

The influence of sublayer material on surface properties of electrodeposited nickel with periodical structures

E. FATARAITE¹, E. GRIŠKONIS^{1,2}, A. GUOBIENĖ¹, P. NARMONTAS¹, I. PROSYČEVAS¹,
S. TAMULEVIČIUS^{1*}

¹ Institute of Materials Science of Kaunas University of Technology, Savanoriu 271, LT-50131 Kaunas, Lithuania

² Department of General Chemistry, Kaunas University of Technology, Radvilenu 19, LT-50254 Kaunas, Lithuania

Surface topography, hardness and microstructure of nickel coatings electrodeposited on Si master with a periodical structure have been studied depending on vacuum deposited sublayer material (Ni, Cu or Ag). It is shown that the quality of replication of silicon master in a nickel shim electrodeposited simultaneously on different sublayers is dependent on the material used. All types of the analyzed coatings enabled transfer of lateral dimensions and showed good replication quality of the tested periodical structures (2 μm period), while the structure replicated using the coatings deposited on the Ni sublayer exhibited the worst roughness. The hardness of the electrodeposited layers was found to be dependent on the sublayer material used as well as on the side of deposit. Despite the fact that the backside of nickel shim had the same hardness for all the sublayers used, it was found that the hardness of the working surface with periodical structure (the side that has been in contact with the vacuum deposited layer) is dependent on the sublayer material: the Ni and Cu sublayers increased the hardness of Ni coating, while for the nickel shim deposited on Ag sublayer the hardness was reduced.

Keywords: *electrodeposition, replication, periodical structure, hardness, nickel shim*

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1. Introduction

Microlayered composite materials, consisting of two different metal layers have gained general recognition as a result of their unusual and sometimes outstanding combination of properties [1]. Deposition of multilayer is carried out mainly by vacuum deposition. On the other hand, nowadays electrolytic deposition has become more and more popular [2]. Different types of techniques can be used for electrodeposition. Typical technologies include electrodeposition using dual baths or single bath technique. Different authors [2–6] present both advantages and disadvantages of this technique. The main disadvantage of the multilayered structures formation using single bath technique is the long duration of electrodeposition (as time consuming processes usually are not practically applicable). Another mentioned disadvantage is the difficulty in achieving the appropriate structure, which must replicate the surface of the master. This is due to

potential contaminants during the transfer from one bath to another. Contrary to the dual bath technique, deposition from a single bath containing electrolytes of different compositions is far more promising [1–6]. On the other hand, this method presents several advantages as well: the electrodeposition process has high deposition rate, low cost and the experimental system used is much simpler.

The main task during electrodeposition of a coating is control of the process to produce the required micro- or nanostructure. The electrodeposition conditions determine the coating growth mechanism, microstructure of the coating and the mode of internal stresses. It is clear that the nature of the substrate also influences the growth of the coating, surface regularity and roughness, morphology, mechanical properties etc. [6, 7]

Production of electrodeposited periodical structures for multifunctional purposes appears to be an even more complicated task. In addition to the aforementioned requirements for the coatings, the electrodeposited surface must fully replicate the surface of the substrate (master). The pitting, delami-

*E-mail: Sigitas.Tamulevicius@ktu.lt

nating, air bubbles, and variations in thickness and other defects must be eliminated. Furthermore, the replicated structure must exhibit specific mechanical properties. Dependent on the field of applications, it must be elastic, hard (but not fragile) and wear resistant [8–11]. The most widely used approach in the replication technique is to electroform the original to produce a nickel foil (shim) or plate (stamper). This technique is routinely used for production of holograms and diffractive foils, as well as in the production of compact discs [12]. In this technique, the first Ni shim fabricated from an original recording can be recombined to form a large shim with multiple copies of the element. Typical flexible shims used for hot embossing have a thickness of about 100 μm , whereas the stampers used for injection moulding elements are typically of 0.5 to 1 mm. The optical planarity or shape is generally lost in this process [13].

In this way, the use of single layer structures does not always make it possible to achieve the required goals. Production of multilayered structures combining two methods: vacuum deposition for production of sublayer and single bath electrodeposition can be considered as an effective solution to this problem [14]. In this approach, the vacuum deposited sublayer ensures replication quality and also acts as a release layer during the removal of the structure from the master after electrodeposition. In addition, the electrodeposition provides the required and uniform thickness of the coating on the entire surface.

