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TEST REPORT 1/2018

Testing of ADR®TEX electromagnetic field shield properties
at room temperature

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The results of the test apply to the test objectives only.

1. Test Objective

The test objective was to determine the properties of ADR®TEX electromagnetic field (EMF) shielding at room temperature (RT). The surface properties of ADR®TEX were studies using optical and scanning electron microscopy. Dielectric absorption in the frequency range from 1×10^{-1} Hz to 2×10^{6} Hz was measured for an ADR®TEX sheet placed between two dielectric spacers in a measuring condenser at room temperature in the relative air humidity of 62%. The samples of the ADR®TEX shield were supplied by ADR Technology Stanislaw Wosiński.

2. Material

The ADR®TEX shield is based on ADR®Technology and is a complex nano/microcomposite. The basic matrix is a fabric woven from three kinds of threads: Rayon (viscose), Nylon (polyamide 66) and Schöller thread (PES yarn with 20% metallic filling) in respective weight ratio of 44:36:20. After weaving (broken twill weave) the fabric was soaked with ADR®Sol, dried in air and reached the basic weight of 110 g/m.

3. Measuring Methods and Instrumentation

3.1. Optical Polarizing-Interference Microscopy (Zeiss) in both transmission and reflection mode was used to image the surface of the ADR®TEX shield.

3.2. Scanning Electron Microscopy (SEM) microimaging of the ADR®TEX shield with a FEI Nova NanoSEM apparatus was used to give information on the surface potential distribution. The experiments were performed in a high vacuum (HV) with a low incident of electron energy.

3.3. Energy Dispersive X-Ray Spectroscopy (EDS) experiments for elemental microanalysis and mapping of ADR®TEX shield were made with a FEI Nova NanoSEM equipped with an EDS sensor.

3.4. Dielectric Losses. Dielectric material is characterized by relative complex dielectric permittivity: $\varepsilon^* = \varepsilon' - j\varepsilon''$ where 'relative' refers to the normalization by the permittivity of free space ε_0 = 8.85×10^{-12} F/m. Dielectric losses: $\varepsilon'' = \varepsilon''_p + \frac{\sigma_{dc}}{\varepsilon_0 f}$ are sum of dielectric polarization losses ε''_p and losses $\frac{\sigma_{dc}}{\varepsilon_0 f}$ due to the Ohmic conduction (σ_{dc} denotes the dc conductivity and f is the frequency of measuring field). The dielectric permittivity ε^* as well as the dielectric loss tangent: $\tan\delta(f) = \frac{\varepsilon''(f)}{\varepsilon'(f)} = \frac{\left(\varepsilon''_p(f)+\frac{\sigma_{dc}}{\varepsilon_0 f}\right)}{\varepsilon'(f)}$ are both dependent on the measuring frequency f.

Dielectric response of the samples was studied using an Alpha-A High Performance Frequency Analyzer (Novocontrol GmbH). ADR®TEX sample was placed between two dielectric spacers in a measuring condenser tanδ measurements were performed in the air of relative humidity 62%, at room temperature (293 K) and the frequency was scanned from 10^{-2} to 10^{7} Hz at the oscillation voltage of 1 V. The measured dielectric permittivity data were collected and evaluated by WinDETA impedance analysis software and a WinFit V 3.2. Program.

3.5. Magnetic Properties. Magnetic hysteresis loops of ADR®TEX shield samples were measured with a Physical Property Measurement System (PPMS, Quantum Design) in the temperature range 290 – 300 K. The hysteresis loops were displayed at constant temperatures, stabilized to within ± 1 K.

4. Results

4.1. Optical Polarizing-interference Microscopy

Fig. 1 and 2 show optical microscope imaging of ADR®TEX shield sample in the reflection mode and transmission mode, respectively. It appears that the weaving of the shield is of broken twill weave type.

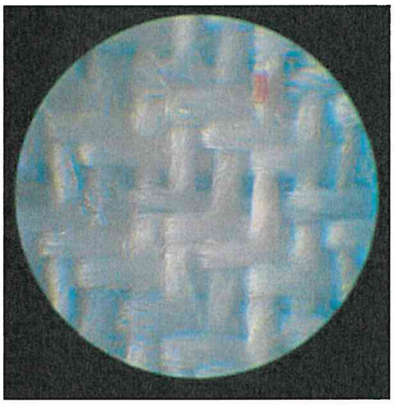

Fig. 1. Optical microscopy image of an ADR®TEX shield in reflection mode. Magnification 200× (Zeiss polarizing-interference microscope).

