

Research article

The effect of the anodization voltage changes on the electric resistance of porous anodic alumina barrier layer and its thickness used in strain sensing devices

Abolfazl Salehi Amnieh Khuzani*

Department of Physics, Khomeinishahr Branch, Islamic Azad University, Isfahan, 84175-119, Iran

*salehi.aba@gmail.com

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Abstract

Strain sensing is the aptitude to measure the mechanical/electrical and other responses towards strain in a material. In this regard, various types of nanocomposite strain sensors have been developed. For strain sensing applications, various aluminum nanomaterials and other nanoparticles have been used. To investigate the influence of aluminum anodization voltage on the barrier layer thickness of porous anodic alumina (PAA) and its electric resistance used in strain-sensing technology, samples in different voltages, and various conditions of mild and hard anodization were fabricated. Measurement of electrical resistance of barrier layer during the voltage change shows that there is a transition region in which electric resistance of samples is located between hard and mild anodized region. The SEM investigation reveals that the barrier layer thickness is linearly proportional to the anodization voltage. But the barrier layer resistance in hard anodization is lower than that one in mild anodization. The observed paradox can be related to higher ionic mobility of ions in the barrier layer which is formed in hard anodization.

Keywords: Porous anodic alumina, Strain sensing devices, Mild and Hard anodization, Electrical resistance, Sensor resistance, Barrier layer thickness.

1- Introduction

Aluminum and its alloys are the most commonly used structural materials in aerospace industries. Anodized porous anodic alumina is expansively attended because of its industrial applications and production of nanostructures. Its applications can be mentioned as strain sensing devices [1-2], producing catalysts [3], metallic nanowires [4], magnetic memories [5], and producing electronic or optical devices [6].

In different researches, diverse anodic alumina thicknesses have been tested and their effect on protection against humidity is studied by monitoring sensor resistance. Strain gauge-based pressure sensors are popular choices among pressure measurement devices [7]. One type of transducer employs a strain gauge bonded to a diaphragm, whereas another method is based on the deposition of a thin film strain gauge directly on the diaphragm. The deflection of the diaphragm directly induces strain on the thin film, causing a

change in the resistance values corresponding to tensile and compressive load. This can be measured using a Wheatstone bridge configuration and is therefore related to the pressure being measured [8]. Even in the case of adverse working conditions and for increased mechanical strain (up to 1.2%), it is found that an 11-nanometer alumina layer provides sufficient sensor protection, while the proposed model remains valid. This certifies the appropriateness of the proposed strain-sensing technology for demanding applications, such as e-skin and pressure or flow sensing, as well as the possibility of developing a comprehensive computational tool for NP-based devices [1].

Anodization is an electrochemical process that is done to increase the thickness of the oxide layer on an alumina substrate. In 1995, a regular array of an anodic alumina nanoporous was introduced by Masuda and Fukada [9]. According to Fig. 1, we have an alumina template with hexagon tight-packed cells that have nanoporous at their centers. The porous diameter is variable from several nanometers to several hundreds of nanometers by changing the conditions of the anodization process [10]. Due to the nature of the electrolyte as well as the applied voltage, the anodization process is done in two ways of mild and hard anodization. By mild anodization, the structure of porous is regular but the process is done too slowly [9]. So, hard anodization is introduced to produce high-order alumina nanoporous in a short time. Hard anodization is done at high current and voltage [10].

This article tries to investigate the effect of voltage changes of mild and hard anodization, on the electric resistance of

the porous anodic alumina barrier layer and its thickness.

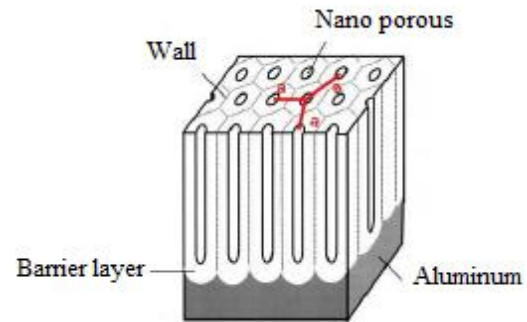


Fig. 1 configuration of produced nanoporous by anodization on an alumina template.

2- The Experiment Method

Before the anodization process, the aluminum sample should be prepared. At first, the sample should be washed well with acetone in an ultrasonic bath. Then the sample is electropolished in the mixture of ethanol and hydrochloric acid in a ratio of four to one. During the electropolishing process which is done under a potential of 20 volts, the aluminum surface reacts with perchloric acid and minimizes the roughness of the aluminum surface. The acidic solution for the anodizing process is 0.3 M oxalic acid. The anodization process is done at a temperature of zero degrees Celsius and this temperature is maintained by the cooling bath during the anodizing process in which a lot of heat is generated. To investigate the influence of aluminum anodization voltage on the barrier layer thickness of porous anodic alumina and its electric resistance, samples in different conditions related to mild and hard anodization starts at 40 volts and continues up to 130 volts. Measurement of electrical resistance of barrier layer during the voltage change is recorded by a computer and shows that there is a transition region in which electric resistance of samples is

located between hard and mild anodized region. The diagram data of the changes in current density, resistance and thickness of barrier are plotted in figures 2 to 5 respectively.