The present investigation aims at defining relations between the type of sublayer used in the electrodeposition of a nickel shim on crystalline silicon and physical properties as well as planarity of the replicated periodical structure. Currently, crystalline silicon is one of the key materials used in the micromachining and microtechnologies and such kind of knowledge could contribute to new types of applications. E.g. the integration of ferromagnetic films with semiconductors is essential for realization of a number of prospective devices based on spin dependent transport. In this case the semiconducting substrates are used to integrate a convenient method for fabricating thin magnetic films with silicon technology [15].

Table 1. The composition of nickel sulphamate bath solution.

Material	Concentration
Nickel sulphamate ($\text{Ni}(\text{NH}_2\text{SO}_3) \cdot 4\text{H}_2\text{O}$)	390–510 g/l
Nickel chloride (NiCl_2)	5–30 g/l
Boric acid (H_3BO_3)	35–40 g/l (saturated electrolyte)
Sodium dodecylsulphate $\text{CH}_3(\text{CH}_2)_{11}\text{OSO}_3\text{Na}$ (PAM)	0.05–0.25 g/l
Brightener (organic compound)	1.5–2.3 ml/l

2. Experimental

Formation of a Ni shim with periodical micro- or nanostructures is a multistage process. In our experiment it included production of a master in crystalline silicon (University Wafer, USA), vacuum evaporation of sublayer material and finally electrodeposition of Ni layer. This replica can be used in the following technological steps that include recombining of the structure on high areas or alternatively it can be employed as a stamper in transferring micro or nanostructures to a multilayer polymer system [12].

Cu, Ni or Ag coatings, of 100 nm thickness (sublayer materials) were thermally evaporated in vacuum on a Si master surface with a regular micro pattern (trenches with a 2 μm period and a 0.5 μm depth) that was produced by micro-contact lithography combined with reactive ion etching. During the vacuum evaporation, residual gas pressure in the vacuum chamber was 2×10^{-4} Pa and the substrate temperature was 20 $^\circ\text{C}$.

Typical technology for electrodeposition of nickel shim was used. The galvanostatic electrodeposition of nickel was carried out from mechanically agitated nickel sulphamate bath at $T = 50 \pm 1$ $^\circ\text{C}$. The composition of this bath is presented in Table 1. Boric acid was used as a buffering agent to stabilize pH value of the electrolyte in the range from 3.8 to 4.2 (optimal value 4.0). Sodium dodecyl sulphate was used as an antipitting agent. The titanium basket filled with Nickel S-Rounds (by INCO) and coated with a polypropylene fabric was used as an anode. The current efficiency of the main cathodic

process – electrodeposition of nickel coating – was higher than 96 %. The cathode current efficiency fell up to 4 % due to hydrogen evolution. The current efficiency of the anodic process – solubility of Nickel S-Rounds – was equal to 100 %.

The electrodeposition was performed on $20 \times 20 \text{ mm}^2$ area of a silicon master with vacuum deposited sublayer (Ag, Cu or Ni), protecting the rest of the surface. To eliminate the edge effect, the edges of the master (fixed on a glass plate) were additionally coated with a layer of silver paint (Agar Scientific). The cathode current density was 10 mA/cm^2 during the first 15 min of electrolysis and later smoothly increased up to 35 mA/cm^2 for the remaining time of electrolysis. The final thickness of the electrodeposited layer was $50 \text{ }\mu\text{m}$ and the average deposition rate was approximately $37 \text{ }\mu\text{m/h}$. All the silicon samples with different sublayers were electroplated simultaneously under the same conditions.

After the electrodeposition of Ni coating, the double-layer compositions (vacuum deposited sublayer and electrodeposited nickel) were detached from the Si master and the hardness and phase composition as well as replication quality of the microstructures were evaluated.

Atomic force microscopy (AFM) and scanning electron microscopy analysis were applied for the detailed inspection of both master and replicated periodical structures. In the AFM analysis, the microscope NANOTOP-206 (Minsk, Byelorussia) working in a contact mode was used. The area of the analysis was $12 \times 14 \text{ }\mu\text{m}$. SEM images were recorded using an emission scanning electron microscope (SEM) Hitachi S-4800 (Japan).

