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Overcoming Strength-Ductility Trade-Off at Cryogenic Temperature of Low Carbon Low Alloy Steel via Controlling Retained Austenite Stability

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Abstract: Stress–strain behavior of a low carbon low alloy multiphase steel with ferrite, tempered bainite, and retained austenite was studied at different cryogenic temperatures. Results indicated that both strength and ductility were enhanced with decreasing tensile testing temperature. The enhancement of both strength and ductility was attributed to the decreased mechanical stability of retained austenite with decreasing temperature, resulting in sufficient transformation induced plasticity (TRIP) effect for increasing work hardening rate.

Keywords: multiphase steel; retained austenite stability; work hardening rate; ductility; low temperature property



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1. Introduction

High strength low alloy (HSLA) steels have attracted great interest for wide fields of applications, due to their excellent strength-toughness balance, economy, and weldability [1–3]. With applied fields of HSLA steels extending to low temperature environment, low temperature toughness is of significant importance in consideration of safety. Generally, yield strength of HSLA steels increases dramatically to be higher than fracture strength with decreasing temperature, thus sudden brittle fracture takes place without ductile plastic deformation [4]. This strength-ductility trade-off greatly limits their application in low temperature environments. Therefore, it is very important to simultaneously enhance yield strength and impact toughness with decreasing temperature for HSLA steels by tailoring microstructure. Recently, it has been suggested that the strength-ductility trade-off can be overcome though the controlled deformation-induced martensitic transformation from face-centered cubic to body-centered cubic via metastability engineering in CoCrFeNiMo medium-entropy alloys [5]. The purpose of this work is to introduce multiphase microstructure containing retained austenite to a low carbon low alloy steel for enhancing the work hardening ability at low temperatures, resulting in high fracture strength and high ductility.

A lot of efforts have been devoted to improving strength, ductility, and toughness of HSLA steels by tailoring microstructure, and progress has been made. It has revealed that refinement of prior austenite grains via increasing high angle grain boundaries (HAGBs) is helpful for decreasing ductile-to-brittle transition temperature (DBTT), because HAGBs can retard brittle crack propagation [6,7]. Control of multiphase microstructure via producing metastable retained austenite in low carbon low alloy steels was able to significantly improve ductility and low temperature toughness [2,8–11]. Metastable retained austenite was suggested to afford TRIP effect during tensile or impact to enhance the plastic deformation ability before fracture. The stability of retained austenite is crucial to performance in service [12,13]. The main concern is that once retained austenite in the steel is unstable and transforms into martensite thermally or mechanically due to a small deformation in

low temperature service, sudden brittle fracture takes place, resulting in disaster. Hence, the objectives of the present work are to study the stability of retained austenite in a low carbon low alloy multiphase steel produced by the two-step intercritical heat treatment, and to elucidate the effect of retained austenite stability on stress–strain behavior at cryogenic temperature.

2. Material and Methods of Experiments

The chemical composition of the experimental steel was Fe-0.07C-0.36Si-1.78Mn-0.51Ni-0.3Mo-0.78Nb-0.02Ti in weight percent (wt%). The steel was melted in vacuum induction furnace and cast into ingot of weight 25 kg. The ingot was reheated to 1200 °C and held for 2 h, then forged into rods with diameter of 16 mm, and air cooled to ambient temperature. The forged rods were intercritically annealed at 740 °C for 30 min, subsequently air cooled to ambient temperature. Next, the annealed specimen was tempered at 680 °C for 30 min, and finally air cooled to ambient temperature. Mechanical properties in terms of strength and elongation of the treated samples (φ 5 mm standard tensile test samples) were measured at an extension rate of 2.5 × 10⁻³ s⁻¹ with an extensiometer of 25 mm at cryogenic temperatures of -80, -120, -160, and -196 °C, respectively. The reason why the tensile test starts at -80 °C is that the material has excellent room temperature properties and good low temperature toughness [8]. The model of cryogenic tensile testing machine is SANS CMT5000. The specimen was cooled to the set temperature with liquid helium (he), then kept for 10 min, and then tensile test was carried out, as shown in Figure 1.



Figure 1. Schematic diagram of cryogenic tensile test process.

