 UDK621.3:(53+54+621+66), ISSN0352-9045

AES CHARACTERIZATION OF PROTECTIVE THIN LAYERS ON THE AgNi0.15 CONTACT MATERIAL AFTER ITS TREATMENT IN THE RF PLASMA

L. Koller
Institute for Electronics and Vacuum Technics, Ljubljana, Slovenia
and
M. Bizjak
Iskra - Stikala, Kranj, Slovenia

Keywords: electrical contacts, Ag contact materials, silver based contact materials, passivation, passivation thin films, RF plasma, AES analysis, Auger Electron Spectroscopy analysis, low cost implementations

Abstract: Well-known surface protection of Ag-contacts with the thin gold layer is rather expensive. We studied some cheaper types of protective layers. Thickness of the layer must be optional to avoid the corrosion effect of the surroundings and at the same time to insure sufficiently good electric conductivity between the two coated surfaces. Material that is used for the layers must be at least as good electric conductor as the ground material unless the contact characteristics deteriorate. Protection of contact surfaces is therefore a compromise between the increase of the contact stability and the deterioration of the initial contact characteristics. Three different protective layers were deposited on the AgNi0.15 contact material. After their treatment in the radio frequency plasma they were analyzed with the Auger spectrometer.

AES karakterizacija nanosov tankih zaščitnih plasti na AgNi0.15 kontaktnem materialu po obdelavi v RF plazmi

Ključne besede: kontakti električni, Ag materiali kontaktov srebrni, pasivacija, plasti tanke pasivacijske, RF plazma radiofrekvenčna, AES analize s spektroskopijo elektronsko po AUGER metodi, izvedbe cenene

Povzetek: Znana površinska zaščita Ag kontaktov s tankim nanosom zlata je cenovno neugodna. Študirali smo nekatere cenejše pasivacijske plasti, ki jih lahko nanesemo dovolj tanko, da preprečimo korozijski učinek okolice in hkrati ohranimo dovolj veliko električno prevodnost stika med pasiviranima površinama, ki ustreza dobremu električnemu kontaktu. Zaščitna plast na kontaktni površini poslabša kontaktne lastnosti, če ni iz električno bolj ali enako prevodnega materiala kot osnovni kontaktni material. Zato pomeni uvedba pasivacije kontaktnih površin kompromis med povečanjem stabilnosti kontakta in verjetnim poslabšanjem začetnih kontaktnih lastnosti. Tri različne pasivacijske tanke plasti smo nanesli na AgNi0.15 kontaktni material. Nanose tankih plasti po obdelavi v radiofrekvenčni plazmi (RF) smo analizirali s spektrometrom Augerjevih elektronov.

1 Introduction

The requirements for the stability of contact characteristics /1,2,3/ of switching devices are fulfilled if the parameters of the electric contact are within the limits that ensure the contact to work properly. The conductivity of the contact spot between the two surfaces should be sufficiently constant and the contact material should be conducting and chosen according to the known contact force. The contact material should not be affected by the surroundings that produce the corrosion layers and cause the deposition of foreign particles and layers. For testing contact material the AgNi0.15 alloy was chosen as a typical representative of the silver based material. Stability of AgNi0.15 contacts is affected primarily by the formation (and its growing with time) of Ag_2S surface layer in the industrial atmosphere.

2 Experimental

Test samples were made of the contact material AgNi0.15 in the form of the miniprofile-strip with the cross section of 2.6x0.3 cm^2. 50 mm long parts were cut from the strip. Contact surface was half-cylindrical in shape with the curve radius of 9 mm. Three different types of protective layers deposited on the test contacts were studied:

- Passivation by waxing. Test samples were treated in the water solution named Silverbrite /4/.
- Passivation by chromizing. Layer was made by anode oxidation according to the receipt /5/.
- Passivation by the solid layer. Solid layer Ti+TiN was formed by sputtering /6/.

