Journal of the Serbian Chemical Society
JSCS-info@shd.org.rs • www.shd.org.rs/JSCS

J. Serb. Chem. Soc. 84 (7) 729–741 (2019)
JSCS–5222

UDC 546.56:531.004.12+544.654.2:
621.793+534–8
Original scientific paper

Mechanical characterization of copper coatings electrodeposited onto different substrates with and without ultrasound assistance

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(Received 3 October 2018, revised 7 March, accepted 11 March 2019)

Abstract: The mechanical properties of systems consisting of copper coatings electrodeposited on both brass sheet (BS) and thick electrodeposited nickel coating (ED Ni) substrates have been investigated. The electrodeposition of copper coatings was performed with and without the ultrasound assistance. The ultrasound application decreases root mean square (RMS) roughness of deposited Cu coating on both applied substrates, as obtained from non-contact AFM measurement. The coating roughness is highly dependent on the substrate roughness, being the smallest for the Cu coatings deposited on ED Ni substrate with the ultrasound mixing. The hardness and adhesion properties were characterized using the Vickers microindentation test. Model of Korsunsky was applied to the experimental data for determination the film hardness and the model of Chen-Gao was used for the adhesion evaluation. The introduction of ultrasonic agitation caused the changes in the film microstructure, and consequently in the mechanical properties. The copper coatings on both substrates, have higher hardness when deposited from electrolyte with ultrasound agitation. Although the type of the substrate has the major influence on the adhesion strength, it can be said that Cu electrodeposition with ultrasonic mixing contributes to an increase in adhesion.

Keywords: Cu electrodeposition; ultrasonic agitation; composite hardness; coating adhesion.

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https://doi.org/10.2298/JSC181003023M

INTRODUCTION

Thin copper coatings are widely used material in electronic industry for the fabrication of contacts in integrated circuits, realization of HAR (high aspect ratio) channels or fabrication of different structures with copper as a sacrificial layer material.[1,2] They are widely utilized in filling and covering flat substrates with regular holes of micro and nano-dimension (damascene and through silicon *via* (TSV) technologies).[3,4,5] Electrodeposited copper films have found their use in the fabrication of microelectromechanical (MEMS) devices for a wide range of applications.[6]

The copper electrochemical deposition (ED) is a low-temperature and easy--controlled technique with relatively high deposition rate. Electrolytes that are commonly used for the copper deposition are sulphate based, with the possibility of adding different additives. The suppressor additives like polyethylene glycol (PEG) and chloride ions inhibit the copper deposition, while the accelerator additives like 3-mercapto-1-propanesulfonate acid (MPSA) enhances the rate of the copper deposition.[7,8]

The introduction of ultrasound (US) into electrochemical deposition is a known way to improve the microstructural and mechanical properties of metal coatings of chromium, cobalt, silver, nickel, iron etc. The ultrasonic mixing of an electrolyte leads to changes in the film microstructure in terms of changing the direction of grain growth. The grains grow preferentially in the manner parallel to the substrate surface. The ultrasound-assisted electrodeposition is a method that can contribute to the improved surface morphology, adhesion and fatigue strength, tensile stress and hardness of the coatings.[9,10]

The two important mechanical properties of thin metallic coatings are hardness and adhesiveness. The adhesion strength of metallic coatings on various substrates is a serious problem in realization of MEMS devices due to the delamination of the coatings under stress. Therefore, a new ways to achieve improved mechanical properties of electrodeposited metallic coatings are actively being researched.

A coating and a substrate can be considered together as a composite system, the properties of which depend not only on particular material properties of the coating and the substrate, but also on the composite parameters such as good adhesion, controlled residual stresses, good corrosion resistance, etc.

Hardness testing is a widely used technique for assessing the structural and mechanical properties of the composite systems. As the thickness of the coating is very small, the influence of the substrate must be considered during the hardness determination.

The measured hardness of composite systems is influenced by a number of factors such as coating thickness, indentation depth, coating and substrate hardness and hardness ratio as well as adhesion. It has been shown that the micro-

hardness testing can be a useful technique in assessing the adhesion of thin films to the substrate.[11–15]

The aim of the study was to analyze the hardness response of the selected composite systems and analyze the results of the quantitative assessment of coating adhesion based on the measured composite hardness.

