

Photoluminescence and diffuse reflectance of porous anodic alumina (PAA) anodized in oxalic acid and phosphoric acid mixtures

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Introduction

Porous anodic alumina (PAA) is used as a template for fabrication of nanostructures and to prepare photonic devices¹. When excited by ultraviolet radiation, PAA exhibited a broad luminescence band in blue region². The comprehension and control of the optical properties are important in biosensors³. In this work, the effect of the addition of phosphoric acid in oxalic acid electrolyte during anodisation of the PAA films was investigated by modification of the molarity of the phosphoric acid in the mixture. The films were characterized by fluorescence and diffuse reflectance spectroscopy. Different emission and diffuse reflectance spectra were obtained indicating the influence of electrolyte impurities on the optical properties of the PAA films.

Results e Discussion

The PAA films were prepared by potentiostatic anodisation of Al foils (1150 alloy, 99.5%) at 20 °C for 3600 s. The anodizing conditions are described in Table 1.

Table 1. Anodizing conditions of Aluminum.

Sample	Electrolyte composition / molL ⁻¹		Applied Potential / V
	[H ₂ C ₂ O ₄]	[H ₃ PO ₄]	
A1	0.15	0.05	45
A2	0.15	0.25	45
A3	0.15	0.05	20
A4	0.15	0.25	20
A5	0.30	-	45

Measurements of photoluminescence emission and diffuse reflectance of the PAA films were performed. Figure 1(A) depicts the photoluminescence emission spectra of the PAA films using a 357 nm excitation light. It was observed that the introduction of phosphoric acid in the electrolyte affected the intensity and shape of the emission spectra. The films prepared in electrolyte mixtures presented the Fabry-Pérot oscillations. This interference effect was not observed in the PAA film anodized in oxalic acid (sample A5). Figure 1(B) shows the diffuse reflectance spectra of PAA films. Note in these spectra the presence of interferences in samples prepared in electrolyte mixture. However, the concentration of phosphoric acid had no effect on interference patterns or intensity. On the other

hand, the applied potential affected the intensity of reflectance. AAP films produced at 20V presented high intensity of reflectance and low frequency of oscillations than those anodized at 45V.

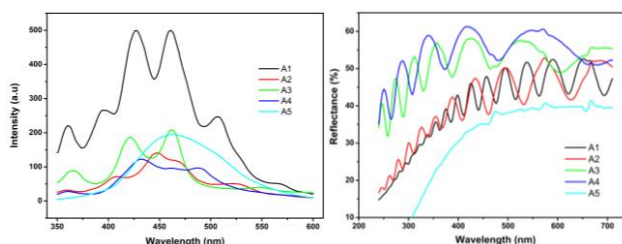


Figure 1. (A) Photoluminescence emission and (B) reflectance spectra of the PAA films. $\lambda_{exc} = 357$ nm.

SEM micrographs of PAA surfaces showed the changing in pore diameter with the anodizing conditions, *i. e.*, the electrolyte composition. These changes can lead to different morphological and optical properties.

Conclusions

The introduction of phosphoric acid affected the intensity and shape of the photoluminescence emission spectrum. The phosphoric acid also provided interference patterns in diffuse reflectance spectra of these samples, since this phenomenon was not observed at AAP films prepared only in oxalic acid. However, the intensity of reflectance was affected by the applied potential during the film growth.

The differences observed in patterns can be ascribed by changes in the pore structure and thickness of the pore layer attached on the aluminum substrate and it is a consequence of increasing the phosphoric acid content in the electrolyte during the anodisation and the applied potential.

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¹Grzegorz, D.S.in: Eftekhari, A. (Eds.), *Nanostructured Materials in Electrochemistry* (1st Edition), Wiley-VCH, Berlin, 2008, p. 1-116.

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³ Santos, A.; Alba, M.; Marsal, L.F. *et al. Nanoscale Res. Lett.* **2012**, *7*, 228.