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In-situ TEM Study of Sintering of Capped Silver Nanoparticles

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Nanoparticles possess unique properties that are different from that of bulk material, providing new applications in many areas of science and technology. For example, nanoparticles are currently used as catalysts for chemical reactions and for patterned metallic conductors in microelectronics. However, due to their large surface area to volume ratio, nanoparticles have a strong tendency to agglomerate and sinter during processing or usage over short time scales and at temperatures lower than bulk sintering temperatures. This in turn leads to a change in their properties.

In this work, we investigate how particle size, temperature and nanoparticle capping layer influence sintering of silver nanoparticles using a transmission electron microscope (TEM) equipped with a novel Protochips Aduro™ heating stage to carry out *in-situ* heating experiments. This heating stage enables very fast heating rates (10^6 °C/s) with an extremely low thermal drift even at high temperatures due to its low mass [1]. We use commercially available silver nanoparticles (from Nanotechnologies Inc.) that typically have a surfactant or organic capping layer on the surface to prevent agglomeration of the nanoparticles.

A sequence of *in-situ* heating TEM images showing sintering of two 15 nm silver nanoparticles at 200 °C is shown in Fig. 1. From these *in-situ* TEM images, the particle radius, neck radius and dihedral angle were measured (Fig. 2) using a procedure that has been described previously [2]. These values were subsequently used to calculate the surface diffusion coefficient. For surface diffusion, the change in neck radius (x) with time (t) for a given particle radius (r) and temperature (T) is given by the following expression [3, 4]

$$x^7 = \frac{56\Omega r^3 \gamma_s D_s \delta_s t}{KT} \quad (1)$$

where Ω = atomic volume, γ_s = surface energy, D_s = surface diffusivity, δ_s = surface diffusive width and K = Boltzmann's constant. Also, an *in*-situ TEM heating experiment was performed on two 40 nm silver nanoparticles at 400 °C and a similar analysis was done to determine the sintering parameters. From these experiments, the surface diffusion coefficient, D_s of silver nanoparticles with an organic capping layer was calculated to be in the range 1.04 - 1.55 x 10^{-21} m^2/s at 200 °C and 2.76 - 5.11 x 10^{-20} m^2/s at 400 °C. These values of surface diffusivity when compared with data obtained from bulk material at high temperatures and extrapolated to lower temperatures are significantly lower than the bulk silver estimates. This is believed to be due to the surfactant or capping layer on the nanoparticles that retards the sintering of the nanoparticles. To test this hypothesis, this data will be compared in the future to similar experiments conducted on silver nanoparticles that are free of capping layers.

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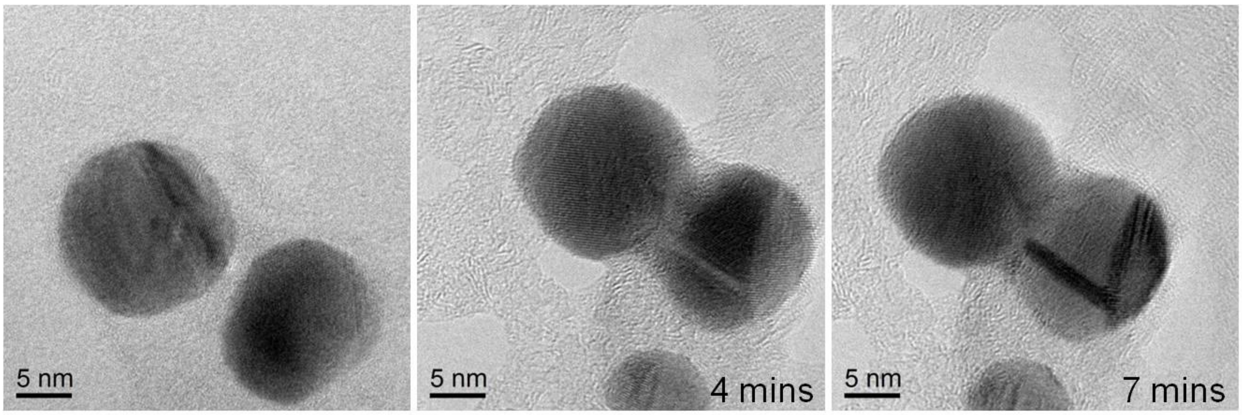

Figure 1: A sequence of in-situ TEM heating images showing sintering of silver nanoparticles at (a) RT (b) 200 °C after 4 minutes (c) 200 °C after 7 minutes

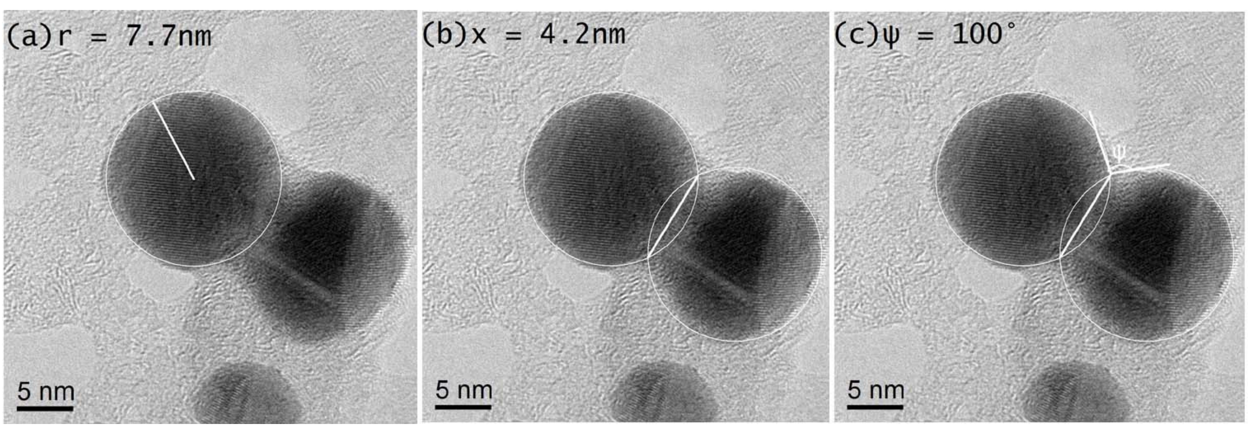

Figure 2: *In-situ* heating TEM images of silver nanoparticles from Fig. 1b showing the measurements of (a) particle radius (b) neck radius (c) dihedral angle.

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