

## ABSTRACT

A low-cost experimental setup for electrochemical anodization and electro-polishing of metals was designed, which is capable to control experimental variables, like voltage, time, temperature, and stirring rate. The nanoporous anodic aluminium oxide and titanium oxide films were prepared on commercially pure aluminium (99.593 wt.%) and titanium (99.618 wt.%) substrates by using 0.3M oxalic acid and 2% HF aqueous solution as electrolytes, respectively. The effects of formation parameters, namely anodization voltage (10 to 55 V), electrolyte temperature (-10 to 30 °C), and anodization time (5 – 40 min), on the kinetics of ions movement inside anodization chamber in terms of total current passed, average current density, total charge transferred, film thickness, and growth rate, was measured and analyzed. It was also revealed that the thickness of anodic oxide film calculated by Faraday's Law (0.2 – 6  $\mu\text{m}$ ) was far less than that measured by SEM (21 – 131  $\mu\text{m}$ ). The surface morphology of anodic oxide films was studied by SEM. Various structural parameters, like pore diameter (20 – 120 nm), interpore distance (70 – 165 nm), pore circularity (0.74 – 0.90), pore density (42 – 570  $\mu\text{m}^{-2}$ ), and porosity (5 – 51 %), were determined by using software ImageJ. Values of these parameters were examined as a function of anodization temperature, voltage and time. The activation energy of the rate process of anodization was determined from the Arrhenius plot of current density for both aluminium (0.182 eV) and titanium (0.476 eV). The observations are in good agreement with those reported in the literature for expensive high-purity aluminium and titanium.

A detailed structural investigation of the metallic substrates was done using their XRD patterns with the help of Harris's analysis and Williamson – Hall analysis. XRD studies of the aluminum substrate shows that (220) and (311) are the preferentially oriented crystallographic planes. The intensity of diffraction peak pertaining to preferentially oriented crystallographic plane (311) is decreased whereas FWHM is increased on anodization; the extent of variation depends on the values of anodizing parameters, i.e. temperature, voltage, and time. The values of crystallite size  $D$  and lattice strain  $\epsilon$  in the substrate are also changed on anodization. Both  $D$  and  $\epsilon$  vary with the change

in a given anodizing parameter in an identical manner. However, in the case of titanium substrate (002) and (103) are the preferentially oriented crystallographic planes. The intensity of diffraction peak pertaining to preferentially oriented crystallographic plane (103) is increased whereas FWHM is decreased on anodization; the extent of variation depends on the values of anodizing parameters, i.e. temperature, voltage, and time.