



Template synthesis of low-density gold foams: Density, microstructure and compressive strength

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ABSTRACT

High Z metallic foams with low density are required in high energy-density physical experiments. In this study, cylindrical Au foams with relative density as low as 5.6% were synthesized from a templating approach. Microspherical polystyrene (PS) was employed as the sacrificial template and aqueous solution of HAuCl₄ as gold source. Au nanoparticles were successfully coated onto the surface of PS template from a two-step deposition process. Cylindrical PS/Au monoliths were formed by filter-casting of the Au coated PS beads. PS template was completely removed and self-supported Au foams were produced after drying, demolding and calcination. The monolithic Au foams demonstrate density as low as 1.08 g/cm³ and compressive strength as high as 3.2 Mpa.

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1. Introduction

Low-density metallic foams, which represent monolithic metallic materials with porosity higher than 90%, are of great interests as they combine the property characteristics of metals and porous materials [1]. The metallic nature leads to high thermal and electrical conductivity while the porous structure contributes high porosity, high surface area and light weight. This combination of metallic nature and porous structure makes metallic foams promising in applications of filtration, separation, thermal engineering, electrode and shielding materials. In the field of metallic foams, noble metallic foams are sought-after materials as they exhibit high sensitivity in electrical, optical, catalytic and sensor properties, which can find applications in supercapacitors, plasmonic, catalytic, hydrogen storage, sensors and lightweight components [2–6]. One significant motivation for the development of noble metallic foams is the requirement in high energy-density laser experiments. Noble metals or alloys, which usually hold high atomic number, high density and high radiation opacity (high Z), are mostly utilized as the hohlraum walls in inertial confined fusion (ICF) to reduce energy dissipation and elevate energy

transfer efficiency [7]. Compared with bulky high Z metallic walls, hohlraum walls made of low-density foamed counterparts demonstrate much lower X-ray energy loss and higher radiation temperature from theoretical and experimental calculations [8,9]. As pure Au is the mostly utilized high Z metal, Au foams with desirable density must be a desirable candidate of hohlraum wall in ICF facilities.

Several well-established techniques, such as dealloying [10–12], templating [13–15] and laser etching [16], have been created to prepare thin films of nanoporous metals. However, the approach to synthesize pure high Z metallic foams with low density and monolithic structure is limited [1]. One seemingly straightforward route, which was developed by Lawrence Livermore National Laboratory [3,17], has combined the dealloying and templating methods to synthesize hierarchically porous Au monoliths with ultralow density of 0.28 g/cm³. Other approaches, such as sol-gel assembly of prefabricated nanoparticles [18], pyrolysis of metal salt/dextran pastes [19], elimination of pore forming agents [20,21] and combustion synthesis [22], have also been developed to synthesize highly porous metallic foams. In ICF experiments, millimeter sized high Z metallic foams with definite density and shape are required as hohlraum wall targets. Thus, the as-synthesized metallic foams must be clamped and machined to accommodate the assembling of hohlraum construction, which demands a degree of mechanical strength for the prepared foams. This requirement causes another challenge for the synthesis of high Z metallic foams with low density and monolithic structure.

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In this study, we demonstrate a bottom-up route of templating and filter-casting to synthesize low-density gold foams with cylindrical shape and a degree of mechanical strength. Polystyrene (PS) microspheres and HAuCl_4 were adopted as the sacrificial template and Au source. Atomic-level Au nanoparticles were synthesized and introduced as the reaction spots of seeding and deposition. After deposition of Au nanoparticles onto the PS template, self-supported pure Au foams were prepared by filter-casting forming of PS/Au microspheres and heat-treatment of the preformed monolithic samples. Density of the as-synthesized Au foams was analyzed by adjusting the addition volume of Au source. Compressive strength of Au foam in 5.6% relative density was characterized to investigate its machinability for subsequent applications.

2. Experimental details

Au nanoparticle hydrosol, which plays the seeding role in subsequent two-step deposition process, was synthesized in advance according to previous report [23]. Aqueous solution of chloroauric acid (HAuCl_4) in concentration of 0.4 mol/L was prepared as Au source. Negatively charged PS microspheres (Nano-Micro Tech. Corp., China) with particle size of 10 μm were purchased as the sacrificial template. The bottom-up synthesis of self-supported low-density Au foams is schematically illustrated in Fig. 1.

