massive formation of austenite that might transform into fresh martensite during quenching. The schematic drawings of all processing routes are presented in Fig. 1.

2.3 Materials Testing and Microstructure Characterization

Uniaxial tensile tests were conducted to evaluate the mechanical response of the WarmR, WRA, WRAg, and WRAA specimens. Dog-bone-shaped samples with a thickness of 1.5 mm and a gauge length of 30 mm were employed. The tests were performed on a ZWICK Z4204 universal testing machine (Zwick GmbH & Co. KG, Ulm, Germany) at an initial strain rate of $1 \times 10^{-3} \text{ s}^{-1}$ under the constant crosshead speed. Additional specimens were tested under identical conditions to ensure reproducibility of the results.

Electron backscatter diffraction (EBSD) analysis was carried out on the rolling direction-normal direction (RD-ND) plane to characterize the microstructural morphology, grain size, and phase fraction. Prior to EBSD assessment, the samples were ground using SiC abrasive paper and mechanically polished using suspensions down to 1 μm , followed by final polishing with an oxide polishing suspension to remove any traces stemming from surface deformation. EBSD mapping was performed using a Zeiss SIGMA field-emission scanning electron microscope (FE-SEM) equipped with an EBSD detector (Oxford Instruments Inc.). Measurements were conducted at an accelerating voltage of 20 kV, step size of 0.05 μm and working distance $\approx 15 \text{ mm}$. Post-processing and quantitative analysis were conducted using the AztecCrystal software package.

Three-dimensional atom probe tomography (3D-APT) analysis was performed on all four conditions (WarmR,

WRA, WRAg, and WRAA) using a Local Electrode Atom Probe 4000X-HR system (LEAP 4000XHR, CAMECA). Needle-shaped specimens, with $\approx 320 \text{ nm}$ length and $\approx 50 \text{ nm}$ curvature, were fabricated by employing lift-out and annular milling techniques with a FEI Helios NanoLab™ 660 dual-beam SEM-focused ion beam (FIB) system. The APT analyses were carried out in laser-pulsed mode at a base temperature of 60 K, with a laser energy of 30 pJ and a pulse frequency of 200 kHz. The three-dimensional reconstruction and quantitative compositional evaluations were performed using Integrated Visualization and Analysis Software (IVAS module, AP Suite version 6.0, CAMECA).

3 Results

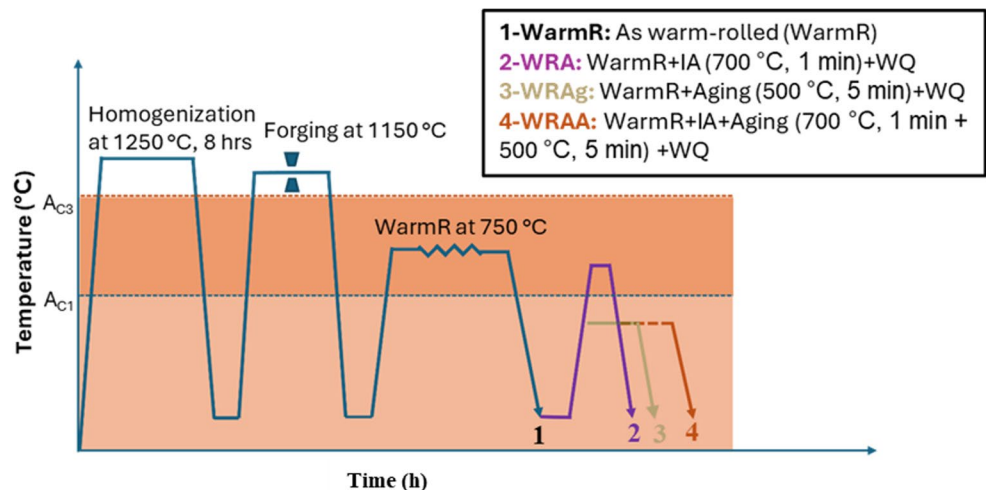
3.1 Microstructural Characterization by EBSD

The micro-scale structural information for WarmR MMnS and its heat-treated variants (WRA, WRAg and WRAA) was investigated by EBSD, focusing on phase fraction, grain size and morphological development of ferrite and austenite. Figure 2 shows the EBSD phase maps, where ferrite and austenite are depicted in blue and red, respectively, and low-angle grain boundaries (LAGB, 2° - 15°) and high-angle grain boundaries (HAGB, $>15^\circ$) are shown as green and black lines, respectively. Phase boundaries are indicated by light blue lines.

In the as-WarmR condition, the microstructure consisted predominantly of elongated ferrite grains aligned along the rolling-direction, with a ferrite phase fraction of 79.3 vol% and an austenite phase fraction of 20.7 vol% (Fig. 2a). The average grain size of ferrite and austenite phases was measured as 0.51 μm and 0.38 μm , respectively.

After direct IA (WRA condition), a slight increase in the austenite phase fraction to 22.6 vol% and a corresponding reduction in ferrite to 77.4 vol% was observed (Fig. 2b). The

Fig. 1 Schematic drawings of the heat treatment routes in the present study. WQ: Water Quenching; IA: Intercritical Annealing



