In-situ Neutron Diffraction Study on Work-hardening Behavior in a Ferrite-Martensite Dual Phase Steel

Satoshi Morooka 1) Naoko Sato 2) Mayumi Ojima 3) Stefanus Harjo 4)
Yoshitaka Adachi 5) Yo Tomota 6) Osamu Umezawa 7)

1.7) Yokohama National University, Department of Materials Science and Engineering
79-5 Tokiwadai, Hodagaya-ku, Yokohama 240-8501, Japan
2) Kyusyu University
744 Motooka, Nishi-ku, Fukuoka, 819-0395, Japan
3) University of Tokyo, Department of Materials Engineering
7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656, Japan
4) Japan Atomic Energy Agency, MLF/J-PARC Center
2-4 Shirakata Shirane, Tokai, Naka-gun, Ibaraki 319-1195, Japan
5) National Institute for Materials Science
1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan
6) Ibaraki University, Department of Science and Engineering
4-12-1 Nakanarusawa, Hitachi, Ibaraki, 316-8511, Japan

Received on July 10, 2011
Presented at the JSAE Annual Congress on May 18, 2011

ABSTRACT: Strength and work-hardening in steels are discussed from the viewpoint of heterogeneous deformation. In-situ neutron diffraction studies made it clear that misfit strains between grains accompanied grain-scaled internal stresses (intergranular stress). In a dual phase steel, the intergranular stress was superposed on the phase stress. These results show good agreement with the predictions of a simple dual-phase material model: the strong martensite phase yields higher stress than the macro-yield stress, resulting in high strengthening of (ferrite + martensite) dual phase steels. Both long-range internal stress and short-range ones such as forest dislocation hardening may cause resistance to dislocation motion in the steels. Therefore, work-hardening takes place more effectively with higher internal stress and larger volume fraction.

KEY WORDS: (Standardized) materials, high-strength steel sheet
(Free) in-situ neutron diffraction, intergranular stress, phase stress [D3]

1. INTRODUCTION

Microstructural design in steels is key to provide a good balance of high strength and high ductility. Strength and work-hardening in steels are discussed focusing on heterogeneous deformation behavior which was experimentally found by means of in-situ neutron diffraction. Plastic deformation of a steel starts at the most preferential site for dislocation motion, i.e., the softest site. Hence, misfit plastic strains are essentially after the onset of local plastic flow, leading to the generation of an internal stress distribution. The phase stress has been measured by a neutron diffraction technique for plastically deformed steels. In order to understand the tensile behavior of a dual-phase alloy, the Eshelby ellipsoidal theory 1) and the Mori-Tanaka mean field concept 2) have been employed. It will therefore be useful to obtain direct evidence of lattice strains and lattice rotation (texture) of the constituents in a dual phase alloy in-situ during deformation, which has been realized by employing neutron diffraction for the bulk average 3).

In the case of a single phase steel, grain to grain yielding occurs at the beginning of deformation because differently oriented grains have different yield strengths. A single phase polycrystalline material behaves as if it would be an extreme of a composite material. That is, this means that stress partitioning occurs between harder phase and softer phase. The misfit strains between grains accompany the grain-scaled internal stress called intergranular stress. In a dual phase steel, this intergranular stress is superposed on the phase stress 4). Even in a single crystal, a strong region such as a dislocation cell wall develops during plastic deformation resulting in the generation of internal stress 5).

The resistance to dislocation motion in steels may therefore be the sum of long range internal stress and a short range one such as forest dislocation hardening. It is believed that the former corresponds to the "athermal stress component" and the latter to the "thermal component".
From an engineering point of view, the balance of strength and ductility/toughness is very important. In order to obtain uniform elongation in strengthened materials, an increase in work-hardening is a key issue.

Hence, this study aims to examine work-hardening behavior during tensile deformation of high strength and high ductility ferrite-martensite dual phase steels at relatively small strain focusing on stress partitioning between the ferrite and martensite and among ferrite grains or martensite packets (blocks) with different orientations, using in-situ neutron diffraction.

