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Morphology and Electromagnetic Shielding Effectiveness of PP Nonwovens Modified with Metallic Layers

DOI: 10.5604/12303666.1161763

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Abstract

Polypropylene (PP) nonwoven was used as a substrate for CuSn and CuZnNi deposited layers. Nonwoven metallization was carried out using the DC magnetron sputtering process at various process parameters (e.g. effective power dissipated in the target, velocity of the substrate drift, number of cycles). The studies aimed at defining the surface morphology of PP nonwoven modified by metallic deposition, the crystallographic structure of the thin metallic layers and the effect of the layers' crystallinity on the electromagnetic (EM) shielding effectiveness of the two-component metal/PP composites. The morphology studies were covered by scanning electron microscopy. Furthermore the crystalline character of components CuSn and CuZnNi was examined in grazing-incidence angle X-ray diffraction experiments. It was found that CuSn/PP composites with a crystalline structure of the metallic layer exhibit the highest values of shielding effectiveness (44-45 dB at 27,12 MHz and 38-39 dB at 1795 MHz).

Key words: polypropylene nonwoven, metallic thin layers, magnetron deposition, surface morphology, crystalline structure, EM shielding effectiveness.

Introduction

Intensive development of PVD (Physical Vapour Deposition) processes allows to exploit, on an industrial scale, the specific properties of thin layers. Such coatings are increasingly used to improve the properties of various materials or to impart specified functions. Therefore the coatings obtained by PVD techniques are applied in many fields, such as optics, microelectronics, biomedicine, aeronautics, the aerospace industry, power engineering, the automotive industry, the construction industry, mechanical engineering, etc.

The thin layers owe their beneficial properties to their structure, which is usually amorphous and/or is characterised by the small size of the crystalline grains. Their properties are also significantly influenced by technical parameters of the deposition process, such as the pressure of working gases inside the chamber, the temperature of the process, and the distance between the substrate and sputtered material.

The coatings produced in PVD processes exhibit good mechanical properties, such as hardness and resistance to friction, as well as good adhesion to the substrate. The latter is one of the most important properties of thin layers produced by PVD techniques. If it is inadequate, the assumed quality of the coating cannot be achieved. The adhesion of the coating to the substrate is influenced by its microstructure, the type and structure

of the substrate, process conditions, and the purity and proper preparation of the substrate before the deposition process.

Almost in all PVD techniques, coatings are formed in a stream of ionized plasma which is led to the substrate by an electric discharge. Such processes are called plasma assisted PVD (PA PVD) and are finding increasing practical use. The large kinetic energy of particles of the deposited material inside the chamber provides better adhesion of the coating to the substrate. High energy of the plasma at the initial stage of the process additionally allows to clean the surface of the substrate prior to the coating deposition. Plasma also plays an important role in crystallisation occurring within the layer.

One of the methods utilising PA PVD processes is magnetron sputtering. A magnetron or magnetron launcher is a device employing, in its action, both electric and magnetic fields. Sputtering involves the "knocking out" of atoms from the surface of the electrode material (target) by high-energy particles of the working gas and subsequent depositing of them onto the substrate. Using magnetron systems, it is possible to obtain conductive [1, 2], insulating or magnetic coatings. Coatings obtained in PVD processes can be simple – formed by one type of material, or complex – multi-component, multi-layer, multi-phase, composite, etc [3].

The coatings deposited can be divided, due to their potential application, into:

- functional, giving certain properties such as mechanical, electrical or thermal conductivity and thermal resistance to the products [4],
- protective, e.g. anticorrosive,
- decorative and/or protective, bestow the product an aesthetic appearance (colour, gloss, texture), fluorescent or phosphorescent properties, etc.

Technologies for coating flat and uniform substrates are relatively well-mastered. However, depositing thin layers on products with complicated shapes is much more difficult. The most problematic seems to be the deposition of coatings on various textile substrates, due to their specific three-dimensional structure and less stable construction. Thus obtaining coatings with the desired properties on such materials creates many difficulties.

The textile barrier materials against electromagnetic fields (EMF) that are the subject of the research were developed at the Textile Research Institute - IW (Lodz, Poland). These materials are manufactured according to innovative technology consisting in depositing in a continuous way thin (in the nanometer scale) conductive layers onto textile media by magnetron sputtering (work on different textile barriers to EMF have been carried out at IW since 1998).

The medium is a textile product with a properly chosen structure, and the coating is metal or alloy, depending on the material shielding effectiveness

assumed. The significant element of novelty here is the use of the magnetron sputtering technique in such a way that the textile product obtained exhibits not only high electrical conductivity but also a suitably arranged “grid” of conductive paths formed by the metallised fibres.

The aim of this work was to define the surface morphology and crystallographic structure of coatings deposited from metallic alloys at various process parameters (e.g. effective power dissipated in the target, velocity of the substrate drift, number of cycles). Furthermore an attempt was made to determine the effect of these features (particularly of the crystallographic structure) on the EM shielding effectiveness of the “metallic layer – nonwoven” composites. It is expected that the results obtained will be useful in the selection of parameters for the metallisation process of polypropylene (PP) nonwovens.

Experiments, results and discussion

Plasma metallisation of PP nonwoven

Earlier studies indicated the possibility to obtain light, formable materials shielding against EMF by magnetron sputtering deposition of metal layers on textiles [4 - 7]. The Textile Research Institute (IW), as the first in Poland, developed an original device for depositing metal, alloy and oxide coatings by the magnetron sputtering technique on various textile products. This device, made according to the idea and construction guidelines of IW, enables “roll to roll” deposition of thin layers or multilayer systems on one or both sides of a textile tape with a width of 60 cm and a length varying from a few to several meters (**Figure 1**).

