Lithuanian Journal of Physics, Vol. **46**, No. 1, pp. 79–84 (2006)

# SURFACE CHARGE DECORATION METHODS *

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Received 30 June 2005

Charge distribution on layer surface is determined by homogeneity of a layer. Surface charge can also be used for information recording. Charge distribution in layers with high surface potential is investigated by the method of layer decoration in air with tungsten oxide. In such a case, round particles of tungsten oxide from 1 to 10 $\mu$m in diameter are formed. The larger the surface charge of a layer, the more rounded are particles of tungsten oxide. In addition, surface charge of a layer stimulates joining of decorating particles into chains. Increase of tangential electric field causes increasingly regular orientation of those chains in the direction of the field. Layers with a lower surface potential are decorated in vacuum with Se islets. This method makes it possible to measure layer surface charge distribution with precision higher than 1 $\mu$m. If the decorated layer is charged negatively, action of the surface charge causes Se islets to form chains, whereas positive surface charge causes a decrease of Se islets by a factor of 2 to 7 in comparison with size of Se islets in areas without surface charge. This effect can be used for information recording. Positive surface charge of a layer is arranged in circular zones.

**Keywords:** surface charge, decoration, defect

**PACS:** 68.37.Lp, 68.55.Ac, 52.80.-s

## 1. Introduction

Charged layers are used in various equipment. Charge distribution on a layer surface may determine properties of the layer [1]. For example, if the surface of an electrographic layer is charged homogeneously, then electrophotographic copies obtained using such a layer will be defect-free. However, non-homogeneous charge distribution on a layer surface can be useful, too. For example, if it were possible to form small charged areas on a layer surface, then this layer could be used for information recording. For these and similar reasons, it is important to be able to accurately determine the distribution of layer surface charge. One of the most common methods of layer surface charge visualization is the development of layer potential relief using an electrographic developer. However, this technique only provides information about macroscopic charge distribution on the layer surface, because size of developer particles is from several to several hundred microns. The goal of this work is the creation of technique that allows precise measurement of charge distribution on the layer surface. In this work decoration methods of electron microscopy are used. The decoration method can be used for investigation of layer structure [2], finding carbon nanotubes in the sample [3], detecting layer defects [4] and dislocations [5]. The literature on surface potential measurement using the decoration technique is practically non-existent. This work demonstrates that the decoration technique makes it possible to measure surface charge distribution with an accuracy higher than 1 $\mu$m. Knowledge of the exact relief of layer surface potential facilitates understanding of various processes taking place on the surface of a charged layer (e. g., movement of positive or negative charged particles [6, 7]), makes it possible to locate small (smaller than 1 $\mu$m) defects on layer surface, as well as to develop new methods of recording the information on a layer surface that are based on microdistribution of surface charge.

## 2. Experimental details

4 $\mu$m to 50 $\mu$m thick polyethyleneterephthalate (lavsan) layers were used for investigations of layer surface charge. The layers are charged in air using the corona discharge [8]. These layers were chosen for the following reasons. They retain a high value of layer potential long after charging, they are not sensitive to light, their surface is smooth, without

* The report presented at the 36th Lithuanian National Physics Conference, 16–18 June 2005, Vilnius, Lithuania.


ISSN 1648-8504

defects, and investigation of layer surface using an electron microscope is relatively easy. These layers are discharged using a radioactive source that ionizes ambient air. Charge carriers created in the process of ionization neutralize surface charge of the layer.