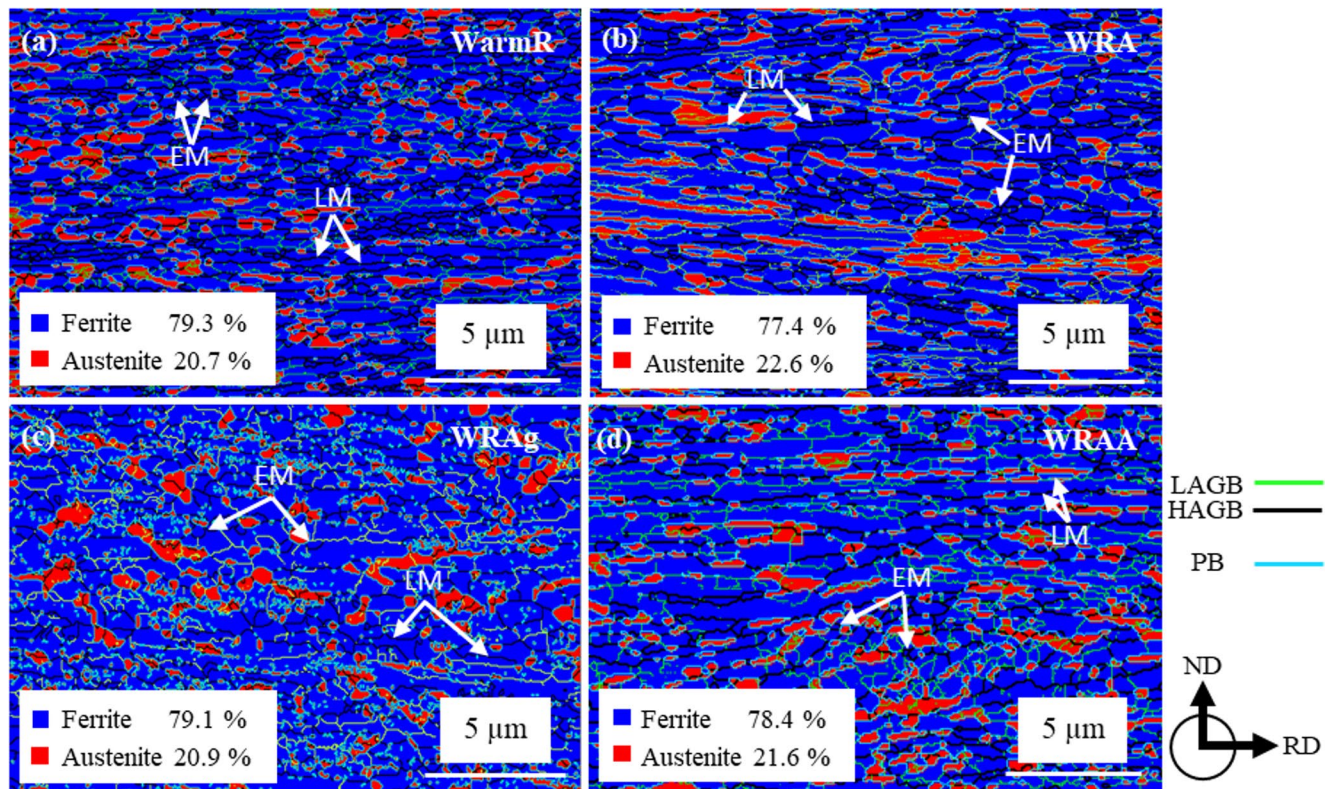


Fig. 2 EBSD phase maps of ferrite and austenite phases in MMnS at different heat treatment conditions; (a) WarmR, (b) WRA, (c) WRAg and (d) WRAA. LM: lamellar morphology; EM: equiaxed morphology; LAGB: low-angle grain boundary; HAGB: high-angle grain boundary

increased austenite fraction is attributed to the reverse austenite transformation at 700 °C, but due to the short IA duration, the reverse austenite transformation is not complete. The austenite might mainly form at preferential sites, e.g., *along the interfaces*, where Mn/C local enrichment prevails. The microstructure also exhibited partial recovery, as indicated by the increased mean grain sizes of ferrite (0.61 μm) and austenite (0.41 μm). For the WRAg specimen, the ferrite and austenite fractions were measured as 79.1 vol% and 20.9 vol%, respectively (Fig. 2c). The average grain sizes of ferrite (0.58 μm) and austenite (0.40 μm) remained similar to those of the WRA condition. The two-step heat-treated condition, i.e., WRAA specimen, exhibited a ferrite phase fraction of 78.4 vol% and an austenite phase fraction of 21.6 vol% (Fig. 2d). The ferrite and austenite grains are characterized by slight coarsening, with mean grain sizes of 0.77 μm and 0.48 μm, respectively.

Overall, the EBSD results indicate that the austenite fractions of the four specimens differ only slightly, within a range of ± 1.5 vol%. Besides the WarmR specimen that exhibited a heavily deformed structure and most pronounced substructures, eventually leading to a smaller EBSD-measured grain size, the other specimens (WRA, WRAg, and WRAA) showed no significant differences in austenite morphology or grain size. Consequently, variations in austenite fraction,

size, and morphology are not the primary factors controlling the tensile behaviors detailed before.

3.2 Tensile Properties

The mechanical properties of the WarmR, WRA, WRAg, and WRAA specimens were evaluated through uniaxial quasi-static tensile testing. The corresponding engineering stress-strain curves are presented in Fig. 3.

The WarmR specimen is characterized by a limited total elongation (TE) of $6.0 \pm 1.2\%$, while exhibiting a relatively high yield strength (YS) of 926.2 ± 17.8 MPa. The subsequent direct aging increased the YS, without sacrificing the ductility. As a result, WRAg specimen showed a high YS of 1179 ± 17.6 MPa and maintained a TE of $5.7 \pm 1.5\%$. Both WarmR and WRAg specimens suffered a lack in strain hardening capacity, exhibiting only minor uniform plastic deformation. The almost horizontal stress-strain curves of WarmR and WRAg indicate the inactivity of the transformation-induced plasticity (TRIP) effect. The ultimate tensile strengths of WarmR and WRAg specimens were 1126 ± 16.2 MPa and 1265.8 ± 13.8 MPa, respectively. The elevated strength of the WarmR condition was primarily attributed to the presence of a significant fraction of deformed ferrite and increased mechanical stability of the fine austenite grains

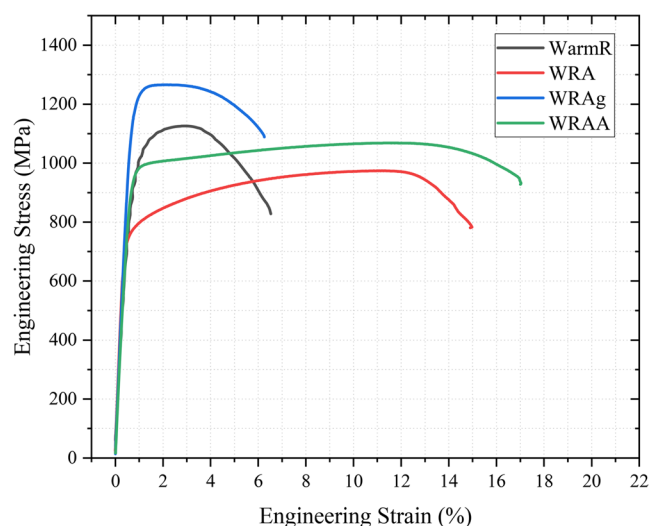


Fig. 3 Engineering stress-strain curves of the investigated WarmR, WRA, WRAg and WRAA MMnS

with an inherent high dislocation density, a characteristic microstructure of the warm-rolled state [45]. Furthermore, the increase in strength observed for the WRAg specimen is associated with the microstructural evolution within the ferrite phase. Here, the aging temperature is well below the A_{c1} temperature, which will be explicitly discussed in following chapters.

The intercritically annealed (WRA) sample showed distinct tensile properties. The YS and UTS of WRA specimens were lowered to 743.7 ± 25.1 MPa and 974.2 ± 3.9 MPa, respectively. The TE of the WRA specimens increased from $6 \pm 1.2\%$ to $14.5 \pm 2.6\%$ (compared to merely the warm-rolled specimen), indicating an improved balance between strength and ductility. IA+aging processed sample (WRAA) denoted a similar trend in influencing the mechanical properties. The WRAA condition exhibited YS of 938 ± 11.2 MPa, UTS of 1068.5 ± 19.0 MPa, and TE

elongation of $16.7 \pm 0.9\%$. Compared with the WRA specimen, direct aging after IA led to a significant improvement in the WRAA sample, where the ductility increased slightly (TE: $+2.2\%$), while the YS increased substantially by approximately 195 MPa. Relative to the sample subjected to warm rolling followed by direct aging (WRAg), the WRAA condition showed a reduction in YS, nevertheless, it still retained a comparatively high strength level among all MMnS conditions considered here, eventually reaching 938 ± 11.2 MPa. In comparison with the WRAg specimen, the most pronounced advantage of the WRAA sample is the significant enhancement in TE, increasing by nearly a factor of three ($5.7 \pm 1.5\%$ to $16.7 \pm 0.9\%$). The detailed relation between microstructure, nanostructure and properties will be discussed in the following chapters.

