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Investigation of Nanostructured Objects Using Cross-section Techniques

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Abstract - **The article represents possibilities for investigation of nanostructured objects parameters using cross-section preparation. Described are the most important process parameters and its possibilities. Stated are the investigation results of several objects based on nanostructured oxide at different preparation steps.**

Keywords – **nanostructured materials, cross-section preparation, microscope observation**

I. Introduction

In the last ten years have been published a great number of articles concerning nanotechnologies and their application [1]. Nanotechnologies can be used for realization of direct functional devices, as nanoelectrical components [2] or nanomanipulators [3], as well as constructive materials. As constructive material nanocomposite [4] one could point e.g. nanostructured anode oxide of aluminum [5].

In every case it is necessary to investigate the parameters of these materials. Investigation methods that are mostly used in nanomaterial analysis are electron microscopy [6] and atomic force microscopy [7].

There are also cases where micro parameters of nanomaterials should be examined. A technique of cross-section preparation, which is used in such cases, is described in the article below.

II. Conventional Method for Cross-section Preparation

The technology of cross-section preparation includes specimen preparation, reliable fixing in desired position and resin moulding in special mounting cups. A complete moulded sample is shown on Figure 1.

As a moulding material in most cases acrylic or epoxy resins are used. After the required curing time and extraction of the moulding the preparation process includes plane and fine grinding, and polishing. During the grinding process special grinding papers (SiC) are used with grit size between P180 and P4000. Usually at this step it is enough to use water as a cooling agent. There is a large variety of consumables, that can be used instead of conventional SiC papers [8].

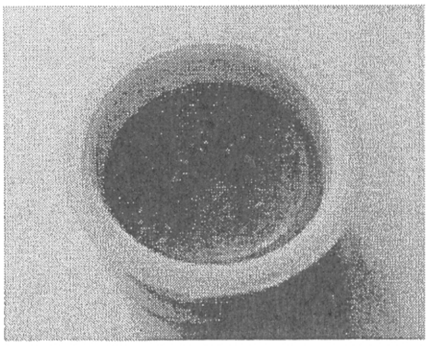

Figure 1. Specimen moulded in mounting cup

Most of them are more expensive, but have excellent improved properties as absolute planeness, lack of edge rounding and relief. The final step of the mechanical preparation is polishing. Normally it is done in the same way as grinding, but on a special polishing clothes and use of diamond pastes/suspensions (6μm to below 1μm). Different types of lubricants are available. Another important issue is the mechanical preparation process parameter as push force, rotates per minute and rotating direction. Finally the cross-section is observed on a light microscope at magnifications 50x-1000x. Figure 2 represents a cross-section for evaluation of intermetallic phases.

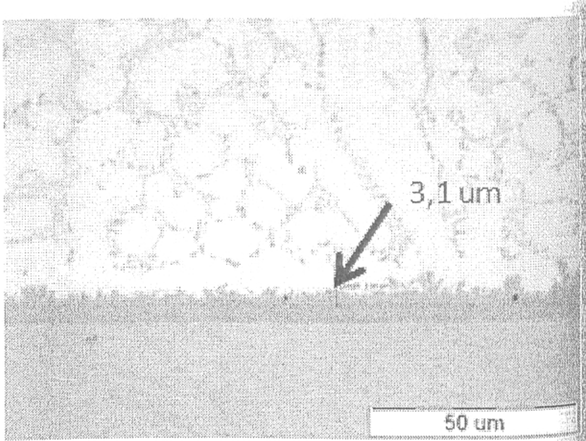

Figure 2. Evaluation of intermetallic phases

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III. Parameters of Nanostructured Materials

For nanostructured materials we can define electrophysical and physical-mechanical parameters. Electrophysical parameters are for example electrical conductivity, permeance and permittivity. Physical-mechanical parameters are hardness, density etc.

Sometimes it is necessary to perform comparatively fast analysis for nanocoating thickness, deformation etc.

It is interesting to know, what is the variation of thickness at different grinding/polishing steps, as well as the vertical distribution. Scope of interest can be also the data for the layers of nanostructured materials one can obtain using metallographic cross-section.

IV. Metalographic Cross-section of Nanostructured Materials

To evaluate the possibilities of cross-section process and define nanostructured material parameters we will focus on anode oxide of aluminum. Its formation is as a porous material with pores in range 50 – 200 nm. Figure 3 represents a typical structure of that oxide.

