INTERFACE FORMATION CONTROLLED BY AN ELECTRO-MAGNETRON

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Formation of the interface between sputtered aluminum and glow discharge deposited carbon films (a-C:H) was controlled by using an "electro-magnetron". A pair of rod type electrodes equipped with multi-pole electromagnets and overlaid with aluminum foils were placed in a bell jar reactor. They were driven at about 20 kHz symmetrically and floating against ground. Effective Al sputtering takes place in case of full excitation of the electromagnets. Switching off the magnet current and introducing carbon containing monomer during the same vacuum run results in a transition from sputter deposition to PECVD formation of carbon layers. In a subsequent annealing procedure in high vacuum the carbon film resistivity is lowered down to 1 kΩ cm. Brief system description, process parameters, ESCA and SIMS results as well as voltammetric measurements are presented.

Introduction

The main motivation for this work is to prove experimentally a novel idea which is expected to improve the adherence between metallic layers and plasma deposited films. It is based on the combination of magnetron sputtering and PECVD processes in the same reaction chamber. The magnetron effect is stimulated by electro-magnets. The ratio between sputtering and PECVD portions of the deposition rate can be adjusted by variation of the magnet excitation current during the process continuously. Hence, an "interfacial mixing" can be obtained between two types of layers with a transition region controllable via the magnet current. Generalized, the proposed techniques is expected to be applicable for very many combinations between two types of films which could only be formed with two separate processes and where the adhesion is usually poor.

With respect to the growing interest in the application and properties of carbon films [1] and in accordance with the needs for the technology of thin film integrated micro sensors, a study of the above mentioned principle has been started at an example of a layer pair comprising aluminum and plasma deposited carbon films.

Deposition System

Details of the construction and electrode configuration of the deposition equipment as well as its operation principles for magnetron enhanced PECVD have been published in detail previously [2], [3]. The main structure of the system is formed by a pair of rod shaped electrodes, each 30 mm in diameter and 180 mm long. They are positioned with the long axes in parallel at an interdistance of 70 mm and vertical to the base plate of a bell jar reactor. Six dipoles of electromagnets having the polarity sequence NS, SN, NS... are embedded into the rods performing the magnetron effect. Both electrodes were overlaid with pure aluminum foils as sputtering material source

In this study one point of major attention was the sputtering mode of operation. In order to permit ion motion to follow the electric field a source with about 20 kHz (AF) and a linear amplifier with a maximum power of 250 W was used for excitation. A series resonance circuitry and a multi-tap transformer performed impedance matching. For fine tuning of matching operation frequency was changed within about +/- 2 kHz. Electrodes were driven symmetrically to the ground. Two separate DC power supplies with variable output excited the magnets. Choke coils decoupled DC and AF sources from each other. Substrates were mounted on top of a glass carrier which was placed midway between the electrodes. Hence, the substrates were on floating potential.

Deposition Procedure

Depositions were carried out on top of microscope slices which were covered by a thin film of PECVD silicon nitride or on top of pieces of polished silicon wafers. In the first process sequence aluminum was sputter deposited at 50 mbar Ar pressure in the reactor with an AF power of 250 W and full excitation of both electromagnets (8 A). After 10 min. a layer thickness of about 70 nm was reached. During the following 30 seconds Ar flow rose linearly giving a reactor pressure of about 100 mbar and resulting in decrease of the sputtering rate. Within this period discharge power (250 W) and the magnetic field excitation (8 A) remained constant. The immediate next step was a ramp continuing 2 minutes where linear reduction of the magnet current from 8 A to 0, linear increase of Ar and acetylene flows from 10 sccm to 50 sccm and from 0 to 4 sccm, respectively, took place. During this ramp period of two minutes a gradual change from sputtering of aluminum towards to PECVD of amorphous carbon is claimed to take place. The process continued with the final parameters of the ramp further 20 min. as PECVD of a-C:H. Sputter deposition data which are specific for the configuration used for these experiments are depicted in Fig. 1.