3.3 Near-atomic Characterization of Cu Precipitation and Local Chemistry by APT

3.3.1 WarmR - warm-rolled Specimen

Figure 4a presents the 3D-APT reconstruction illustrating the spatial distribution of major alloying elements Fe, Mn, C, Si, Al, Cu and Ni. Analysis of the elemental distributions showed that no Cu precipitates were detected when constructing iso-concentration surfaces (at%) for Cu. Similarly, iso-concentration surfaces generated for Al, Ni, and Mn revealed no noticeable enrichment. The frequency-distribution analysis further confirms the chemically uniform distribution of Cu, Al, Ni, and Mn. In contrast, C exhibits clear local enrichment within the APT dataset, consistent with its known tendency to segregate to dislocations (Fig. 4b). The high dislocation density seen in the warm-rolled specimen corroborates this behaviour [45].

Based on the Mn and Al distribution, the analysed APT specimen contained only a single phase. Therefore,

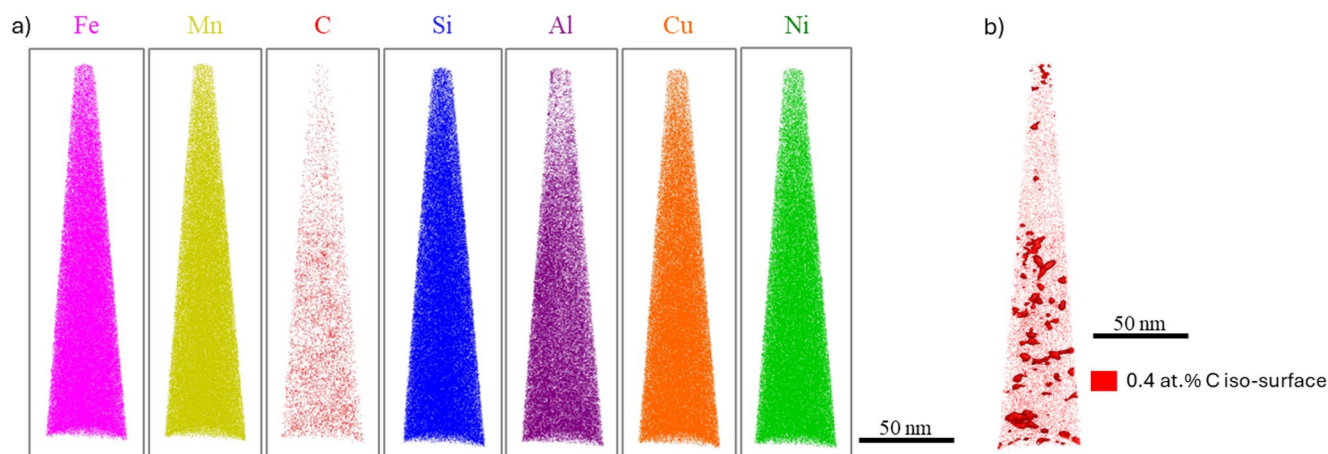
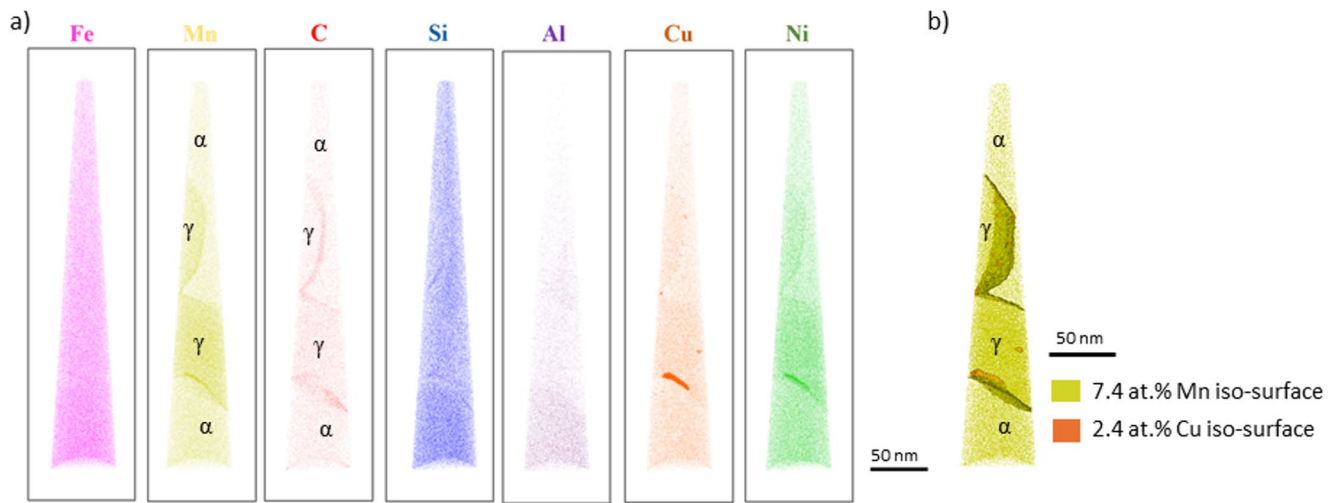


Fig. 4 APT analysis of WarmR specimen: (a) elemental map of Fe, Mn, C, Si, Al, Cu and Ni; (b) C elemental map imposed with 0.4 at.% C iso-concentration surfaces

Table 2 Chemical analysis of APT dataset in the WarmR specimen revealing the composition of ferrite phase

α -ferrite	Fe	Mn	C	Si	Al	Ni	Cu
At.%	Bal.	6.47	0.084	2.93	2.58	1.20	0.99
Wt.%	Bal.	6.55	0.019	1.52	1.28	1.30	1.16

**Fig. 5** APT analysis of WRA specimen: (a) elemental map of Fe, Mn, C, Si, Al, Cu and Ni; (b) Mn elemental map imposed with 7.4 at.% Mn iso-concentration surfaces and 2.4 at.% Cu iso-concentration surfaces**Table 3** Average chemical compositions of austenite and ferrite in the WRA specimen

α -ferrite	Fe	Mn	C	Si	Al	Ni	Cu
At.%	Bal.	5.03	0.077	4.36	2.81	0.82	0.45
Wt.%	Bal.	5.14	0.017	2.28	1.41	0.89	0.53
γ -austenite	Fe	Mn	C	Si	Al	Ni	Cu
At.%	Bal.	10.43	0.436	3.06	1.83	1.35	0.72
Wt.%	Bal.	10.56	0.096	1.58	0.91	1.46	0.84

a compositional analysis of the APT tip was conducted (considering peak decomposition), and the results are summarised in Table 2. The depletion of C and Mn compared to nominal composition confirms that the obtained APT dataset shows only the ferrite matrix in the WarmR specimen, no Cu precipitates are present.

