Development of an Environmental TEM and specimen holders for a wide range of atmospheres and temperatures

Toshie Yaguchi¹, Akira Watabe¹, Yasuhira Nagakubo¹, Kouta Ueda¹, Munetoshi Fukui¹, Takeo Kamino², and Tadahiro Kawasaki³

¹Hitachi High Technologies Corp., 882, Ichige, Hitachinaka, Ibaraki, 312-8504 Japan
²Fuel Cell Nanomaterals Center, University of Yamanashi, Takeda, Kofu, 400-8511, Japan
³Department of Engineering and Computer Science, Nagoya University, Nagoya 464-8603, Japan

The role of an environmental TEM has become more and more important to study the behavior of the nanostructural materials under controlled atmospheres and temperature. In response to the requirements, we have developed an environmental TEM based on the H-9500 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 pump with the pumping speed of 260 l/s (Fig.1). 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. Though the construction of the inside the column has been changed due to the modification of the pumping system, the analytical capabilities of both EDX and EELS systems have been kept unchanged from the original TEM.

In order to improve the experimental capability of the environmental TEM, we are developing various types of specimen holders and environmental cells. All of the developed specimen holders are possible to use in high resolution objective lens pole-piece with the lens gap of 4 mm.

In this paper, the features of a specimen heating holder with gas injector and metal evaporator (Fig.2a) and environmental cell (Fig.2b) are discussed.

In the field of catalyst, the specimen heating holder equipped with a gas injector and an evaporator can be used for both synthesis of metal oxide support and deposition of catalyst nano-particles in situ. All the procedure from the synthesis of support material to the deposition of catalyst as well as the behavior of the catalyst nano-particles on the support can be observed at near atomic resolution.

The 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. The gas pressure inside the environmental cell can be continuously varied from \(10^{-5}\) Pa to atmospheric pressure in the normally evacuated specimen chamber. Depending on the kind of gas introduced to the cell and heating temperature, various kinds of separating membrane in various thickness can be chosen. A low magnification TEM image of the SiN-membrane window is shown in Fig.2c.

Fig.3 shows an example of HR-TEM image of Si particle heated to 600 °C in the air atmosphere at the pressure of \(10^{4}\) Pa using the 15 nm-SiN membrane. The lattice fringes of Si(111) plane with the distance of 0.314 nm are clearly observed.
References

FIG 1. Differential pumping system of the H-9500 analytical environmental TEM

FIG 2. Specimen heating holder with gas injection nozzle and metal evaporator (a) developed environmental cell (b) and TEM image of the SiN-membrane window(c).

FIG 3. HR-TEM image of Si particle heated to 600 degree Celsius at a pressure of 10^4 Pa using the 15 nm-SiN membrane.