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Effect of austenitizing temperature on the transformation of banded dual-phase structures

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Abstract

The dual-phase (DP) banded microstructure in HSLA steels, particularly API X52, often results in anisotropic mechanical performance due to non-uniform phase distribution. This study investigates the effect of austenitizing and intercritical annealing treatments on the morphology and mechanical properties of DP microstructures. API X52 steel samples were austenitized at 950, 1050, and 1150 °C, followed by intercritical annealing (Step Quenching, SQ) at 740, 780, and 800 °C to form ferrite-martensite dual-phase structures. Microstructural analysis revealed that increasing the austenitizing temperature significantly reduced banding. At 1150 °C, the banded structure was fully dissolved, producing a more homogeneous martensite dispersion. Hardness increased with intercritical temperature due to higher martensite content, while excessive austenitizing led to grain coarsening and reduced hardness. Charpy impact tests revealed a general decrease in absorbed energy with increasing austenitizing temperature, except at 1150 °C, where microstructural isotropy resulted in a slight recovery of toughness. SEM fracture surfaces confirmed ductile behavior, with a dimple morphology indicative of a uniform microstructure. These results demonstrate that austenitizing at 1150 °C followed by intercritical annealing is an effective route to suppress banding and improve mechanical isotropy in dual-phase API X52 steel.

Key words: austenitization, ferrite, banded structure, Charpy impact, hardness, dual phase

1. Introduction

High-strength low-alloy (HSLA) steels, such as API X52, are widely used in pipeline, automotive, and structural applications due to their excellent mechanical properties, good weldability, and cost-effectiveness. The mechanical behavior of these steels is strongly influenced by their microstructure, which can be tailored through thermal treatments. Among these, austenitizing – a process involving heating into the austenite phase field – is a critical step that governs grain size, phase transformation kinetics, and the distribution of alloying elements in solid solution.

The choice of austenitizing temperature has a significant impact on the dissolution of carbides, the growth of austenite grains, and the homogeneity of chemical composition. At lower austenitizing temperatures, 900 °C, the resulting microstructure is typically fine-grained, consisting of polygonal ferrite and pearlite, which provides good toughness but limits strength. At higher temperatures, 1200 °C, increased atomic mobility leads to substantial grain coarsening and partial or complete dissolution of pearlite. These changes influence the conditions under which dual-phase (ferrite + martensite) microstructures can form during subsequent intercritical annealing and quenching treatments.

However, excessive grain growth at high austenitizing temperatures can be detrimental, as it reduces the density of grain boundaries that act as nucleation sites for ferrite during cooling. This results in sluggish transformation kinetics and promotes mechanical anisotropy, particularly when banded microstructures develop. Banded structures are often associated with microsegregation, especially of elements like manganese (Mn), and with the weakening of the grain

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boundary pinning effect typically exerted by microalloying precipitates, such as niobium carbide (NbC) or titanium nitride (TiN). Recent studies have shown that these precipitates tend to coarsen or dissolve at elevated temperatures, thus reducing their effectiveness in controlling grain growth [1, 2].

Among the various microstructures that can be achieved in HSLA steels, the dual-phase (DP) structure is particularly attractive. Comprising a soft ferritic matrix reinforced with islands of hard martensite, this microstructure offers a favorable balance between strength, ductility, and strain hardening capacity [3, 4]. However, during thermomechanical treatments such as rolling followed by intercritical annealing and quenching, DP steels often exhibit a banded microstructure. In this configuration, ferrite and martensite phases become aligned into elongated, alternating layers or bands, usually parallel to the rolling direction [5]. This microstructural banding is primarily attributed to chemical segregation, particularly of alloying elements like Mn, which stabilize austenite in specific regions and promote anisotropic transformation upon cooling [6].

While banded structures may form naturally during steel processing, they can adversely affect mechanical performance by introducing anisotropy in deformation behavior. Specifically, these bands act as stress concentrators under loading, promoting early crack initiation and directional fracture propagation, especially under multiaxial or dynamic conditions such as impact or fatigue loading [7]. Moreover, the mechanical anisotropy induced by banding has been linked to variations in yield strength, ductility, and toughness depending on the orientation of applied stress relative to the band alignment [8]. Recent studies have highlighted that finer and more uniform distributions of martensite, achieved by optimizing intercritical heat treatment parameters, can significantly reduce band intensity and improve both strength and toughness [9, 10].

Recent studies have underscored the detrimental effects of banded microstructures on the mechanical anisotropy of HSLA steels, particularly in dual-phase (DP) configurations. Banded structures, characterized by alternating ferrite and martensite layers aligned along the rolling direction, can significantly compromise isotropic toughness, ductility, and fatigue resistance. For instance, Zidelmel et al. [11] demonstrated that in API-grade steels, the alignment of phases in bands reduces toughness in directions transverse to the band orientation due to early crack initiation and preferred crack propagation paths. This anisotropy is particularly pronounced under dynamic loading or multiaxial stress conditions.

The morphology, size, and spatial distribution of ferrite and martensite in dual-phase steels are strongly influenced by the thermal history, particularly during the austenitizing and intercritical annealing stages. Austenitizing at higher temperatures promotes the dissolution of cementite and other segregated phases, leading to a more homogeneous solid solution. This also enhances the recrystallization of deformed grains and the redistribution of alloying elements such as manganese (Mn), which is known to segregate and stabilize austenite during intercritical annealing [12, 13].

