In Situ Neutron Diffraction during Tension-Compression Deformation for Nodular Graphite Cast Irons

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1. Introduction

Work-hardening behavior of real engineering materials has been elucidated by means of in situ neutron diffraction during deformation.1–3) To understand work-hardening mechanism more deeply, the change in deformation path is effective to be examined.2–5) A typical test for such purpose is a tension-compression test. Harjo et al. have reported that the tension-compression deformation behavior of austenite-ferrite duplex stainless steels by using an angular dispersion neutron diffraction with a reactor neutron source.5) They have made clear three deformation stages based on the changes in phase stresses during deformation and claimed that the phase stress enhances the Bauschinger effect.6) In their work, however, only (111) austenite and (110) ferrite diffraction peaks were simultaneously tracked using a position sensitive detector. Another drawback of their experiment was a step by step loading manner in order to increase statistic reliability of diffraction profile. Creep deformation occurs during the external stress holding, while stress relaxation during stopping the cross head movement. If we could use a time of flight (TOF) method with a spallation neutron source, we would obtain more insights on heterogeneous deformation behavior. The proton beam power at MLF/J-PARC increased from 18.5 kW in 2009, 116 kW in 2010, to 218 KW in 2011 (finally planned to be 1 MW). With increasing of neutron beam intensity, the time slicing measurement is becoming a powerful tool to study dynamic behavior of engineering materials. The 218 KW beam enabled us to perform in situ neutron diffraction during tension-compression test without stopping for data acquisition.

Claussen et al. have claimed that intergranular stress plays an important role for work hardening including the Bauschinger effect using an EPSC model analysis and in situ TOF neutron diffraction for a polycrystalline austenitic steel.1,2) It is, therefore, postulated that the phase stress and the intergranular stress must be overlapped to cause the Bauschinger effect in engineering materials like cast irons which consist of ferrite, cementite and graphite. Hence, in this paper, tension-compression deformation behavior was studied for three cast irons with different microstructures using in situ TOF neutron diffraction, which could be applied to cyclic deformation, i.e., low cycle fatigue behavior in near future.

2. Experimental Procedures

The chemical compositions of cast irons used in this study are 3.71C–2.70Si–0.31Mn–0.02P–0.008S in mass% and balanced Fe. Test specimens with 5 mm diameter and 4 mm gauge length were prepared from cast blocks after heat treatment. In situ neutron diffraction profiles were measured during tension-compression test at room temperature using an engineering neutron diffraction meter, TAKUMI, at MLF/J-PARC. A specimen was deformed in tension firstly with a stress increasing rate of 0.5 MPa/s in an elastic deformation regime and then 0.05 MPa/s in an elasto-plastic deformation regime up to a certain plastic strain followed by unloading and continuously deformed in compression. An event mode of data acquisition system employed at MLF enables us to analyze the obtained data by changing data slicing time interval after measurement. Taking statistic sufficiency into consideration, the time interval can be decreased with increasing of neutron beam intensity. In 2009 and 2010, some diffraction profiles were obtained by keeping the applied stress constant in a step by step loading manner to increase the statistic reliability. The diffraction profiles corresponding to the axial and transverse direction were obtained by two 90 degrees banks, as reported in the previous paper.

The analysis of neutron diffraction enables us to determine microscopic parameters, like lattice strain, ε\text{lat}1–5) The single-peak fitting6) and the Rietveld refinement for multi-peaks7,8) were employed to determine (hkl) intergranular strain and phase strain, respectively.

3. Experimental Results and Discussion

Figure 1 shows optical microstructures of three nodular graphite cast irons which were made by changing heat-treatment conditions: the microstructure of specimen A consists mostly of ferrite, B pearlite-ferrite mixture and C mostly pearlite. The microstructural parameters of these three irons are tabulated in Table 1. The volume fractions of ferrite, pearlite and graphite were measured using an image processing software for optical micrographs. In diffraction profiles obtained at the continuous loading test, cementite peaks could be detected clearly as the beam intensity became higher in 2010.

Stress-strain curves of the present cast irons obtained by the in situ neutron diffraction experiments are shown in Fig. 2(a). As seen, both the yield strength and tensile strength increase with increasing of pearlite volume fraction. The phase strains determined by the Rietveld refinement for ferrite, cementite and graphite were plotted as a function of the applied stress in Fig. 2(b). As is observed, the graphite strains are always nearly zero during loading, suggesting that the graphite does hardly bear stress. Similar result was reported for the graphite phase in ADI.13) The graphite has a layered structure. The carbon-carbon bonding energy between the layers (van der Waals bonding) is approximately 1/20 of that in (0001) layer14) and hence sliding or decohesion along (0001) plane is thought to occur quite easily. The phase strains of ferrite and cementite increase with increasing of the applied stress linearly in the elastic deformation stage (stage I) commonly observed for specimens B and C. The onset of stress (strain) partitioning is observed at a lower applied stress in specimen B than in C, corresponding to their 0.2% proof stresses, 318 MPa and 642 MPa, respectively. After the onset of plastic flow, i.e., stage II (see Fig. 2(a)), the phase strain of cementite increases more rapidly whereas that of ferrite slowly. Because the volume fraction of cementite is higher, stress partitioning occurs more

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remarkably resulting in higher work-hardening in specimen C. On the other hand, in specimen A, such phase strain could not be detected because cementite volume fraction is extremely low.

