



Direct sputtering- and electro-deposition of gold coating onto the closed surface of ultralow-density carbon-hydrogen foam cylinder



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HIGHLIGHTS

- The surface pores of P(DVB/St) foam cylinder are sealed by CVD method.
- Gold film was deposited on the surface of foam cylinder by magnetron sputtering.
- Electroless plating was excluded in the present experiments.
- The gold coatings were thickened through the electrodeposition process.

ARTICLE INFO

Article history:

Received 30 June 2016

Received in revised form 13 October 2016

Accepted 16 October 2016

Keywords:

Ultralow-density foam cylinder

Poly-*p*-xylylene

Magnetron sputtering

Electrochemical deposition

ABSTRACT

This work aimed to fabricate a gold coating on the surface of ultralow-density carbon-hydrogen foam cylinder without electroless plating. Poly (divinylbenzene/styrene) foam cylinder was synthesized by high internal phase emulsion, and chemical vapor deposition polymerization approach was used to form a compact poly-*p*-xylylene film on the foam cylinder. Conducting gold thin films were directly deposited onto the poly-*p*-xylylene-modified foam cylinder by magnetron sputtering, and electrochemical deposition was adopted to thicken the gold coatings. The micro-structures and morphologies of poly (divinylbenzene/styrene) foam cylinder and gold coating were observed by field-emission scanning electron microscopy. The gold coating content was investigated by energy-dispersive X-ray. The thicknesses of poly-*p*-xylylene coating and sputtered gold thin-film were approximately 500 and 100 nm, respectively. After electrochemical deposition, the thickness of gold coating increased to 522 nm, and the gold coating achieved a compact and uniform structure.

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1. Introduction

With the emphasis on environmental protection in numerous countries throughout the world, conventional fossil fuels are increasingly becoming less suitable for highly technology-oriented society because of their high carbon dioxide emission [1,2]. Nuclear fusion energy, which is derived from heavy hydrogen stored abundantly in seawater, is predicted to be a permanent, powerful, and clean source of energy [3,4]. Scientists face considerable challenge regarding the practical application of nuclear fusion: the fusion reaction must be a stable and controllable process [5,6]. Mainly, direct- and indirect-drive methods have been suggested in previous reports to solve this challenge in inertial confinement fusion [7–12].

In fusion physical experiments, Z-pinch, which obtain high-efficiency thermal X-ray energy from electrical energy, have been demonstrated to be an advantageous tool for indirect-drive of the heavy hydrogen device [13,14]. In 1999, D. Hoarty et al. [15] designed a TRIDENT experimental assembly, in which a carbon-hydrogen foam with a density of 30 mg/cc and thickness of 300–500 μm attached a gold cylinder on the end of gold hohlraum. The result showed that an initial front propagation speed using 30 mg/cc foam was about 14 cm/us, and this assembly was of interest to Z-pinch applications.

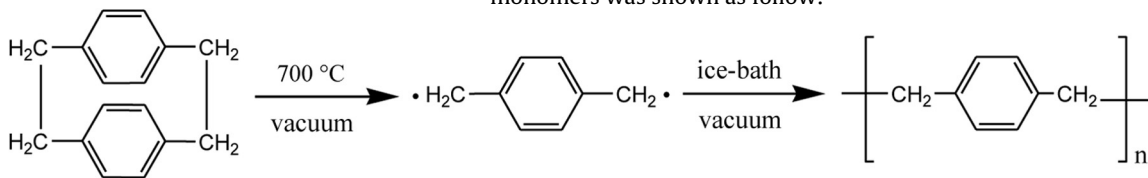
Poly (divinylbenzene/styrene) (P (DVB/S)) [16,17] and poly (4-methyl-1-pentene) (PMP or TPX) [18,19] are the two main polymers for synthesizing the carbon-hydrogen foams that are applied in Z-pinch devices. The traditional metallization method of non-active surfaces, such as plastics, employes electroless plating process [20,21]. Prior to electroless deposition, surface activation is required to initiate electroless metal deposition [22–24]. However, the activation processes result in the existence of impurity ele-

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ments, including Pd, Sn, Cl, O, and others, in the composites, which are likely to affect the diagnostic results of inertial confinement fusion (ICF) experiments [25,26].

In the present work, the surface pores of P (DVB/St) foam cylinder prepared by high internal-phase emulsion (HIPE) approach were sealed by chemical vapor deposition (CVD) polymerization of poly-*p*-xylylene (PPX, only containing C and H elements) coatings, and then magnetron sputtering was introduced to deposit gold



thin-films with high conductivity onto the surface of samples. The gold coatings were thickened by the electrodeposition process to complete the construction of P (DVB/St) foam/gold coatings.

