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Time-Resolved EDS Studies with Rapid Heating and FEG-SEM

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Conventional heating stages for electron microscopy use relatively large heating elements and their slow response times and substantial drift often precludes their use for time-resolved, in situ heating studies. In the Aduro System from Protochips, standard heating elements are replaced with a microfabricated, semiconductor-based heating device, and this approach provides extremely accurate and well-controlled sample heating (up to 1,000,000°C per second and up to temperatures of 1200°C) with virtually no drift. Because of the extremely fast response times, novel, in situ experiments in the electron microscope are now possible. Using this technology and two JEOL FEG-SEMs (JSM-7600F and JSM-7001F TTLS), time-resolved EDS spectra have been collected from Cu/Ag specimens heated for intervals only seconds in duration from 800˚C to 1200˚C.

A micron-scale Cu/Ag specimen was applied via solvent dispersion onto a heating device for the Aduro system and loaded into a JEOL FEG-SEM. The specimen was then imaged at room temperature and EDS spectra were collected. The specimen was then subjected to multiple heat cycles and returned to room temperature for additional SEM imaging and EDS spectra collection. All images and EDS spectra were collected at 6500x and with 300 second acquisition time. Figures 1-5 show the SEM images Cu spectra and Ag spectra at room temp and then after the specimen was heated in sequence to 800˚C for 1 minute, 900˚C for a total of 20 seconds, 950˚C for 10 seconds and 1200˚C for 20 seconds.

We have observed that upon heating the outer shell of Ag is slowly undergoing two processes - evaporation as well as creation of CuAg intermetallic compound. This suggests behavior that follows the basic bulk phase diagram, especially in the regime where the percentage of Ag is relatively small. With increasing temperature the particles coalesce and coarsen eventually forming a large ball, essentially following Ostwald ripening mechanism, at 1200˚C. When the temperature is lowered to 500˚C, the ball is quenched nucleating individual facets. Cycling the temperature further reforms these facets.

We have shown that dynamic heating and quenching experiments can be performed in FEG-SEM using the Aduro system. Such experiments can be invaluable for observation of the behavior of nanoparticulate systems.

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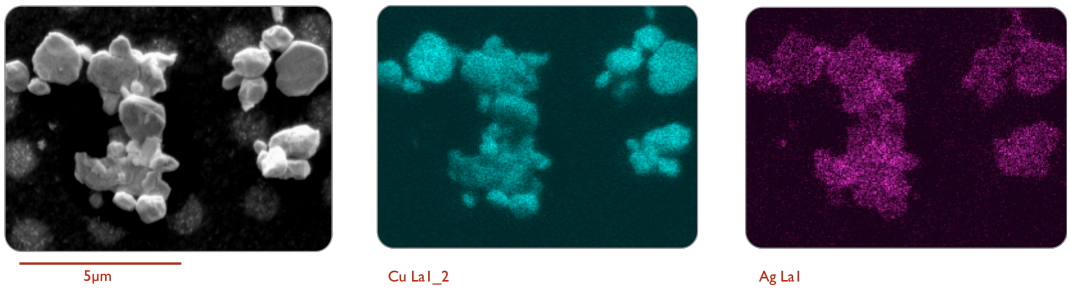

Figure 1: Room temperature SEM image and Cu / Ag EDS data

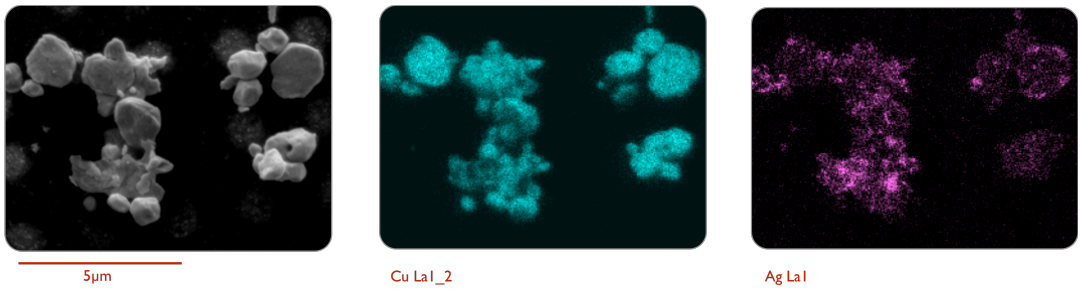

Figure 2: SEM image and Cu / Ag EDS data of sample after heating to 800˚C for 1 minute

