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## NANOMATERIALS FOR OPTICAL MICROELECTROMECHANICAL SYSTEM TECHNOLOGY

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The research is focused on the nanostructured materials processing and their application in microelectromechanical systems (MEMS), in general, and optical MEMS, in particular. In the framework of LIGA-like (Lithografie, Galvanoformung, Abforming) technology, anode and cathode cost effective technologies are developed. One of approaches to solve the fundamental problem of friction and wear of MEMS elements is application of the composite materials, in particular, cathode codeposited metal and alloy with inert hard particles by electroless or electrochemical processes. Wear resistance of elements increased in 2-2.5 times, microhardness increased in 2 times, coefficient of friction and corrosion current were reduced by factor 1.5 and 1.6 respectively. Application of composite materials for moveable microelement with high reliability is the only way for real MEMS development. Anodic processes of aluminum and silicon anodizing result in nanopore structure. Anisotropy of the pores determines the unique properties of the films during chemical selective etching and provides aspect ratio up to 50 for MEMS microstructures.

**Keywords:** microrelief, microelectromechanical systems (MEMS), codeposition

### Introduction

MEMS provide sensation and treatment information, communication and interaction with the world. Micromechanical elements of MEMS are 3d structures with the possibilities to mechanical micromovement.

MEMS technologies are widely applied in display industry [1]. Bulk micromachining is used as well as surface. As for MEMS-based displays the modulation and contrast creation for the DMD and the GLV are redirecting light and for iMoD – absorbing light. For implementation of the functional principles for displays it is necessary to form precision micro- and nanoreliefs. Moreover, obtained moulds and reliefs can be both MEMS elements and forms for following deposition of metal. Finished metal element is received after removing the form. Mechanical properties of materials, surface interactions are dominant for final determination about reliability of MEMS-based Displays [1]. The aim of this investigation is working out the cost effective technique for microrelief fabrication for MEMS and display technology, developing new nanoheterogeneous materials of high reliability [2, 3].

## Experimental details

In this work three technologies of microrelief formation were worked out: polyimide, porous silicon, and porous alumina.

Polyimide films based on pyromellitic dianhydride and diaminodiphenyloxide were applied on substrate by spinning. Imidization process was carried out in the nitrogen medium at a temperature of 620 K. Resistive mask on the surface was received using standard methods of photolithography. Thus, plane pattern corresponding future quasi-3D microstructure was formed. Process of reactive ion beam etching was carried out on the modified wide-aperture ion source with cold hollow cathode. Final relief was obtained by etching of polyimide in oxygen and its mixture with argon at ion beam current density  $j=0,2-0,8 \text{ mA/cm}^2$  and voltage of ion extraction  $U=400-2000 \text{ V}$ .

Porous Si was received by anodic treatment of high-doped Si with p-type conductivity in alcoholic solution of hydrofluoric acid. Cr layer was used as primer. Common photolithography provided selectivity of Cr etching and consequently chemical etching of porous Si in solutions of KOH and HF to form microreliefs and elements.

Porous  $\text{Al}_2\text{O}_3$  was got by anodizing process of aluminum foil in solution of oxalic acid. Free film of porous alumina was prepared using saturated solution of  $\text{HgCl}_2$ . Priming underlayer of Mo, Ni, Cr, SiO were used for exception of photoresist penetration into pores. Application of free alumina films allows excluding priming masks. Final reliefs were obtained by chemical etching of porous  $\text{Al}_2\text{O}_3$  in solution of chromic anhydride and phosphoric acid.

Nanocomposite coatings containing ultra-fine particles were plated from sulfate, glycine, acetic, and Watts bathes. Soft magnetic (NiFe, CoFeP, CoP) and hard magnetic (CoNiP, CoW, CoP) alloys as well as conductive matrix of Cu and Ni were investigated. Concentration of ultra-fine particles was varied from 0 to  $10 \text{ g}\cdot\text{dm}^{-3}$  (dry substance). Diamond, alumina and aluminium monohydrate ultra-fine particles were used. The amount of codeposited particles was determined both by integral Coulombmetric analysis on express analyser AH-7529 (USSR) and by local Auger spectroscopy (PHI-660 Perkin Elmer Corp., USA). The Vickers microhardness of coatings was measured at a load of 0.5 N with MICROMET-II (Buehler-Met, CH). The structure of the deposits was examined by TEM (EM-125, USSR). The coefficient of friction and the wear were evaluated by a FRETTE II test machine (KU Leuven, BE). Wear volumes were estimated by RM600 laser profilometry (Rodenstok, D) after 100,000 fretting cycles.

