

In situ neutron diffraction study of the microstructure and tensile deformation behavior in Al-added high manganese austenitic steels

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Abstract

In situ neutron diffraction experiments were performed to measure the tensile deformation behavior of high manganese austenitic steels with different Al contents (0, 1.5, 2.0, 3.0 wt.%). Significant variations of peak shift, broadening and asymmetry of the diffraction peaks were observed in the plastic region with the measurement. Diffraction peak profile analysis was applied to determine microstructural parameters such as stacking/twinning fault probabilities, dislocation density and stacking fault energy (SFE). These parameters are quantitatively correlated to the yield strength, serrated flow and strain hardening rate during tensile deformation. The main results showed that the twin/stacking fault probability considerably decreases from 0.05 to 0.01 and dislocation density from 10^{16} to $4 \times 10^{15} \text{ m}^{-2}$ as a function of Al addition, while SFE (γ) increases from 20 to 45 mJ m^{-2} with the relationship of $\gamma = 8.84 \text{ wt.\% Al} + 19.0 \text{ mJ m}^{-2}$. Such microstructural parameters are also in good agreement with the results of the misorientation and pattern quality map obtained by the electron backscatter method.

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1. Introduction

High manganese austenitic steels have been extensively studied for their superior work hardening capacity and tensile strength combined with exceptional ductility based on the twinning induced plasticity (TWIP) phenomenon [1]. They are potentially attractive for automobile applications involving press formed parts for energy absorption and structural reinforcements. In the microstructure perspective, it has been argued that the excellent mechanical properties of TWIP steels mainly originate from dynamic Hall–Petch effect, which means the formation of mechanical twins gradually decreases the effective mean free path of

dislocations and results in a high strain hardening behavior [2–4]. Due to the poor formability and delayed fracture in TWIP steels, a few studies have recently highlighted the influence of Al addition to enhance the formability and suppress the delayed fracture in cup deforming specimens [5,6]. It is known that the Al alloying increases stacking fault energy (SFE) and degrades the progressive formation capability of twin faults in TWIP steels [7,8]. However, the detailed mechanism of Al influence on microstructure evolution and deformation properties has not been fully understood yet.

Electron interaction basis methods, for example, electron backscatter diffraction (EBSD) and transmission electron microscopy (TEM), can be utilized to obtain direct images of microstructure, including the deformation structures. However, there are difficulties in the quantitative analysis of the microstructure parameters under tensile deformation [9]. Neutron diffraction is another promising

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method to resolve the problem. The deep penetration capability of neutrons into most metallic materials and the unique volume-averaged mm-scale bulk measurements characteristic of the scattering beam make neutron diffraction a powerful tool for measuring deformation behavior [10,11]. Furthermore, in situ neutron diffraction can provide a clear feature of the microstructure evolution under deformation with the real-time quantitative analysis capability based on Bragg's diffraction and peak profile analysis [11]. In this regard, we employed the in situ neutron diffraction method under loading for the quantitative analysis of the microstructural parameters such as mechanical twinning, stacking faults, and dislocation density in high Mn austenitic steels.

The purpose of this study is the quantitative determination of the microstructural parameters under tensile deformation in order to explain the different microstructure-induced mechanical properties in Al-added high Mn austenitic steels. We measured five (*hkl*) Bragg peaks as a function of strains (engineering strain, $\varepsilon = 0\text{--}0.5$) and analyzed the characteristics of the observed diffraction peak profiles such as the peak position, breadth, and asymmetry. This enabled us to obtain useful microstructural parameters including probabilities of twin/stacking fault formations, SFE, and dislocation density. It can also provide insight for understanding the extraordinary deformation behavior in high manganese austenitic steels.

