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Electromagnetic interference shielding of layered linen fabric/polypyrrole/nickel (LF/PPy/Ni) composites

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ABSTRACT

In this paper, layered linen fabric/polypyrrole/nickel (LF/PPy/Ni) composites were successfully fabricated by combining a chemical in-situ polymerization approach with an electroless plating method. Owing to the multiple characteristics of both wave-absorption and wave-reflection, the resultant composites showed great potential as electromagnetic interference (EMI) shielding material to realize electromagnetic compatibility. Various experimental conditions including initiator concentration, polymerization time and synthesis cycles were optimized in the in-situ doping polymerization process. Under the optimized conditions, the coating amount of PPy was adequate for generating the inter-fiber connection. Afterwards, Ni layer was deposited on the as-made PPy coated LF (LF/PPy). X-ray diffraction (XRD) analysis indicated that the Ni layer had a characteristic face-centered cubic (FCC) crystalline structure. Vibrating sample magnetometry (VSM) analysis revealed that the resultant LF/PPy/Ni composites exhibited strong magnetic properties. Furthermore, comparative study of shielding effectiveness (SE) among nickel plated LF (LF/Ni), LF/PPy and LF/PPy/Ni was conducted by using a Spectrum analyzer at a frequency range of 30 to 1000 MHz. The result indicated that the highest SE was obtained in the LF/PPy/Ni sample, which had already achieved civilian product standard.

1. Introduction

Electromagnetic (EM) waves from electronic instruments have an adverse effect on the performance of neighboring equipment and even can cause mutual malfunction [1–5]. Hence, EM compatibility of an electronic circuit at broadband EM wave is of great importance in the design of integrated circuits. External EM radiations should not interfere with the internal circuit. Meanwhile, the internal circuit should not radiate EM energy to disturb other neighboring circuits. The most efficient method to realize such an EM compatibility is to house the circuit in an enclosure utilizing electromagnetic interference (EMI) shield, which can attenuate the radiations from the internal circuit and prevent external radiations interfering with the internal circuit [6].

Conductive textile emerging as a burgeoning material have been applied extensively in EMI shield due to their advantages such as prominent flexibility, high EMI shielding effectiveness (SE), good electrostatic discharge and light weight. To manufacture a conductive fabric featuring with high SE, a variety of conductive components has been introduced, such as metals and intrinsically conducting polymers (ICPs) [7–12]. Herein, SE is a notion to characterize the effectiveness of EMI shield in terms of decibels (dB). When an incident EM wave encounters the conductive fabric, it will be blocked via three forms, namely reflection, absorption and internal multiple reflections. Therefore, the EMI SE can be expressed by the following equation:

$$SE = A + R + B \quad (1)$$

where A, R and B denote absorbed wastage, reflected wastage and multiple internal reflected wastage, respectively [12]. ICPs such as polyaniline (PAN), polyacetylene (PA) and polypyrrole (PPy), are typically EM wave-absorption dominated material due to their tunable electrical/dielectric properties and non-transparency toward EM radiations [13–15]. Conversely, metals are EM wave-reflection dominated material due to their abundant mobile charge carriers [16–19]. To transform the textile from insulating to conductive, various methods have been proposed [20–22], such as a mixture of metal powders during melt or wet spinning process, twisting or wrapping insulating fibers with metallic wires during mechanical spinning process and selecting ICPs for chemical spinning, etc. However, those aforementioned methods are always sophisticated and involve in numerous problems, such as importing conductive components during spinning will result in filaments or yarns with poor flexibility, selecting ICPs for spinning is difficult to form homogeneous and mechanically strong filaments. Be different from those spinning-stage methods, directly forming metal or ICPs coating on textile production (fiber, yarn, fabric) may be more suitable due to their low cost, labor-saving and simplicity of processing. Qin et al. successfully manufactured copper coated polyester fabric by autocatalytic copper plating process using glyoxylic acid as a reducing agent.

