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Letter

Ga-In liquid metal nanoparticles prepared by physical vapor deposition

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ABSTRACT

Controlled synthesis and appropriate characterization of nanoscale particles of gallium-based liquid metals are critical to fulfilling their broad range of applications in the field of flexible, stretchable, and printable micro-/nanoelectronics. Herein, we report a new way to synthesize surfactant-free gallium-indium nanoparticles with controlled particle size on a variety of substrates through a facile physical vapor deposition method. It was found that with prolonged deposition time the liquid metal nanoparticles gradually grew from near-monodispersed small particles with a diameter of ~25 nm to bimodal distributed particles. A nucleation, growth, ripening and merging process was proposed to explain the observed evolution of particle size. Atomic force microscopy measurement indicates that the fabricated liquid metal nanoparticles demonstrate elastic deformation with a certain range of loads and the scanned particle size is dependent on the applied loads. We further investigated the gradual breaking process of the core-shell structured liquid metal nanoparticles, which was evidenced by multiple kinks on the force-separation curve. This work presents a new bottom-up approach to prepare nanoscale liquid metal particles and demonstrates that atomic force microscopy is a suitable technique to characterize the synthesized liquid metal nanoparticles.

1. Introduction

As a special form of metals, liquid metals have combined the softness and flowability of liquids, and the excellent electrical and thermal conductivity of metals. In particular, gallium-based (Ga-based) liquid metals have received intensive investigation due to the advantageous features such as low toxicity, low viscosity, and tunable melting temperature [1–3]. They have been used as conductors for stretchable and wearable electronics [4–7], self-healing and reconfigurable circuits [8,9], deformable antennas [10], and functional microfluidic devices [11].

Compared to conventional bulk liquid metals, microscale and nanoscale liquid metal particles facilitate the fabrication of micro/nanoelectronic devices through facile printing technology [12,13] and expand the application into other fields such as chemical catalytic reactions [14], nanoplasmonics [15], and thermal management of microelectronics [16]. So far, liquid metal microparticles or nanoparticles have been generally prepared by top-down methods. Methods such as mechanical fracturing [17], sonication [18–20], emulsion shearing [21], template molding [22], and flow-focusing [23–25] have been employed to break down Ga-based bulk liquid metal particles into small particles. Most frequently, these approaches can only generate micrometer-sized particles. Although nanoparticles have also been obtained with long-time high-power ultrasonication and high-speed shearing techniques, the sizes of the obtained particles are often polydispersed. Additionally, it needs the addition of surfactants such as polyvinylpyrrolidone, polyvinylalcohol, trifluoroacetic acid or alkyl thiols to stabilize the particles. These organic coatings, however, may affect the electrical or thermal properties of the pristine liquid metal particles and limit their associated applications.

Appropriate characterization of liquid metal particles is of great importance for analyzing their properties and exploring potential applications. To date, scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray scattering, energy dispersive spectroscopy (EDS), X-ray photoelectron spectroscopy (XPS), low-energy ion-scattering spectroscopy (LEIS) have been utilized to characterize the liquid metal particles [26,27]. While these tools can provide rich information on particle size, chemical composition of the liquid metal particles, obtaining more surface-related properties is challenging even though they played important roles in governing the wetting and flowing behavior, mechanical and electrical properties of the liquid metal particles [26–28]. By comparison, atomic force microscopy

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(AFM) is a simple but effective tool that can offer unique information related with the surface properties of the liquid metal particles. However, characterization of liquid metal nanoparticles with AFM has been rarely reported so far probably due to the technical difficulty in probing the soft liquid particles [13].

In this work, we report the use of physical vapor deposition (PVD) technique as a new bottom-up method to fabricate gallium-indium (Ga-In) liquid metal nanoparticles. By varying the deposition duration, we investigated the growth kinetics of the Ga-In liquid metal particles and proposed the corresponding mechanism to explain the observed particle size evolution. By carrying out AFM measurement under the quantitative nano-mechanical (QNM) mode, we analyzed the nanomechanical response of the Ga-In liquid metal nanoparticles. Finally, we studied the breaking behavior of a single particle with a diameter of ~300 nm by continuously increasing the load on the AFM tip, which indicated an inner liquid core and an outer oxide shell structure for the Ga-In liquid metal nanoparticles synthesized by the PVD process.

2. Experimental

2.1. Fabrication of Ga-In liquid metal nanoparticle

Pure gallium and indium were purchased from Sinopharm Group Co., Ltd., China. Ga-In liquid metal nanoparticles were prepared by first obtaining the eutectic gallium-indium (EGaIn) alloys by melting 74.5 wt% gallium and 25.5 wt% indium at 120 ℃, then depositing the alloy on to a substrate through a controlled PVD technique. In a typical experiment, 5 g of EGaIn alloys were placed within a tungsten boat that sits at the bottom of the chamber within the vacuum coater (JSD400, Anhui Jiashuo Vacuum technology CO., LTD, China). The vacuum of the chamber was kept below 1.0×10^{-3} Pa. During the deposition process, a constant deposition rate of 0.1 nm/s was used. The thickness of the deposited film was monitored by a film thickness gauge.

