Scripta Materialia 140 (2017) 45–49

Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.elsevier.com/locate/scriptamat

Regular article

Multi-scaled heterogeneous deformation behavior of pearlite steel studied by *in situ* neutron diffraction

Yanxu Wang [a,b,*], Takahisa Ohnuki [c], Yo Tomota [b], Stefanus Harjo [d], Takahito Ohmura [a,b]

[a] Kyushu University, 774 Motooka, Nishi-ku, Fukuoka 819-0395, Japan
[b] Research Center for Structure Material, National Institute for Materials Science, 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan
[c] Tokyo Metropolitan College of Industrial Technology, 8-17-1, Minami-Senju, Arakawa, Tokyo 116-8523, Japan
[d] J-PARC Center, Japan Atomic Energy Agency, 2-4, Shirane Shirakata, Tokai, Ibaraki 315-1195, Japan

ARTICLE INFO

Article history:
Received 19 June 2017
Received in revised form 6 July 2017
Accepted 6 July 2017
Available online xxxx

Keywords:
Neutron diffraction
Pearlite steel
Heterogeneous deformation
Diffraction profile
Stress partitioning

ABSTRACT

Heterogeneous deformation of Fe-0.8 mass%C pearlite steel was studied using *in situ* high-resolution neutron diffraction during tensile deformation, characterized by the superposition of multi-scaled stress partitioning behaviors between the ferrite and cementite phases (phase stress), ⟨hkl⟩-oriented grain families with respect to the tensile direction (intergranular stress), and colonies with different lamellar alignments (colony stress). The colony stress was evaluated by analyzing profile asymmetry. The influence of lamellar spacing on such stress partitioning was clarified, and large peak broadening of cementite was also determined in a deformed specimen.

Pearlite steel is widely used in industrial applications, and has particularly attractive properties, because it exhibits high strength with effective ductility, even following cold wire-drawing [1], having recorded the highest strength above 6 GPa [2]. Pearlite consists of a two-phased lamellar structure, with a brittle cementite (θ) and ductile ferrite (α) phase, which is formed by diffusional eutectoid transformation from the austenite phase, super-cooled below A_1 temperature (727 °C for the Fe-C binary system). The pearlite structure is composed of blocks that are sub-divided into lamellae, known as colonies. A block has classically been identified as the region with a certain α crystallographic orientation [3], while recent studies using the electron backscatter diffraction (EBSD) method have revealed that the crystallographic orientation is slightly rotated within an individual block [4–6].

Using *in situ* neutron diffraction during tensile deformation, Tomota et al. [7,8] and Oliver et al. [9] elucidated the stress partitioning between the α and θ phases (phase stresses) for α-θ lamellar or θ-spheroidized steel specimens, where internal stresses are accompanied by misfit plastic strains between the two phases. The macroscopic yield strength of pearlite corresponds to the preferential onset of plastic flow in the α matrix. Similar behavior has recently been reported by Zheng et al. for θ-spheroidized steels, using *in situ* synchrotron X-ray diffraction [10]. The misfit plastic strains between the α and θ phases yield phase stresses that are influenced by the morphology of θ. Furthermore, plastic flow in the individual α grain is dependent on the crystallographic orientation, ⟨hkl⟩, with regard to the tensile direction, as a result of the Schmid factor for the slip-system and strong elastic anisotropy of the bcc crystal. This is generally known as "intergranular stress" and should be referred to as "block stress" in pearlite. Therefore, phase and block stresses are superimposed in a plastically deformed α matrix, and potentially in θ. The elastic anisotropy of θ is also strong [11] and the real morphology of the θ grain is not of the simple plate [12]; recent scanning electron microscopy (SEM)/EBSD observations with the serial sectioning method have revealed the complicated topological features of the cementite particle [13]. According to recent reports on the change in specimen surface with deformation (in plane stress conditions) employing a high-precision marker technique [14] or digital image correlation method [15], certain colonies that aligned at approximately 45° with regard to the tensile direction show extremely preferential plastic flow, suggesting a further scaled microstructural heterogeneity. It should be made clear whether or not such heterogeneous plastic deformation takes place, even within the specimen.