The anodizing process starts at 40 volts and continues for ten minutes at this voltage to form a protective layer on the aluminum substrate. For hard anodizing, the voltage rises at a constant rate. In this experiment, three samples were examined, the first sample is made under mild anodizing at 40 volts and the other two samples are made at 90 and 130 volts under hard anodizing conditions. The current and voltage of the sample are recorded by two multimeters connected to a computer. Finally, scanning electron microscope images were prepared from the samples, and along with the current-voltage curve with respect to time, the effect of different anodization voltages on the thickness of the alumina barrier layer and consequently its electrical resistance was investigated.

3- Discussion

The voltage- current diagram is shown in Fig. 2. This chart can be divided into three areas: In the first 600 seconds, the current before the red circle is mild anodization and the anodizing current is low (about Milli ampere on Square centimeters).

After this period, with a gradual increase in voltage, the current begins to increase, and then this increase takes on an exponential state, and after reaching a maximum value, it gradually begins to decrease. The anodizing phase changes from mild to hard as the current increases exponentially. The area marked with a red circle is a saturated area where the anodization current is transferred from the mild phase to the hard

phase and a phase transition occurs during the process.

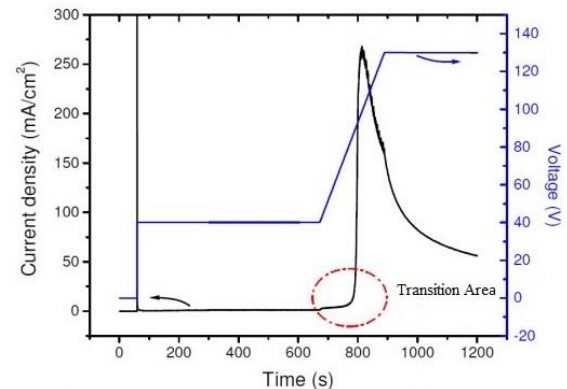


Fig. 2 Graph of current and voltage density over time during the anodizing process.

After the red area, the sample enters the hard anodization. The voltage-resistance curves with respect to time are shown in Fig. 3. The three regions marked in Fig. 2 are well visible in Fig. 3 as three different resistance regions. The remarkable note about these experiments is that as the voltage increases, the resistance of the sample starts to decrease and reaches an almost constant value.

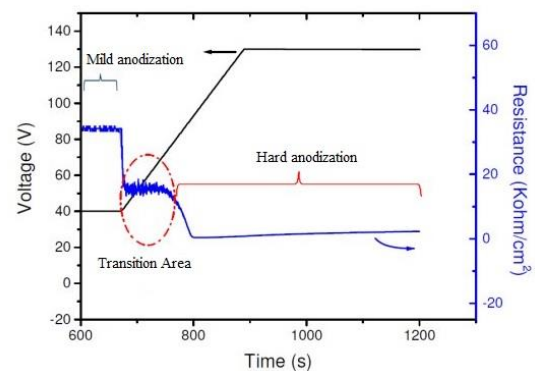


Fig. 3 The voltage-resistance graph with respect to time.

Due to the anodizing current trend, it is predicted that the resistance changes of the sample are severe; and these resistance changes can be studied with a rough and simple approximation. The simple approximation is to assume that the barrier

layer behaves like an ohmic resistance, whose area is marked with the red circle.

The resistance of the transition zone is approximately between the soft and hard anodization regions. After this area, the current shows a downward behavior again, and reaches a minimum value at the voltage of 90 volts and then slowly starts to increase. Given that all chemical reactions take place in the barrier layer area, to examine the specimens more closely, scanning electron microscope images have been prepared from the cross-sectional area of the barrier layer of the specimens made at 40, 90, and 130 volts, which are shown in Fig. 4.

Based on scanning electron microscope images and the diagram in Fig.3, the thickness of the barrier layer and its resistance at these three voltages in terms of anodization voltage is plotted in Fig. 5.

As shown in Fig. 5, the thickness of the barrier layer is almost linearly related to the anodizing voltage, but the resistance of specimens made in hard anodizing conditions (90 and 130 volts) is much lower than the resistance of specimens made in mild anodizing conditions (40 volts), although the thickness of the soft anodization samples is much larger.

This indicates that the material of the barrier layer in the hard-anodizing process is different from the barrier layer made in mild anodization, as other researchers have said, the electrical mobility of the anions forming the barrier layer of the hard anodizing is faster than the anions created in the mild anodizing conditions. Therefore, it is predicted that this is an effective factor in reducing its electrical resistance compared to the barrier layer of mild anodizing [11-12].