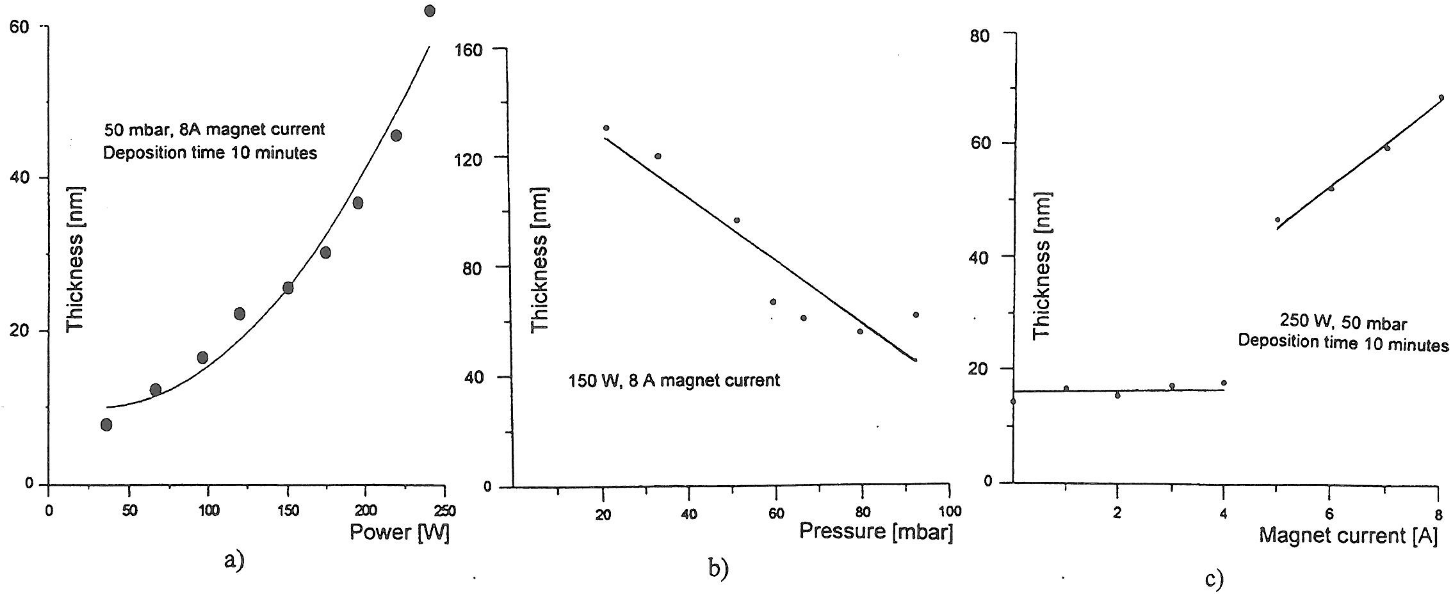

Fig. 1 Quantitative values of the deposition equipment. Sputtering rates in
Argon depending on: a) excitation power (50 mbar, 8A magnet current)
b) pressure (150 W, 8 A magnet current)
c) magnetic excitation (250 W, 50 mbar)
Deposition time 10 minutes

Results

The resulting films were insulating (10^7 Ω cm) as deposited. Thermal annealing, 30 minutes long at 600 °C in a high vacuum furnace, reduced the film resistivity down to 10^3 Ω cm. Compositional analysis of the resulting films was carried out by ESCA and SIMS, before and after annealing. SIMS depth profiling results are depicted in Fig. 2. They indicate very clearly the interfacial transition