The heterogeneous plastic deformation of pearlite steel is likely much more complicated than our current understanding thereof. Hence, in this paper, *in situ* neutron diffraction measurements during tensile deformation of pearlite steels were re-examined, using high-resolution neutron diffraction, with the aim of clarifying (1) the

* Corresponding author at: Kyushu University, 774 Motooka, Nishi-ku, Fukuoka 819-0395, Japan.
E-mail addresses: WANG.Yanxu@nims.go.jp (Y. Wang), ohnuki@metro-cit.ac.jp (T. Ohnuki), TOMOTA.Yo@nims.go.jp (Y. Tomota), stefanus.harjo@j-parc.jp (S. Harjo), OHMURA.Takahito@nims.go.jp (T. Ohmura).

http://dx.doi.org/10.1016/j.scriptamat.2017.07.004
1359-6462/

superposition of multi-scaled heterogeneous plastic flows, (2) the effect of lamellar spacing, and (3) diffraction peak broadening of the θ as well as α phase.

The steel used in this study was created by induction heat melting in a vacuum, and the chemical compositions in mass% were 0.801C-0.01Mn-0.01Si-0.01P-0.01S. The ingot was hot-forged at 1473 K to rods with a 32 mm diameter, and the rods were annealed at 1103 K for 3.6 ks, followed by air cooling. Using salt baths, the rods were austenitized at 1103 K for 3.6 ks in a vacuum and then isothermally held at 673 K or 823 K for 3.6 ks, in order to obtain full pearlite structures with different lamellar spacings. Dog-bone-shaped specimens, with a diameter of 3 mm and gauge length of 30 mm, were prepared.

The *in situ* neutron diffraction measurements were performed using a high-intensity, high resolution ($\Delta d/d = 0.2\%$ (d: lattice spacing)) time-of-flight (TOF) neutron diffractometer, TAKUMI (BL19), at the Materials and Life Science Research Facility (MLF) of the Japan Proton Accelerator Research Complex (J-PARC). A 20 kN tensile tester was mounted on the diffractometer in such a way that the loading axis became 45° with the incident beam. Therefore, diffraction profiles for two scattering vectors in terms of the tensile (axial) and transverse directions were measured simultaneously by two detector banks set at ±90° against the incident beam. The specimens were deformed in tension at room temperature in a step-by-step manner in the elastic deformation region, and subsequently a continuous manner in the elasto-plastic region, using a strain rate of 10^{-5} s^{-1}. The strain was measured and controlled using a strain gauge that was glued onto the specimen surface. Neutron diffraction intensities were continuously recorded by means of a tensile test with an event-mode data acquisition system. The data were time-sliced with 900 s intervals for analyses following measurement. The *Z*-Rietveld software [16] was employed to analyze the time-sliced data, whereby the diffraction profiles were fitted with the convolution of a pseudo-Voigt (Gaussian and Lorentzian) function and instrumental parameters. Asymmetric diffraction peaks were analyzed by assuming two ferrite phases with different lattice parameters, and experimental errors were calculated from curve-fitting errors.

The SEM microstructures of specimens are shown in Fig. 1. The wide-view characteristic is found in (a), observed with a low magnification, while the difference in lamellar spacing between the two specimens prepared at different transformation temperatures is found in (b) and (c), with a higher magnification. The average lamellar spacings in (b) and (c) are 82 nm (hereafter referred to as fine pearlite) and 160 nm (coarse pearlite), respectively. The average colony size is approximately 6.97 μm, which is determined by the intercept method. Assume a spherical colony, then, the number of colonies in the gauge volume ($5 \times 5 \times 5$ mm^3) for neutron diffraction is estimated approximately 6.9×10^{10}. Fig. 1(d) displays the details of the diffraction profile at around α 110 peak, obtained from the tensile direction. Low-intensity peaks are completely indexed for an orthorhombic crystal structure, given by the space group Pmna (with lattice parameters c < a < b-axis) [17]. Here, both the α and θ peaks are found to exhibit not only shifting, but also broadening, following deformation. Such cementite peak broadening has been reported for deformed bainitic steel [18,19] and fine-grained α–speroidized θ steels [10], and was apparently confirmed to occur similarly in lamellar pearlite steel in this study.