3.3.2 WRA - warm-rolled +Intercritically Annealed Specimen

Figure 5 presents the 3D-APT reconstruction illustrating the spatial distribution of alloying elements in the WRA specimen. The elemental maps reveal enrichment of Mn, C, Ni, and Cu within one region corresponding to the austenite (γ) phase, accompanied by a depletion of these elements in the ferrite (α) phase. Iso-concentration surfaces of 7.4 at% Mn and 2.4 at% Cu are superimposed with a Mn elemental map in Fig. 5b. The Mn iso-surfaces represent the phase boundary between austenite and ferrite phases, eventually pinpointing the distinct concentrations of Mn in different phases. Herein, different Mn

iso-concentration thresholds (at%) were employed to distinguish between the austenite and ferrite phase, and for subsequent compositional analysis. Austenite is characterized by elevated Mn (and C) concentrations, while ferrite is characterized by lower Mn (and C) concentrations relative to the nominal alloy composition. Cu iso-surfaces indicate the existence of Cu-rich precipitates in the microstructure. According to the distribution, most of the Cu precipitates with spherical morphology are located at the austenite/ferrite interfaces, and only one Cu precipitate is observed inside the austenite phase. To quantify the chemical compositions of austenite and ferrite phase, regions of interest (ROIs) were placed inside individual austenite and ferrite phase regions. The average chemical compositions derived from these ROIs are summarized in Table 3. Compared to the chemical composition in the WarmR specimen, obvious elemental partitioning was observed. At 700 °C (above A_{C1}), austenite stabilizers (Mn, C, Ni, Cu) partitioned from the ferrite phase to the austenite phase, while ferrite stabilizer (Al, Si) exhibited the opposite partitioning behavior.

Nano-sized Cu precipitates were found in the WRA specimen, as shown in Fig. 6. Most of the Cu precipitate were of small size, spherical morphology and decorated the austenite/ferrite interface. A Cu precipitate with a plate-like morphology was observed, characterized by a markedly larger size compared to the remaining precipitates. Figure 6b-e explicitly shows the distribution and chemical concentration profiles of these representative Cu precipitates. In the proxygram concentration profiles (Fig. 6d), the distance-axis represents the spatial distance (in nm) measured normal to the iso-concentration surface and used to visualize the precipitate. Negative and positive distances correspond to the matrix region and progression toward the precipitate core (low concentration to high concentration of selected element type), respectively, with the zero position representing the precipitate-matrix boundary. The differing morphologies and number densities of Cu precipitates observed along the same type of interface can be attributed to precipitate coalescence and abnormal (discontinuous) growth. At the austenite/ferrite interface in Fig. 6b, the Cu precipitates display a high number density, spherical morphology and small dimensions, with radii between 1.1 nm and 1.8 nm. Their core compositions ranged within 30–50 at%, indicating early-stage clustering/precipitation (Fig. 6d). At the interface shown in Fig. 6c, only a single Cu precipitate was present; however, it is considerably larger and

has coarsened along the interface with a thickness ≈ 5 nm. This precipitate exhibited a high Cu concentration, reaching up to 75 at% (as shown in Fig. 6e). The IA at 700 °C is not expected to promote Cu precipitation since the upper precipitation window is 640 °C according to the thermodynamic calculation for this alloy [41]. The observed Cu precipitates at the austenite/ferrite interface were considered to form assisted by heterogeneous nucleation and fast grain boundary diffusion [48–50].

Besides the Cu precipitates located at austenite/ferrite interfaces, there was one exception where a Cu precipitate, with near spherical morphology, was found in the austenite phase, as shown in Fig. 7. Although a FCC-structured Cu precipitate is energetically favorable, many studies have reported the BCC-9R-3R-FCC Cu transformation in different steels [48, 51]. Due to low misfit between the original BCC-Cu and the ferrite phase, Cu precipitates are more frequently formed in ferrite phase within the duplex microstructure. The Cu precipitate in Fig. 7 is therefore considered as undissolved Cu precipitate (during reverse austenite transformation).

3.3.3 WRAg - warm-rolled + Aged Specimen

Figure 8 shows the 3D-APT reconstruction of major alloying elements in the WRAg specimen and the 3.5 at% Cu

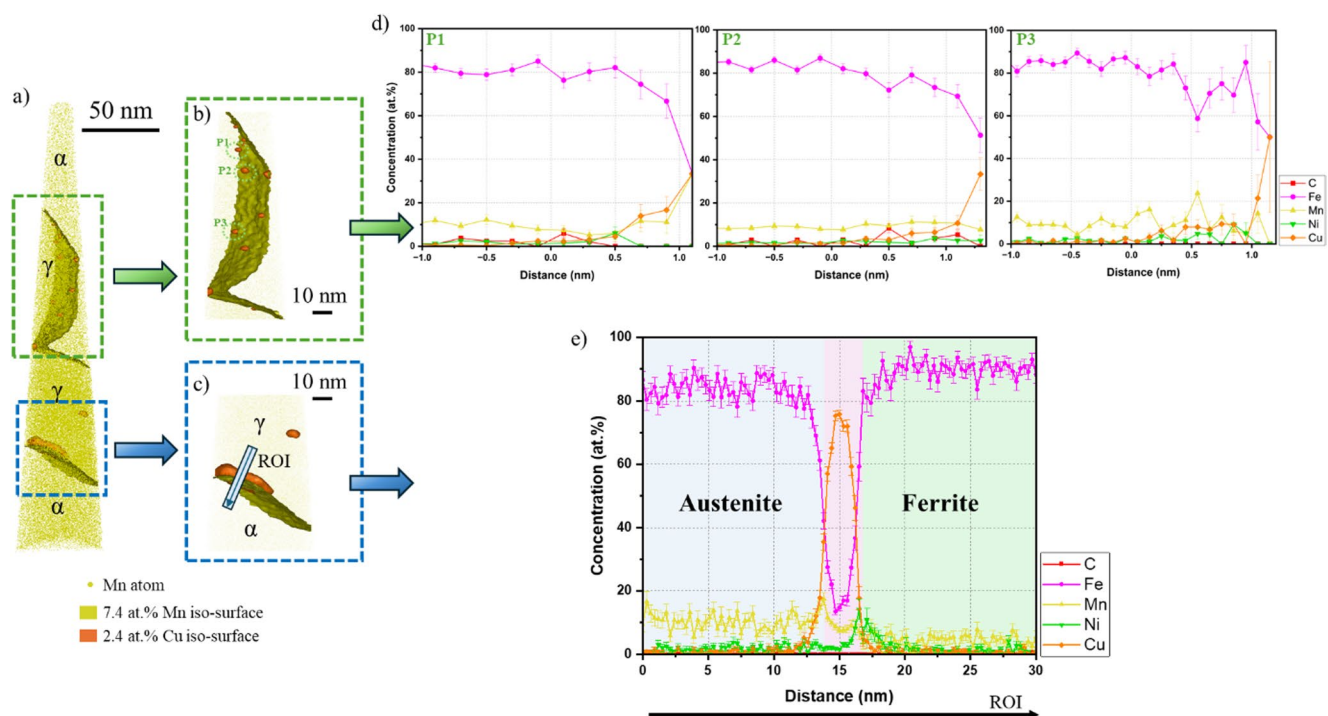


Fig. 6 APT analysis of a nano-size Cu precipitate located at the austenite/ferrite interfaces: **(a)** Mn elemental map imposed with 7.4 at.% Mn iso-concentration surfaces and 2.4 at.% Cu iso-concentration surfaces; **(b)** enlarged view of green box in **(a)**, three precipitates (P1-P3) are

marked for concentration profiles; **(c)** enlarged view of blue box in **(a)**, ROI is marked for concentration profiles; **(d)** proxygram concentration profiles of P1-P3 as marked in **(b)**; **(e)** concentration profiles along the arrow direction as marked in **(c)**

