OUTGASSING PROPERTIES OF Au ELECTROPLATED CONTACT MATERIAL ARGELEC 181 FOR USE IN HERMETIC RELAYS

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Abstract: The purpose of our study was to establish the degree of outgassing of gold plated material (produced with the use of citrate and phosphate electrolytes) still permitting the use of that material in hermetic relays. Systematic investigations of vacuum outgassing of Au electroplated Argelec 181 alloy are performed by Auger Electron Spectrometer which was additionally equipped with quadrupole mass spectrometer. The AES depth profile analysis revealed that the oxide layer on the Au electroplated Argelec 181 alloy is negligible and this is the reason for very low outgassing rate.

Razplinjevalne lastnosti elektrokemijsko pozlačenega kontaktnega materiala ARGELEC 181 za hermetične releje

Ključne besede: deli sestavni elektronski, releji miniaturni, releji hermetični, kontakti električni, zlatenje elektrokemično, čiščenje površin kontaktov, razplinjevanje vakuumsko, stopnje razplinjevanja, AES Augerjeva spektroskopija elektronska

Povzetek: Namen našega dela je bil določiti stopnjo razplinjevanja pozlačenega kontaktnega materiala, ki še dovoljuje njegovo uporabo za hermetične releje. Zlata kontaktna površina na osnovnem materialu Argelec 181 je bila nanešena v Au-citratni in Au-fosfatni kopeli. Pri sistematičnih raziskavah vakuumskega razplinjevanja elektrokemijsko pozlačene kontaktne zlitine Argelec 181 smo uporabili Augerjev elektronski spektrometer (AES), dodatno opremljen s kvadrupolnim masnim spektrometrom. AES globinska profilna analiza je pokazala, da je oksidna površinska prevleka na merjencu zanemarljiva, kar je tudi vzrok za zelo nizko stopnjo razplinjevanja.

Introduction

Recently, the properties of the outgassed material used for the hermetically encapsulated electronic parts draw much attention of the scientific community. One of the main serious causes of failure of electronic components is the film made from the contaminating products on the surface of electric contacts. Effects of a film are the increasing of the contact resistance and the decreasing of the electronic components reliability. The most common types of contamination are oxide and corrosion products, particulates, films formed by thermal diffusion processes, debris produced by mechanical wear and fretting, outgassing and condensation of contact surfaces. Contamination of the gold layer deposited on the contact alloy depends also on the electroplating process technology. The gold electroplated contact layers used in hermetic relays are very important because they act in decreasing the contact resistance and increasing the lifetime.

Experimental

Citrate and phosphate electrolytes (Kemijska tovarna Podnart) were used for electroplating /1,2,3,4/ of contact alloy Argelec 181 (Ag with 0.3 % of Mg). In both cases the thickness of the obtained Au layers was 0.5 μm. The citrate electrolyte as well as the phosphate one was found suitable for the electric currents up to 20 A and did not cause the contact sticking. To examine the degree of contamination and the surface structure of the gold layer obtained with the use of the two different baths the Auger Electron Spectroscopy (AES) was used. The PHI Spectrometer SAM, model 545A was used for the AES analysis. Experiment parameters were: static electron beam of 3 keV, 0.5 μA, 40 μm in diameter at an incident angle of 47° and the ion sputtering rate of Ni/Cr was 10 nm/min.

The surface with the dimension of 5mm x 5mm was etched by two ion guns (3 keV Ar^+). Figures 1 and 2 show profile diagrams where the contamination layer thickness obtained from the ion etching parameters is plotted against the concentration of the elements. The both types of gold electroplated surfaces were outgassed /5,6,7,8/ for several hours in high vacuum of $1x10^{-6}$ mbar at the temperature 135°C. The experimental high vacuum system used for our study was developed and built at the Institute for Electronics and Vacuum Technics. Outgassing products were analysed by the quadrupole mass spectrometer LEISK 1000M.

Results and discussion

The profile diagram (Fig. 1) shows the concentration profile of the surface gold layer deposited to the contact material Argelec 181 using phosphate electrolyte bath. It can be seen that the surface gold layer is covered with the very thin contamination film (~ 5 nm) formed of carbon containing traces of calcium and oxygen. Below