The hardness of the formed coatings (as well as pure metals – for comparative analysis) was measured according to Vickers method that is based on the evaluation of the imprint size of a diamond pyramid on the investigated surface [16]. For the Vickers hardness (HV) measurements, a hardness meter equipped with an optical microscope P-3 (Russia) was used. The normal force of 0.49 N (load 0.050 kg) was applied to the both sides of the nickel shim. The indentation time of the diamond pyramid was 5–10 s. The hardness measurement from the vacuum evaporated metal sublayer side was made

on a flat surface, in the vicinity of periodical structures. As a result of each HV test, the average of 15 measurements was calculated. The confidence interval of HV mean with 5 % probability of statistical error was calculated also.

The qualitative surface phase analysis of the nickel shim (on both sides – of the vacuum evaporated sublayer and electrodeposited nickel) was carried out by means of X-ray diffraction (XRD), performed with a diffractometer DRON-3 for the 2θ ranging from 20° to 100° and with Ni- filtered $\text{CuK}\alpha$ ($\lambda = 1.5418 \text{ }\text{\AA}$) radiation. The voltage and current of the diffractometer X-ray tubes were 30 kV and 30 mA, respectively. The NIST SRM660a (lanthanum hexaboride LaB6) was used as a line profile standard to determine instrumental broadening. The parameters such as peak position, full width at half maximum (FWHM) and intensity were extracted from all XRD diagrams using WinFit V1.2 (by S. Krumm) software package. The average size of the crystallites was calculated from the XRD spectra in a conventional way according to the Scherrer formula.

3. Results

Fig. 1 presents the AFM images of periodical structures with a period of $2 \text{ }\mu\text{m}$ in a crystalline silicon master (Fig. 1a) and the structures replicated in Ni shim (Fig. 1b, 1c, 1d). One can see that for all replicated structures, independently of the sublayer material used, the surface relief was transferred without any significant lateral distortion. On the other hand, vertical parameters (depth of the trenches as well as roughness of the surface) of the replicated structures are highly dependent on the sublayer used. Despite the fact that the structures formed on Ni and Ag sublayer exhibit a similar shape like the ones of the master surface, the electrodeposited shims are rough and the height of the replicated structures varies with the sublayer material used. For the structures electrodeposited on Cu sublayer, the rounded profiles of the ridges dominate.

Three surface roughness parameters R_a (average roughness), R_q (root mean square roughness) and R_{sk} (skewness) were calculated to describe quanti-

