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In situ TOF neutron diffraction during phase transformation in an engineering steel

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ABSTRACT

Phase transformations on heating and cooling or during thermo-mechanically controlled processing (TMCP) for a low alloyed steel were studied by means of *in situ* neutron diffraction. Cylindrical specimens were quenched after austenitization and hence the starting microstructure was martensite (bct). Tempering behavior accompanying cementite precipitation and ferrite (bcc) to austenite (fcc) reverse transformation were observed upon heating. During cooling, austenite to ferrite and pearlite transformations were observed in diffraction profiles, where nearly equilibrium microstructural changes were confirmed. In TMCP simulation measurements, plastic deformation of austenite at an elevated temperature was found to accelerate the ferrite transformation.

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1. Introduction

It has been recognized that *in situ* neutron diffraction is a powerful tool to investigate martensitic transformation in high alloyed steels or some shape memory alloys because of their athermal character, i.e., diffusionless transformation. The statistic reliability can be improved by increasing the measuring time [1,2]. Particularly, Time Of Flight (TOF) measurements with multi-detectors provide useful data on phase volume fraction, texture, phase and intergranular strains (stresses), which cannot be obtained by any other experimental techniques. This method is expected to be applied to diffusional transformations and recrystallization in different steels and such a challenge has been reported [3,4]. Although the time resolution is not enough to follow the quick transformation kinetics at the present, the situation must be improved in near future at J-PARC. In this study, the microstructural evolution during step-by-step heating and cooling or thermo-mechanically controlled processing (TMCP) for a low alloyed steel was monitored by means of *in situ* TOF neutron diffraction.

2. Experimental procedures

A low alloyed steel (0.19C–1.95Mn–0.01Si in mass%) was used in this study. A cylindrical specimen with 7 mm in diameter and

50 mm in length was solution treated at 1273 K followed by quenching into water. This as-quenched specimen with martensite structure was heated up to 1173 K and then cooled to RT in a step-by-step manner. At each holding temperature for 3.6 ks, neutron diffraction profiles were measured using SIRIUS at KENS/KEK. Next, the effect of austenite deformation to ferrite transformation at an elevated temperature was examined using ENGIN-X at ISIS. Here the compression specimens with 10 mm in diameter and 15 mm in length were used. An Instron hydrostatic loading jig and a radiation heating with gas cooling system were employed to simulate TMCP, where strain was measured by a high temperature extensometer.

3. Experimental results

3.1. Phase transformations and precipitation of cementite during heating and cooling

Fig. 1 shows changes in (110) ferrite (or martensite) and (111) austenite diffraction profiles on heating and cooling. The starting microstructure is martensite (bct), so that the full width at half maximum (FWHM) for (110) ferrite profile is large (see the datum at 298 K). With heating, tempering of martensite takes place accompanying cementite precipitation although it is not clearly recognized in Fig. 1 because of a small volume fraction and FWHM in (110) ferrite profile becomes smaller due to decreasing of tetragonality of bct martensite. The peak shift with increasing of temperature observed in Fig. 1 is caused mainly by thermal

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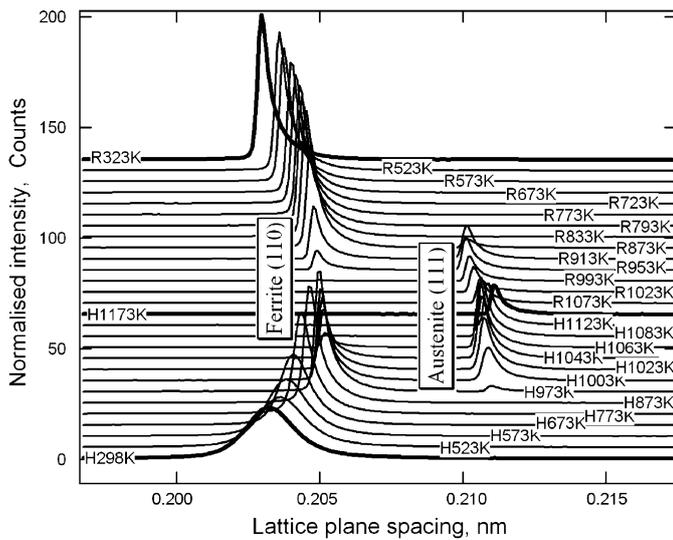


Fig. 1. Change in diffraction profile with heating and cooling. Here, H and R refer heating and cooling, respectively.

