Properties of Au nanolayers on polyethyleneterephthalate and polytetrafluoroethylene

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Gold nanolayers deposited by sputtering on polytetrafluoroethylene (PTFE) and polyethyleneterephthalate (PET) films were studied in this work. The influence of sputtering time on the layer thickness, surface morphology and roughness was determined by various methods (i.e. atomic absorption spectroscopy, focused ion beam and scanning electron microscopy). Sheet resistance and concentration and mobility of free charge carriers in Au nanolayers were determined using the Van der Pauw method. Surface morphology was determined using atomic force microscopy (AFM). After gold deposition on PET, the surface roughness increases and the surface morphology changes in contrast with PTFE coated under the same conditions. With the increasing sputtering time, the layer resistance decreases rapidly for both polymer substrates. Electrically continuous coverage is achieved for the gold layer with an average thickness of ca 4 nm for PET and ca 5 nm for PTFE. Continuous layers on both polymers exhibit the same concentration of free charge carriers. Lower mobility of the charge carriers was found on rougher Au/PTFE. Copyright © 2009 John Wiley & Sons, Ltd.

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Introduction

Microelectronic technology ever tends to a higher integrity and density of components. One of the technological steps affecting this effort is preparation of well-defined thin metal layers on different substrates. The advantage of polymer substrates is their high flexibility and elasticity. Metal layers on polymers are used in the production of LCD displays, where high optical transparency is required. Polymers were also recently used as a promising substrate for the production of organic transistors. [1,2] Microwave susceptors (composed of a metal layer and a polymer) are used in the microwave heating of food.[3] Another application field of metal–polymer composites is in the pharmaceutical industry.[4] Properties of metal–polymer composites have already been studied in connection with the fabrication of MIS (metal-insulator-semiconductor) transistors suitable for the construction of computer data storage.[5] Metal layers on polymer substrates are used as a part of solar cells[6] and in a vast range of other applications, such as microelectromechanical systems and nanoelectromechanical systems (MEMS and NEMS),[7,8] sensors,[9,10] electronic textiles,[11] in bioengineering[12] and as a part of polymer-based memories.[13]

The study of metal–polymer composites is important for fundamental reasons and for the potential applications mentioned above, as well. Most recent studies have been devoted to systems with gold and silver.[14] It has been shown that the morphology and electrical properties of the metalized polypropylene depend on the thickness of the metal layer.[15] Surface morphology, the mechanical and electrical properties of thin metal films on polymers are all of primary importance for the functionality of these structures in different applications.[16–19] Migration and aggregation of metal atoms on metal–polymer interface, which can be affected by deposition conditions, are also of great importance.[20–24] The surface properties of polymer can be effectively changed in several ways by plasma treatment.[19,25–27]

The present study was performed on polyethyleneterephthalate (PET) and polytetrafluoroethylene (PTFE), the polymers that have broad potential applications in microelectronics. Another reason for this choice is the high thermal stability of PTFE and the great difference in the surface roughness of both polymers. In this work, the gold layers are deposited onto PET and PTFE films by sputtering. Thickness, surface morphology, roughness and electrical properties of the deposited layers, as a function of the deposition time, are examined by different methods.

Experimental

Materials and Au deposition

Gold layers were deposited onto PET (Goodfellow, 50 µm) and PTFE (Goodfellow, 25 µm) films from standard gold targets (purity 99.99%, supplied by Goodfellow Ltd.) by diode sputtering on a BAL-TEC, SCD 050 device. Deposition parameters were as follows: room temperature, deposition times from 0 to 500 s, total Ar
pressure (purity 99.995%) about 5 Pa, the electrode distance of 50 mm and current of 20 mA. The power density of Ar plasma in our case was 0.13 W/cm² and the average deposition rate was 0.15 nm s⁻¹.

Analytical methods

Measurement of the layer thickness

The layer thickness was determined using two independent techniques: first, an indirect method based on the determination of gold content on a defined area of the sample surface, and second using the scanning electron microscope (SEM) image of a cross-section perpendicular to the sample surface. The first technique comprised complete removal of the gold layer from a well-defined part of the sample surface, and the determination of gold content using atomic absorption spectroscopy (AAS) on a Varian AA 880 device using a flame-atomization technique at a wavelength of 242.8 nm.[16] Each measurement was performed three times. The mean layer thickness was determined from the gold content. Direct measurement of the layer thickness was accomplished on a SEM JSM-7500F. The specimen for SEM examination was prepared by cross-sectioning of the metal–polymer sample on a standard cross-section polisher (CP), with a focused ion beam (FIB, 6 kV acceleration voltage, 1-h operation).

Electrical properties of Au layers

Sheet resistance (Rₛ) of the gold layers was measured by the Van der Pauw method. Two gold contacts (about 50 nm thick) on the layer surface were prepared by sputtering. We define an electrically continuous layer as a layer, where the declining sheet resistance reaches a saturated minimum. Concentration and mobility of free charge carriers were determined from the measured resistance and the Hall constant measured by the Van der Pauw method. The measurement was accomplished with d.c. current and a homogeneous d.c. magnetic field, with a polarity commutation of both quantities. Keithley 2400 served as a source of constant current. Voltage response was measured with Keithley 2010 multimeter. The magnetic field (B = 0.2 T) was generated by an electromagnet fed from the Keithley 2440 source. Computer code, working under the LabView 8.5 system, was used for the experiment control and data evaluation.