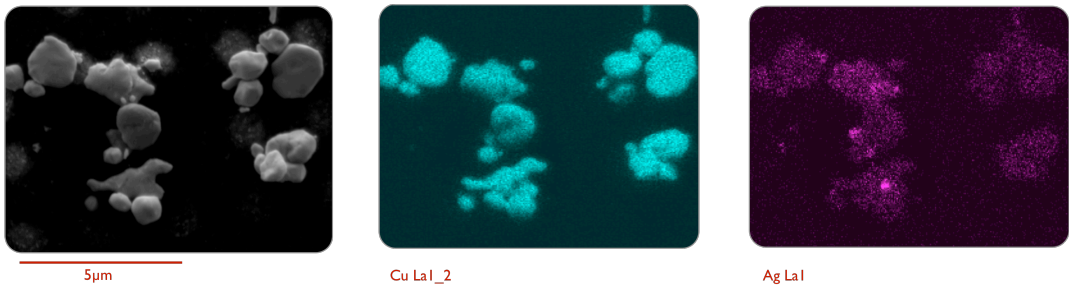

Figure 3: SEM image and Cu / Ag EDS data of sample after heating to 900˚C for a total of 20 seconds

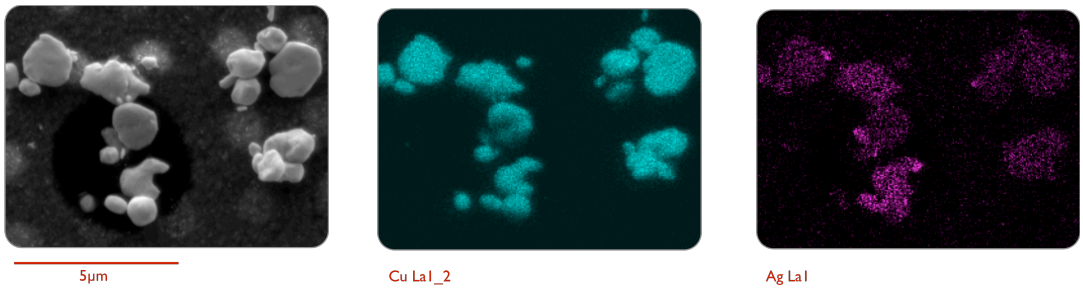

Figure 4: SEM image and Cu / Ag EDS data of sample after heating to 950˚C for 10 seconds

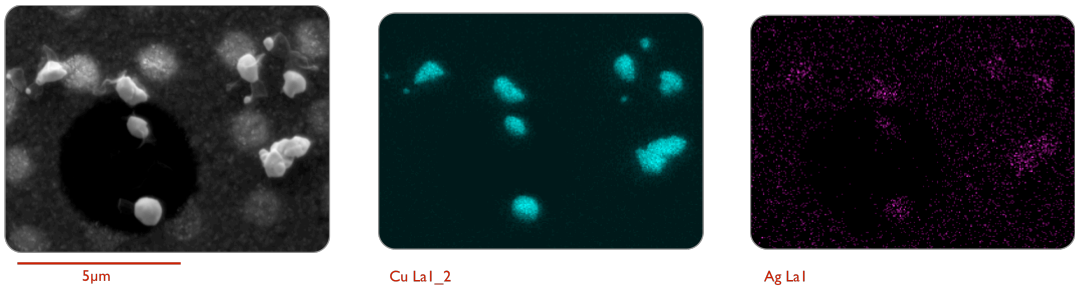

Figure 5: SEM image and Cu / Ag EDS data of sample after heating to 1200˚C for 20 seconds

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