The versatility of composite systems was achieved by combining various substrates and copper coatings. The change of coating microstructure and hardness was performed using the electrodeposition with and without the ultrasonic assistance.

The selected thickness of the coatings allowed the analysis of the composite hardness in a large load range, from low loads when the hardness of the film in the measured composite hardness is dominant, to higher loads when the influence of the substrate hardness is primary.

The adhesion estimate, quantitatively expressed over a critical reduced depth (the ratio of the plastic zone radius to the indentation depth), was made based on the measurement of the composite hardness for all the composite systems.

Theory of composite hardness and adhesion models

There is a problem of determining the coating hardness separately from the measured composite hardness. The composite and the coating hardness values depend on the applied loads. The change of the composite and the coating hardness with the load depends on the composite system structure.

The composite hardness model of Korsunsky was found to be appropriate for the experimental data analysis and film hardness determination.[11]

According to this descriptive model, the correlation between composite hardness, H_c, coating hardness, H_f, and substrate hardness, H_s, is given as:

$$H_c = H_s + \left[\frac{1}{1+k'\left(\frac{d^2}{t}\right)}\right]\left(H_f - H_s\right);\ k' = \frac{k}{49t} \tag{1}$$

where t is the thickness of the film, d is the indent diagonal and k' is a dimensionless material parameter related to the composite response mode.

For the evaluation the adhesion properties of thin coatings, Chen-Gao (C-G) method was chosen.[12–15] This method introduces the composite hardness as a function of the critical reduced depth, b, beyond which the material will have no effect on the measured hardness. The critical reduced depth b represents the ratio between the radius of the plastic zone beneath the indentation and the indentation depth. A large value of the critical reduced depth corresponds to the good adhesion, while low values indicate poor adhesion of the coatings, as shown in Fig. 1.

According to C-G model, the correlation between composite, coating and substrate hardness values and the critical reduced depth *b* is given by:

$$H_c = H_s + \left[\frac{(m+1)t}{mbD}\right](H_f - H_s) \quad (2)$$

where *D* is the indentation depth and *m* is the power index. Critical reduced depth *b* has different values for various coating-substrate systems.

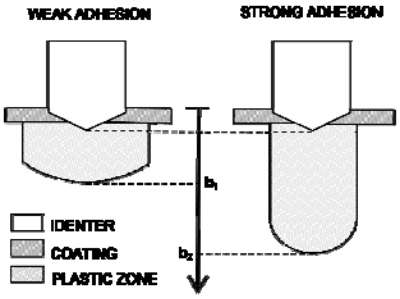

Fig. 1. Schematic representation of deformation associated with indentation in a coated substrate.[12]

The appropriate value for the power index *m* is found to be 1.8 for a system of soft film on a hard substrate. This value is the intermediate between the value predicted by assuming an area law of mixtures ($m = 1$) and the mixtures of low volume of ($m = 2$).[16,17] Then, introducing the diagonal *d* of the indentation with $d = 7D$, for a Vickers indentation test and $\Delta H = H_s - H_c$, Eq. (2), it becomes:

$$\Delta H = \left[\frac{7(m+1)(H_s + H_f)}{mb}\right]\frac{t}{d} \quad (3)$$

The critical reduced depth *b* can be calculated by using Eq. (3) with experimental values of H_c, H_f, *t* and *d*.

EXPERIMENTAL

Two types of substrates were employed for experimental work. The first substrate was 125 μm-thick brass foil (2601/2 hard, ASTM B36, K&S Engineering) and this substrate is denoted with BS in the further text. The second one was 50 μm-thick Ni coating electrodeposited on brass foil, and this substrate is further denoted with ED Ni.

ED Ni substrate was prepared by Ni electrodeposition from sulphamate electrolyte consisting of 300 g L^{-1} $Ni(NH_2SO_3)_2 \cdot 4H_2O$, 30 g L^{-1} $NiCl_2 \cdot 6H_2O$, 30 g L^{-1} H_3BO_3, 1 g L^{-1} saccharine on brass foil. Prior to deposition, the brass foil was degreased and chemically polished in acid mixture of HNO_3:H_3PO_4:CH_3COOH of 4:11:5 volume ratio. Electrochemical deposition was carried out using direct current (DC) galvanostatic mode with the current density value maintained at 50 mA cm^{-2}. The temperature and pH-value were maintained at 50 °C and 4.20, respectively.

