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Fabrication of glass fibers uniformly coated with metal films by magnetron sputtering by a two step process involving fixation by a photoresist

Mingming Lan*, Huiqin Li, Dong Wang, Yan Li, Guangyin Xu

College of Mechanical and Electrical Engineering, Henan Agricultural University, Zhengzhou 450002, PR China

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A B S T R A C T

In this paper, glass fibers with high aspect ratio were used as inner-core templates to prepare core–shell functional particles by way of magnetron sputtering by a two step process involving fixation by a photoresist. The morphologies and constituents of the metal-coated glass fibers were characterized using a scanning electron microscopy and an energy dispersive spectrometer. The crystal structures were characterized by employing X-ray diffraction. The surface roughnesses of glass fibers before and after magnetron sputtering were measured by an atomic force microscope. The results show that the glass fibers after magnetron sputtering have smooth and compact surface coatings, which exhibit face-centered cubic (fcc) structures. The RMS values of the metal-coated glass fibers are 4.20 ± 0.5 nm and 5.74 ± 0.5 nm for aluminum and silver respectively, which are significantly higher than that (1.36 ± 0.2 nm) of original glass fibers. Furthermore, the surface coatings of the glass fibers after electroless silver plating are very poor and there are lots of tiny fragments on them, resulting in high surface roughness (47.70 ± 5 nm). By comparison, the coating qualities of magnetron sputtering are significantly superior to that of electroless plating, indicating that magnetron sputtering is an excellent preparation method of core–shell particles.

1. Introduction

Metal-coated core–shell functional particles are widely used in industrial applications (defense, optical, magnetic, electrical and electronic fields, etc), owing to their lightweight characteristic, shape diversity and excellent performance [1]. According to the process features of the fabrication of core–shell functional particles, it consists of three parts: the inner-core templates, the functional materials and the deposition methods. Glass fibers with high aspect ratio are excellent templates for the fabrication of core–shell functional particles [2–5]. Aluminum and silver have been applied widely in electromagnetic wave shielding and thermal infrared suppression for their high electrical conductivity and low infrared emissivity [6–8]. The electroless plating method is characterized in simple process and convenient operation, which is appropriate for core–shell particles preparation in large scale production [9,10]. However, the surface coatings obtained by electroless plating have some drawbacks, such as high roughness, poor uniformity and low adhesion, which restrict significantly their further performance improvement and practical applications.

Magnetron sputtering is a vacuum process which has been widely used to deposit thin films on various substrates. The advantages are the coatings with high homogeneity and minimal impurity, easily controllable process parameters and flexibility in fabricating a wide variety of materials. However, magnetron sputtering is a vapor deposition method, which is not applicable to the fabrication of core–shell functional particles. There are only few reports on depositing thin films on microparticles surface using the magnetron sputtering technique. The existing research mainly focused on the modifications of the conventional magnetron sputtering apparatus, which is mainly suited to the deposition of spherical or near-spherical microparticles. Senos A.M.R. and coworkers [11–13] introduced a chamber with rotational and vibrational movement into a D.C. magnetron sputtering equipment, favoring the homogeneous nickel and stainless steel coatings of WC powder particles. Takayuki Abe and coworkers [14,15] deposited WC and Pt films on the surface of SiO_2 particles and spherical PMMA powders by using a sputtering system with a barrel-type powder sample holder (the barrel sputtering system). Hell J and coworkers [16,17] coated Cu, Pt and Mo films on the surface of

* Corresponding author. Tel./fax: +86 371 63554632.
E-mail address: lan_mingming@163.com (M. Lan).

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diamond particles and hollow glass spheres by using a sputtering system based on a rotating plate with three rotatable cups. Shen Zhigang and coworkers [18,19] deposited metal films (Co, Ni, Cu and Ag) on cenosphere particles and SiC particles by using a magnetron sputtering system in which a newly designed sample stage equipped with an ultrasonic vibration generator was implemented. Nevertheless, the above methods usually increase the complexity of magnetron sputtering apparatus, and it is unsuited to the deposition of non-spherical microparticles.

In this paper, a magnetron sputtering method based on a two step process involving fixation by a photoresist is proposed and used to deposit metal films (aluminum and silver) onto the glass fibers, and the corresponding sketch of the core–shell system in cross section is shown in Fig. 1. The characteristic properties of the obtained metal-coated glass fibers are investigated in detail. Moreover, the glass fibers metal-coated by magnetron sputtering are compared to the metal-coated glass fibers by electroless plating.