2. EXPERIMENTAL PROCEDURES

2.1. Experimental materials

The chemical composition of the steel used in this study was 0.15C, 0.10Si, 1.00Mn, 0.011P, 0.005S, 0.86Ni, 0.76Cr, 0.25Mo, 0.003t-Al in mass%. The specimen machined from a cold rolled steel plate with 1 mm thickness and aged at 737°C and 775°C for 1.8 ks to obtain a ferrite-martensite dual phase microstructure with different martensite volume fractions, which were named as 737-DP and 775-DP steel, respectively.

The microstructures of the two steels were observed using scanning electron microscopy (SEM) (KEYENCE VE-8800). The martensite volume fraction was 46.5 and 76.2 vol.% respectively which was determined by 3 dimensional (3D) topological analysis. The martensite carbon content is 0.33 and 0.2 mass%C (ferrite carbon content calculated by thermo-calc.), respectively as determined by a mass balance calculation. The gauge portion of the tensile test specimen was 4 mm in width, 36 mm in length, and 1mm in thickness. Tensile tests were performed at room temperature with a constant cross-head speed of 0.5 mm/min and an initial strain rate of $2.3 \times 10^{-4}/s$.

The plate tensile test specimens for in-situ neutron diffraction experiments with a gauge length of 50mm, width of 5mm and thickness of 1mm were machined from the as-received treated plates. In-situ diffraction experiments during tensile deformation were carried out at room temperature using an engineering material diffractometer “TAKUMI” at the Japan Proton Accelerator Research Complex facility, Tokai (J-PARC/MLF) (Fig. 1). The instrument is equipped with a 20 kN tensile tester mounted on the diffractometer, with its loading axis turned 45 degrees with respect to the incident beam. There are two detector banks, which measure time-resolved diffraction patterns at fixed horizontal scattering angles of ±90 degrees. Each [hkI] reflection in the diffraction pattern (Fig. 2) is generated by a distinct family of polycrystalline grains similarly oriented with respect to the load axis. The two detector banks thus measure diffraction patterns from grains oriented in axial and transverse geometry with respect to the applied tensile stress. The in-situ diffraction experiments during tensile testing were conducted using stress control, with counting times of approximately 240 s.

Stress diffraction measurements in crystalline materials are based on the precise measurement of the deviation of the lattice spacing ($d_{\text{hkl}}$) of particularly oriented {hkI} crystallographic planes or the lattice parameter obtained from the average lattice spacing ($a_{\text{ph}}$) deviation due to residual or applied stresses. The lattice strain ($\varepsilon_{\text{hkl}}$) and the average phase strain ($\varepsilon_{\text{ph}}$) can be determined from measured changes in $d_{\text{hkl}}$ and $a_{\text{ph}}$, respectively, according to equation (1):

$$\varepsilon_{\text{hkl}} = \frac{(d_{\text{hkl}} - d_{\text{hkl}}^0)}{d_{\text{hkl}}^0}, \quad \varepsilon_{\text{ph}} = \frac{(a_{\text{ph}} - a_{\text{ph}}^0)}{a_{\text{ph}}^0}$$

where $d_{\text{hkl}}^0$ and $a_{\text{ph}}^0$ are the particular lattice spacing and lattice parameter in the stress-free sample, respectively. Individual reflections in the diffraction pattern (Fig. 2) from both detector banks were analyzed by multi-peak fitting to determine the ferrite peak position and the martensite peak position, from which the lattice strains ($\varepsilon_{\text{hkl}}$) during the tensile test were evaluated.
Fig. 3 displays the fitting results of the overlapping (200) peaks along the axial direction (load direction) at 0 and 784 MPa, respectively. As seen in the inserted fitting curves for ferrite and martensite, single-peak fitting was not adequate and hence multi-peak fitting was employed for analysis of the profiles. For such multi-peak fitting, first, the mean carbon concentration of martensite was estimated from the mass balance calculation. The carbon concentration was estimated to be 0.33 and 0.2 mass% for 737-DP and 775-DP steel. Then, $c/a$ of bct martensite was postulated from equation (2) to be 1.014 and 1.009 for 737-DP and 775-DP steel, which was assumed not to change during tensile deformation.

\[ c/a = 1.000 + 0.045 \times X_c \text{ (mass%)} \]  

(2)

