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# Enhancement of low temperature toughness of nanoprecipitates strengthened ferritic steel by delamination structure



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# ABSTRACT

This study investigated the effects of aging and thermomechanical treatments on the microstructure evolution and mechanical properties of a nanoprecipitates strengthened ferritic steel. The toughness of steel at various temperatures was measured carefully and correlated with microstructural features. Tensile tests show that aging can improve the mechanical strength without scarifying the ductility. With high yield strength of ~1000 MPa, excellent low temperature Charpy impact energy more than 300 J at -80 °C can be obtained. The ductile brittle transition temperature (DBTT) is lower than -80 °C. The high strength can be contributed by the nanocluster precipitation as determined by small angle neutron scattering and transmission electron microscopy. The excellent low temperature toughness is attributed to the delamination structure of the steel, which blunts the cracks and restrains the crack propagation.

### 1. Introduction

As for the most high-strength low alloy steels, carbon is commonly applied for increasing strength [1]. However, the high carbon content would lead the bad plasticity and weldability. With the addition of alloying elements (Cu, Ni, Al and Mn), the nanoscale-precipitates consisting of a Cu-enriched bcc core partially encased by a B2-ordered Ni (Mn, Al) can be introduced into matrix, which significantly strengthen the steel without sacrificing the ductility and toughness [2–4]. Due to the high strength, excellent weldability, good plastically and prominent ductility of the nanoprecipitates strengthened ferritic steels (NSFS), these kind of steels can be widely used in automobile, construction, bridging industry, etc. [5–7].

The low temperature toughness (LTT) is one of the most important mechanical properties of the steels. When the operating temperature is lower than the ductile-to-brittle transition temperature (DBTT), the steel would suffer catastrophic fracture and cause serious accidents, such as the steel structure buildings in cold region and polar ocean voyaging vessels [8]. Therefore, enhancing the LTT and reducing the DBTT of high strength steels deserves attention. It is known that there are multiple factors effecting LTT, including alloying elements, thermomechanical parameters, grain size, dislocation density, microstructure, quantity and distribution of impurities or brittle phases [9–15]. Recent results indicate that the delamination structure in some structural metals can effectively increase the LTT by blunting the crack tip and hindering the crack propagation, thus leading to high impact energy at low temperatures [16].

In this study, through the utilization of precise thermo-mechanical control process (TMCP) technology, the microstructure was successfully controlled. Remarkably high impact energy at low temperatures and a low DBTT were obtained. The size and distribution of nanophase were also evaluated.

### 2. Experiment

The experimental steel used in this study was produced by an induction melting furnace. About 40-kg alloy was melted in a protective argon atmosphere and cast to a rectangular ingot. The chemical compositions of the steel are listed in Table 1. The thermo-mechanical control process (TMCP) was applied to control the deformation amount precisely [17]. The ingot with a thickness of ~170 mm was homogenized at 1200 °C for 2 h and hot-rolled to 80 mm at 1000 °C with 4 passes. And then the 80 mm thick plate was rolled at 900 °C down to 12 mm with several passes, followed by water spray cooling to 100 °C.

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#### Table 1

Chemical composition of the steel (wt.%).

	-				
C	Si	Mn	P	S	Cu
< 0.05	0.2	1	< 0.004	< 0.002	2
Ni	Al	В	Nb	Ti	Fe
4	0.5	0.005	0.03	0.05	bal

Aging processing was applied to control the precipitation of nanoscale precipitates to strengthen the steel. Based on our previous studies [18], aging at 550 °C for 1 h can provide a comprehensive properties of the steel. At the same time, this aging temperature will not affect the matrix microstructure of the steel. Thus, all the specimens were aged at 550 °C in air for 1 h followed by water quenching to investigate the effect of matrix microstructure on the mechanical properties, specifically the LTT and DBTT.

The tensile tests were evaluated using INSTRON 5565 on rod tensile specimens with tensile direction along the rolling direction. The tensile specimens are with a 5 mm in diameter and 25.4 in gauge length. The tensile tests were conducted at room temperature in air with a strain rate of  $\sim 1.3 \times 10^{-3} \text{s}^{-1}$ . Hardness measurements were conducted under a 1000 g applied load and a dwell time of 15 s. The average hardnesses from six different measurements were reported. To evaluate the effect of temperature on the hardness, hardness at various temperatures were determined.

The Charpy impact tests were conducted on the Zwick/Roell 450 J impact machine (RKP450) coupled with the testXpertII software. The Charpy V-notch specimens with size of  $10 \text{ mm} \times 10 \text{ mm} \times 55 \text{ mm}$  were prepared by milling machine and grinding machine, and the notch place was on the rolling plane with the angle and deepness of 45° and 2 mm, respectively. The impact test was conducted over a temperature range of -80-20 °C. In order to reduce deviations in data interpretation, a regression analysis on absorbed energy curve was done by a hyperbolic tangent curve fitting method.

The phase components of the specimens were determined by X-ray diffraction (XRD) with the following experimental parameters: a Cu Target K alpha ray, 40 kV tube pressure,  $20^{\circ}-90^{\circ}$  scanning angle, and  $5^{\circ}$ /min scanning speed. The optical microscopy (OM) and scanning electron microscope (SEM) were also used to characterize the microstructure of the specimens. After mechanical polishing, the samples for OM analysis were etched with 4 vol% natal solution. And the fracture surfaces after the impact and tensile failure were also examined by SEM. Small angle neutron scattering (SANS) experiments were conducted at the China Academy of Engineering Physics (Mianyang, China). High-quality SANS data covering a wide *Q* range from 0.004 to 0.8 Å<sup>-1</sup> was measured with detector-to-sample distances of 2.041



Fig. 2. X-Ray diffraction patterns of the as-rolled and aged samples.

and 9.186 m, using 4.4 Å neutrons with 15% wavelength spread. The SANS samples were 10 mm square discs and 1 mm in thickness. The SANS data was amended for transmission, background, empty scattering, and detector sensitivity [19]. The transmission electron microscope (TEM) was used to observe the distribution of precipitated phase. The preparative procedure of samples was as follows: 1 mm thick foils were cut by EDM, and all of the foils were obtained from inside of the speciments to avoid surface effects. Then the foils were grinded into 50–80  $\mu$ m thickness and discs with 3-mm diameter were punched from the foils and thinned by GATA-691 ion milling. Then the discs were inspected with a JEM-2100F TEM operated on 200 kV for nanoprecipitation.

# 3. Results

## 3.1. Microstructure and phase analysis

Fig. 1 shows the optical microstructures of the as-rolled and aged samples. It is very clear that the elongated grain structures were introduced along the rolling direction. Upon aging, recovery of deformation microstructure occurred.

Fig. 2 shows the X-Ray diffraction results of the as-rolled and AG550–1 h samples. The pattern shows that there are mainly three peaks, which were (110), (200) and (211) crystal faces representing ferrite. Similar with the above metallography results, no extra peaks were observed, indicating that no second phase was introduced after



Fig. 1. Optical microstructures: (a) As-rolled (b) AG550-1 h.

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