Effect of Tempering Temperature on Microstructure and Mechanical Properties of 5.5Ni Cryogenic Steel: Postprint

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Abstract

The 5.5Ni steel was characterized by XRD, SEM, TEM, and other techniques to investigate changes in the content, morphology, and size of reverted austenite at different tempering temperatures, and the effect of tempering temperature on the mechanical properties of 5.5Ni steel was studied. The results show that: after tempering at 580-600°C, the tensile strength and yield strength of 5.5Ni steel exhibited no significant change; after tempering at 620°C, the tensile strength increased slightly, while the yield strength decreased significantly, and the elongation continuously increased; when tempering at 580-620°C, although the volume fraction of reverted austenite in 5.5Ni steel gradually increased with increasing tempering temperature, the impact energy continuously decreased. The lamellar reverted austenite with high stability and a finely, uniformly dispersed distribution is the main reason for the impact energy reaching as high as 148 J after tempering at 580°C. There are two types of reverted austenite in the steel: one is lamellar, with a width of 20 nm and varying lengths, which is beneficial for improving the low-temperature toughness of the steel; the other is blocky, distributed in clusters with a size of approximately 200 nm, which is detrimental to the low-temperature toughness of the steel.

Full Text

Preamble

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Abstract

The volume fraction, morphology, and size of reversed austenite in 5.5Ni steel tempered at different temperatures were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM). The influence of tempering temperature on the mechanical properties of 5.5Ni steel was systematically investigated. The results show that when tempered between 580℃ and 600℃, both tensile strength and yield strength remained essentially unchanged. At 620℃, tensile strength increased slightly while yield strength decreased significantly, with elongation continuing to rise. Although the volume fraction of reversed austenite (γ) increased gradually with tempering temperature from 580℃ to 620℃, impact energy decreased continuously. The exceptionally high impact energy of 148 J at 580℃ is primarily attributed to the presence of highly stable, finely dispersed lamellar reversed austenite. Two distinct types of reversed austenite were identified: lamellar austenite with a width of approximately 20 nm and variable length, which enhances low-temperature toughness, and blocky austenite occurring in clusters with a size of about 200 nm, which is detrimental to low-temperature toughness.

KEY WORDS: metallic materials, 5.5Ni steel, tempering temperature, microstructure, mechanical properties

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1. Introduction

Appropriate heat treatment of cryogenic steels can produce a certain amount of reversed austenite (γ) [1-3]. This phase significantly improves low-temperature toughness through several mechanisms, including crack tip blunting, localized transformation-induced plasticity, and "matrix purification" [4]. The beneficial effects are particularly pronounced when the γ is finely dispersed and stable [2, 5-7]. Heat treatment parameters, especially tempering conditions, substantially influence the microstructure and mechanical properties of cryogenic steels, as γ primarily forms through the reverse transformation of martensite during tempering [6-8]. Therefore, investigating how tempering temperature affects the content, morphology, and stability of γ is essential for microstructural control and optimization of heat treatment processes to achieve superior comprehensive mechanical properties.

9Ni steel is an ideal material for manufacturing LNG storage tanks for ultra-low temperature pressure vessels, but its high nickel content increases costs considerably. Consequently, developing new Ni-saving cryogenic steels with excellent comprehensive performance and lower cost is crucial for enhancing industrial competitiveness. In this study, XRD, SEM, and TEM were employed to investigate the effects of tempering temperature on the microstructure and mechanical properties of a 5.5Ni steel.

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2. Experimental Procedures

The chemical composition of the experimental steel was (mass fraction, %): C 0.046, Si 0.049, Mn 1.0, Al 0.023, Ni 5.32, Mo 0.11, Cr 0.32, P 0.0064, S 0.0037, with Fe balance. The steel was melted in a 50 kg vacuum induction furnace and forged into billets measuring 120 mm × 110 mm × 60 mm. The thermo-mechanical controlled processing (TMCP) and cooling schedule are illustrated in [Figure 1: see original paper]. The billets were homogenized at 1150℃ for 1 hour, followed by two-stage rolling (roughing and finishing) with a finishing temperature of 830℃, and then water-quenched to room temperature. The as-quenched steel was subsequently annealed at 690℃ for 1 hour, water-quenched, and tempered at 580℃, 600℃, or 620℃ for 1 hour, followed by air cooling to room temperature.