Polyethyleneterephthalate layers can be charged in air up to (2000–3000) V. In order to obtain exact distribution of surface charge, the charged layer should be decorated in a vacuum equipment (VUP-4). However, it is impossible to place a charged layer immediately into high vacuum. Vacuum must be increased gradually. At a certain value of pressure, glow discharge begins in a vacuum chamber and a part of layer surface charge leaks into the substrate. For this reason, layers charged to (2000–3000) V can not be decorated in vacuum. However, layers charged to a high potential can be decorated at normal pressure. Before decorating these homogeneously charged layers, a desired potential relief is formed on their surface. This is done by touching the charged surface of a layer with a grounded needle electrode. Thus, a part of layer surface looses its charge. Both tangential and normal electric fields are formed in such region of the surface [9]. The layer surface potential at the point of contact with the needle becomes zero. Layer potential grows with an increasing distance from that point, approaching the potential of a homogeneously charged layer. Therefore the potential gradient (and electric field strength) around the point of contact with the needle has a component that is parallel to the surface of the layer. Further on, this component of electric field will be called tangential electric field, whereas the component that is normal to the layer surface will be called normal electric field. As layer potential varies from 100 V to 2000 V, tangential electric field strengths from 0 V/m up to $10^6$ V/m can be reached. Those tangential electric fields are calculated using the results presented in [9]. Charged layers are decorated in air by placing them at a distance of (3–5) cm from the evaporator into the rising smoke of tungsten oxide emitted by a hot tungsten spiral, which oxidizes intensely when heated. The precision of layer relief measurements increases with decreasing size of tungsten oxide particles. The smallest particles are obtained at evaporation rate (1–1.5) $\mu$m/min and substrate temperature equal to room temperature. Those conditions were identical in all experiments involving evaporation of tungsten oxide. Therefore, it is possible to investigate influence of layer surface potential on formation of those particles. Tungsten oxide has been chosen because tungsten is one of the heavier elements and, consequently, it gives good contrast in an electron microscope (for the same reason, tungsten is used for production of replicas [10]).

The potential relief of layers charged to (50–100) V is investigated by decorating them in vacuum by Se islets. This material has been chosen because it is possible to grow Se islets of 0.1 $\mu$m size (by Se evaporation at room temperature, with evaporation rate of (7–10) $\mu$m/min), and Se does not cause charge leakage (as metals do, for example). Se islets are always hemispheric in shape, regardless of evaporation rate and substrate temperature.

The images formed by decorating islets have been investigated using the electronic microscope EVM-100BR.

## 3. Results and discussion

In order to determine the influence of layer surface charge on decorating particles, one has to know the effects of decoration of uncharged polyethyleneterephthalate layer surface in air. For this purpose, layers discharged by a radioactive source are decorated. In this case, a small number of tungsten oxide particles are formed on the layer surface. These particles are crystalline in form (rectangular, rhombic, or hexagonal). A similar image is obtained after decorating the surface of a metal substrate (which has no surface charge, too) in air. During decoration of a charged layer, a large number of round and crystalline particles of tungsten oxide of various sizes are formed on its surface (Figs. 1, 2). Distribution of crystalline particle diameters is weakly dependent on whether the layer is charged or not (Fig. 2). However, when the layer has surface charge, round particles are formed, which have been practically absent in the uncharged layers. Concentration of those particles is about 10 times higher than concentration of crystalline particles (Fig. 2). The maximum diameter of round particles can exceed the maximum diameter of crystalline particles in uncharged layers by a factor of 7–10.

Therefore, the existence of round particles with a maximum diameter of about (3–7) $\mu$m after decorating the layer with $W_2O_3$ particles serves as the evidence that the layer has a surface charge. In our opinion, formation of round particles is a result of charge accumulated in them. The total concentration of particles in charged layers increases with growing surface charge (Fig. 3). This happens because concentration of round particles increases, whereas concentration of crystalline particles is weakly dependent on surface charge of the layer.

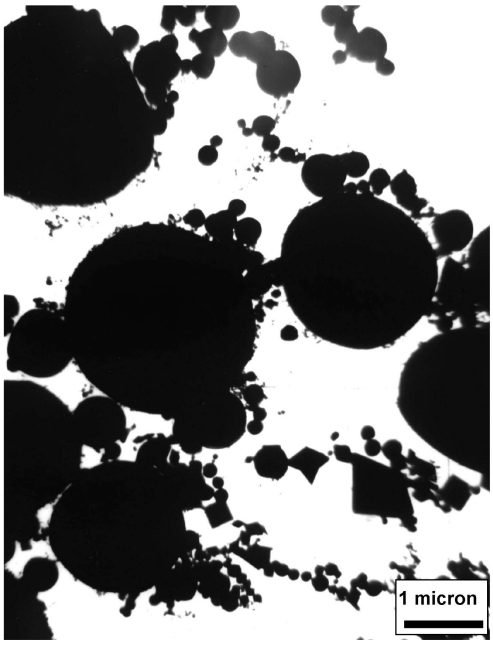


Fig. 1. A 9 $\mu$m thick polyethyleneterephthalate layer, which was charged negatively and decorated in air with tungsten oxide.