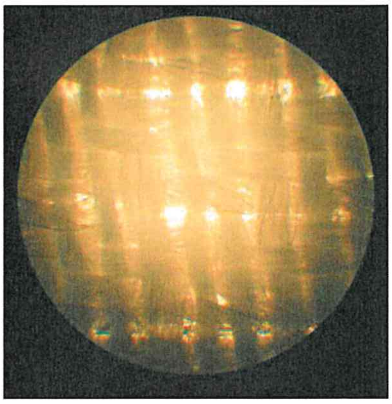

Fig. 2. Optical microscopy image of an ADR®TEX shield in transmission mode. Magnification 200× (Zeiss polarizing-interference microscope).

4.2. Scanning Electron Microscopy Imaging

ADR®TEX shield is based on ADR®Technology and is a complex nano/microcomposite. The basic matrix is a fabric woven from three kinds of threads: Rayon (viscose), Nylon (polyamide PA66) and Schöller thread (PES yarn with 20% metallic filling) in respective weight ratio of 44:36:20. **Viscose rayon** is a fiber of regenerated cellulose and can be produced from a variety of plants (soy, bamboo, and sugar cane). Cellulose is a linear polymer of β-D-glucose units with the empirical formula $(C_6H_{10}O_5)_n$.

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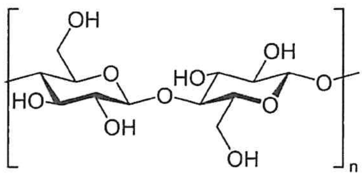
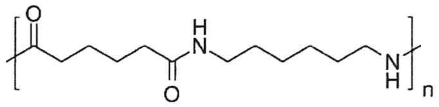


Polymers/copolymers obtained by reacting difunctional monomers containing equal parts of amine and carboxylic acid are termed Nylons. The most common Nylon for textile is **PA66** consists of two monomers each containing 6 carbon atoms: hexamethylenediamine and adipic acid.



Synthetic polymer made of purified terephthalic acid (PTA) or its dimethyl ester dimethyl terephthalate (DMT) and monoethylene glycol (MEG) is termed polyester (PET, **PES)** with the sum formula of H-[$C_{10}H_8O_4$]-n=60–120 OH and mol. unit weight: 192.17.

Fig. 3. SEM image of ADR®TEX (E_e = 2 keV). Viscose, polyamide and PES threads are visible in bright contrast, black contrast is due to metallic filer of Schöller thread. (FEI Nova NanoSEM).

4.3. Energy Dispersive X-Ray Spectroscopy

Energy dispersive X-ray spectroscopy (EDS) was used to obtain information on distribution of various elements within the ADR®TEX shield. The fabric was soaked with ADR®Sol, dried in air and reached the basic weight of 110 g/m. As discussed in 4.2. all kinds of threads (viscose, PA66 and PES) are built of carbon atoms which are visible in the microanalyse image as having green contrast. In Fig. 5. EDS shows green contrast of carbon atoms in viscose rayon, PA66 and PES together with the magenta, yellow and blue contrast of chromium, iron and nickel atoms, respectively in the Schöller yarn.

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Fig. 4. EDS imaging of carbon (green) in ADR®TEX shield. Carbon is the element having the greatest impact on the molecular structure of viscose rayon, PA66 and PES.

Fig. 5. EDS imaging of carbon C in (Rayon, PA66 and PES) and chromium Cr (magenta), iron Fe (yellow) and nickel Ni (blue) in ADR®TEX shield.

Distribution of metal elements within the ADR®TEX shield is shown in Fig. 6. The chromium, iron and nickel thin wires are placed along the PES yarn and in the fabric one can observe the wires broken into 40 – 400 µm long pieces.

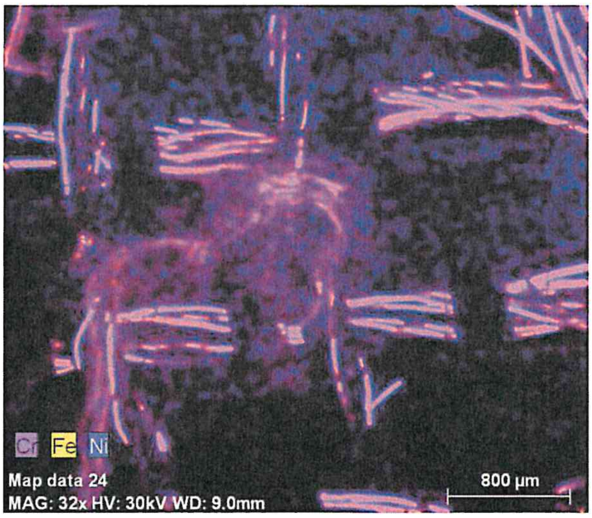

Fig. 6. EDS imaging of chromium Cr (magenta), iron Fe (yellow) and nickel Ni (blue) in ADR®TEX shield.