## Results and discussion

Classical LIGA (Lithografie, Galvanoformung, Abforming) technology is the basis for MEMS and MEMS-based display elements production. LIGA uses synchrotron X-ray radiation. It makes industrial application of classical LIGA technology practically impossible. Low-cost LIGA-like technologies for precision microrelief were developed using common microelectronics materials and equipment.

Multi-step spinning and imidization of polyimide was used for required layer thickness. Various conditions of reactive ion beam etching were investigated. Etching of polyimide in mixture of  $\text{O}_2$  and Ar showed insufficient results. Optimal conditions are medium of  $\text{O}_2$ , ion beam current density of  $j=0,5 \text{ mA/cm}^2$  and voltage of ion extraction  $U=800 \text{ V}$ . Received microrelief structures have thickness up to  $50 \text{ }\mu\text{m}$  and aspect ratio up to 50 (Fig. 1a).

The process of production of high-ordered structure of porous anodic alumina is sensitive to procedures of anodization and pre-treatment processing of anode surface. Observance of optimal conditions leads to formation of periodical self-organized nanostructure, which is perpendicular to plane of substrate. SiO showed good results for porous  $\text{Al}_2\text{O}_3$  as priming underlayer which are sputtered by vacuum method. Film of silicon monoxide with thickness  $0,2 \text{ }\mu\text{m}$  has the best adhesion properties. Anisotropy of form provides anisotropy of selective chemical etching, i.e. high aspect ratio. Angle of side wall is near  $90^\circ$ . Obtained microrelief has aspect ratio up to 50 at thickness up to  $500 \text{ }\mu\text{m}$  (Fig. 1 c,d).

Classical type of substrate for MEMS technology is Si. Investigations of porous Si were carried out for integration of electronic and micromechanical elements. Formation of relief for MEMS

elements provided with good solvability of porous silicon in weak alkaline solution. Pore anisotropy is of a great important as well. Aspect ratio is up to 20 at thickness more than 100  $\mu\text{m}$  (Fig. 1b).

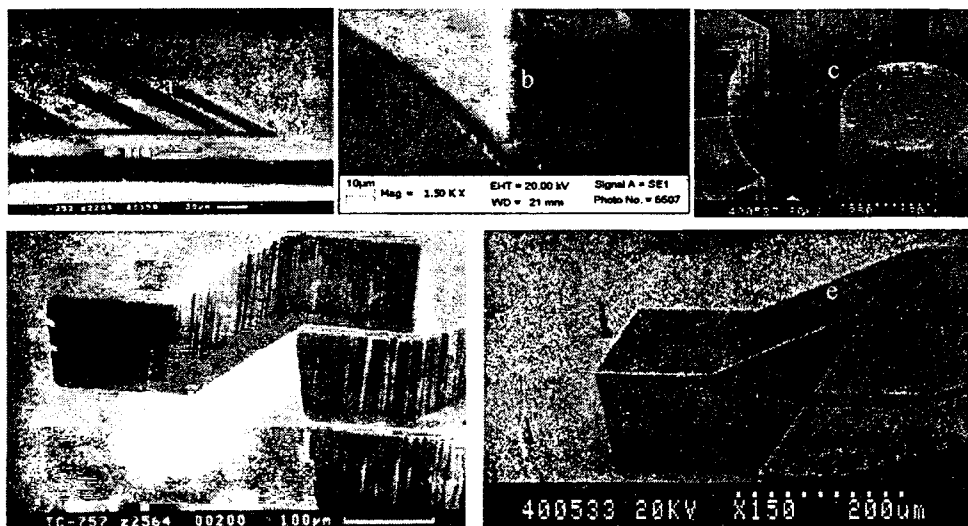


Fig. 1. Images of MEMS microreliefs and microelements: *a* – based on polyimide; *b* – based on porous Si; *c, d* – moulds based on porous alumina; *e* – metal element of winding

Subsequent deposition of metal into mould and its dissolution lets to form microsystem elements of required shape (Fig. 1, *e*). Nanocomposite materials were plated instead of homogeneous metals to provide high reliability [4,5].

