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Indentation behaviour of „soft film on hard substrate“ composite system type

ABSTRACT

This investigation has been carried out in order to analyse and compare the hardness response of different composite systems of the same type, named „soft film on hard substrate“ composite system. Composite systems of mono- and multilayered electrodeposited Ni and Cu thin films on (100) and (111)-oriented monocrystalline Si wafers and 100 µm-thick electrodeposited Ni film as the substrates were fabricated.

The indentation behaviour of these composite structures was characterized using Berkovich nanohardness and Vickers microhardness testing.

The measured hardness is so-called „composite hardness“, because the substrate participates in the plastic deformation during the indentation process. The contribution of the substrate to the measured hardness starts at indentation depths of the order of 0.07- 0.2 times the film thickness.

Dependence of nanohardness and microhardness values on electrodeposition process conditions, substrate and film microstructure, total film thickness, layer thickness and Ni/Cu layer thickness ratio for different composite systems ratio was investigated. Composite hardness model of Chicot-Lesage (C-L) was chosen and applied to experimental data in order to determine absolute film hardness.

Keywords: *composite hardness, nanohardness, vickers microhardness, hardness model, Ni/Cu electrodeposition, Ni/Cu multilayers*

1. INTRODUCTION

Thin metallic monolayer or multilayer film structures are often used in fabrication of different microelectronic and micromechanical devices. Thin film and substrate constitute a composite system which properties depend not only on material parameters of the film and of the substrate but also on the composite parameters such as the residual stress and adhesion. It is possible to fabricate composite systems of multilayer thin film of different materials on substrates, with small layer thickness (less than 1µm), that have specific mechanical, magnetic and optical properties [1-3].

Development of the reliable techniques for metal layer deposition in controlled manner is of great interest in the MicroElectroMechanicalSystem (MEMS) technologies. An interesting approach for growing of metal and alloy layers is the use of electrochemical deposition technique. Electrochemical deposition (ED) is fully compatible with MEMS technologies and has several advantages in comparison to another deposition processes: it is a low-temperature deposition technique with easy-controlled high deposition rate that allows a wide thickness range, the easy control of thickness and residual stress, chemical composition of the layer and microstructure (grain size) of the deposits [4-6].

Ni and Cu thin films are recognized and used for applications in microsystem technologies owing to their low resistivity, low cost and easy to deposit electrochemically. Also, thin Ni/Cu multilayer film is technologically interesting system that attracted great attention owing to high mechanical strength, high wear and corosion resistance and giant magnetoresistance behaviour [7-11].

Indentation testing is reliable test method for the evaluation of mechanical properties of bulk and thin-film materials over a wide range of size scales. Nanoindentation has become the standard technique used for nanomechanical characterization of materials where force-displacement curves obtained during the indentation process give informations about hardness and elastic modulus pro-

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perties. During hardness determination of thin films and coatings by indentation method, the influence of the substrate must be considered. Above a certain critical indentation depth, the measured „composite“ hardness is a complex value depending on the relative indentation depth and structural and mechanical properties of both the composite film and the substrate.

There is a need to obtain the hardness of the film solely from the experimental composite hardness measurements. Change of the composite and film hardness with applied loads depends on the composite structure (especially on the substrate type). The composite hardness model of Chicot-Lesage (C-L) was chosen and applied to experimental data in order to analyse the influence of the substrate for different composite systems [12,13].

2. THEORY OF COMPOSITE HARDNESS MODEL

The model proposed by Chicot and Lesage (C-L) [12,13] is constructed on the analogy between the variation of the Young’s modulus of reinforced composites in function of the volume fraction of particles, and the variation of the composite hardness between the hardness of the substrate and that of the film.

Hardness value calculated from an indentation test is not constant because hardness is load-dependent. Meyer’s law is an empirical relation between the variation of the size of the indent in function of the applied load *P*. For the particular case of a film-substrate couple, the relation between the measured diagonal and the applied load and Meyer’s relation are similar in appearance:

$$P = a^* d^{n^*} \tag{1}$$

The variational part of the hardness number with load is represented by the factor *n**. Then they adopted the following expression:

$$f\left(\frac{t}{d}\right) = \left(\frac{t}{d}\right)^m = f \quad where \quad m = \frac{1}{n^*} \tag{2}$$

