Study The Influence of Immersion in The Synthesis of Thin Layers on a Composite Substrates.

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Abstract: *Dip coating is a common liquid deposition technique used in research and also for industrial production to produce polymeric, hybrid, and inorganic thin films of controlled thickness. During liquid deposition, the substrate withdrawal rate allows, in principle, easy tuning of the deposited film thickness. However, experimentally, unexplained thickness irreproducibility or strong fluctuations of sol-gel films are often observed when coating large substrates, which is a critical problem for optical coatings such as anti-reflective/reflective coatings. In this study, we present improved coatings obtained by sol gel-like multilayer structures composed of* ZrO_2-Y_2O_3 *coating. The phase composition and morphology were analyzed by X-ray diffraction spectroscopy (XRD), X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM) suitable for characterizing the surface properties of the obtained materials*.

Keywords: *DIP-COATING, SOL-GEL, THIN FILMS, MONOLITIC SUBSTRATES*

1. Introduction

Solution dip coating is a widely used method for depositing thin films on various substrates. With this liquid coating process, a homogeneous layer is easily deposited on the surface of the substrate and evaporation of the solvent leads to the formation of a solid film with good control over structure and thickness [1–4]. In principle, both organic and inorganic thin films can be obtained by this method and explored for many applications ranging from electronics [5–8], optical coatings [9], photocatalysis [10, 11] and more [12–14]. Despite the widespread use of sol-gel coatings both in industrial production and in research laboratories, physicochemical phenomena such as location during the coating process are still not fully understood. The first definition of the drain and drag phenomena involved in dip coating was published by Landau and Levich (LL) in 1942 for polymers[15], which built a theoretical model to describe the film formation mechanism and predict the final thickness under certain conditions. Solvent evaporation significantly affects the deposition process, its rate becoming a critical parameter that radically affects the thickness of the deposited film. Capillary mode enables the preparation of thick films with highly dilute solutions at ultralow withdrawal rates and can be used when the deposition species cannot be dissolved or dispersed in high concentrations.[10-15]

The present publication is aimed at the deposition of sol-gel coatings by the dipping method on composite substrates.

2. Experimental part

Eight multi-layer zirconia-yttria protective coating systems were deposited by the sol-gel method on 2 neobium-aluminum alloy substrates. Zirconium-based coatings are obtained from Zr butoxide; $Zr(OC_4H_9)_4$, a small amount of nitric acid was dissolved in 2-propanol. Acetic acid and acetylacetone were added as complexing agents. Finally, 5 wt.% polyethylene glycol added as a structural directing agent is added. The substrates were each immersed in the solution and withdrawn at a speed of 40 mm/min. After each deposition, the samples were sequentially dried at 100°C for 1 hour. The dipping-drying procedure is repeated three times. The deposits were then immersed in a yttrium solution. The final treatment of the samples was carried out at 400°C with a heating rate of 5°C/min.

3. Results and discussion

The phase composition of the samples was investigated by X-ray diffraction (XRD) with CuKα-radiation (Apparatus Philips PW 1050). Chemical composition and morphology were investigated by SEM analyses. X-ray photoelectron spectroscopy (XPS) was applied.

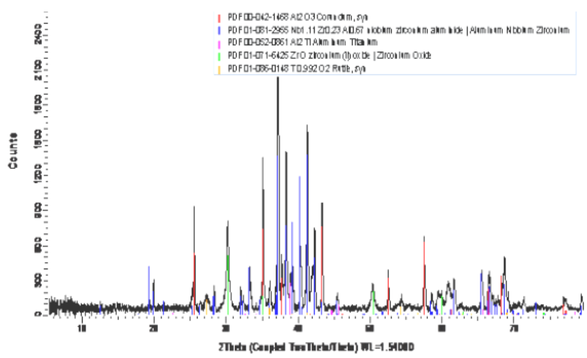
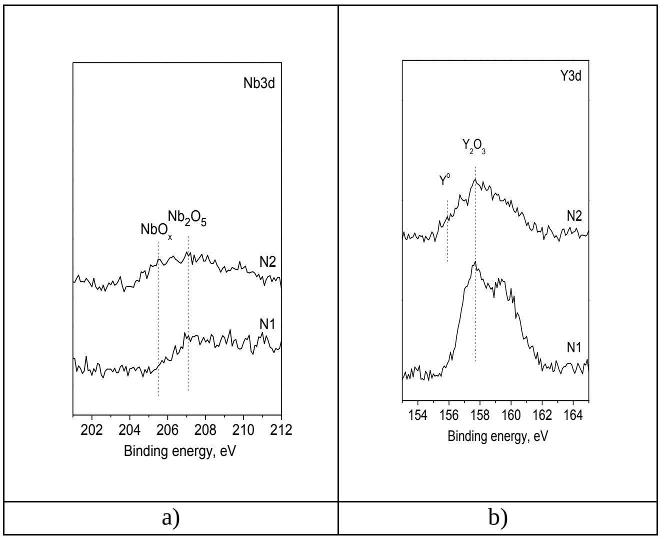
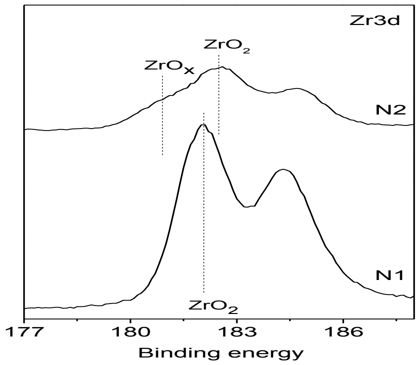