All the three types of protective layers were tested for the resistance to sulfating in the K_2S solution. Samples were exposed to the wet atmosphere containing H_2S (climate test). Afterwards samples were treated by the radio frequency (RF) low pressure plasma /7/: first cleaning of the samples in the hydrogen plasma (1 minute of exposition, 1 mbar), then surface oxidation in the reactive oxygen plasma (0.5 minute, 0.6 mbar). Both procedures were performed in the standard discharge vessel which was a cylindrical glass tube with the diameter of 3 cm. Plasma was created by an inductively coupled RF generator. Hopkins liquid nitrogen cooled trap was used. Temperature of the samples was not measured.

After the treatment of all the three protective layers in plasma condition of the surface was examined /8/ with the Auger spectrometer (Physical Electronic Ind. SAM 545 A) with the static primary electron beam with the energy of 3 keV, the beam current of 0.5 μA, and the beam diameter of about 40 μm. Etching was performed on the surface area of 10 mm x 10 mm with the two Ar^+ ion beams with the energy of 1 keV. The incidence angle of the ion beam was 47°. The etching velocity was about 1.7 nm/min and was calibrated with the standard Ni/Cr multilayer sample. For determining the element concentration (except for the nitrogen concentration where the factor was calculated from the standard sample of stehiometric TiN) the sensitivity factors were taken from the spectroscope manufacturer (PEI) manual. Results of the analysis were presented in the profile diagrams (Fig. 1-6).

3 Results and discussion

We found that the thin Silverbrite layer on the surface of the AgNi0.15 sample is resistant to the sulfading with K_2S (according to the standard test) and also to the wet atmosphere. Protective substance is attached to the Ag base material only by adhesion therefore after the treatment in hydrogen plasma it can not be detected by the Auger spectrometer any more. Very clean surface with only the traces of oxygen can be seen in Fig. 1. After additional exposition of the sample to the oxygen plasma very uniform (black) silver oxide coating is obtained. Formation of silver oxide was confirmed by the Auger analysis. The concentration of oxygen at the surface of the layer is 13 at.% while on the other side the concentration falls to 6 at.%. Thickness of the layer is more than 25 nm and is very clean (Fig. 2). Passivation layer of chromized AgNi0.15 sample is resistant to K_2S and to wet atmosphere containing H_2S. Thickness of the coating layers of the samples exposed to hydrogen plasma was less than 1 nm. Coating contains Cr and small concentrations of N, P and Ca on the surface (Fig. 3). After the additional exposition to oxygen plasma traces of carbon could be found on the surface, while the inner part of the layer is oxidized with less than 10 at.% of oxygen (Fig. 4). Chromium is removed by the oxygen plasma. AES analysis of the AgNi0.15 with the sputtered thin TiN layer after the treatment with hydrogen plasma indicates that only the surface of the layer is oxidized (Fig. 5). There are carbon atoms inside the TiN layer. They were built-in during the deposition of TiN. When the same sample is treated also with the oxygen plasma the TiN layer surface was oxidized to the high degree (Fig. 6). It is evident that carbon was removed from the surface region. Estimated thickness of this sample's oxide layer is 5 nm. Nitride layer is so

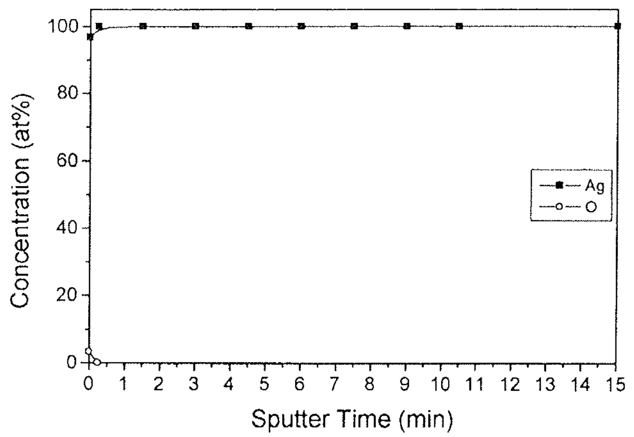

Fig. 1. Sample AgNi0.15 with the Silverbrite layer after exposition to hydrogen plasma

