### Stability of Structure, Phase and Elemental Composition of Chemically Deposited and Electrodeposited Gold Layers in Multilayer Systems Au<sub>chemically reduced; electrodeposited</sub> /Co-W/Cu<sub>sputtered</sub> / Glass-Ceramics

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**Abstract:** The changes in the structure and elemental composition of Au and Co-W thin layers after thermal treatment of 200 h at 200-300 °C and 50 h at 450 °C were systematically studied using the X-ray microanalysis of scanning electron microscope XL-30 ESEN Philips. The phase stability was evaluated by X-ray diffraction (XRD). The electrodeposited Co-W layer (W 46.0-53.0 wt. % depending on the electrodeposition regime) is amorphous and maintains its amorphous state after heating at 200 °C for 200 h and subsequent heating (+) at 300 °C for 150 h. Co-W amorphous layers prevent interdiffusion of Au and Cu atoms and formation of intermetallics AuCu in the systems of layers Au <sub>chemically reduced</sub>; Au <sub>electrodeposited</sub> /Co-W/Cu <sub>sputtered</sub> / glass-ceramics. The chemically reduced 0.4 µm Au layers maintain their structure and phase in the systems of layers upon heating at 200 °C for 200 h and subsequent heating (+) at 300 °C for 50 h. The Au layer maintains its Au content 85-86 wt. % upon heating at 450 °C for 50 h, the cobalt content of the layer up to 8.6 wt. % and content of oxygen bonded to Au of 8.6 wt. %. The electrodeposited Au layers (0.8 – 1.0 µm) maintain their structure at heating at 200 °C and 300 °C for 200 h. After additional heating at 450 °C for 30-50 h gold content in the layer decreases from 98.4 wt. % to 85.0 wt. %, cobalt content increases to 7.6 wt. % and oxygen to 7.2 wt. %.

Keywords: Multilayer systems, gold coating, interdiffusion, barrier layer, elemental composition, structural properties.

### **1. INTRODUCTION**

Different thin metal layer systems, such as Au/Ni/Cu, Au/Fe-Ni, Au/Pd/Ni, Au/Co, Au/Pt/Ti/InP, Au/Ni/Ti/InP, Au/Ru/Ti/InP, Au/Ru/Ti, Cu/Co-Ni/Cu, Co-Pt/Cu are used in microelectronics, electronics and semiconductor products [1-5].

The formation of systems involving the metal layers sputtered on ceramics and of  $0.2-3.0 \ \mu m$  thick functional metal layers thereon using the electrochemical methods is very actual in microelectronics today [6-8].

Multilayer metal systems must provide the multifunctionality of microelectronics products, their high electrophysical and magnetic properties, solderability, low contact resistance of surfaces and high corrosion resistance. To ensure this, the metal layer systems must possess high thermal stability and mutual adhesion [1, 6, 9-14].

Functional properties of the above multilayer systems deteriorate by mutual diffusion of metal atoms and formation of brittle intermetallics. The latter may give rise to the formation of monolithic films, cause the deterioration of mutual adhesion of the layers and complete reactions of the upper conducting Sn, Au layers with the base metal Ni, Cu, Ti, Sn [15, 16]. This diffusion can worsen the properties of very thin (0.5-1.0  $\mu$ m) metal layers electrodeposited on a Cu layer (1-2  $\mu$ m) sputtered on glass-ceramics [15].

Gold is one element whose atoms can form intermetallic compounds with metals Ni, Cu, Al, Zn, Pb, Cd, Sn, In, Ga as

a result of diffusion even at room temperature [17]. Similar diffusion properties are characteristic of Cu and Ni atoms, also [17-20].

To prevent the mutual diffusion of the interlayer metal atoms, the investigations on formation of interlayers or the so-called barrier layers between the copper substrate and functional Au, Sn coatings have become very actual throughout the world in the last 15 years. In the last two decades of the 20th century, such investigations mainly covered the properties of the barrier layers of Ni-P, Ni, Ni-B, Pd, Pt, Co and their alloys [16, 21-24].

Further investigations concerning the properties of barrier layers are related to the use of X-ray amorphous Ni-W, Co-W, etc. alloys. These investigations are governed by global economic politics, namely, the economy of gold and the tendency to decrease the thickness of gold layers. Along with this, the requirements are increased for providing the functional properties of thin metallic films, such as thermal stability of adhesion, preservation of structure and phase composition under conditions of continuous current and heat. This is why the investigations concerning the electrowinning and properties of nanostructured thin layers of X-ray amorphous Co-W alloys are very actual today [21, 23, 25-27].

