

Original Article

ZrO₂-TiO₂ Multi-layered Nanostructured Coatings on AA5052 Substrate as Corrosion and Thermal Barrier Coatings

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Abstract - Nowadays, 80–90% of the components of airplanes are composed of aluminum alloys. Another material with a high level of corrosion resistance and is utilized in aircraft is AA5052. When exposed to the natural environment, AA5052 forms an oxide layer that shields the aluminum alloy from corrosion. However, this oxide layer is eroded and corrodes more quickly in the harsh weather conditions where an aeroplane operates. On the AA5052 substrate, multilayered nanostructured ZrO₂-TiO₂ coatings will be formed utilizing a spin coating and chemical bath deposition techniques to protect against this corrosion and heat resistance. Using an X-Ray Diffractometer (XRD), the produced coatings are examined for their crystallinity properties, surface morphological properties using Scanning Electron Microscope (SEM), elemental composition using Energy Dispersive X-ray Analysis (EDAX), optical properties using UV-visible spectroscopy and corrosion properties using salt spray technique.

Keywords - AA5052, Corrosion, Salt spray technique, ZrO₂-TiO₂ coating.

1. Introduction

The metal that is most frequently utilized in daily life is aluminum [1]. It is seen in producing anything from kitchenware to essential aeroplane parts. Pure aluminum can't be used to make the components [2] of an aeroplane since it lacks primary stability and is so sensitive. Because of its outstanding bendability, it may be machined successfully regularly and reinforced further by adding various metals such as Cu, Mg, Mn, and Zn, among others. It is estimated that 75 and 80 percent of aeroplane parts are constructed [3] of aluminum these days. Amazingly lightweight materials with strong strength and excellent erosion resistance are aluminum composites. Various applications call for various aluminum amalgams utilized in an airplane's wings, rudder, doors, flooring, exhaust pipes, motor turbines, and cockpit instruments [4]. Despite having considerable corrosion resistance, it loses this feature in extremely harsh weather. Therefore, ZrO₂ and TiO₂ are used to coat AA5052 to preserve and improve its corrosion resistance.

Zirconium [5] possesses excellent frictional conductivity, high strength, high break resistance, and high hardness. A few characteristics like Electrical protection, Low, warm conductivity, Corrosion resistance in acids and salts, almost Steel-Like Steel Flexibility Modulus, and almost Iron-Like Warm Extension Coefficient make Zr suitable for heat-reflective coatings [6].

A brilliantly developed metal with a silvery tone, low thickness, and exceptional strength is titanium. Utilization of titanium is impossible in seawater, regenerated water, or chlorine. Titanium is impressively less dimensional than steel while being just as strong. It is employed in various applications by alloying it with various materials to give it diverse qualities. They are used in aircraft, rockets, and other devices owing to their thinness and great temperature resistance. Additionally, it's employed in things like bicycles, bolsters, paint, PCs, anti-glare reflectors, and golf clubs.

In these experiments, the coatings [7] are made utilizing chemical bath deposition [8] and spin coating techniques, but they are prepared using the sol-gel approach. The sol-gel method is a type of wet synthetic process or compound arrangement testimony procedure [10] for the fusion of colloidal oxide scatterings that have been transformed into powders, strands, and thin film sandstone [11] monuments. According to the interaction, sol-gel coating [12] is a cycle of arranging single or multi-part oxide covering that may be glass, clay, or glasslike ceramic [13].

Spin coating [26] is an increasingly popular method of applying a uniform, thin fluid sheet to fragmented substrates, particularly small level plates, for the same purpose. The primary stream is unstable outspread waste, in which radial and goeey forces compete, so conventional (Newtonian) fluid of constant thickness [15] tends toward a uniform layer



that continuously becomes thinner. Unpredictable solvents [16] are typically used because it is frequently possible to separate shrinking by spin-off [17] from subsequent shrinking and hardness by drying. Even if they are hypothetically expected today, thickness and consistency are sensitive to speed, gas conditions, and the rheology of concentrating hardening fluids. Turn covering performs brilliantly for the rheology of photoresist covering in microelectronics. The interaction requests more cautious control for suspension coatings in attractive circle innovation; it is normally adjusted.