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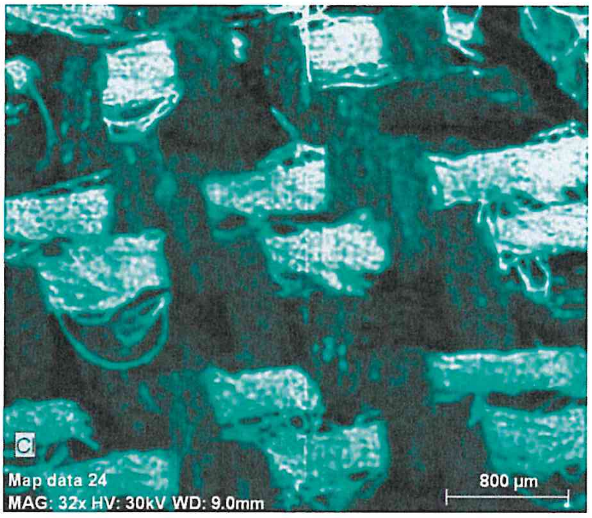

Fig. 7. EDS imaging of chlorine Cl (cyan) in ADR®TEX shield.

The presence of chlorine atoms is related to $MgCl_2$ hydrated salt contained in ADR®Sol used to soak the Rayon/Nylon/Schöller thread matrix. It appears that the Cl atoms are attached to the PA66 threads.

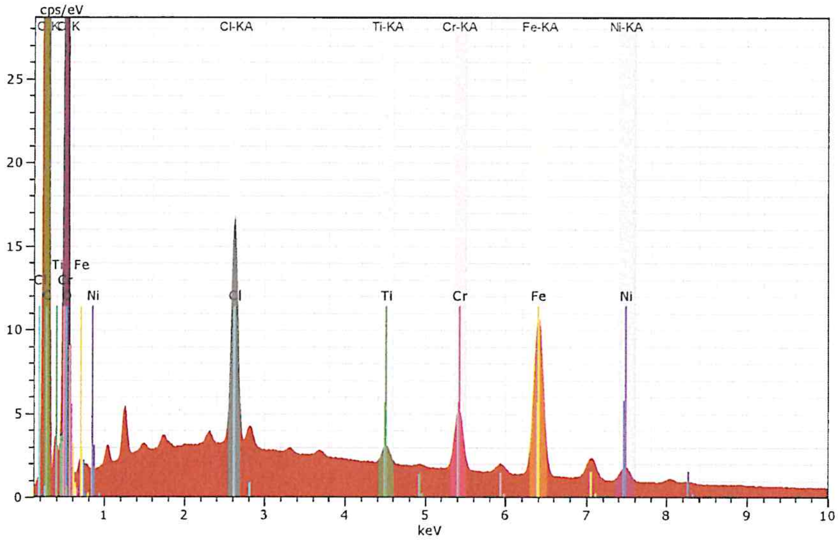

Fig. 8. Energy Dispersive X-ray Spectrum of a sample of ADR®TEX shield.

4.4. Dielectric Losses

Dielectric properties of ADR®TEX shield have been measured in radiofrequency range. A sample of ADR®TEX shield was placed between two dielectric spacers in a measuring condenser and frequency dependence of dielectric losses was scanned at room temperature in the 1-st, the second and the third run (each lasted 30 min) in the air of relative humidity 62%. Fig. 9 shows the dispersion of tanδ of the ADR®TEX shield obtained in sequent measurements together the frequency dependence of tanδ of the dielectric spacer.

