Electroless deposition over polished alumina using wet surface treatment

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1. Abstract

Surface treatment using wet process is very important for the adhesion of metal layers obtained by electroless deposition technique. The treatment performed consist of four stages: cleaning, coarsening activation and pos-activation. Results showed a good film adhesion of Copper (Cu) and Nickel Phosphorus (Ni-P) by electroless deposition. The uniformity of grain structure, its density and the grain size were characterized by scanning electron microscopy (SEM). Contact angle was also measured before and after coarsening.

2. Introduction

Electroless Deposition (ED) technique is an important metallization method for non-conductive material [1]. It is also one of the simplest methods for generating thin metallic films on ceramic or polymeric materials compared to other methods (CVD/PVD, plasma spraying or electrodeposition) [2]. It was found to be very useful for interconnection application on several micro technology assemblies. ED offers high selectivity that allows self-aligned deposition, hence saving lithography steps. ED methods are also very versatile and can produce multi component films. In addition, many metals and alloys can be produced with good quality thin films [3]. Other advantages of ED are: high metal purity, low operating temperature, planar topography, low cost and applicability for mass production [4]. The purpose of the treatment is to generate nucleation sites for the metals atoms to deposit on. The treatment process before plating involves four stages: (i) acid detergent (to remove organic and fingerprint contamination), (ii) coarsening step (to make the compact and dense surface of ceramics rough, in order to increase the adhesive strength of the deposit metal film to the ceramic), (iii) activation (Sn, Pd catalyst). In the activation step, the redox reaction takes place between Pd^{2+} and Sn^{2+} on the substrate where Pd^{2+} is reduced to Pd^0 to catalyse the following ED.[1,5] (iv) Post activation step (to remove excess of ionic tin from colloid-covered surface, which surrounds the palladium catalyst)[6].

The effect of the treatment is verified by comparing the contact angle before and after the treatment performed on the ceramic surface. After that, ED of copper and nickel on alumina ceramics was carried out and scanning electron microscopy (SEM) was perform to evaluate the morphology of the deposited films.

3. Experimental

Polished 99,9% Al_2O_3 (Coorstek) with dimension of 2,5 x 2,5 x 0,6 cm was used as substrates.

Fig. 1 shows the schematic of electroless deposition of Cu onto alumina particles using the surface treatment and ED processes. The surface treatment process consist of four stages of cleaning, coarsening, activation and pos-activation.

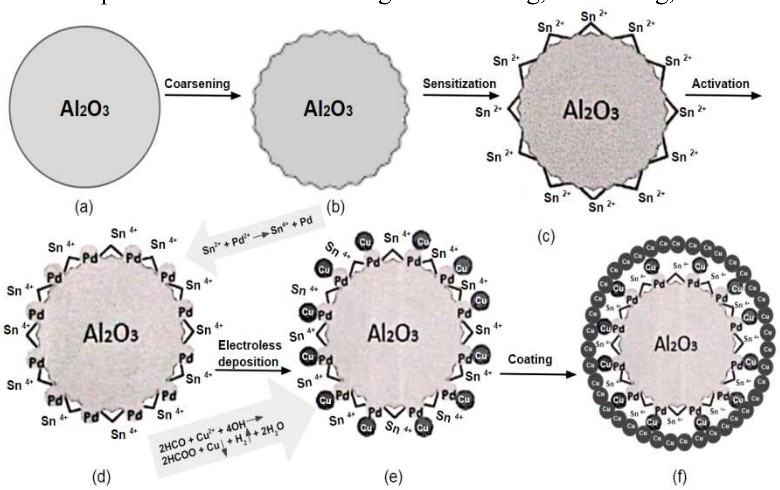

Fig.1 schematic of Cu deposition on the surface Al_2O_3 particles.[7].

After surface treatment, a non-commercial solution of electroless Ni-P and Cu is used. Ni and Cu solutions contain: ion source, reducing agent, complexing agents, buffer and stabilizer. Depositon temperature for Ni and Cu was 40^0 and 30^0 C respectively, while Ph for Ni 5,8 and 12,7 for Cu. Processes are carried out under magnetic agitation.

Surface wettability was evalueted by a contact angle system OCA 15 plus, model OCA 15 (Dataphysics instruments GmbH). Fig.2 shows the contact angle before (a) and after (b) the treatment on the alumina surface.
The morphology of the surface after deposition of metallic films was evaluated by SEM (Tescan, model mira 3xMU) used 5KV and the images was obtained by SEM-FEG.