Moreover, increasing the austenitizing temperature accelerates the dissolution of alloy carbides (e.g., Fe_3C, NbC), thereby increasing the carbon content in the austenite phase. This elevated carbon content contributes to the formation of a harder martensitic phase upon quenching, while also influencing the morphology of the resulting dual-phase microstructure [14]. Studies by Zhang et al. (2017) [1] and Stefan et al. (2022) [2] confirm that elevated austenitizing temperatures lead to coarser austenite grains and higher carbon enrichment in solid solution, which in turn affects transformation kinetics and the final phase distribution. However, this grain coarsening can also suppress ferrite nucleation during intercritical treatment, reduce boundary density, and, if not properly controlled, exacerbate banding [15, 16].

Furthermore, the suppression of band formation through optimized thermal treatments has been shown to improve mechanical isotropy. For example, [14] reported that steels austenitized at 1150 ℃ followed by intercritical annealing exhibited a more dispersed martensitic phase with reduced band intensity, which correlated with improved impact toughness and uniform mechanical behavior in all directions.

These findings highlight the importance of balancing austenitizing parameters to simultaneously control grain size, carbon distribution, and phase morphology, thereby minimizing anisotropy and optimizing the mechanical performance of HSLA steels.

The objective of this study is to investigate the influence of austenitizing temperature on the evolution and mitigation of banded dual-phase microstructures in API X52 high-strength low-alloy (HSLA) steel. To this end, samples were subjected to austenitizing at three temperatures – 950, 1050, and 1150 ℃ – followed by intercritical annealing at 740, 780, and 820 ℃, and subsequently treated using a Step Quenching (SQ) process.

The resulting microstructures were characterized by using optical microscopy to assess phase morphology and the extent of banding. In addition to hardness testing, Charpy impact tests were conducted to evaluate the material's fracture toughness and to investigate the relationship between banding intensity and impact energy absorption. This study aims to propose an optimized thermal treatment strategy that minimizes microstructural banding and enhances isotropy in both strength and toughness, thereby im-

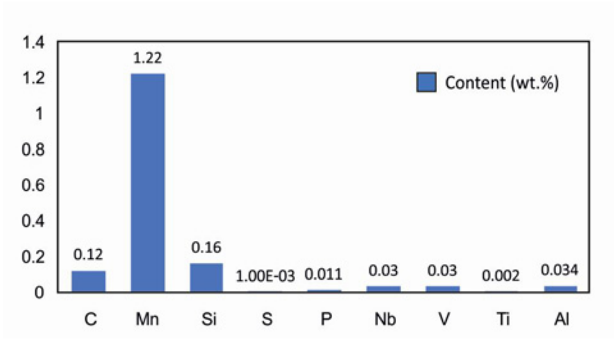

Fig. 1. Chemical composition of steel X52 (wt.%).

proving the performance reliability of pipeline-grade steels under dynamic loading conditions.

2. Experimental procedure

This study utilizes X52 high-strength steel, provided by Alpha Pipe in Ghardaïa, Algeria. The chemical composition of the steel, as determined by optical emission spectroscopy, is presented in Fig. 1.

The steel was received in the form of 14 mm thick plates, which were machined into specimens with dimensions of 20 mm × 20 mm × 5 mm for microstructural and electrochemical analyses. Steel samples were sectioned into rectangular coupons of dimensions 10 mm × 10 mm × 5 mm using precision cutting. All samples were ground progressively using SiC papers up to 1200 grit and polished with 1 µm diamond suspension to obtain a mirror-like finish for microstructural analysis.

The heat treatment process involved two main stages to develop and modify the dual-phase microstructure. In the first stage, the steel samples were austenitized at three different temperatures: 950, 1050, and 1150 ℃. Each sample was held at the respective temperature for 30 minutes to ensure complete transformation into austenite and allow for the homogenization of alloying elements. After austenitization, the samples were cooled in air. In the second stage, the austenitized samples were subjected to intercritical annealing at temperatures of 740, 780, and 800 ℃ – selected within the two-phase $(\alpha + \gamma)$ region of the phase diagram. The holding time at each intercritical temperature was 30 minutes, after which the samples were immediately quenched in agitated water at room temperature. This sequence, known as Step Quenching (SQ), as shown in Fig. 2, was designed to produce a dual-phase microstructure consisting of a ferritic matrix with a martensitic second phase. By varying the austenitization temperature prior to intercritical annealing, the influence on band structure modification and martensite distribution was investigated.

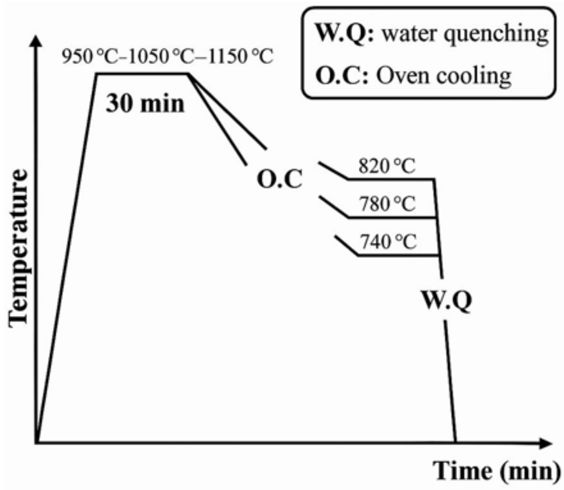

Fig. 2. The SQ thermal cycle applied to X52 steel in the dual-phase condition.

Metallographic examination was carried out on all heat-treated samples. After etching with 4 % Nital solution, the microstructure was observed using an optical microscope at magnifications ranging from 200× to 500×. Selected samples were further examined by scanning electron microscopy (SEM) to reveal the morphology and distribution of martensite within the ferritic matrix.

Vickers microhardness measurements were performed using a 500 g load with a dwell time of 15 seconds. For each sample, five indentations were taken at different locations, and the average value was reported.