Composite hardness can be expressed by the next relation:

$$H_C = (1-f)/\left(1/H_S + f\cdot\left(\frac{1}{H_F} - \frac{1}{H_S}\right)\right) + f\cdot\left(H_S + f\cdot\left(H_F - H_S\right)\right) \tag{3}$$

Hardness of the film is the positive root of the next equation:

$$\begin{aligned} &A\cdot H_F^2 + B\cdot H_F + C = 0 \\ &A = f^2\cdot(f-1) \\ &B = \left(-2f^3 + 2f^2 - 1\right)\cdot H_S + (1-f)\cdot H_C \\ &C = f\cdot H_C\cdot H_S + f^2\cdot(f-1)\cdot H_S^2 \end{aligned} \tag{4}$$

The value of m (composite Mayer’s index) is calculated by a linear regression performed on all experimental points obtained for a given film substrate couple and deduced from the relation:

$$\ln d = m\cdot \ln P + b \tag{5}$$

With the known value of m, only the hardness of the films remains to calculate.

3. EXPERIMENTAL

3.1. Substrates and film deposition

For the hardness measurement experiments two different substrates were prepared: single crystalline Si(100) and Si(111)-oriented wafers and 100-μm thick fine-grained films of electrodeposited Ni.

The plating base for the Si wafers were sputtered layers of 100Å Cr as the adhesion film and 800Å Ti as the nucleation film. Electrochemical deposition (ED) was performed under DC galvanostatic regime. Nickel was electrodeposited from a sulphamate bath consisting of 300 g/l $Ni(NH_2SO_3)_2\cdot 4H_2O$, 30 g/l $NiCl_2\cdot 6H_2O$, 30 g/l H_3BO_3, 1 g/l saccharine, and Cu from a sulphate bath consisting of 240 g/l $CuSO_4\cdot 5H_2O$, 60 g/l H_2SO_4. Ni and Cu layers in multilayer films were alternately electrodeposited from two above-mentioned electrolytes (dual bath technique, DBT) [14]. The current density values were maintained at 10 mA/cm^2 and 50 mA/cm^2. According to the plating surface and plating conditions, projected thickness of deposits was determined.

3.2. Micro and nanoindentation tests

Nanoindentation tests on the substrates and film samples were performed using the Nanoindenter Hysitron Triboindenter with a Berkovich diamond tip and in-situ imaging scaning mode. The indentation maximum load was set on 8mN for all samples and a minimum of five indents were performed. The hardness and reduced elastic modulus were calculated from the indentation curves using the Oliver and Pharr method [15]. The loading, hold and unloading times at the peak force were set to 15s, 30s and 5s, respectively.

The mechanical properties of the composite systems were characterized using Vickers microhardness tester „Leitz, Kleinharteprufer DURIMET I“ with loads ranging from 1.96 N down to 0.049 N. Three indentations were made at each indentation load from which the average composite hardness could be calculated. Experimental data were fitted with GnuPlot, v 4.2 (http://www.gnuplot.info/).

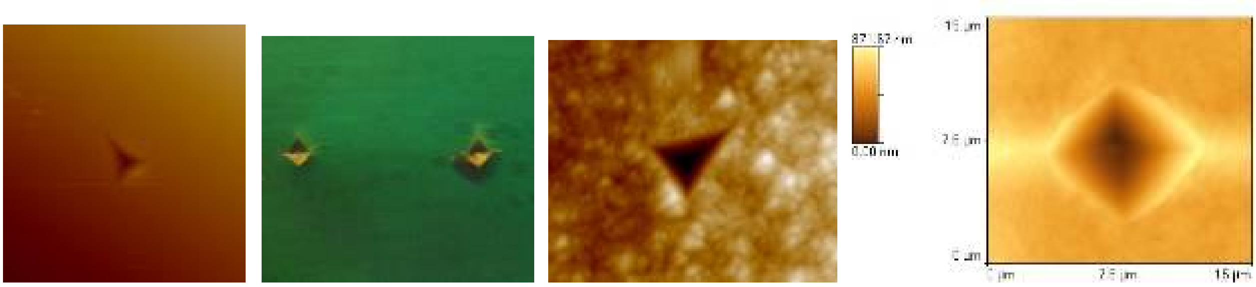
Topographic examination was done by metallografic (Carl Zeiss microscop „Epival Interphako“) and atomic force microscopy (AFM, TM Microscopes-Veeco, non-contact mode).

