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# Electromagnetic shielding efficiency of plasma treated and electroless metal plated polypropylene nonwoven fabrics

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As the use of commercial and scientific electronic devices and communication instruments increases, there is an increased interest in shielding against electromagnetic radiation. The lifetime and efficiency of electronic devices can be increased through effective electromagnetic interference (EMI) shielding. To produce materials with high EMI shielding efficiency (EMISE), various metal coating techniques have been suggested and commercially used, as metal foils and laminates, conductive paints and lacquers, sputter coating, electroless plating etc. Among them, electroless plating is a promising way to produce metal-coated fabrics. This technique has advantages such as homogeneous metal deposition, excellent conductivity and high EMISE, and applicability to non-conductors and complex shaped materials. In the standard electroless procedure a catalytically active substance (usually $SnCl_2$/$PdCl_2$ mixed solution) is first deposited on the surface of substrate. For polymer fibers, an efficient surface treatment process is required before Pd chemisorption, due to their low surface activity. Their surface can be activated either by alkali or acid etching [1, 2] or by plasma treatment [3]. In this work, plasma activation of polypropylene nonwoven fabrics (PPNF) using atmospheric pressure surface barrier discharge and the subsequent electroless copper plating were studied. Preliminary results of EMISE are discussed.

Industrial spunbonded polypropylene nonwoven fabrics supplied by Nippon Nonwoven Fabrics, Co., Ltd. (Japan) were used as a substrate. Properties of fabrics are shown in Table I.

The experimental arrangement used for the surface treatment of PPNF is sketched in Fig. 1. The reactor is based on a discharge with a high density of streamers generated on a dielectric surface. Such a discharge system was invented by Masuda and his coworkers [4]. The discharge system consists of two electrodes separated by $100 \times 100 \times 0.6$ mm$^3$ $Al_2O_3$ plate. The discharge electrode (1.5 $\mu$m-thick) consists of interconnected strips 1-mm-wide and 3 mm strip-to-strip distance. The inductive electrode is square-shaped ($90 \times 90$ mm$^2$), 1.5 $\mu$m-thick. Both electrodes were made of molybdenum. The electrode system with a sample was housed inside of a glass enclosure to allow containment of the process gas. The experiments were performed in nitrogen gas of technical purity (supplied by Air Liquide) at atmospheric pressure.

A sinusoidal high voltage with a frequency of typically 2.1 kHz and peak-to-peak value 9 kV was applied between the electrodes. This produced a stable surface discharge covering uniformly the ceramic surface. After stabilization of the discharge current (approximately 5 s), the PPNF sample was brought into contact with the discharge plasma layer using a rotatable sample carrier. The treatment time was defined as the contact time of the sample with the plasma. The average value of the power dissipated in the discharge, calculated from voltage and current waveforms, was approximately 0.3 W per 1 cm$^2$ of the specimen surface.

Electroless copper plating was realized by multistep process using commercial metallization baths (supplied by Kamiya Riken Co., Ltd., Japan). The plating procedure consisted of following steps:

1. Soaking in solution of HCl (50–100 ml/l) with an addition of a wetting agent.
2. Catalyzation in solution of $PdCl_2$ (50–200 mg/l), $SnCl_2$ (5–20 g/l) and HCl (100–200 ml/l) at 25–30 °C for 1–3 min.
3. Acceleration in HCl (80–120 ml/l) at 30–40 °C for 1–3 min.
4. Electroless copper plating in solution of $CuSO_4$ (8–10 g/l), EDTA (24–30 g/l), NaOH (6–8 g/l),

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TABLE I Types and properties of PPNF used as a substrate

| Type | Density ($g/m^2$) | Thickness (mm) |
|---|---|---|
| SP-1020E | 20 | 0.21 |
| SP-1030E | 30 | 0.27 |
| SP-1050E | 50 | 0.40 |
| SP-1070E | 70 | 0.48 |
| SP-1090E | 90 | 0.55 |