The impact toughness (resilience) of the heat-treated API X52 steel samples was evaluated using Charpy V-notch tests in accordance with ASTM E23 standards. Standard specimens measuring 55 mm × 10 mm × 10 mm with a 2 mm deep V-notch were machined from each heat-treated condition, with the notch oriented perpendicular to the rolling direction, to assess the effect of band orientation on crack propagation. The tests were conducted at room temperature using a pendulum impact testing machine with a capacity of 300 J. For each condition, three specimens were tested, and the average absorbed energy was recorded.

3. Results and discussion

3.1. Microstructure characteristics

Figure 3 illustrates the microstructural features of X52 steel in its as-received condition, observed through optical microscopy. The microstructure consists of ferrite (white regions) and pearlite (black re-

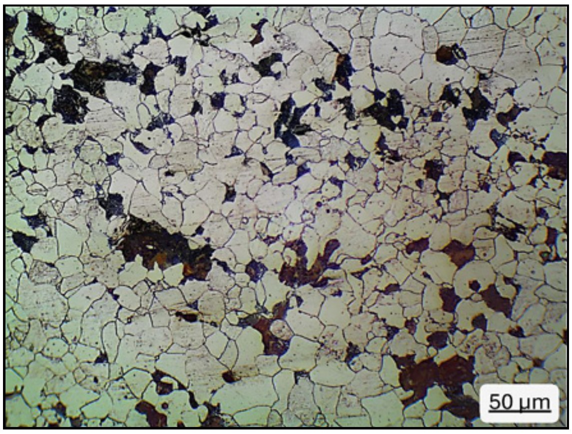

Fig. 3. The optical micrograph of as-received steel.

gions). Ferrite, a soft and ductile phase, dominates the structure with a calculated proportion of 77.81 % (14 µm), while pearlite, a harder and stronger phase consisting of alternating layers of ferrite and cementite, accounts for 22.19 %. These proportions were determined using *ImageJ software*.

The optical microstructures of X52 steel subjected to different austenitizing temperatures are illustrated in Fig. 4. At the lower austenitizing temperature of 950 ℃ (Fig. 4a), the microstructure is characterized by a fine-grained mixture of polygonal ferrite and pearlite. The ferrite grains are relatively small and equiaxed, and the pearlite colonies are well dispersed, indicating limited grain growth and moderate diffusion activity. As the austenitizing temperature increases to 1050 ℃ (Fig. 4b), partial dissolution of pearlite occurs, and the microstructure becomes more homogenized, with a noticeable increase in ferrite grain size. The remaining pearlite colonies appear more spheroidized, and the contrast between the phases begins to diminish. At the highest austenitizing temperature of 1150 ℃ (Fig. 4c), the microstructure exhibits substantial grain coarsening, with large polygonal ferrite grains and a significant reduction in pearlitic features due to their full or near-complete dissolution. This coarsening is attributed to enhanced atomic mobility at elevated temperatures, which promotes grain boundary migration and reduces the number of ferrite nucleation sites during subsequent cooling [17].

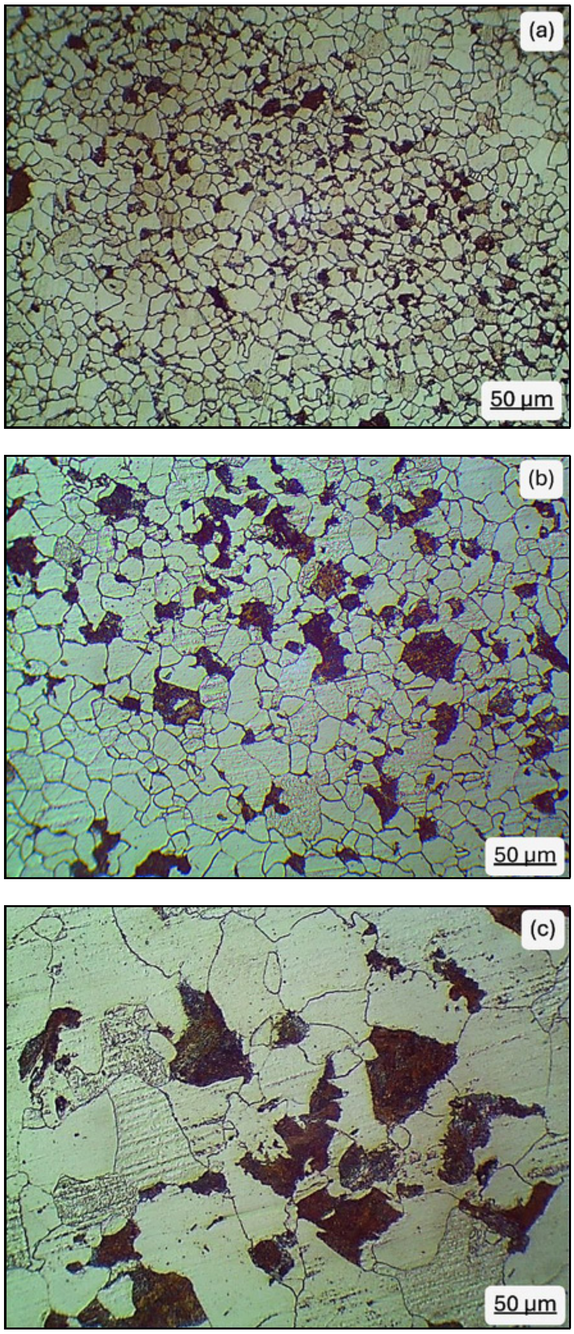

Fig. 4. Microstructure of X52 steel at different austenitizing temperatures: (a) $T = 950$ ℃, (b) $T = 1050$ ℃, and (c) $T = 1150$ ℃.

