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High Resolution In Situ Heating TEM Study of Nanocrystals

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In recent years, the development of high resolution in situ transmission electron microscopy (TEM) has moved the characterization of nanomaterials from static states to dynamic states such as at elevated temperatures or under a force load. In the in situ heating TEM field, materials growth, oxidation-reduction, solid/liquid/gas reactions, and structural modifications of nanoscale catalytical materials are among the most interested topics, for which atomic resolution observation of dynamic structural processes is increasingly demanded. A group of sample heating holders have thus been developed by Hitachi High Technologies Corporation [1], which, in conjunction with a 300 kV high-resolution transmission electron microscope (H-9500) facilitates the atomic resolution in-situ heating TEM studies. A simplified differential pumping system on the H-9500 microscope also allows gas injection directly into the specimen chamber when needed [2, 3].

Fig. 1 shows a picture of the tip part of a Hitachi gas injection-heating sample holder. The gas injection is controlled by an external needle valve, and the gas is directly spread onto specimen in the specimen chamber without using a window-type cell. The temperature of the specimen can be varied from room temperature to above 1000^{o}C. A video recording system is set to record the continuous images during the whole in situ heating TEM experiments.

The two lattice images in Fig. 2 were cropped from a movie file recorded during heating a Ni thin film on a Si substrate. Growth mechanism of a nanoscale island is studied in this case.

Fig. 3 shows a metallic nanoparticle heated with injection of air into the H-9500 microscope specimen chamber. The newly grown atomic layers are seen on the surface of the particle, and the ledge flow growth mechanism left facets on the surface.

In summary, the presented in-situ heating TEM results on nanomaterials demonstrate unique information and knowledge obtained from the atomic resolution dynamic TEM studies. Such research programs may have a great impact on nanoscience and nanotechnology associated with catalysts, fuel cells, nanomaterials environmental safety, gas sensors, geochemistry, and toxicity.

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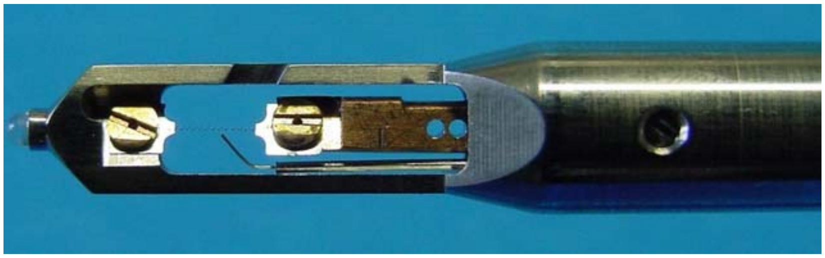

Fig. 1. Tip part of a Hitachi gas injection-heating sample holder. A heating filament and a gas nozzle can heat the sample while generating a gas environment around it.

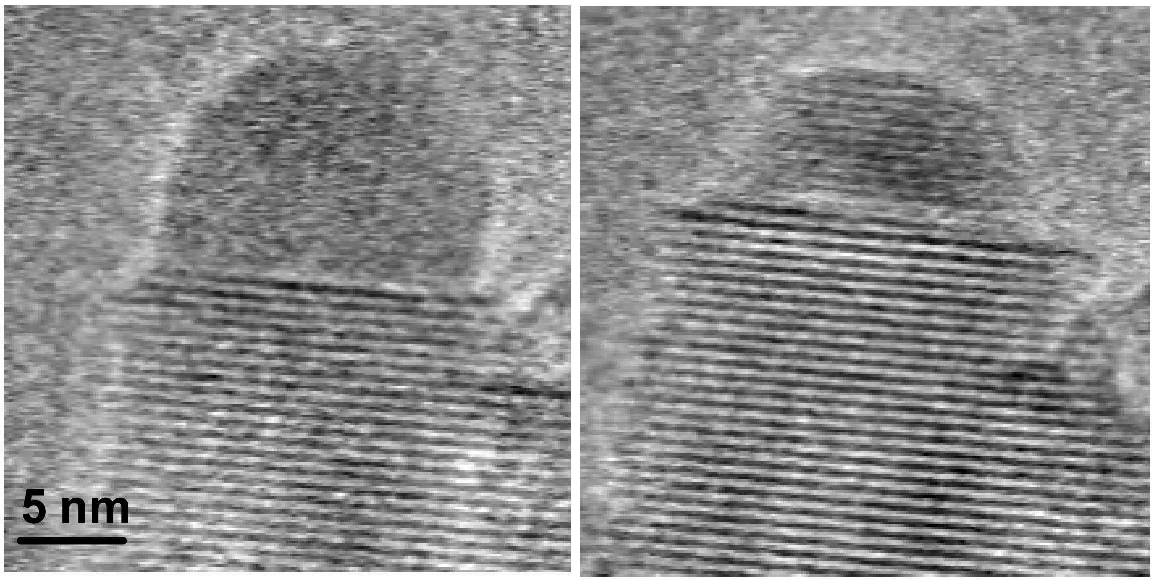

Fig. 2. In-situ heating a Ni thin film on Si substrate to 650^{o}C in an H-9500 transmission electron microscope. Growth of a nanoscale island was observed with a spiral growth mechanism.

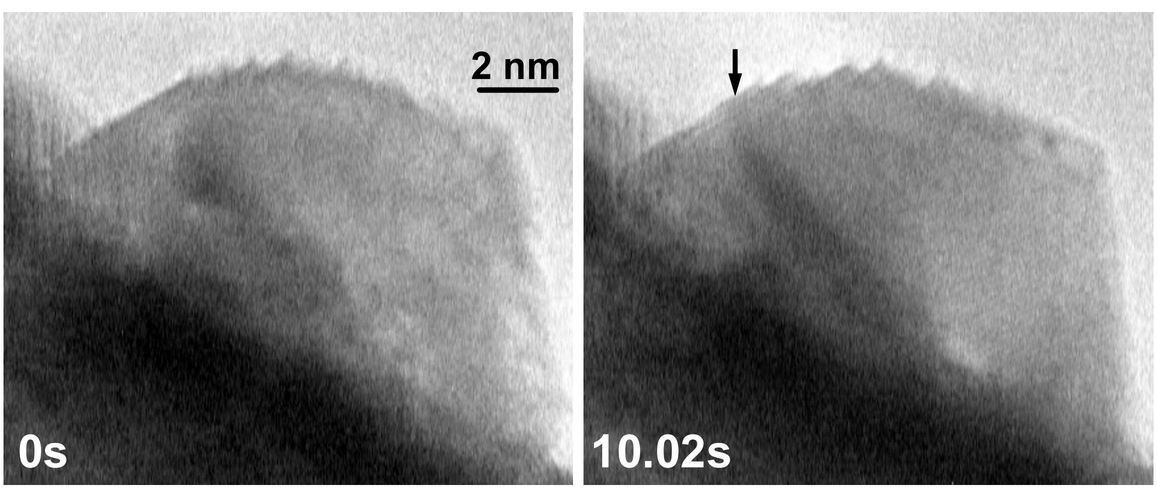

Fig. 3: A metallic nanoparticle was heated to 500^{o}C in an H-9500 transmission electron microscope while air was injected to the specimen area. The arrow indicates a new atomic layer growing on the surface.

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