Copper coatings were electrodeposited on the both substrates from the sulfate electrolyte consisting of 240 g L^{-1} $CuSO_4 \cdot 5H_2O$, 60 g L^{-1} H_2SO, 0.124 g L^{-1} NaCl, 1 mg L^{-1} polyethylene glycol (PEG), 1.5 g L^{-1} 3-mercapto-1-propanesulfonic acid (MPSA) and deionized water. This

electrolyte was used because it enables electrodeposition of Cu in the form of mirror bright coatings.[18,19]

DC-galvanostatic mode was used for the electrochemical deposition, with the current density value maintained at 50 mA cm^{-2}. The process temperature and pH-value were maintained at 25 °C and 0.30, respectively. The deposition rates of the Cu coatings were determined for the deposition performed under different mixing conditions: without stirring and with the assistance of agitation in ultrasonic bath (40 kHz, Bransonic 220 ultrasonic cleaner). Then, the time of the deposition was determined according to the plating surface, current density value and projected film thickness of 20 μm.

The thickness of the coatings was controlled by measuring the mass of the samples before and after the deposition process. The cross-sections of several samples were prepared and the thickness of the coatings was measured and checked by optical microscopy. The results of the measurement showed good agreement.

The roughness and topographic details analysis of the two used substrates and electrodeposited copper coatings on them without and with ultrasound assistance was done by atomic force microscopy (AFM, TM microscopes-Vecco in non-contact mode). The root mean square (RMS) roughness parameter, that represents the standard deviation of the distribution of surface heights and which is sensitive to large deviation from the mean line, was taken to express the roughness of the substrates and electrodeposited coatings.

The mechanical properties of the composite systems were characterized using Vickers microhardness tester "Leitz, Kleinharteprufer DURIMET I" with loads ranging from 1.96 down to 0.049 N. Three indentations were made at each load from which the average value of composite hardness could be calculated.

RESULTS AND DISCUSSION

Surface morphology and roughness analysis

The surface morphology and the roughness of used substrates and copper coatings electrodeposited on them, without and with ultrasound assistance, obtained by the AFM technique are shown in Figs. 2–4, respectively.

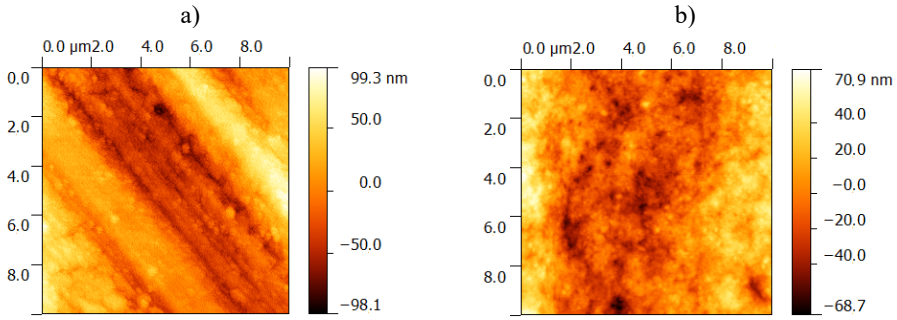

Fig. 2. Substrates in the processes of electrodeposition: a) brass (BS); b) ED Ni.

Surface roughness of the substrates and the coatings was expressed by their root mean square (RMS) roughness derived from the AFM images for a scanned area of 100 μm^2. Results given in Table I show the influence of ultrasound mix-

ing of electrolyte on RMS roughness. From Table I, it can be noticed that the RMS roughness for ED Ni substrate were about two times smaller than the same values for the BS substrate.