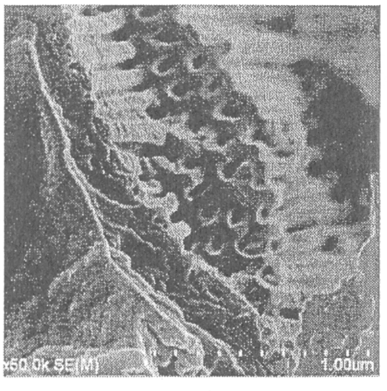

Figure 3. Structure of aluminum anode oxide

To examine the influence of preparation method different specimens have been moulded in a single cup. Figure 4 represents sample that have been treated with SiC grinding paper P320. Moulded are aluminum base, selective grown nanoporous oxide base, nanoporous oxide with grown copper layer, membrane and membrane with metal core.

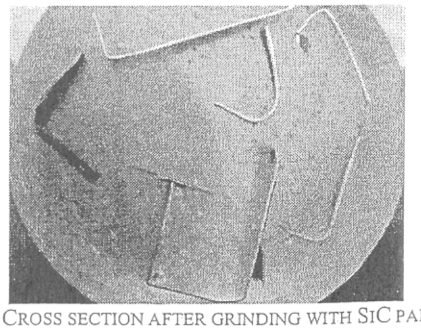

Figure 4. Cross section after grinding with SiC paper P320

On the picture taken in macro mode (2cm), traces caused by the grinding process are evident. They are uniformly distributed over the complete grinded area. Some additional grinding steps have been performed with papers up to P4000. Polishing has been performed using series of different diamond polishing suspensions in the range 6um – 1 um.

For the evaluation of process results it is also important to use appropriate observation method. On a microscope with reflected light two modes are mainly used – bright field (when the surface is illuminated perpendicular) and dark field (the surface is illuminated at angle). Dark field is the proper mode when rough surfaces are observed or to find traces over mirror surfaces.

V. Experimental Results

Figure 5 represents comparison of complex specimen after one and the same process step, but different observation method.

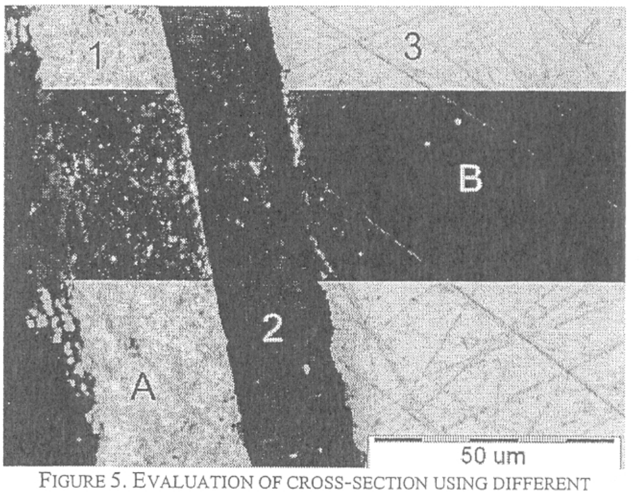

Figure 5. Evaluation of cross-section using different observation method.

Area A is observed in bright field mode and area B in dark field mode. Possition 1 is an aluminum, position 2 is nanoporous oxide with grown copper. Possition 3 is electrochemical layer of copper. The surface has been treated with diamond suspension 1 um and the image magnification is x500. Depending of the material hardness [9] there is a different level of smoothness and distinguishing ability. Different material outlines are easily distinguished.

Copper inclusions in oxide pores has different distinguishing in bright field and dark field modes. It is necessary to use color observation.

Figure 6 represents a comparison of surface at different process steps.

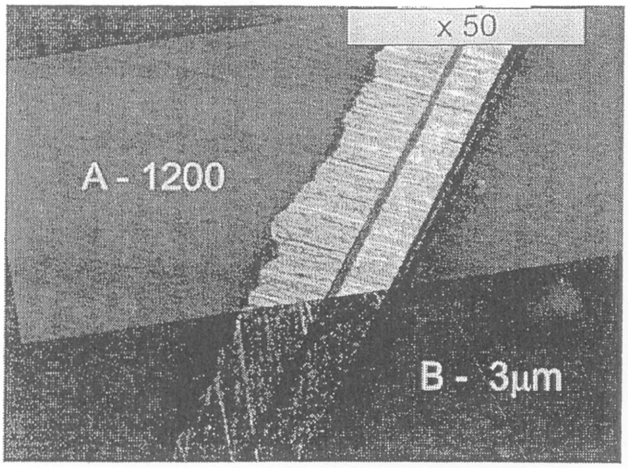

FIGURE 6. COMPARISON OF DIFFERENT LEVELS OF PREPARATION

Observed are layer of electrochemical copper, nanoporous oxide and aluminum. Area A is prepared with SiC grinding paper P1200, and area B is polished with diamond suspension of 3μm.

