



Fabrication and Photocatalytic Performance of Au/ZnO Layered Structure on Silk Textile for Flexible Device Applications



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ABSTRACT

This study integrated ZnO/Au layered structure on silk textiles to realize a flexible photocatalytic composite material for wearable devices. Supercritical carbon dioxide promoted electroless plating and cathodic deposition were used to metallize and deposit photocatalytic metal oxides on silk textiles. Silk was used as the substrate for its flexibility and stretchability. Au was selected due to its high biocompatibility, electrical conductivity, and ductility. ZnO was chosen for its photocatalytic activity and biocompatibility to fulfill the application. Surface morphology, composition, crystal structure, electrical resistance and photocatalytic activity assessments were evaluated. With the assistance of supercritical carbon dioxide, smooth, strong adherence and uniform coverage of Au layer was successfully electroless plated on the silk. ZnO was homogeneously deposited on the Au metal layer. The coatings on the silk have been confirmed to be metallic Au phase and ZnO wurtzite phase. The ZnO modified specimen with 0.2 wt. % H₂O₂ in the electrolyte showed the highest crystallinity and performed 11.5 times enhancement in the photocurrent density when comparing to the H₂O₂-free one.

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1. Introduction

The global market of wearable devices is predicted to keep prospering in the future [1]. In the meanwhile, wearable devices are diversified into different functions such as biomedical devices, supercapacitors, and photocatalytic devices [2] due to diverse requirements in the next-generation technology. The integration of functional and flexible materials turns out to be the most critical step in fabrication of the component used in wearable devices. For example, integration of photocatalytic, electrical conductive, and flexible materials is needed to realize wearable devices capable of harvesting solar energy.

Flexible materials such as cloth textiles are usually non-conductive, and the flexible material can be made electrically conductive by depositing metal coatings on the non-conductive material through electroless plating [3]. Electroless plating

consists of a pretreatment step to clean the substrate, a catalyzation step to activate the substrate, and a metallization step to metallize the substrate. In the conventional catalyzation step, catalyst-contained corrosive aqueous solution is used to activate the substrate by depositing the catalyst on the substrate; meanwhile, the substrate structure is often damaged during the process. Due to the high surface tension and polarity of the aqueous catalyzation solution, the catalyst is only inlaid on the substrate surface resulting in poor adhesive property. On the other hand, supercritical carbon dioxide (sc-CO₂) can be introduced to the catalyzation step to enhance the plating characteristics [4,5]. Carbon dioxide develops into supercritical phase owning properties in the midway of gas phase and liquid phase when both the temperature and pressure go beyond its critical point [6]. In this case, sc-CO₂ can be used as the solvent, and an organic-metal complex would be used as precursor of the catalyst. Due to the affinity to non-polar material [7], low surface tension [8], and high self-diffusivity [6] of sc-CO₂, sc-CO₂ thus can convey the organic-metal complex into the textile structure while remains the substrate structure intact, and the catalyst could be formed after a reduction process.

Regarding the photocatalytic functionality, there are various synthetic methods to deposit photocatalytic metal oxides, which

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include high-temperature vapor deposition [9], polymer-mediated chemical reaction [10], hydrothermal-annealing process [11], and one-pot solution-based method [12]. Among the synthetic techniques, cathodic deposition allows a simple and effective process to produce metal oxides [13–15]. In addition, cathodic deposition can cooperate with lithography to allow further applications such as patterning [16]. In this study, the photocatalyst/metal/cloth textile flexible composite material was realized by *sc*-CO₂ assisted electroless plating and cathodic deposition for applications in wearable devices.

A slight disturbance from the rigid component in a wearable device can be annoying to the user, and hence a flexible material is preferred in wearable devices. Silk, a common clothing material, was chosen in this study due to its flexibility and stretchability. Since wearable devices come into contact with the skin frequently during the employment, a thin Au layer, which is known to be highly biocompatible, was selected as the electrically conductive layer. Moreover, Au also shows high ductility [17], which is an essential requirement for wearable devices. There are many materials that can play the role as the photocatalyst in the composite material, such as TiO₂ [18], CuO [19], and ZnO [20]. ZnO was designated as the photocatalyst due to its comprehensive applications, requirement of low fabrication temperature to have high crystallinity, and simple fabrication process. There are several reports on preparation of flexible photocatalyst materials, such as metal-organic chemical vapor deposition of GaN on graphene film [21] and electrospinning combined with hydrothermal method to deposit TiO₂ on polyacrylonitrile [22]. However, due to the aforementioned requests in next generation wearable devices, flexible composite materials composed of highly biocompatible cloth textile, conductive metallic layer, and photocatalyst are needed. Here, we report a facile fabrication process in realization of a flexible and photocatalytic ZnO/Au/silk layered composite material toward applications such as solar energy harvesting wearable devices. Fundamental properties required for wearable devices such as electrical resistance and photocatalytic activity evaluations are demonstrated.