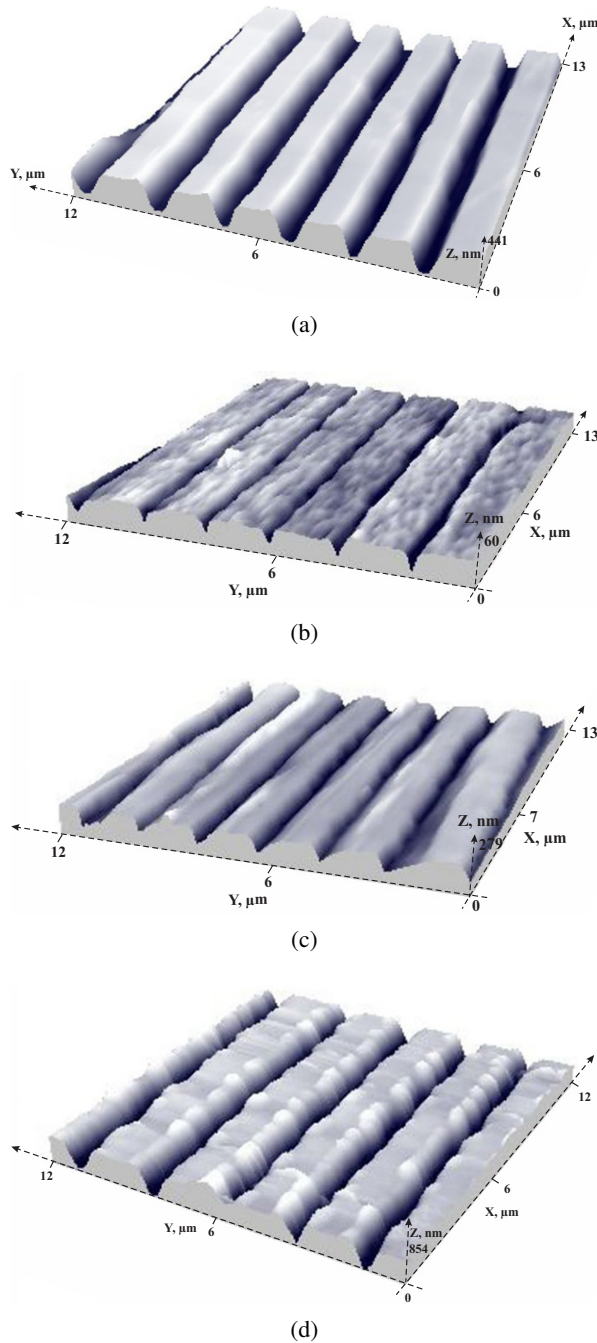


Fig. 1. AFM images of the periodical structures in crystalline Si (a) and nickel shims replicated in various vacuum deposited sublayer materials: (b) Ag, (c) Cu, (d) Ni.

tatively the morphology of the master and the replicated nickel shim surface [17].

The values of these parameters for the top part (ridged) of the master and replicated structures in

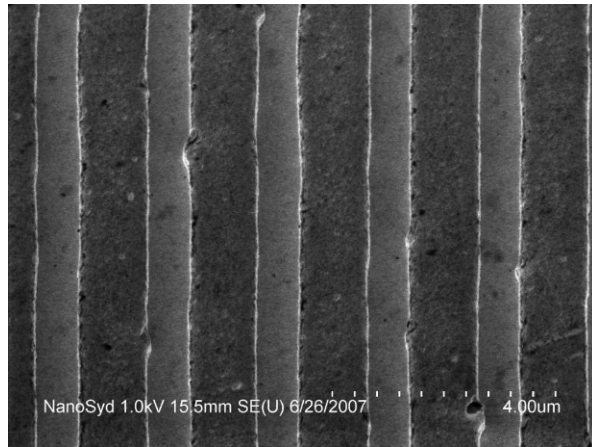
Table 2. Surface roughness parameters of the top part (ridged) of master and structures replicated in various vacuum deposited sublayer materials.

Roughness parameter	Si master	Nickel shim electrodeposited on different sublayers		
		Ag	Cu	Ni
R_a , nm	0.8	0.86	0.76	19.39
R_q , nm	1.06	1.1	0.99	27.84
R_{sk} , nm	-0.64	0.01	0.11	1.12
d_{av} , μm	2.01	1.96	1.90	2.43

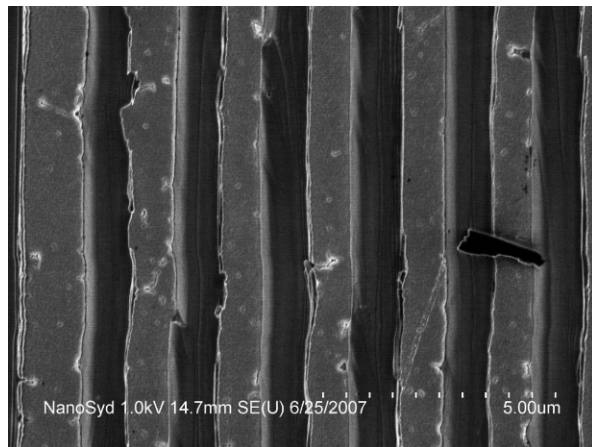
dependence of the used sublayer material are presented in Table 2. The values of the average period defined at half height of the structure (d_{av} , squares of data deviations $0.004 \mu\text{m}$) in silicon master and electrodeposited nickel shim are shown as well.

Fig. 2 presents SEM micrographs of the replicated periodical structures for various vacuum deposited sublayer materials. It is evident that the top view of the periodical structures coincides well with those obtained from AFM images in the case of Ni and Ag sublayers. The SEM micrographs also indicate that the Ag sublayer enables the production of electrodeposited periodical structures with the geometrical dimensions (the width of the bottom and top parts) that are close to those of Si master. On the other hand, the SEM image illustrates quite different shapes of the structures with a Cu sublayer, indicating a clear tetragonal shape of the ridges.

