Studying ultrafast dynamics of temperature-dependent dislocation in Fe-0.1mass%C using femtosecond X-ray diffraction

In the iron and steel industry, the development of new alloys and the improvements in manufacturing processes have resulted in the commercialization of several new materials systems. Specifically, the process control during steel production has attracted significant attention in the automobile industry. In the steel manufacturing process, the heat treatment plays an extremely important role. During heat treatment, nonequilibrium states may be produced as a result of ultrafast microstructural changes directly affecting the final properties of the metal, resulting in new alloys [1]. However, the difficulty of observing atomic diffusion and dislocation recovery during such ultrafast microstructural changes hinders the physical and kinetic understanding of these processes. The martensitic transformation is an extremely important phenomenon in physical metallurgy. Many studies have focused on the rapid heating of low-carbon steel, and improved ductility and strength by the refinement of crystal grains has been reported [1]. However, a complete understanding of the phenomenon remains elusive owing to a lack of an operand measurement. To the best of our knowledge, the microstructural changes under ultrafast heating rates greater than 10^4 °C·s^{-1} have not been observed directly in steel.

To observe these ultrafast and irreversible changes in the steel microstructure, single-shot X-ray diffraction (XRD) measurements are highly effective. Generally, high intensity X-rays obtained from synchrotron radiation require a very short exposure time (10 ms) for recording the XRD patterns [2]. However, this exposure time is far greater than the temporal resolution of the target (which is less than 1 μs) required for the direct observation of the microstructural changes in iron and steel. Therefore, X-ray beam with an intensity four orders of magnitude higher than that of synchrotron X-rays is required. X-ray free-electron laser (XFEL) is capable of producing such high intensity X-rays. In this study, time-resolved XRD measurements were carried out to clarify the changes in the dislocation density and carbon concentration during the martensitic transformation of steel at ultrafast heating rates of 10^3–10^4 °C·s^{-1}. The experiments were carried out at SACLA **BL3**. The influence of the ultrafast heating on the formation of the microstructure is discussed within the context of the dislocation migration [3].

Figure 1 shows the experimental setup of XRD measurements using femtosecond XFEL pulses. For a steel sample under ultrafast heating, a two-dimensional XRD pattern was recorded using a multiport charge-coupled device (MPCCD) detector. The ultrafast electrical heating, the temperature measurement, and the X-ray detection were synchronized with a trigger signal from the XFEL source. Both edges of the sample (15 mm × 5 mm × 0.5 mm) were affixed to copper electrodes to facilitate resistive heating. The sample had a known mass composition of 0.1 mass%C-2 mass%Mn-bal.Fe with a martensitic microstructure, and it was cold-rolled for a 50% reduction in thickness. The XRD line profiles were obtained by integrating the diffraction images in the circumferential direction. Ungár *et al.* proposed that the X-ray line profiles could be analyzed by considering the effects of anisotropic lattice strains along the crystallographic directions, and the strength of the lattice strains around the dislocations [4]. These characteristics are used to deduce the optimal relationship between the dislocation density and the X-ray line profiles.

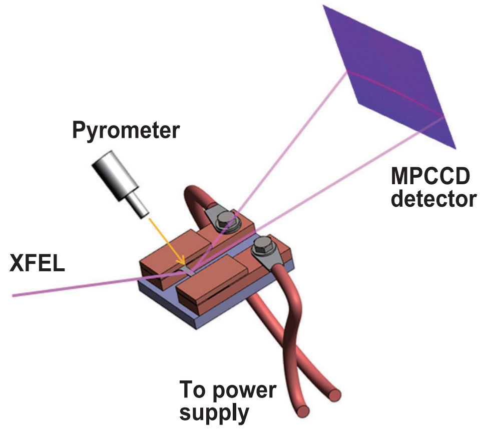

Fig. 1. Experimental setup of femtosecond XFEL.