4. RESULTS AND DISCUSSION

4.1. Mechanical properties of the substrates

Nano- and microindentations were performed on uncoated substrates of single-crystal Si(100) and Si(111) wafers and polycrystalline ED Ni film in order to observe their response to indentation according to their different microstructure and load range (Fig.1).

Brittle fracture of single crystalline Si is evident for high indentation loads and cracks that occur around the indent in order to release the strain energy can be noticed on Fig.1.b. Evidence of plastic deformation and pile-up phenomenon for composite system of electrodeposited nickel film on hard Si substrate is shown on Fig.1.d.



(a) *(b)* *(c)* *(d)*

Figure 1 - Images of nanoindentation and microindentation response on Si wafer (a) and (b) and on 5µm-thick ED Ni film on Si substrate (c) and (d)

The load-displacement nanoindentation curves obtained with 8 mN load in single crystal Si and polycrystalline Ni and Cu films on silicon substrates is shown in Fig.2.

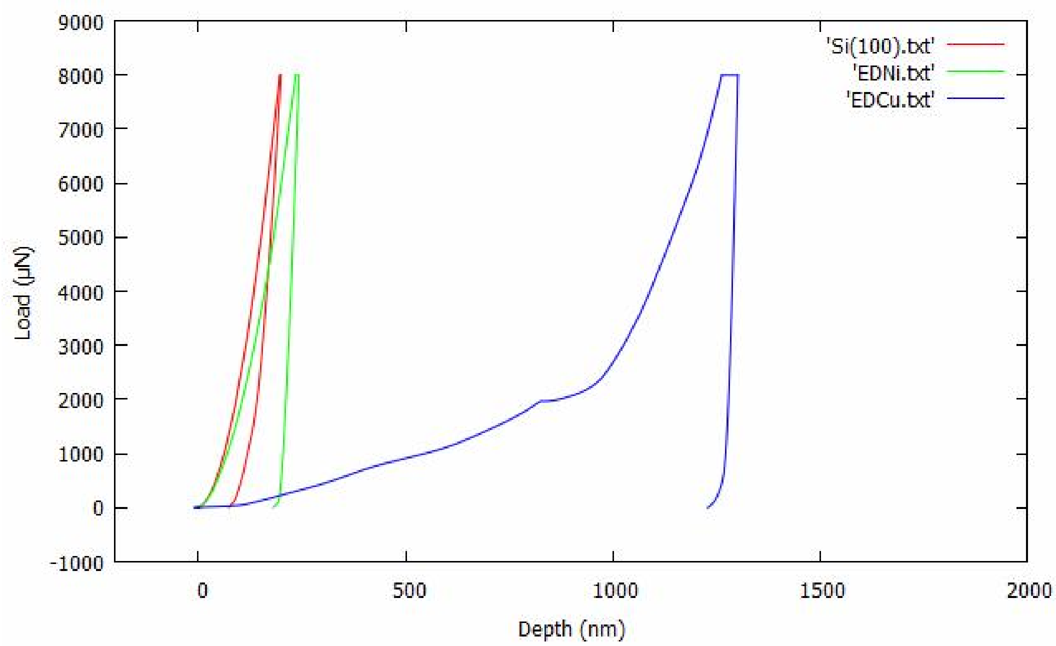


Figure 2 - Load-depth curves for 8 mN indent in Si(100) substrate and ED Ni and ED Cu films on Si substrate

The unloading curve for the Si(100) substrate shows high amounts of elastic recovery indicating mainly elastic deformation. For the nickel film and especially for the copper film, there is very little of elastic displacement in the unloading curves, indicating mainly plastic deformation. An evident displacement shift in the loading curve („pop-in phenomenon“) that was observed for the ED Cu film is interpreted to be a consequence of the nucleation of dislocations.

Table 1 - Mechanical properties of Si(100) and Si(111)-oriented wafers and ED Ni film as the substrates obtained from nanohardness measurements

Mechanical properties →	Nanohardness /GPa	E_R /GPa	h_c/nm
Substrates ↓			
Si(100)	11.2	146.9	154.3
Si(111)	11.9	152.7	151.0
ED Ni	5.8	164.9	223.6

Experimental results of nanoindentation on the substrates are presented in Table 1.