The substrate (textile tape) was a polypropylene nonwoven of the spunbond type of an area weight per unit of 150 g/m². Metallisation of the PP nonwoven was carried out by the magnetron sputtering of CuSn (the composition by weight: 80% Cu, 20% Sn) and CuZnNi (the composition by weight: 53.5 – 56.5% Cu, 25 – 30% Zn, 17 – 19% Ni) alloy targets. Because of their specific size and shape, the targets were made for the project on request. The principle of selection of the composition of the targets, as well as the textile substrate is the subject of a new patent application. Layer deposition of the exemplary processes

were conducted in an argon atmosphere (0.10 Pa) under the conditions listed in **Tables 1** and **2**, respectively. During tests, different velocities of the substrate drift as well as different effective power were applied. At the same velocity, different values of effective power were tested, or at the same or similar power, different velocities of the substrate drift were applied.

Surface morphology of metallised PP nonwoven

Microstructural characterisation of the samples was performed using scanning electron microscopy (SEM). SEM images (**Figures 2** and **3**) were obtained on a 5500 LV microscope (JEOL Company) equipped with X-ray microanalyser, Oxford Instruments. The microscope observations were conducted in a vacuum environment at a pressure equal to $2 - 5 \times 10^{-4}$ Pa. A voltage accelerating an electron beam (probe) of 20 kV was applied. Each of the images obtained for both types of materials includes information on the scale and magnification that was used during the measurements. Furthermore in the description of the results obtained by SEM, the term of “weave” has been introduced as the equivalent of the crossing points of the polypropylene fibres.

Images obtained for PP nonwovens coated with CuSn layers reveal, in each case investigated a three-dimensional structure of the substrate with well separated single polypropylene fibres. Similar results were achieved in earlier studies



Figure 1. Magnetron device (general view).

performed for the CuSn/PP- system [8]. Maintenance of the starting construction of the material in the weave region (see **Figure 2.a**, SEM image obtained for uncoated polypropylene nonwoven, as a reference sample) enables the covering of both surface fibres as well as those located deeper in the structure of the material. The exceptions are places where the “shadowing effect” of individual fibres occurs. It can be also presumed that the deeper coverage of fibres gets smaller and smaller, due to increasingly hindered penetration of the substrate by the metal particles. In order to reduce the “shadowing effect”, a specially shaped electrode facilitating the separation of fibres inside the nonwoven tape during its sliding under the target was applied in a magnetron

Table 1. Selected parameters of CuSn deposition processes; * Effective power – power generated directly onto sputtered material in intervals (pulse phases). Changing its value, one can modify the sputtering effectiveness. ** Flying power – a value which describes “mismatching” between the output resonant circuit of the feeder and the impedance of glow discharge. The value of flying power can be affected by the gas type and its composition, e.g. content of reactive gas, changes in total pressure, electric properties of reaction products covering the target surface, changes in the density of effective power resulting from variations in the target geometry due to its gradual etching. These definitions apply only to pulse feeding due to the feeder with a resonant circuit.

Sample	Velocity of the substrate drift, mm/s	Effective power*, kW	Flying power**, kW	Number of cycles
CuSn/PP-1	15	2.02 – 2.07	1.20 – 1.23	30
CuSn/PP-2	15	3.99 – 4.05	2.40 – 2.50	15
CuSn/PP-3	30	2.05 – 2.06	1.01 – 1.10	15

Table 2. Selected parameters of CuZnNi deposition processes; *,** - see **Table 1**.

Sample	Velocity of the substrate drift, mm/s	Effective power*, kW	Flying power**, kW	Number of cycles
CuZnNi/PP-1	15	2.00 – 2.03	0.58 – 0.60	15
CuZnNi/PP-2	50	1.90 – 2.10	0.50 – 0.60	15
CuZnNi/PP-3	50	2.02 – 2.07	0.68 – 0.72	40

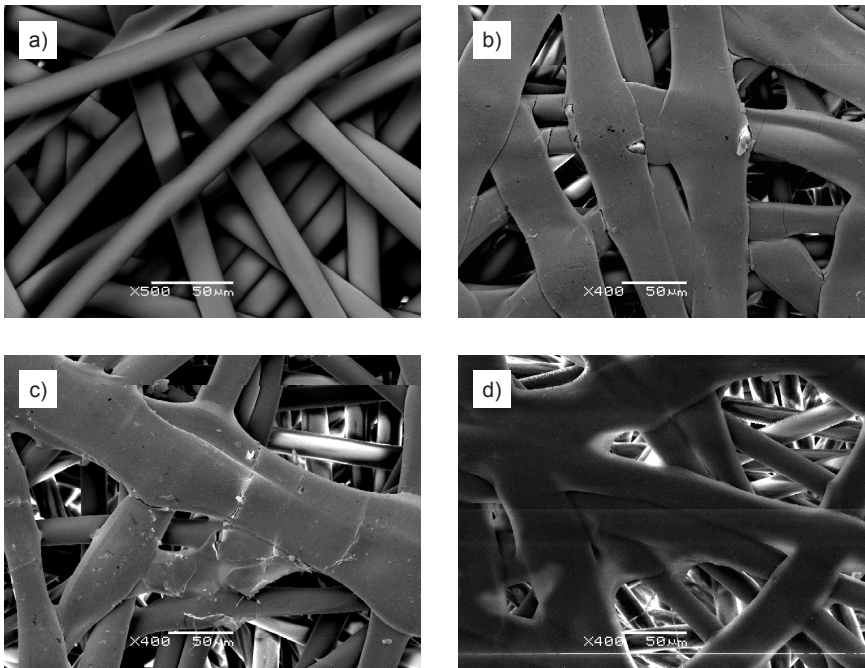


Figure 2. SEM images showing the surface of (a) PP nonwoven, (b) CuSn/PP-1, (c) CuSn/PP-2 and (d) CuSn/PP-3.

