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# Investigation of thermophysical properties of thin-layered paint

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## ABSTRACT

The present study reports an investigation of the thermophysical properties of a camouflage coating paint (brand name: EC Paint) applied in thin layers onto a sample substrate. Since the thin-film form of the specimen substantially impedes traditional thermophysical measurements, a dedicated investigation procedure was developed to address this issue. This tailored approach comprises: SEM characterisation; density determination; DSC measurements; laser flash experiments; and a numerical parameter estimation procedure for determining thermal conductivity/diffusivity transversal to the coated surface. Experimental studies were carried out on multi-layered specimens of the EC Paint deposited onto molybdenum (Mo) or aluminium (Al) foil substrates of known thermophysical properties. The key measurements of thermal transport properties were performed on paint layers of varying thickness. In addition, the supporting gravimetric and calorimetric experiments were performed at temperatures ranging from about 20–120 °C. The resultant values of thermophysical properties of the EC Paint layer fall within the range typical for insulators. Furthermore, the study also revealed the presence of a moisture sorption effect, which has been further characterised. Finally, the obtained qualitative and quantitative results confirmed the correctness and effectiveness of the elaborated procedures.

## 1. Introduction

Knowledge of the thermophysical properties of thin films such as camouflage paints/surface coatings is critical for a wide range of their applications [1]. The two complementary parameters necessary for a complete thermophysical description are: specific heat (alternatively, heat capacity) $c_p$, which characterizes heat accumulation phenomena; and thermal conductivity,[1] which characterizes heat transport phenomena [2]. By combining the two above-mentioned quantities with density, a third parameter can be defined: thermal diffusivity [3]. However, typical thermophysical measurements are challenging to perform on thin film specimens. Thermal transport properties are particularly difficult to determine, and especially the values of cross-plane (out of plane) thermal diffusivity, because of extremely low values of the characteristic dimension of the studied specimens (that is, layer thickness) [4–8]. Since the mid-1960s, when the effect of layer thickness on thermal transport properties was first revealed [9,10], it has been well known that thermal conductivity of a thin layer can be substantially different from that of the bulk material. Depending on the type of the material, the effect can be attributed to: decreased mean free path of different carriers (electrons, phonons etc.[2]); structure defects at the layer boundary; interfacial thermal resistance; etc. [7,8]. In addition, for non-homogenous materials of thicknesses ranging from a few up to tens of microns, the complexity of structure becomes a significant factor in shaping their thermophysical properties. A growing interest in studying the above-mentioned phenomena and an increasing demand for thermophysical characterisation of thin-layered structures have resulted in the development of dedicated measurement methods [1,7,11–17]. However, the two most powerful of these techniques − 3 ω and thermoreflectance – are burdensome regarding specimen preparation and require sophisticated, often custom-designed instruments. Even when commercial apparatus is available [18–20], measurements are difficult to perform, especially when the specimens under study are irregular in planar shape or of non-homogeneous composition. Fortunately, if the specimen thickness is around tens of microns some of the traditional techniques of thermal transport measurements can be accommodated for thin specimen investigation [6,21–23]. In the case of a very popular laser flash (LF) methodology, the classical Parker approach [24–27] can be adapted for such measurements by applying more complex multi-layer analytical models for the inverse parameter estimation procedure [28–35]. The alternative method is to employ numerical simulations of the thermal response of a multi-layer

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[1] Alternatively - apparent/effective thermal conductivity, when non-homogeneous structures are regarded or coupled heat transfer phenomena occur.

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specimen to infer said parameters. [36–40]. The latter of the above-described approaches had been successfully applied by the authors before, in a study of the thermal diffusivity of black coating [41]. A similar procedure was therefore applied in the present study to determine the thermal diffusivity of a camouflage colorant, the EC Paint.

The investigated paint is used primarily to provide the coated surfaces with certain desired reflectance properties at visual and near-infrared spectrum [42]. The EC paint is typically delivered in 400 ml pressurised cans, to be applied by spraying [42]. This results in layers of approximately 10–50 μm thickness, which renders it suitable for the modified LF technique. Since it is difficult to obtain a free-standing layer of the paint, the substance was investigated after deposition onto metal foil substrates. In order to obtain the necessary data for the parameter estimation routine and to complement the material characteristics, additional measurements had been performed prior to main studies. These were: density measurements, microcalorimetric heat capacity measurements, microstructural and thermogravimetric (TG) investigations. The combined results of thermal diffusivity, specific heat and density measurements were further employed to calculate the thermal conductivity of the EC Paint.