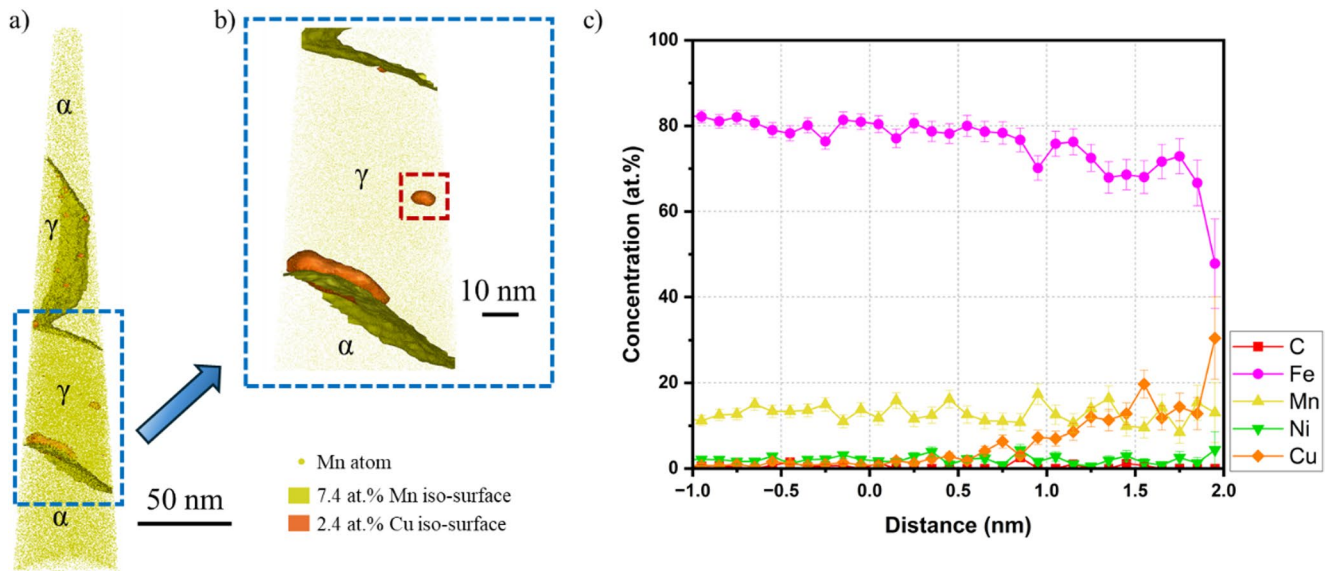


Fig. 7 APT analysis of a nano-size Cu precipitate in austenite: (a) Mn elemental map superimposed with 7.4 at.% Mn iso-concentration surfaces and 2.4 at.% Cu iso-concentration surfaces; (b) enlarged view of

the blue box in (a); (c) proxygram concentration profiles of the precipitate marked in (b)

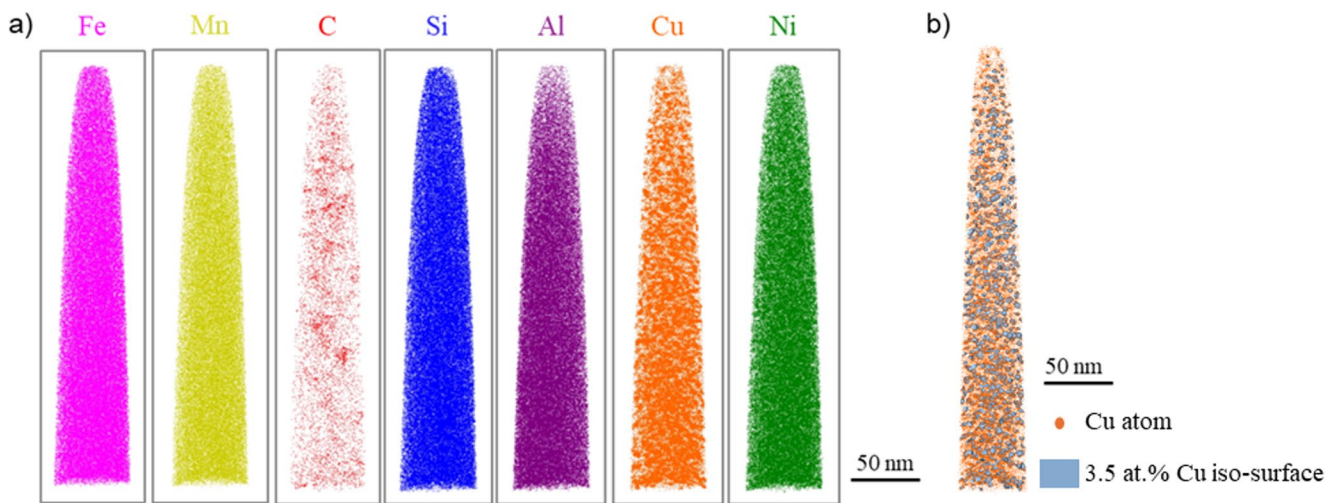


Fig. 8 APT analysis of WRAg specimen: (a) elemental map of Fe, Mn, C, Si, Al, Cu and Ni; (b) Cu elemental map superimposed with 3.5 at.% Cu iso-concentration surfaces

Table 4 Average chemical analysis of the matrix showing the composition of the ferrite phase

α -ferrite	Fe	Mn	C	Si	Al	Ni	Cu
At.%	Bal.	6.27	0.066	3.00	2.50	0.97	0.46
Wt.%	Bal.	6.35	0.015	1.55	1.24	1.05	0.54

iso-concentration surfaces superimposed in the Cu element map. The elemental maps reveal a uniform distribution of Fe and Mn throughout the analysed volume, while localized enrichment of C and Cu is evident. A large number density of the Cu precipitates could be visualized using 3.5 at.% Cu iso-concentration surfaces (Fig. 8b). To examine the chemical composition of the matrix embedding the Cu precipitates, two cylindrical ROI were placed inside the APT tip

while excluding any Cu precipitates. The average composition of the matrix is shown in Table 4. From the chemical composition with depleted C and Mn compared to nominal composition, the matrix in the APT dataset is confirmed as ferrite phase.

Figure 9 in addition shows the spatial distribution and chemical composition of the nano-sized Cu precipitates within the ferrite phase. A statistical analysis of fifteen randomly

selected particles yielded a mean radius $\approx 1.7 \pm 0.4$ nm. Five precipitates (P1-P5) were analysed in detail, and their proxygram concentration profiles are presented in Fig. 9b-f. Precipitate P1 exhibits Cu enrichment up ≈ 38.5 at%, and P2 ≈ 16.2 at%, P3 ≈ 30.8 at%, P4 ≈ 23.8 at%, and P5 ≈ 16.6 at% within their respective cores. All precipitates distribute homogeneously within the ferrite phase and display nearly spherical morphologies with compositional gradients characteristic of diffusion-controlled nucleation and early-stage growth.