The stress-strain curves recorded during the *in situ* neutron diffraction measurements are illustrated in Fig. 2(a). The yield strength was defined by 0.2% proof stress ($\sigma_{0.2}$). It can be seen that the square and circle marks indicate the data acquisition steps of neutron diffraction during the tensile test. Both the fine and coarse pearlite specimens exhibit continuous yielding, with high work hardening. These results are similar to those reported in Refs. [7,18,20]., but differ from those for fine-grained α–spneroidized θ steels [10], in which discontinueous yielding with a Lüders band was observed. The Lüders deformation is a macroscopic heterogeneous deformation mode that is affected by various microstructural factors in α–θ steels, and is strongly related to microscopic

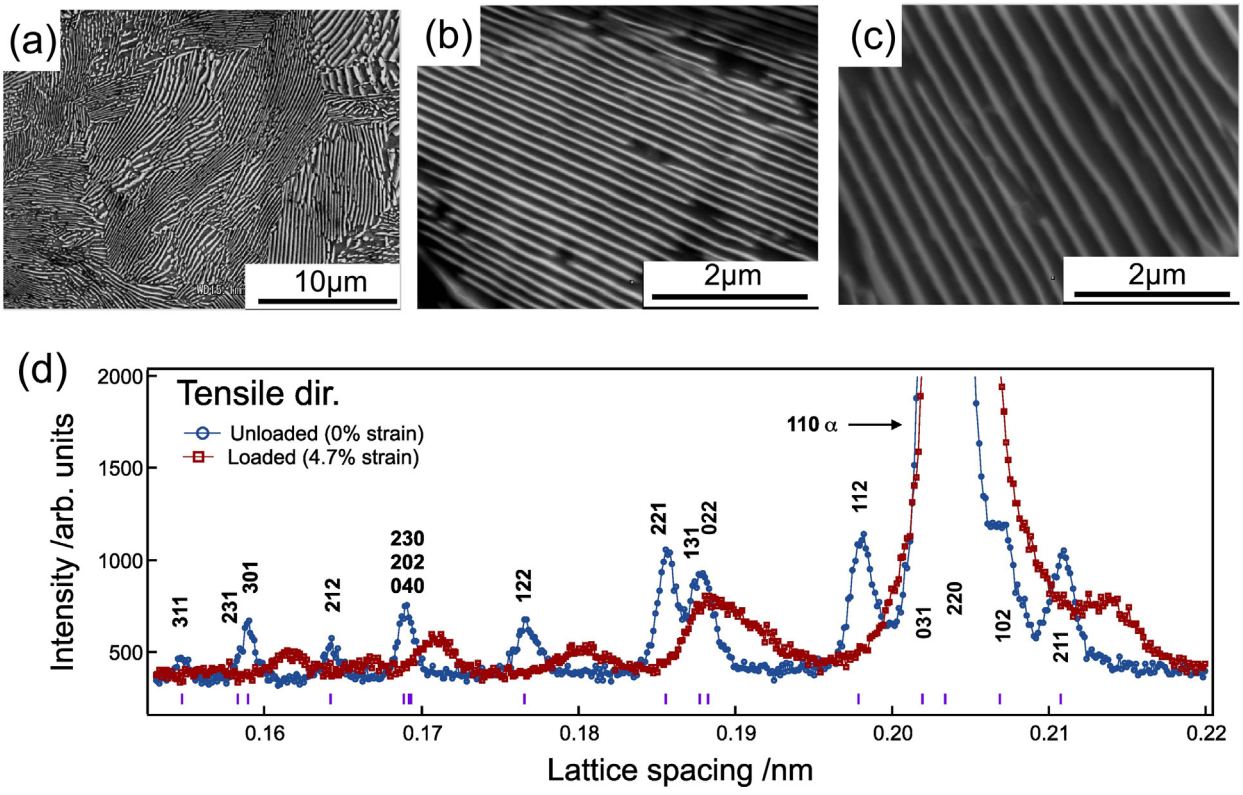

Fig. 1. Microstructures and neutron diffraction profiles of pearlitic steel: (a) SEM micrograph with a low magnification (b) that with a high magnification for fine pearlite and (c) the same for coarse pearlite; (d) neutron diffraction profile obtained from the tensile direction magnified around the ferrite 110 peak for fine pearlite to show cementite peaks, where low intensity peaks were indexed as cementite for unloaded profile.