The carbon concentration probably differed from grain to grain. Hence, as a first approximation, the obtained diffraction peaks were fitted using two voigt curves with fixed $c/a$ for martensite, as in Fig. 3. A similar separation technique for the ferrite and martensite or ferrite and bainite diffraction peaks was successfully made for synchrotron X-ray and neutron diffraction from a dual phase steel by Cong et al.\(^\text{(11, 12)}\) and Ohnuki et al.\(^\text{(9)}\), respectively. These studies revealed the stress partitioning behavior between the two similar crystal structure phases using such profile analysis. Obviously, the broader peak is from the martensite due to various crystal defects and the narrow peak is from the ferrite. Under a given applied stress, the martensite peak is more shifted, indicating the larger tensile stress in this phase. Such shifting of peaks with loading was examined for other diffraction peaks of (110), (211), (220), and (310) for both steels.

3. RESULTS AND DISCUSSION

3.1. Microstructural characterization and mechanical properties

Characterization of the structure was performed with the aim of determining the ferrite and martensite distribution homogeneity and morphology across the specimens. Fig. 4 shows the microstructures of the steel sheets. 737-DP and 775-DP steel are dual-phase types with martensite volume fractions of 46.5 and 76.2 vol.%, respectively.

Fig. 5 shows the nominal stress-strain curves of the two dual phase steels. Since both steels were stored at room temperature before tensile straining, dislocation locking by segregation of solute carbon did not occur, and the occurrence of a yield point was suppressed. The 775-DP steel has a tensile strength of 915 MPa which is about 80 MPa above that of the 737-DP steel. The yield strength is higher as well. The higher strength levels are generally attributed to the higher phase fraction of the hard second phase. Uniform elongation is affected by the martensite volume fraction.

Fig. 4 Microstructure of the (a) The 737-DP steel with a martensite volume fraction of 46.5 vol.% and (b) The 775-DP steel with a martensite volume fraction of 76.2 vol.% produced by aging at 737°C and 775°C for 1.8 ks.

Fig. 5 Nominal stress-strain curves of the steels with different martensite volume fraction. MVf: martensite volume fraction; LYS: low yield strength; YS: 0.2% proof stress; TS: tensile strength; U.EL: uniform elongation; T.EL: total elongation.
Fig. 6 shows the work-hardening rate as a function of true strain in both steels. The initial work-hardening rate in both dual phase steels is very high. In particular, in the beginning, the 775-DP steel with high martensite volume fraction has a higher work-hardening rate than the 737-DP steel. However, with increasing plastic strain, the work-hardening rate of the 775-DP steel was lower than that of the 737-DP steel.

3.2. Stress partitioning among \{hkl\} family grains

The unique feature of the in-situ neutron diffraction experiment is that it provides information on the evolution of lattice strains and phase fraction in families of distinctly oriented grains during mechanical loading. The dependence on the applied stress of the lattice strains and integrated intensities of individual reflections of the ferrite matrix and martensite are hereafter referred to as lattice plane responses. This allows us to follow the load partitioning not only between different phases but also between sets of similarly oriented grains. Fig. 7 (a) and (b) show the evolution of lattice strains $\epsilon_{hkl}$ of ferrite matrix family grains \{110\}_f, \{200\}_f and \{211\}_f and martensite family grains \{110\}_m, \{200\}_m and \{211\}_m during the tensile test on the 737-DP steel in the axial direction. Throughout the following discussion, for the sake of simplicity and consistency, we will focus solely on the axial response (i.e. diffraction grains oriented similarly along the loading axis). As seen in Fig. 7 (a), the slope is dependent on \{hkl\} indicating different Young’s modulus. The selected ferrite \{hkl\} diffraction elastic modulus shows good agreement with the prediction by the Kröner model for a ferritic steel[14,15]. An indicated two black arrows in Fig. 7 (a), the deviation from linear elastic the line takes place earlier in [110] oriented family grains than [200] family grains. This implies that [110] family grains are softer for plastic flow than the [200] ones, leading to generation of intergranular stresses operating over a ferrite grain as deformation proceeds. When unloaded after tensile plastic deformation, all the ferrite family grains had residual compressive lattice strain (Fig. 8 (a)). As seen in Fig. 8 (b), all martensite \{hkl\}_m were shown to have residual tensile lattice strain in the axial direction. The \{200\}_m residual lattice strain was the highest among these strains. Therefore, the martensite phase has different crystal orientation as well as compared to the ferrite matrix.