4.2. Absolute hardness of the substrates

Microhardness values are always below their nanoindentation hardness values, even the two kinds of hardness exibit similar load effect, owing to the indentation size effect (ISE)[16-18].

Microhardness testing was performed both on uncoated substrates and on different composite systems. Model of Li and Bradt named Proportional Specimen Resistance (PSR) model [19] was chosen for analyzing the variation of substrate microhardness with the load:

$$P = a_1 d + \left(P_C / d_0^2\right) \cdot d^2 \qquad (6)$$

Value of P_C is the critical applied load above which microhardness becomes load independent and d_0 is the corresponding diagonal length of the indent. A plot of *P/d* against *d* gives a straight line, the slope of which gives the value for the calculation of load independent microhardness.

The average values of the indent diagonal *d* (in m), were calculated from several independent measurements on every specimen for different applied loads *P* (in N). The absolute substrate hardness and composite hardness values, *H* (in GPa), were calculated using the formula:

$$H_C = 0.01854 \cdot P \cdot d^{-2} \qquad (7)$$

where 0.01854 is a constant, geometrical factor for the Vickers indenter.

On Fig.3., *P/d* values are plotted against *d* for the tested substrates: single-crystal Si and 100-µm thick Ni film electrodeposited with 50 mA/cm^2 current density.

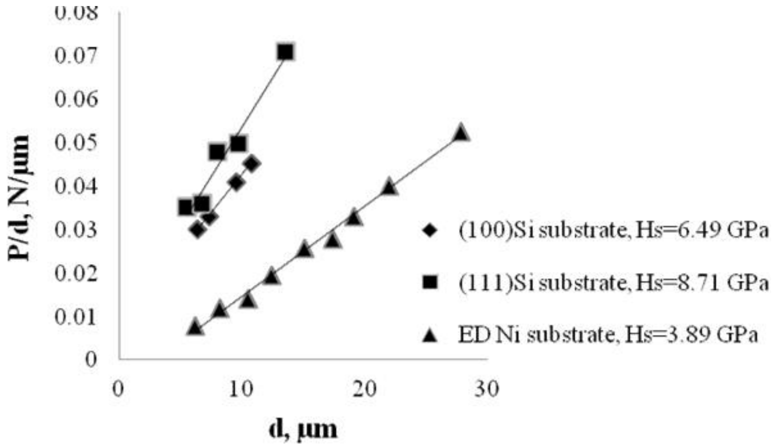


Figure 3 - PSR plot of applied load (in N) through indent diagonal (in µm), P/d, versus indent diagonal, d, for different hard substrates

Absolute hardness of the substrates H_S, is 6.49 GPa and 8.71GPa for (100) and (111)-oriented Si single crystal substrates respectively, and 3.89 GPa for thick ED Ni film (100 µm, 50 mA/cm^2) as the substrate.

4.3. Composite and film hardness of monolayer thin film composite systems

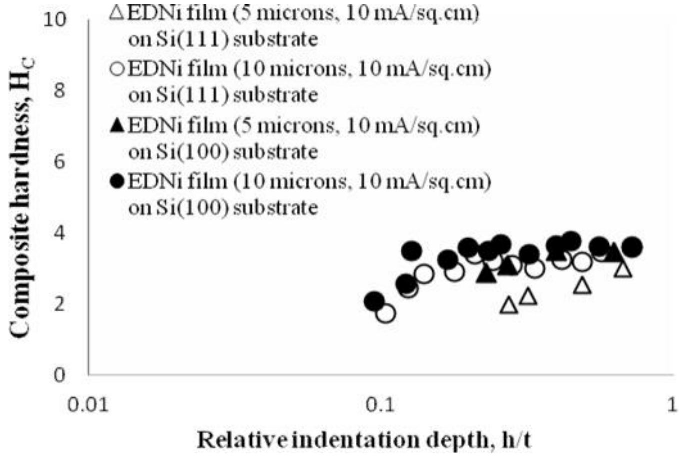
Two different composite systems were investigated: electrodeposited Ni films on Si(100) and Si(111)-oriented substrates and electrodeposited Cu films on thick harder electrodeposited Ni films (100 µm, 50 mA/cm^2) as the substrates.

Variation of the composite H_C, and film hardness H_F, of electrodeposited Ni films with different thickness (5µm and 10µm) on single-crystalline Si substrates, with relative indentation depth *h/t*, where *h* is indentation depth and *t* is total film thickness, is shown on Fig. 4.

