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Compression Stability of Reversed Austenite in 9Ni Steel

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The effect of compressive stress on the stability of reversed austenite in 9Ni steel was investigated by uniaxial and hydrostatic compression. It was found that the uniaxial compressive pressure promoted the $\gamma \rightarrow \alpha$ transformation, while the hydrostatic pressure suppressed the $\gamma \rightarrow \alpha$ transformation. The pressure dependent transformation behavior can be explained according to thermodynamic analysis.

KEY WORDS: 9Ni steel; Martensite; Reversed austenite; Hydrostatic pressure; Uniaxial compressive pressure

1. Introduction

The ferrite 9Ni steels have been widely used as a structural material at cryogenic temperatures, for which a typical quenching, larmellarizing and tempering (QLT) heat treatment has been introduced. The intermediate (L) treatment is an intercritical temper in the range of the two-phase ($\alpha+\gamma$) field. During the QLT heat treatment, a fine admixture of austenite is precipitated along the martensite lath boundaries and prior austenite grain boundaries[1–3]. It is widely accepted that the excellent cryogenic mechanical properties are due to the precipitation of the reversed austenite, which is thermally stable but mechanically unstable. Correlations between the mechanical stability of the reversed austenite and the cryogenic mechanical properties are well established by several models[4 –7].

However, the thermal stability of the reversed austenite after heat treatment is not yet well understood. Generally, the austenite formed at high temperature transforms into martensite below the M_s temperature. For the 9Ni steel, an enrichment of solute elements such as Ni, Cr, Mn, and Si in the reversed austenite caused the suppression of M_s from 561 °C to 70 °C after 1-hour tempering[8]. However, the reversed austenite formed after tempering does not transform into martensite even cooled down to liquid nitrogen temperature, so the changes in reversed austenite composition are less able to account for the austenite stability. Fultz *et al.*[9,10] correlated the stability of the austenite particles with dislocation structures and considered that the reversed austenite is in a state of compression because of the plastic accommodation of the transformation strain. Therefore, it is argued that the compression could contribute substantially to the stability of the reversed austenite. We seek to determine the extent to which the change in austenite stability is attributable to the compression. Thus, we undertook a study of compressive test on the reversed austenite of 9Ni steel.

2. Experimental

The 9Ni steel used in this study was obtained from a 12 mm thick sheet with the chemical composition listed in Table 1. The steel sheet was processed by QLT heat treatment (Q: 1073 K for 60 min, water cooling, L: 943 K for 60 min, water cooling, and T: 858 K for 60 min, water cooling). Specimens with a dimension of 5 mm×5 mm×5 mm were cut from the sheet for compression test and X-ray diffraction measurement. The specimens were chemically etched in a solution of 28%H_2O+3%HF +69%H_2O_2 for 1–2 min to remove the mechanical effect of the cut surface.

The measurements of the volume fraction of the reversed austenite and the phase analysis were performed by X-ray diffraction (XRD) with a Rigaku

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Table 1 Chemical composition of 9Ni steel (mass%)

C	Ni	Mn	Si	Cr	S	P	Fe
0.054	9.45	0.32	0.15	0.089	0.005	0.006	Bal.

D/max-2400PC X-ray diffractometer. 2θ diffraction scan was carried out in the range of 40–100 deg. at room temperature using Cu$K\alpha$ radiation. The (200) and (220) austenite peaks were compared with the (211) and (200) martensite peaks to determine the volume fractions of the reversed austenite[11].

A uniaxial compression test and a hydrostatic pressure test were conducted on a computer servo-controlled Schimadzu AG-I universal testing machine at room temperature. Two smooth boards were placed on the above and below surface of the specimens to avoid damaging the surface. The hydrostatic pressure test was carried out on a piston-cylinder pressure vessel with an internal diameter of 10 mm. The specimen was surrounded by NaCl powder as a pressure medium, so that a nearly hydrostatic condition prevailed. The specimen was compressed at a pressure which was 50 MPa larger than the desired pressure for 15 min to minimize the pressure reduction induced by structure relaxation during the experiment.

