

Preparation of Ultrafine Au-Pb Particles by Gas-evaporation Technique

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Abstract

Ultrafine Au-Pb particles prepared by two methods, (1) simultaneous evaporation of Au and Pb in inert gas and (2) subsequent vapor condensation of Pb in a differentially evacuated tube onto flying Au nanoparticles prepared by gas-evaporation technique, were observed by electron microscopy. In the method (1), the particles that grew at the region where the two smoke masses converged, consisted of alloy phases. In the method (2), the particles consisted of two or three phases of Au, Au₂Pb, AuPb₂ and Pb phases in turn from the inner part, Pb-rich particles being composed of only two phases of AuPb₂ and Pb.

Keywords : ultrafine particles, Au-Pb alloy, gas-evaporation technique, electron microscopy, crystal growth

1. Introduction

Recently, in order to apply nanoparticles to new industrial materials and to examine their unique properties, various kinds of alloy and/or composite nanoparticles have been produced by a variety of preparation methods such as gas-evaporation¹⁾, arc-plasma, chemical reaction and mechanical alloying. The aim of the present study is to examine the growth of Au-Pb alloy and/or composite nanoparticles prepared by two methods; (1) simultaneous evaporation of Au and Pb in inert gas and (2) subsequent vapor condensation of Pb in a differentially evacuated and heated-up quartz tube onto flying Au nanoparticles prepared by gas-evaporation technique.

2. Experimental and Results

2.1 Au-Pb particles prepared by method (1)

Figure 1 shows smoke formed by evaporating Au and Pb metal from two evaporation sources (6x80 x0.1 mm) at each temperature $T_{Au} = 1800\text{ }^{\circ}\text{C}$ and $T_{Pb} = 980\text{ }^{\circ}\text{C}$, separated by 14mm in height for I and by 20 mm in the side for II, respectively, in an argon atmosphere of pressure $P_{Ar} = 2.6\text{ kPa}$. Figure 2 shows electron micrographs of particles which were prepared by the method (1)-I and observed by electron microscopy, where those grown at the positions indicated by (a), (b) and (c) in Fig. 1 were Au containing a small amount of Pb, Pb containing a small amount of PbO and the nearly same quantity of Pb and PbO, respectively. Au-Pb alloy phases were seldom observed for the particles.

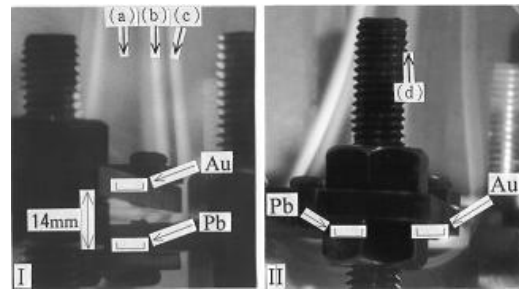


Fig. 1. Smoke formed by evaporating Au and Pb metal from two evaporation sources.

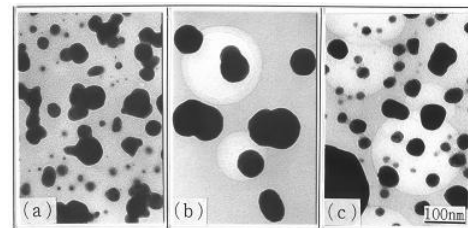


Fig. 2. Electron micrographs of particles grown at the positions indicated by (a), (b) and (c) in Fig. 1.

On the other hand, alloy phase of AuPb₂ was observed for particles grown at the position (d) of smoke, where the two smoke masses converged, formed by the method (1)-II.

2.2 Au-Pb particles prepared by method (2)

Figure 3 shows a schematic illustration of experimental equipment used for the preparation of ultrafine Au-Pb particles by subsequent vapor condensation of Pb onto flying Au nanoparticles produced in advance by gas-evaporation technique.²⁾

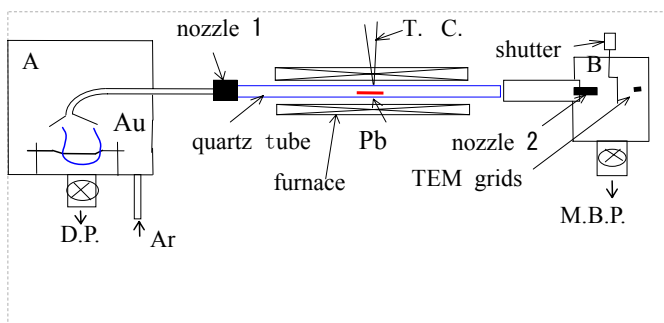


Fig. 3. A schematic illustration of experimental equipment used for the method (2).