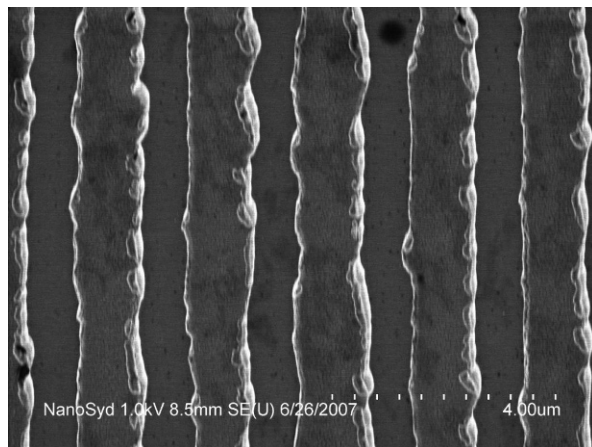
The results of hardness measurements on both sides of the nickel shim are presented in Fig. 3. One can see that the hardness of nickel deposited on Cu and Ni sublayers is higher for the vacuum coated side of the shim as compared to the electrodeposited one (backside). In the case of Cu, this difference reaches almost 30 % and for the structures with Ni sublayer this effect is negligible (within the measurement error). On the other hand, the opposite effect was observed for the nickel shim with an Ag sublayer. The electrodeposited surface (backside) showed more than 60 % higher hardness value than that for the vacuum coated side. For comparative analysis, the measured HV values of high-purity Ag (Cp-999, Russia, Ag – 99.90 %), Cu (M1, Russia, Cu – 99.90 %) and Ni (H111, Russia, Ni – 99.70 %) are presented in Table 3 as well.



(a)



(b)



(c)

Fig. 2. SEM images of the periodical structures in nickel shims replicated in various vacuum deposited sublayer materials: (a) Ag, (b) Cu, (c) Ni.

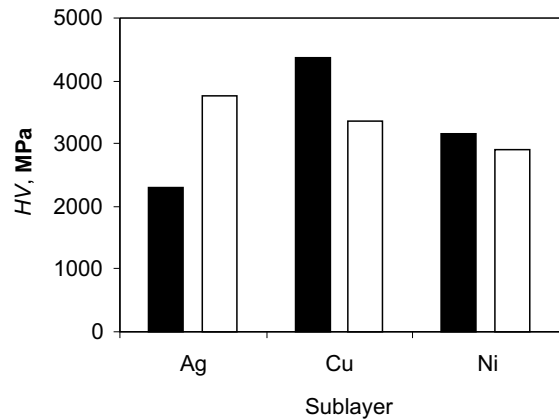


Fig. 3. Hardness of the nickel shim (50 μm thickness) on various vacuum deposited sublayer materials (Ag, Cu, Ni – 100 nm thickness) measured on different sides: ■ – vacuum deposited sublayer side; □ – electrodeposited side (backside).

Table 3. Hardness of high-purity metals.

Metal	HV, MPa
Ag	505.97 ± 27.30
Cu	928.27 ± 33.95
Ni	1566.07 ± 60.16

X-ray diffraction (XRD) patterns of a nickel shim (measured from the vacuum deposited sublayer side) for various sublayer materials are presented in Fig. 4. The diffraction pattern of the high-purity nickel (HIP1, Russia, Ni – 99.70 %) for comparative analysis is shown as well (curve 4). All the patterns show peaks that are typical of polycrystalline Ni. The (111), (200), (311) peaks typical of a face-centred cubic Ni structure with intensities similar to the random grain orientation were recorded. No structural differences between the coatings electrodeposited on a Cu and Ni sublayer were observed. Only for the nickel shim electrodeposited on an Ag sublayer, the new relatively weak peaks appeared at the diffraction angles of 38 and 85 degrees. The summary of the experimental XRD data (for both sides of shim) is presented in Table 4. One can see that, apart from the structures with Ag sublayer, Ni lattice parameters are constant and independent of the