3. Results and Discussion

Figs. 1 and 2 show the XRD patterns of the above surface of samples normal to the compression direction after compression tests. It indicates that the individual diffracting planes from the bcc-martensite (α) and fcc-autenite (γ) can be clearly identified and labeled in the figure. The diffraction patterns of the samples under different pressures between 0 and 600 MPa after the uniaxial compression tests are shown in Fig. 1. Hydrostatic pressure tests are conducted in the pressure range of 0–1 GPa. Texture is generated at the pressure of 1 GPa according to the diffraction patterns[12], because hydrostatic pressure is not achieved and deformation happens at high pressure. The diffraction patterns of the samples under different pressures between 0 and 400 MPa after the hydrostatic pressure test are shown in Fig. 2. The applied stress does not change the type of phases present. However, they affected the relative amount of martensite and reversed austenite.

Quantitative analyses were performed to obtain the volume fractions of the reversed austenite and the result is shown in Fig. 3. The amount of the reversed austenite decreases linearly with the increase of applied pressure under the uniaxial compressive test. However, the amount of the reversed austenite is almost invariant under the hydrostatic pressure test.

The stability of the reversed austenite depends on the stress state according to the above results. In order to explain the stability of the reversed austenite under external applied stress, the role of applied stress was analyzed after each test. The work (U)

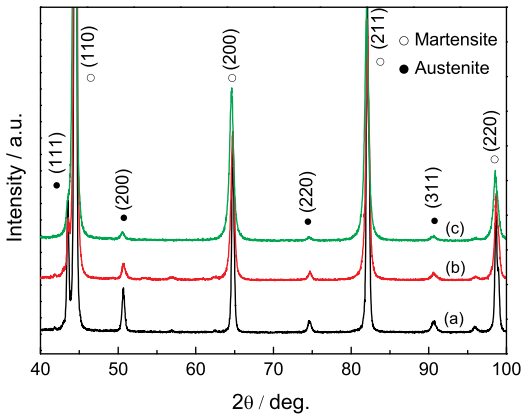

Fig. 1 XRD patterns of the samples after the uniaxial compression test: (a) p=0 MPa, (b) p=300 MPa, (c) p=600 MPa

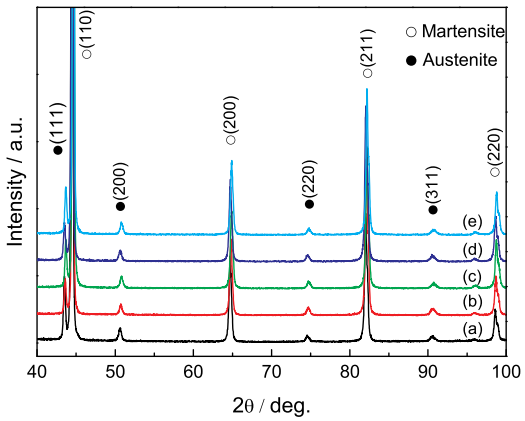

Fig. 2 XRD patterns of the samples after the hydrostatic pressure test: (a) p=0 MPa, (b) p=100 MPa, (c) p=200 MPa, (d) p=300 MPa, (e) p=400 MPa

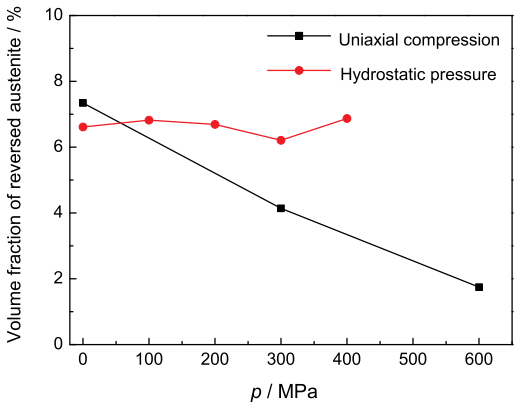

Fig. 3 Variation of austenite volume fraction after uniaxial compression test and hydrostatic pressure test