2. Experimental

2.1. Chemicals

A piece of silk in the dimension of 2 cm × 4 cm was used as the substrate, and Pd(II) acetylacetonate (Pd(acac)₂) (98.0%, Tokyo Chemical Industry Co., Ltd., Japan) was used as the organic-metal complex. Carbon dioxide gas (99.99%, Nippon Tansan Gas Co., Ltd. Japan) was used as the source of *sc*-CO₂. A commercially available acidic Ni-P electroless plating electrolyte (Okuno Chemical Industries Co., Ltd., Japan) consisted of nickel chloride (1.8 wt.%), sodium hypophosphite (2.4 wt.%), complexing agent (2.4 wt.%), and ion-exchanged water (93.4 wt.%) was used in this study. The Au electroless plating electrolyte used in this study was a commercially available electrolyte provided by MATEX JAPAN Co., Ltd. The Au electroless plating electrolyte was composed of ammonium gold sulfite (0.2 wt.%), reducing agent (0.3 wt.%), complexing agent (0.3 wt.%), and ion-exchanged water (99.2 wt.%). The electrolyte for cathodic deposition of ZnO comprised of 0.25 wt.% ZnCl₂ (98.0%, Sigma-Aldrich, USA), 3.0 wt.% NaCl (99.0%, Sigma-Aldrich, USA), 1.5 wt.% NaNO₃ (99.0%, Sigma-Aldrich, USA), 0–0.3 wt.% H₂O₂ (30%, Sigma-Aldrich, USA), and ion-exchanged water.

2.2. *Sc*-CO₂ assisted catalyzation

No pretreatment was carried out before the *sc*-CO₂ catalyzation step. Pd(acac)₂ was used as the precursor in formation of the Pd catalyst seed used in metallization of a Ni-P layer on the silk, and 25 mg of the Pd(acac)₂ catalyst was used with respect to a 50 ml reaction cell to maintain the Pd(acac)₂ concentration at the saturation point throughout the process. A piece of the silk textile was hooked from inner ceiling of the 50 ml stainless reaction cell [23], and *sc*-CO₂ and the Pt(acac)₂ were introduced into the reaction cell afterward. A high-pressure apparatus (Japan Spectra Company, Japan) was used to pressurize CO₂ into the reaction cell, and the details could be found in a previous study [23]. The catalyzation was executed at 80 ± 1 °C and 15 ± 0.1 MPa with

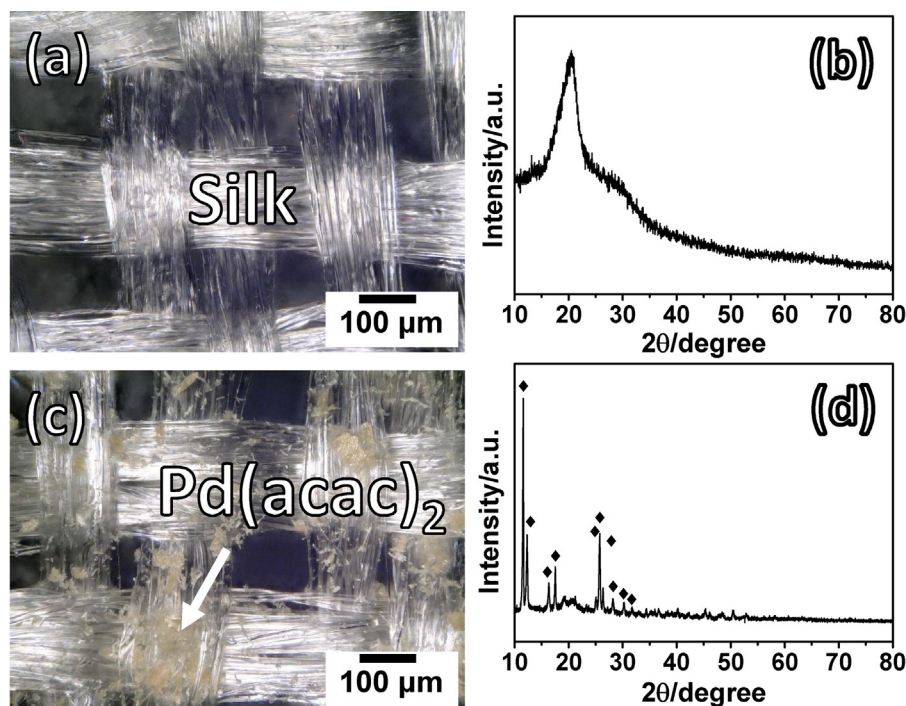


Fig. 1. (a) OM image and (b) XRD pattern of the as-received silk, and (c) OM image and (d) XRD pattern of the catalyzed silk (diamond symbols: Pd(acac)₂).

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