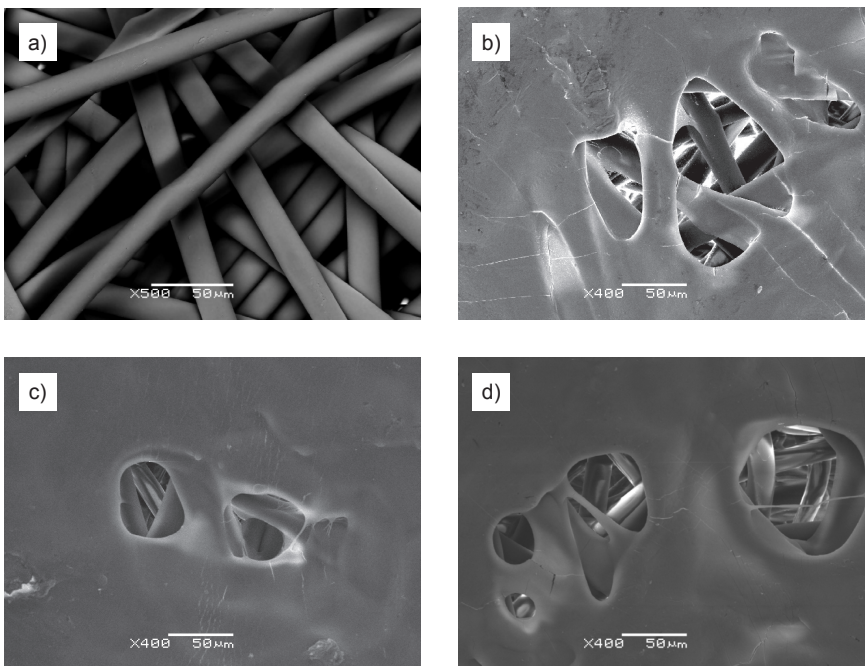


Figure 3. SEM images showing the surface of (a) PP nonwoven, (b) CuZnNi/PP-1, (c) CuZnNi/PP-2 and (d) CuZnNi/PP-3

device of own construction (*Figure 1*). However, despite the use of such a solution, the possibility of the presence of areas not covered by the CuSn layer (and thus non-conductive) cannot be completely ruled out. This is particularly related to the CuSn/PP-3 sample obtained at a high velocity of the substrate drift – 30 mm/s, which results in a smaller mass deposition of the sputtered material in a single cycle.

In *Figure 2.b - 2.d*, one can observe that surface fibres of the PP nonwoven have been partially deformed, which is particularly evident for CuSn/PP-1 and CuSn/PP-2. It may be assumed that applying lower velocity of the nonwoven drift (15 mm/s), and in the case of the CuSn/PP-2 sample also higher effective power of the target sputtering (3.99 – 4.05 kW), results in the increasing temperature of the PP substrate above its softening point. This effect is not so

obvious for the CuSn/PP-3 sample. Additionally the CuSn/PP-1 and CuSn/PP-2 metallic layers exhibit a few cracks extending perpendicular to the long axis of the fibre that arose due to tensile stresses. Studies carried out by other authors [9 - 12] on thin metal layers deposited onto polymeric substrates showed that the layers are capable of larger deformation under stress than the same material in the bulk. Amongst others, Lacour *et al.* [11] studied gold films on an elastomer substrate and observed that at about 8% elongation, small cracks appeared at the edges of the film, but did not propagate further into the film. Therefore one can assume that the cracks present in the CuSn layers, particularly those visible in *Figure 2.b*, do not cause the total disruption of the metallic layer continuity. Furthermore the absence of large areas of delamination might suggest good adhesion of all deposited layers to the substrate.

Similar to the metallic layers obtained from the CuSn alloy for a series of samples coated with CuZnNi, the surface morphology was depicted with a scanning electron microscope and compared with the morphology of the substrate – polypropylene nonwoven (see *Figure 3*). It was observed that during the CuZnNi deposition process, almost complete melting of polypropylene surface fibres occurred; hence the structure of the weaves now recalls the points of nonwoven thermal welding. In this case this effect appears to be independent of both the velocity of the substrate drift and the number of cycles. On the other hand, it might be connected with the high melting point of the alloy used for the CuZnNi target preparation. The CuZnNi/PP-1 and CuZnNi/PP-2 metallic layers are marked by numerous cracks and point defects. However, due to the surface melting of PP fibres, the damage would not have a significant impact on the continuity of their structure.

X-ray studies for CuSn metallic layers

Studies of the crystallographic structure and phase composition of the metallic layers deposited onto the polypropylene nonwoven substrate were carried out based on X-ray diffraction measurements. Grazing-incidence angle X-ray diffraction experiments were performed on a X'Pert MPD diffractometer (PANalytical, the Netherlands) in the 2θ range of 20 – 100 deg. The X-ray source was a copper X-ray diffraction tube operating at 45 kV and 40 mA.