One of the pioneering studies on the application of Co-W alloys for stabilizing the structure of Au [26] has shown that mutual diffusion of Fe and Ni atoms into a 1.0-1.25  $\mu$ m thick Au coating in the system of layers Au/Co-W/Fe-Ni is prevented by the layer of Co-W alloy 1  $\mu$ m thick. The diffusion did not occur upon heating at 500 °C for 5 min, at 450 °C for 5-10 min nor at 300 °C for 70 h. The content of

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Co in the layer amounted to 50 wt. % but the content of W should be at least 40 wt. %.

Formation of X-ray amorphous Co-W alloy depends on the composition of the electrolyte and electrodeposition regime. Co-W alloy layers with a W content of 40 wt. % [26], 22-38 wt. % [28] 36 wt. % [29], 23-40 wt. % [30] were prepared.

As a result of our investigations, thin layers of X-ray amorphous Co-W alloy containing 46-55 wt. % of W have been deposited from the complex salt electrolyte depending on its composition and pH of the solution. The investigations have shown that the 1.0-1.25  $\mu$ m thick thin layer of Co-W alloy provides the phase and structure stability of gold coatings in the system of layers Au/Co-W/Cu (foil) upon heating at 400 °C for 200 h and at 500 °C for 15 h [31, 32].

The problem of structure and phase stability of both 0.2-0.5  $\mu$ m thick gold layers obtained by the chemical reduction method and 0.8-1.0  $\mu$ m thick electrodeposited gold layers on the sputtered copper on ceramics, when exposed to current flow or heat remains very actual. For this reason our further investigations are focused on the possibility of using the electrodeposited amorphous Co-W (W 50 %) layer as a barrier layer to prevent the interdiffusion of atoms in the system of layers Au/Co-W/Cu (sputtered)/glass-ceramics.

### 2. MATERIALS AND METHODOLOGY

The changes of structure of Au and Co-W thin layers upon thermal treatment - on duration and temperature of thermal treatment - were studied by means of scanning electron microscopes MREM-100-005, XL-30 ESEN Philips and transmission electron microscope JOEL 100S. The phase stability was evaluated using X-ray diffraction (XRD) with Diffractometer D8 ADVANCE (Bruker AXS GMBH) and DIFFRAC plus Evaluation Package Release 2007-EVA V13 programme. The changes in the elemental composition in the thin layers due to the possible mutual diffusion of atoms were determined using the X-ray microanalysis of electron microscope XL-30 ESEN Philips.

The following electrolyte was used to electrodeposit the thin layer of Co-W amorphous alloy (g·dm<sup>-3</sup>): CoSO<sub>4</sub>·7H<sub>2</sub>O 55-63;  $Na_2WO_4 \cdot 2H_2O$ 56-58; disodium salt of ethylenediaminetetraacetic acid  $C_{10}H_{14}N_2O_8Na_2\cdot 2H_2O$ (EDTA) 26.0-28.0; citric acid 65-70. The electrolyte did not contain ammonium ions nor ammonium salts. The electrolyte pH was 6.9-7.5. The cathode current density during the electroplating process was 0.8-3.0 A/dm<sup>2</sup>; electrolyte temperature was 62-68 °C.

Co-W layers were electrodeposited from the abovementioned electrolyte on vacuum sputtered copper layers (thickness 0.5-1.0  $\mu$ m). The composition of Co-W alloy depending on the electrolyte pH and cathode current density was controlled using the methods of chemical analysis [31, 32].

Tungsten content in the layer of Co-W alloy was 48-52 wt. %. The following electrolyte (g·dm<sup>-3</sup>): K[Au(CN)<sub>2</sub>] 12-16; diammonium citrate 15-20; citric acid 1-2; benzyl alcohol 2-4% (by volume), was used to deposit the chemically reduced gold thin layer (0.3-0.5 µm thick).

Electrolyte temperature was 50-70 °C, and electrolyte pH was 4-9 [31, 32].

The following electrolyte was used to electrodeposit gold layers 0.6–1.5  $\mu$ m thick (g·dm<sup>-3</sup>): K[Au(CN)<sub>2</sub>] 17-6; citric acid 140.0. The electrodeposition was performed under the following conditions: cathode current density from 0.3 to 1.0 A/dm<sup>2</sup>, electrolyte temperature 30 °C.

Gold layers were deposited by chemical reduction and electrowinning on electrodeposited Co-W layers (thickness 0.8-1.0  $\mu$ m). The temperatures of thermal treatment (200, 300, 400 and 500 °C) and duration of thermal treatment (up to 200 h) were close to the test conditions applied by the authors of Refs [14, 17, 26, 29, 30].

### **3. RESULTS AND DISCUSSION**

### 3.1. System: Co-W/Cu

of cobalt-tungsten allov The formation and coprecipitation of tungsten with cobalt in the electrochemical process depends on electrolyte pH (Fig. 1). The content of W in the electrodeposited Co-W alloy layers (thickness 1.0-1.25 µm) is 46-53 wt. % at pH varying from 6.9 to 7.3 and cathode current density of 0.8-1.2 A/dm<sup>2</sup>. The decrease of the electrolyte pH to 6.5 causes the decrease of W content in the alloy to 32 wt. %. Elevation of electrolyte pH to 7.3 causes the increase of W content to 55 wt. %. Such changes in tungsten content in the alloy are due to the different stability of Co and W citrate complexes.