Heat treated samples were mechanically polished and etched with 4% nital using standard metallographic procedures for TESCAN MIRA3 field emission scanning electron microscopy (SEM, TESCAN, Brno, Czech Republic). To obtain information on distribution and morphology of retained austenite, electron backscatter diffraction (EBSD, Oxford Instruments, Oxford, UK) analysis was carried out after mechanical and electrolytic polishing using TESCAN MIRA3 field emission scanning electron microscope (FE-SEM, TESCAN, Brno, Czech Republic) at an acceleration voltage of 20 kV, with a step size of 60 nm. EBSD data was post-processed by HKL CHANNEL 5 flamenco software (Oxford Instruments, Oxford, UK) to acquire necessary information. The amount of retained austenite was also evaluated using magnetic measurements [14]. For these measurements, samples were cut with approximately 150 mg. The M(*H*) curves were obtained at 300 K using a PPMS (Physical Property Measurement System, Quantum Design, San Diego, USA). The hysteresis loops were carried out using the following sweep rates: 0.4 kOe/min for |H| < 0.5 kOe, 1.8 kOe/min for 0.5 < |H| < 5 kOe and 10 kOe/min for 6 < |H| < 15 kOe.

3. Results and Discussion

Representative SEM micrograph of the heat-treated sample is given in Figure 2a. Multiphase microstructure consisting of ferrite, tempered bainite (TB), and retained austenite (RA) was obtained. Polygonal ferrite (PF) with diameter of $\approx 10 \,\mu\text{m}$ was observed along prior austenite grain boundary. Lath tempered bainite (TB, tempered at 680 °C) and intercritical ferrite composited as lamellar structure. The distribution and morphology of retained austenite are presented by EBSD image in Figure 2b. It was seen that retained austenite distributed mainly in the lamellar structure with fine blocky and thin film-like morphologies. The stabilization of retained austenite as a result of two-step heat treatment was reported in our previous work [8,15].



Figure 2. (a) SEM micrograph showing multiphase microstructure and (b) EBSD image showing morphology and distribution of retained austenite (RA) in the multiphase microstructure of the studied steel, in which red areas represent retained austenite. (PF: polygonal ferrite, TB: tempered bainite).

Figure 3a shows the engineering stress–stain curves of the studied steel measured at temperatures of -80, -120, -160, and -196 °C. It was found that both yield strength and tensile strength were increased with decreasing tensile temperature. The yield strength and tensile strength were 507 and 805 MPa at -80 °C, respectively. When the test temperature was decreasing to -196 °C, the yield strength and tensile strength were increased to 846 and 1083 MPa, respectively. More importantly, the uniform and total elongations also presented a trend to increase with decreasing the tensile temperature, except that there was a slight decrease of total elongation at -160 °C. The studied steel exhibited high uniform elongation of 16.1-19.1% and high total elongation of 30.0-33.3% at tensile temperature range of -80 to -196 °C. This atypical phenomenon of stress–strain behavior in this work may be ascribed to the presence of the TRIP effect of retained austenite, rather than the classical deformation mechanism by dislocation glide and dislocation interactions [16,17].

To understand the ductility of the studied steel at low temperatures, work hardening behavior at examined temperatures was studied. The work hardening rates are plotted in Figure 3b, according to the calculation results from the Hollomon equation [18,19]. Three-stage work hardening behavior was characterized for samples tensile tested at all temperatures, namely the work hardening rate firstly decreased, then increased, and finally decreased to failure with increasing strain, as observed in TRIP-aided steels at room temperature [20,21]. It can be found that the decrease of work hardening rate in the first stage was stopped at the end of the yield plateau for all four testing temperatures. This suggested that the first stage of work hardening behavior was dominated by dislocation glide in ferrite due to breaking away from Cottrell atmosphere. The increase in the second stage was ascribed to the TRIP effect of retained austenite [21]. In the third stage, the downtrend of work hardening rate became slow, leading to delaying the occurrence of

necking and enhancing the ductility. This indicated that retained austenite manifested variations in mechanical stability under different loading temperatures. At relative high temperature of -80 °C, some of the retained austenite had high mechanical stability, they were stable to supply the TRIP effect till failure. With decreasing tensile testing temperature, the mechanical stability of retained austenite decreased, such that more retained austenite afforded the TRIP effect during strain to provide sustainable work hardening rate. This was the reason for the atypical phenomenon of increase in ductility with decreasing temperature.



Figure 3. (a) Engineering stress–strain curves and (b) true stress–strain curves and work hardening rate of the studied steel at low temperatures.