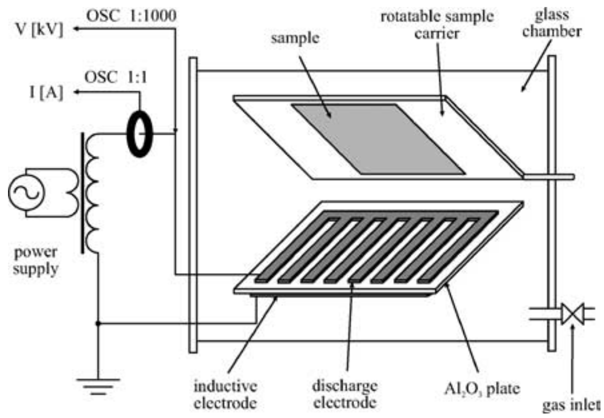


*Figure 1* Schematic view of the experimental set-up.

formaldehyde (5–7.5 ml/l) and stabilizer at 45–50 °C for 20 min.

Every step of the plating procedure was followed by rinsing in distilled water. The average plating rate was 1 $\mu$m/20 min.

The microstructure of electroless copper plated PPNF was examined using a Jeol JSM-6320F scanning electron microscope. A standard industrial peeling test ("Scotch Tape Test" [5]) was used to determine the quality of the copper adhesion to the substrate. The EMISE of fabrics was measured by using ASTM D4935-99 technique [6]. The EMISE of material is defined as a ratio of the power of incident ($P_I$) and transmitted ($P_T$) electromagnetic wave:

$$\text{EMISE} = 10\log(P_I/P_T)$$

The unit of EMISE is given in decibels (dB). The frequency range for EMISE measurements was from 50 MHz to 1.5 GHz.

Our previous experiments indicated that nonwoven fabrics could be best activated by using the surface barrier discharge generated in nitrogen. Optimal treatment time to get the highest hydrophilization of fabrics was 1–2 s [7]. More homogeneous copper layer and higher value of EMISE is achieved for both-sides plasma treated fabrics [8]. Based on these results, samples were plasma treated for 2 s each side. The thickness of copper layer estimated from plating rate was 1 $\mu$m.

As it is shown in Fig. 2a, a nonuniform metal layer on the untreated samples is observed. Apparently, even when the untreated samples are hydrophobic, their partial wetting and metallization is possible since the activation bath contains a wetting agent. In contrast, the plasma treatment renders the fiber surfaces hydrophilic and facilitates absorption of the Sn catalyst sensitizer and Pd catalyst in order to provide a catalytic surface for the electroless deposition of copper. Fig. 2b shows that a uniform copper coating on the individual fibers is obtained for plasma-treated PPNF.

