

This article appeared in a journal published by Elsevier. The attached copy is furnished to the author for internal non-commercial research and education use, including for instruction at the authors institution and sharing with colleagues.

Other uses, including reproduction and distribution, or selling or licensing copies, or posting to personal, institutional or third party websites are prohibited.

In most cases authors are permitted to post their version of the article (e.g. in Word or Tex form) to their personal website or institutional repository. Authors requiring further information regarding Elsevier's archiving and manuscript policies are encouraged to visit:

<http://www.elsevier.com/copyright>



Contents lists available at ScienceDirect

# Nuclear Instruments and Methods in Physics Research A

journal homepage: [www.elsevier.com/locate/nima](http://www.elsevier.com/locate/nima)

## Advances in anodic alumina MCP development

Gleb Drobychev<sup>a,\*</sup>, Andrei Barysevich<sup>a</sup>, Kirill Delendik<sup>b</sup>, Patrick Nédélec<sup>c</sup>, Daniel Sillou<sup>d</sup>, Olga Voitik<sup>b</sup>

<sup>a</sup> Institute for Nuclear Problems, 11, Bobruiskaya Str., Minsk 220030, Belarus

<sup>b</sup> Institute of Physics, 22, Logojsky Trakt., Minsk 220090, Belarus

<sup>c</sup> IPNL, Domaine scientifique de la Doua, Université C. Bernard, 4, Rue Enrico Fermi, 69622 Villeurbanne, France

<sup>d</sup> Laboratory d'Annecy-le-Vieux de Physique des Particules, 9, Chemin de Bellevue, F-74941 Annecy-le-Vieux, France

### ARTICLE INFO

Available online 21 May 2009

#### Keywords:

Microchannel plate  
Electron amplification

### ABSTRACT

The development of microchannel plates (MCP) on the basis of anodic aluminum oxide (AAO) started in the recent years. High electric resistivity of AAO up to several GΩ makes impossible to obtain an amplification of the electrons current. Several approaches to increase the electric conductivity of AAO were studied by the authors. One of these approaches was successful and new AAO samples present a resistance around tens of MΩ, which can vary in a wide range depending on the production parameters. An etching technology, which has a characteristic “anisotropy” due to the porous structure of the AAO, is developed in order to achieve AAO MCP with optimal operational aspect ratio (OAR). This technique allows the production of open-ended channels that keep a constant diameter along the full length of a plate.

© 2009 Elsevier B.V. All rights reserved.

### 1. Introduction

Conventional microchannel plates (MCP) produced from lead silicate glass (LSG) have high gain ( $10^5$ – $10^6$ ), a sensitivity to single electron, a time resolution of the order of 100 ps, a high spatial resolution and the ability to work in strong magnetic fields. However, the existing production technology is expensive, and their production parameters are subject to large deviation.

In Ref. [1] we reported about the development of a low-cost, highly reproducible technology for MCP production based on anodic aluminum oxide (AAO). AAO is formed by electrochemical oxidation of aluminum in moderately dissolving electrolytes. AAO structure consists of regular, parallel hexagonal cells, normal to the surface of the aluminum substrate. Every cell is closed on the aluminum anode side by an oxide barrier layer (see Fig. 1a and b). According to our estimations AAO-based MCP will present low production cost, high reproducibility, the possibility to produce large-area (up to  $5 \times 5 \text{ cm}^2$ ) MCPs, large working temperature range, and high radiation hardness [1].

### 2. Doping of anodic aluminum oxide by metallic ions

Natural AAO presents a high electric resistivity (up to several GΩ) and so cannot afford the necessary gain to work as MCP. We studied different methods of AAO doping by metallic ions in order

to modify this resistivity. The main difficulty has been to implant a significant amount of metallic ions into the AAO structure in order to sensibly modify the electric conductivity without destroying the channels structure. Three methods were studied: (1) growth of AAO MCP from alloys of alumina with other metals; (2) modulation of voltage during electrochemical production of AAO; and (3) deposition of conductive layers on the MCP surface and on the walls of channels.

Several series of AAO samples were produced from alumina alloys (method 1) (see Table 1). The results of the tests have shown that the resistivity was not affected in comparison with pure AAO. The spectral analysis shows (see Fig. 2a) that metals other than Al do not incorporate into AAO from alloy. A possible reason being that during the alloy electrochemical oxidation, other metals form ions, which enter into solution and deposit on the cathode.

In the second method, for AAO production with a modulated voltage generator, a metal-containing compound (salt, acids, complexes) was introduced in the electrolyte. The AAO growth takes place during the anodic half-period and deposition of ions on AAO happens during the second one.

