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In-situ TEM Study of Cu and Ag Nanoparticle Interaction

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Nanoparicles (NPs) possess unique properties that are often different from that of bulk materials. For example, the melting temperature of metallic NPs is typically lower in comparison to bulk materials presumably due to their highly reactive surface properties. This has led to new applications in many areas of science and technology such as new joining techniques that bond at lower processing temperatures but remain viable at higher temperatures [1]. Controlling metallic NP interactions plays a vital role in the joining applications. Several studies that utilize *in-situ* transmission electron microcopy (TEM) to study interaction among same kind of NPs at the elevated temperature have been reported [2, 3]. However, no work has been reported on study of NP interaction among different types by *in-situ* TEM.

In this work, we report our effort to study Cu and Ag NP interaction by *in-situ* TEM in order to develop a fundamental understanding of the reaction processes and associated atomic mechanisms. A novel Protochips Aduro^{TM} *in-situ* heating stage was used for the heating experiments. An aberration-corrected scanning TEM (AC-STEM) (FEI Titan^{TM} G2 80-200) equipped with SuperX energy-dispersive x-ray spectroscopy (EDS) i.e. quadruple windowless silicon drift EDS detectors, were used to follow the compositional change as a result of the interactions. The atomic-scale molecular dynamic (MD) modeling was also used to understand the reaction mechanism.

Figure 1a shows the Cu-Ag NPs deposited on a carbon film before the heating experiment. The Cu NPs and Ag NPs have size of about 15 nm and 6 nm, respectively, before the heating. We have found that thermal heating at a temperature as low as 300^{o}C can introduce significant interaction between Cu and Ag NPs and the interaction can be significantly affected by electron beam. Figure 1b show a TEM micrograph from an area without electron beam pre-exposure, where the thermal annealing by a heat pulse of 60s at 300^{o}C has led to change in particle size and morphology, and formation of Cu/Ag interface in some particles. The areas which were exposured to electron beam during the same heat pulse, on the other hand, show no visible changes (Fig.1c). The formation of graphitic shell on NPs under electron beam is likely the cause of different observation [4]. The effect, however, makes it difficult to observe initial stage of the interaction, critical for the alloy formation.

The resulting microstructure of the interaction at 300^{o}C was studied by STEM-EDS spectral imaging. Figure 2 shows the result of EDS mapping. The Cu/Ag interface show a shape transition from Cu to Ag, as expected due to limited mutual solubility. However, the Cu atoms appear to be around the Ag particles (Fig.2d), forming a Cu-shell and Ag-core structure. Further work is needed to understand the phenomena [5].

References:

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[3] M.A. Asoro, *et al.*, Microsc. Microanal. 17, 486 (2011).
[4] E. Sutter *et al.*, Nano Lett. 5, 2092 (2005).

[5] Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the US Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000.

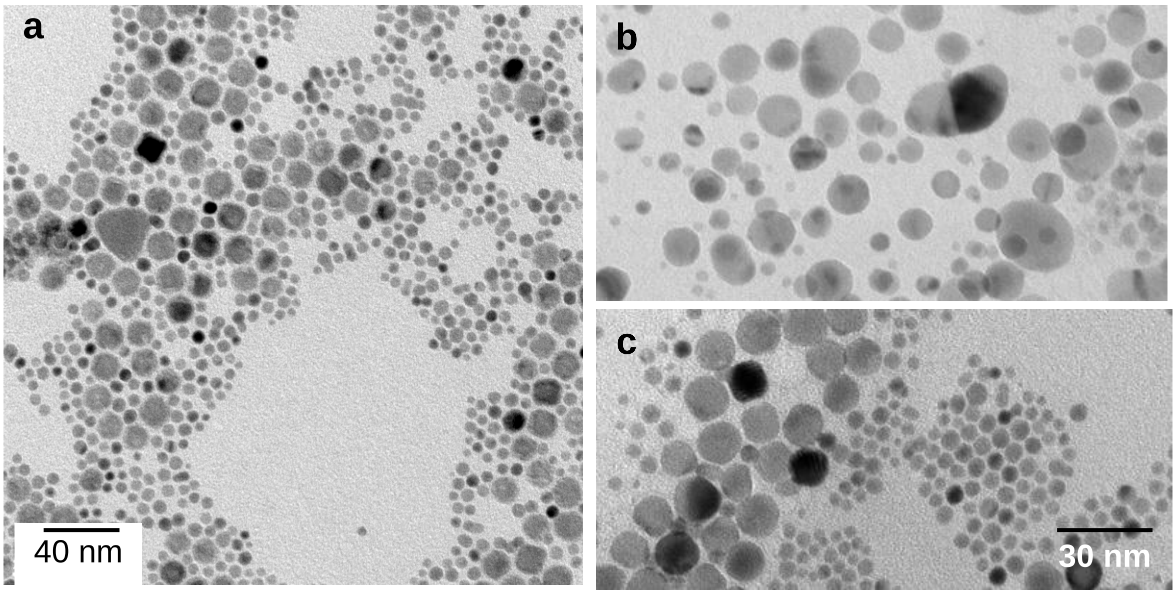

Figure 1: TEM micrographs showing (a) mixture of Ag-Cu NPs deposited on carbon film before heating; (b) an area after 60 second, 300^{o}C heating without electron beam exposure during the heating; and (c) an area after 60 second, 300^{o}C heating but with electron beam on during the heating.

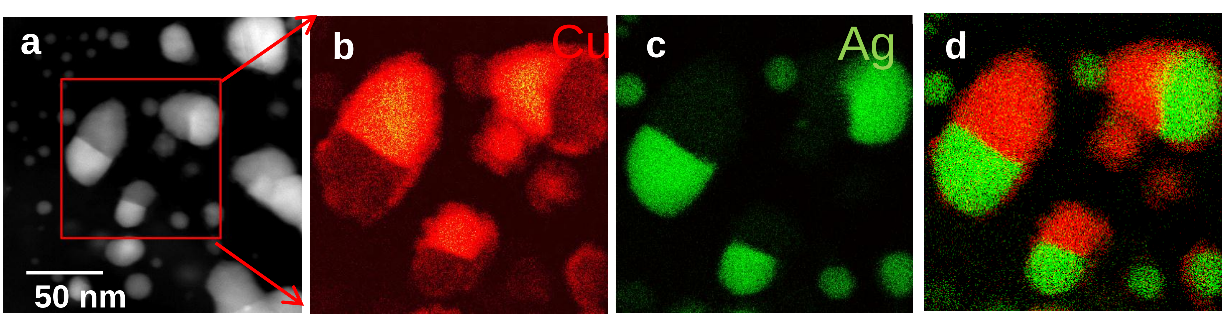

Figure 2: (a) HAADF imaging showing an area used for EDS compositional map; (b) EDS map of Cu (K_{α}) and (c) EDS map of Ag (L_{α} and L_{β}), and (d) composite map of Cu and Ag.