2.2. Characterization of Ga-In liquid metal nanoparticle

The deposited samples were characterized by both SEM and AFM. SEM (Nova Nano SEM 230 FEI) that is equipped with an energy dispersive spectrometer (Oxford AZtec X-max 80 T) was used to map the morphology and the composition of the liquid metal particles. All the AFM measurements including morphology images and force curves were performed using the Dimension Icon system (Bruker) at room temperature and under stable ambient humidity. Considering that the fragile oxide layer of the nanoparticle could be easily scratched by the AFM tip [18], here we utilized a mode named Peak Force QNM that can minimize the load [29,30]. The scanning size is ranging from 10 μm × 10–1 μm × 1 μm with a resolution of 256 × 256 pixels. The force-separation curves were obtained with the point and shoot module in the AFM analysis software [31]. All the height images were filtered by the first order plane fit. And all the raw force curves were converted into force-separation curves with the off-line data analysis software. The Si AFM tip and the nitride cantilever (SNL-10, with a tip radius of 10 nm) were treated with a plasma cleaner (Harrick, PDC-32 G) for 2 min before use. Prior to each scanning the deflection sensitivity of the cantilever was calibrated on the sapphire substrate, and the spring constant was corrected by the thermal tune method.

3. Results and discussion

3.1. Fabrication of Ga-In liquid metal nanoparticles

Fig. 1a shows the schematic for the synthesis of Ga-In liquid metal nanoparticles by PVD technique, which was carried out within a vertical vacuum chamber. The EGaIn was placed within a tungsten boat to provide gallium and indium source. Under resistive heating, the EGaIn alloys are vaporized into gallium and indium atoms, diffusing towards and condensing on the cold target substrate, which is 25 cm above the heater. Fig. 1b shows the photographs of a bulk EGaIn alloy particle on glass substrate and the deposited Ga-In nanoparticles on different substrates. It was found that the Ga-In nanoparticles could be homogeneously deposited on three kinds of substrates including silicon, glass and mica, which could enable the widespread application of the PVD approach for preparing liquid metal nanoparticles.

The Ga-In nanoparticle sample that was deposited on Si wafer (deposition time: 25 min) was characterized with SEM. As shown in Fig. 1c, the Ga-In nanoparticles were ball-like and homogeneously

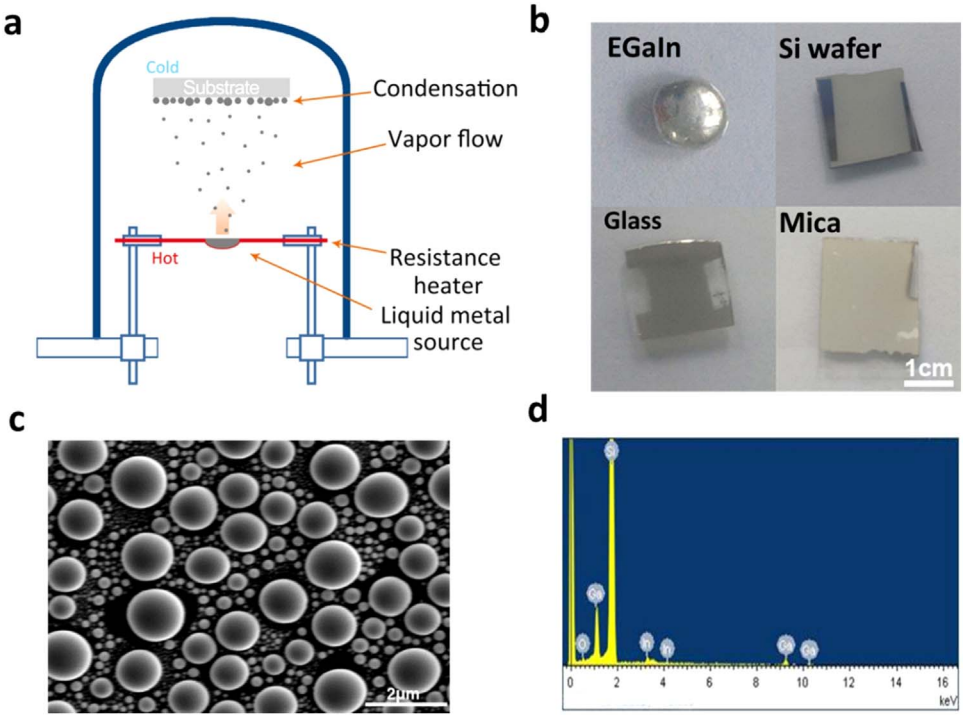

Fig. 1. (a) A schematic for preparation of Ga-In liquid metal nanoparticles through physical vapor deposition technique; (b) Photographs of EGaIn and liquid metal nanoparticles coated on Si, glass and mica substrates. (c) An SEM image of Ga-In nanoparticles deposited on Si substrate. (d) An EDS spectrum of the Ga-In nanoparticles.

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