Chemical Bath Deposition [18], sometimes referred to as Chemical Solution Deposition or CBD, is a thin-film deposition method that creates solids from a solution or gas using an aqueous precursor solution. Heterogeneous nucleation is used in Chemical Bath Deposition to produce homogenous thin films of metal chalcogenides [27] (mainly oxides, sulphides, and selenides) and a variety of uncommon ionic compounds (the deposition or adsorption of water ions onto a solid substrate). Films can be consistently produced by Chemical Bath Deposition [20] using a straightforward process that needs little equipment, at a low temperature (100°C), and for an affordable price. Additionally, batch processing [21] and continuous deposition over a large area are also possible with chemical bath deposition. CBD films are commonly employed in semiconductors, solar cells, and supercapacitors, and there is growing interest [22] in employing Chemical Bath Deposition to make nanomaterials.

Spin coating is a rapid and simple method for applying thin coatings on materials [23] that are typically flat. When the substrate is spun, the coating solution spreads out and leaves behind a very uniform coating of the chosen material on the surface of the substrate [24]. A rotating fixture keeps the substrate in place, frequently using a vacuum to hold the substrate. The spin coating technique has extensively studied the parameters [25] that influence coating deposition and deposit thickness.

2. Materials and Methods

2.1 Materials Used

Titanium tetra-iso-propoxide (TTIP), Zirconium butoxide, Propan-2-ol, Ethanol, Nitric acid, and Acetyl acetone.

2.2 Synthesis

Firstly, we deposited the TiO₂ layer on the AA5052 substrate using chemical bath deposition. Later, the ZrO₂

layer was deposited on top of the TiO₂ layer through the spin coating technique.

2.2.1 TiO₂ Layer

8ml of propan-2-ol and 4ml of titanium tetra-iso-propoxide (TTIP) were combined, and the mixture was agitated for 10 minutes. 32ml of ethanol was added, and the mixture was agitated for 1.5 hours. TiO₂ solution was later made. We applied a 20-minute chemical bath deposition coating of TiO₂ to AA5052 substrates. At room temperature, the substrates were dried out for 60 minutes.

2.2.2 ZrO₂ layer

40ml of ethanol and 10ml of nitric acid were combined to create the ZrO₂ solution, which was then consistently agitated for 15 minutes with a magnetic stirrer. After that, 9ml of zirconium butoxide was added, and 30 minutes were spent stirring. 5.3ml of acetylacetone was also added afterward, and the mixture was agitated for an hour. The final product was a ZrO₂ solution that was pale orange. Furthermore, employing the spin coating process, the ZrO₂ layer was applied to TiO₂-coated AA5052 substrates. Coated substrates underwent a 3-hour annealing process at 300° C.

3. Results and Discussion

3.1. X-Ray Diffraction (XRD)

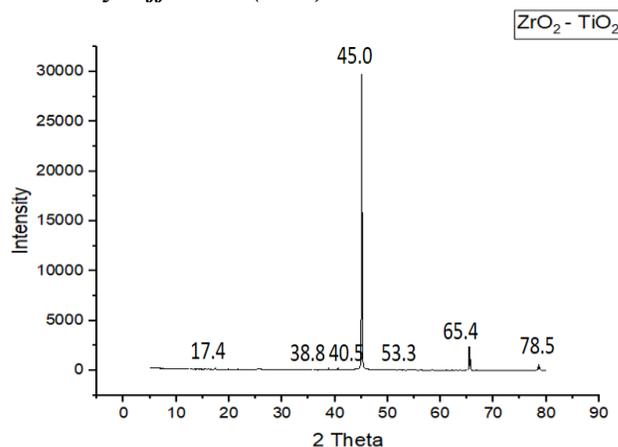


Fig. 1 XRD image of ZrO₂ - TiO₂ coated AA5052 substrate

Fig. 1 represents the XRD analysis of ZrO₂ - TiO₂ coated AA5052 substrate. We obtained peaks at 17.4°, 38.8°, 40.5°, 45°, 53.3°, 65.4° and 78.5° at the various intensities of 134.16, 182.15, 140.48, 17.79, 29327.72, 5.56, 2307.45 and 628.35 respectively. The maximum peak obtained was 40.5° at an intensity of 29327.72