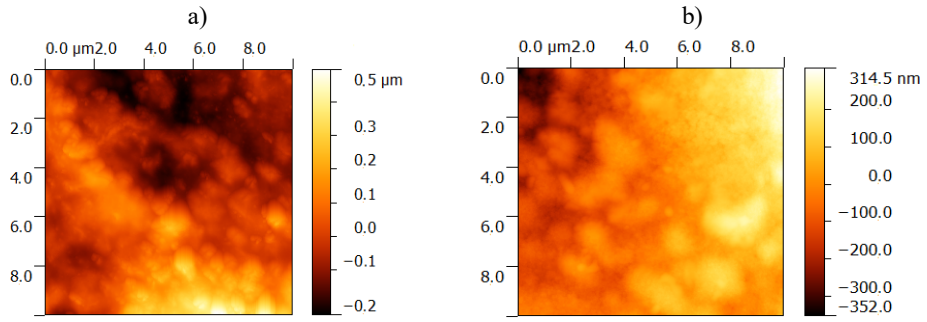

Fig. 3. AFM images of copper coatings electrodeposited on BS substrate: a) Cu coating deposited from silent bath, b) Cu coating deposited from ultrasonically mixed electrolyte.

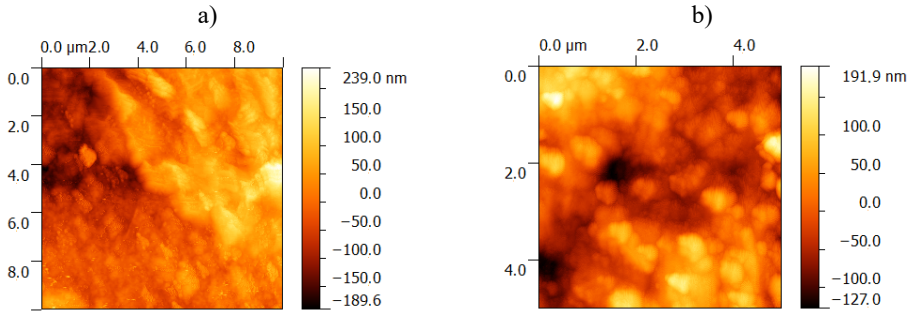

Fig. 4. AFM images of copper coatings electrodeposited on ED Ni substrate: a) Cu coating deposited from silent bath; b) Cu coating deposited from ultrasonically mixed electrolyte.

TABLE I. Surface roughness values of substrates and Cu coatings electrodeposited with and without the ultrasound agitation

Substrate	Coating	Ultrasound	RMS roughness, nm
BS	–	–	34.1
BS	Cu	–	126.3
BS	Cu	+	119.5
ED-Ni	–	–	18.1
ED-Ni	Cu	–	66.6
ED-Ni	Cu	+	52.8

At the first sight, it can be mentioned the considerable increase of RMS roughness for Cu coatings in relation to the same values for the substrates. In the case of Cu electrodeposition on BS substrate without application of ultrasound, the values of RMS roughness were 3.70 times larger than the corresponding values for the BS substrate. With ultrasound assisted electrodeposition, these

values were 3.50 times larger than the RMS roughness for the brass substrate. The similar changes are also observed with use of ED Ni substrate. Without the ultrasound assisted electrodeposition, the values of RMS roughness were 3.7 times larger than the values for ED Ni substrate. However, when Cu electrodeposition was performed in the presence of ultrasound on the ED Ni substrate, the RMS roughness were 3.0 times larger than the values for this substrate. Although the values obtained in the presence of ultrasound were smaller than those obtained without the ultrasound agitation, it is necessary to note that there is no any significant difference between the values obtained with and without application of ultrasound.

According to expectations, the finest morphology of the electrodeposited Cu film was achieved on the fine-grained 50 μm-thick ED Ni film, as the substrate in the presence of ultrasound, what is a result of useful effects of both the addition of additives and the application of electrolyte stirring on the metal electrodeposition process.[20]

Absolute hardness of the substrates

The indentation tests were performed on brass foils and 50-μm thick ED Ni coatings as the substrates in order to observe their response to indentation, due to their different microstructure. The load-independent microhardness values of the substrates were calculated according to the proportional specimen resistance (PSR) model:[17,20]

$$P = a_1 d + \left(\frac{P_c}{{d_0}^2} \right) d^2 \tag{4}$$

Parameter P_c is the critical applied load above which microhardness becomes load independent and d_0 is the corresponding diagonal length of the indent. The measured values and linear fit of P/d against d are shown in Fig. 5.

