



A comparative study of electrochemical barrier layer thinning for anodic aluminum oxide grown on technical purity aluminum



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ABSTRACT

Anodization of AA1050 foil in 0.3 M oxalic acid at 50 V was performed and nanoporous alumina was formed. Four various approaches of electrochemical barrier layer thinning with varied voltage steps were applied to optimize the experimental conditions. Scanning electron microscopy was employed to investigate the bottoms of the anodic alumina after electrochemical barrier layer thinning. Values of the barrier layer thickness derived from Bode plots extrapolations confirmed the FE-SEM observations. For the most suitable barrier layer the thinning procedure was applied to successfully form nickel nanowires via pulse electrodeposition.

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1. Introduction

Nanoporous anodic aluminum oxide (AAO) due to its highly-ordered hexagonal morphology is very often applied as a template for nanofabrication [1]. To form hexagonally arranged anodic aluminum oxide, numerous electrolytes have been applied, including sulfuric [2,3], oxalic [2–5], phosphoric [2], chromic [6], selenic [2,7,8], acetylenedicarboxylic [9], croconic [10], citric [11,12], malic [13], malonic [11], rhodizonic [10], squaric [14], sulfamic [15,16] and tartaric acid [11] at various voltage ranges. Moreover, various strategies like hard anodization and anodization in solutions containing alcohol or in non-aqueous solutions were also developed to find more optimal strategy for anodization [17–19] and control of geometrical features of anodic alumina [17–21]. After self-organized, two-step anodization, providing well-ordered periodically aligned nanopores, unoxidized aluminum beneath oxide layer is still present [1]. To obtain templates suitable for nanofabrication, through-hole membranes have to be fabricated [1,2]. For this purpose, firstly aluminum is chemically removed by immersion in saturated water solution of HgCl_2 [22–24], SnCl_4 [25], or CuCl_2 dissolved in concentrated aqueous solution of HCl [26,27]. Then, due to the redox reaction, aluminum is oxidized and mercury, tin or copper cations are reduced. Next, to open the barrier layer at

pore bottoms, anodic alumina layer has to be immersed in diluted phosphoric acid [22–27]. Furthermore, gold has to be sputtered on the bottom of so prepared membrane and short electrodeposition of gold has to be performed to provide electric contacts at the bottom of every pore [22,24,26]. Therefore, template-assisted fabrication, employing anodic aluminum oxide does not require expensive equipment. However it is very time consuming process due to the many steps of through-hole membrane preparation. Nevertheless, such membranes have been successfully commercialized and are available on the market [28]. Another approach is detachment of anodic alumina employing $\text{HClO}_4\text{:C}_2\text{H}_5\text{OH}$ mixture, commonly used for aluminum electropolishing [29]. Chen et al. applied 3 s long voltage pulses to detach AAO from unoxidized aluminum. However, this method has not been employed yet to technical purity aluminum. Nevertheless, such procedure is also another step applying chemicals and time.

To shorten the preparation procedure, electrochemical barrier thinning was worked out. Furneaux et al. proposed in 1989, before Masuda and Fukuda published their results about two-step self-organized anodization [30], how to open the barrier layer of anodic aluminum oxide using electrochemical approach [31]. The method, known in the literature as barrier layer thinning (BLT) is based on a gradual voltage decrease and it is being re-discovered now by the researchers working on the AAO template assisted nanofabrication. The whole trick of the successful electrochemical BLT consist in the observation of current–time curve. The voltage has to be lowered

