

Radiation Synthesis and XRD Study of Binary and Ternary Alloyed Nanoparticles

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Numerous methods have been developed for the synthesis of alloyed Au-Ag nanoparticles in aqueous solutions. Here we report a new synchrotron based X-ray irradiation to synthesize binary and ternary nanoparticles in aqueous solution. The X-ray source was the high brightness superconducting wavelength shifter (SWLS) at the BL01A beam line [1, 2]. We used a white beam with no optical elements except one set of beryllium and Kapton windows. A slit system produced a transversal beam cross section of $13 \times 9 \text{ nm}^2$. The calculated X-ray photon flux absorbed by the precursor solutions was centered at $\approx 12.5 \text{ keV}$ and ranged in photon energies from 6.5–30 keV. The delivered dose rate was estimated to be $\sim 4.7 \times 10^5 \text{ Gy/sec}$ with an uncertainty of 2–6% by measuring methylene blue bleaching [3].

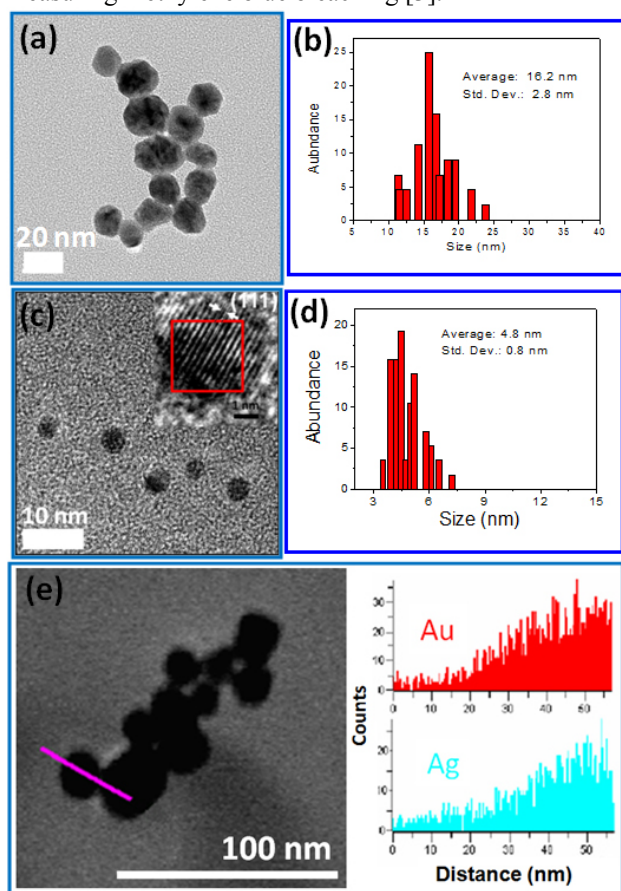


Fig. 1: TEM micrograph and size distribution of AuAg alloyed nanoparticles (a-b) and PEG-AuAg nanoparticles (c-d) with the elemental line profile of Au-Ag nanoparticles by EDX analysis (e).

We investigated the crystal structural analysis of colloidal samples (binary AgAu, AgCd, AuCd, AuZn and

AgZn and ternary alloyed nanoparticles) based on high photon energy synchrotron XRD measurement at BL12B2 (Spring 8). The beam size was 150 micrometer and the photon energy was tuned to 22.5421 keV. The crystal sizes were estimated from the line broadening of reflection peak (111) using Scherrer's equation. The measured breadths were corrected by excluding the FWHM of LaB₆ standard as the instrumental broadening.

As shown in Fig. 2, the reflection peaks of the AuAg alloyed particles can be indexed as AuAg alloy with a molar ratio of 1:1 demonstrating a typical FCC crystal structure. For the Cd- and Zn- containing alloyed nanoparticles, however, the reflection patterns didn't match with the responding Heusler phases and resembled well with that of binary AuAg alloyed nanoparticles indicating that the ternary phase was not formed. Combining the data of both XRD and UV-VIS spectra indicated that the phase composition of the "AuAgCd₂" and "AuAgZn₂" are expected to be a mixture of Au+AuAg and Ag+AuAg, respectively. The crystal sizes were estimated from the line broadening of reflection peak (111) using Scherrer's equation and thus derived the crystal size of 2.2 nm, 2.2 nm and 3.9 nm for AuAg, AuAgCd₂ and AuAgZn₂ nanoparticles, respectively. As judged by the XRD data, the attempt for ternary AgAu based Heusler alloy is not successful and the obtained particles are mainly composed of mixtures of either Au+AuAg (Au-Ag-Cd) or Ag+AuAg (Au-Ag-Zn).

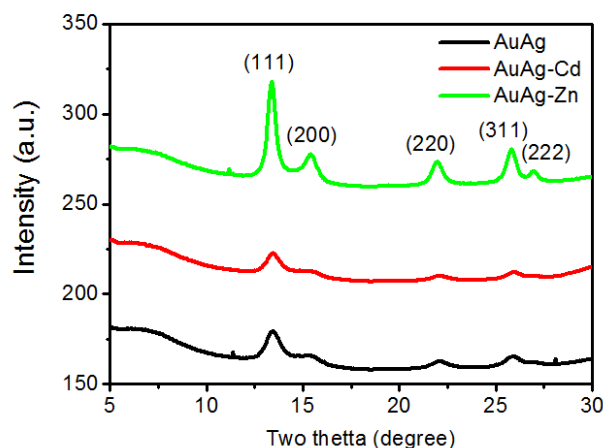


Fig. 2: XRD spectra of AuAg, AuAgCd₂ and AuAgZn₂.

- [1] S. Baik *et al.*, Rev. Sci. Instr. **75**, 4355 (2004).
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- [3] C. J. Liu *et al.*, J. Synchrotron Radiation **16**, 395 (2009).