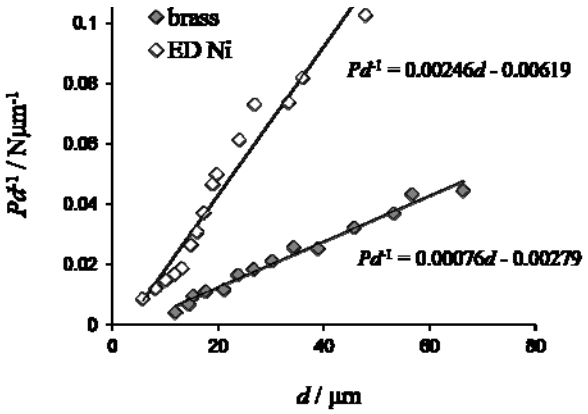

Fig. 5. PSR plot of applied load trough indent diagonal, Pd^{-1}, *vs.* indent diagonal, d, for BS substrate and for ED Ni substrate.

The value of P_c/d_0 for the brass substrate was calculated as 7.6×10^{-4} GPa/μm and for the thick ED Ni substrate was 2.4×10^{-3} GPa /μm.

Three independent measurements of indent diagonal size for each applied load were performed and the average values were calculated. The absolute substrate hardness and composite hardness values, H (in GPa), were calculated using the equation:

$$H = 0.01854\, P\, d^{-2} \tag{5}$$

where 0.01854 is geometrical factor for the Vickers indenter.

Variation in composite and coating hardness

It is supposed that the systems of electrodeposited copper coatings on brass and thick ED Ni coatings as the substrates belong to the "soft film on hard substrate" composite system type. The thickness of the electrodeposited nickel coatings of 50 μm is sufficient in terms of the hardness value to allow the coating to be chosen as the substrate.[20] Dependence of the composite hardness, H_c, on the relative indentation depth (*RID* – the ratio between indent depth and coating thickness) for the mentioned systems is given in Figs. 6. and 7.

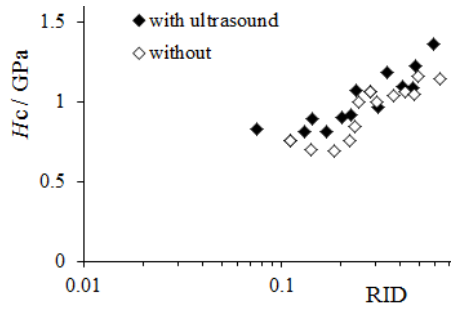

Fig. 6. Composite hardness, H_c, variation with relative indentation depth, *RID*, for 20 μm thick Cu coating on BS substrate with and without ultrasound assistance.

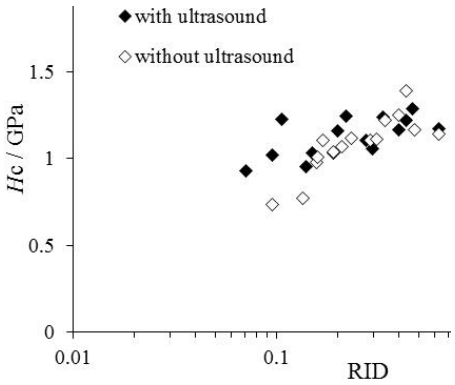

Fig.7. Composite hardness, H_c, variation with relative indentation depth, *RID*, for 20 μm thick Cu coating on ED Ni substrate with and without ultrasound assistance.

As shown on Fig. 6, the hardness of thin copper coatings electrodeposited with 50 mA cm^{-2} current density on BS substrate increases with introducing the ultrasound agitation into an electrolyte. The relative indentation depth between 0.1 and 1 corresponds to the hardness response of the whole composite system.

The increase of the composite hardness values for the system of electrodeposited copper on ED Ni substrate in the presence of ultrasound or without it, is also recorded. It is shown on Fig. 7.

The tendency of composite hardness H_c with *RID*, as shown in Figs. 6 and 7 is characteristic for the "soft film on hard substrate" type of composite systems. With the increase of the relative indentation depth above 1, the hardness values of the system will approach the hardness of the substrate for both systems.[20]

Korsunsky model was applied to experimental data in order to determine the absolute hardness of copper coatings, H_f. The fitting results are presented in Table II.

