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HIGH TEMPERATURE IN-SITU ELECTRON MICROSCOPY USING A DEDICATED SCANNING TRANSMISSION ELECTRON MICRSCOPE

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To understand mechanism of structural changes of materials under heat treatment, in-situ high temperature observation using analytical transmission electron microscope(TEM) is quite useful for its imaging and analytical performances[1]. One of the advantages of TEM in in-situ study is the possibility of dynamic observation and recording of materials behaviors using high resolution TV-system. Recently, we tried the same kind of observation using a dedicated scanning transmission electron microscope (STEM). The microscope is of 200kV cold field emission gun and equipped with bright field STEM, high angle annular dark field (HAADF) STEM and secondary electron microscopy (SEM) detectors. In this microscope, simultaneous observation of SEM and STEM images is possible and it allows collection of structural information not only from inside of the specimen thorough transmitted electron but also from the surface through secondary electrons This is important when three dimensional characterization is required. TV camera system for electron diffraction pattern observation is also newly developed and attached. The electron diffraction pattern is displayed near by SEM image on the standard STEM monitor simultaneously.. . Analytical systems such as energy dispersive X-ray spectrometer (EDX) and electron energy-loss spectrometer(EELS) for real time energy filtered imaging are also attached. The specimen heating holder employed in the experiment is of double tilt mechanic developed for standard size of TEM specimen(3mm diameter). Specimen tilting angles of the holder are $\pm 20^\circ$ (α) and $\pm 10^\circ$ (β). Highest heating temperature of 1500℃ is obtained at heating power of approximately10 W[2]..

Figure 1 shows external view of recently modified double tilt specimen heating holder. The frame of the heating stage and the specimen fixing threaded ring are made of tantalum. Heating temperature as a function of heating power is shown in Figure 2. Highest temperature of 1500℃ is obtained at 10W. Example of STEM observation of behavior of dislocation in Cu thin foil heated to 600℃ is shown in Figure 3. Slip of the dislocation is clearly observed

References

[1] T.Yaguchi, T.Sato, T.Kamino, Y.Kuroda, T.Hashimoto, K.Motomiya, K.Tohji and K.Kasuya, Proc. Microsc. Microanal. 8 (Suppl .2) (2002) 1152CD

[2] T.Kamino, T.Yaguchi, and K.Hidaka, Proc. 54 th Annual Meeting of Japan Society of Electron Microscopy (1995) 65

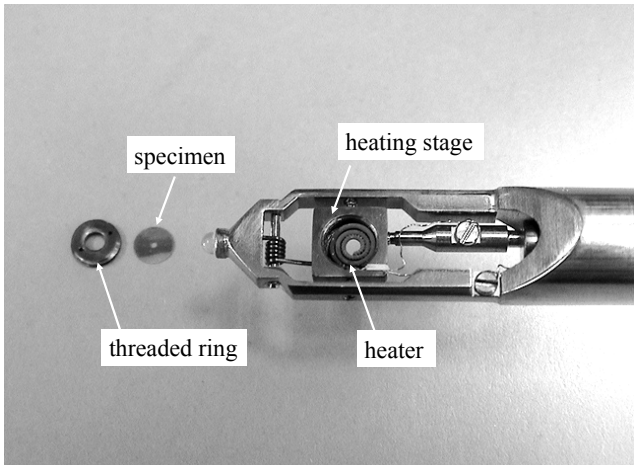

Figure 1. Double tilt specimen heating holder for standard TEM specimen(3mm dia.).

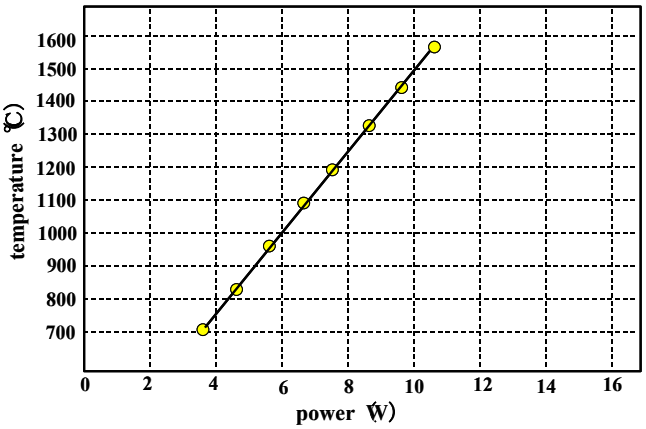

Figure 2. Heating temperature (℃) as a function of heating power (W)

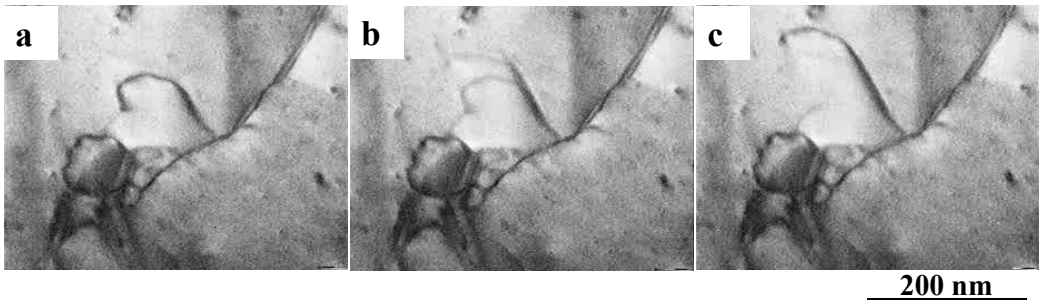

Figure 3. Bright field STEM image observation of dislocation slip in Cu thin foil at 600℃. (a:0s, b:0.5s, c:1.0s)

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