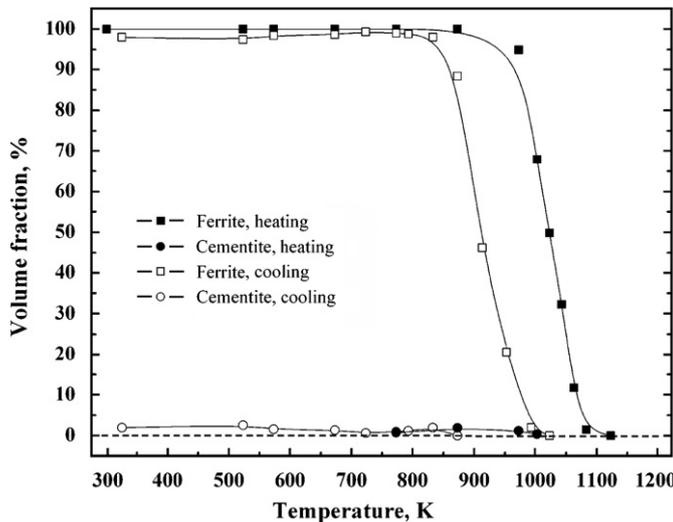


Fig. 2. Volume fraction change of the constituents with heating and cooling.

expansion. The volume fractions of ferrite (or martensite) and cementite determined by the Rietveld refinement are presented in Fig. 2 as a function of temperature, where the volume fraction of austenite is the balance from those of ferrite and cementite. Austenite is firstly generated with dissolution of cementite particles. The lattice parameters of ferrite and austenite are plotted as a function of temperature in Fig. 3. It is found that the lattice parameter of austenite is large in the beginning (see austenite data at “Heating”) because of high carbon concentration caused by cementite dissolution. With further heating, the austenite volume fraction increases and the lattice parameter changes, being influenced by thermal expansion and decreasing of carbon concentration. To be noted here is the deviation from the linear thermal expansion line for ferrite, meaning the generation of phase stresses caused by misfit strains between ferrite and austenite. The misfit strains are yielded by transformation strains and different thermal expansion coefficients of the two constituents. After reaching 1173 K, the microstructure becomes single austenite. Then, during the cooling, austenite to ferrite transformation and pearlite transformation took place

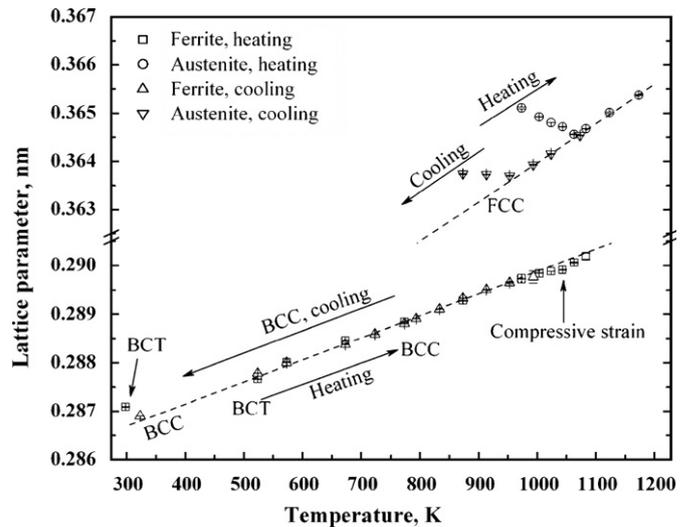


Fig. 3. Lattice parameters as a function of temperature.

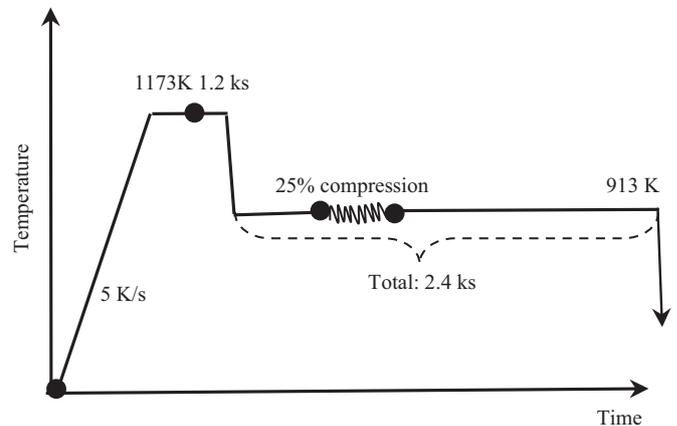


Fig. 4. Schematic drawing of a TMCP schedule employed.