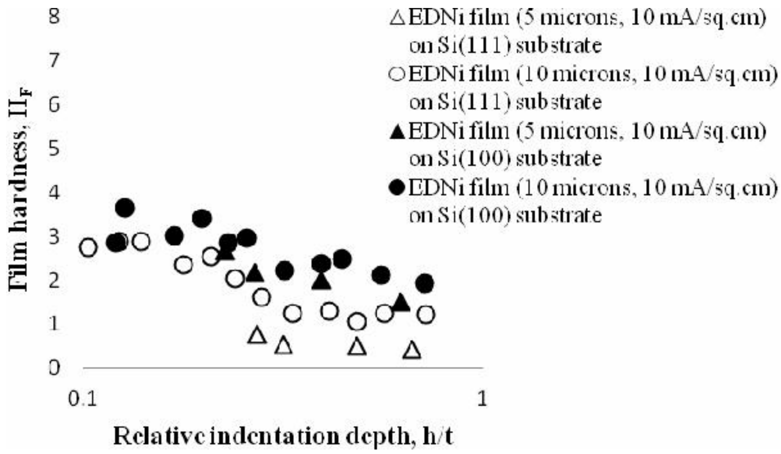
For shallow indentation depths (h/t ≤ 0.1), the response of the system is mostly response of the film. For indentation depths between 0.1 and 1, hardness response belongs to the whole composite system.

The system of ED Ni film on Si(100) substrate (which is is softer than Si(111) substrate) has slightly higher values of composite and film hardness than the system of ED Ni film on Si(111) substrate. Hard but brittle monocrystal Si substrates overwhelmed cracks formation and for these systems film hardness has descending character [20].

Change of the composite and film hardness, *Hc* and H_F, with relative indentation depth, *h/t*, for the system of soft ED Cu film on hard 100 µm-thick ED Ni film as the substrate, is shown on Fig.5. Projected thickness of the ED Cu films was 10 µm, and the current density values were maintained at 10 mA/cm^2 and 50 mA/cm^2. Increase in current density values has led to grain size refinement and hardness increasing. In distinction from the system of ED Ni film on single-crystal Si substrate, both composite *Hc* and film hardness H_F have ascending characters, because the deformation hardening of the polycrystalline fine-grained ED Ni substrate occured.



a)



b)

Figure 4 - Variation in composite hardness H_C *(a) and film hardness* H_F *(b) with normalised depth* h/t*, for electrodeposited Ni film on Si substrates*

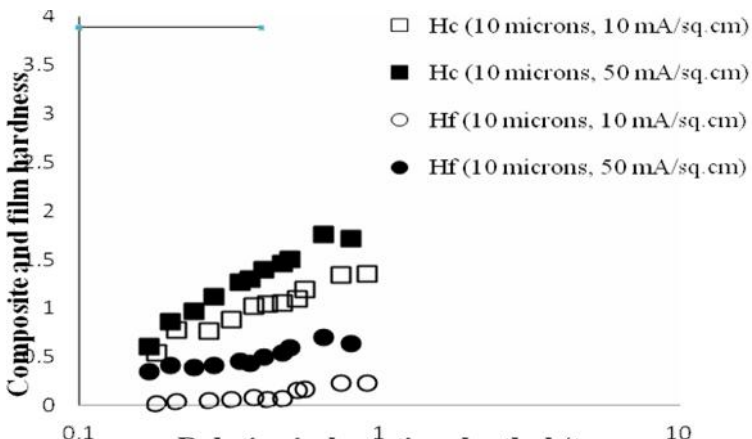


Figure 5 - Variation in composite hardness Hc and film hardness H_F, with relative indentation depth, h/t, for 10-µm electrodeposited Cu films on 100 µm-thick ED Ni films as the substrates.

4.4. Composite and film hardness of multilayer thin film composite systems

Introducing film with highly-densified parallel interfaces by depositing layers at a very narrow spacing into film-substrate system, gives rise to high strength and hardness of the composites.