2. Experimental procedures

Four kinds of high Mn austenitic steel plates were prepared with different Al alloying compositions, Fe–18 Mn–*x* Al–0.6 C (in wt.%) with $x = 0, 1.5, 2.0$ and 3.0 . Note that the concentrations of other elements were kept less than 100 ppm. Each composed specimen was made by casting method with an induction melting furnace under Ar atmosphere. The specimens were soaked at 1150 °C for 2 h and hot-rolled into 2.5 mm plates with a final rolling temperature of 850 °C. Each plate was electrical-discharge-machined into the plate-type tensile specimens. The dimension of the tensile specimen had a total length of 100 mm. The parallel section part of the gauge was 25 mm in length, 2 mm in thickness, and 3 mm in width.

The in situ neutron diffraction experiment has been performed using the Residual Stress Instrument (RSI) at the Korea Atomic Energy Research Institute (KAERI) [12]. The gauge length direction of the specimen (longitudinal direction) was located between the incident beam and detector and the longitudinal component was measured during tensile loading. Note that the loading direction is parallel to the rolling direction (RD) of the base metal plate. Measurements have been done with the diffraction angle of $2\theta = 41^\circ, 47^\circ, 68.8^\circ, 83^\circ$ and 87.6° . These diffraction angles correspond to the reflections of the (111), (200), (220), (311) and (222) sets in the austenite specimen, respectively. The scattering gauge volume of the neutron beam was defined by 5 mm width, 4 mm height

input slit and 2 mm width detector slit. Thus, the spatial resolution of the beam was ~ 2 mm of the sample along the loading direction. Neutron diffraction peaks were measured for a total of seven different loading stages under the static loading condition including without loading. The measurement time was 5 min for each (*hkl*) reflection at each loading stage and less than 50 MPa of the stress relief was observed under the static loading condition. Note that the constant strain rate was $4 \times 10^{-4} \text{ s}^{-1}$ at room temperature.

The electron backscatter diffraction method (EBSD) has been also applied to obtain microstructural features and preferred orientations of twinned grains normal to tensile directions in each specimen. The observed surface was cross-section of the tensile specimen after polishing with colloidal silica. Note that high angle grain boundaries are defined with misorientation angle of $\theta > 15^\circ$ and the twin boundaries defined as $\Sigma 3 = 60^\circ \langle 111 \rangle$ for which a 60° rotation about $\langle 111 \rangle$ pole brings a lattice back into partial self-coincidence with 1/3 coincidence sites in the (111) net. The maximum tolerance of the misorientation angle is identified by $\Delta\theta \leq 6^\circ$ from the exact axis-angle relationship by the Palumbo-Aust criterion (i.e. $\Delta\theta \leq 15^\circ \Sigma^{-5/6}$) [13]. Consequently, they construct a misorientation map which can present the coherent twin boundaries and high angle grain boundaries in microstructure. In addition, the EBSD results were reconstructed into a pattern quality to represent the accumulated defects in the microstructure [14].

3. Tensile and microstructure analysis

Fig. 1 shows the true stress–strain curve and strain hardening rate under tensile loading in high Mn austenitic steels as a function of Al contents (0, 1.5, 2.0 and 3 wt.% Al). The values of tensile and yield strengths and total elongation are summarized in Table 1. Compared with the 0 wt.% Al specimen, 1.5 wt.% Al added specimens show the higher yield strength and lower tensile strength, total elongation, and strain hardening rate. Interestingly, the yield strength and tensile strength decrease further as the Al content increases from 1.5 to 3.0 wt.%. This can be attributed to the changes in microstructure, e.g. less resistance to the dislocation motion under plastic deformation due to the increased SFE by Al addition [1]. It should be mentioned that the serration phenomenon (known as dynamic strain aging) is the more obvious in the plastic deformed region of the 0 wt.% Al specimen compared to the Al-added specimens. It has been reported that the serration behaviors of TWIP steels occur due to the strong interaction of solute atoms with stacking faults [15].

Fig. 2 shows the misorientation map in the four specimens at the engineering strain, $\varepsilon = 0.5$, by using EBSD. It is clear that the number of twin boundaries decreases as Al concentration increases.

In Fig. 3 we show the reconstructed pattern quality (H_i) of the crystallographic orientation map and the average values of the pattern quality, $\langle H_i \rangle$. H_i can be determined

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