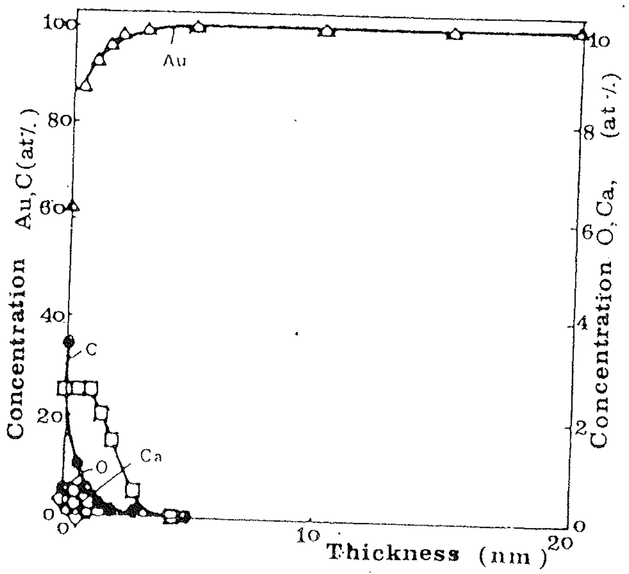

Fig. 1: Profile diagram of the gold surface layer (0.5 μm Au) deposited from the phosphate electrolyte.

this a high peak typical for Au is detected. The next profile diagram (Fig. 2) deals with the gold deposit from the citrate electrolyte. The surface contamination film has the thickness of about 2 nm and contains twice less

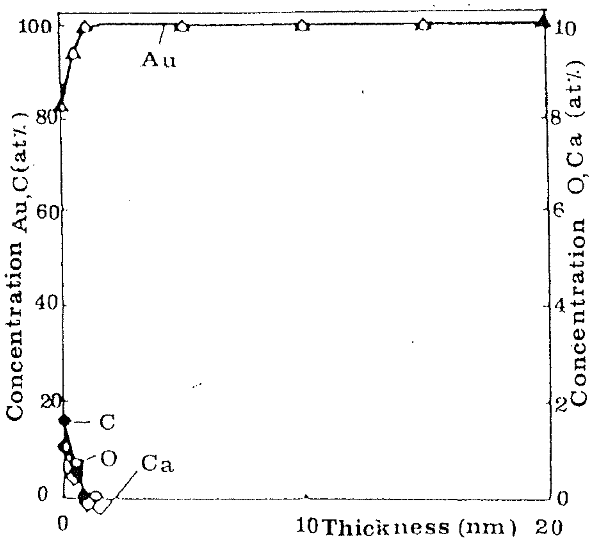

Fig. 2: Profile diagram of the gold surface layer (0.5 μm Au) deposited from the citrate electrolyte.

carbon than the former but greater concentrations of calcium and oxygen. Again, the gold layer below the contamination film is very clear. Then the analysis of gases outgassed from the samples of gold electroplated contact alloy obtained with the use of two different baths (citrate and phosphate electrolyte) was performed. Samples were heated in the experimental device shown in Fig. 3, 4, 5 and 6. After 24 hours heating at 135°C it was evident hat carbon, oxygen, water vapour and the remainings of the cleaning agents (ethanol and isopropyl alcohol) which have been adsorbed on

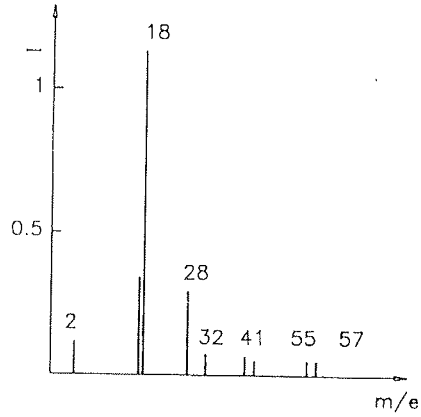

Fig. 3: Mass spectrum of the outgassed empty chamber (150°C, 24 hours of outgassing).