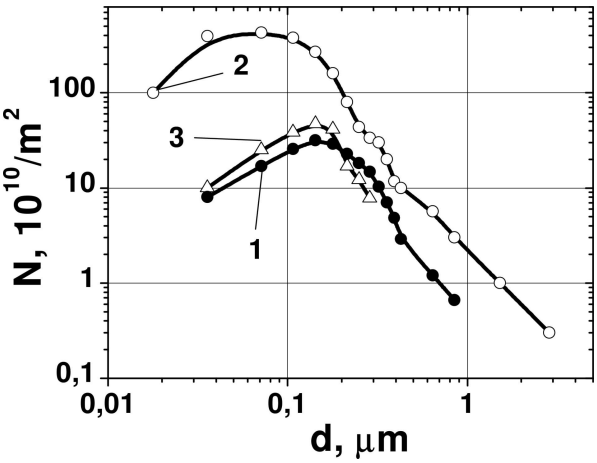


Fig. 2. Dependence of $W_2O_3$ particle concentration on their diameter. A 9 $\mu$m thick layer without potential (*1*) or negatively charged to 2000 V (*2*, *3*) and decorated with tungsten oxide in air. *1*, *3* are for crystalline and *2* is for round particles.

Tangential field on the layer surface causes the formation of particle chains. While this field is weak, the chains are branched and without any preferential direction (Fig. 4). With increasing tangential field, the chains become less branched and they increasingly tend to orient along the direction of tangential electric field (Fig. 5). Therefore, after determining the chain geometry, it is possible to determine electric field strength in a given place of the layer surface. When tangential electric field reaches $(3-7)\cdot 10^5$ V/m, the particles (both single ones and particle chains) disappear from the layer surface completely.

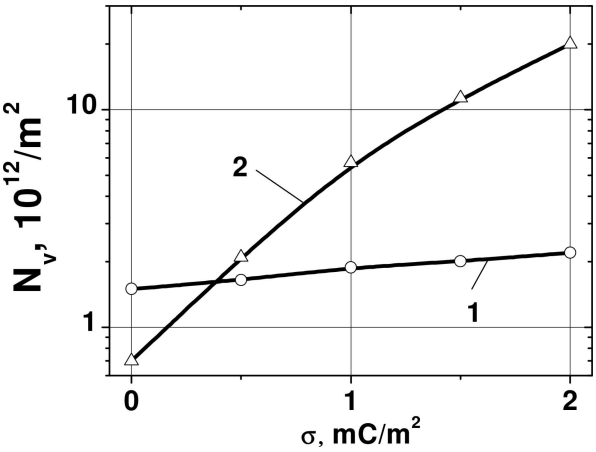


Fig. 3. Dependence of the total particle concentration on surface charge density. *1* for crystalline particles, *2* for round particles.

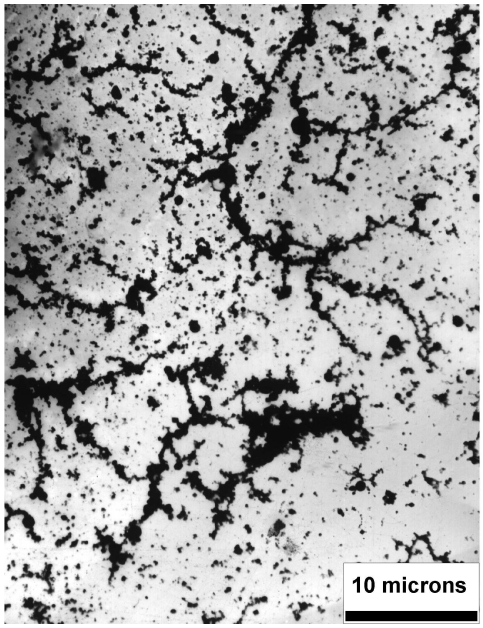


Fig. 4. 9 $\mu$m thick layer positively charged to 2000 V and decorated with tungsten oxide in air. Tangential electric field strength is $6\cdot 10^3$ V/m.