Results and Discussion

Thickness of Au nanolayers

The thickness of the gold layers deposited onto PET and PTFE (as determined from AAS measurement, as a function of the sputtering time) is shown in Fig. 1. As could be expected, the thickness is an increasing function of the deposition time. For the deposition times below ca 150 s, the thicker layers are deposited onto PET, whereas for longer deposition times, the thicker layers are found on PTFE. The difference may be due to the different surface morphology of pristine PET and PTFE that is discussed below.

The CP FIB method was chosen (see above) as an alternative method for the thickness determination. The morphology of the CP FIB cross-section is illustrated in the SEM image of a Au–PET sample shown in Fig. 2(a and b). Two samples were analyzed with gold layers deposited for 500 and 30 s, with a layer thickness of...
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Figure 3. AFM scans of pristine (PET), PET sputtered with 20 nm Au (PET/Au), pristine (PTFE) and PTFE sputtered with 20 nm Au (PTFE/Au). \( R_a \) is average surface roughness in nm. This figure is available in colour online at www.interscience.wiley.com/journal/sia.

Figure 4. Dependence of the Au layer sheet resistance on layer thickness measured on PET and PTFE with the Van der Pauw technique. Dash-dot lines represent fit of our experimental values. This figure is available in colour online at www.interscience.wiley.com/journal/sia.

Figure 5. Dependence of surface free carrier volume concentration on Au layer thickness sputtered on PET and PTFE measured by the Van der Pauw method. This figure is available in colour online at www.interscience.wiley.com/journal/sia.

Surface morphology and roughness

The AFM images that illustrate the surface morphology and average roughness \( (R_a) \), determined from the AFM measurements of pristine and gold-coated PET and PTFE, are shown in Fig. 3. It is clear that pristine PET and PTFE exhibit different surface morphology and roughness, the difference being due to the different fabrication techniques used for oriented PET and cut PTFE films. After deposition of a 20 nm thick gold layer onto PET, the surface roughness increases (from 0.5 to 1.9 nm) and the surface morphology changes. In contrast, the same deposition

68 and 8 nm, respectively. The thickness determined from the SEM image for the 500 s sputtering time is in excellent agreement with that determined by the AAS metod (see Fig. 1). For the deposition time of 30 s, however, the SEM thickness (8 nm) is twice that determined indirectly from the AAS data (about 4 nm). The difference is obviously due to the fact that the layer deposited for 30 s is not continuous and homogeneous (see Fig. 2(b)), and by the AAS method, only, a thickness is determined over large surface area averaging.
onto PTFE does not result in any significant changes in the surface morphology and roughness (15.1 nm for pristine PTFE and 15.1 nm for PTFE with Au).

Sheet resistance of Au layers

Dependence of the sheet resistance (measured by the Van der Pauw method) on the layer thickness for PET and PTFE is seen from Fig. 4. Because of technical reasons, only resistances of the order of 10^2 Ω and below can be measured in the present experimental setup. With increasing layer thickness, the sheet resistance of the gold layer decreases for both polymers. First, a sharp drop (over the first nanometers) is evident, then a variation inversely proportional to the thickness that is characteristic of an electrically continuous layer is perceptible. We fit our experimental values for previously mentioned two intervals as a function of the thickness layer. We found the values of thickness for individual polymers, when the electrically continuous layers are formed. The intercept points of the dashed-dot lines indicate the creation of electrically continuous gold coverage on the polymer. In the case of PET (see Fig. 4), this value is about 4.3 nm, whereas for PTFE, about 5.3 nm. The difference may be due to the different initial surface morphology of both polymers (see Fig. 3).

Free carrier volume concentration and Hall mobility

Free carrier volume concentration and their Hall mobility significantly affect the electrical conductance of materials. Dependence of free carrier concentration and mobility on the layer thickness for PET and PTFE substrates is shown in Figs 5 and 6. As can be seen from Fig. 5, with increasing layer thickness, the carrier concentration increases dramatically and the layers become conductive (see also Fig. 4). The values of thickness for these transitions (3.7 nm for PET and 4.9 nm for PTFE) are in a good correspondence with the measurement of sheet resistance (see previous paragraph). For both polymers and electrically continuous gold layers, the carrier concentration remains constant regardless of the layer thickness. The carrier mobility changes dramatically with increasing layer thickness (Fig. 6). The mobility first declines up to the moment when an electrically continuous layer is formed. The decline may be due to the fact that in the discontinuous layer, the mobility mechanism differs from classical electron conductivity common in metals. For PTFE, thanks to its higher initial average roughness, the continuous layer is formed after longer deposition time than for PET. For thick electrically continuous gold layers and both polymers, the mobility is a slowly increasing function of the layer thickness. In this region, the mobility in the Au layer on PTFE is significantly lower than that in Au—PET. There is a clear correspondence between the mobility (Fig. 6) and resistance (Fig. 4) data obtained on PTFE and PET.

Summary

It has been observed that mainly the roughness of the PET and PTFE substrates has a significant influence on the sputtered Au layer. In contrast to PTFE, sputtering of an electrically continuous Au layer on PET has a significant effect on the surface morphology and roughness of the Au—PET system. The deposition of a continuous Au layer increases the roughness of Au—PET dramatically.

The electrical properties of the sputtered gold layers on PET and PTFE were studied in dependence on the average layer thickness. It has been found that the concentration and mobility of the charge carriers change rapidly for lower thicknesses of the Au layer. This is mainly valid for the electrically discontinuous Au coverings. The reason is probably due to the different mechanisms of the charge carrier transport, where the quantum effects could also be present in case of an island structure. For the electrically continuous layer in both substrates, the concentration declines slowly and the mobility increases monotonously. The change from discontinuous to an electrically continuous layer was proven by the Van der Pauw electrical measurements.

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References