2. Experimental

2.1. Materials and characterization

Divinyl benzene (DVB, A.R. grade) and styrene (St, A.R. grade) were purchased from Chengdu Kelon Chemical Reagent Company. We used 10% sodium hydroxide solution to rinse the two reagents for a minimum of five times to remove inhibitors, followed by distilled under reduced pressure. Others reagents were of analytical grade. Span-80, potassium persulfate ($K_2S_2O_8$), anhydrous calcium chloride ($CaCl_2$), alcohol (CH_3CH_2OH), potassium gold chloride ($KAuClO_4$), tri-ammonium citrate, and sodium sulfite (Na_2SO_3) were also purchased from Chengdu Kelon Chemical Reagent Company. Xylene (A.R. grade) was received from Chengdu Institute of Chemical Reagents. 2, 2-Paracyclophan was purchased from Aladdin Industrial Corporation.

The p (DVB/St) foam cylinders and gold coatings were observed using Inspect F field emission scanning electron microscopy (FESEM) to investigate the micro-structures and morphologies. An X'pert Pro X-ray diffractometer was used to characterize the crystallinity of gold coatings, and energy-dispersive X-ray spectroscopy (EDS) was used to measure chemical compositions.

2.2. The preparation of p (DVB/St) foam

In a typical process, 1.5 mL DVB, 0.37 mL St, 0.37 g Span-80, and 6 mL xylene were mixed and continuously stirred to form an oil phase. A 0.16 g portion of the initiator potassium persulfate ($K_2S_2O_8$), 0.6 g anhydrous calcium chloride ($CaCl_2$), and 60 mL deionized water were added into a 100 mL beaker as a water phase. Then, the water phase was added dropwise to the oil phase at a rate of 200 drops/min under magnetic stirring for 20 min. The prepared emulsion was transferred to glass tubes with a diameter of 3 mm and polymerized at 60 °C for 48 h. Finally, the polymer was cleaned with ethanol at 50 °C for 48 h and then dried in vacuum oven at 60 °C for another 48 h.

2.3. Hole-sealing process of polymer foam cylinder

CVD polymerization of PPX was used to undertake the hole-sealing process of polymer foam cylinders. Similar processes described in previous references [27,28] were adopted to carry out CVD polymerization. First, 3 g of the precursor paracyclophan was sublimated at 180 °C under a pressure of 5×10^3 Pa from a ceramic crucible placed in a quartz tube. Second, the monomers

were converted into the reactive intermediate diradicals at 700 °C. (The purpose of 700 °C is to form a thin-film on the surface of foam cylinders within a short time). Finally, diradicals were introduced into the deposition chamber equipped with an ice bath, and the polymerization reaction of PPX occurred on the surface of the rotated foam cylinders. As the carrier gas, argon was fluxed through the quartz tube with a flow of $0.5 \text{ cm}^3 \text{ s}^{-1}$ (sccs). The polymerization mechanism of PPX thin film derived from paracyclophan monomers was shown as follow:

2.4. The gold thin-film by magnetron sputtering

As conducting layers for gold plating, ultrathin gold films were directly deposited onto the PPX-modified foam cylinders by magnetron sputtering. The experiments were performed in a vacuum chamber attached with a rotating foundation support and operated under a base pressure of 1×10^{-3} Pa. As the working gas, argon was controlled at a pressure of 1 Pa and a flow rate of 5 sccm. The PPX-modified foam cylinders were loaded into a copper crucible. A gold disk (Au 99.9%, 51 mm in diameter and 2 mm in thickness) was used as target, and the distance between target and samples was 8 cm. The fabrication sputtering time was limited to a range of 3–4 min. The thickness of Au films deposited onto foam was determined by X-ray thickness meter.

2.5. Gold electroplating in cyanide-free baths

Gold electroplating was carried out on the surface of foam cylinder after magnetron sputtering to increase the thickness of gold coatings for the formation of a compact gold layer. The gold bath composition and operating conditions, which was similar to those described in the reference [29], were presented in Table 1. As shown in Table 1, tri-ammonium citrate performed key functions as a complexing agent and a buffer in gold plating solution. Sodium sulfite is an essential element for stabilizing gold ions, which improves the stability of gold plating baths. The pH value of this solution was maintained at about 5.5, and the electroplating time was 10 min. A cylindrical gold with a thin-wall thickness of 0.1 mm and a diameter of 20 mm was employed as an anode.

3. Results and discussion

3.1. Characterization of p (DVB/St) foam

The SEM images of p (DVB/St) foam cylinder synthesized by high internal-phase emulsion are shown in Fig. 1. The surface of foam cylinder is smooth and uniform under low magnification. However, numerous micron-grade holes are clearly observed under high magnification. The average density of the foam cylinder is found to

Table 1
Composition and operating conditions of the gold plating bath.

Composition	Concentration
Potassium gold chloride ($KAuCl_4$)	25 g/L 100 g/L
Tri-ammonium citrate ($(NH_4)_3C_6H_5O_7$) sodium sulfite (Na_2SO_3)	68.5 g/L

Operating conditions: pH 5.5; temperature 50 °C; anode material Au; cathodic current density 1.5 mA/cm^2 ; cathode/anode area ratio 1:3; the plating time is 10 min.

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