Decoration of charged layers in air causes formation of tungsten oxide particles with sizes of (0.2–20) $\mu$m. For these reasons, the accuracy of electron microscopy investigations of layers decorated in air is limited by sizes of (10–30) $\mu$m. However, even such accuracy is about 10 times higher than that achieved using electrographic developers.

In order to increase the precision of investigations of layer surface charge distribution, decoration of the layer should be done in vacuum, because this allows one to achieve much smaller sizes of islets of decorating material. They can be smaller than 0.03 $\mu$m. However, as mentioned above, if a layer charged to (2000–3000) V is placed into vacuum, then the glow discharge

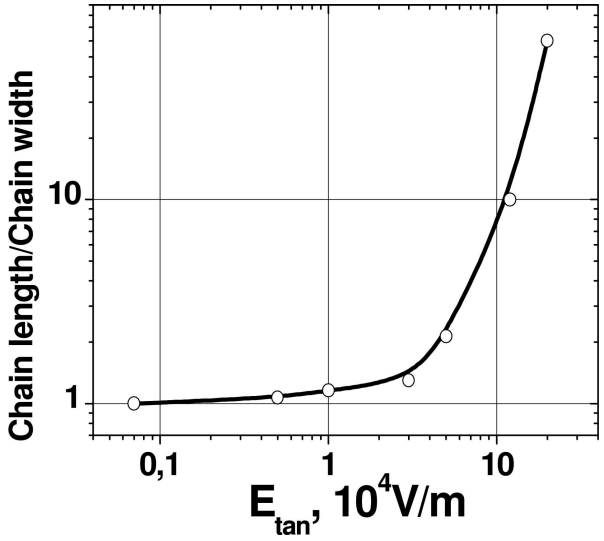


Fig. 5. $W_2O_3$ particles join into chains after formation of tangential field. This figure shows dependence of chain length to width ratio on tangential electric field strength.

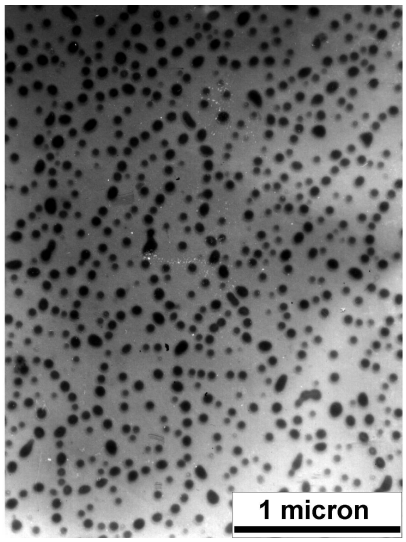


Fig. 6. A 9 $\mu$m thick polyethyleneterephthalate layer, which was charged negatively and decorated in vacuum with selenium. Tangential component of electric field is $3 \cdot 10^3$ V/m.

causes a decrease of its potential to (50–100) V. The latter value is practically independent of layer thickness. Thus, thin layers retain a larger charge than thick layers. Consequently, decoration of surface charge of thin layers in vacuum leads to better results than in the case of thick layers.