Firstly, the PS latex microspheres (0.75 ml, 10 wt% in water, DVB: 10%) were extracted and dispersed in 7.5 ml deionized water. The prefabricated Au nanoparticle hydrosol (10 ml) was added into the PS solution dropwisely under vigorous agitation. The Au nanoparticles adhere firmly onto the PS surface by electroplating deposition. The Au nanoparticle coated PS beads were extracted by centrifugation and then dispersed in deionized water. 1.1 g polyvinylpyrrolidone (PVP, $M_{\text{av}} = 10,000$) and 0.2 g hydroxylamine hydrochloride were incorporated into the PS/Au aqueous solution as stabilizing and reduction agents. HAuCl_4 solution in volumes of 0.6, 0.7, 0.8, 0.9, 1.0 and 1.1 ml (represented as GF-06, GF-07, GF-08, GF-09, GF-10, GF-11 samples) were subsequently added to further introduce Au onto the surface of PS beads by electroless deposition. After washing and centrifugation, the Au coated PS beads were extracted and dispersed in 1–2 ml deionized water. Self-supported PS/Au foams were formed by a filter-casting method using $\Phi 5$ mm Teflon tube as the forming mold and compacted plaster of Paris (CaSO_4) as the water-absorbing agent [20]. After drying and demolding, the PS/Au foams were calcined at 400 $^\circ\text{C}$ for 12 h in flowing N_2 atmosphere. The PS template was

completely eliminated and self-supported cylindrical Au foams were produced.

Microstructure of the prefabricated Au nanoparticle hydrosol was characterized by high-resolution transmission electron microscope (HRTEM, JEOL JEM-2010). Particle size and surface charge of the Au nanoparticles were measured by laser granular analyzer (Brookhaven 90plus) and Zeta potential analyzer (Brookhaven Zeta PALS). Surface charge of the as-purchased PS template was analyzed as well. Ultraviolet-Visible spectrophotometer (Shimadzu UV-3150) was employed to characterize the Au nanoparticle hydrosol before and after electroplate deposition. Morphology of the prepared Au foams was observed using field-emission scanning electron microscope (FESEM, JEOL S-4800 and Carl Zeiss Ultra 55). Particle size of the coated Au nanoparticles was calculated using image analysis software (Image-Pro Plus) from the FESEM images. Room temperature compressive stress-strain curve of GF-09 sample was carried out on MTS810 materials testing machine with a loading speed of 0.5 mm/min. Three compression tests were performed and the average value of compressive strength was calculated from the stress–strain curves.

3. Results and discussion

3.1. Characterization of Au nanoparticle hydrosol

The Au nanoparticles were firstly synthesized in toluene and then be transferred into deionized water using equivalent volume of 4-dimethylaminopyridine (DMAP) solution [23]. Compared with Au nanoparticles synthesized in organic agent, the nanoparticles prepared in aqueous solution display higher concentration and dispersity with predictable particle size and shape. Fig. 1(a) and (b) demonstrates the HRTEM images of the as-synthesized Au nanoparticle hydrosol. The Au nanoparticles are in near spherical morphology with particle size centered at 5.5 nm, which is in accordance with previous reports [23,24]. The surface of Au nanoparticles is positively charged with Zeta electric potential of +5.40 mV. The surface charge can be attributed to the existence of stabilizer ligands derived from DMAP. Single Au nanoparticle exhibits distinct lattice fringes in the HRTEM image (Fig. 2b). Fringe distance of the grain crystal is calculated to be 0.235 nm, which is in agreement with the lattice constant of Au and reveals the Au nanoparticles are in crystalline structure.

Surface charge of the purchased PS latex microspheres were also characterized by Zeta potential analyzer with the SEM image illustrated in Fig. 2(c). It can be observed that the PS beads are in high uniformity and monodispersity with particle size of 10 μm and spherical morphology. The PS surface was negatively charged with Zeta electric potential of -2.51 mV because of the existence of residual sulfate radicals. The Au nanoparticles were readily deposited onto the surface of PS beads by electrostatic interaction as they were contrarily charged. The Au nanoparticle hydrosols before and after PS coating were characterized by UV spectrophotometer as the resultant spectrums shown in Fig. 2(d). The as synthesized Au hydrosol shows a narrow absorbance peak centered at 517 nm, which locates at the visible wavelength region. The absorbance peak exhibits a slight broadening and red shift to 526 nm after deposition. From Mie theory in plasmonic resonance absorption, small particles demonstrate different peak positions according to their particle size [25]. Red shift and broadening occur in the absorption peak as the particle size increase. Thus, we can deduce that the remnant Au nanoparticle hydrosol after deposition contains Au nanoparticles with larger mean particle size than the as synthesized sample. This phenomenon can be easily explained as nanoparticles in smaller size adhere preferentially onto the PS surface under the effect of surface tension. Meanwhile, the absorbance intensity of Au

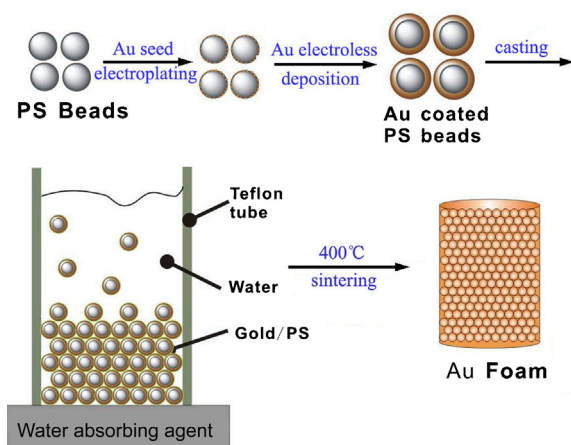


Fig. 1. Schematic illustration of the templating synthesis route.

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