Fig. (1). Content of tungsten in the Co-W alloy depending on electrolyte pH and cathode current density at electrodeposition:  $1 - 0.8 \text{ A/dm}^2$ ;  $2 - 1.2 \text{ A/dm}^2$ .

The formation of X-ray amorphous alloy is controlled by the very complicated cathodic process. W cannot be electrochemically deposited on the cathode from the solutions of ions or complex anions. The electrochemical formation of X-ray amorphous alloy thin layers is mainly due to a reduction of Co and W on the cathode from highly dispersed combined hydrated Co-W oxides formed in the layer adjacent to the cathode under the influence of hydrogen evolution [25]. The ratio of the previously mentioned hydrated Co and W oxides, in turn, is influenced and determined by the stability of the complexes in electrolyte.



Fig. (2). Changes in structure and phase composition of electrodeposited Co-W alloy thin layer ( $\sim 1.0 \mu m$ ) depending on the duration and temperature of thermal treatment (RT – room temperature, h - duration).

The X-ray phase diagrams and micrographs show that the Co-W alloy containing 48.4 wt. % of Co and 49.5 wt. % of W is X-ray amorphous (Fig. 2d). The amorphous state of the Co-W alloy is preserved upon heating at 200 °C for 200 h and subsequent heating (+) at 300 °C for 150 h. Changes in the layer structure occur at additional heating at 200 °C 200 h+300 °C 150 h+450 °C 30 h. The formation of crystals of CoWO<sub>4</sub> and Co<sub>3</sub>O<sub>4</sub> can be observed in the structure and phase diagram of the Co-W alloy thin layer (Fig. 2c, e). Changes in elemental composition take place in the layer, too. The content of Co and W is decreased gradually from 48.4 and 49.5 wt. % to 41.4 and 42.9 wt. %, respectively (Fig. 3a-c); the amount of oxygen bound in the Co-W alloy layer is increased from 2.1 wt. % to 15.9 wt. %.

# 3.2. System: Au <sub>chemically reduced</sub> /Co-W/Cu <sub>sputtered</sub> / Glass-Ceramics

The micrographs of structure of the chemically reduced Au layers show that the structure of the chemically reduced 0.4  $\mu$ m thick Au layer is retained upon heating the layer system at 200 °C for 200 h and subsequent heating at 300 °C for 50 h (Fig. 4a, d). Under such a regime of thermal treatment the gold layer retains its Au content of 86 wt. % (Fig. 5b). After additional heating at 450 °C for 50-80 h, the spontaneous growth of Au crystals begins (Fig. 4b, e). The latter is favoured by the incomplete crystallization of gold in the course of the chemical reduction process; crystallization is continued on heating of the layer system.



Fig. (3). Changes in elemental composition (wt. %) of the electrodeposited Co-W thin layer (~1.0  $\mu$ m thick) depending on the duration and temperature of thermal treatment.



**Fig. (4).** Changes in structure of the chemically reduced Au thin layers (0.4  $\mu$ m) depending on the duration and temperature of thermal treatment in the system of layers: Au <sub>chemically reduced</sub> /Co-W/Cu <sub>sputtered</sub> / glass-ceramics: **a**, **b**, **c** – scanning electron micrographs; **d**, **e** – transmission electron micrographs of structure of gold layers.

The changes in the elemental composition of the thin layer subjected to the two regimes of thermal treatment - heating at 450 °C for 50 h - show that under conditions of prolonged heating the cobalt content in the layer increases from 5.3 wt. % to 8.6 wt. % and oxygen content - to 6.0 wt. % (Fig. **5b**).

If the sputtered Cu film has pores or other defects, they are repeated in the electrodeposited Co-W layer and chemically reduced gold layer (Fig. 4c). In such spots diffusion of copper atoms starts, and finally the amount of copper in the gold layer may reach 50 wt. % (Fig. 5c).

According to the XRD pattern, Au retains its phase and the diffusion of Cu atoms through the Co-W barrier layer is not observed. Also the broadening of reflections corresponding to gold, the decrease of their intensity and the formation of intermetallics Au Cu are not observed (Fig. **6ad**).



Fig. (5). Changes in elemental composition of the chemically reduced Au thin layers (0.4  $\mu$ m) depending on the duration and temperature of thermal treatment in the system of layers: Au chemically reduced/Co-W/Cu sputtered / glass-ceramics.



Fig. (6). Stability of phase composition of chemically reduced Au thin layers depending on the duration of thermal treatment and spontaneous growth of Au crystals in the system of layers: Au chemically reduced/Co-W/Cu sputtered / glass-ceramics.