Microhardness testing was performed on two different composite systems. Multilayered Ni/Cu films, with total thickness of 5 µm, were electro-deposited with 10 mA/cm^2 current density on Si(111)-wafers and 100 µm-thick ED Ni films as the substrates. Ni layer thickness was projected at 100 nm and layer thickness ratios were chosen to be Ni:Cu=1:1 and 1:4,

Change of the composite hardness H_C and film hardness H_F with relative indentation depth *h/t*, for Ni/Cu film electrodeposited on Si(111) substrate is shown on the Fig.6. This system has better hardness response in comparison to monolayer Ni film on the same Si(111) substrate. Composite and film hardness values have ascending character because of higher hardness of multilayer Ni/Cu film and increasing the layer thickness ratio Ni:Cu of the Ni/Cu film from 1:1 to 1:4, leads to increase of the composite and film hardness.

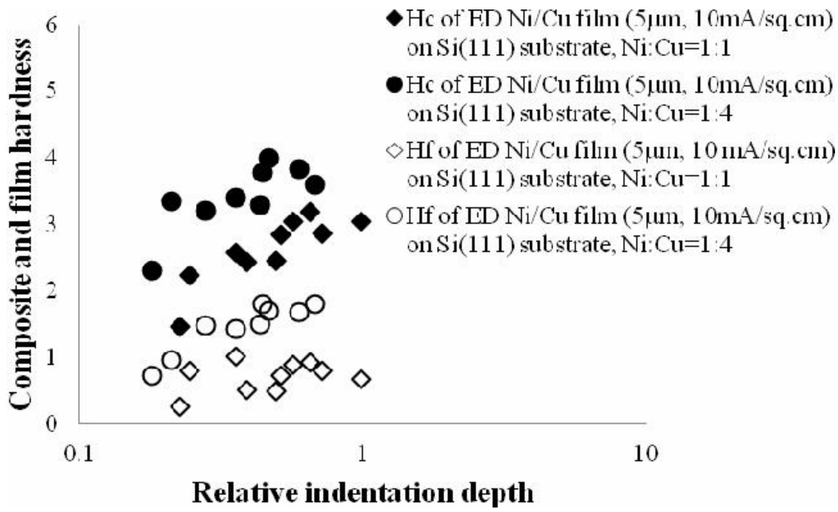


Figure 6 - Change in composite H_C and film hardness H_F, with relative indentation depth h/t, for ED Ni/Cu films on Si(111) substrate. Total thickness of the film is 5µm, Ni layer thickness is 100 nm and the layer thickness ratios Ni:Cu are 1:1 and 1:4.

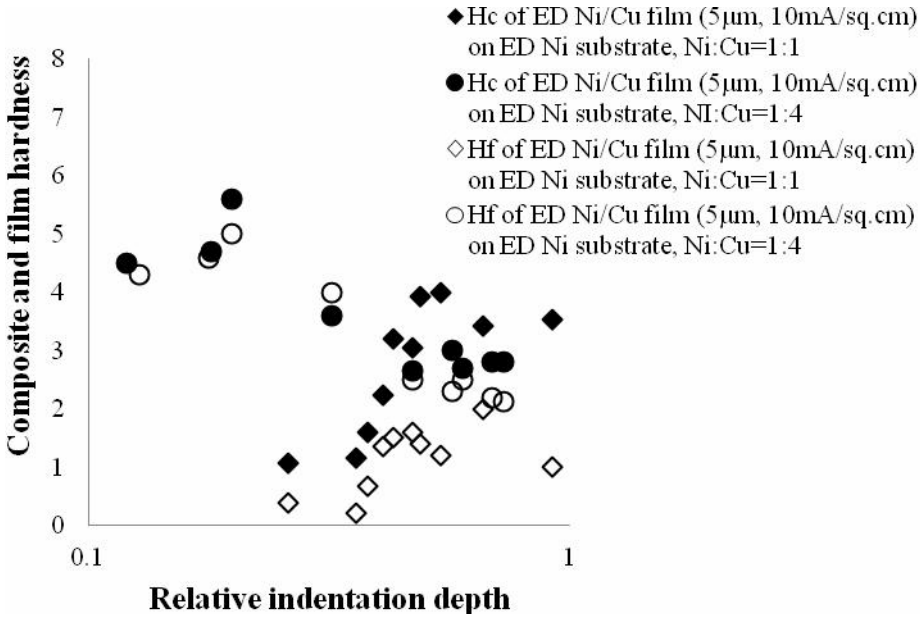


Figure 7 - Change in composite H_C and film hardness H_F, with relative indentation depth h/t, for ED Ni/Cu films on thick ED Ni (100 µm, 50 mA/cm^2) substrate. Total thickness of the film is 5µm, Ni layer thickness is 100 nm and the layer thickness ratios Ni:Cu are 1:1 and 1:4.