TABLE II. Absolute hardness of 20 μm thick ED copper coatings, according to Korsunsky model

Substrate	Ultrasound	H_s / GPa	H_f / GPa	$k'\times10^6$
BS	+	1.41	0.7355	47.41
BS	–	1.41	0.6333	58.71
ED Ni	+	4.63	1.0700	0.984
ED Ni	–	4.63	0.9786	1.811

The dimensionless material parameter k' from Korsunsky model, is related to the response mode of the composites and defined in Eq. (1).

As shown in Table II, the ultrasonic agitation contributes to the increase of the electrodeposited copper coatings hardness for coatings, which have been deposited on the same substrates. Coatings deposited on ED Ni have higher hardness in general, but the tendency of the hardness increase for coatings deposited under ultrasound agitation is preserved. Higher absolute hardness for Cu coatings, deposited on ED Ni substrates, in comparison with Cu coatings deposited on BS substrates under the same deposition and mixing conditions, can be explained by higher adhesion energy for Cu coatings on ED Ni than for Cu coatings on BS, as discussed in next section.

Composite hardness and adhesion

The evaluation of the interlayer adhesion strength of 20 μm-thick copper coatings electrodeposited on different substrates was performed according to the composite hardness model of Chen-Gao.[12] The composite hardness of the coating/substrate system is expressed by Eq. (2) and in the form of Eq. (3) was used to calculate the critical reduced depth b (the ratio between the radius of the plastic zone beneath the indenter and the indentation depth). Substrate (H_s) and com-

posite (H_c) hardness were calculated using directly measured indent diagonals on substrate and coating surfaces, respectively. The hardness of the ED Cu coatings, H_f, was obtained as the result of the applied model of Korsunsky (Table II).

In Fig. 8, the measured values of $\Delta H = H_s - H_c$ are plotted *vs.* td^{-1} (ratio between the coating thickness and the indentation diagonal). A linear fit of experimental data was performed, based on Eq. (3), and the values of the fitted curve slope k are reported in the same figure.

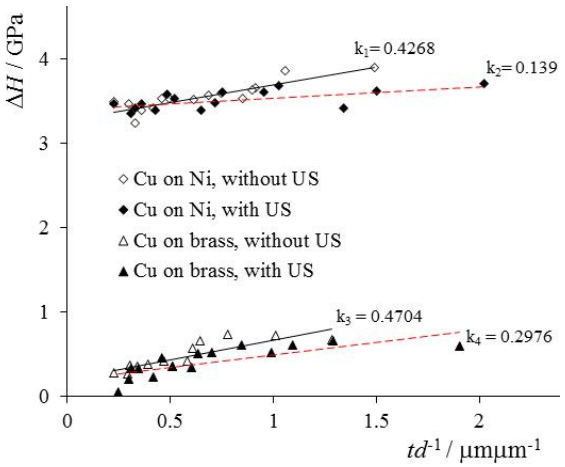

Fig. 8. The micro-hardness difference, $\Delta H = H_c - H_s$, *vs.* the ratio of coating thickness to indentation diagonal, td^{-1}, for electrolytically obtained Cu coatings on BS and ED Ni substrates with and without ultrasound. The slope values (k) are indicated.

By using m = 1.8 as the appropriate value of the power index, values of b were calculated and given in Table III.

TABLE III. Critical reduced parameter, b, for 20 μm thick ED copper coatings on different substrates

Substrate	Ultrasound	m	k	b
BS	+	1.8	0.2976	24.679
BS	–	1.8	0.4704	17.986
ED Ni	+	1.8	0.1390	278.88
ED Ni	–	1.8	0.4268	93.158

The good adhesion properties correspond to the increasing values of the plastic deformation zone radius around the indentation and the critical reduced depth, b. High values of the critical reduced depth correspond to better adhesion properties. It is obvious that the values of b are significantly higher for the ED Cu coating on ED Ni substrate than for the Cu coating electrodeposited under same conditions on BS substrate. For both systems, the adhesion increased with the use of ultrasound agitation, which is more noticeable for the ED Cu coating system on ED Ni substrate, due to more similar microstructures between substrate and coating.

