



Electret films with extremely high charge stability prepared by thermal evaporation of Teflon AF

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ABSTRACT

We report on the preparation of Teflon AF based electret films by thermal evaporation and corona discharge. Due to a special construction of the evaporator, smooth films without micropores could be deposited even at rates above 10 nm/s. The influence of film thickness, substrate temperature, charge density and type of charge carrier on the charge stability was investigated together with the mechanism of charge decay. Negatively charged films of 9.5 μm evaporated Teflon AF 1600 turned out to exhibit a much higher charge stability compared to commercial 25 μm Teflon FEP electret foils at almost 1/3 thickness and proved to be also superior to spin-coated Teflon AF 1600 films. This is discussed in terms of absence of discharge via the surface. Charge decay was shown to take place by internal drift and diffusion through the bulk of the film and not along the surface or via external discharge mechanisms as reported for Teflon FEP.

1. Introduction

Electrets contain permanent charges generating internal and external electrical fields and are the electrostatic equivalent to permanent magnets. Particularly polymer-based electrets have been studied extensively due to hosts of applications. They are generally produced as polymer films and foils used in applications such as electret capacitor microphones, electrostatic filters and radiation dose meters [1–3] or as voided charged polymers with excellent piezoelectric properties, which are explored for acoustic transducers and energy harvesting systems, for instance [4,5]. For applications, charge stability is a critical issue since the permanent charges have to remain in the polymer for many years even if the polymers are exposed to elevated temperatures or humid environment [1]. Discharging mechanisms discussed in the literature can be subdivided into internal and external processes [1]. External processes originate from charge compensation via ions attracted from the ambient air in the permanent electrical field, while internal mechanisms are based on charge transport and diffusion along the electret surface or through the bulk. Polar water molecules, attracted by the permanent electrical field are also expected to play a significant role in internal charge decay. Therefore, polymers used for electret applications should be unpolar and exhibit very low water uptake. Typically, Teflon and other fluoropolymers [6] are used when high charge

stability is required [1]. Among those, Teflon AF is particularly attractive. Teflon AF is a co-polymer of tetrafluoroethylene and 2,2-bis(trifluoromethyl)-4,5-difluoro-1,3-dioxole. It is produced by Du Pont as Teflon AF 1600 and Teflon AF 2400 (see Fig. 1). Unlike Teflon, the bulky side group prevents crystallization which, in particular, improves adhesion to other materials and eliminates charge transport along crystallite boundaries. The dielectric constant of 1.9 is even lower than that of Teflon, and the water absorptivity is below 0.01%, which is excellent for electret applications to avoid discharging by ionic diffusion.

For fabrication of MEMS and microelectronic devices, vapor phase deposition is very attractive [7]. Moreover, it allows preparation of solvent free, high-purity films [8–10] and can be combined with co-deposition of one or more components for the deposition of nanocomposite films [11,12]. Vapor phase deposition has also been investigated for Teflon AF via vacuum pyrolysis [9], laser ablation [8], and a direct liquid-injection and atomization process [13]. Vacuum pyrolysis was carried out from a graphite crucible which was heated radiatively by a hot filament. It was suggested that pyrolysis leads to chain cleavage between two dioxole fragments because the resultant free radical is stabilized by the interaction with an adjacent O atom (see Fig. 1). The volatile fragments were assumed to repolymerize on the substrate with little change in the overall chemical structure. Very low

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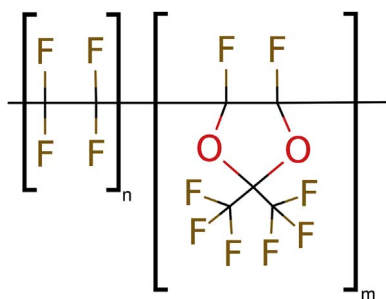


Fig. 1. Chemical Structure of Teflon AF, which is a copolymer of tetrafluoroethylene (TFE) and 2,2-bis(trifluoromethyl)-4,5-difluoro-1,3-dioxole (PDD). For Teflon AF 1600, $n/m = 1/2$, and for Teflon AF 2400, $n/m = 1/7$.

deposition rates below 0.1 nm/s were achieved, and the resulting films contained micropores whose depth increased at increasing deposition rate. The films were not considered for electret applications. Here we report on high-rate vapor phase deposition of Teflon AF electret films by evaporation. Teflon AF was chosen for its unique combination of properties with respect to electret applications and decomposition behavior upon heating, allowing well controlled deposition of high-quality thin films. Using a special labyrinth type evaporator, we were able to deposit very smooth micropore-free films even at high deposition rate. Subsequent charging by corona discharge was employed to obtain electret films with very high charge stability far above much thicker commercial Teflon FEP electret foils and also above spin-coated Teflon AF of the same polymer. We also studied the charge decay mechanism. In contrast to Teflon FEP, where external discharge via ions from the atmosphere was reported to play a crucial role [1], discharge for evaporated Teflon AF films is shown to occur entirely via internal drift and diffusion through the bulk.