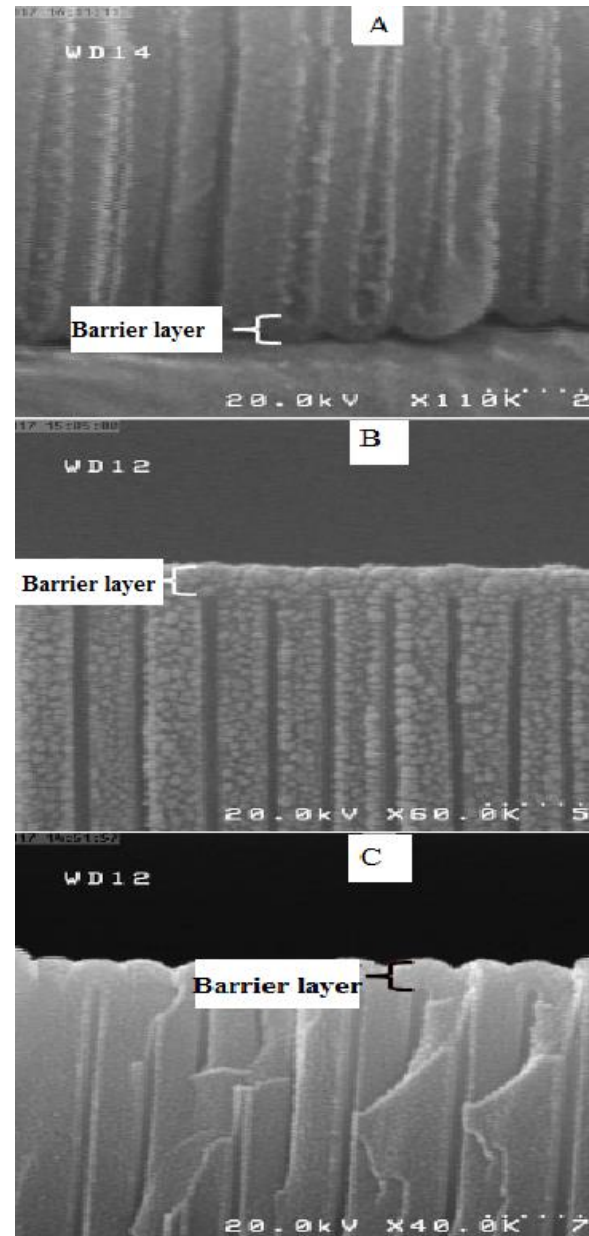


Fig. 4 Scanning electron microscope images of the cross-section of nanoporous alumina made at mild and hard voltages: a) 40 volts b) 90 volts and c) 130 volts.

Another noteworthy point is the slight increase in the resistance of the barrier layer of the template made under the voltage of 130 volts compared to its resistance at the voltage of 90 volts, which can be due to the difference in the length of the spread between them. In fact, after the voltage is fixed in the hard anodization, due to the increase in the depth of the holes, the propagation length increases and

this leads to a gradual increase in resistance [11].

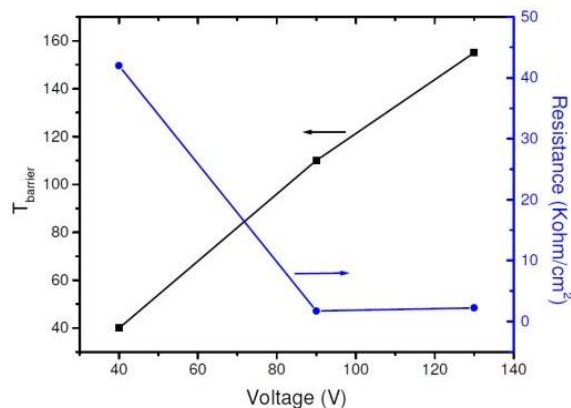


Fig. 5 Diagram of the thickness of the barrier layer and its electrical resistance at voltages of 40, 90 and, 130 volts.

4- Conclusion

To investigate the effect of aluminum anodizing voltage on electrical resistance and thickness of the barrier layer of porous anodic alumina, samples were made in mild and hard anodizing conditions. Measurement of the resistance of the samples shows that during the change of the anodization voltage from mild to hard, there is a transition zone whose resistance is between the resistance of the mild and hard anodization zone. The thickness of the barrier layer is almost linearly related to the anodizing voltage, but the resistance of specimens made in hard anodizing conditions is much lower than the resistance of specimens made in mild anodizing conditions, although the thickness of the soft anodization samples is much larger. This difference is related to the difference in electrical mobility of anions in the barrier layers formed in mild and hard anodizing processes.

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