The idea was that metal would deposit on AAO the barrier layer and thus be incorporated into the AAO structure. Several metals were tested: Zn, Sn, Ni, Mg, Cu, W, Ta, Mo, and Pb. To date, we succeeded in incorporating lead (see Fig. 2b) and molybdenum. Measurements have shown that the amount of deposited metal may be well controlled and that the distribution of metal in the AAO volume is homogeneous. However, the resistance of the produced samples is close to the pure AAO's one. An explanation of this result could be that the metals are implemented into the

\* Corresponding author. Tel.: +375 17 226 42 21; fax: +375 17 2 26 51 24.  
E-mail address: [Gleb.Drobychev@cern.ch](mailto:Gleb.Drobychev@cern.ch) (G. Drobychev).

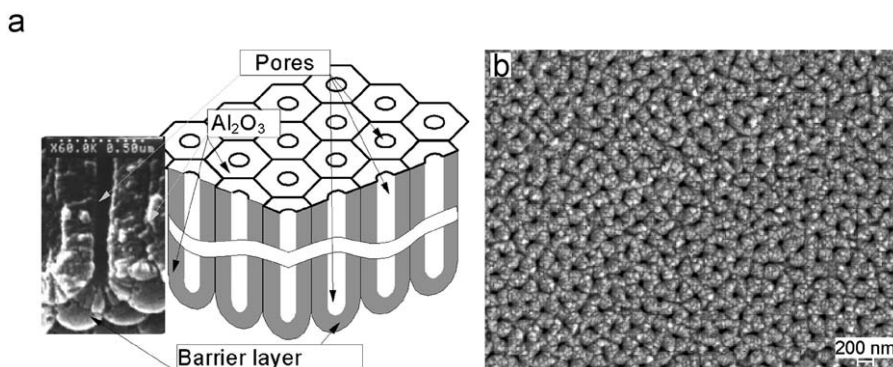


Fig. 1. Anodic aluminum oxide: (a) AAO structure and (b) electronic microscopy photography.

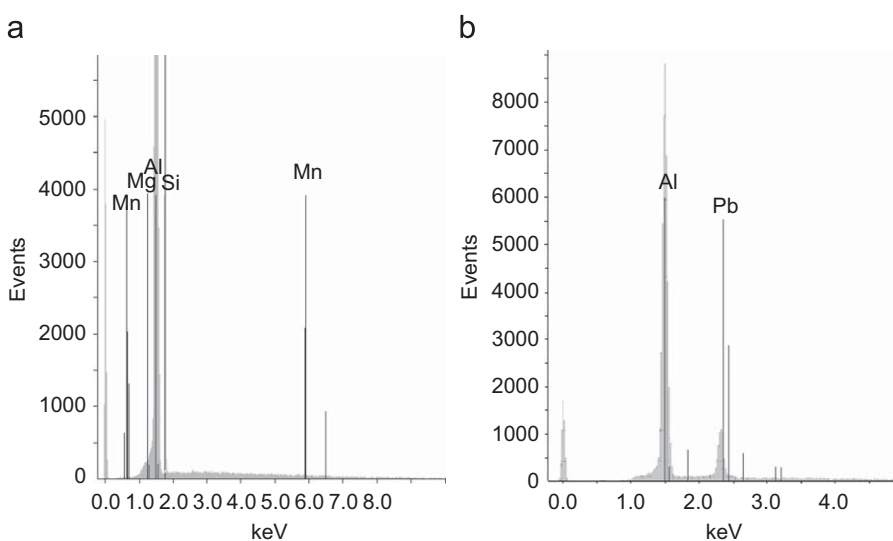


Fig. 2. Spectrograms of AAO: (a) sample produced from alloy and (b) sample doped with lead ions using a modulated voltage generator.

Table 1

The list of alloys used for production of AAO samples.

| Alloy | Doping  |         |         |
|-------|---------|---------|---------|
|       | Mg (%)  | Si (%)  | Mn (%)  |
| AL-8  | 9.3–10  | –       | –       |
| AMG-3 | 4.5–5.5 | 0.8–1.3 | 0.1–0.4 |

AAO structure under the form of oxides and it is necessary to de-oxide them partly to affect resistance of AAO. A reactivation can be performed by annealing in hydrogen atmosphere or in vacuum. The optimization of the annealing process is under work now.

The best results were obtained by deposition of conductive material on walls inside AAO channels (method 3). Among all possible methods, we chose to perform either metal sputtering in vacuum and oxidation, or deposition from metal-organic solutions followed by annealing. Nickel and magnesium oxide were tried. Good results were obtained with nickel films. The electric resistivity and the secondary emission coefficient were studied. Electric resistivity at room temperature was measured in the range from 40 MΩ/mm to GΩ/mm depending on density of conductive deposit in MCP. To date we reached a good reproducibility of MCP resistance within 20%. However, the secondary

emission coefficient of the produced samples is around one; so, further research for technology optimization is still required.