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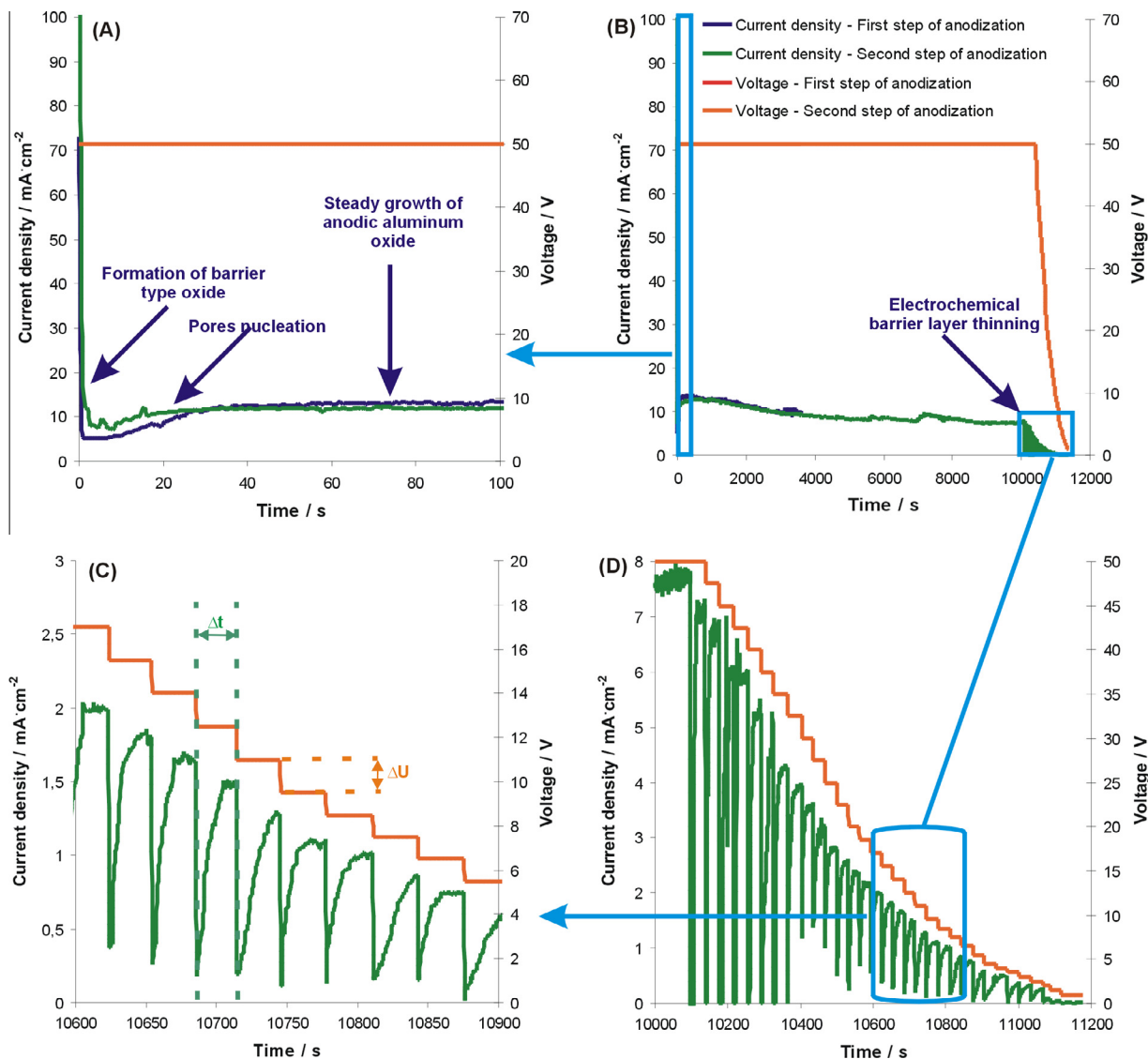


Fig. 1. Current density and voltage versus time for the first and second step of anodization. First phase of anodic oxide growth rate (a) as well as whole current and voltage curves (b) are shown with zoomed in electrochemical barrier layer thinning process (c and d).

Table 1
Various attempts of the electrochemical barrier layer thinning.

Number	Voltage range (V)	ΔU (V)	Current curve	FE-SEM images
I	50.0–20.0	2.0	Fig. 2a	Fig. 3a and b
	20.0–10.0	1.0		
	10.0–5.0	0.5		
	5.0–1.0	0.1		
II	50.0–20.0	2.5	Fig. 2b	Fig. 3c and d
	20.0–9.5	1.5		
	9.5–4.5	1.0		
	4.5–1.0	0.5		
III	50.0–30.5	1.5	Fig. 2c	Fig. 3e and f
	30.5–20.5	1.0		
	20.5–10.0	0.5		
	10.0–5.2	0.3		
	5.2–1.0	0.1		
IV	50.0–29.0	3.0	Fig. 2d	Fig. 3g and h
	29.0–19.0	2.5		
	19.0–11.0	2.0		
	11.0–5.0	1.5		
	5.0–1.0	0.5		

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