To study thermal and mechanical stability of retained austenite, the volume fraction of retained austenite was determined by saturation magnetization (M_s) for the undeformed and uniform deformed area of samples after testing at different temperatures. The obtained magnetization (M) curves as a function of applied magnetic field (H) are shown in Figure 4. It can be seen that the M_s was identical for samples without strain at different temperatures. This indicated that retained austenite in the studied steel had high thermal stability in temperature range of -80 to -196 °C. After the maximum uniform deformation, the M_s increased obviously. There was a tendency that the increment of M_s increased with decreasing testing temperature. This effect was attributed to the decreasing of non-ferromagnetic phase of retained austenite. Hence, it can be concluded that the mechanical stability of retained austenite in the studied steel decreased with decreasing temperature. Therefore, more retained austenite transformed to martensite during strain, resulting in sustainable work hardening rate due to sufficient TRIP effect. Given that all retained austenite transformed to martensite at -196 °C, the M_s at -196 °C was taken as $M_s(f)$ for the austenite-free sample. Therefore, based on the calculation method in [14], the value of the last point of the magnetization curves $(M_s(c))$ obtained at different conditions is used to calculate the volume fraction of retained austenite (f_{γ}). The calculation method is presented in Equation (1).

$$f_{\gamma} = \frac{M_s(f) - M_s(c)}{M_s(f)} \times 100\%$$
 (1)

Thus, the volume fraction of retained austenite can be calculated as \approx 21.1% for samples undeformed at -80 to -196 °C. After maximum uniform deformation at -80 and -120 °C, the volume fraction of retained austenite was \approx 4.5–5.2%. When the testing temperature decreased to -160 °C, the volume fraction of retained austenite decreased to \approx 1.8%. This confirmed that the enhancement of both strength and ductility with decreasing temperature was ascribed to the decreased mechanical stability of retained austenite, resulting in sufficient transformation induced plasticity (TRIP) effect for sustainable work

hardening rate. The main reason why retained austenite can be stable to low temperature (-196 °C) is ascribed to the highly enrichment of Mn and C during the two-step intercritical heat treatment. Atom probe tomography analysis in our previous studies revealed that the Mn and C contents were ≈ 6.6 and 0.4 wt%. In addition, the obtained retained austenite was very fine, the average size was ≈ 300 nm in diameter. Hence, the retained austenite was very stable thermally up to -196 °C [8,15].



Figure 4. Magnetization (*M*) curves as a function of applied magnetic field (*H*) for samples without deformation and after uniform deformation in the range of -80 to -196 °C.

To reveal the failure mechanisms at different testing temperatures, the fracture surfaces were examined using SEM. The macroscopic and microscopic fractographs are shown in Figures 5 and 6. It can be seen from the Figure 5 that with the decrease of temperature, the anisotropic fracture morphology gradually changes to the isotropic fracture morphology. When the temperature decreases to -196 °C, the fracture surface presents complete brittleness. This phenomenon can be attributed to the change of microstructure with the decrease of temperature and the conditions of testing. From Figure 6, it can be seen that several main cracks with edge step were always observed along the diameter direction. Full ductile fracture with dimples was characterized for samples tensile tested at -80 and -120 °C. Based on our previous study, this effect could be caused by comprehensive influence of texture evolution, the redistribution of grain boundaries, and the state of three-dimensional stress with large tensile plastic deformation during necking [22]. When testing temperature decreased to -160 and -196 °C, the main crack was not obvious, and some small cracks and brittle fracture were observed within the matrix. This indicated that after large uniform deformation, the plasticity during necking decreased at -196 °C. This may ascribe to the full consumption of retained austenite during uniform deformation. Therefore, without the help of TRIP effect from retained austenite to release the stress, the brittle fracture occurred during necking. This result suggested that some stable retained austenite that remained after uniform deformation could suppress brittle collapse and ensure safety in service.





Figure 5. SEM images showing the macroscopic fractographs of samples tested at different temperatures: (a) -80 °C, (b) -120 °C, (c) -160 °C, and (d) -196 °C.



Figure 6. SEM images showing the microscopic fractographs of samples tested at different temperatures: (a) -80 °C, (b) -120 °C, (c) -160 °C, and (d) -196 °C, corresponding to the marked area in Figure 5.

4. Conclusions

The stability of retained austenite was studied in a low carbon low alloy steel processed by the two-step intercritical heat treatment. The retained austenite exhibited high thermal stability in range of -80 to -196 °C. Whereas, mechanical stability decreased with decreasing temperature from -80 to -196 °C. The decrease in mechanical stability of retained austenite was helpful for enhancing the uniform elongation via sufficient TRIP effect during uniform deformation, resulting in simultaneous enhancement of strength and ductility at low temperatures. However, the full consumption of retained austenite during uniform strain may raise risk of sudden brittle collapse during necking.

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