In general, during the electrolytic codeposition, the suspended inert nanoparticles interact with the surface of the growing film due to hydrodynamic, molecular and electrostatic forces and incorporate into the film. Based on the experimental data, the qualitative codeposition model of the composite coatings with the ultra-fine particles is proposed. The specific characteristics the behavior of the ultrafine particles are considered in the model. The model worked out is based on the assumption that the codeposition of ultrafine particles proceeds through the following stages: (1) coagulation of ultrafine particles in the plating solution; (2) formation of quasistable aggregates and, therefore, a change in the system dispersion constitution; (3) transport of the aggregates to the cathode surface by convection, migration, and diffusion; (4) disintegration of the aggregates in the near-cathode surface; (5) weak adsorption of ultrafine particles and aggregate fragments onto the cathode surface; (6) strong adsorption of spersion fraction (embedding).

This complex process results in the formation of nanocomposite coatings. Auger profiles, local X-ray analysis and cross-section show that ultra-fine particles are effectively incorporated into the meal matrix (Fig. 2).

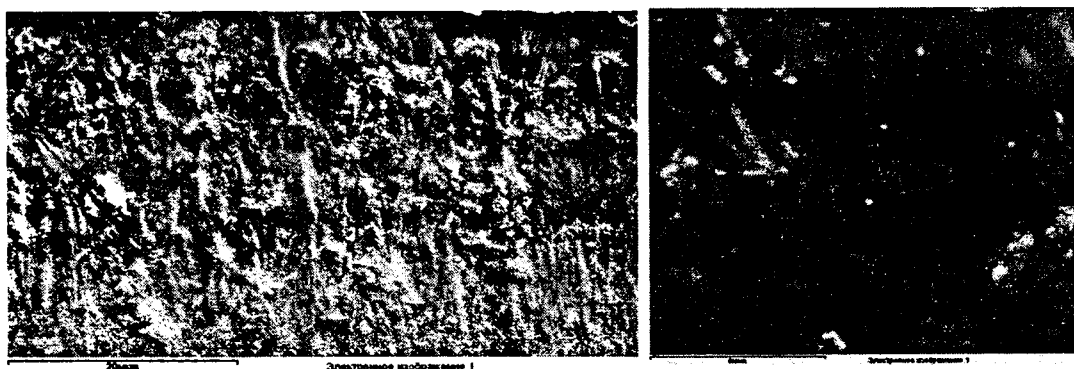


Fig.2. Cross-section SEM images of nanocomposite Ni-Al<sub>2</sub>O<sub>3</sub> coating

Structural investigation shows that pure Ni coatings contain twins, dislocation aggregates inside the grains, and a concentration of solitary dislocations and dislocation walls of 20 nm thick along the grain boundaries. The average grain size is about 500 nm. As for composite coatings the grain size reduces up to 30-100 nm, i.e. nanocrystalline Ni electrodeposites were formed.

Related to microreliefs and MEMS elements functionality, the most important properties are friction and wear resistance. For the composite systems, mechanical properties are determined by the phase composition of materials, i.e. matrix and particles ratio. Incorporation of the particles into metal matrixes increases microhardness of the films and MEMS elements (Fig. 3).