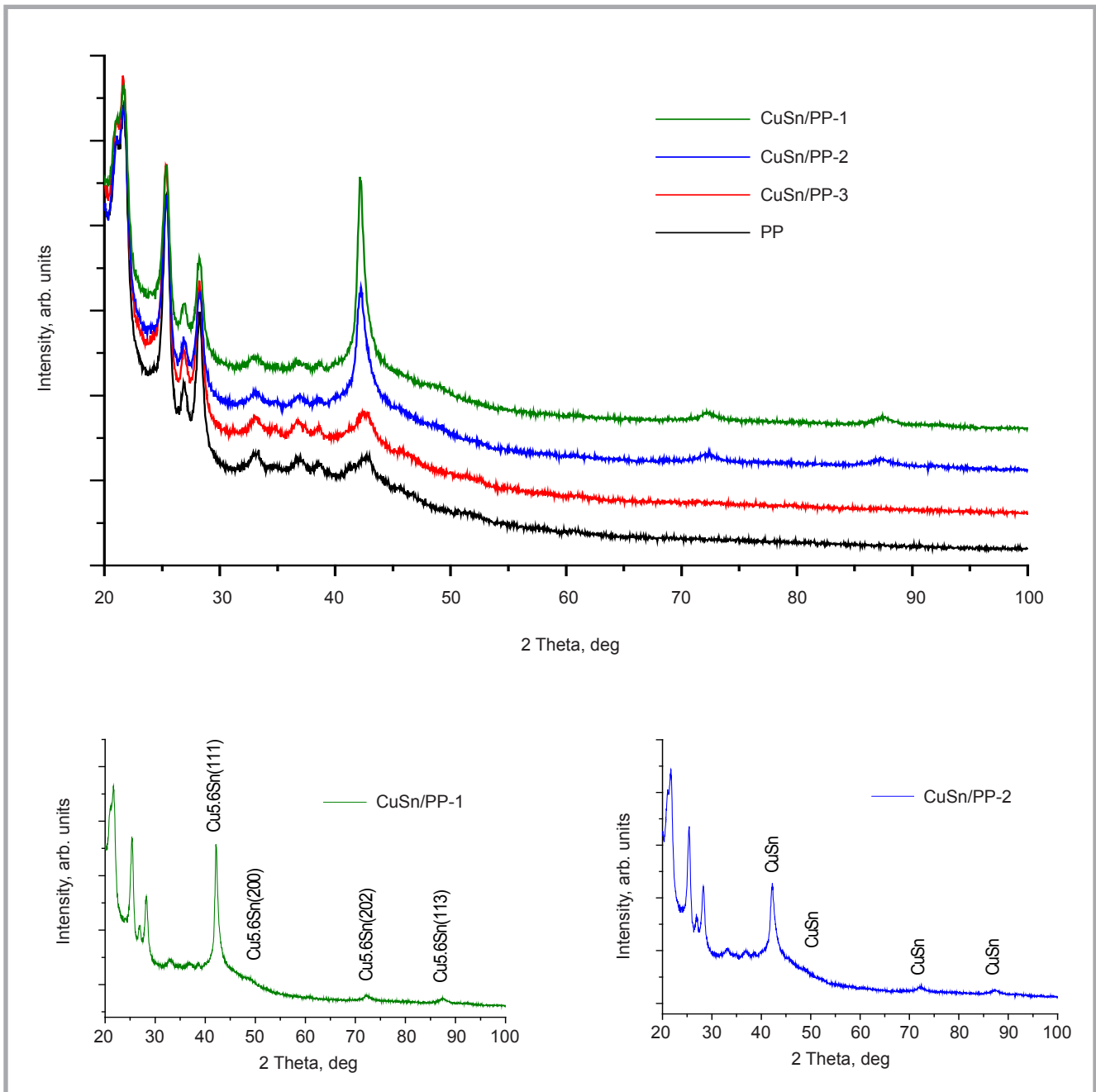


Figure 4. GI-XDR profiles obtained for CuSn metallic layers on PP nonwoven and uncoated PP nonwoven (as a reference sample).

A grazing incidence angle of 3 deg. was applied. During the measurements, parallel beam geometry, with a parabolic Cu mirror in the incident beam, was used. Such a configuration is particularly effective for measuring samples with an uneven surface. The span of the incident beam was 0.5 deg., and the width of the beam at the sample – 10 mm. Identification of the metallic layer phase compositions was carried out using the HighScore programme. Measurements were performed for both samples coated with metallic layers as well as for uncoated polypropylene nonwoven as a reference sample. In the case selected (CuZnNi/PP-1 sample), the size of crys-

tallites formed in the metallic layer was calculated using the Scherrer's formula. Half-widths of the diffraction peaks were obtained by fitting the diffraction patterns with the use of the ProFit 1.0c programme (Philips Electronics N.V.) The presence of maxima originated from both the metallic phase and PP substrate and were taken into account (the maximum in the diffraction pattern of the metallic layer/nonwoven composite was fitted as a superposition of the peaks originating from the metallic phase and PP).