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The copper coated polyester fabric possessed SE of 40–50 dB at the frequency of 6 GHz to 18 GHz. However, the metal coated fabric had reflection as its major shielding form, which limited its development in SE [23]. Parveen et al. proposed composite absorbers based on conducting fabrics, which possess moderate conductivity and dielectric/magnetic properties. The absorbers were prepared by in situ incorporation of nanoparticles of $BaTiO_3$ or Fe_3O_4 within coated polyaniline matrix. The specific SE value of the composite absorber was 17–20 dB cm^3/g, which demonstrated that these fabrics exhibited great potential as promising microwave-shielding material [24]. Although metal plated fabrics or ICPs coated fabrics for EMI shielding have been studied recently, researches about combination of them two to manufacture conductive fabrics have not been revealed to date. If the contribution of absorbance or reflectance to the total EMI SE can be controlled by the appropriate array of ICPs and metal, an extremely high SE will certainly be achieved theoretically. Inspired by the aforementioned analysis, ICPs and metal were successively deposited on the natural textile to fabricate EMI shielding materials in this paper.

In the present work, linen fabric (LF) was selected as the substrate to manufacture layered linen fabric/polypyrrole/nickel (LF/PPy/Ni) composites via a PPy deposition followed by an electroless nickel plating. LF/PPy/Ni composites were synthesized in two successive steps: (I) PPy layer was constructed on LF substrate by self-polymerization of pyrrole monomers. (II) Nickel film was grown on 3-aminopropyltrimethoxysilane (APTMS) self-assembled monolayers (SAMs) modified LF/PPy by electroless nickel plating. In step (I), pyrrole was used as monomer, Ferric chloride ($FeCl_3$) was designed as oxidant, and sodium p-toluenesulfonate (STS) was selected as dopant. Prior to the step (II), LF/PPy was modified by APTMS. The APTMS used here was a kind of trifunctional alkoxy silanes ($R'Si(OR)_3$, where R and R′ are alkyl groups), which acted as a molecular bridge between organics (PPy film) and inorganics (Ni layer) [25]. It was confirmed that the APTMS-SAMs could enhance the adhesive strength between metal layer and substrate by chemical sorption instead of the physical sorption in the conventional sensitizing-activation method [26–29]. In step (II), electroless nickel plating was performed by multistep processes including modification, activation and nickel deposition followed by rinsing and drying. The synthetic route of the LF/Ni/PPy composites was shown schematically in Fig. 1.

Optimization study was conducted in order to achieve the optimum experimental conditions in the polymerization process. Herein, the optimum PPy layer was evaluated in terms of SE (30–1000 MHz). Fourier transform infrared spectroscopy (FTIR) measurement was conducted to investigate the interaction mechanism between LF substrate and PPy. The surface morphologies of the samples in each step were investigated by scanning electron microscopy (SEM), and the crystal structures of the external nickel layer were detected by X-ray diffractometer (XRD) measurement. Surface resistance (Rs) was measured by the four probe method described in ASTM F 390. Magnetic properties of the resultant LF/PPy/Ni composites were investigated by vibrating sample magnetometry (VSM). Peel test and tensile test were utilized to evaluate the reliability of the composites. Additionally, SE values of LF/Ni, LF/PPy and LF/PPy/Ni were recorded and compared in this study by using a Spectrum analyzer.

2. Experimental

2.1. Materials

The linen fabrics (45 × 45 count/cm^2, 24 mg/cm^2) were purchased from Taicang Biqi Novel Material Co., Ltd., and were precisely cut into rectangular patch (6 × 6 cm or 5 × 15 cm). Pyrrole, sodium p-toluenesulfonate (STS), 3-aminopropyltrimethoxysilane (APTMS) and hexahydrate ferric chloride ($FeCl_3 \cdot 6H_2O$) were obtained from Sinopharm Chemical Reagent Co., Ltd. Particularly, pyrrole was distilled under reduced pressure and stored at 4 °C for the following usage. All other reagents were of analytical grade and were used without further purification unless otherwise mentioned. Moreover, the distilled water was purified by a Milli-Q system (Milford, MA, USA).