Overall, the APT results of the WRAG MMnS confirm that a short aging treatment at 500 °C for 5 min promotes the formation of uniformly dispersed nano-sized Cu-rich precipitates within the ferrite phase.

3.3.4 WRAA - warm-rolled + Intercritically Annealed + Aged Specimen

Figure 10 shows the 3D-APT reconstruction of major alloying elements of the specimen subjected to a short IA step for 1 min at 700 °C and a subsequent aging step for 5 min at 500 °C, i.e., the *WRAA specimen*. The elemental maps reveal the enrichments of C and Mn along with the depletion of Al in the top region of the APT tip (marked as γ in Fig. 10a), representing the austenite phase. The bottom

region is characterized by the opposite enrichment/depletion behaviours of C/Mn and Al, eventually representing the ferrite phase. For better visualization, iso-concentration surfaces corresponding to 4 at% Mn (green, ferrite phase) and 10 at% Mn (red, austenite phase) were generated, as shown in Fig. 10b.

Quantitative compositional analysis was performed for representative ROIs in both phases, and the results are summarized in Table 5. As the austenite phase in the WRAA specimen was located near the apex of the APT tip, unstable field evaporation can occur during measurement, occasionally leading to insufficiently precise compositional data. Therefore, elemental partitioning behaviour is primarily inferred by comparing the compositional differences within the ferrite phase, while the austenite phase composition is used as supporting evidence. The additional aging process at 500 °C altered the elemental partitioning behaviours between ferrite and austenite phases, compared with WRA specimen (Table 3). During aging, although C, Mn, Cu and Ni are all austenite stabilizer, Mn and C tended to be further enriched in the austenite phase, while Cu and Ni were found to be enriched in the ferrite phase.

Nano-sized Cu precipitates with near spherical morphology, were found at the austenite/ferrite interfaces and inside the ferrite phase, as shown in Fig. 11. Cu precipitate

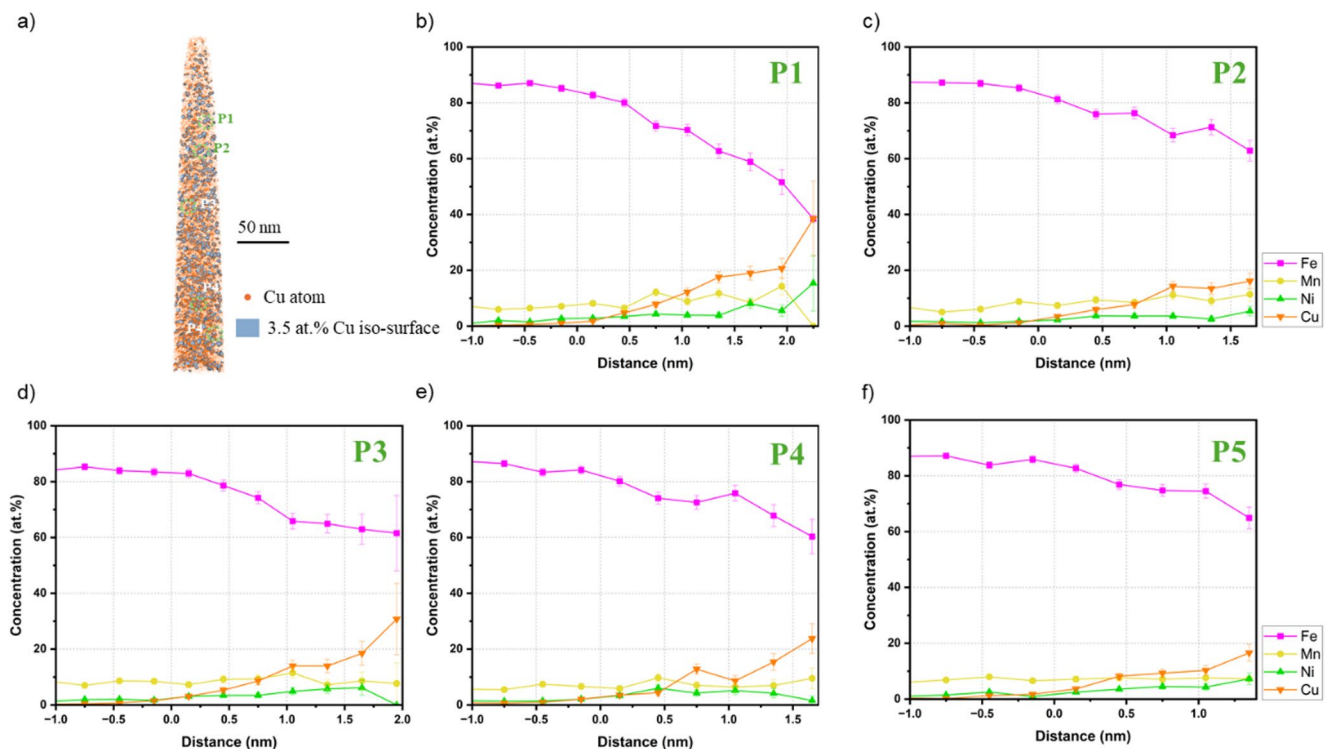


Fig. 9 APT analysis of nano-sized Cu precipitates in WRAG specimen: (a) reconstructed APT tip containing 3.5 at.% Cu iso-concentration surface; (b)–(f) proxygram concentration profiles of five selected nano-sized Cu precipitates as marked in (a)

