

# In situ observation of structural alteration process of filmy quenched carbonaceous composite

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**Abstract.** The thermal alteration process of filmy quenched carbonaceous composite (filmy QCC) has been studied in situ by high-resolution transmission electron microscopy (HRTEM). HRTEM images of the as-prepared filmy QCC showed the typical amorphous carbon film structure. By heating above 300 °C, the structural alteration takes place. Curled graphene structure started to appear at 300 °C. Distorted onion-like structure similar to dark QCC appeared above 500 to 700 °C. The distorted onion-like structure that appears at 700 °C after heating for 30 minutes also appeared by heating at 450 °C for 2 hours.

**Keywords.** Methods: laboratory, dust, plasmas

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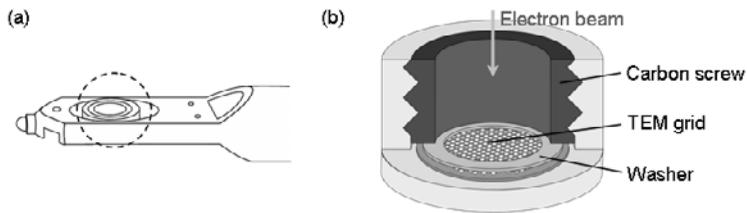
## 1. Introduction

Many types of carbon and other carbonaceous materials have been proposed to explain the 217.5 nm feature. Sakata *et al.* (1983) had proposed and analyzed a quenched carbonaceous composite (QCC) material which is condensed from a hydrocarbon plasma. A brown-black carbonaceous materials named 'dark-QCC' shows a 217 nm absorption maximum (Sakata *et al.* 1983). A yellow-brown filmy material named 'filmy QCC' is collected on a wall surrounding the plasma beam. By thermal treatment at 500-700 °C, the filmy QCC is carbonized and shows a 217 nm absorption maximum (Sakata *et al.* 1994). The dark QCC is a coagulation of carbonaceous onion-like particles as elucidated by high resolution transmission electron microscopy (HRTEM) (Wada *et al.* 1999). In the present paper, the structural alteration of filmy QCC has been examined by in situ HRTEM observation in the temperature range of 300-700 °C.

## 2. Heating of filmy QCC

The filmy QCC was deposited onto a KCl cleavage crystal surface. The KCl was dissolved in water, and the isolated sample was collected onto carbon holey film supported by a standard electron microscopic grid. The samples were heated in an electron microscope using a special specimen holder in  $6 \times 10^{-6}$  Pa. Heating was carried out by controlled the rate of temperature rise to 5 °C per minute and maintained for 30 minutes at 100 °C intervals. Figure 1 shows the schematic presentation of the furnace specimen holder.

In a previous similar study, the structural alteration was different between the periphery and the central part of the film, i. e., turbostratic graphite structure and onion-like

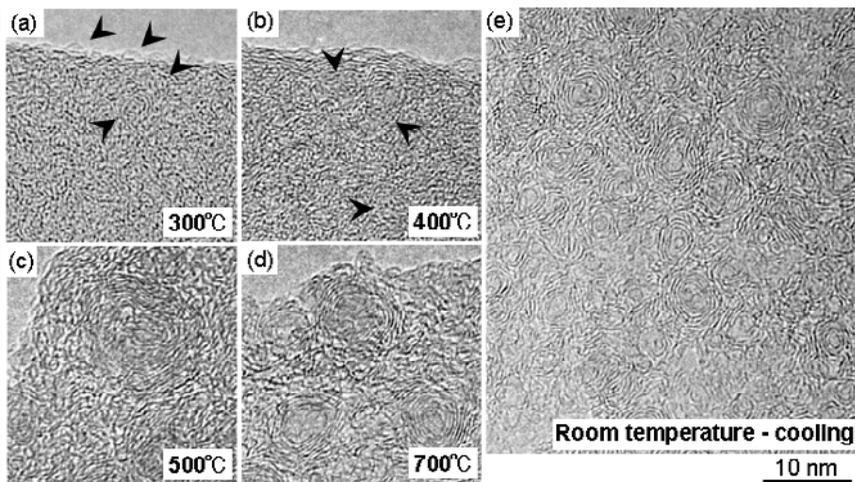


**Figure 1.** Schematic images of a heating head of the TEM holder (a). Broken line circle of the detail is shown as (b).

structure were seen, respectively (Kaito *et al.* 2003). We found in the present study that the periphery structural alteration was only seen in the strong electron irradiated region.

Figure 2 shows the filmy QCC of the regions which were heavily exposed to electron radiation. The difference between the periphery and inner parts are small. Upon heating to 300 °C, curled graphene was seen as indicated by arrows. By heating at 400 °C, the void contrast of the onion-like structure started to appear as indicated by arrows. By heating at 500 and 700 °C, onion-like spherule growth became apparent (Figure 2c and 2d). Figure 2e shows the results after heat treatment at 700 °C. The onion-like spherules are seen throughout the film. The basic structure is like that of dark QCC.

When the heating experiment was done at 450 °C for 2 hours, similar structural alteration, with the same onion-like spherules, was obtained.



**Figure 2.** In situ images of heated filmy QCC at 300 °C, 400 °C, 500 °C, and 700 °C, and at room temperature after cooling.

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