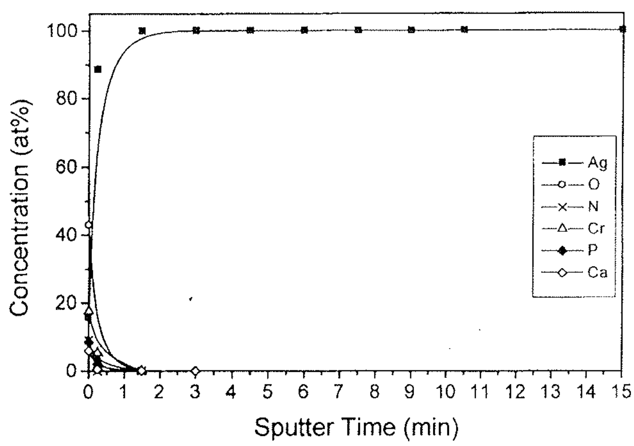

Fig. 3. Chromated AgNi0.15 sample after exposition to hydrogen plasma

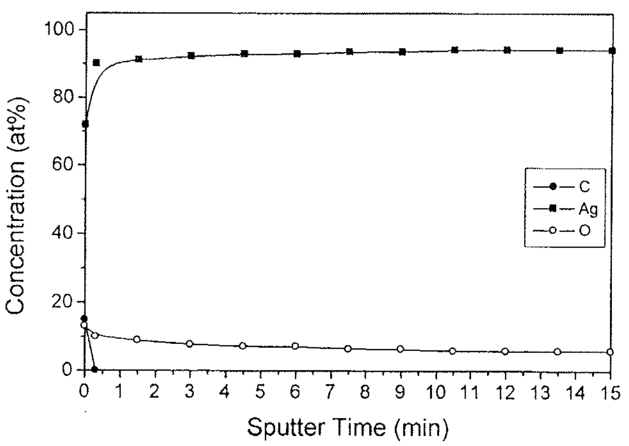

Fig. 2. Sample AgNi0.15 with the Silverbrite layer after exposition to hydrogen and oxygen plasma, respectively

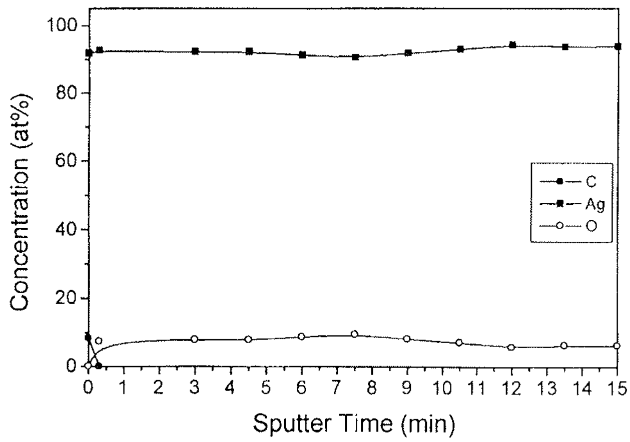

Fig. 4. Chromated AgNi0.15 sample after exposition to hydrogen and oxygen plasma, respectively

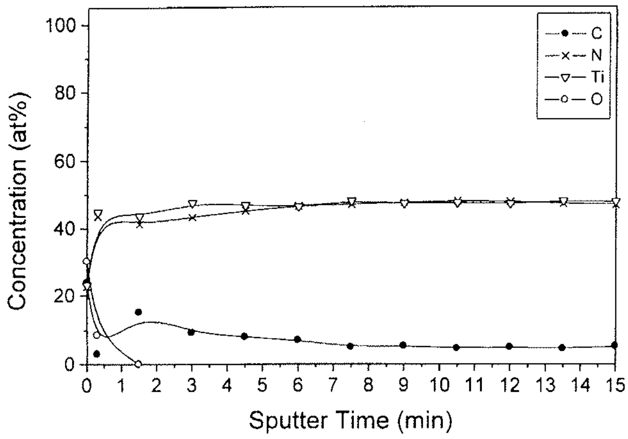

Fig. 5. Sample AgNi0.15 with the TiN thin layer after exposition to hydrogen plasma