Figure 5 presents the optical microstructures of X52 steel subjected to a 30-minute SQ treatment, illustrating the formation and evolution of a dual-phase (ferrite + martensite) structure. The micrographs reveal a distinct banded morphology characterized by alternating ferrite and martensite layers aligned along the rolling direction. This banded distribution becomes more prominent with increasing intercritical annealing temperature, due to the thermally activated phase separation mechanisms. During SQ, the steel is initially fully austenitized before being cooled to the intercritical region ($\alpha + \gamma$), where ferrite begins to nucleate heterogeneously along the prior austenite grain boundaries and grows into the austenite matrix [18]. The resulting microstructure consists of separated regions rich in ferrite and retained austenite [19].

The formation of bands is primarily attributed to chemical segregation, particularly of manganese (Mn),

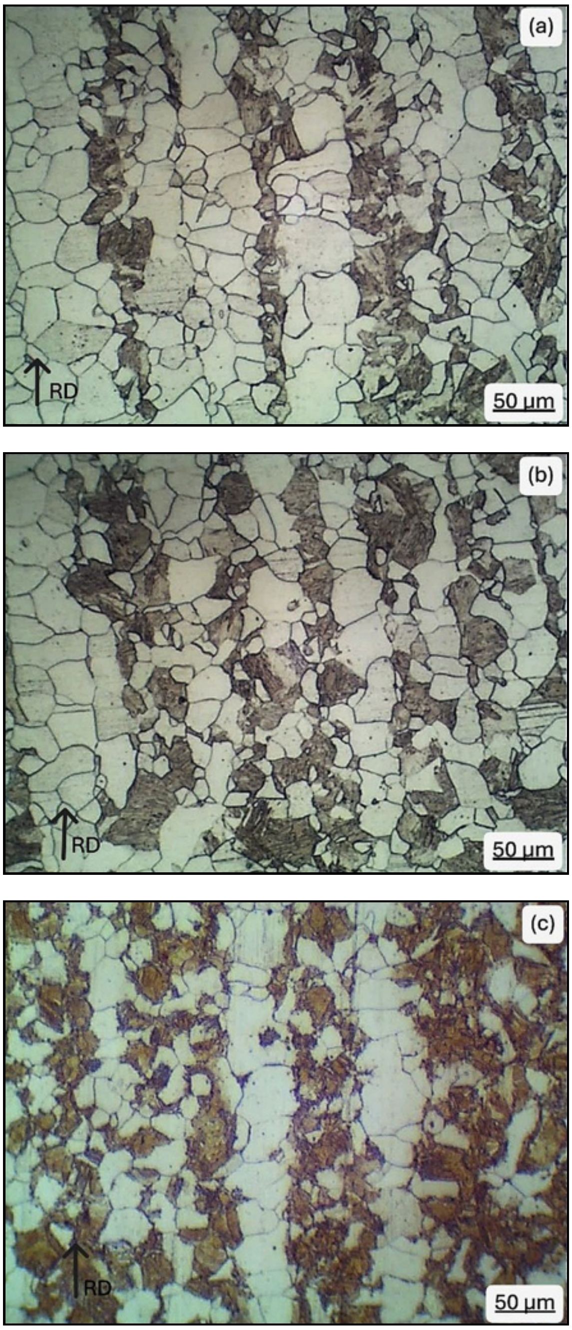

Fig. 5. Optical micrographs of X52 steel of SQ treatment with austenitizing at 950 ℃: (a) $T = 740$℃, (b) $T = 780$℃, and (c) $T = 820$℃.

Table 1. Volume fraction (%)

Temperature (℃)	740	780	820
	Volume fraction (%)		
Martensite	29.77	46.51	60.1
Ferrite	70.33	53.49	39.9