3.2. Scanning Electron Microscope (SEM) Analysis

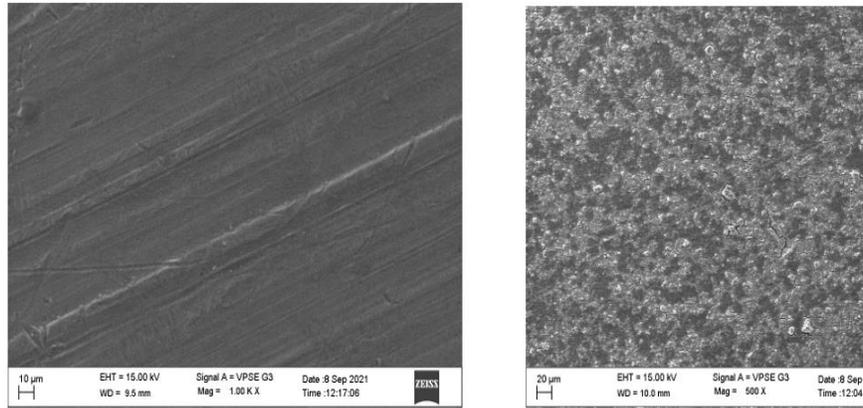


Fig. 2 SEM analysis of the a) Bare and b) ZrO₂-TiO₂ coated AA5052 Substrate

The above figures show the SEM images of bare and ZrO₂-TiO₂ coated AA5052 substrate. From the image of the coated substrate, we can observe multilayered ZrO₂-TiO₂ coatings. The coatings were non-uniform, non-homogeneous, and contained some particle agglomerations.