Decoration of layers in vacuum can be done using Se. This material has been chosen because of a high mobility of Se molecules, which makes formation of the round amorphous islet Se layer quite sensitive to the tangential electric field. In addition, leakage of layer surface charge through the islet Se layer into the substrate is slower than in the case of metallic decorating

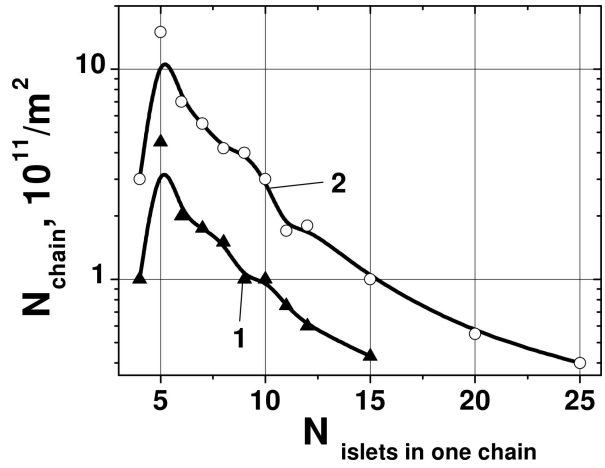


Fig. 7. Dependence of concentration of Se islet chains on the number of Se islets in a chain. Tangential electric field strength is $3 \cdot 10^2$ V/m (*1*) or $5 \cdot 10^3$ V/m (*2*). The polyethyleneterephthalate layer thickness was 3 $\mu$m, the layer was charged negatively.

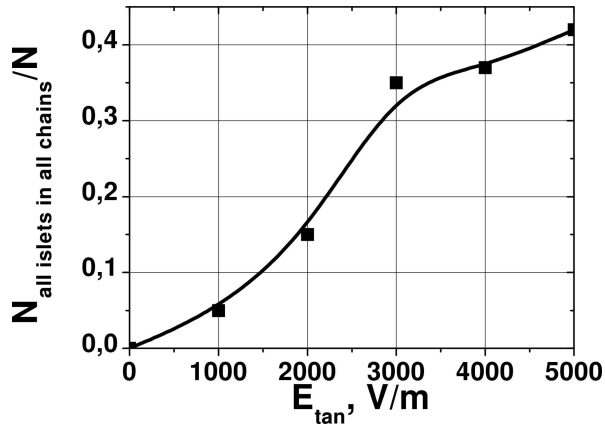


Fig. 8. Dependence of the ratio of Se islet chain concentration and the total Se islet concentration on tangential electric field strength. Polyethyleneterephthalate layer thickness was 3 $\mu$m, the layer was charged negatively.

material. Thus, the layer surface charge influences the formation of Se islet layer for a longer time, which improves decoration results.

In the case of decoration by selenium islets, different images are obtained for positively and negatively charged layers. In the case of decoration by $W_2O_3$, the final result is practically independent of the surface charge polarity. Results of decoration of negatively charged polyethyleneterephthalate layers in vacuum indicate that the action of layer surface charge causes formation of chains of Se islets (Fig. 6), similarly to the case of decoration with $W_2O_3$ of charged layers in air. In this case, the particles that form the chains do not touch each other. The number of such chains decreases with increasing number of particles in a chain (Fig. 7). As tangential electric field increases, those chains

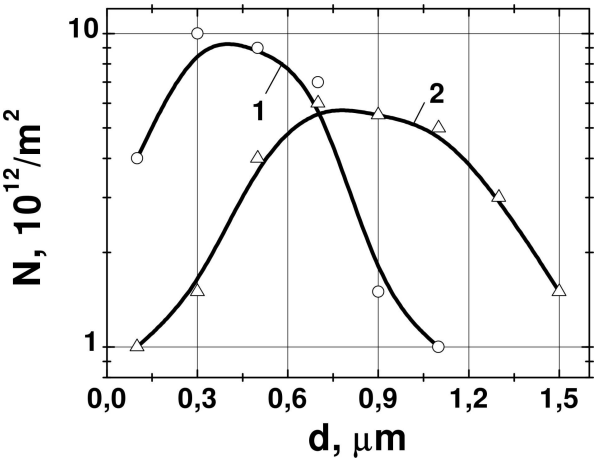


Fig. 9. Distribution of Se islet diameters. *1* for surface positive charge density of 0.8 $\mathrm{mC/m^2}$, *2* for uncharged layer.

become more straight and the number of islets in them grows (Fig. 8). However, the number of Se islets that do not belong to chains remains large, too. The size of joined Se islets is almost the same as the size of isolated Se islets. These results indicate that the main effect of decoration of a negatively charged layer is joining of Se islets into chains that become more straight with increasing tangential electric field on the layer surface.