The Ae_1 and Ae_3 temperatures were calculated to be 595℃ and 729℃, respectively. After grinding and polishing, specimens were electrolytically etched to remove surface stresses. The volume fraction of retained austenite at room temperature was measured using a PANALYTICAL-MPD X-ray diffractometer (XRD) according to national standard GB8362-87. The calculation formula is:

$$V_\gamma = \frac{I_\gamma}{I_\gamma + G \cdot I_\alpha}$$

where V_γ is the volume fraction of retained austenite, I_α and I_γ are the integrated intensities of diffraction peaks from ferrite/martensite and austenite, respectively, and G is the ratio of integrated intensity factors corresponding to fcc crystal planes (hkl)γ and bcc crystal planes (hkl)α.

Room-temperature tensile properties were measured using a WE-300 hydraulic tensile testing machine with standard specimens of 5 mm diameter. Charpy impact tests were conducted at -196℃ using a JBN-300B impact tester with V-notched specimens of 10 mm × 10 mm × 55 mm. Microstructural observation was performed using a S-4300 cold field-emission scanning electron microscope (SEM) and an H-800 transmission electron microscope (TEM) after mechanical grinding, polishing, and etching with 4% nital solution.

3. Results

3.1 Microstructure

[Figure 2: see original paper] presents SEM micrographs of the experimental steel after tempering at different temperatures. The matrix structure in all tempered specimens consisted of tempered martensite with indistinct lath structures. White contrast features distributed throughout the matrix represent reversed austenite (γ) and some "fresh" martensite that transformed from unstable γ during cooling. After quenching at 690℃ and tempering, the microstructure comprised tempered martensite, γ , and a small amount of fresh martensite. The γ appeared as fine, discontinuous particles predominantly along prior austenite grain boundaries and martensite packet boundaries, while fresh martensite was more randomly distributed and coarser. At 580℃, γ existed mainly as fine granular and lamellar morphologies. Compared with the 580℃ condition, the amount of γ increased significantly at 600℃ and 620℃, with a predominance of blocky morphology. Notably, after tempering at 620℃, the quantity of fresh martensite was markedly higher and slightly larger in size than at 580℃ [Figure 2c: see original paper].

[Figure 3: see original paper] shows the XRD spectra and volume fraction of γ at different tempering temperatures. The diffraction peaks (111)γ , (200)γ , (220)γ , and (311)γ correspond to austenite, while (110)α, (200)α, and (211)α belong to martensite. These results confirm the presence of γ in all tempered conditions, with increasing peak intensities as tempering temperature rises. The volume fraction of γ increased progressively from 580℃ to 620℃. After liquid nitrogen immersion, the γ volume fraction decreased in all specimens, but the change was minimal for the 580℃ tempered sample. The austenite volume fraction decreased by 4.6% after liquid nitrogen immersion for the 600℃ specimen, and by approximately 10% for the 620℃ specimen [Figure 3b: see original paper], indicating that higher tempering temperatures produce less stable γ .

TEM analysis was conducted to further characterize the distribution and morphology of γ . Generally, γ exhibits two distinct morphologies: irregular blocky particles primarily at high-angle grain boundaries, and lamellar plates typically distributed between martensite laths [9]. The latter morphology is particularly important for enhancing low-temperature toughness. [Figure 4: see original paper] presents TEM images of the 5.5Ni steel after tempering at various temperatures. After tempering at 580℃, γ appeared as fine lamellar plates distributed between tempered martensite laths, aligned parallel to the surrounding martensite laths, with a width of approximately 20 nm and variable length [Figure 4a: see original paper]. At 600℃, the lamellar γ width increased to about 50 nm while maintaining similar length, and a certain amount of blocky γ emerged [Figure 4b: see original paper]. Selected area electron diffraction confirmed that these lamellar γ plates obeyed the Nishiyama-Wassermann (N-W) orientation relationship with the martensite matrix: [011]γ-Fe // [001]α-Fe and (111)γ-Fe // (110)α-Fe. At 620℃, substantial blocky γ formed in clusters with sizes around 200 nm [Figure 4c: see original paper]. [Figure 4d: see original paper] shows fresh martensite in the 620℃ specimen, primarily located at intersections of prior austenite or tempered martensite high-angle grain boundaries, with a size of about 400 nm. This fresh martensite contained numerous fine parallel martensite laths that transformed from coarser γ during cooling.