Diffractograms obtained for the nonwoven coated with CuSn layers are shown

in **Figure 4**. The creation of the crystalline phase within the metallic layers is manifested by the presence of peaks located at angles higher than $2\theta = 40$ deg., while the signals recorded at 2θ between 33.14 and 42.70 deg. were identified as those originating from the structure of the substrate ordered (PP nonwoven). It was found that under the process conditions applied (shown in **Table 1**), the growth of crystallites occurred in the CuSn/PP-1 and CuSn/PP-2 layers, whereas in CuSn/PP-1 – crystallites of the Cu_{5.6}Sn phase. Additionally the X-ray microanalysis (EDX) carried out for CuSn/PP-1 showed the presence of

Table 3. Results of EDX microanalysis of CuSn/PP-1*; *The results obtained should be considered as an indication of the proportion between the various components of the layer. **It was assumed that a carbon contribution at a level of 6% may appear due to contamination of the compounds from the vacuum environment (the vacuum is generated with the use of an oil pump) and as the carbon compounds desorbed from the surface of the working chamber of the microscope.

Elements	Wt % (average values calculated from four measurements)	Wt % (average values calculated after correction for carbon contribution)
C	21.21	6.0**
O	2.10	2.51
Cu	63.54	75.82
Sn	13.15	15.67
Total	100.0	100.0

Table 4. Results of EDX microanalysis of CuSn/PP-2*; *,** - see Table 3.

Elements	Wt % (average values calculated from four measurements)	Wt % (average values calculated after correction for carbon contribution)
C	12.37	6.0**
O	1.58	1.69
Cu	73.31	78.65
Sn	12.74	13.66
Total	100.0	100.0

oxygen at a level of 2.5% by weight (see **Table 3**). The oxygen contribution leads to the hypothesis that there is a presence of the Cu₂O phase in the sample. The occurrence of this phase may result from a longer time of the deposition process (in this case, the number of cycles - 30).

The longer duration of action of heat radiating from the target toward the nonwoven substrate can favour the desorption of gases (including oxygen) that were not released from the nonwoven under the vacuum generated in normal temperature conditions.

The hypothesis of oxygen release from the substrate may be evidenced by the quantitative X-ray microanalysis made for the CuSn/PP-2 sample (see **Table 4**). It appears that the greater power of target sputtering and shorter exposure time result in a smaller content of oxygen (approximately 1.6% by weight) in relation to CuSn/PP-1. In both cases, one can observe a slight disproportion to the initial target composition, and a reduced level of Sn amounting approximately to 16% for CuSn/PP-1 and 14% for CuSn/PP-2. On the other hand, it is known that sputtering processes comprise two complementary mechanisms: kinetic, consisting in the extraction of target components from its surface, and thermodynamic - leading to the evaporation of components within the micro-regions. Therefore one of the reasons for tin depletion may be the difference in the vapour pressure of alloy components at the melting point, being higher for copper. Another may result from a reduction in the Sn to Cu ratio at the target surface during its sputtering, which is caused by a far greater share of copper [14]. Taking into account the above factors, which play an important role in the magnetron sputtering process, it can be concluded that, in fact, the qualitative and quantitative compositions of the CuSn layers deposited correspond well to the composition of the target used.

In the case of sample CuSn/PP-3, signals originating from the CuSn metallic layer were not observed during the X-ray studies. Therefore one can assume that the high velocity of the substrate drift, and thus the variation in the thermodynamic condition during deposition of the alloy components were not conducive enough for an effective nucleation process and/or for further growth of the crystallites.

Referring to the results of the surface morphology studies and assuming that the magnetron sputtering of the CuSn and CuZnNi alloys proceeded at a low temperature (less than 200 °C), which was confirmed by the low degree of polypropylene fibre deformation (in the extreme conditions, only their partial melting), and further based on the CuSn phase equilibrium system (see **Figure 5**), the occurrence of the eutectoid of the α + ε phases in the coatings studied cannot be also excluded.

Assessing the effect of the crystalline phases on the mechanical properties

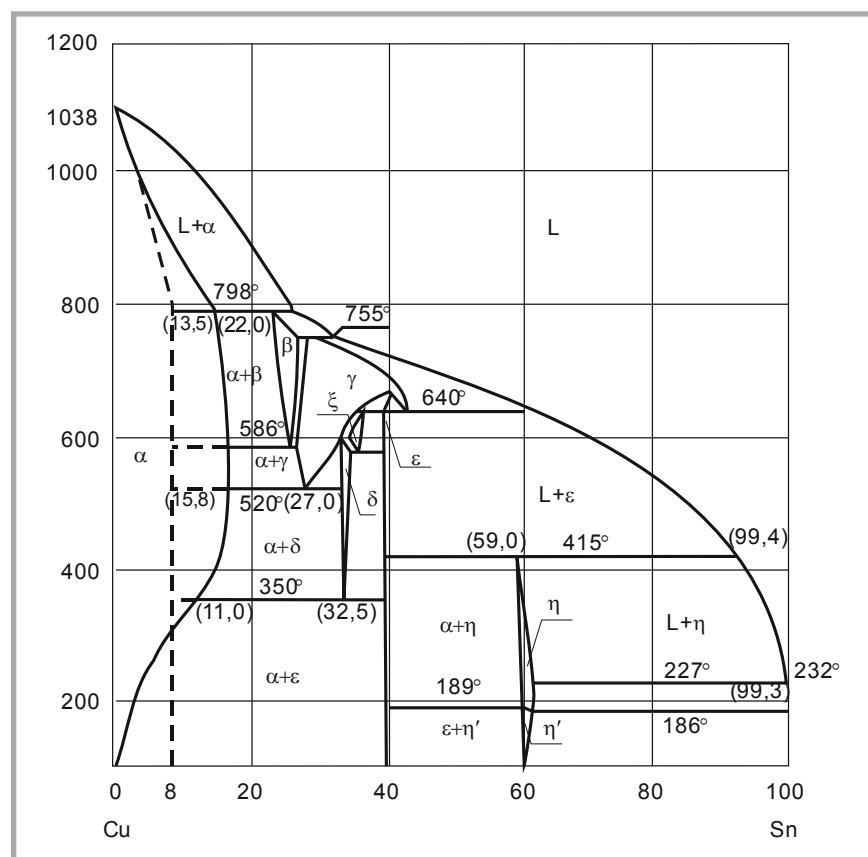


Figure 5. CuSn phase equilibrium system [12].

