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Development of an environmental cell for the H-9500 *in-situ* TEM and its application

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For the characterization of the materials under a wide range of different ambient atmospheres and temperatures, we have developed an environmental TEM based on a 100-300 kV analytical TEM. It employs high resolution objective lens with the point-to-point resolution of 0.18 nm. The microscope column is differentially pumped using three sets of high speed turbo molecular pumps with a pumping speed of 260 l/s. The gas pressure at the specimen area can be varied from 10^{-5} Pa to 100 Pa without increasing the pressure at the electron gun area that is constantly evacuated by an ion pump with the pumping speed of 60 l/s. Related to the modification of the pumping system, the construction of the inside the column has been changed but the analytical capabilities such as EDX and EELS systems have been kept unchanged.

In order to improve the experimental capability of the environmental TEM, we are developing various types of specimen holders [1], [2] and environmental cells.

In this paper, the features of an environmental cell（Fig.1a）and its application are reported.

The developed environmental cell is a side entry type with a built-in specimen heater of a spirally shaped tungsten wire which is used as the standard heater for high temperature specimen heating in principle. All of the developed holders are possible to use in the high resolution objective lens pole-piece which has the lens gap of 4mm. Depending on the kind of gas introduced to the cell and heating temperature, we use various kinds of separating membranes in various thicknesses. The gas pressure inside the environmental cell can be continuously varied from 10^{-5}Pa to atmospheric pressure in the normally evacuated specimen chamber. A low magnification TEM image of the SiN-membrane window is shown in Fig.1b.

Fig.2 shows an example of HR-TEM image of carbon graphite heated to 600 degree Celsius under high vacuum condition using the 15nm-carbon membrane [3]. The lattice fringes of graphite(002) with the distance of 0.34nm were clearly observed. It is confirmed that the membrane was not damaged after heated up to 1000 degree Celsius.

An example of TEM image and electron diffraction pattern of Si particle obtained at various pressures is shown in Fig.3. A 200nm-thick SiN-membrane was used as the window. At a pressure of about $1x10^{4}$Pa, details in the TEM image are difficult to observe but it is still possible to observe the electron diffraction pattern.

References

[1] T.Kamino, et al.,Journal of Electron Microscopy **54(6)** (2005) 497-503

[2] T.Kamino, et al.,Journal of Electron Microscopy **55(5)** (2006) 245-252
[3] T.Kawasaki, et al, Microscopy & Microanalysis **13(2)** (2007)644-645CD

(a) (b)

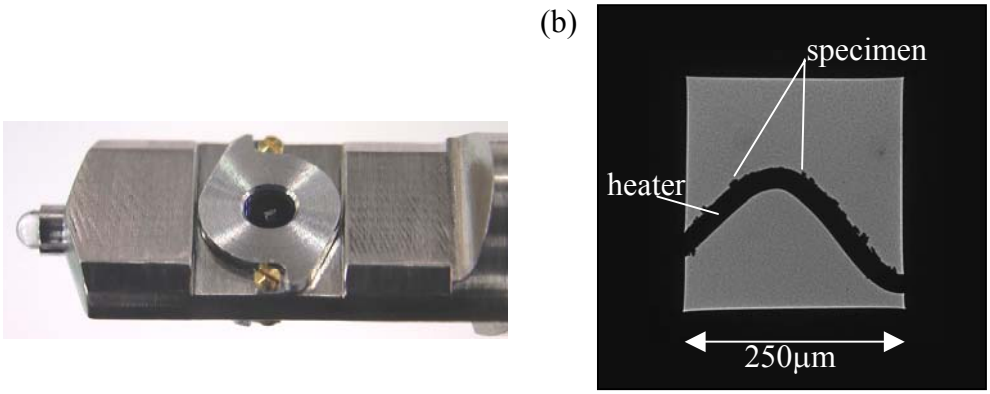


Figure 1 Developed environmental cell(a) and low magnification TEM image of the SiN-membrane window (b)

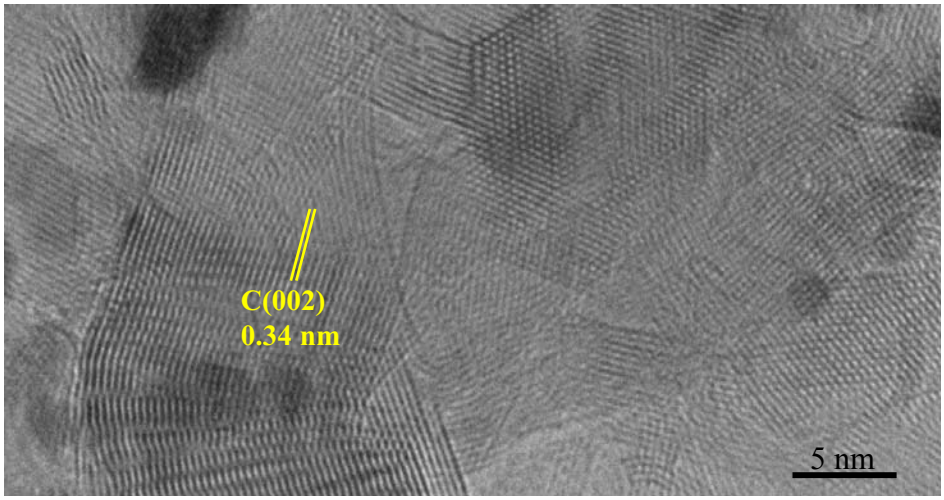
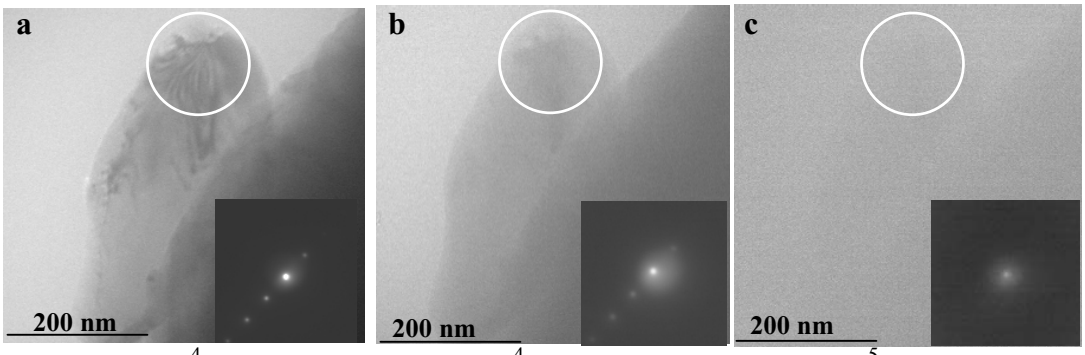


Figure 2 HR-TEM image of carbon graphite observed at 600 degree Celsius under high vacuum condition using the 15nm-carbon membrane.



5×10^{-4} Pa 1×10^{4} Pa 1×10^{5} Pa

Figure 3 TEM images and electron diffraction pattern of Si particle observed at various air pressures.

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