According to Fig. 8, it can be concluded that the quality of adhesion can be assessed based on the microhardness measurements. The difference of the substrate hardness and composite hardness, $\Delta H = H_s - H_c$, decreases more rapidly with the increase of the indentation load, for poor adhesion.

CONCLUSION

Copper was electrodeposited from sulfate electrolyte with addition of additives for leveling and brightness on brass (BS) and thick electrodeposited nickel coatings (ED Ni) substrates. DC-galvanostatic electrodeposition was performed with and without ultrasonic agitation of sulfate electrolyte. The analysis of the influence of the substrate type and ultrasonic mixing on microstructure and composite hardness properties was performed.

The tests of microindentation were performed on BS and ED Ni substrates to observe their hardness response. The thickness of nickel coating (50 μm) electrodeposited on BS was sufficient to allow the coating to be considered as the substrate. The BS substrate hardness was calculated as 1.41 GPa, and 4.63 GPa for the ED Ni substrate.

Considering experimental results, the composite hardness model of Korsunsky was applied to calculate the coating hardness. It is shown that Cu coatings electrodeposited on the ED Ni substrates have higher values of the hardness, than Cu coatings electrodeposited on the brass substrates. Higher values of the hardness were obtained for the ultrasound-assisted electrodeposition in comparison with those obtained without application of ultrasound.

The composite hardness model of Chen-Gao was used for the adhesion assessment of Cu coatings on different substrates through the values of microhardness. The system obtained by Cu electrodeposition on ED Ni substrate had significantly better adhesion strength than the system obtained by electrodeposition of Cu on BS (brass) as the substrate, with high values of critical reduced depth, b, as the adhesion parameter. An increase in the adhesion strength was observed for the coatings electrodeposited under ultrasound mixing. The quality of adhesion can be assessed based on microhardness measurements. The microhardness difference $\Delta H = H_s - H_c$ decreases more rapidly with the increase of the indentation load, for poor adhesion.

The coating roughness values depend on the substrate type and agitation conditions. The best morphology of the Cu coatings was achieved with ultrasonic-assisted electrodeposition on the fine-grained ED Ni substrate, with the average roughness value of 43 nm.

Acknowledgement. This work was funded by Ministry of Education, science and Technological Development of Republic of Serbia through the projects TR 32008, TR 34011 and III 45019.

ИЗВОД

КАРАКТЕРИЗАЦИЈА МЕХАНИЧКИХ СВОЈСТАВА ПРЕВЛАКА БАКРА ЕЛЕКТРОХЕМИЈСКИ ИСТАЛОЖЕНИХ НА РАЗЛИЧИТИМ ПОДЛОГАМА УЗ ПРИМЕНУ И БЕЗ ПРИМЕНЕ УЛТРАЗВУЧНОГ МЕШАЊА

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Испитивана су механичка својства композитних система који се састоје од електрохемијски исталожених превлака бакра на месингу (BS) и дебелим електрохемијски исталоженим превлакама никла на месинганој фолији (ED Ni) као супстратима. Превлаке бакра на наведеним супстратима су исталожене из електролита без мешања или са ултразвучним мешањем. Неконтактна микроскопија атомских сила (AFM) је показала да храпавост, изражена средњом вредношћу квадратног одступања (*RMS*), исталожених превлака бакра на обе врсте супстрата опада са применом ултразвучног мешања. Храпавост превлака у највећој мери зависи од храпавости супстрата, при чему су електрохемијски исталожене превлаке бакра са најмањом храпавошћу реализоване на супстратима Ni електрохемијским таложењем из електролита мешаног применом ултразвука. Механичка својства тврдоће и адхезије превлака су анализирана Викерсовим тестом утискивања са малим оптерећењима. За израчунавање апсолутне тврдоће превлака коришћен је модел Korsunsky, док је за процену адхезије коришћен модел Chen-Gao. Примена ултразвучног мешања током процеса електрохемијског таложења бакра довела је до промена у микроструктури превлака, па самим тим и промена у механичким својствима превлака. Превлаке бакра на оба супстрата имају већу тврдоћу када се таложе из електролита уз ултразвучно мешање. На адхезију превлаке на подлогама највише утиче тип супстрата, али се може рећи да примена ултразвучног мешања доприноси побољшању адхезије електрохемијски исталоженог бакра на наведеним супстратима.