Smoke consisting of Au nanoparticles is produced in chamber A by gas-evaporation technique and the smoke is introduced into a differentially evacuated quartz tube with the argon flow, and there, Pb vapor, which is vaporized at a furnace temperature T_F (700-730°C), condenses onto the flying Au nanoparticles. The specimen for the observation using a transmission electron microscope (TEM) was collected directly onto TEM microgrids in chamber B.

Figure 4 shows an electron micrograph (a) and the corresponding electron diffraction pattern (b) of particles, which were prepared under the conditions of $P_{Ar} = 2.6$ kPa,

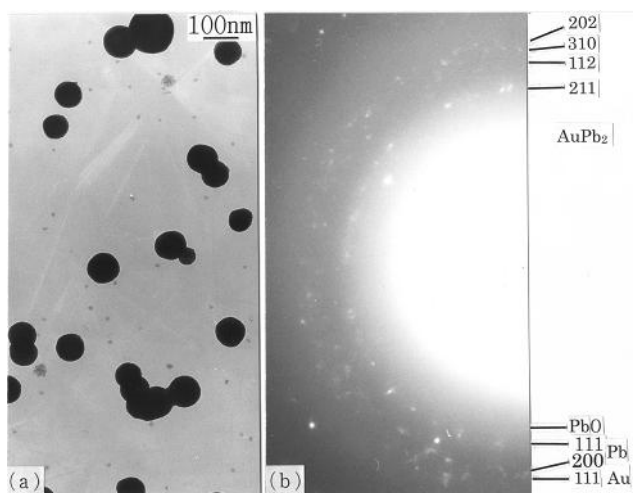


Fig. 4. Particles prepared by the method (2), showing the presence of three phases of Au, Pb and AuPb₂.

$T_{Au} = 1700^\circ\text{C}$ and $T_F = 725^\circ\text{C}$, showing the presence of three phases of Au, Pb and AuPb₂.

Figure 5 shows electron micrographs of a Au-Pb particle prepared under the same conditions as the particles shown in Fig. 4. The images (c) and (d) were taken for the particle tilted by 42° against (a). The dark field images (b) and (d) were taken with 202 and 332 reflections of AuPb₂, respectively. Judging from the particle diameter of bright and dark field images and the result obtained in Fig. 4, it is inferred that the particle consists of three shells of Au, AuPb₂ and Pb in turn from the inner part.

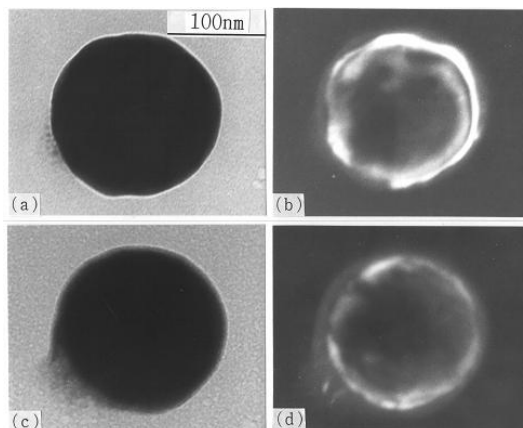


Fig. 5. Particle consisting of three shells of Au, AuPb₂ and Pb in turn from the inner part.

In case the evaporation amount of Pb is more than or less than that of the particles grown in Fig. 4, particles were Pb-rich or Au-rich, respectively; that is, the particles consisted of two phases of Pb and AuPb₂ in the former, and Au and Au₂Pb in the latter.

3. Summary

Ultrafine Au-Pb particles prepared by two methods of gas-evaporation technique. In case of the simultaneous evaporation of Au and Pb, alloy particles grew at the region where the two smoke masses converged. In case of the subsequent condensation of Pb vapor onto flying Au nanoparticles, the composition of the particles differed reflecting the evaporation amount of Pb.

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4. References

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