2.2. In-situ polymerization of pyrrole

The pristine LFs were degreased in alkaline solution (60 g/L NaOH + 30 g/L Na_3PO_4 + 15 g/L Na_2CO_3) to remove oils and other organic chemicals, followed by rinsing with distilled water until the pH value reached neutral and drying in an oven at 50 °C. Afterwards, a two-stage bath process was performed for the in-situ polymerization of pyrrole. In the first stage, LFs were soaked in the pyrrole monomer

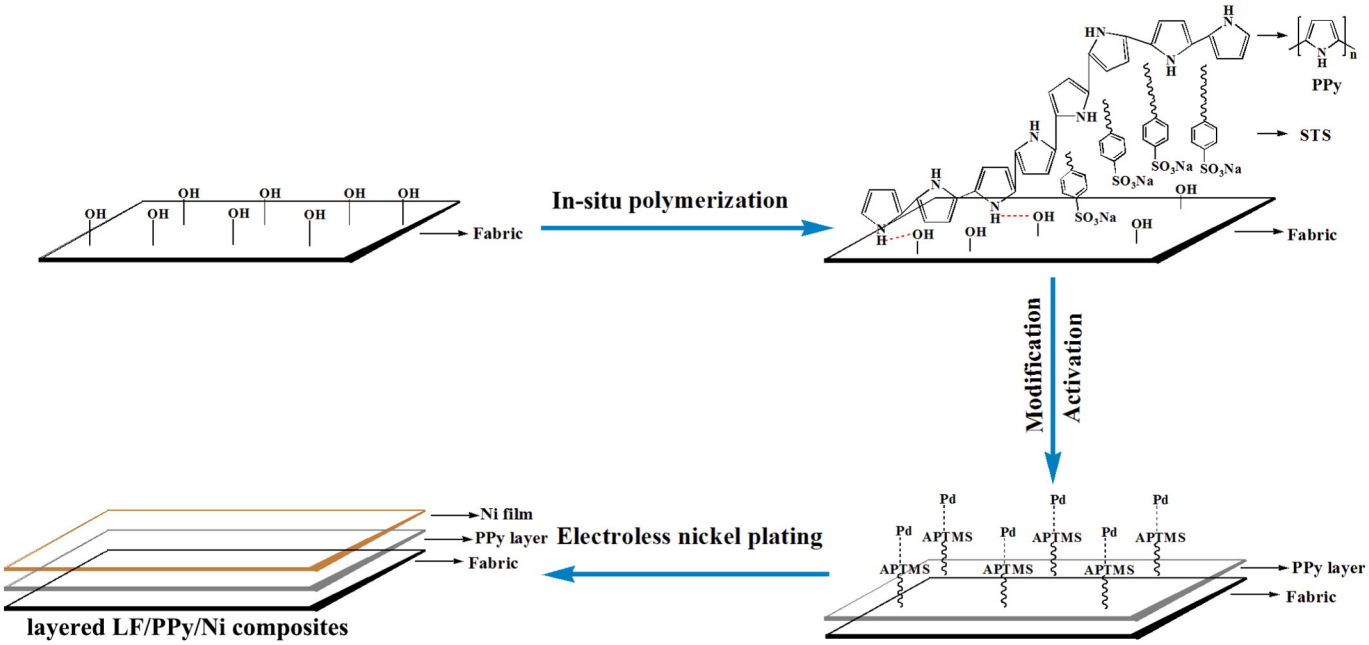

Fig. 1. The synthetic route of the layered LF/PPy/Ni composites (APTMS and STS are the abbreviations of 3-aminopropyltrimethoxysilane and sodium p-toluenesulfonate, respectively).