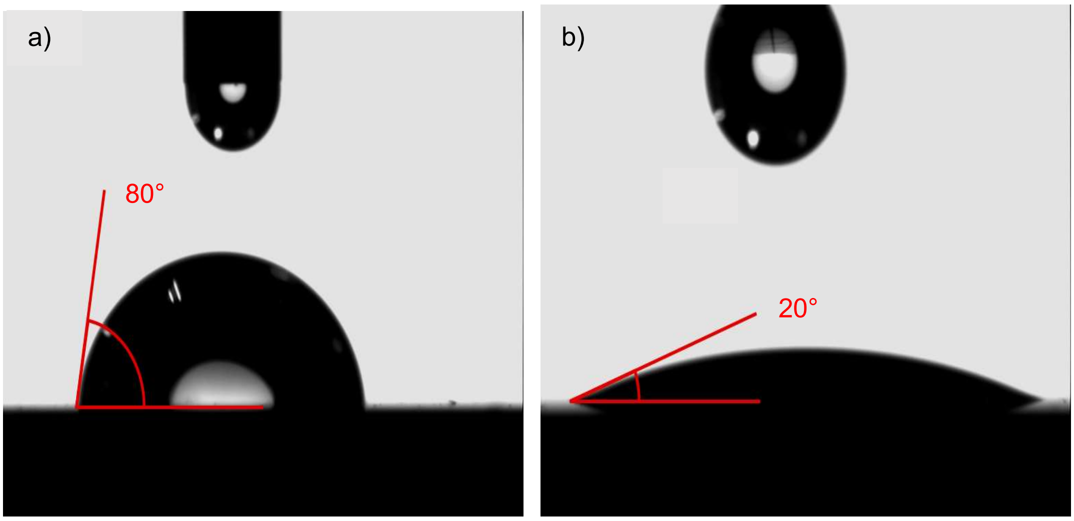

Fig.2 contact angle images of: (a) before treatment; (b) after treatment

4 - Results and Discussion

Electroless deposition onto insulator materials demands the formation of seeds onto the surface so to the deposition can occur. There are no metallic bonds between the polished alumina and metal coatings but unstable mechanical combination [1]. So, acid detergent and coarsening is used to leave the surface free of organic residues and increase the micro roughness of the surface to obtain a hydrophilic surface in order to acquire good adherence for metallic coatings on the surface after nucleation. A widely used procedure for this nucleation involves "sensitization" and "activation" in a stannous chloride and a palladium chloride solution, respectively [8]. Excess tin is usually removed with a solvent for ionic tin [6]. Palladium nuclei should then initiate the autocatalytic plating process.

Contact angle images (fig. 2) showed that the surface of the polished alumina prior to the treatment was very hydrophobic so the drop practically did not spread on the surface, unlike the treated surface, which was quite hydrophilic where the drop greatly spreads.

Morphology and structure of metallic Cu and Ni were evaluated through SEM images shown on Fig. 3. Fig. 3 a) shows alumina covered with Ni thin film and fig. 3 b) shows alumina covered with Cu, both deposited after surface treatment

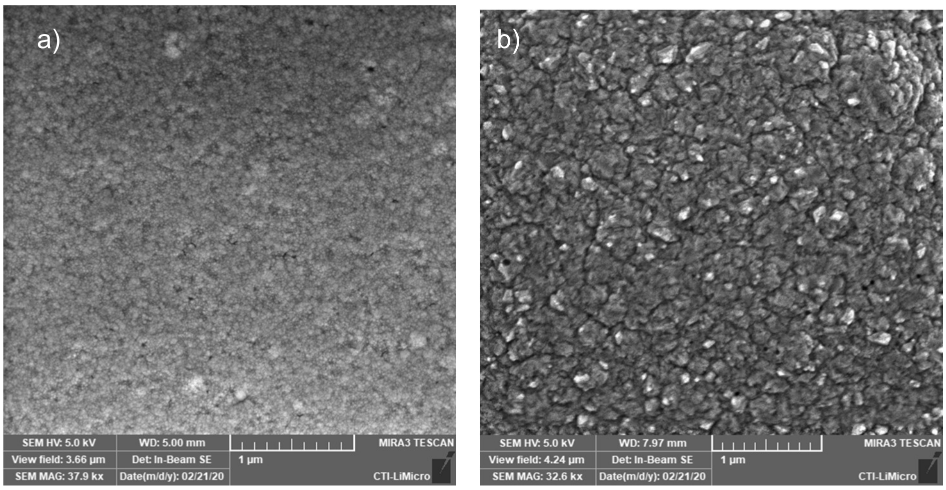

Fig.3 SEM images of a) Ni b) Cu deposited after surface treatment

The average grain roughness (Ra) obtained using the software gwyddion v2.47 was 0.094 and 0,082 microns respectively for Cu and Ni films and no cracks were observed on the evaluated samples. Both films showed a dense structure and few cavites/holes of very small dimensions after the ED.

5- Conclusions

As metallic films, in general, do not present good adherence when deposited directly over polished alumina substrate, we proposed a surface treatment, as an alternative to other more expensive methods (CVD/PVD, plasma spraying or electrodeposition.

The four stages treatment process permitted the development of a fully wet Ni and Cu deposition process, being a good alternative to obtain thin high performance flexible films. The surface treatment also showed a high performance in the ED film at low temperature 40 and 30º C for Ni and Cu respectively obtaining more compact metal films and with small grain size, when observed through the SEM images.

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