Fig. 1 X-ray diffractogram of deposited layers ZrO_2-Y_2O_3

Figure 1 shows an X-ray diffraction pattern. Angle 2θ varies in the interval 20-80°C. Peaks at approximately 38.5^O, 40.1^O correspond to $Nb_{1.11}$ ZrO_{23} $Al_{0.67}$ are illustrated.
The peaks at 39.1^O and 45.1^O show the link between Al_2Ti, while at 31^O, 51^O and 60^O the presence of ZrO_2 is proved. This study proves the deposition of the layer on the substrate despite the non-uniform structure of the substrate.

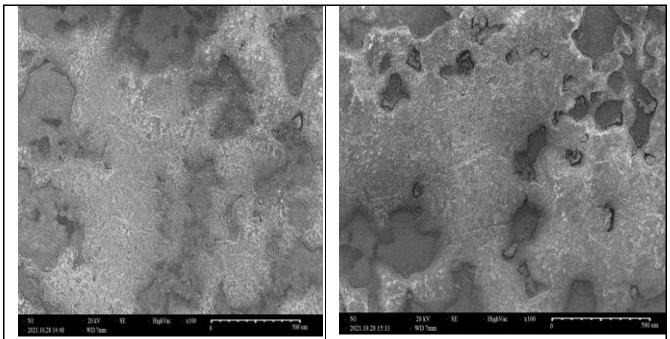




c)

Fig. 2 XPS analysis a),b),c)ZrO_2 modificran s Y_2O_3 prepared by zol-gel method and immersion deposition

The XPS analysis corroborates the results of the X-ray study. XPS spectrum of ZrO_2 modified with Y_2O_3 prepared by zol-gel method and dip deposition on monolithic Nb/Al sample. The sample was found to contain indeed ZrO_2, Y_2O_3 Figure 2(b) and (c). The Y_2O_3 peak at about 158.8 eV for sample N1 is more pronounced compared to sample N2. The second peak at 282.5 eV can be attributed to a characteristic peak for ZrO_2.
SEM (scanning electron microscopy) fig. 3 was conducted on a Scanning Electron Microscope (SEM) is “HIROX SH-5500P” with EDS analysis attachment included “Energy-Dispersive X-Ray Spectroscopy” (EDS) system “QUANTAX 100 Advanced” –Bruker



a)	b)

Fig. 3 SEM images of ZrO_2-Y_2O_3 thin layers

Fig. 3 a) and b) shows SEM images of yttria modified ZrO_2 thin films with eight deposits. It can be observed that these films are not uniform, have very low porosity and not very good adhesion to the substrate. Poor coverage is observed in places, which is due to the fact that the pad does not have a smooth surface. To overcome this problem, when applying the layers, an aging time of about 24 hours will be given, after which the next layer will be applied.

4. Conclusions

A stable sol of ZrO_2 - Y_2O_3 was synthesized and thin films deposited by the dip-coating method. X-ray Diffraction Spectroscopy (XRD), X-ray Photoelectron Spectroscopy (XPS), Scanning Electron Microscopy (SEM) studies were presented to characterize the results, which provided a clear insight into the preparation of the coatings and their deposition on a neo-alumina substrate.

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