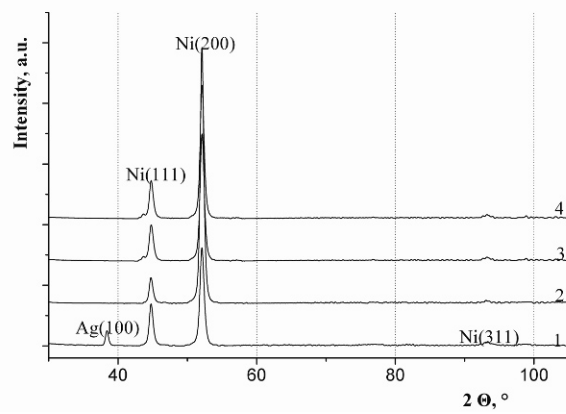


Fig. 4. XRD patterns of electrodeposited nickel shim (recorded from the vacuum deposited sublayer side) for various vacuum deposited sublayer materials: 1 – Ag; 2 – Cu; 3 – Ni; and for comparison 4 – high-purity Ni.

Table 4. Lattice parameters of the nickel shims electrodeposited on various sublayer materials.

Sublayer materials	Side ^a	Lattice parameters		
		d_{311} , Å	a_0 , Å	Δa_0 , %
Ag	V	1.069	3.546	+0.62
	E	1.061	3.520	
Cu	V	1.061	3.520	
	E	1.062	3.521	
Ni	V	1.060	3.520	
	E	1.062	3.521	
high-purity Ni		1.062	3.524	

^aE – measurements performed from the electrodeposited side (backside); V – measurements performed from the vacuum deposited sublayer side.

sublayer material used as well as of the side of shim (electrodeposited [backside] or vacuum coated).

The measurements of the full width at half maximum (FWHM) of the peaks at the 52° (Ni (200)) indicate that FWHM equals to 0.572° (Ag sublayer), 0.609° (Cu sublayer), 0.637° (Ni sublayer) and 0.352° (high-purity Ni). Taking into account instrumental broadening and neglecting microstrains, according to the Scherrer formula, these FWHM correspond to the crystallite size of 15.5, 14.5, 13.9 and 25.1 for the Ag, Cu, Ni sublayer and high-purity Ni, respectively i.e. the electrodeposited nickel in all cases has crystallite size on the nanometric scale.

It should be noted that grain refinement of electroplated nickel into the nanometric range (<100 nm) results in unique, in many cases, improved properties (e.g. hardness) compared to conventional polycrystalline nickel [18] which are important in mechanical applications of Ni shim. It is worth mentioning that although all the layers were deposited simultaneously, at identical plating conditions and bath composition, the grain size is only to a small degree dependent on the sublayer used.

4. Discussion

It is known [1] that in electrodeposition of three-dimensional layers the cross sectional morphology displays a sequence of three different regions from the substrate outwards: narrow interfacial region, where nucleation and initial growth stages take place; transition zone, where the number, dimensions and shape of the deposit tend to change progressively towards the third zone, in which the structure of the deposit is no longer influenced by the substrate, but depends only on the electrodeposition conditions. In our experiment during electrodeposition of thick Ni shim, the formation of all these three zones occurred. The first zone (where the sublayer is expected to play the most important role) appears to be important for the surface microrelief transfer and working surface hardness, which is dependent on the surface composition as well as grain size of the electrodeposited shim. According to our results, the use of sublayer metals of different resistivities could influence the different electrodeposition rates of Ni at the initial moment of electrolysis (initial growth and interfacial region). Probably, electrodeposition of Ni on the Ag and Cu sublayers begins with the higher rate than that on the Ni sublayer, because resistivity of Ag and Cu (respectively $1.6 \cdot 10^{-8} \Omega \cdot m$ and $1.7 \cdot 10^{-8} \Omega \cdot m$) is about 4 times lower than the resistivity of Ni ($7 \cdot 10^{-8} \Omega \cdot m$) [19]. This can contribute to the increase in local current density, enhance the velocity of arriving particles and finally the growth of bigger crystallites of Ni electrodeposited on Ag and Cu sublayers. This assumption is in accordance with the experimental investigations of cathode polarization during electrodeposition of Ni on Cu and Si substrates where it was demon-