of the CuSn coatings, one can only assume that they cause a slight increase in the tensile strength, and thus would decrease the elasticity of the coatings. A spectacular confirmation of this hypothesis are cracks of the metallic layers of samples CuSn/PP-1 and CuSn/PP-2, which were disclosed in the study of the surface morphology (see **Figure 2**). These defects were probably formed due to the stress generated by the shrinkage and stretching of polypropylene fibres during the nonwoven drift in the deposition process.

Considering the structural properties of the coatings deposited on the polypropylene nonwoven as a result of CuZnNi target sputtering, one can conclude that a well-built crystalline phase is present only in the metallic layer of the CuZnNi/PP-1 sample (see **Figure 6**). With high probability, this feature can be associated with the presence of Cu crystallites. For this sample, the velocity of the substrate (nonwoven) drift was more than three times smaller – 15 mm/s compared to 50 mm/s for the CuZnNi/PP-2 and CuZnNi/PP-3 samples. Leaving the nonwoven for a longer time in the target sputtering zone favours the deposition of a higher mass of the spattered material. At the same time, a longer cycle time, due to the slower velocity of the substrate drift, promotes the initiation of nucleation and growth of crystallites, whose size (assessed on the basis of the diffraction patterns) reaches up to 8 nm. Such conditions are not provided in the case of increased velocity of the nonwoven drift, where the mass of the material deposited in one cycle is lower, and the cycle time is insufficient for crystal growth. Increasing the number of cycles (40), as took place in the case of the CuZnNi/PP-3 sample, led only to the emergence of small Cu crystallites, as evidenced by slightly sketched signal strengthening visible above the angle $2\theta = 40$ deg. in the diffraction pattern obtained.

Additionally, considering the energy losses during CuZnNi/PP-1 deposition, evidenced by the value of the flying power, which equals 25% in relation to that of the effective power (**Table 2**), one can assume the occurrence of partial and/or momentary target “poisoning”. As a result of this phenomenon, oxides are formed on the target surface (copper, in particular, as the most reactive target component). The resulting oxides, on the one hand, narrow the area of active sputtering, but on the other make it

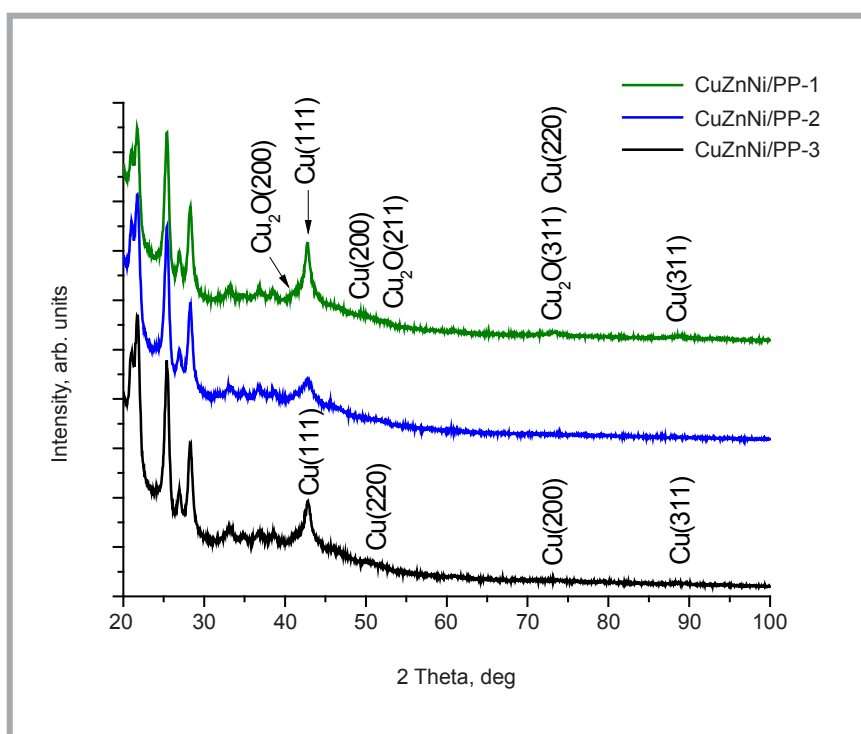


Figure 6. GI-XDR profiles obtained for CuZnNi metallic layers on PP.

possible to deposit a layer with a contamination of small Cu₂O crystallites. In the case of increased velocity of the substrate drift (such as for the CuZnNi/PP-2 and CuZnNi/PP-3 samples), the phenomenon of target “poisoning” can also occur, but there are no favourable conditions for nucleation and growth of crystalline structures – lack of clear signals on the diffraction patterns (**Figure 6**).

