Controlling Interferometric Properties of Anodically Formed Nanotubular Tantalum Oxide Films

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Introduction

It has been known for decades that metals with a thin oxide coating are capable of producing interference colours by a Fabry-Perot interference phenomenon (1). The colour produced by constructive interference of light reflecting from the air/oxide and oxide/metal interfaces is directly related to the oxide film thickness, and this property has been used as a convenient way to determine oxide film thickness by the unaided eye. Recently, interference colours of nanostructured metal oxides formed at Al, Ti, and Si have been reported. The observed colour of these nanostructured oxide films is dependent on film thickness, surface modification, and porosity of the nanostructures. These parameters have been investigated for potential applications in sensing and displays (2,3). In this study, the interference colours of Ta oxide nanotubular films ranging in length from 20-500 nm are investigated in order to develop a method of nanotube length calibration.

We have recently reported that thin films of highly ordered Ta_2O_5 nanotubes (NTs) can be formed by the anodization of Ta foil (4). The Ta_2O_5 NTs were prepared by anodizing a Ta foil in a solution containing HF, H_2SO_4 and H_2O in a two-electrode cell configuration. Ta_2O_5 NT diameter was controlled by the applied voltage, and NT length was varied by controlling the anodization time and HF concentration (5).

Methods and Results

Figure 1 shows aligned NTs with good adhesion to the Ta substrate (bottom), formed by anodization of Ta foil for various times.

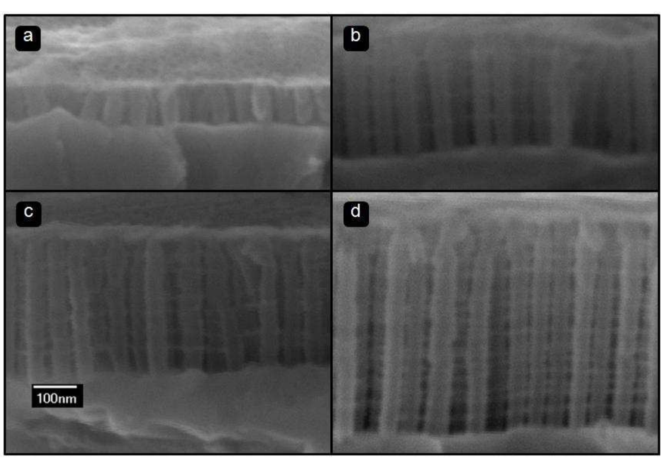

Fig. 1: Cross-sectional FE-SEM images of mechanically fractured Ta oxide NTs, formed by anodization of Ta at 15 V in 16 M H_2SO_4 + 0.6 M HF for (a) 5.85; (b) 18.3; (c) 23.9; and (d) 37.6 seconds.

Upon decreasing the length of the Ta_2O_5 NTs, it was seen that Ta NTs as short as ~20 nm are coloured. The colour of the NT film changes with NT length, progressing through brown, deep purple, deep blue, yellow, orange, purple, and blue as the NT length increases from 20-500 nm. Beyond a length of ~500 nm, teal and pink colours dominate the visible appearance of the oxide films.

Knowing the length (and variation in length) of NTs is important for many applications, such as the use of Ta_2O_5 NTs as a template for an ordered biosensor. Currently, determining NT length requires SEM imaging of each sample (such as in Fig. 1), which is time-consuming, expensive, and destructive of samples. To evaluate colour as a method of determining the thickness of the nanostructed Ta oxide film, diffuse reflectance spectra in the visible wavelength range were obtained for several coloured samples. Reflectance peaks shift to longer wavelengths as the NT length increases, and new (higher order) peaks appear in the shorter wavelength region (shown in Figure 2), with peaks corresponding to the observed colour of the sample.

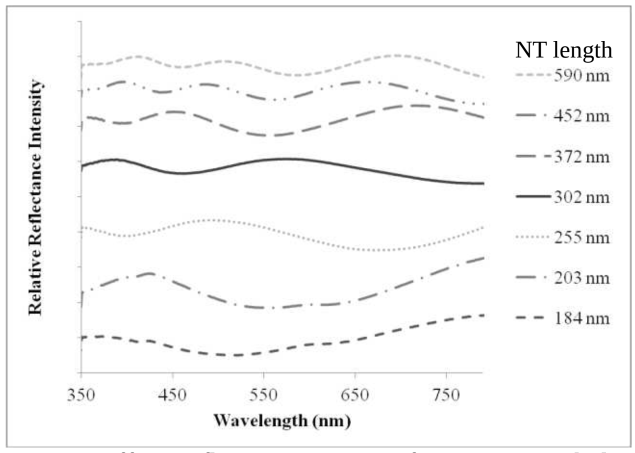

Fig. 2: Diffuse reflectance spectra of Ta_2O_5 nanotubular films of varying thickness, ranging from 185 to 590 nm.

The peaks and valleys present in the diffuse reflectance spectrum were used to calculate the effective refractive index of the Ta_2O_5/air nanostructured layer, and also the nanotubular film thickness. A good correlation is seen between the position and spacing of adjacent spectral peaks and the NT length, as determined by SEM imaging. The many interesting optical properties of nanostructured materials that were observed in this study, such as the visible colour change upon NT infiltration with different analytes, make these materials interesting candidates for visual sensors.

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