Figure 2 shows the temperature dependence of the screw and edge components of the dislocation densities for a heating rate of 1.2×10^4 °C·s^{-1}. In the α' phase, the total dislocation density is approximately constant below 500°C. However, it is noted that in this temperature region (100–500°C), the edge dislocations are decreased while the screw dislocations are increased along with the increase in the temperature. This behavior is attributed to the migration of the high mobility edge dislocations towards the deformed dislocation loop, indicating that the edge dislocations are unstable below 500°C. Furthermore, the edge dislocation component decreases drastically above 600°C, while the screw dislocation component decreases insignificantly above 600°C. Therefore, the screw dislocations are predominant dislocations

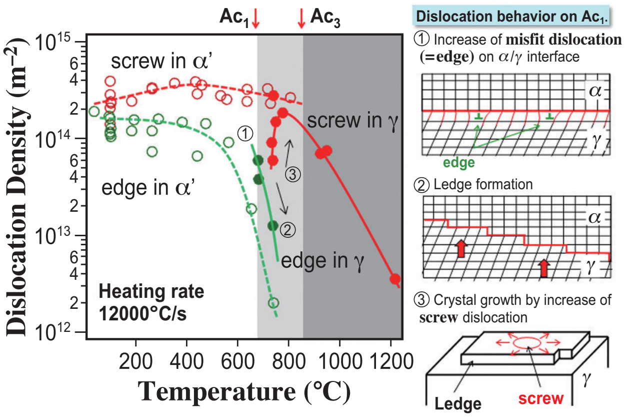

Fig. 2. Temperature dependence of the dislocation densities of the screw and edge dislocations at 1.2×10^{4} °C s^{-1}.

in the α' phase at temperatures above the perlite transformation (Ac_1). On the other hand, the edge dislocations are predominant in the γ phase around Ac_1 region. As the temperature increases, the dislocation type immediately changes from the edge dislocations to the screw dislocations. The screw dislocation component in the γ phase decreases rapidly in 100% austenite structure above austenitizing temperature (Ac_3). It is seen that, the edge dislocation which are unstable at the high temperature, fall in number and the screw dislocations are introduced in the lattice for promoting the three-dimensional screw growth. The dislocation multiplication in the γ phase of the steel, thus results in the crystal growth of the γ phase.

Figure 3 shows the microstructure formation mechanism. In this study, the dynamic changes in the carbon concentration and the dislocation densities were successfully observed for a timescale shorter than 0.1 s. These changes indicate the precipitation of fine particles of θ-Fe_3C and a delay in dislocation recovery. At the heating rate of 10^4 °C·s^{-1}, the diffusion distance of carbon is extremely short and thus, the growth of θ-Fe_3C particles is restricted before the α' to γ phase transformation takes place.

The dislocation density is one of the key factors in producing high-strength steel. Fine grains of γ phase with high dislocation densities result from the transformation of the α' phase with a high dislocation density, at a high heating rate. The dislocation density is further multiplied by a martensitic transformation with rapid cooling. This study shows that the direct observation of a dynamic change in the dislocation density enables our understanding of kinetics in the microstructure, under steep thermal gradients, and contributes to the further development of the functional steels and new manufacturing processes.

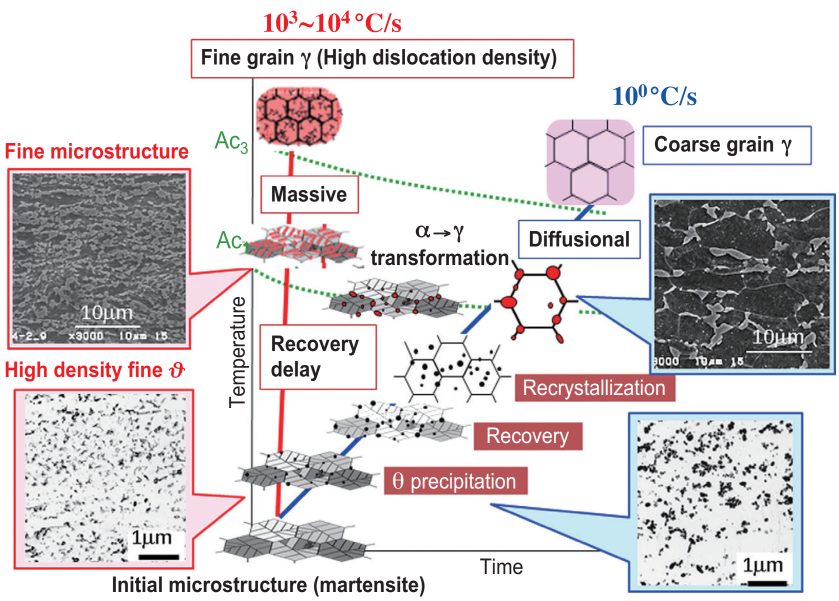

Fig. 3. Microstructure formation mechanism.

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References

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