(Примљено 3. октобра 2018, ревидирано 7. марта, прихваћено 11. марта 2019)

REFERENCES

1. W. H. Teh, L. T. Koh, S. M. Chen, J. Hie, C. Y. Li, P. D. Foo, *Microelectron. J.* **32** (2001) 579 (https://doi.org/10.1016/S0026-2692(01)00035-0)
2. A. Maciossek, B. Lochel, H. J. Quenzer, B. Wagner, S. Schulze, J. Noetzel, *Microelectron. Eng.* **27** (1995) 503 (https://doi.org/10.1016/0167-9317(94)00154-M)
3. F. Wang, P. Zeng, Y. Wang, X. Ren, H. Xiao,W. Zhu, *Microelectron. Eng.* **180** (2017) 30 (http://dx.doi.org/10.1016/j.mee.2017.05.052)
4. W. P. Dow, M. Y. Yen, C. W. Liu, C. C. Huang, *Electrochim. Acta* **53** (2008) 3610 (https://doi.org/10.1016/j.electacta.2007.12.048)
5. K. Kondo, N. Yamakawa, Z. Tanaka, K. Hayashi, *J. Electroanal.Chem.* **559** (2003) 137 (https://doi.org/10.1016/S0022-0728(03)00110-4)
6. J. Lamovec, V. Jović, I. Mladenović, M. Sarajlić, V. Radojević, in *Proc. 57th ETRAN Conference*, 3–6 June, 2013, Zlatibor, Serbia, pp. MO3.3
7. L. Bonou, M. Eyraud, Y. Massiani, *Electrochim. Acta* **47** (2002) 4139 (https://doi.org/10.1016/S0013-4686(02)00356-0)
8. M. Datta, D. Landolt, *Electrochim. Acta* **45** (2000) 2535 (https://doi.org/10.1016/S0013-4686(00)00350-9)

9. C. T. Walker, R. Walker, *Electrodepos. Surface Treat.* **1** (1973) 457 (https://doi.org/10.1016/0300-9416(73)90029-1)
10. R. Cui, Y. He, Z. Yu, W. Shu, J. Du, in *Proc. of 11th International Conference on Electronic Packaging Technology & High Density Packaging*, 16-19.8, 2010, China, p.847
11. A. M. Korsunsky, M. R. McGurk, S. J. Bull, T. F. Page, *Surface Coat. Technol.* **99** (1998) 171 (https://doi.org/10.1016/S0257-8972(97)00522-7)
12. M. Chen, J. Gao, *Modern Phys. Lett., B* **14** (2000) 103 (https://doi.org/10.1142/S0217984900000161)
13. J. L. He, W. Z. Li, H. D. Li, *Appl. Phys. Lett.* **69 (**1998) 1402 (https://doi.org/10.1063/1.117595)
14. H. Qingrun, J. Gao, *Modern Phys. Lett., B* **11** (1997) 757 (https://doi.org/10.1142/S0217984997000931)
15. L. Magagnin, R. Maboudian, C. Carraro, *Thin Solid Films* **434** (2003) 100 (https://doi.org/10.1016/S0040-6090(03)00469-3)
16. Q. R. Hou, J. Gao, S. J. Li, *Eur. Phys. J., B – Cond. Matter Complex Sys.* **8** (1999) 493 (https://doi.org/10.1007/S100510050716)
17. H. Li, R. C. Bradt, *J. Mater. Sci.* **28** (1993) 917 (https://doi.org/10.1007/BF00400874)
18. N. D. Nikolić, Z. Rakočević, K. I. Popov, *J. Electroanal. Chem.* **514** (2001) 56 (https://doi.org/10.1016/S0022-0728(01)00626-X)
19. N. D. Nikolić, Z. Rakočević, K. I. Popov, *J. Solid State Electrochem.* **8** (2004) 526, (https://doi.org/10.1007/s10008-003-0467-8)
20. J. Lamovec, V. Jović, D. Randjelovic, R. Aleksic, V. Radojevic, *Thin Solid Films* **516** (2008) 8646 (http://dx.doi.org/10.1016/j.tsf.2008.06.035).