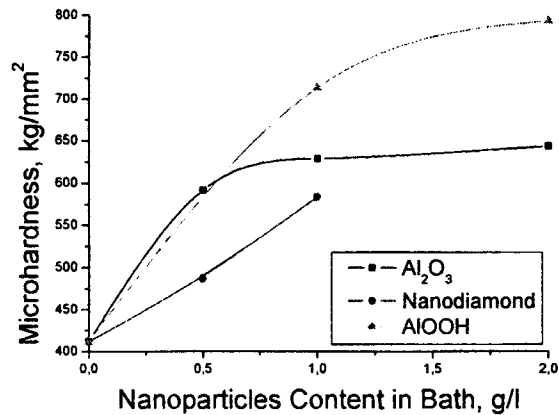


Fig. 3. Microhardness vs concentration of particles in bath

Composite nickel coatings containing ultra-fine particles show the lower coefficient of friction in comparison with pure one. The wear volume for pure Ni and composite coatings is shown for fretting tests operated for 100,000 cycles (Fig. 4). The amount of particles in the coatings affects the wear rate. The friction behaviour of multiphase materials has been described in the literature. The main problem with these approaches is that they are static ones and applied to ideal surfaces because during wear of a multiphase material the topography changes in practice continuously. Localised wear of the matrix takes place in the first phase. After that, the particles become more loaded. This dynamic process may lead to an increased wear resistance of composite materials. Of course, the fretting wear properties of composite coatings are also influenced by the size, shape and distribution of the reinforcing phase.

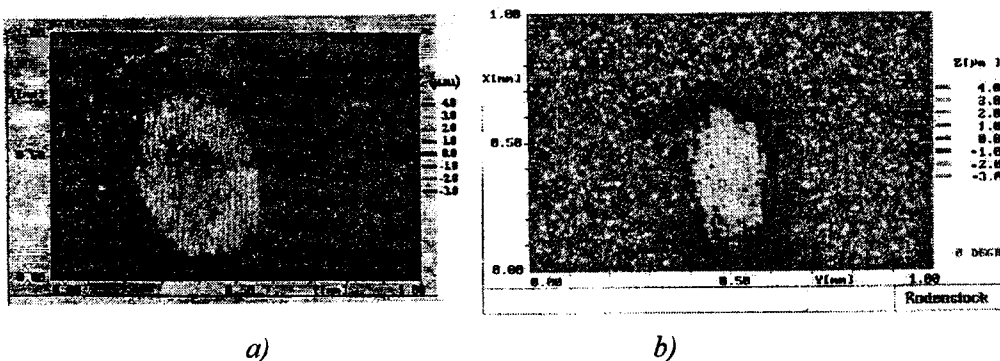
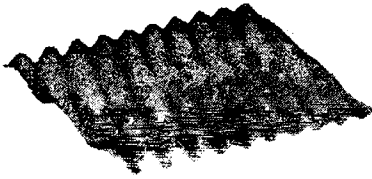


Fig. 4. Wear track of pure (a) and nanocomposite (b) Ni-nanodiamond coating after 100 000 fretting cycles

Electroforming is technology for mould and relief. Nanomodified electroforming is used in display and holographic industry for production of optical elements. As a rule the images are replicated by mechanical pressing micromoulds into polymeric layer. Hence advanced microhardness and wear resistance are required. To plate the moulds (reliefs, elements), the above-mentioned codeposition technology is used to overcome these problems. Holographic nanoreliefs before and after mechanical replication are shown in Fig. 5. Test results show the length of holographic imprint on poly-

meric tape of 5500 m for nanocomposite material and 1800 m for pure material (imprint quality is controlled by estimation of diffraction pattern).

12.2 x 12.2  $\mu\text{m}$   
 $\lambda = 832.9 \text{ nm}$   
 $R_a = 144.9$   $R_q = 173.3$



11.6 x 11.4  $\mu\text{m}$   
 $\lambda = 168.2 \text{ nm}$   
 $R_a = 13.1$   $R_q = 15.8$



Fig. 5. Nanoreliefs before (left) and after (right) replication.

### Conclusion

LIGA-like technologies for MEMS-based displays were carried out: polyimide, porous Si, porous  $\text{Al}_2\text{O}_3$  and nanocomposite plating. The developed processes and materials are cost effective, provide flexibility, wide range of thickness, acceptable aspect ratio and high reliability.

### Литература

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