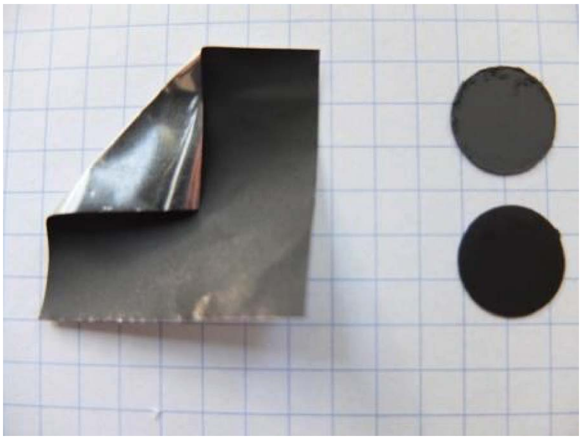

**Fig. 1.** View of the investigated specimens of the EC Paint deposited onto the 11.25 μm Al foil (on the left; gravimetric, TG and DSC measurements) and 96 μm Mo foil (on the right; LF studies).

**Table 1**
Mo and Al foil data: thickness and density measured, specific heat – room temperature data from [43].

| Material | Thickness μm | Density kg m$^{-3}$ | Specific heat J kg$^{-1}$ K$^{-1}$ |
|---|---|---|---|
| Mo | 96 | 9999 ± 168 | 251 |
| Al | 11.25 | 2656 ± 154 | 896 |

## 2. Experimental

### 2.1. Specimen preparation and structural imaging

The EC Paint was investigated after its deposition onto a metal foil substrate (Fig. 1) of known physical and thermophysical properties (listed in Table 1). Two substrate materials were employed, depending on the type of measurements performed: a stiff 96 μm thick molybdenum foil; and a pliable 11.25 μm thick aluminium foil. The aluminium foil was utilized for specimen preparation for gravimetric, thermogravimetric and differential scanning calorimetric (DSC) measurements. All of the experiments listed above were performed on specimens of a uniform thickness of the paint layer. The molybdenum foil was used for specimen preparation for thermal diffusivity measurements. For the purpose of these experiments, six composite specimens were prepared of incrementally increased paint layer thickness (note that even the thinnest EC Paint layer required several sprayings). In addition, the opposite, laser-facing side of the molybdenum foil was coated with a layer of black conductive coating, GRAPHIT 33, in order to ensure appropriate laser flash absorbance.

Layer thickness uniformity was controlled by both micrometric measurements and by scanning electron microscopy (SEM) imaging. SEM inspections were performed using the Hitachi TM3030Plus apparatus working in energy dispersive X-ray spectroscopy (EDX) mode with 15 kV accelerating voltage, standard charge-up reduction, and a magnification of 1000 (Fig. 2a) or 800 (Fig. 2b). This microscope is equipped with highly sensitive low-vacuum secondary electron detector, capable of revealing fine sample surface detail information. Inspection of the resulting images revealed a disperse composite structure of the paint bulk and showed that the front surface is rugged.

### 2.2. Gravimetric and thermogravimetric measurements

All gravimetric measurements were performed with an AT261 Delta Range Mettler-Toledo analytical microbalance, providing 0.01 mg resolution. The balance equipped with a Density Kit was also employed to determine the density of aluminium and molybdenum foils, as reported in Table 1. These measurements were performed using the buoyancy technique, with distilled water applied as an immersing fluid. The density of the EC Paint was derived from gravimetric and dimensional measurements of an EC Paint/Al foil specimen. Thickness measurements were performed with a micrometer on folded strap specimens after the appropriate weight measurements. Calculations of the EC Paint density resulted in the following figure:

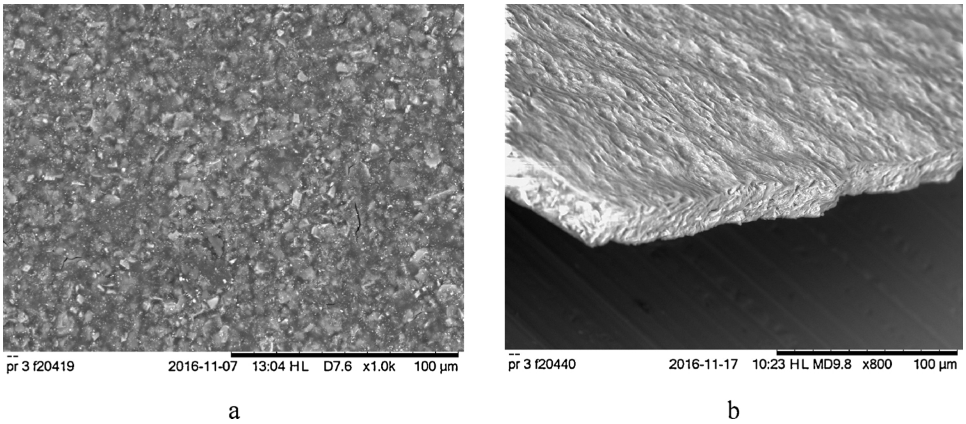

**Fig. 2.** SEM images of the investigated EC Paint layer: (a) reflection image of the front surface of an EC Paint/Al foil specimen, observed at EDX(15 kV) mode, magnification = 1.0k, (b) reflection image of an isolated EC Paint layer observed at EDX(15 kV) mode, magnification = 0.8k.

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