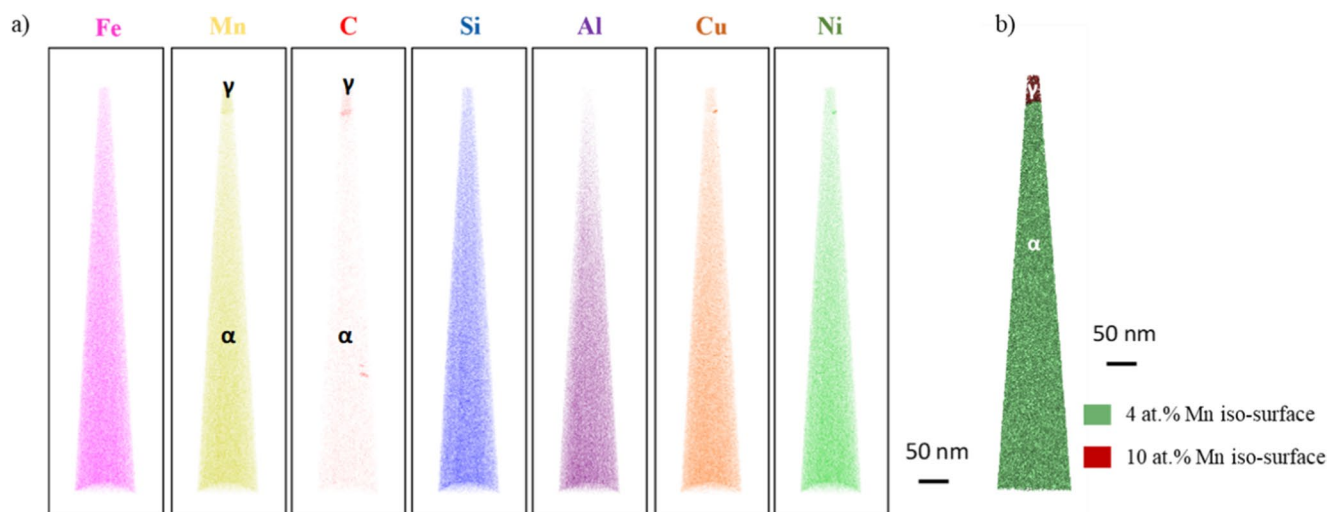
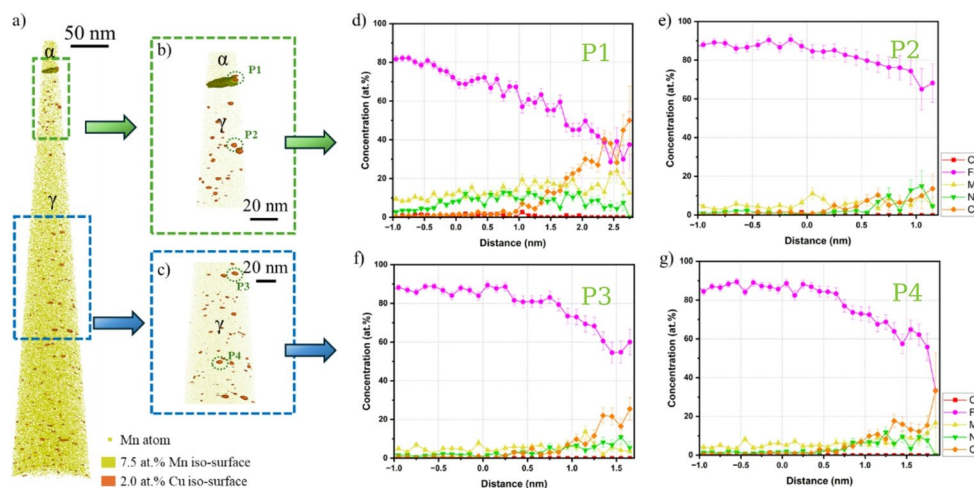


Fig. 10 APT analysis of WRAg specimen: (a) elemental map of Fe, Mn, C, Si, Al, Cu and Ni; (b) Green region indicates 4 at.% Mn iso-concentration surfaces, representing the ferrite phase, and red region indicates 10 at.% Mn iso-concentration surfaces, representing the austenite phase

Table 5 Chemical compositions of austenite and ferrite in the WRAA specimen

α -ferrite	Fe	Mn	C	Si	Al	Ni	Cu
At.%	Bal.	4.30	0.020	3.61	3.26	1.01	0.57
Wt.%	Bal.	4.38	0.004	1.88	1.63	1.10	0.67
γ -austenite	Fe	Mn	C	Si	Al	Ni	Cu
At.%	Bal.	10.07	0.580	3.38	1.78	1.24	0.55
Wt.%	Bal.	10.22	0.129	1.75	0.89	1.34	0.65

Fig. 11 APT analysis of nano-size Cu precipitate in WRAA specimen: (a) Mn elemental map imposed with 7.5 at.% Mn iso-concentration surfaces and 2.0 at.% Cu iso-concentration surfaces; (b) enlarged view of green box in (a), two precipitates (P1-P2) are marked for concentration profiles; (c) enlarged view of blue box in (a), two precipitates (P3-P4) are marked for concentration profiles; (d) proxygram concentration profiles of P1; (e) proxygram concentration profiles of P2; (f) proxygram concentration profiles of P3; (g) proxygram concentration profiles of P4



P1 was located at an austenite/ferrite interface, exhibiting a size ≈ 2.6 nm and Cu concentration ≈ 50 at% in the precipitate core. Statistical analysis of sixteen randomly selected Cu precipitates in the ferrite phase yielded an average radius $\approx 1.5 \pm 0.2$ nm. Cu precipitates P2-P4 were randomly selected for analysing the concentration profiles. Cu concentrations of these Cu precipitates ranged between 13 at% and 33 at%. In comparison with the Cu precipitates seen in the WRAg specimen, those in the WRAA specimen exhibited smaller sizes, lower number density, and reduced Cu concentrations in the core.

4 Discussion

4.1 Effect of Processing on Microstructural Evolution

The warm-rolling process for MMnS is lean and energy-efficient, and most importantly able to establish mechanical properties similar to those achieved by conventional hot-rolling-cold-rolling-annealing routes [45]. However, it also promotes microstructural heterogeneity, which necessitates

further microstructural optimisation. In the present work, a multi-scale characterisation approach was employed to elucidate the effects of direct short-time IA, direct aging, and continuous two-step IA + aging treatments on the warm-rolled microstructure, local chemistry, and Cu precipitation behaviour and, thus, to derive process-microstructure-property relationships.

The microstructure of the WarmR specimen contains about 20.7 vol% austenite and retains a high density of dislocations and substructures as a result of deformation [45]. The ferrite grains are refined (0.51 μm) and exhibit a lamellar morphology. APT analysis reveals that the ferrite phase contains relatively high amounts of Mn and C (the Mn concentration is only ≈ 0.5 wt% lower than the nominal alloy composition, Tables 1 and 2), indicating that the austenite phase has relatively low Mn and C concentration. The austenite in the WarmR condition is stabilised at room temperature primarily through grain refinement and the increased dislocation density. Introducing an additional short-time IA step slightly increases the austenite fraction (to 22.6 vol%), reflecting that limited reverse austenite can be retained at RT upon quenching from 700 °C. Furthermore, precise compositional analysis by APT shows that IA strongly promotes elemental partitioning, resulting in a much more pronounced Mn and C concentration contrast between ferrite and austenite. Since the IA temperature exceeds the recrystallization temperature, recovery and recrystallization reduce the dislocation density and promote the formation of equiaxed grains. The direct aging treatment at 500 °C after warm-rolling promotes the evolution of a less pronounced microstructural alteration compared with the 700 °C IA step. Because the temperature does not exceed A_{C1} , no reverse austenite transformation occurs, and the austenite fraction remains unchanged from the WarmR specimen. A comparison of the ferrite compositions (Tables 2 and 4) shows that the Mn, C, Si, and Al contents in the WarmR and WRAg ferrite phases are nearly the same. However, after aging, the ferrite phase exhibits a pronounced decrease in Ni and Cu concentrations, rationalized by the formation of Cu precipitates. Since 500 °C is below the recrystallization temperature, the deformation-induced substructures are not fully eliminated during aging.

The continuous combination of IA and aging further modifies the microstructure. The austenite fraction decreases slightly compared with the WRA specimen, reaching 21.6 vol%, which is attributed to the austenite-to-ferrite transformation occurring at the aging temperature. Owing to the preceding 700 °C IA step, elemental partitioning between austenite and ferrite has already taken place. Similar to the WRA condition, pronounced differences in Mn, C, Al, and Ni concentrations are observed between the two phases, consistent with thermodynamic expectations. Compared

with the WRA specimen, the subsequent aging step further drives Mn and C partitioning into austenite, while Ni and Cu partition in the opposite direction, i.e., *from austenite into ferrite*. This behaviour and its implications for Cu precipitation will be discussed in the following section.