Effect of CuSn crystalline structure on EM shielding effectiveness of metallised PP nonwoven

The EM shielding effectiveness (EM SE) was studied at Wrocław University of Technology according to the modified ASTM D4935-99 method [15]. Results of the measurements chosen and presented in **Tables 5 & 6** and **Figures 7 & 8** (see page 90, copies of the results from the measuring device) show exemplary plots of EM SE in the full range of frequencies tested.

Table 5. EM shielding effectiveness for CuSn/PP samples registered at two different frequencies.

Sample	EM SE, dB	
	27.12 MHz	1795 MHz
CuSn/PP-1	45	39
CuSn/PP-2	44	38
CuSn/PP-3	25	20

The above-described studies of the crystalline structure revealed that in the case of samples obtained from the CuSn alloy, the application of a larger number of cycles – CuSn/PP-1 or larger target sputtering power (effective power) – CuSn/PP-2 at the same low velocity of the substrate drift promotes the growth of crystallites within CuSn layers. The shielding properties of these samples are almost two times better with respect to CuSn/PP-3 in both ranges of EMF frequency investigated (27.12 MHz and 1795 MHz). This is probably due to the presence of the crystalline phase in these coatings, which was not found within the CuSn/PP-3 metallic layer.

Similarly for samples with CuZnNi metallic layers, the best shielding properties were obtained for CuZnNi/PP-1, during the preparation of which the most favorable process parameters for the formation and growth of the Cu crystalline phase were applied.

Table 6. EM shielding effectiveness for CuZnNi/PP samples registered at two different frequencies.

Sample	EM SE, dB	
	27.12 MHz	1795 MHz
CuZnNi/PP-1	37	31
CuZnNi/PP-2	24	19
CuZnNi/PP-3	28	22

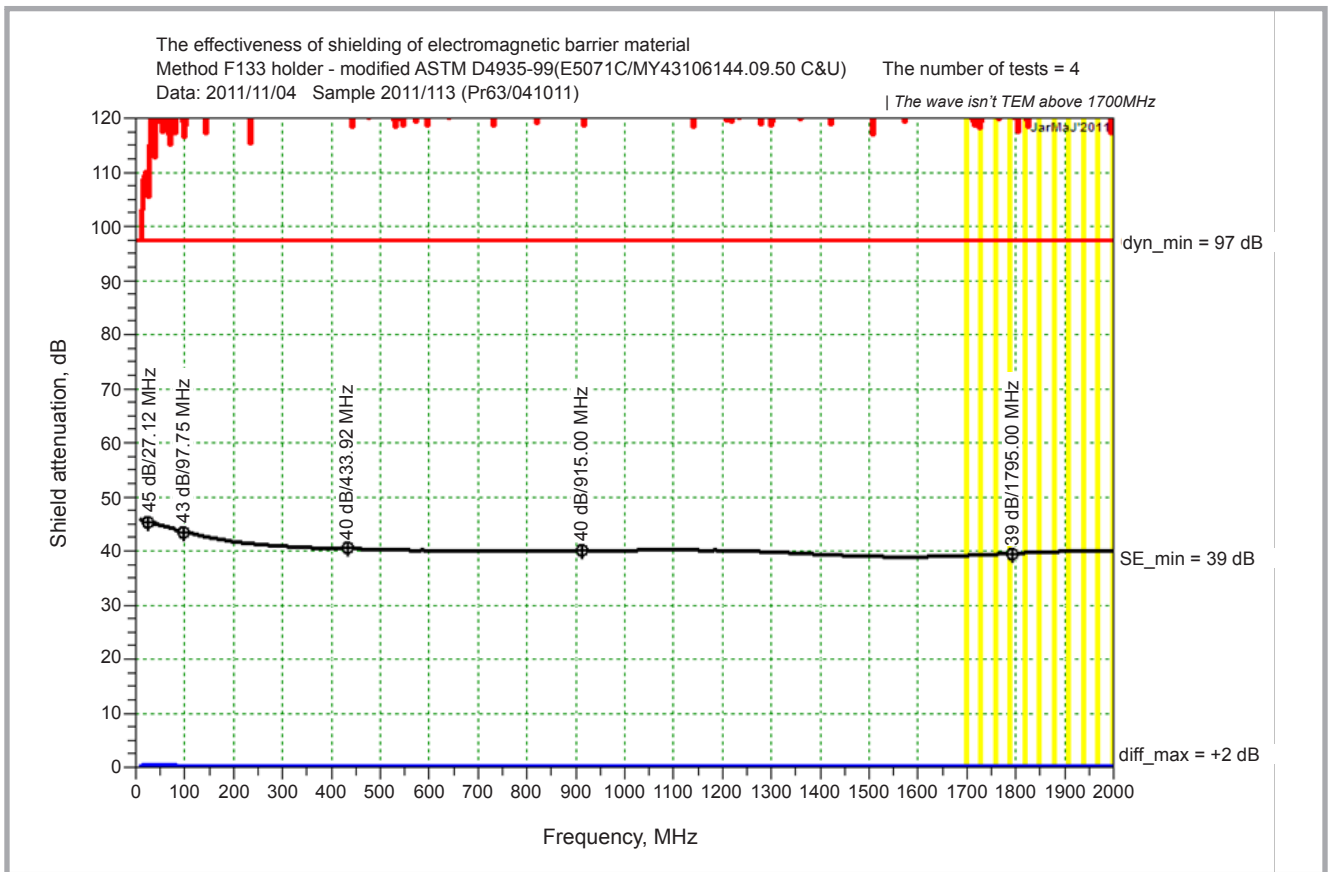


Figure 7. Shielding effectiveness of the CuSn/PP-1 sample.