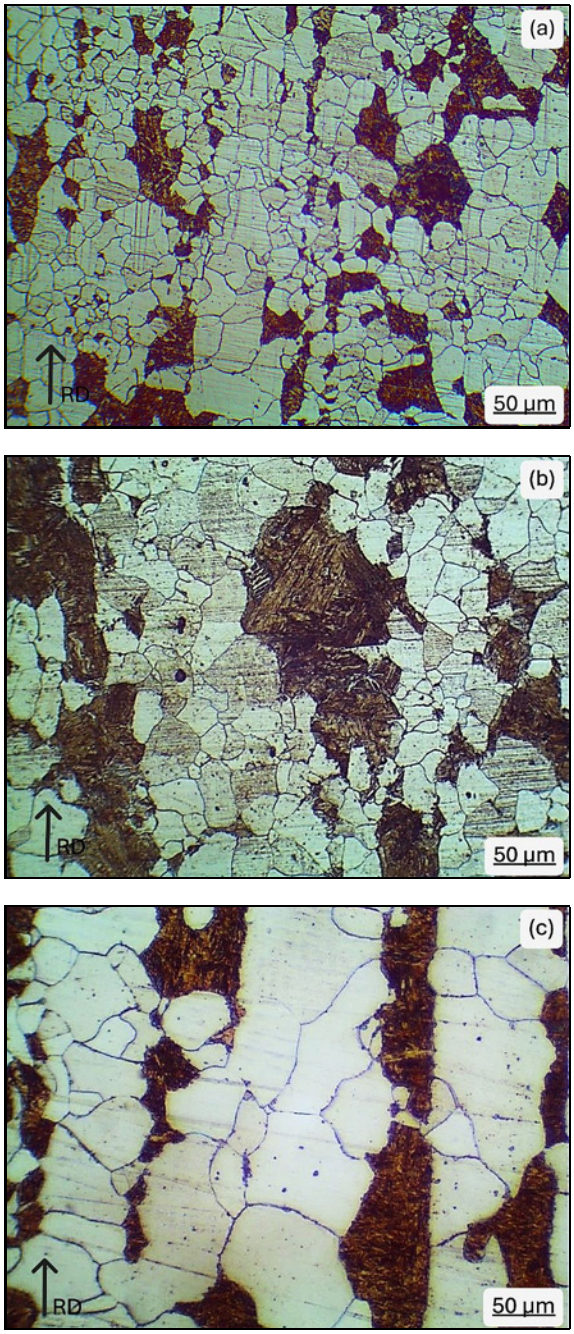

Fig. 6. Optical micrographs of X52 steel of SQ treatment with austenitizing at 1050 ℃: (a) $T = 740$℃, (b) $T = 780$℃, and (c) $T = 820$℃.

which diffuses slowly during solidification and remains segregated even after hot deformation. According to Offerman et al. [20], Mn-rich zones stabilize austenite, while Mn-depleted areas favor ferrite nucleation. During slow cooling, carbon atoms tend to diffuse from Mn-lean to Mn-rich areas, enhancing this phase contrast. Upon subsequent quenching, the Mn-rich retained austenite transforms into martensite, resulting in a persistent banded structure of alternating ferrite and martensite. Verhoeven [21] supports this the-

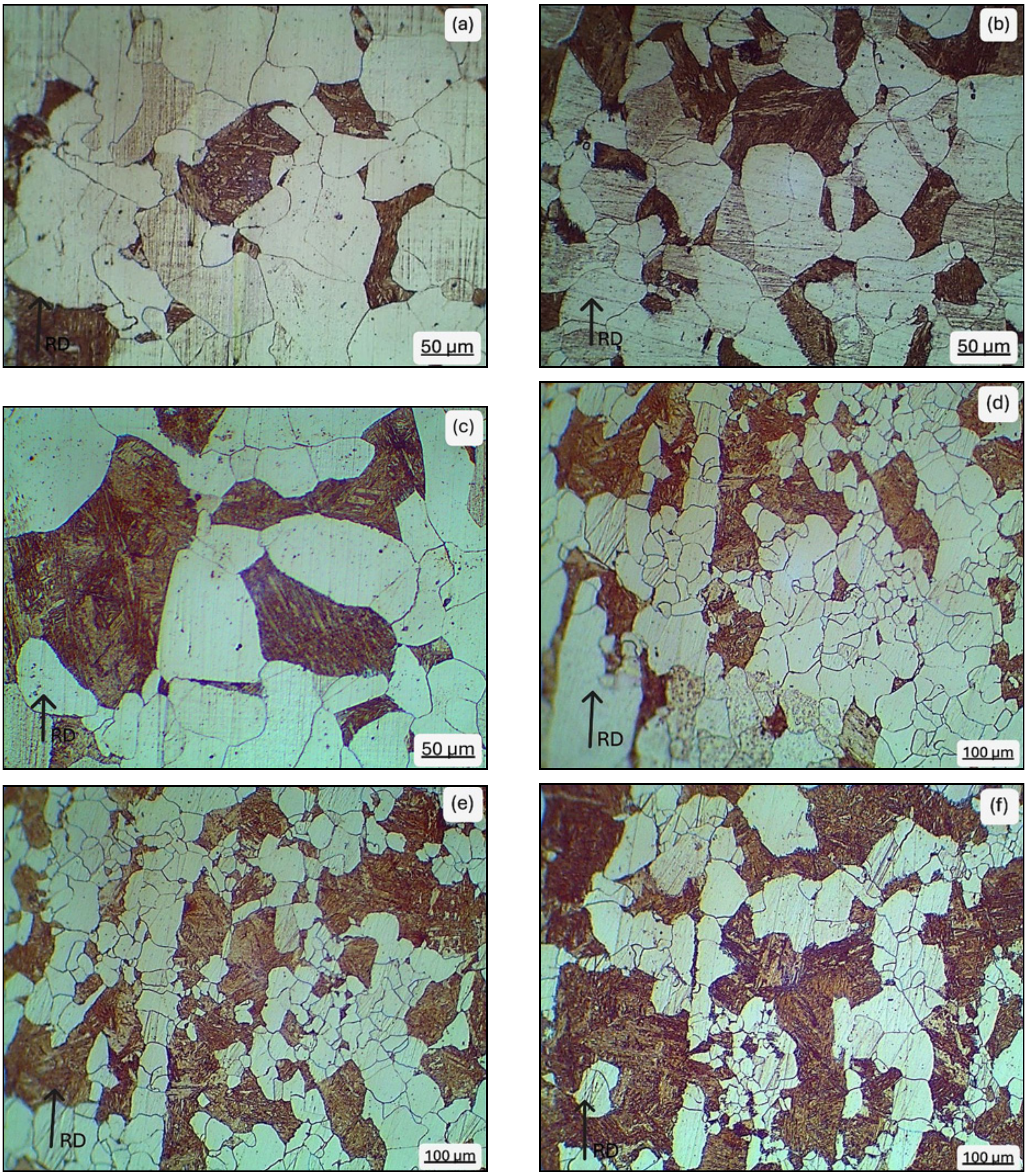

Fig. 7. Optical micrographs of X52 steel of SQ treatment with austenitizing at 1150 ℃: (a) and (d) $T = 740$ ℃, (b) and (e) $T = 780$ ℃, (c) and (f) $T = 820$ ℃.

ory by explaining that the thermodynamic stability of austenite in Mn-rich regions causes a delay in the ferrite transformation, reinforcing the observed morphological alignment.