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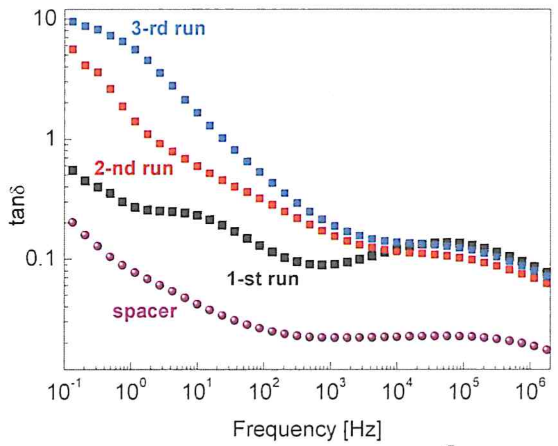

Fig. 9. Room temperature frequency dependence of tanδ for a sample of ADR®TEX shield placed between two dielectric spacers. tanδ dispersion of the dielectric spacer is also apparent.

The results obtained in the 1-st run measurements yield information on the dielectric absorption widows only: below 1 kHz absorption related to water dispersed in the composite and intrinsic dielectric absorption in the range 10 kHz – 1 MHz.

To yield more information on the origin of the intrinsic dielectric absorption of AD®TEX shield we measured frequency dependence of tanδ of a fabric woven from two kinds of threads: Rayon (viscose) and Nylon (polyamide 66) in weight ratio of 55:45 without the Schöller thread (PES yarn with 20% metallic filling). After weaving (broken twill weave) the fabric was soaked with ADR®Sol, dried in air. Fig. 10 shows the dispersion of tanδ of an EMF shield made from Rayon/Nylon 55/45 fabric with ADR®Technology. One can observe a broad maximum centered at ~1 kHz, which is due to water dispersed in the Rayon/Nylon/ ADR®Sol composite. Thus, dielectric absorption in the frequency range 10 kHz – 1 MHz of the ADR®TEX shield (Fig. 9) is an intrinsic feature related to the presence of the Schöller thread (PES yarn with 20% Cr/Fe/Ni filling).

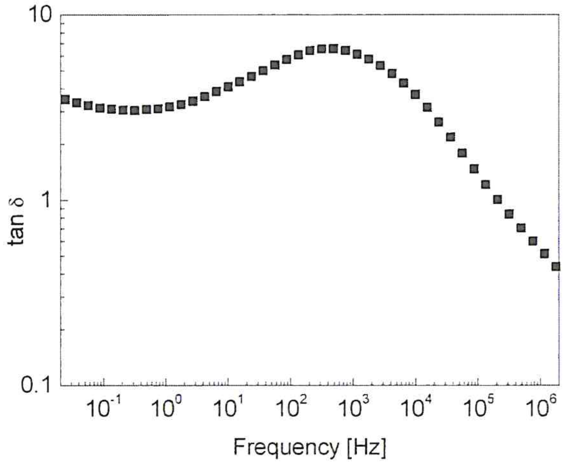

Fig. 10. Room temperature frequency dependence of tanδ for a sample of Rayo/Nylon 55/45 fabric shield made with ADR®Technology.

4.5. Magnetic Properties

As apparent in Fig. 5 and Fig. 6 the ADR®TEX shield should exhibit magnetic properties. Fig. 11 shows ferromagnetic hysteresis loops of an ADR®TEX shield in the temperature range 290 – 310 K.

