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High-speed in-situ observation of a pulse homogeneous nucleation process in vapor phase

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The gas evaporation method has a history of almost half century. Study of the products has been performed energetically mainly using a transmission electron microscope and elucidated that nanoparticles have different physical properties from that of the bulk material, have a crystal habit reflected their crystal structure as well as bulk crystals, and so forth. On the other hand, there is almost no report concerning a nucleation in a smoke in view of crystal growth. Recently, we firstly achieved an in-situ observation of a nucleation process in a smoke using a Mach-Zehnder type interferometer and clearly showed that smoke particles condense only in very high-supersaturation environment homogeneously. In case of preliminary experiment using tungsten trioxide, it condenses with a degree of supersaturation as high as ~10^6. In this process, since evaporant is continuously supplied into surrounding of an evaporation source, flow of a smoke (i.e., nucleation and growth of nanoparticles) has been simply considered as a consecutive process. However, the nucleation and growth of smoke particles should be a rapid process (~ms) due to high supersaturation. In such case, it is possible that the concentration of the evaporated vapor around the evaporation source is frequently changing. No one ever observed the detail of smoke formation process so far. There is only a report, which includes a serial photographs of a smoke and flow velocities were estimated as a function of gas pressure (Yatsuya et al. 1984). In this report, we tried to see a motion of a smoke using a high-speed camera (Keyence VW-9000) with a high-speed color camera (Keyence VW-600C) combined with a zoom lens of VH-Z20W to know the nucleation process of smoke particles.

A small vacuum chamber was newly constructed based on a new concept to do smoke experiments flexibly. The work chamber used was a stainless-steel cylinder 76 mm in diameter and 16 cm in length with two view ports of ICF70 for optical observation, a ports of ICF34 for temperature measurements using pyrometer, two electrodes and two tubes with quarter inch diameter for introduction of a thermocouple and a vacuum and gas system. Tungsten wire (99.95% in purity) with 0.3 mm in diameter and 70.0 mm in length was prepared as an evaporation source between the electrodes. After evacuating the chamber down to ~10^{-2} Pa, Ar gas (99.9999% in purity) of 3.6x10^4 Pa and oxygen (99.999% in purity) gas of 4.0x10^3 were introduced into the chamber. The pressure was monitored by a capacitance manometer (ULVAC CCMT-1000D) and a pirani/cold cathode combination gauge (Preiffer PKR 251).

When a tungsten wire is electrically heated in the gas atmosphere, a tungsten wire reacts with oxygen and tungsten oxide smoke particles are produced. The smoke follows a convection current from bottom to top generated by a hot source. The high-speed camera was set in a direction of certainly parallel to the evaporation source and observed the smoke with a rate of 4000 frames per sec. As a result, we found a fluctuation with a constant frequency with ~11 ms of concentration of a smoke. We will discuss the reason why the concentration of a smoke fluctuates at the presentation. It can be assumed that decrease of a supersaturation is the reason. But, why? There is some possibilities; temperature increase due to latent heat by condensation, depletion of oxygen around the evaporation source and decrease of the concentration due to nucleation, which eats ambient tungsten oxide molecules immediately. We intend further experiment using gold, which will elucidate the reason more clearly.

Keywords: nucleation, dust, nanoparticle, in-situ observation