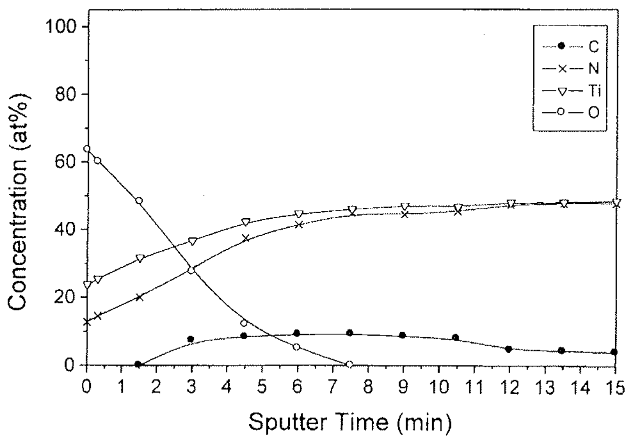

Fig. 6. Sample AgNi0.15 with the TiN thin layer after exposition to hydrogen and oxygen plasma, respectively

tightly bound to the base material that it is not removed during the treatment in the hydrogen RF plasma. Chemical resistance to the influence of the aggressive agents of the surroundings is excellent.

4 Conclusions

With the spectroscopy of Auger electrons three types of pasivation thin layers on the base contact material AgNi0.15 were analyzed after the treatment of samples in the hydrogen and oxygen RF plasma.

Results of pasivation of the contact material AgNi0.15 with respect to contact performances are very satisfactory therefore we suggest to consider also other contact materials on the silver basis.

TiN pasivation layer beside good contact performances exhibits also fair slide and wear properties. Therefore this type of layer is recommended for contacts in heavy duty switches (large number of switchings under difficult environmental conditions).

When Ag contact material is exposed to low pressure oxygen plasma oxidation to Ag oxide is possible. If the pasivation with plasma oxidation will prove to be effective it will be possible to clean and to protect the contacts in just one technological procedure.

References

/1/ M. Bizjak, L. Koller, K. Požun and J. Leskovšek, ICEC 98 Offenbach, VDE-Verlag (1998) 47-51.

/2/ L. Koller, M. Mozetič, K. Požun, M. Bizjak and S. Vrhovec, Kovine, zlitine, tehnologije, 32, 3-4 (1998) 255-257.

/3/ J. Schimkat, H-J. Gewatter and L. Kiesewetter, F&M, 104, 7-8 (1996) 515-518.

/4/ Doduco Datenbuch, 2. Aufl., Pforzheim, 1997.

/5/ Manual "Cromating AG-797100", Kemična tovarna Podnart.

/6/ B. Navinšek, P. Panjan and J. Krušič, Service and Coating Technology, 98 (1998) 809-815.

/7/ M. Mozetič and B. Praček, Informacije Midem, 28, 3 (1998) 172-174.

/8/ B. Praček, Kovine, zlitine, tehnologije, 30, 1-2 (1996) 53-55.

Lidija Koller, dipl. ing.
Institute for Electronics and Vacuum Technics
Teslova 30, 1000 Ljubljana, Slovenia
tel.: (+386 61) 177 6658
fax: (+386 61) 126 4578
e-mail: joze.koller@uni-lj.si

Doc. dr. Martin Bizjak, dipl. ing.
Iskra - Stikala, Kranj
Savska loka 4, 4000 Kranj, Slovenia
tel.: (+386 64) 372 226
fax: (+386 64) 372 259
e-mail: iskra.stikala.rd@siol.net

Prispelo (Arrived): 8.3.99 *Sprejeto (Accepted): 9.6.99*