3.2 Mechanical Properties

[Figure 5: see original paper] illustrates the mechanical properties of the experimental steel at different tempering temperatures. As tempering temperature increased from 580℃ to 600℃, tensile strength (Rm) and yield strength (Rp0.2) showed negligible change, while elongation (A5) increased slightly [Figure 5a: see original paper]. From 600℃ to 620℃, Rm increased modestly, but Rp0.2 decreased substantially, resulting in a significantly lower yield ratio and slightly improved elongation. Impact energy decreased linearly with increasing tempering temperature, dropping from 148 J at 580℃ to 107 J at 620℃ [Figure 5b: see original paper]. All impact energies exceeded 100 J, meeting or surpassing the technical requirements for 9Ni steel.

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4. Discussion

4.1 Effect of Tempering Temperature on Strength and Ductility

The 5.5Ni steel exhibited high tensile and yield strengths after various tempering treatments [Figure 5a: see original paper]. At tempering temperatures of 580℃ and 600℃, strength values remained stable with high yield ratios, indicating strong resistance to deformation. At 620℃, the slight increase in Rm and significant decrease in Rp0.2 occurred primarily because the higher γ content absorbed more impurity elements, enhancing matrix purification and promoting formation of a softer martensitic matrix. Additionally, higher tempering temperatures reduced dislocation density, contributing to lower yield strength. During tensile deformation, slip in austenite leads to macroscopic yielding, so higher austenite content corresponds to lower yield strength, as also reported by Zhao et al. [10].

Some researchers [11] have used Thermo-Calc software to predict room-temperature austenite volume fractions after different heat treatments and established corresponding models. [Figure 6: see original paper] shows calculated phase fractions for the 5.5Ni steel tempered at various temperatures, indicating that the maximum predicted room-temperature austenite volume fraction occurs between 600℃ and 620℃. At 620℃, γ stability decreased, causing partial transformation to fresh martensite during cooling, consistent with [Figure 4d: see original paper]. As shown in [Figure 3b: see original paper], higher tempering temperatures resulted in more γ transforming during liquid nitrogen immersion, with γ being least stable at 620℃. This suggests a greater likelihood of martensitic transformation under plastic deformation conditions after tempering at higher temperatures.

Experimental data demonstrate that the tensile strength of multiphase structures generally follows a rule of mixtures [12]: TS = fM1 · TSM1 + (1-fM1) · TSM2, where TSM1 and TSM2 are the tensile strengths of constituent phases M1 and M2, and fM1 and (1-fM1) are their respective volume fractions. When the soft phase volume fraction is not excessive, tensile strength depends primarily on the hard phase. In this steel, martensite is the hard phase. As shown in [Figure 6: see original paper], blocky γ formed at 620℃ partially transformed to fresh martensite during subsequent cooling, thereby increasing tensile strength. Additionally, more metastable γ transformed to martensite during tensile deformation, further enhancing strength.

With increasing tempering temperature, the γ volume fraction increased, and uniform elongation followed the same trend. When steel contains a certain volume fraction of metastable austenite, deformation-induced martensitic transformation occurs during straining, generating work hardening that forces plastic deformation to transfer to surrounding softer regions. This delays necking initiation and propagation, thereby improving ductility, particularly uniform ductility [13]. Consequently, the increased γ volume fraction at higher tempering temperatures led to correspondingly higher elongation, confirming that γ volume fraction is a primary parameter determining material plasticity.

4.2 Effect of Tempering Temperature on Impact Energy

Research [14] has shown that significant stress concentration exists at crack tips during impact fracture. When a crack tip encounters γ , the austenite undergoes martensitic transformation, absorbing substantial energy and effectively relaxing stress concentration, thereby delaying crack nucleation. Furthermore, when reversed austenite exhibits high stability, more γ is retained after loading during the crack nucleation stage, and the "TRIP" effect during fracture delays crack propagation, ultimately improving low-temperature toughness. Although γ content increased with tempering temperature [Figure 3b: see original paper] and the steel' s performance met 9Ni steel specifications, impact energy decreased with higher tempering temperatures [Figure 5b: see original paper].

