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# Contact electrification and charge decay on polyester fibres: A KPFM study

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#### ABSTRACT

Surface contact electrification experiments have been performed on insulating polyester fibres in contact with a conductive fibre using biased atomic force microscopy tips. With positive tip bias, charge spots have been deposited in a reproducible manner. The density of deposited charges on the fibre surface was observed by Kelvin probe force microscopy as a function of time, charging position and relative humidity. Two main charge dissipation mechanisms were identified with different importance depending on the relative humidity value.

#### 1. Introduction

Contact electrification and electrostatic discharge (ESD) are known as serious risks in many domains. Antistatic fabrics are thus widely used in many applications, such as sophisticated microelectronic devices in electronic industry, in the chemical industry and in industries using filters [1,2]. In antistatic materials, the most widely used method consists in dispersing conductive fibres inside the highly resistive base fabrics. This method has the advantages to provide effective conductivity and long-term durability. The key is that the accumulated charges should be safely and quickly dissipated. However, nowadays the understanding of the mechanisms of charge generation and dissipation behaviours on surface and in bulk of insulating materials, especially the insulating fibres, is far from being completed [3,4].

Many efforts have been done to perform surface charging experiments on polymer materials at the macroscopic scale in order to understand these mechanisms [5–9]. In these works, surface charging was achieved by different techniques applied for different purposes, such as corona charging [6,10–12], electron beam [13–15], contact electrification (with or without friction) [16–20], etc. The charging mechanisms depend largely on the technique used for charging the surface, depending on the source of carriers. For example, X-rays and electron beams are used to trigger a number of reactions initiated by breaking covalent bonds and forming free-radicals and ions. Corona discharge is a powerful way to produce atmospheric ions, together with light and unstable chemicals such as ozone [21].

Kelvin probe force microscopy (KPFM) was first reported in 1991 [22] and is used to measure the distribution of surface potential and detecting charges on a wide range of materials, such as metals, semi-conductors and insulating materials, under different experimental conditions [23–28]. Recently, KPFM has also been used to perform

contact electrification experiments [19,28-36]. The use of biased AFM probes to induce charge electrification and to create electrostatic patterns on insulating films gain widespread interests. Some mechanisms have been proposed in the scope of KPFM results. An electrochemical charging mechanism with ionic charge carriers mediated by the fieldadsorbed water meniscus on the biased probe was discussed by Knorr and co-workers, for dried amorphous polymer films, in particular hydrophobic polymer surfaces [30,37]. Belarni et al. attributed the charging to the generation of space charge polarization, which may arise from trap assisted tunnelling of electrons from the tip to defects in the dielectric [35]. Baytekin et al. obtained new insight into the charging mechanisms and demonstrated that contact charging phenomena derive from the microchemical and possibly micromechanical properties at and near the surface of the contacting polymers [33,38]. Regarding the charge dissipation mechanisms, the surface conductance and water absorption and desorption events have been proposed as the key parameters [21,34].

In this study, contact electrification experiments have been performed for the first time on the surface of insulating fibres by KPFM in order to investigate charge generation and dissipation mechanisms at the microscopic scale in antistatic felts. Contact charging studies previously reported in the literature were always realized on flat insulating film glued or spun directly on a metallic support, allowing a good contact between the sample and the electrode. One of the main challenge in the present study was to obtain a reliably electrical contact of the cylindrical polyester fibres presenting a rough surface with the back electrode. With biased AFM probes, charges were successfully deposited on the surface of polyester fibres in contact with a stainless steel conductive fibre. The deposited charges and charge dissipation were studied as a function of time, relative humidity, and distance between the charging point and the contact with the conductive fibre.

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#### 2. Materials and methods

Two types of industrial fibres were used in this study: insulating polyester fibres and conductive stainless steel fibres. The brand name of the conductive stainless steel fibres is Bekinox fibres produced by the company Bekaert in Belgium. The fibres are commercially available and are used along with textile (polyester) fibres for manufacturing antistatic filters for filtering different kinds of powders. Both the stainless steel fibres and the polyester fibres were received from the company Sioen Nordifa (Belgium). The diameter of the stainless steel fibres varies between 7 and  $13\,\mu\text{m}$ , and that of polyester fibres varies between 10 and  $20\,\mu\text{m}$ . The fibres were stored under ambient conditions. Prior to the electrification experiments, the fibres were rinsed with distilled water and dried for 12 h in ambient air in order to remove contaminants from the surface.

KPFM experiments (one-pass mode) were performed using a Picoplus 5500 Atomic Force Microscope (Agilent Technologies) equipped with a scanner of maximum scanning area of  $100\,\mu\text{m}$ , with a MAC III AC module and an extender electronic module. Pointprobes probes coated with a layer of Pt/Ir on tip and detector sides were obtained from Nanosensors. The typical spring constant and resonance frequency of the cantilevers were  $1.2–29\,\text{N}\,\text{m}^{-1}$  and  $75–260\,\text{kHz}$ , respectively. The single-pass scan acquires topographical and surface potential images simultaneously [39].