Variations in composite H_C, and film hardness H_F, with relative indentation depth *h/t*, for the system of ED Ni/Cu film on thick ED Ni film (100 µm, 50 mA/cm^2) is shown on Fig.7. Hardness of the Ni/Cu film is sensitive to variations of the layer thickness ratio Ni:Cu. Change of the layer thickness ratio Ni:Cu from 1:1 to 1:4, with Ni layer thickness of 100 nm kept constant, has led to increase of the composite and film hardness. For the layer thickness ratio Ni:Cu=1:4, higher hardness value for the film than for the substrate is achieved, and then change of composite system hardness response occured.

The difference in behaviour of the composite systems and tendency of the composite hardness H_C with relative indentation depth *h/t*, can be expressed through composite Meyer's index *m* and $(t/d)^m$ parameter. When the composite hardness tends to that of the film (for the low load values), parameter $(t/d)^m$ is almost independent of the substrate type. With increasing load and relative indentation depth, influence of the substrate in composite system hardness response becomes evident, but also the change of the composite system type can be noticed. It is shown on Fig.8.

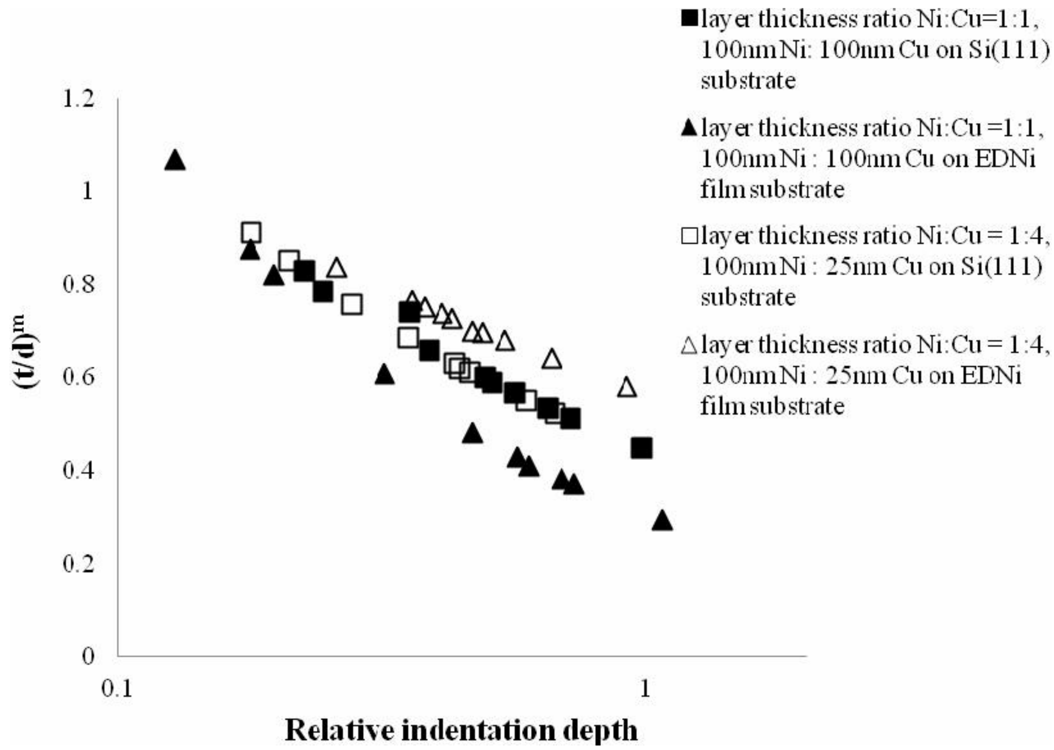


Figure 8 - Comparison of the parameter $(t/d)^m$ with relative indentation depth, h/t, for 5-µm thick ED Ni/Cu multilayer films on single crystal Si(111)-oriented wafer and 100-µm thick ED Ni film as the substrates.