Furthermore, at higher intercritical temperatures, the overall fraction of martensite increases due to the larger volume of austenite available for transformation. However, the stability of band formation remains as long as the segregation remains significant. The persistence of this banded structure is problematic for mechanical performance, particularly in terms of toughness and isotropy, as it may serve as preferential paths for crack initiation and propagation under stress. Thus, understanding and controlling the thermal history – particularly cooling rate, intercritical holding temperature, and austenitizing conditions – is essential to mitigating band formation and promoting a more homogeneous, isotropic dual-phase microstructure. The evolution of martensite content with temperature is quantitatively confirmed in Table 1, which shows a decrease in martensite fraction at excessively high intercritical temperatures, likely due to reduced undercooling and coarsening effects.

Figures 6 and 7 illustrate the evolution of the banded microstructure as a function of the Intercritical Annealing Temperatures (ICT), with prior austenitization performed at 1050 and 1150 ℃ for 30 minutes, respectively.

At an austenitizing temperature of 1050 ℃, the banded microstructure is already well-developed locally. Large martensite islands appear to connect multiple bands, as shown in Fig. 6a. This suggests a heterogeneous distribution of phases, where martensite tends to form within manganese-enriched zones aligned along the bands. Compared to the microstructures obtained at 950 ℃ (not shown here), the grains are significantly coarser, and the pronounced banded morphology is less evident in Figs. 6b,c. This change in morphology highlights the significant role of austenitic grain size in influencing phase transformation kinetics. Specifically, larger austenitic grains reduce the density of nucleation sites available for ferrite formation during cooling, since grain boundaries act as primary nucleation sites for ferrite. Furthermore, the increased diffusion distances within larger grains slow the transformation kinetics, limiting the formation of ferrite bands.

These observations align well with the conclusions of Thompson and Howell [18], who reported that banding diminishes or disappears as the austenitic grain size increases. They emphasized that when grains become sufficiently large, the nucleation effect at grain boundaries surpasses the chemical segregation effects (such as manganese banding), thus preventing the formation of the characteristic dual-phase bands.

At the highest austenitizing temperature of 1150 ℃ (Fig. 7), the banded microstructure typically observed in dual-phase X52 steel is completely eliminated, resulting in a homogeneous dispersion of martensite within the ferritic matrix. This transformation is primarily attributed to the intensified atomic diffusion at elevated temperatures, which enhances the redistribution of alloying elements such as Mn and C, thereby reducing chemical segregation, a key factor in band formation. Moreover, the high temperature promotes the dissolution of pearlite and other segregated phases, while increasing austenite grain size and reducing the influence of localized composition differences. Therefore, during subsequent intercritical annealing and quenching, the nucleation and growth of martensite occur more uniformly across the microstructure rather than along segregated bands. This microstructural uniformity is further supported by low-magnification observations presented in supplemental Figs. 7d–f, which reveal a consistent martensite distribution over a broader field of view, confirming the elimination of morphological anisotropy and the enhancement of isotropy across the transformed regions.

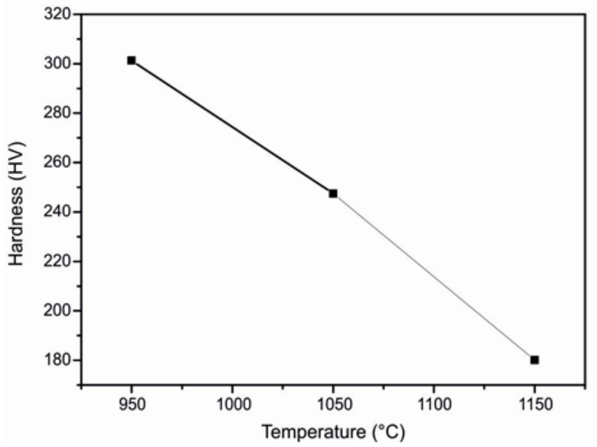

Fig. 8. Evolution of hardness as a function of austenitizing temperature.

3.2. Mechanical properties

The variation in hardness of API X52 steel as a function of austenitizing temperature is presented in Fig. 8. The general trend indicates that hardness increases with increasing austenitizing temperature. This can be attributed to the growth of ferrite grains and the gradual dissolution of pearlite at higher temperatures, resulting in a more homogeneous austenitic phase before cooling. As the austenitizing temperature rises – from 950 to 1150 ℃ – the microstructure becomes increasingly enriched in austenite, with fewer undissolved constituents. Upon air cooling, this austenite transforms into finer pearlite and harder bainitic structures. Additionally, grain growth at higher temperatures may lead to an increase in dislocation density during cooling, contributing to enhanced hardness. Similar observations have been reported in HSLA steels, where elevated austenitizing temperatures promote solid solution strengthening and partial phase transformation, leading to increased hardness values [1, 2].

The relationship between microstructure and mechanical properties in dual-phase steels such as API X52 is complex and influenced by multiple factors, including chemical composition, austenitizing and intercritical annealing parameters, as well as the volume fraction, morphology, and spatial distribution of martensite within the ferritic matrix.

