Development of double-tilt heating holder for dynamical/atomic resolution AC-TEM and WB-STEM observations of ferromagnetic ferrous alloys

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The *in-situ* annealing observation is one of effective methods for visualizing thermal microstructural changes such as phase transformation, coalescence and so on in dynamic time scale. For applying this dynamical characterization to bulk samples fabricated by ion-milling, electro- polishing and FIB mill, it is generally needed to use the heating-pot type system. However, the conventional heater system has some problems at spatial resolution and its temporal stability because of the continuous thermal expansion and contraction with sample drift caused. In addition, the wide gap of pole piece for a double axes of sample tilting gives the large effects of spherical aberration. Therefore, it was necessary for modifying the heating system to obtain dynamical/atomic resolution observation of bulk samples. We here report an initial trial to investigate burring on the spatial and temporal resolution of the *in-situ* annealing observation of bulk samples using a spherical aberration corrected transmission electron microscopy (AC-TEM) with a new thermal control unit.

In our unit, an achieving heating current was controlled using the temperature of a heating pot measured by a direct thermocouple. The fluctuation of temperature were below ±1˚C per 5 seconds at 30 seconds after the holder has reached target temperatures above 350˚C. Figure 1(a) is a spherical aberration corrected transmission electron microscopy (AC-TEM) image of from Au/Pd grating sample at 350˚C. It is indicated that the Au/Pd(200), d = 1.99 Å, was continuously achieved. The information limit as shown by Young fringe in Figure 1(b) showed the 1.5 Å resolution, acquired at the pixel size of 0.6 Å/pixel and the frame rate of 2 fps. Figure 1(c) shows the time dependences of the information limit of the sample maintained the constant temperatures. The information limit had a highly stable to time regardless of the target temperature. While the sample was being heated at a temperature rising rate of +1.0˚C/s, the distance of sample drift acquired by a high-speed CCD camera was below 2.0 Å/s.

In the present study, we also tried to apply the heating system to two kinds of ferromagnetic ferrous alloys. One is a partial vessel steel sample, which was used in a European nuclear reactor for 30 years. The FIB fabricated sample was observed at 400˚C using weak-beam STEM [1], and the *in-situ* annealing clarified that decreasing of dislocations are dominated by initial lattice defects and grain boundaries in real-space as shown in Fig. 2 (a) and (b). Another sample is Fe-15at%Si magnetic alloy sample fabricated by ion-milling. Some bcc phase Fe grains the size of 100 nm formed and moved to the hole fabricated by ion-milling over 600˚C. At the same time, the fine crystals the size of 10 nm precipitated in the matrix phase. These results indicate that the new heating-pot type *in-situ* annealing system can widely apply to bulk samples with high spatial and temporal resolution stability.

[1] K. Yoshida et al. *Microscopy* 66 (2017) 120-130.

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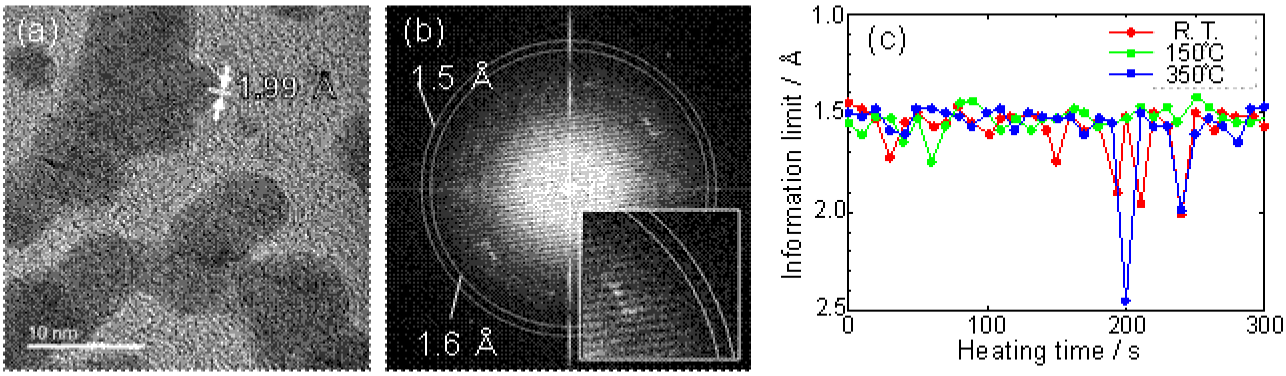

Fig. 1 AC-TEM image of Au/Pd grating (a) and Young fringe pattern(b) at 350˚C. (c) is the heating time dependence of information limit of the grating sample at room temperature, 150˚C and 350˚C.

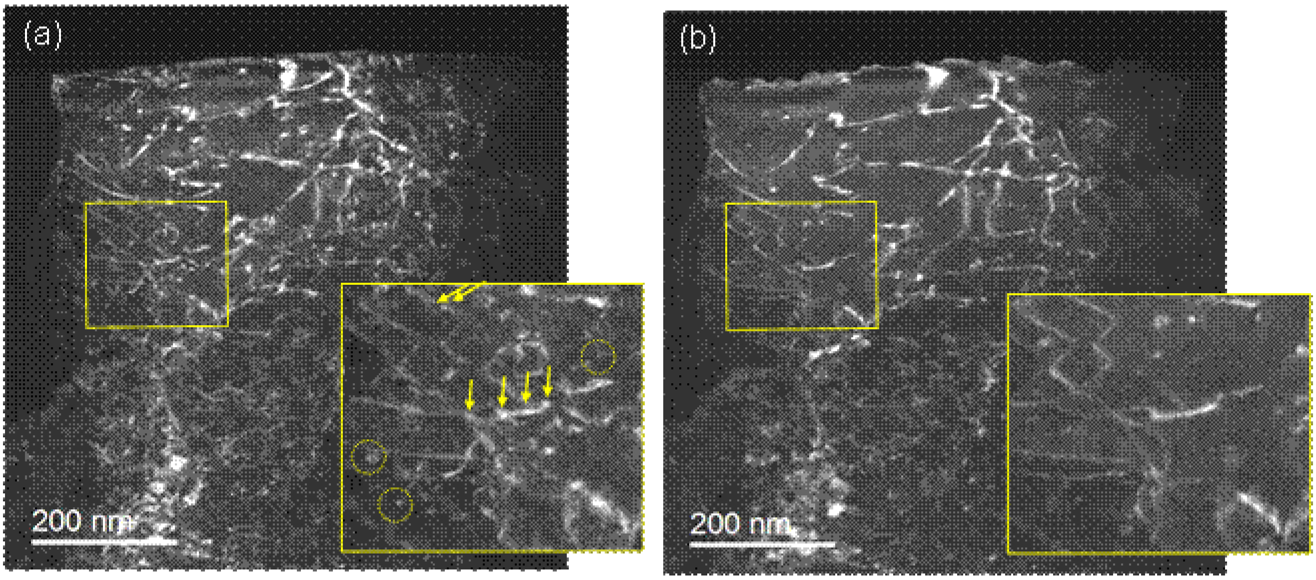

Fig. 2 Weak-beam STEM images of steel sample before annealing (a) and after 30 min (b) from the time when temperature reaches 400˚C. Insets are magnified images of the areas denoted in (a) and (b). Some of dislocations as shown in inset image of (a) are disappeared after 30 min.