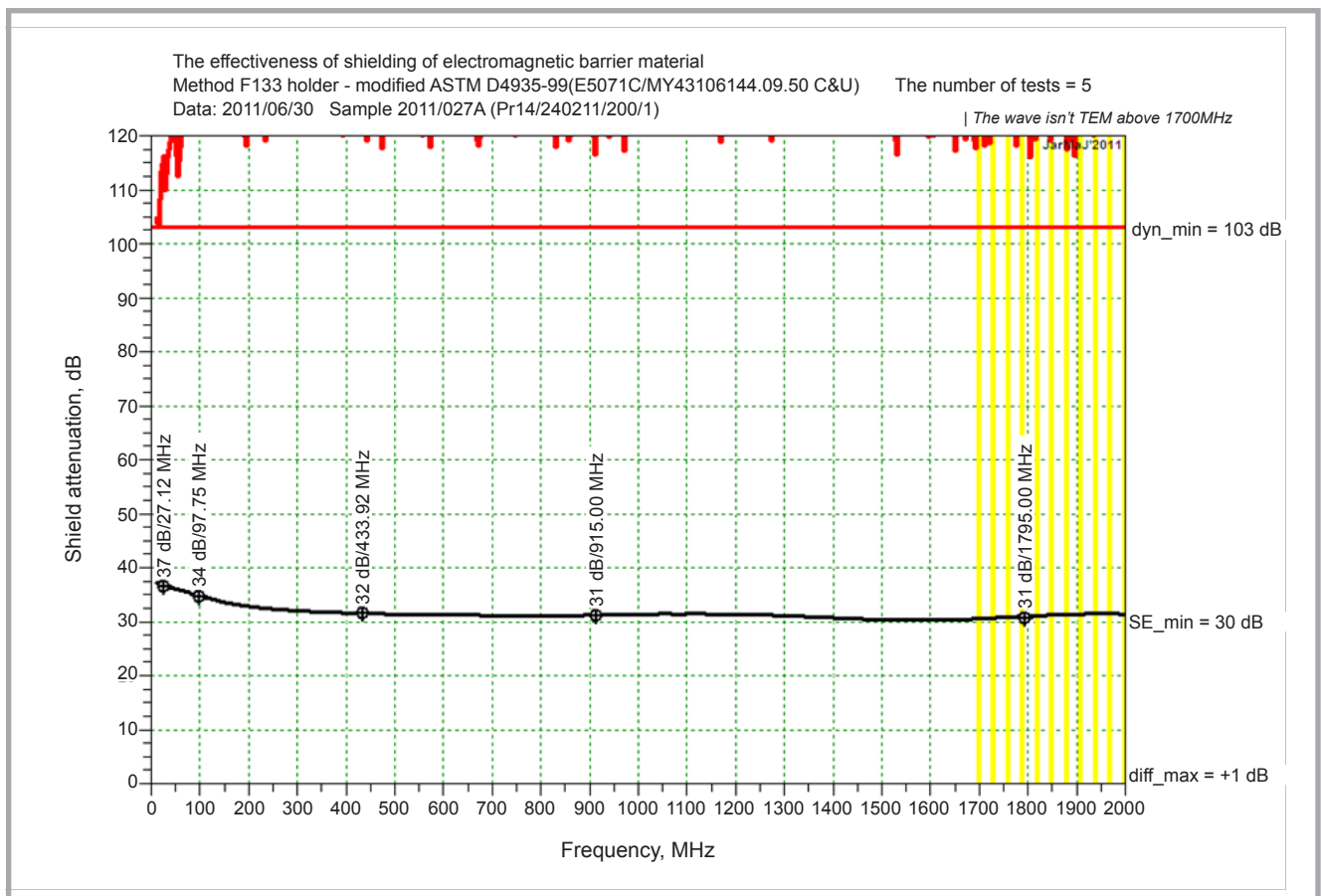


Figure 8. Shielding effectiveness of the CuZnNi/PP-1 sample.

Basic differences in the EM shielding effectiveness between the two types of coatings (CuSn, CuZnNi) result from their material composition. Other properties of the shielding materials presented in this article will be the subject of further publications.

Conclusions

On the basis of the morphology and crystalline structure studies performed for CuSn and CuZnNi metallic layers deposited onto a PP nonwoven substrate, it was found that:

- there are no large areas of delamination of the coatings deposited that might suggest good adhesion of all metallic layers to the substrate,
- the samples with metallic layers for which the process parameters applied were favorable for the nucleation and growth of the crystalline phase (CuSn/PP-1, CuSn/PP-2, CuZnNi/PP-1) exhibit better shielding properties than the other samples,
- metallic layers CuZnNi/PP-1 and CuZnNi/PP-2 are marked by numerous cracks and point defects. This damage would not have a significant impact on the continuity of the layers' structure, and thus on their conductivity. However, for much larger areas than those observed under a microscope, the presence of such defects (together with the impact of individual layer components) results in lower EM SE values than those obtained for samples with CuSn layers.
- The studies conducted justify the differences in EM SE values obtained for the barrier materials developed and may be helpful in verification of the parameters used during the magnetron sputtering processes.



Acknowledgments

The research was carried out within the key project – PO IG no. 01.03.01-00-006/08 acronym ENVIROTEX, co-financed from funds of European Regional Development Fund within the framework of the Operational Programme Innovative Economy 2007-2013.

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Received 09.12.2013 Reviewed 09.03.2015

Institute of Textile Engineering and Polymer Materials



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