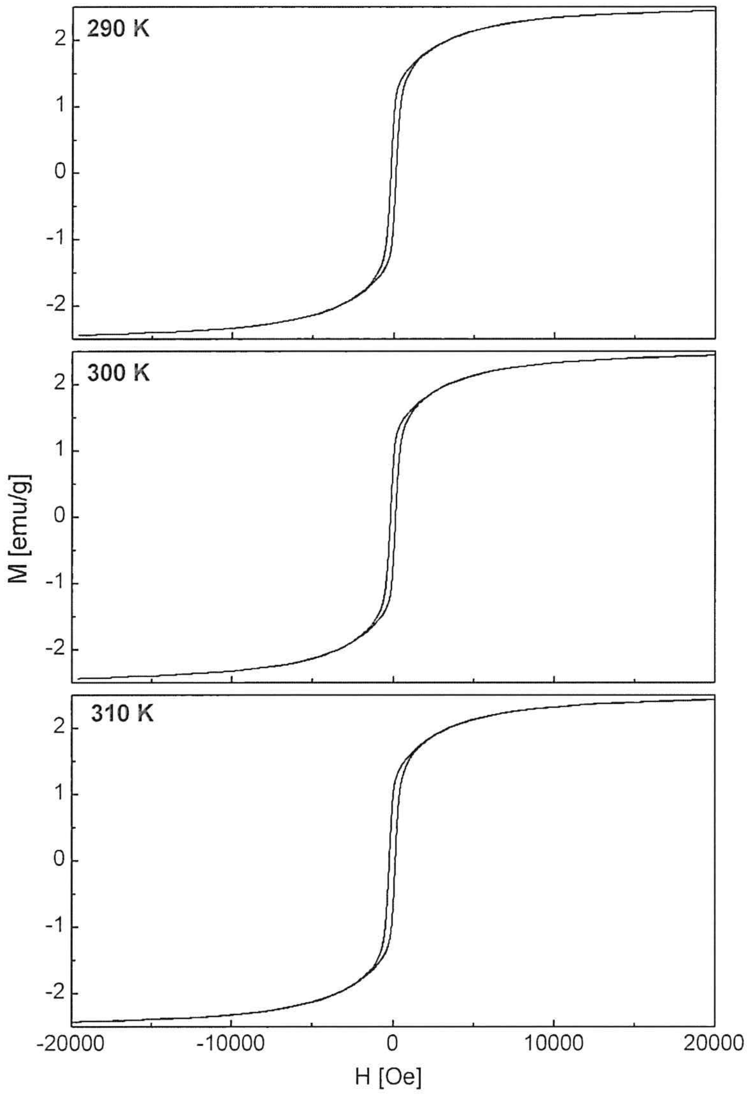

Fig. 11. Ferromagnetic hysteresis loop of an ADR®TEX shield at 290, 300 and 310 K. (Physical Property Measurement System, Quantum Design).

One can observe that ADR®TEX shield exhibits magnetic properties characteristic of soft ferromagnetic material with remnant magnetization of M_r = 0.83 emu/g and coercivity of H_c = 160 Oe at room temperature. A small increase in M_r and H_c values was observed on heating to 310 K (Fig. 12.). The saturation magnetization of ADR®TEX in magnetic field of $\mu_0 H$ = 2T amounts to M_{2T}= 2.47 emu/g at room temperature.

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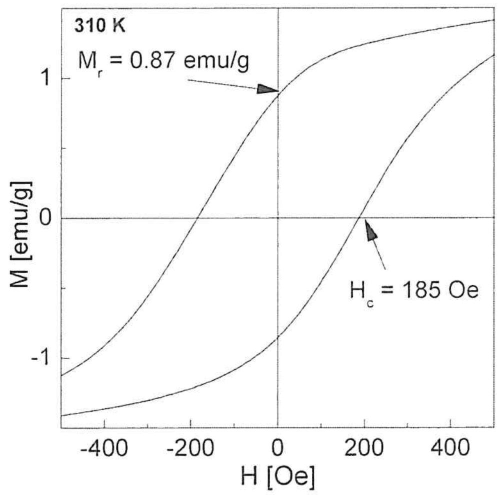

Fig. 12. Ferromagnetic hysteresis loop of an ADR®TEX shield. (Physical Property Measurement System, Quantum Design).

5. Conclusions

Evaluation of the results of characterization of ADR®TEX shield described above yields the following conclusions:

i) The ADR®TEX shield is a composite with a Rayon/Nylon/ Schöller thread (PES yarn with 20% metallic filling) fabric as a matrix which is soaked with ADR®Sol.

ii) Optical and scanning electron microscopy imaging reviled a broken twill weave of the fabric.

iii) Energy Dispersive X-ray Spectrum of carbon atoms in Rayon (viscose), Nylon (Polyamide 66) and PES (polyester) enabled us to imagine the the distribution of the threads.

iv) EDS imagine revealed the distribution of chromium, iron and nickel atoms within the Schöller thread. The thin metal wires are placed along the length of PES yarn and in the fabric 40 – 400 μm long metal pieces are apparent.

v) The presence of Cr, Fe and Ni is responsible for soft ferromagnetic properties at room temperature (magnetic remanence of M_r = 0.83 emu/g and coercivity of H_c = 160 Oe).

vi) Dielectric absorption studies of ADR®TEX shield reviled two absorption windows in the radiofrequency range. Below 1 kHz dielectric absorption of the ADR®TEX shield is due to water dispersed in the composite and intrinsic dielectric absorption in the range 10 kHz – 1 MHz may be related to the properties of the ADR®TEX matrix.

vii) We recommend a direct measurements of the screening efficiency of the ADR®TEX shield with ESM-100 3D H/E by Maschek Elektronik.

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