It is interesting in this case, that the distinguishing of material outline is better during grinding process. A explanation for that effect can be lower level of smearing.

The role of magnification used for examination of material details is shown on Figure 7. To determine the grow profile of pure nanoporous oxide, high magnification has to be used.

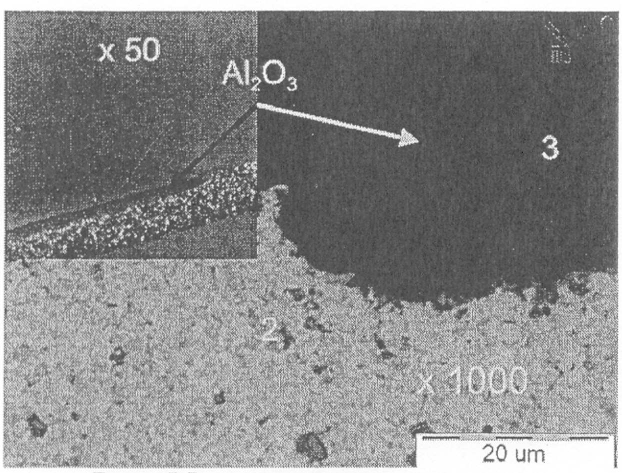

FIGURE 7. PROFILE OF SELECTIVE GROWN OXIDE

The shown specimen is a profile of porous oxide grown using mask of own oxide.

Figure 8 represents cross-section of nanostructured oxide membrane with metal core having thickness of 0,5 um. On the picture it is clearly visible the different oxide structures, which is grown without preliminary fuze preparation.

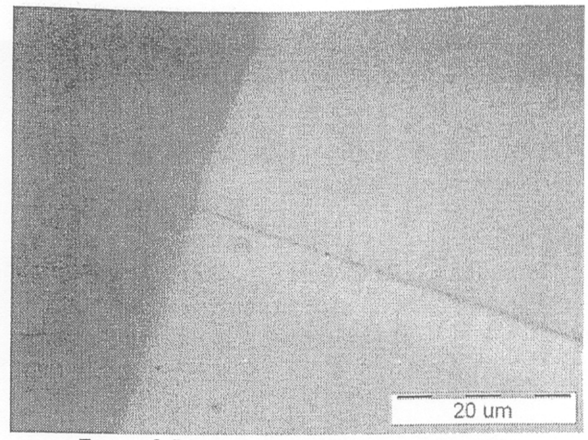

FIGURE 8. PROFILE OF SELECTIVE GROWN OXIDE

VI. CONCLUSIONS

In the article have been presented experimental results from cross-sectioning of different nanostructured materials. Different levels of preparation as well as different modes of observation have been applied. Shown is that when using appropriate observation mode, magnification and preparation, microparameters of nanostructured materials can be evaluated. For improved results further investigations are necessary with levels of polishing below 1 μm. Also the parameters of the moulding can be optimized – different coefficient of deformation and viscosity of resins.

REFERENCES

[1]http://www.nanoword.net/library/links/publications.php open 05.2010

[2] M. H. Lankhorst, B. W. Ketelaars, and R. A. Wolters, *Low-Cost and Nanoscale Non-volatile Memory Concept for Future Silicon Chips*, Nat. Mater. 4, No. 4, (2005) pp. 266-267

[3]http://www.icems.ist.utl.pt/functionalisation/nanomanipulation/ open 5.2010

[4] A.A. Poznak. *Modified anodic alumina and composite materials based on it,* BSU, Minsk, 2007 ISBN 978-985-476-561-7 (in Russian)

[5] Socol V.A. *Electrochemical technology of hybrid integrated circuits,* Besprint, Minsk, 2004, ISBN 985-6767-04-0 (in Russian)

[6] A. Bogner, G. Thollet, D. Basset, P.-H. Jouneau, C. Gauthier, *Wet STEM: A newdevelopment in environmental SEM for imaging nano-objects included in a liquid phase*, Ultramicroscopy V. 104 Issues 3-4 (2005), pp 290–301

[7] F. Sarioglu, M. Liu, K. Vijayraghavan, A. Gellineau, O. Solgaard, E. L. Ginzton, *Nanoscale Material Characterization with Differential Interferometric Atomic Force Microscopy,* http://www.stanford.edu/group/nnincomputing/talks/O.Solgaard.pd, open 06.2010

[8] http://www.struers.com

[9] Struers Metalog Guide