5. CONCLUSION

Analysis of the composite and film hardness of different composite systems of the same type („soft film on hard substrate“) was performed. Metallic monolayer and multilayer films of Ni and Cu were electrodeposited on different substrates: brittle single-crystalline Si(100) and Si(111)-oriented wafers and thick film of polycrystalline electrodeposited Ni (100 µm, 50 mA/cm^2). Electrodeposition (ED) was carried out using DC galvanostate mode. Ni and Cu layers in multilayer films were alternately electrodeposited from two different electrolytes (dual bath technique, DBT), Ni from a sulphamate bath consisting of 300 g/l $Ni(NH_2SO_3)_2 \cdot 4H_2O$, 30 g/l $NiCl_2 \cdot 6H_2O$, 30 g/l H_3BO_3, 1 g/l saccharine, and Cu from a sulphate bath consisting of 240 g/l $CuSO_4 \cdot 5H_2O$, 60 g/l H_2SO_4. Deposition time was determined according with plating surface and projected thickness of deposits.

Nano- and microindentation measurements were performed on uncoated substrates and film-substrate composite systems in order to observe their hardness response according to their different structures. Composite hardness model of Chicot-Lesage (C-L) was found to be appropriate for the analysis of hardness response on different indentation loads for this type of composite systems.

Composite and film hardness of Ni monolayer films on Si substrate and Cu monolayer films on thick ED Ni film as the substrate were determined. Different microstructures and deformation response and consequently, different hardness values of the substrate and the film, as their relative difference

H_F/H_S, are most important parameters that influence the composite hardness value, H_C.

Microhardness testing was performed on composite systems of multilayer Ni/Cu films on Si(111)-wafers and thick ED Ni film as the substrates. Influence of parameters such as microstructure of the substrates and structure of multilayer films (layer thickness ratio) was investigated. Great number of parallel interfaces in thin films gives rise to high strenght of the composites. Ni/Cu multilayer films have Cu layers with much lower hardness than Ni layers, but it is possible to achieve high composite hardness value with appropriate change of layer thickness ratio Ni:Cu (from1:1 to 1:4) and even to change the composite system type.

Composite Meyer's index *m* and parameter $(t/d)^m$ characterize the way in which the composite hardness, H_C , varies with the relative indentation depth, h/t. For low indentation depths, parameter $(t/d)^m$ is almost independent on the substrate type, but with increasing load, parameter $(t/d)^m$ depends on the substrate type and on the composite system type („soft film on hard substrate“ or „hard film on soft substrate“).

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IZVOD

PONAŠANJE KOMPOZITNIH SISTEMA TIPA „MEK FILM NA TVRDOM SUPSTRATU“ NA TESTU UTISKIVANJA

Ovo istraživanje je izvršeno u cilju analize i upoređivanja odziva različitih kompozitnih sistema koji pripadaju istom tipu sistema nazvanom „mek film na tvrdom supstratu“, pri merenju tvrdoće. Formirani su kompozitni sistemi elektrodepozicijom jedno- i višeslojnih Ni i Cu tankih filmova na (100) i (111)-orijentisanim monokristalnim Si pločicama i 100-µm debelom elektrodeponovanom Ni filmu kao supstratima.

Ponašanje ovih kompozitnih struktura pri utiskivanju je okarakterisano merenjem nanotvrdoće po Berkoviču i mikrotvrdoće po Vikersu.

Izmerena vrednost mikrotvrdoće se naziva „kompozitnom tvrdoćom“ jer prilikom testiranja utiskivanjem dolazi i do plastične deformacije supstrata. Doprinos deformacije supstrata izmerenoj tvrdoći počinje na dubinama utiskivanja koje su reda 0.07-0.2 puta manje od debljine filma.

Istraživana je zavisnost vrednosti nanotvrdoće i mikrotvrdoće od procesnih parametera elektrodepozicije, mikrostrukture supstrata i filmova, ukupne debljine filma, debljine pojedinačnog sloja u filmu i odnosa debljina Ni/Cu pojedinačnih slojeva u filmu za različite kompozitne sisteme. Radi određivanja apsolutne tvrdoće filma u sistemu, odabran je i na eksperimentalne rezultate primenjen model kompozitne tvrdoće Šiko-Lezaž (C-L).

Ključne reči*: kompozitna tvrdoća, nanotvrdoća, vikersova mikrotvrdoća, model tvrdoće, Ni/Cu elektrodepozicija, Ni/Cu višeslojni filmovi.*

Naučni rad

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