In the case of decoration of positively charged layers in vacuum, Se islets in some places of the layer are smaller, whereas their number is larger (Fig. 9). The smaller Se islets form round zones. It follows that positive charge on the surface of a polyethyleneterephthalate layer forms separate round zones.

Results presented above indicate that in the case of layer decoration in vacuum, the positive charge on the layer decreases the diameter of a Se islet (for example, from 0.9 $\mu$m in the absence of surface charge to 0.3 $\mu$m when surface charge exists). This phenomenon can be used for information recording. If the mean distance between Se islets is 0.5 $\mu$m, then the amount of information that can be written onto a standard-size CD equals 4 GB, which exceeds the amount of information that is currently stored on such disks by a factor of 6. Decreasing islet size can increase the maximum amount of recorded information. Assuming that the material on which the surface charge is formed can withstand electric fields up to $10^8$ $\mathrm{V/m}$, the maximum amount of information that can be recorded on a standard-size CD would be 10 TB. Therefore, in our opinion, the information recording method proposed in this work holds much promise.

## 4. Summary

1. In the case of layer decoration in air with tungsten oxide, formation of large (from 1 to 20 $\mu$m in diameter) round islets of tungsten oxide serves as an indicator of surface charge on the layer surface. The larger this charge, the more rounded are the particles of tungsten oxide. In addition, surface charge on a layer stimulates joining of the particles into chains.
2. In the case of layer decoration in air with tungsten oxide, prevailing orientation of chains of tungsten oxide particles in one direction serves as an indicator of tangential electric field on layer surface. The larger the strength of tangential electric field on the layer surface, the more straight are the chains of tungsten oxide particles.
3. In the case of layer decoration in vacuum with selenium, negative surface charge of a layer causes joining of Se islets into chains that become straighter with increasing tangential electric field on the surface.
4. In the case of layer decoration in vacuum with selenium, positive surface charge of a layer causes a decrease of the size of Se islets by a factor of 2 to 5 in comparison with regions without surface charge. This effect can be used for information recording. Positive surface charge is arranged in ring-like circular zones on the surface of a layer.

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## SLUOKSNIO PAVIRŠINIO KRŪVIO DEKORAVIMO METODAI

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**Santrauka**

Krūvio pasiskirstymą sluoksnio paviršiuje lemia sluoksnio vienalytiškumas. Sluoksnio vienalytiškumą galima ištirti, užgarinant ant jo paviršiaus įvairias medžiagas, t. y. dekoruojant sluoksnį. Esant aukštoms sluoksnio paviršinio potencialo vertėms (apie 2000 V), krūvio pasiskirstymo vaizdas išryškinamas, ant sluoksnio paviršiaus užgarinus volframo oksidą. Ryškinimo metu susidaro nuo 1 iki 10 $\mu$m didumo volframo oksido dalelės, kurios, didėjant paviršiniam krūviui, darosi vis apvalesnės ir jungiasi išsišakojančiomis grandinėlėmis. Nustatyta, kad elektrinio lauko stiprio tangentinis sandas orientuoja grandinėles lauko kryptimi tuo stipriau, kuo šis laukas stipresnis. Esant mažesniems sluoksnio paviršiniams potencialams (šimtams voltų), paviršinio krūvio pasiskirstymo vaizdas išryškinamas, ant sluoksnio vakuume užgarinus seleną. Nustatyta, kad neigiamas paviršinis krūvis sukelia seleno salelių grandinėlių susidarymą, o teigiamas krūvis mažina seleno salelių skersmenį ir iš jų sukuria atskiras sritis. Toks sluoksnių tyrimo būdas leidžia įvertinti krūvio pasiskirstymą sluoksnio paviršiuje 1 $\mu$m tikslumu. Salelių formų kitimo reiškinys gali būti taikomas optinei informacijai užrašyti.