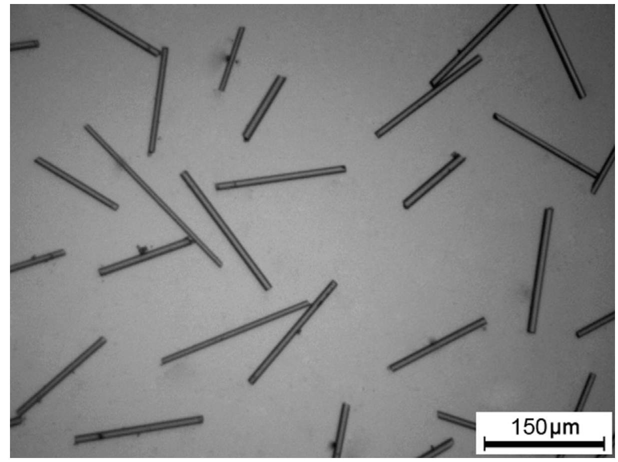

Fig. 2. OM image of glass fibers.

2. Experimental details

2.1. Materials

The glass fibers with high aspect ratio are selected as inner templates in this experiment, which were purchased from Hebei dingxing xuanyue science and technology Co., Ltd. In general, the fiber length is about 120–180 μm, the fiber diameter is about 15–20 μm, the density is about 2.45 g/cm^3, as shown in Fig. 2.

2.2. Magnetron sputtering

The magnetron sputtering method of glass fibers coated with metal films is based on a two step process involving fixation by a photoresist: the positive photoresist can be stripped in developer solution by way of UV irradiation, while the negative photoresist can be cured and not stripped in developer solution by way of UV irradiation.

A flat stainless steel plate (200 mm in diameter and 5 mm in thickness) was selected as the substrate. The process flowchart of glass fibers coated with metal films by magnetron sputtering based on a two step process involving fixation by a photoresist is shown in Fig. 3. The process of coating the glass fibers could be described as follows: Step 1: The stainless steel substrate holder was covered by Al foil (simply commercially available Al foil) for demoulding the photoresist easily; Step 2: a thin layer of positive photoresist was spin coated onto the Al foil, and fibers were dispersed on the photoresist layer in the spin-coating process to avoid serious agglomeration; Step 3: the positive photoresist was heated to 110 °C and cured to fix the fibers, then a metallic layer was sputtered onto the immobilized fibers. Step 4: samples were removed from the chamber and a thin layer of negative photoresist was spin coated onto the metallized fibers and the sandwich positive photoresist/metallized fibers/negative photoresist, which still is fixed on the Al foil, was removed from the stainless steel holder and turned; Step 5: The Al-foil is removed, the positive photoresist is UV irradiated and then stripped. Now the other side of the fibers is free and they are fixed on the negative photoresist. The exposed side of the fibers is again metallized. Finally, the completely metallized fibers are stripped from the negative photoresist by remover solution. The process parameters of magnetron sputtering metal films onto glass fibers are shown in Table 1. The throughput of the process is about 5–6 g per batch.

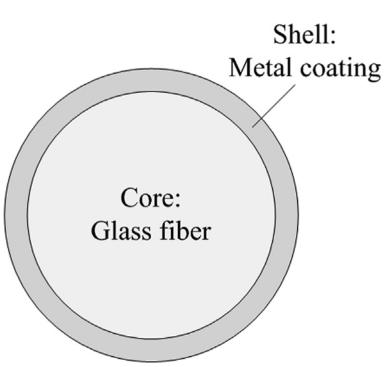

Fig. 1. Sketch of the core–shell system in cross section.

2.3. Morphology characterization

The morphologies of the glass fibers were examined by an optical microscope (OM, XSY-1) with photographic camera (SONY DSC-H50), a scanning electron microscope (SEM, Cambridge CamScan CS3400) and an atomic force microscope (AFM, Bruker Dimension Icon). The contact mode using silicon nitride tips were used and the scan areas of AFM images were 2 μm × 2 μm. The flatten command was used to remove the bow of the AFM images, and the root-mean-square (RMS) roughness values were calculated from AFM images. The composition analysis was made using an energy dispersive spectrometer (EDS, Oxford INCA). In order to guarantee the accuracy of EDS data, the three measurements were taken on the different coated fibers per batch, and the median was selected as EDS result in this paper. The crystal structures were characterized by employing the X-ray diffraction (XRD, Rigaku D/max-3) with Cu-Kα radiation at a voltage of 40 kV, a current of 40 mA, step size 0.02°, scanning rate 8°/min and wavelength 1.5418 Å.

3. Results and discussion

3.1. Morphological characteristics

Fig. 4 shows SEM images of glass fibers before and after magnetron sputtering of the metal films. It can be seen that the appearance morphologies of the glass fibers before and after magnetron sputtering metal films (Al and Ag) are roughly identical, which indicates that the glass fibers after magnetron sputtering still keep their initial shapes with high aspect ratio and there are no ruptures or agglomerates. From the enlarged SEM images of the metal-coated glass fibers (Fig. 5), the metal coatings on the surface