2. Material and methods

Thermal evaporation of the Teflon AF films was carried out in a HV chamber with a commercial evaporator made of Tantalum (Kurt J. Lesker), (see Fig. 2). Its labyrinth system avoids rough film surfaces due to occasional explosive deposition of micro-droplets at elevated temperatures and thus enables deposition at high rates. The evaporator was electrically heated to temperatures between 420 °C and 430 °C for evaporation of Teflon AF 1600 and to 450 °C for Teflon AF 2400. Cleaning was performed at 650 °C. The temperature was measured with a thermocouple attached to the evaporator by spot welding. The base pressure in the chamber was 10^{-7} mbar, rising to 10^{-6} to 10^{-5} mbar during evaporation. The films were deposited mostly onto stainless steel substrates coated with a thin Cr layer as adhesion promoter. The distance between evaporator and substrate could be varied between 60 and 120 mm. The deposition rate was monitored by a quartz crystal microbalance, and a shutter was opened after reaching stable deposition conditions within 10–15 min. The deposition rate was typically in the range of 12–15 nm/s. In this range, no film fracture, delamination or formation of micropores was observed. During deposition the

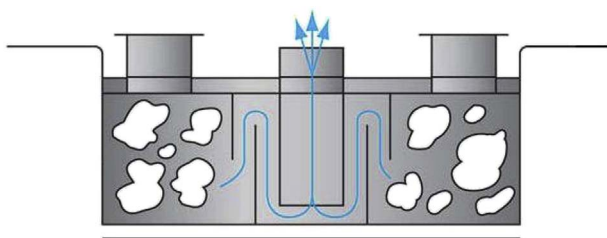


Fig. 2. Sketch of the evaporator cell. The labyrinth-type construction avoids deposition of micro-droplets at elevated temperatures. Thus, smooth films can be deposited even at high deposition rates. The evaporator is filled with polymer granules (white).

substrate was kept at room temperature by backplate watercooling. Higher substrate temperatures showed no improvement in electret properties but turned out to reduce the deposition rate strongly, due to reduction of the sticking coefficient. The film thickness was determined by stylus profilometer measurements (Veeco DEKTAK 8000), which was also used to measure the surface roughness. In addition, surface roughness was studied with atomic force microscopy (AFM, Autoprobe CP5, Park Scientific Instruments). The chemical composition of the films was characterized by means of Fourier transformed infrared spectroscopy (FTIR, 20 DBX, Nicolet) and X-ray photoelectron spectroscopy (Omicron Full Lab). For the FTIR measurements the films were deposited onto a Au coated Si wafer and a 45° single reflection setup was used. For XPS analysis, a Shirley type background was subtracted and the lines were fitted with Voigt type peaks. The glass transition temperature was determined by differential scanning calorimetry (DSC, Perkin Elmer).

A corona-like/arc-type discharge electric set-up was used to charge the Teflon AF films. To create the discharge, a 5 kV AC voltage was applied between a fine tungsten tip and a stainless steel grid. By application of a DC voltage between the grid and the sample, ions could be extracted from the discharge region and accelerated towards the sample. Choosing a positive or negative acceleration voltage, the sample was either charged negatively or positively, respectively. The charge density of the electrets was determined by measuring the DC voltage that was required to compensate the AC voltage induced between a metallized membrane which is aligned parallel to the electret and vibrates at a frequency of 1 kHz. The compensation charge is proportional to the surface potential $V(t)$ of the electret and can therefore be used quantitatively to compare the different electrets. For comparison, corona charging was also applied to commercial Teflon FEP foils of 25 μm thickness (Du Pont) and to spin-coated Teflon AF 1600 films with the same thickness as the evaporated films.

3. Results and discussion

3.1. Structural and chemical properties

Prior to corona charging, the evaporated Teflon AF films were characterized in terms of structure and chemical composition. As expected, X-ray diffraction measurements showed that the films are completely amorphous. After confirming smooth surfaces on a large scale via profilometer measurements, detailed investigations on surface roughness were carried out by means of AFM. All films deposited at rates up to 12–15 nm had a smooth surface without micropores or cracks. A typical AFM image for a Teflon AF 1600 film is shown in Fig. 4. At higher rates, no micropores were observed either, but delamination and crack formation could not always be excluded under the present conditions for thicknesses well above 10 μm . However, excellent electret properties were already obtained for thicknesses < 10 μm (see below). Similar results were also obtained for Teflon AF 2400. Compared to the pyrolysis approach reported by Nason et al. [9], our evaporation method allows fast deposition with an increase in the deposition rate by one order of magnitude without formation of micropores. A smooth film surface is crucial in many applications, particularly in MEMS technology, e.g., for electret microphones or sensors.

The chemical structure of the evaporated films was investigated by FTIR spectroscopy and XPS. A comparison of a FTIR spectrum of an evaporated Teflon AF 1600 film and the corresponding powder is displayed in Fig. 5. The peak at 986 cm^{-1} is ascribed to the CF_3 group and those at 722 , 1145 , and 1203 cm^{-1} are attributed to the fluorinated dioxole component [9]. Whereas the main components of the Teflon AF are clearly present after evaporation one notes changes in the band intensities for the evaporated films which have been already reported and discussed by Nason et al. [9]. This could indicate slight changes in the composition of the Teflon AF films.

Further insight was provided by XPS (although XPS only probes the

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