strated that the Cu substrate is more active than Si for electrodeposition (higher current densities were recorded for Cu substrate) leading to differences in the deposition rate. [20]. The influence of deposition current density on the crystallinity of Ni layer deposited on Cu was found experimentally in [21] as well. It should be noted that the crystallites of Ni electrocoatings on the Cu sublayer are smaller than the ones on the Ag sublayer. One can expect that this is related to the higher resistivity of copper compared to that of silver as well as the increased resistivity of the Cu sublayer due to possible Cu oxidation in air. On the other hand, it appears that surface alloying takes place during nucleation of nickel as it was shown in [3]. For the shim with the Ag sublayer, an increase in Ni lattice parameter by 0.62 % was found (Table 4). The obtained changes in Ni lattice parameters can be attributed to the formation of Ni–Ag solid solution in the sublayer and electrocoating interface zone. The lattice parameter for Ag is significantly higher than those of Ni and Cu which are as follows: $a_0(\text{Ag}) = 4.085 \text{ \AA}$; $a_0(\text{Ni}) = 3.524 \text{ \AA}$; $a_0(\text{Cu}) = 3.615 \text{ \AA}$ [22–24]. The appearance of the deformation results in the increase of the lattice parameter a_0 of the Ni electrocoating. In this way, by changing the sublayer material, one can control surface properties of the electrodeposited coating due to the changes in alloying conditions during electrodeposition. In the case of the Ag sublayer, the hardness measurements taken from the vacuum deposited sublayer side exhibit the lowest HV values of nickel shims in comparison with Cu and Ni sublayers. This is in a good correspondence with the measured hardness of high-purity metals: HV value of Ag is about two times lower than HV value of Cu and more than three times lower than HV value of Ni (see Table 3). Meanwhile, the hardness measurements taken from the backside of the electrodeposited layer show that the Ag and Cu sublayers are responsible for the increased hardness of the electrochemically formed nickel shims in comparison with the Ni sublayer. In addition, the increase in hardness values indicates not only structural changes but also an improvement in the wear properties and an increase in the resistance to plastic deformation [18]. On the other hand, one should keep in mind that an unlimited increase in

the hardness values could be the cause of cracking and final failure of the structure. The importance of the internal stresses must also be taken in account.

Considering the replication of microstructures one should keep in mind that the quality of final product in the used technique is defined by the interfacial region of the shim, where nucleation and initial growth stages take place, as well as by the detaching process of the shim from the crystalline silicon master. It is evident that the surface roughness parameters (R_a and R_q) are close to those of master matrix when Ag and Cu sublayers are used. The skewness values indicate that the surface produced by the Ag sublayer has almost symmetrical amplitude distribution function. On the other hand, one can assume that the vacuum deposited Ni sublayer acts as a primer between the Si matrix and Ni coating. In this case adhesion is higher than the cohesion energy, and after electrodeposition the formed structure cannot be fully removed from the surface. That explains why the high values of R_a and R_q of surface roughness parameters were found in the case of Ni sublayer. Although the depth of relief in the case of Ni sublayer is the highest compared to that of Ag and Cu, the detaching of the shim produces a very rough surface.

5. Conclusions

It is shown that the quality of replication of microstructured silicon master in a nickel shim electrodeposited simultaneously on different vacuum evaporated sublayers (Ag, Cu, Ni) is dependent on the material used. All types of the analyzed coatings were efficient in transferring of lateral dimensions (period of the tested structure was equal to $2 \mu\text{m}$) and showed good replication quality, although the roughness of the structure was the highest while replicating using the coatings deposited on the Ni sublayer.

Ni lattice parameters of the electroplated nickel are found to be constant and independent of the sublayer material as well as of the side of the shim (electrodeposited (backside or vacuum coated) except the silver sublayer. For the shim with an Ag sublayer, an increase in Ni lattice parameter by 0.62 % was observed. The obtained changes in the Ni lattice

parameters can be attributed to the formation of Ni–Ag alloy in the sublayer and electrocoating interface zone.

Although all the nickel layers were deposited simultaneously, the grain size was to a small degree dependent on the sublayer used. The electrodeposited nickel had the crystallite size on the nanometric scale and the crystallite size was found to be 15.5 nm, 14.5 nm, 13.9 nm for the Ag, Cu, Ni sublayer, respectively.

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