### 3. Optimization of channel length to diameter ratio of AAO MCP production

There is a general relation between the channel length to diameter ratio (operational aspect ratio, OAR) and the voltage [2]:

$$\gamma_{opt} = \frac{U_{MCP}}{A}$$

where  $U_{MCP}$  is the potential difference between two elastic interactions of electron,  $A$  is a parameter, the value of which ranges from tens to hundreds of volts for good secondary emitters. For “conventional” lead glass MCP, the OAR is in the range of 40–100. The maximal gain is achieved at OAR values in the range 30–60 depending on voltage and quickly falls to one with increase of OAR [2]. The thickness of AAO plate can vary from several μm to 250 μm and the channels diameter from 10 to 200 nm. The natural AAO MCP OAR is in a region from 100 to 25 000. An acceptable OAR value (from 20 to 90) leads, for 100 nm channels diameter, to a 9 μm or less thickness, which makes it too fragile. This makes it practically impossible to have amplification with natural porosity AAO. For this reason, we studied methods to form channels of larger diameters by etching through. Due to the AAO structure, the

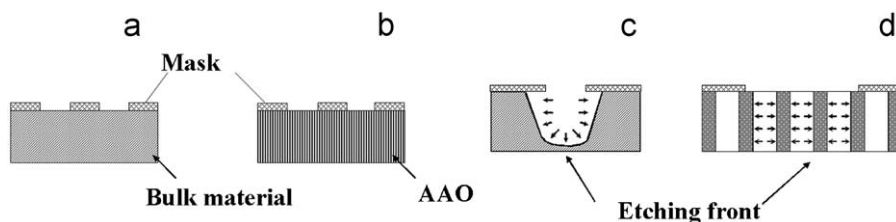


Fig. 3. The difference of etching processes in bulk (a, c) and porous (b, d) materials: (a, b) start of process; (c, d) etching results.

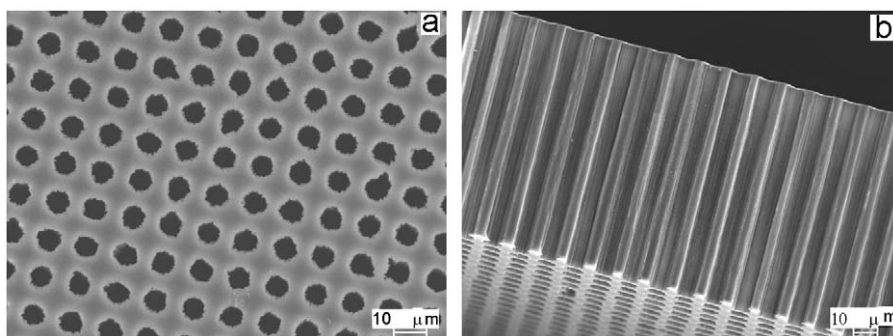


Fig. 4. Etched AAO MCP sample: (a) front view and (b) side cross-section.

etching process has a characteristic “anisotropy”, which allows producing MCP with vertical parallel channels of any diameter (within some technological limits). The difference in the etching processes in bulk and porous materials is illustrated in Fig. 3.

For experimental purposes we produced a photolithographic mask with 5- $\mu\text{m}$ -diameter channels at 5  $\mu\text{m}$  distances. The production procedure consists in growing AAO of a given thickness, separating the alumina plate from the substrate, depositing the mask, protecting the non-operational surface, etching the alumina through mask in multi-component acidic and alkaline, and dismounting and annealing the MCP matrix. During the etching process a very high filling factor of the MCP could cause the destruction of the mask and of the MCP structure. The parameters that were chosen for this production are as follows: diameter 24 mm; working surface diameter 20 mm; thickness 100–120  $\mu\text{m}$ ; channels diameter 5  $\mu\text{m} \pm 10\%$ ; inter-channels distance 4–5  $\mu\text{m}$ ; OAR of 20 or higher; number of channels  $1 \times 10^6$  per  $1 \text{ cm}^2$ . One of such structures is shown in Fig. 4.

#### 4. Conclusion

A technological process has been developed in order to increase the AAO conductivity. Three different approaches were studied and the best one was chosen. The new samples have resistivity that can be varied a wide region from 40 M $\Omega$ /mm to

G $\Omega$ /mm depending on the density of conductive deposit in MCP. To date we reached a good reproducibility of MCP resistance within 20%.

An etching method, which makes use of the porous AAO structure “anisotropy,” allows the production of structures with characteristics suitable for MCP. Resulting channels are open and have a constant diameter all along their full length. An optimization of this technology is still required, in order to etch plates of 150–180  $\mu\text{m}$  thickness conserving the MCP parameters.

#### Acknowledgments

The authors acknowledge French Ministry of Foreign Affairs, the CNRS (France), and Belarusian Republican Foundation Fundamental Research, the support of which, through different programs, was extremely useful in completing this work. Dr. G. Drobychev also wishes to thank Ministry of National Education of France and Savoie University (France) for support.

#### References

- [1] Gleb Drobychev, Andrei Barysevich, Kirill Delendik, Anton Karneyeu, Patrick Nedelec, Daniel Sillou, Olga Voitik, Nucl. Instr. and Meth. Phys. Res. A 567 (2006) 290.
- [2] Joseph Ladislav Wiza, Nucl. Instr. and Meth. 162 (1979) 587.