subsequently. Upon cooling, the austenite lattice parameter decreases before the onset of ferrite transformation. With proceeding of ferrite transformation, carbon is enriched in austenite leading to small change in the lattice parameter of austenite. Finally, the carbon enriched austenite decomposed to pearlite structure, i.e., lamellar structure consisting of ferrite and cementite. Because every temperature holding step was 3.6 ks, the evolution of microstructure could be explained from the equilibrium phase diagram. At the next stage of study, it is needed to monitor non-equilibrium condition in a short time for real engineering heat treatments.

3.2. Thermo-mechanically controlled processing (TMCP)

The final target is to simulate real TMCP. In order to understand quantitatively what is happening during TMCP, an *in situ* neutron diffraction measurement was performed using ENGIN-X at ISIS. The TMCP schedule employed is drawn in Fig. 4. After austenitization at 1173 K (austenite stable temperature), a specimen was cooled down to a certain temperature for isothermal transformation. Some of the specimens were deformed by 25% in compression before or during ferrite transformation. Examples of diffraction profiles obtained in 120 s at various stages are summarized in Fig. 5. As seen, the start microstructure is martensite (see the four martensite peaks in the bottom profile

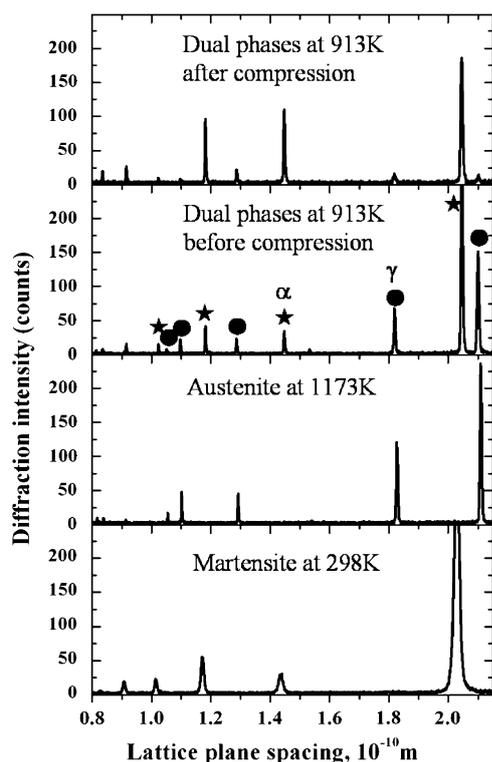


Fig. 5. Diffraction profiles obtained at four stages in the TMCP schedule shown in Fig. 4.

at 298 K), transformed to austenite fully at 1173 K and then ferrite is formed during cooling. In the profile obtained at 913 K (dual phase region) in Fig. 5, the peaks labeled by solid circles are corresponding to austenite (γ : fcc) while those labeled by star marks are ferrite phase (α : bcc). Then, the specimen was plastically deformed in compression and the diffraction profile

was found to change drastically; one is an increase in ferrite volume fraction and the other is the change in (hkl) intensity ratio (compare the profiles before and after compression in Fig. 5). The former result indicates that ferrite transformation is accelerated by plastic deformation, while the latter reveals the evolution of texture both in ferrite and in retained austenite. If the holding step is set at various temperatures, so called TTT (Temperature-Time-Transformation) diagram would be obtained. To evaluate dislocation density using a profile analysis, more statistically sufficient data are required. It would become possible when strong neutron beam can be used in the near future.

4. Concluding remarks

It is evident that *in situ* neutron diffraction is very powerful tool to investigate athermal martensitic transformation. Although there are some difficulties in time resolution for diffusional transformation, the situation must be improved greatly by using a new diffractometer to be installed at J-PARC. The material processing in industry is so complicated that the underlying scientific phenomena are in many cases estimated only by indirect measurements. Therefore, *in situ* neutron diffraction must bring a big impact to develop advanced material processing. The content of this paper was partially reported by oral presentations [5–7] and the full papers are now under preparation.

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