## 3.3. System: Au <sub>electrodeposited</sub> /Co-W/Cu <sub>sputtered</sub> / Glass-Ceramics

The thickness of electrodeposited Au layers is 0.8-1.0  $\mu$ m. The thermal treatment at 200 °C and 300 °C for 200 h does not cause changes in the structure of the gold layer system in comparison with the structure obtained without heating (Fig. **7a**, **b**). After additional heating at 450 °C for 30-50 h, the spontaneous growth of gold crystals begins (Fig. **7c**, **d**). The spontaneous growth of crystals on the surface of the Au thin layer in this case is determined by the physical properties of gold, e.g. by the tendency to form such crystals that is also characteristic of metals Zn, Cd, Sn.



Fig. (7). Changes in structure of the electrodeposited Au layers depending on the duration and temperature of thermal treatment in the system: Au  $_{electrodeposited}$ /Co-W/Cu  $_{sputtered}$ /glass-ceramics.

The elemental composition of gold coating changes upon heating of the layer system. Gold content in the layer decreases from 98.4 wt. % 200 °C 200 h and 93.5 wt. % 300 °C 200 h (Fig. **8a**) to 89.0 wt. % and 85.0 wt. % (Fig. **8b**, c). Cobalt content in the layer increases from 2.7 wt. % to 5.3 wt. % and 7.6 wt. % along with increase of duration and temperature of thermal treatment. Such a phenomenon can be caused by diffusion of Co atoms into the Au layer. The elemental analysis data show that at sites of spontaneous growth of Au crystals, the gold content in the layer is decreased to 73.0 wt. %; diffusion of Cu atoms starts parallel with the diffusion of Co atoms (Fig. **8d**). The gold layer incorporates oxygen from 3.5 wt. % to 10.3 wt. % at all the temperatures of thermal treatment tested (Fig. **8a-c**).

XRD patterns show that the formation of intermetallic Au-Cu layers is not observed in the system of layers Au electrodeposited /Co-W/Cu sputtered / glass-ceramics with the barrier layer of Co-W (Fig. 9).

### 4. CONCLUSIONS

- 1. The different stabilities of Co and W citrate and ethylenediaminetetraacetate (EDTA) complexes were determined as functions of the solution pH in the absence of ammonium ions. The different stabilities of Co and W complexes determine whether it is possible to obtain Co-W alloy having the W content 30-55 wt. %.
- 2. Changes in the phase and elemental compositions of the thin layer of the electrodeposited amorphous alloy W 46.0-53.0 wt. % were determined for different heating times and temperatures. The Co-W layer maintains its amorphous state after heating at 200 °C for 200 h and subsequent heating at 300 °C for 150 h. Crystallization of the Co-W layer with formation of CoWO<sub>4</sub> and Co<sub>3</sub>O<sub>4</sub> crystals starts upon additional



Fig. (8). Changes in elemental composition of the electrodeposited Au layer depending on the duration and temperature of thermal treatment.

heating at 450 °C for 30 h. The Co and W contents in the layer gradually decrease from 48.4 wt. % of Co and 49.5 wt. % of W to 41.1 and 42.9 wt. % respectively. The content of the oxygen bonded in the Co-W layer increases from 2.1 to 16.0 wt. %.



**Fig. (9).** Stability of phase composition of the electrodeposited Au layers after thermal treatment.

- Chemically reduced 0.4 μm thick layers maintain their structure and phase in the system of layers Au<sub>chemically</sub> reduced/Co-W/Cu<sub>sputtered</sub>/glass-ceramics upon heating at 200 °C for 200 h and additional heating at 300 °C for 50 h. The layer maintains its Au content of 86 wt. %. The Co content in the Au layer is 5.3 wt. % and the content of the oxygen bonded to Au 3.9 wt. %. Upon heating at 450 °C for 50-80 h the Co content in the Au layer increases to 8.6 wt. % and oxygen to 6.0 wt. %.
- Electrodeposited Au layers (thickness 0.8 1.0 μm) maintain their structure upon heating at 200°C and 300°C for 200 h. Upon additional heating at 450°C for 50 h, spontaneous growth of gold crystals starts. Under these heating conditions, the Au layers maintain their phase.
- A thin layer (thickness approx. 1µm) of amorphous Co-W alloy prevents interdiffusion of Au and Cu

atoms and formation of intermetallics AuCu in the systems of layers Au <sub>chemically reduced; electrodeposited</sub> /Co-W/Cu <sub>sputtered</sub>/ glass-ceramics upon heating at 200 °C 200 h + (subsequent heating) 300 °C 200 h; 450 °C for 50-80 h; 500 °C for 10 h.

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#### Received: July 13, 2009

Revised: December 10, 2009

Accepted: December 22, 2009

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