The difference in slopes of the hkl responses of both phases below the elastic limit is due to stress partitioning between grains controlled by the cubic elastic anisotropy factor $A_{hkl} = (h^2k^2 + h^2l^2 + k^2l^2) / (h^2 + k^2 + l^2)^2$[14,15]. For cubic steel phases, the greater $A_{hkl}$ implies greater stiffness (diffraction elastic constant, $\epsilon_{hkl}$) of <hkl> family grains aligned axially to the axial direction. In other words, higher $A_{hkl}$ value means the index of easy deformation. Therefore, the axial stiffness of the selected family grain or ferrite matrix increases in the following order: \{110\}_f, \{211\}_f and \{200\}_f, because $A_{200}(0.00) < A_{110} = A_{211}(0.25)$. Similarly, the stiffness of the martensite family grains \{110\}_m, \{211\}_m and \{200\}_m increases in the same order, because $A_{200}(0.00) < A_{110} = A_{211}(0.25)$. This trend can be clearly seen in the ferrite matrix response to applied stress as well as in the martensite responses.
3.3. Stress partitioning between martensite ($\alpha'$) and ferrite ($\alpha$) matrix

To clarify the stress partitioning among the constituent phases, the phase strain of each constituent was estimated by simply averaging the $\{110\}$ and $\{211\}$ reflections lattice strains. This is because the phase strain obtained using the Rietveld refinement is plotted at $A_{hkl} = 0.2^{(16)}$. Hence, the evolutions of the average lattice strain (phase strain) $\varepsilon_{ph}$ of the ferrite matrix and martensite second phase during tensile tests on both the investigated DP steels (737-DP and 775-DP) are plotted in Fig. 9 as a function of the applied stress. As seen in Fig. 9, both ferrite and martensite strains in the axial direction increase linearly with increasing applied stress in the beginning. After the onset of plastic flow, i.e., yielding, phase strain in the ferrite phase hardly changes or slightly increases with increasing applied stress. On the other hand, phase strain in martensite phase increases greatly with an increase in the applied stress after the yielding. These results indicate good agreement with the predictions by a simple dual-phase material model$^{(17)}$; the strong martensite bears a higher stress after yielding, resulting in high work-hardening of the DP steel.

Let us try to draw a comparison between 737-DP and 775-DP steel with regard to the work-hardening mechanism. Fig. 10 shows phase strains in the axial direction as a function of the applied strain. It is found that the phase strain in martensite becomes higher with increasing volume fraction at the same plastic strain level. This means that load transfer occurs more effectively with increasing volume fraction; in other words, plastic relaxation becomes more difficult. As seen in Fig. 10, ferrite phase strain of the 737-DP steel is lower than that of the 775-DP steel. This indicates that the martensite phase with large volume fraction leads to deformation of the ferrite matrix. Thus, ferrite phase strain of the 775-DP steel bore higher stress. And then, martensite phase strain of the 737-DP steel is lower than hat of the 775-DP steel. This must be the effect of the volume faction and it most likely is not the effect of martensite arness.

Therefore, the work-hardening takes place more effectively with higher internal stress and larger volume fraction.

4. SUMMARY

The tensile behavior of ferrite-martensite dual phase steels was studied under in-situ neutron diffraction. The origin of work-hardening behavior was discussed in this paper, leading to the following summary.

1) Misfit strains between grains accompany the grain-scaled internal stress, called intergranular stress. This internal stress also affects the work-hardening behavior.
2) The misfit strains between phases accompany the phase-scaled internal stress which is called the phase stress. This internal stress also affects the work-hardening behavior.

3) Work-hardening takes place more effectively with higher internal stress and larger volume fraction.

ACKNOWLEDGMENTS

This series of investigations has been carried out with the assistance of the instrument scientists at the neutron research facility (J-PARC). The authors highly appreciated useful discussions at member meetings of the ISIJ research group (work-hardening properties and microstructures, leader Prof. Higashida).

REFERENCES


Copyright © 2011  Society of Automotive Engineers of Japan, Inc. All rights reserved