When the single peak fitting method was employed, the deformation behavior of individual [hkl] oriented family-grains were determined as [hkl] lattice strains indicating the generation of intergranular strains (stresses). As a typical example of intergranular strains, the (110), (200) and (211) lattice strains in the ferrite matrix for specimen A were presented in Fig. 2(c). As seen, these strains behave differently. The different slope of the stage I stems from the anisotropic elastic properties of a bcc crystal. After the yielding (see Fig. 2(a)), the deviation from the elastic line is observed particularly in (110) and (200) strains, meaning the onset of stress partitioning among the [hkl] oriented family-grains, i.e., generation of intergranular stress. In cases of specimens B and C, such intergranular stresses are overlapped onto phase stresses. Here, the results of Figs. 2(b) and 2(c) were obtained by the continuous loading test in 2010. These data for ferrite phase showed good agreements with the results obtained by the step-by-step loading test in 2009. Because the neutron beam power increased to 116 kW at J-PARC in 2010 (5.5 times stronger than before), non-stop loading became applicable with an event mode data acquisition system, which encouraged us to perform continuous tension-compression test.

The flow curves of tension-compression continuous loading are presented in Fig. 3 for specimen A and Fig. 4 for specimen C. According to Tomota and Kuroki,15) the characteristics of the Bauschinger effect are evaluated by using either the Bauschinger stress $\sigma_B (= \sigma_T - |\sigma_R|)$ or the Bauschinger strain $\beta_{0.5}$. The Bauschinger stresses obtained here are 123 MPa for specimen A, 259 MPa for B and 610 MPa for C while the Bauschinger strains are 0.22 for A, 0.30 for B and 0.50 for C. These results indicate clearly that the Bauschinger effect becomes more remarkable with increasing of pearlite (or cementite) volume fraction. Similarly to Harjo et al.,5) the introduction of the hard second phase enhances the Bauschinger effect. As can be seen in Fig. 3(a), the hysteresis loop of ferrite (110) strain is much different from that of (200), showing that the intergranular stress built up during the forward deformation accelerates the backward plastic flow resulting in an increase of the Bauschinger stress and Bauschinger strain (some corresponding points in (a) and (b) are denoted by encircled numerals). The phase stress contributes more effectively to enlarge the Bauschinger stress or strain which can be found in Fig. 4(a) for specimen C. Here, the diffraction profile was obtained by collecting every 0.48 ks during continuous loading with the beam intensity of 218 KW in 2011. The phase strains were determined using the Z-code Rietveld refinement and the relevant hysteresis loops obtained are demonstrated both for ferrite and cementite in Fig. 4(a), where five corresponding points in (a) and (b) were denoted by encircled numerals. It is revealed that the ferrite phase deforms plastically in tension as well as in compression preferentially. The phase strain of ferrite is compressive after unloading from tensile loading and tensile after compression loading (see points ③).
and ⑤). The phase stress built up in the forward deformation in ferrite is evidently found to accelerate the reverse deformation, resulting in large Bauschinger effect. The magnitude of phase or intergranular stresses stem basically from the strength difference either between constituent phases or differently oriented grains. The strain difference between ferrite and cementite at the unloaded point ③ after tension in Fig. 4 is 2.6 × 10⁻³, whereas that between [110] oriented grains and [200] ones is 3.5 × 10⁻⁴. The larger the internal stress (or strain) becomes, the larger the Baushinger effect because such internal stresses assist only the plastic deformation in a specific direction.

4. Conclusions

Tension-compression deformation behavior of three nodular graphite cast irons with different microstructures was studied using in situ neutron diffraction. The continuous tension to compression loading with neutron diffraction measurements could be performed at J-PARC. The main conclusions obtained are as follows.

(1) It has experimentally been revealed that graphite particles hardly bear stress. The strength of ferrite phase is mainly contributes to the yield strength whereas pearlite mostly to work hardening.

(2) Phase stresses and intergranular stresses were determined during tension-compression deformation. It is found that the larger the magnitudes of phase and/or intergranular stresses, the larger the Bauschinger effect.

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