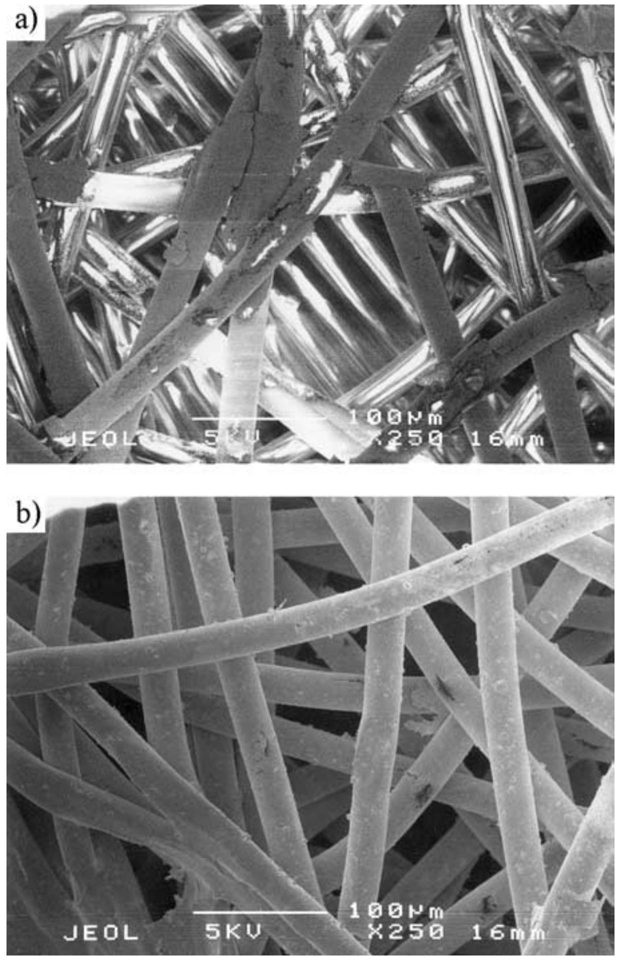


*Figure 2* Scanning electron micrographs of copper-plated PPNF (50 $g/m^2$): (a) untreated and (b) plasma treated before plating (2 s each side).

Fig. 3 shows the result of the peel test. The dark pattern on the picture represents copper peeled off the fabric. Adhesion of the copper layer to untreated PPNF is low and almost all copper layer can be easily peeled off the fabric (Fig. 3a). The adhesion of copper layer to plasma treated fabric is much higher and it is not possible to remove the copper layer without separating the fibers of the fabric (Fig. 3b).

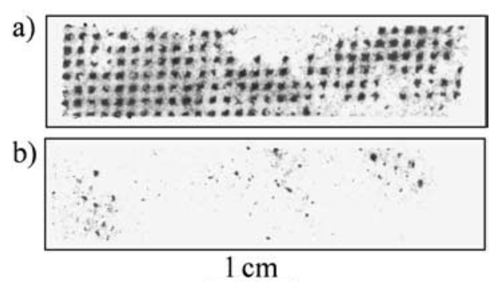


*Figure 3* Adhesion of copper layer to PPNF (picture of adhesive tape after peel test): (a) untreated fabric and (b) plasma treated fabric (2 s each side).

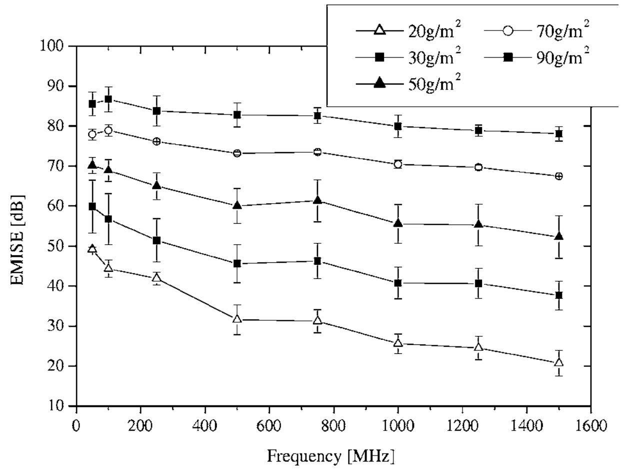


*Figure 4* EMISE of plasma treated (2 s each side) and copper-plated (20 min) PPNF as a function of fabric's density.

As it was reported in our previous work [8], untreated polypropylene fabrics were only partially copper-plated and they showed low values of EMISE (up to 10 dB). Fig. 4 indicates that EMISE increases after surface activation of fabrics by surface barrier discharge and its value is a function of frequency and density of substrate. The EMISE of 20 g/m$^2$ is 49 dB at 50 MHz and this value decreases to 21 dB at 1500 Mz. The EMISE is less sensitive to frequency and reaches higher values with increasing substrate density. It can be explained by fact, that not only the surface layer of fibres is copper plated, but fibers in the whole volume of fabric are homogeneously covered by copper (Fig. 2b). The highest shielding efficiency (85–78 dB) is obtained for 90 g/m$^2$ fabric.

In summary, atmospheric-pressure surface barrier discharge has been found to be very effective in the surface activation of PPNF. Fabrics with high EMISE were produced after 2 s treatment in nitrogen discharge followed by electroless copper plating. The copper layer shows high adhesion to fabrics and is homogeneously deposited on the surface of single fibers.

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