This decrease in impact energy with increasing tempering temperature is related to γ stability. γ stabilizes itself by absorbing interstitial atoms and alloying elements from the matrix, with C, Mn, and Ni being the primary austenite-stabilizing elements. Lower tempering temperatures reduce the diffusion capacity of interstitial atoms like C and N and alloying elements such as Mn and Ni. Consequently, after tempering at 580℃, the relatively low γ volume fraction resulted in higher average concentrations of stabilizing elements in the austenite, yielding higher stability—one reason for the high impact energy at this temperature. As tempering temperature increased, the γ volume fraction increased, but the average concentration of stabilizing elements within the austenite decreased, reducing γ stability. As shown in [Figure 3b: see original paper], more γ transformed during liquid nitrogen immersion at higher tempering temperatures. The 580℃ specimen showed minimal change in γ volume fraction before and after liquid nitrogen immersion, indicating high stability. The 600℃ specimen exhibited moderate stability, while austenite formed at 620℃ was the least stable. As shown in [Figure 6: see original paper] and [Figure 4d: see original paper], γ formed at 620℃ was extremely unstable, with partial transformation to martensite occurring during cooling. During subsequent impact testing at -196℃, even more γ transformed, significantly reducing the effectiveness of unstable γ in improving impact toughness. Additionally, enhanced long-range diffusion of C, Mn, and Ni atoms at higher temperatures reduced microsegregation, increasing the likelihood of martensitic transformation under applied stress —another reason for the lower impact energies at 600℃ and 620℃. Therefore, when γ volume fraction is within a certain range, its stability becomes a critical factor affecting impact energy.

Low-temperature toughness depends not only on γ content and stability but also on its morphology [15] and size [16, 17]. Compared with blocky austenite, lamellar austenite contains higher carbon content, providing greater resistance to martensitic shear and requiring larger chemical driving force for transformation [15]. At 580℃, the predominantly lamellar γ with fine, uniform distribution between martensite laths [Figure 4a: see original paper] experienced enrichment of austenite-stabilizing elements due to constraint from the surrounding hard martensite phase. This made the austenite less susceptible to martensitic transformation under small plastic deformation, requiring more energy or larger deformation for transformation to occur. Consequently, this highly stable γ effectively hindered crack propagation, contributing to the high impact energy at 580℃. At 600℃ and 620℃, although γ content increased, blocky γ appeared. Blocky γ exhibits lower stability and less constraint, making it prone to martensitic transformation during plastic deformation. The coarse fresh martensite formed from blocky γ at 620℃ [Figure 4d: see original paper] did not effectively obstruct crack propagation, severely deteriorating low-temperature toughness. As tempering temperature increased, the proportion of blocky γ increased substantially, leading to continuously decreasing impact energy.

In summary, the high impact energy at 580℃ is primarily attributed to the appropriate volume fraction of highly stable, finely dispersed lamellar γ. Although the γ volume fraction varied only modestly (5.5%-10.8%) across 580-620℃, its morphology and stability were the dominant factors influencing impact energy. The 580℃ tempered specimen exhibited the smallest γ volume fraction but the highest stability, resulting in the highest impact energy.

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5. Conclusions

1. When tempering temperature increased from 580℃ to 600℃, the strength of 5.5Ni steel changed negligibly. However, after tempering at 620℃, tensile strength increased slightly while yield strength decreased substantially, and elongation continued to improve.
2. Although the reversed austenite (γ) volume fraction increased with tempering temperature from 580℃ to 620%, impact energy decreased continuously from 148 J to 107 J. All impact energies exceeded 100 J, meeting the performance specifications of 9Ni steel. The reduced impact energy at higher tempering temperatures is mainly attributed to decreased γ stability and increased volume fraction of unstable blocky austenite.
3. In 5.5Ni steel, lamellar γ with a width of approximately 20 nm and variable length was uniformly dispersed between martensite laths, which is beneficial for low-temperature toughness. In contrast, blocky γ occurring in clusters with sizes around 200 nm is detrimental to low-temperature toughness.
4. The low-temperature toughness of 5.5Ni steel is closely related to tempering temperature and depends not only on γ content but also critically on its morphology and stability. The exceptionally high impact energy of approximately 150 J after tempering at 580℃ is primarily due to the appropriate volume fraction of highly stable, finely dispersed lamellar γ .

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Note: Figure translations are in progress. See original paper for figures.