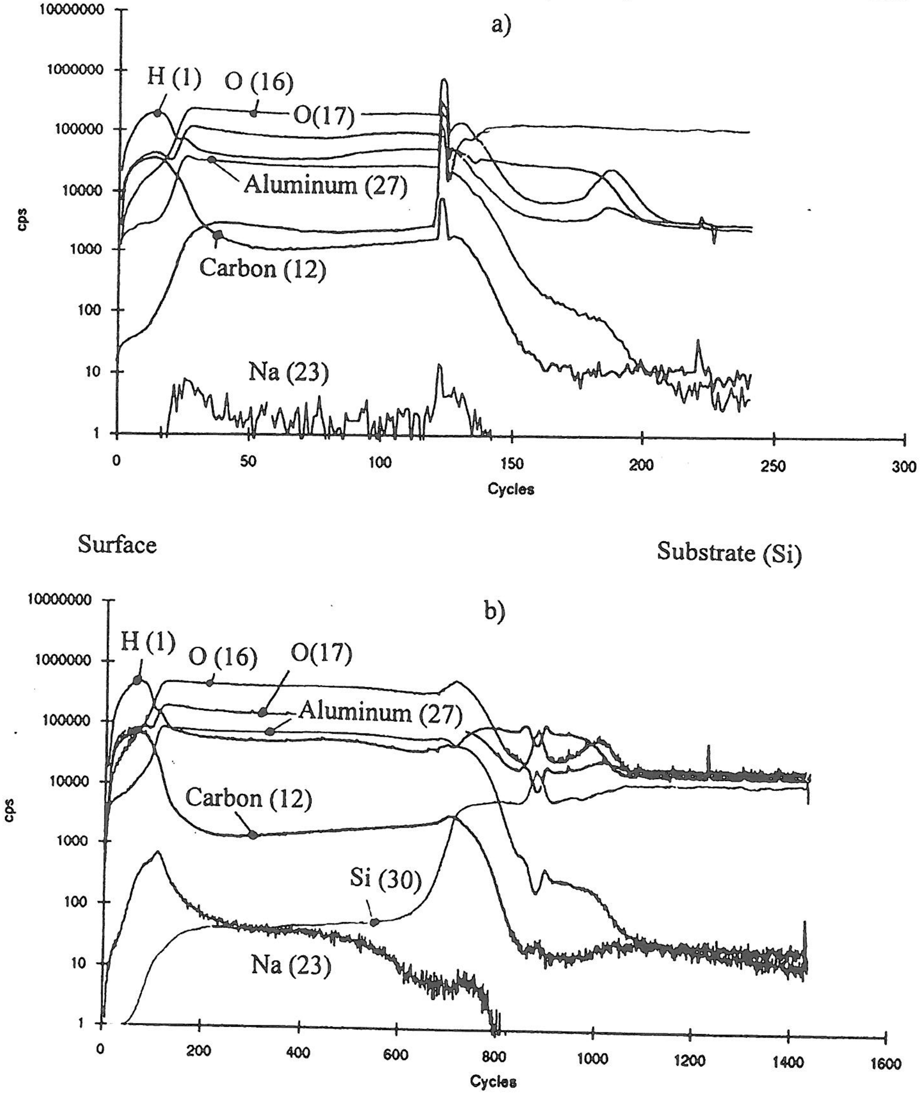

Fig. 2 Shows the SIMS depth profiles with the transition region: a) before, b) after annealing at 600° C

region between the two types of layer formations. However, there is no significant difference between SIMS profiles before and after annealing. XPS analysis of the surfaces at variable angle of incidence between 0° - 60° does also not show any remarkable compositional change of the carbon surface before and after annealing. Therefore, the increase of the conductivity is apparently not caused by a major diffusion of aluminum into the insulating carbon film. Hence, other structural changes must have taken place within the carbon films during the annealing procedure. They should be clarified. Film stability was tested by immersing the samples about 12 hours in sodium chloride of physiological concentration (100 mmol). Neither degradation, swelling, peeling off or cracking was detectable by optical microscopy nor a shift in film resistivity was measured after rinse and drying. Voltammetric evaluation of the structure showed an electro-chemical behavior identical to graphitic carbon. Cyclic voltammograms are shown in Fig. 3.

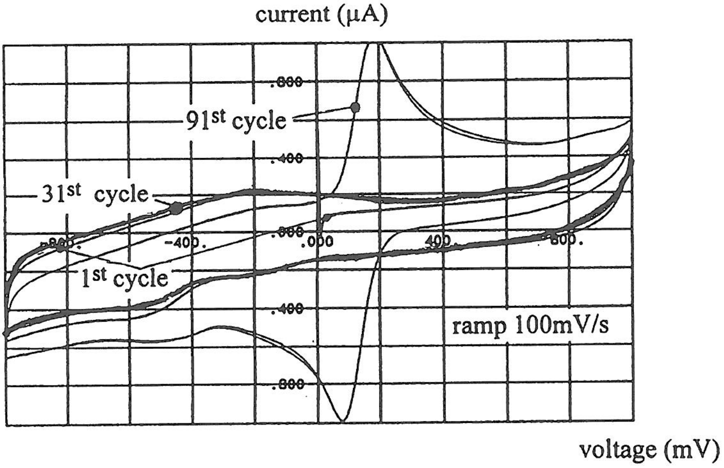

Fig. 3 Cyclic voltammogram taken with a-C:H film as electrode surface. Bold curve shows the inertness of the electrode. The fine curve is electrode response to iron hexa-cyannoferrat II,III. No drift after ninety cycles prove the electrochemical stability

This is a fundamental finding which opens a way for the formation of thin film micro electrode structures for electrochemical measurements and sensor applications. Carbon as electrode material plays a crucial role in this context [4]. The film resistivity obtained after annealing is low enough for the applications where carbon film is a cover on top of a bare or thin film metal electrode and current passes vertically through the film. Up to our knowledge, this is the first PECVD carbon film with sufficient conductivity and with good adherence on top of a metal film. It shows the efficiency of the magnetic field controlled interface formation.

In an outlook one should expect to apply this technique to other material combinations where adhesion improvement is required. Further investigations with other ramp duration of the change of the excitation current and gas mixtures are considered as necessary in order to optimize the effectiveness of the principle. More precise investigations of the interface formation, mechanisms of the adherence increase and the effect of the post deposition annealing will still be interesting questions to work on. Thin film carbon micro electrodes may help indirectly to novel results in sensor applications.

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