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FIM OBSERVATION OF THE POLYETHYLENE TUNGSTEN SYSTEM

Polymer ageing processes of a polymer/metal system in the presence of the electric field originate mostly in the transition region between a metal and a polymer. Generally, the ageing process may be regarded in many cases as a peculiarity of the polymer/metal interface. The hitherto prevailing investigations indicate that the polymer ageing processes may be influenced both by imperfections of the crystal structure of the interface region including the presence of microfissures and by charge transfer and accumulation processes from the metal into polymer. The latter processes are frequently discussed by the adoption of the band concepts developed for semiconductors. So far, no theoretical model has been formulated for the polymer/metal contact.

The idea of this work was to use the melted phase deposition method and a field emission tip as the metal substrate which has the form of a needle, and to examine the polymer coatings and possibly the interface by means of the field-ion microscopy (FIM). The polyethylene/tungsten system was chosen. A field ion microscope enables examining structure and defects of a series of metals, also some semiconductors, by directly viewing the surface atoms and vacancies. The high electric field which is present at the tip surface and frequently becomes a disadvantage in the case of some metals and semiconductors under imaging, could be useful as one of the working factors in investigation of a dielectric polymer.

A conventional glass FIM apparatus was used and it ensured obtaining

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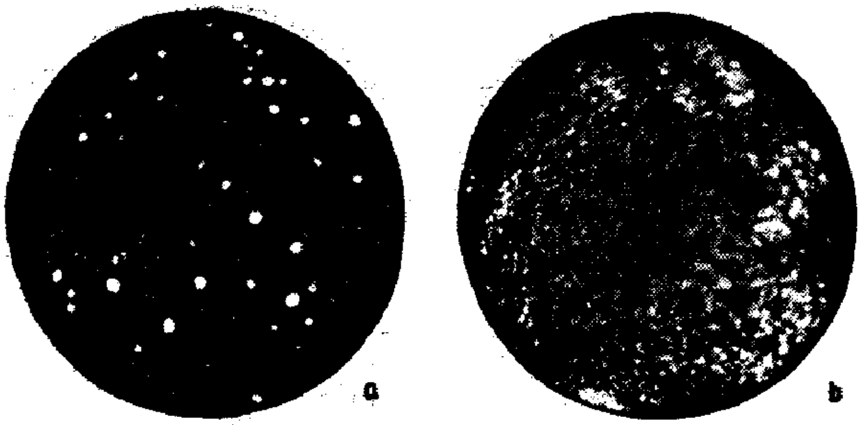
an ultimate vacuum at 10^{-8} Pa level in the absence of polyethylene (PE) source in the FIM tubes. PE of soft type was deposited onto tungsten tips by thermal spreading of melted PE in vacuum from a source located on the tip embedment up the tip apex distant ca. 1,5 mm from the source location. The temperature of the deposition of PE onto the tungsten tip was not directly measured on FIM experiment, and we estimate it was in the range 420 K to 550 K. During the course of PE deposition the pressure (in the space of the measuring Bayard-Alpert gauge) occasionally rose up to some 10^{-4} Pa. Hydrogen field-ion images were photographically recorded via an external image intensifier type T2001 EMI, England.

An essential problem in this investigation was whether it is possible to deal with a dielectric like PE in the field-ion microscope. The observation of the PE-covered tungsten tips, made both in the helium and hydrogen FIM modes, showed an amorphous-like field-ion patterns. Photos 1 a and b have been taken during the course of one hydrogen FIM runs typical for a very wide range of imaging voltages 29 kV to 7 kV. A series of such hydrogen FIM patterns were obtained after the deposition of PE on to the substrate surface and also after either a charge rearrangement or some kind of stimulation process of the polymer had occured at a voltage of ca. 30 kV. In fact, no field-ion image appeared up to this stimulation voltage, though so called "the best image voltage" in hydrogen imaging gas was found to confine in the range 19 kV to 20 kV for this tungsten tip prior to the loading the FIM tube with the PE source. Then, after the stimulation had occurred, a series of hydrogen FIM images of the covered surface were observed in this anomalously wide range of imaging voltages below 30 kV.

Such a hysteresis-type behaviour indicates that the polymer chains were either maintained on the tip or being drawn up on the metal tip surface at very high voltages applied. Let us provisionally consider the hypothesis of the stimulation of the PE layer taking into account that each bright point in the FIM pattern is a site at which the highest intensity of the electric field locally exists. Most likely the observed effect is not associated with breakdown since in that case evaporation of the material and depletion of the very substrate should occur. Instead, weak evidence for field desorption or field evaporation was observed up to voltages in excess of twice "the best image voltage" in the helium FIM operation mode in the case of some other PE tungsten samples [1].



a b

Photo 1. FIM patterns of PE tungsten
a - imaging voltage 14 kV, b - imaging voltage 29 kV

Also, a hysteresis-type run should not be present in the case of breakdown. On the other hand, we believe that the imaging behaviour and the obtained patterns give no evidence for possible complete decomposition of the polymer and the penetration of carbon atoms into tungsten lattice. Charge transport through the adlayer should occur only in certain specific sites or paths. It would be interesting to assume that the apparent similarity of the observed hysteresis-type dependence of the FIM image intensity versus the imaging voltage and the well known S-curve for semiconductors in the high electric fields is not accidental. This might reflect, thus, the relation between the microgeometry of the FIM sample and the electronphonon interaction in the polymerlayer. If this is true, the FIM techniques would give the possibility of the understanding of the behaviour of polymers in the high electric field, particularly as a function of the deposition technology.

In conclusion, we regard that this preliminary work shows that the application of the field ion microscopy techniques to investigations of polymer/metal systems may yield interesting results with respect to ageing processes. It has been found that field-ion imaging appears when a certain value of the stimulating voltage has been reached. This value is unexpectedly high and the range of imaging voltages applied is anomalously wide in comparison to a metal/metal or metal/semiconductors systems.

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R e f e r e n c e s

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