4.2 Effect of Processing on Cu Precipitation

Building on the understanding of the microstructural evolution influenced by different processing routes, combined assessment of the APT results further enables the establishment of solid process-nanostructure (precipitation) relationships.

Starting from the WRA specimen, the heating and cooling cycle covers the Cu precipitation window. As shown in Fig. 6, most precipitates are located along the austenite/ferrite interfaces. This is because recovery and recrystallization significantly reduce the dislocation density in the ferrite phase, limiting the availability of preferential nucleation sites. The homogeneous nucleation for Cu precipitation is thus hindered. Consequently, nucleation occurs preferentially at interfaces, where heterogeneous nucleation is energetically favoured as shown in Fig. 6b. The interfacial regions not only lower the nucleation barrier, but also provide fast diffusion paths, eventually promoting accelerated precipitate growth. The large precipitates seen along the interface in Fig. 6c are likely the result of discontinuous growth and coalescence processes. The lower Cu concentrations measured in both the ferrite and austenite phases, relative to the Cu concentration in the ferrite of the WarmR specimen, further corroborate this behaviour.

The direct aging treatment applied to the WarmR specimen results in pronounced Cu precipitation within the ferrite phase. These Cu precipitates are characterized by small size (1.7 ± 0.4 nm), low-to-medium Cu concentrations, and high number density. The compositional analysis of the WRAg specimen (including both the ferrite matrix and the Cu precipitates) reveals a total Cu concentration of 0.93 at%, which is very similar to the Cu concentration in the WarmR specimen (0.99 at%). Given the high dislocation density present in the WarmR condition, which provides sufficient nucleation sites for Cu precipitation in the entire specimen volume, it is reasonable that the ferrite phase in the WRAg specimen contains a homogeneously distributed and high number density of Cu precipitates.

The precipitation behaviour in the WRAA specimen represents an intermediate state between the two conditions discussed above. The Cu precipitates remain primarily located within the ferrite phase. However, they exhibit smaller sizes (1.5 ± 0.2 nm) and a lower number density. During aging, Cu diffusion enables nucleation and growth within the ferrite matrix. The reduced number density arises

from the suppressed nucleation process compared with the WRAg specimen, which is attributed to the significantly lower dislocation density after IA.

The compositional analysis reveals that Cu partitioning is governed not only by thermodynamic austenite-ferrite phase equilibrium but also by the dynamic evolution of Cu precipitation. A direct comparison of the phase compositions in the WRA and WRAA specimens reveals that, when Cu precipitates form, Cu additionally partitions from austenite into ferrite to compensate for the depletion of Cu in the ferrite matrix caused by precipitation. The partitioning behaviour of Ni follows the same trend, which is consistent with previous studies by the authors on Ni-Cu interactions in the same alloy system [52].

4.3 Influence of Microstructure and Cu Precipitates on Tensile Properties

The mechanical properties of the investigated MMnS are jointly governed by the microstructure and the characteristics of the Cu precipitates.

The WarmR specimen exhibits a high YS of 926 ± 17.8 MPa and UTS of 1126 ± 16.3 MPa, attributable to its heavily deformed microstructure and the high dislocation density. Although the microstructure contains 20.7 vol% austenite, this retained austenite is characterized by a high dislocation density and is therefore mechanically stabilized, suppressing it from contributing to toughening and strain hardening through the TRIP effect [53].

Direct aging after warm rolling (WRAg specimen) effectively increases the YS to 1179 ± 17.6 MPa and simultaneously enhances the UTS to 1265.8 ± 13.8 MPa. The increase of both YS and UTS primarily arises from interactions between Cu precipitates and dislocations. According to the yielding mechanism reported by Ma et al. [54], Sun et al. [55], and Hu et al. [56], macro-yielding in MMnS occurs after micro-yielding within individual phases. Consequently, precipitation strengthening within the ferrite phase is responsible for the elevated YS. The ductility of the WRAg specimen remains low, as the TRIP effect continues to be suppressed. This suppression originates from two factors: (i) aging does not introduce additional austenite, and (ii) the aging temperature remains below the recrystallization temperature, therefore the limited recovery does not substantially reduce the dislocation density.

Short-term IA on the WarmR specimen (WRA) effectively improves the ductility, achieving a total elongation of $14.5 \pm 2.6\%$. However, the YS and UTS of the WRA specimen decrease to 743.7 ± 25.1 MPa and 974.2 ± 3.9 MPa, respectively. After IA, the dislocation densities in both ferrite and austenite are significantly reduced. Owing to the lack of dislocations serving as preferential nucleation sites,

Cu precipitates do not form within the ferrite matrix, and the few precipitates located at the austenite/ferrite interfaces do not contribute to strengthening. The improved ductility is attributed to the activation of the TRIP effect. Following recovery, partial recrystallization, and reverse austenite transformation during IA, the TRIP effect contributes to strain hardening, thereby enhancing the ductility.

The WRAA specimen exhibits a superior combination of ductility and strength. Compared with the WRA specimen, its YS and UTS increase by about 194 MPa and 95 MPa, respectively. Relative to the WRAg specimen, the WRAA specimen achieves a nearly threefold improvement in ductility at a small expense of strengthening. The high strength of the WRAA specimen is likewise attributed to precipitate-dislocation interactions, without contribution from dislocation strengthening. Similar to the WRA specimen, the reduction of dislocation density enables the austenite to effectively activate the TRIP effect, which enhances the strain-hardening rate and, thus, improves the ductility.

5 Conclusions

The present study systematically investigated the process-structure-property relationships in warm-rolled Cu-alloyed MMnS, Fe-7Mn-0.05 C-1.5Al-1.5Si-1.5Ni-1.5Cu (wt%). Through multi-scale characterization, the evolution of the microstructure and precipitates under different heat treatment routes was systematically investigated, and the role of the micro-/nanostructures in controlling the mechanical properties was elucidated. The IA step plays an important role by simultaneously reducing dislocation density, enhancing Mn/C partitioning, and re-activating the TRIP effect that is suppressed in the warm-rolled condition. Direct aging promotes homogeneous Cu precipitation within ferrite due to the high density of deformation-induced defects, while IA hinders precipitation in ferrite matrix. The two-step IA+aging route (WRAA) promotes the evolution of fine Cu precipitates with intermediate number density. Furthermore, the TRIP effect is activated. As a result, the WRAA condition achieves superior mechanical performance, combining TRIP-assisted strain hardening and moderate Cu-precipitation strengthening. Overall, this work provides a mechanistic study that connects processing, micro/nanostructure evolution, and mechanical behaviour, offering an effective strategy for tuning strength-ductility synergy in warm-rolled medium-Mn steels.

Additionally, the processing strategy examined in this study is, in principle, compatible with established industrial processing routes. The combination of warm rolling with short-time IA and aging can be accommodated within existing thermomechanical processing schedules and

continuous annealing lines commonly employed in the steel industry, thereby provides a potential means of improving the strength-ductility balance without requiring substantial changes to processing time or energy consumption.

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Data Availability Data will be available on request.

Declarations

Conflict of interest On behalf of all authors, the corresponding authors state that there is no conflict of interest.

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