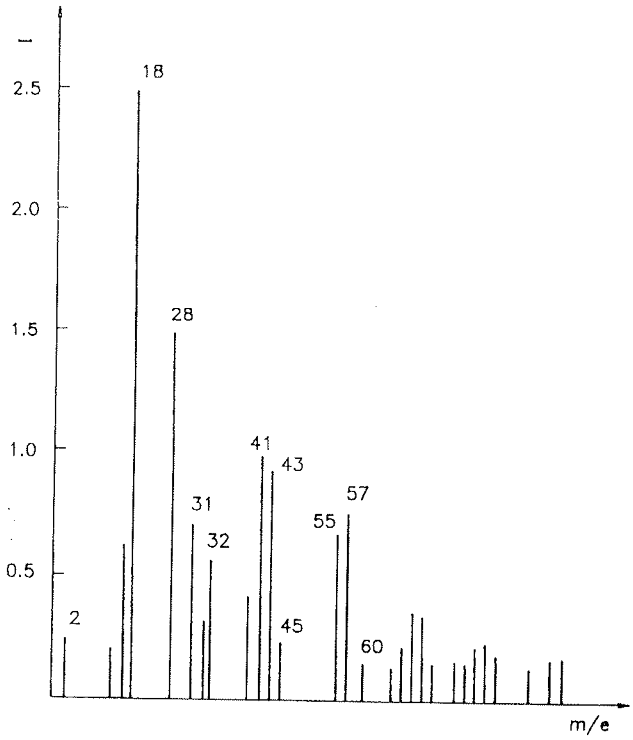

Fig. 4: Mass spectrum of the outgassing products from the electrochemically gold plated (citrate and phosphate electrolyte) contact alloy Argelec 181 (135°C, $1x10^{-6}$ mbar) after 30 minutes of outgassing.

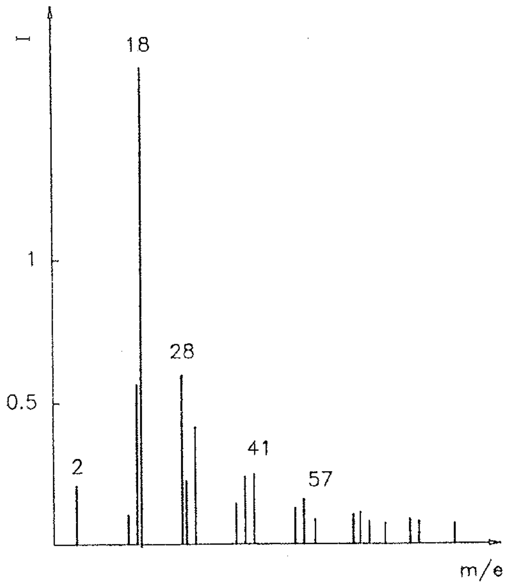

Fig. 5: *Mass spectrum of the outgassing products from the electrochemically gold plated (citrate electrolyte) contact alloy Argelec 181 (135°C, 1x10^{-6} mbar) after 24 hours of outgassing.*

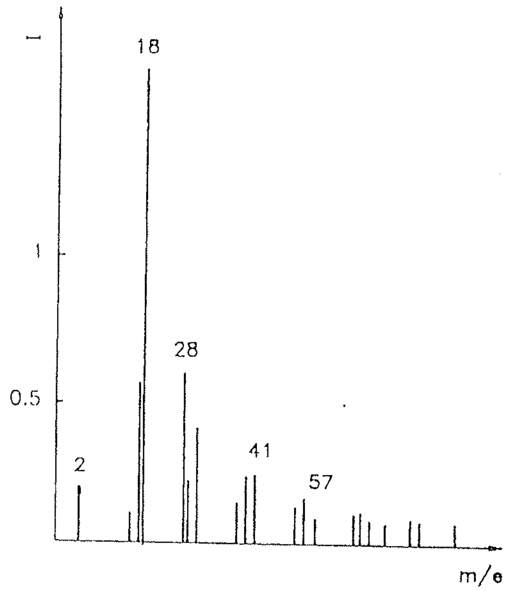

Fig. 6: *Mass spectrum of the outgassing products from the electrochemically gold plated (phosphate electrolyte) contact alloy Argelec 181 (135°C, 1x10^{-6} mbar) after 24 hours of outgassing.*

he surface were completely outgassed. The comparison between the spectra of two outgassed samples and the spectrum of the background - empty chamber (Fig. 3, 5 and 6) proves that. The degree of outgassing for hydrogen was then calculated and compared with the value obtained from the spectra (Fig. 3, 4, 5, 6). The ratio between the hydrogen peaks after outgassing (background subtracted) was f=0.1 for the gold layer from citrate and phosphate bath which is in agreement with the theoretically obtained value being equal to 0.067.

Conclusions

With the AES analysis we established that the contamination of the surface gold layers electroplated on the contact material Argelec 181 is strongly dependent on the technological process.

The degree of surface contamination is greater for the gold layer made with the use of phosphate electrolyte than for the other one. The inner gold layer is very pure in both technologies used.

The degree of outgassing (f) for hydrogen from the gold electroplated contact alloy Argelec 181 (Ag with 0.3% of Mg) from the citrate and phosphate electrolyte is low (about 0.1) which is in good agreement with the theoretically obtained value 0.067.

The contact alloy Argelec 181 electrochemically covered with the gold layer of the thickness of 0.5 μm after outgassing (135°, 24 hours, 1x10^{-6} mbar) is found to be a suitable contact material for use in electronic parts.

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