The variation in the hardness of dual-phase X52 steel with respect to the martensite content, influenced by different intercritical annealing temperatures, is illustrated in Fig. 9. As the intercritical temperature increases, the volume fraction of martensite rises, resulting in a noticeable increase in overall hardness. This

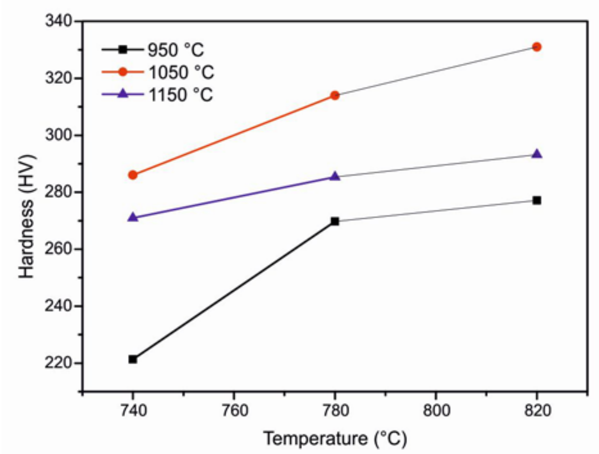

Fig. 9. Evolution of hardness as a function of intercritical temperature at different austenitizing temperatures.

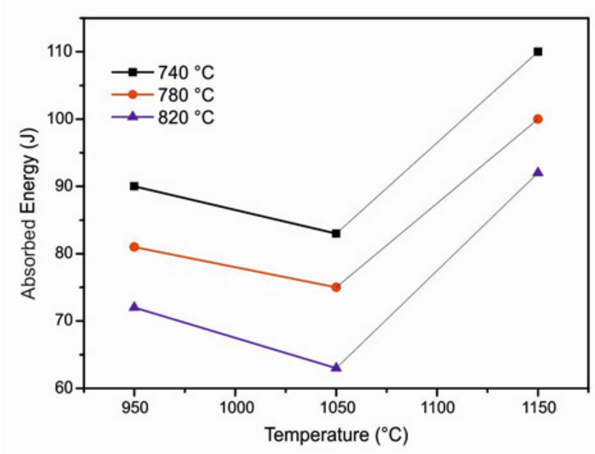

Fig. 11. Variation of absorbed energy as a function of austenitizing temperature for different intercritical annealing temperatures.

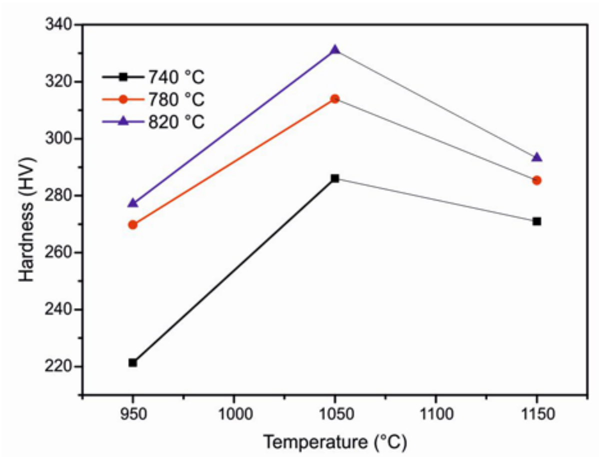

Fig. 10. Variation of hardness as a function of austenitizing temperature for different intercritical annealing temperatures.

is because martensite, being a significantly harder phase than ferrite, contributes directly to the steel's strengthening [22]. This trend holds true regardless of the prior austenitizing temperature or holding time, indicating that the intercritical temperature plays a dominant role in controlling the phase distribution and thus the mechanical response.

Figure 10 illustrates the evolution of hardness in dual-phase X52 steel as a function of austenitizing temperature for three intercritical annealing temperatures. The data presented in Fig. 9 demonstrates a clear correlation between austenitizing temperature and the resulting hardness of dual-phase X52 steel under various intercritical annealing conditions. For all intercritical temperatures, the hardness initially increases with rising austenitizing temperature, reaching a maximum at 1050 ℃, and then decreases at 1150 ℃. This trend is closely linked to microstructural evolution. At lower austenitizing temperatures (e.g., 950 ℃), the steel exhibits a banded dual-phase structure, where ferrite and martensite are aligned due to prior rolling and chemical segregation. As the austenitizing temperature increases, diffusion is enhanced, leading to homogenization of alloying elements, dissolution of segregated phases, and grain refinement. At 1150 ℃, the microstructure becomes fully uniform, with martensite islands finely distributed within a ferritic matrix, and the previously observed banded morphology disappears completely. This uniform distribution of phases correlates with improved isotropy in the microstructure but may lead to a slight reduction in hardness due to grain coarsening and a decrease in phase boundary density, explaining the observed drop in hardness at the highest austenitizing temperature.

The absorbed energy behavior of dual-phase X52 steel varies significantly with changes in austenitizing temperature and intercritical annealing conditions. As shown in Fig. 11, for all three intercritical temperatures (740, 780, and 820 ℃), the absorbed energy initially decreases when the austenitizing temperature increases from 950 to 1050 ℃, reaching a minimum. This reduction in toughness can be attributed to grain coarsening and the development of heterogeneous microstructures, which promote crack initiation and reduce the steel's ability to absorb energy under impact. At 1050 ℃, the microstructure often contains coarse martensite islands with poor distribution within the ferritic matrix, resulting in stress concentrations and reduced ductility.

However, a sharp increase in absorbed energy is observed at 1150 ℃ across all intercritical treatments. This improvement is directly related to the microstructural homogenization induced by the elevated austenitizing temperature. At 1150 ℃, the dissolution of segregated phases and increased atomic diffusion led to the elimination of banded structures and the formation of a uniform dual-phase microstructure, in which martensite islands are finely and evenly dispersed within the ferrite matrix. This uniformity enhances the toughness by reducing anisotropy and limiting preferential crack paths, especially under dynamic loading conditions.