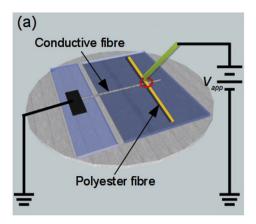
On conductive surfaces, the measured surface potential refers to the surface contact potential due to the difference of work function between the sample and tip materials to which an applied potential may be added [40]. However, it is not so easy to interpret the signals measured on insulating surfaces by KPFM. In a working approximation, one may consider that charge densities which are constant on an area large enough to be resolved by KPFM are proportional to the measured surface potential. Under this assumption, the surface charge density  $\sigma$  located on the insulating surface can be approximated by a capacitor equation [30,33].

$$\sigma(x, y) = \varepsilon \varepsilon_0 V_{SP}(x, y) d^{-1} \tag{1}$$

with  $\varepsilon_0$  the vacuum permittivity,  $\varepsilon$  the dielectric constant of the insulating material,  $V_{SP}$  the measured surface potential and d the average distance between the tip and the surface. This equation expresses the fact that the charge induced in the probe/electrode capacitor by the applied voltage has to match the charge on the insulator surface in order to nullify the electric field between the charge and the probe. Although the absolute value of charge density is still difficult to derive in these experiments on insulating surfaces, the charge density may safely be considered as being proportional to the measured surface potential.

Schemes of the experimental setups used for the biased probe electrification experiments are presented in Fig. 1. Fig. 1(a) shows the sample configuration where a single polyester fibre is in galvanic contact with a grounded conductive fibre, both glued on a AFM standard sample puck with insulating double-face adhesive tape. Fig. 1(b) shows the setup used to study the influence of the distance from the contact point with the conductive fibre. The different positions were identified as follows: P1 was the cross point of the polyester fibre and the conductive fibre, P2 was located 50 µm away from P1, P3 100 µm from P1, and P4 200  $\mu$ m from P1. As mentioned in the introduction, the contact with the grounded electrode is crucial in contact electrification experiments on fibres. If the contact is bad, charges will accumulate on the side facing the conductive support rather than on the top surface [30]. Different setups were thus tested before using the one presented in Fig. 1 where the polyester fibre is in electrical contact with a grounded conductive fibre.

After mounting the fibres as depicted in Fig. 1, all the samples were put in the environmental chamber of the AFM during one night under controlled relative humidity to ensure full stabilization of the setup. Previous studies showed that the surface potential may have a strong



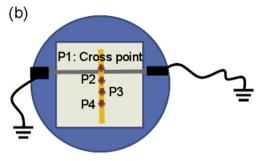


Fig. 1. Scheme of biased probe contact electrification experiments. (a) Sample configuration: a polyester fibre (yellow) in galvanic contact with a grounded conductive stainless steel fibre (gray) are fixed on insulating double-face adhesive tape, a bias voltage  $V_{app}$  is applied to the AFM tip. (b) Measurement positions on the surface of the polyester fibre: P1 = cross point of the polyester fibre and the conductive fibre, P2 is  $50 \, \mu \text{m}$  away from P1, P3 is  $100 \, \mu \text{m}$  from P1 and P4 is  $200 \, \mu \text{m}$  from P1). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

dependence on relative humidity; a low relative humidity reduces the discharge rate [30]. In order to compare the results for different relative humidity (RH) values, different saturated salt solutions were used in the environmental chamber containing the sample: lithium chloride for 15% RH, potassium acetate for 25% RH, potassium carbonate for 46% RH and sodium chloride for 75% RH.

Charge electrification experiments were realized as follows. First, topographic and potential images of the polyester fibre surface were acquired in KPFM mode. Second, the AFM probe was brought into contact with the surface using the AFM contact mode with a controlled applied force (set point equal to  $\approx 0.5 \, \text{V}$ ). Third, a constant bias voltage,  $V_{app} = 10 \, \text{or} -10 \, \text{V}$  was applied to the probe during 60 s. Finally, topographic and surface potential images were acquired continuously to monitor the time variation of the surface potential on the charged spot.

The topographic and surface potential images were treated and analysed using home-made procedures developed on the Igor Pro software (WaveMetrics). From the successively acquired surface potential images, average profiles of the potential spots were obtained as 10 pixels wide profiles at the center of the image, allowing to obtain spot profiles at intervals of 256 s, the first profile corresponding to a time of 128 s. After subtraction of the background value of the surface potential, a gaussian function was used to fit the profiles and the following parameters were obtained: the peak height PH in volts and peak width W in  $\mu$ m. With these two parameters, the peak volume PV (V. $\mu$ m<sup>2</sup>) was estimated as follows

$$PV = \pi W^2 PH \tag{2}$$

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