The data suggests that while higher intercritical temperatures generally increase the martensite content (which can be detrimental to toughness), the positive effects of a well-refined and isotropic microstructure – achieved through higher austenitizing temperatures – can compensate and even enhance the overall impact resistance of the steel. Therefore, selecting an optimal combination of austenitizing and intercritical annealing parameters is critical for achieving a balance between hardness and toughness in dual-phase HSLA steels.

3.3. Failure mode during resilience testing

At lower austenitizing temperatures such as 950 ℃, the microstructure retains fine ferrite and a controlled amount of martensite, which favors ductile fracture behavior (Fig. 12a). This is evidenced by the presence of numerous large and deep dimples on the Charpy fracture surface, resulting from void nucleation, growth, and coalescence – typical of a high-energy absorption fracture mechanism. As the austenitizing temperature increases to 1050 ℃ (Fig. 12b), the coarsening of austenite grains and partial dissolution of precipitates such as NbC and TiN reduce the density of ferrite nucleation sites. This leads to a less refined dual-phase microstructure, where smaller dimples are formed due to decreased strain hardening and localized plastic deformation, indicating a reduction in ductility and absorbed energy.

When the austenitizing temperature reaches 1150 ℃ (Fig. 12c), enhanced atomic diffusion leads to improved homogenization and a more uniform distribution of martensite islands within the ferritic matrix. This reduces the severity of banding and creates a more isotropic microstructure. As a result, although the grain structure becomes coarser, the more even stress distribution during impact allows for the formation of larger dimples once again, reflecting improved toughness relative to the intermediate condition. This evolution in dimple morphology underscores the intricate interplay between grain size, phase distribution, and fracture behavior across various austenitizing temperatures.

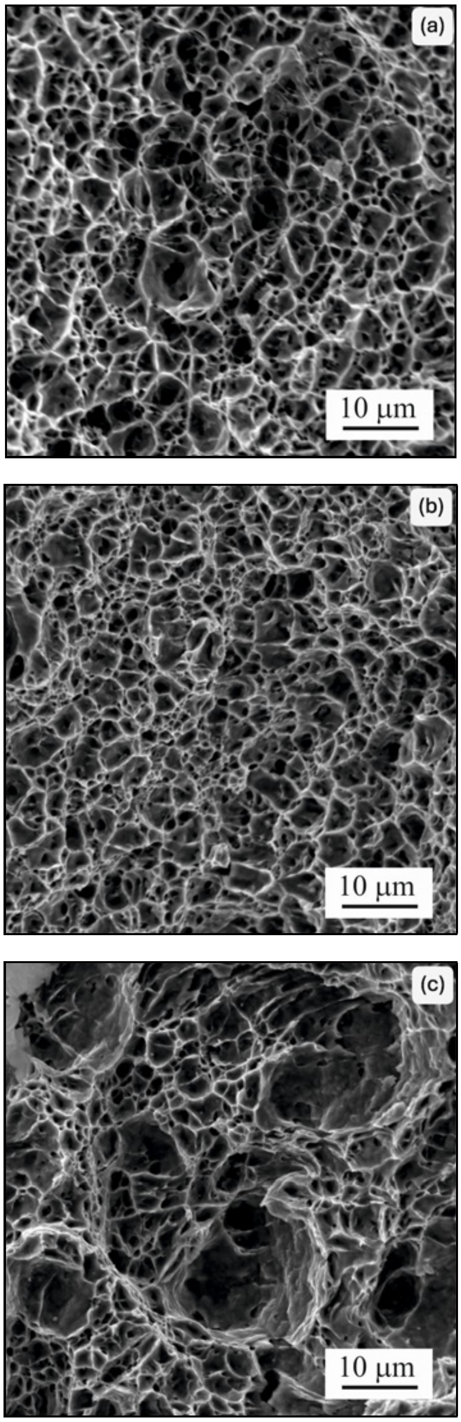

Fig. 12. SEM micrographs of Charpy fracture surfaces at different austenitizing temperatures for samples intercritically annealed at 740 ℃: (a) $T = 950$ ℃, (b) $T = 1050$ ℃, and (c) $T = 1150$ ℃.

4. Conclusions

This study investigated the influence of austenitizing temperature on the microstructure evolution and mechanical response of dual-phase X52 steel subjected to SQ treatment. The key findings can be summarized as follows:

1. Increasing the austenitizing temperature from

950 to 1150 ℃ led to significant changes in the microstructure. At 950 ℃, the microstructure exhibited a clearly banded structure of ferrite and martensite. However, at 1150 ℃, the banded morphology was eliminated, and a more uniform dispersion of martensite islands within the ferritic matrix was achieved due to enhanced atomic diffusion and reduced segregation.

2. The hardness increased with intercritical annealing temperature, correlating with the higher martensite volume fraction. However, at a constant intercritical temperature, increasing the austenitizing temperature resulted in a decrease in hardness due to grain coarsening, reduced nucleation of ferrite, and a diminished phase boundary density.

3. Charpy impact tests showed that absorbed energy generally decreased with increasing austenitizing temperature, particularly at intermediate temperatures (1050 ℃), where partial banding and coarse grains reduced ductility. However, a slight recovery in toughness was observed at 1150 ℃, attributed to the elimination of banded structures and improved microstructural isotropy.

4. SEM observations confirmed ductile fracture in all cases, but the size and shape of dimples varied. Smaller dimples at intermediate austenitizing temperatures indicated reduced ductility, whereas larger dimples at 1150 ℃ reflected the improved uniformity of the dual-phase microstructure.

Overall, austenitizing at 1150 ℃ followed by intercritical annealing at 740–800 ℃ appears to be an effective thermal route for minimizing banding and promoting an isotropic dual-phase microstructure in X52 steel, improving both toughness and structural reliability.

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