3.3. Energy Dispersive X-ray Spectroscopy (EDX) Analysis

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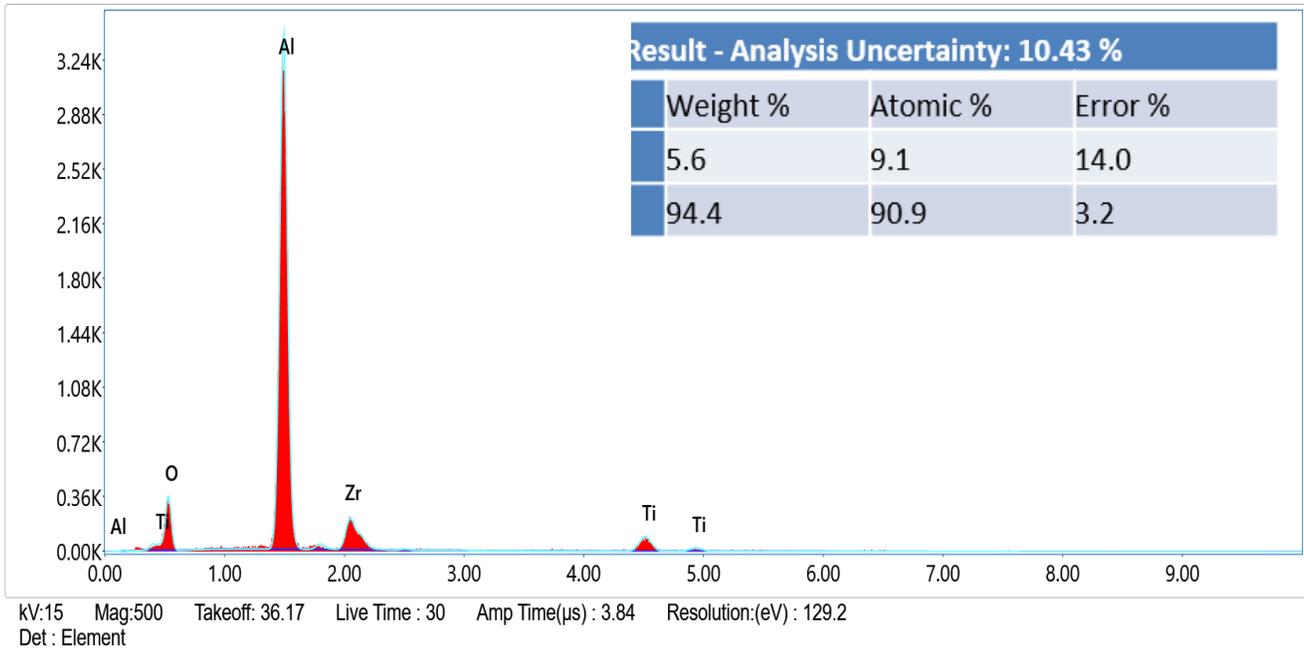
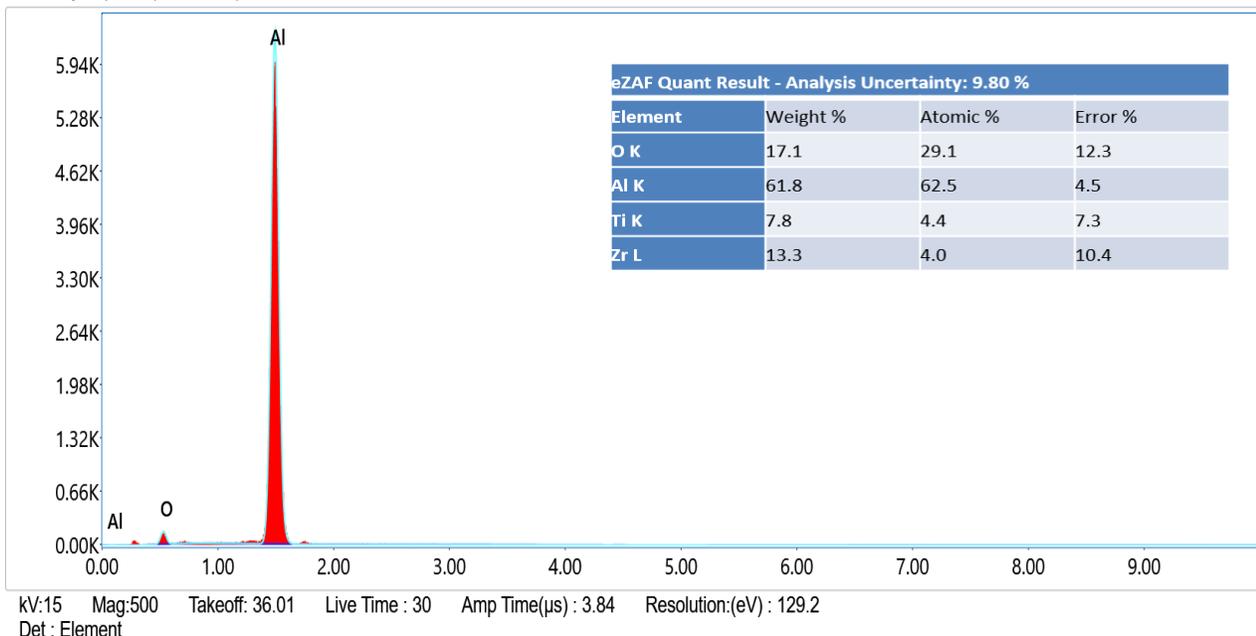


Fig. 3 EDX analysis of the a) Bare and b) ZrO₂-TiO₂ coated AA5052 Substrate

The above figures reveal the EDX analysis of bare and ZrO₂-TiO₂ coated AA5052 substrates. EDX image of bare substrate represents the presence of oxygen and aluminum, having a weight percentage of 5.6 and 94.4, respectively. The

EDX image of the coated substrate showed the presence of oxygen, aluminum, titanium, and zirconium, having a weight percentage of 17.1, 61.8, 7.8, and 13.3, respectively.

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3.4. Electrochemical Analysis

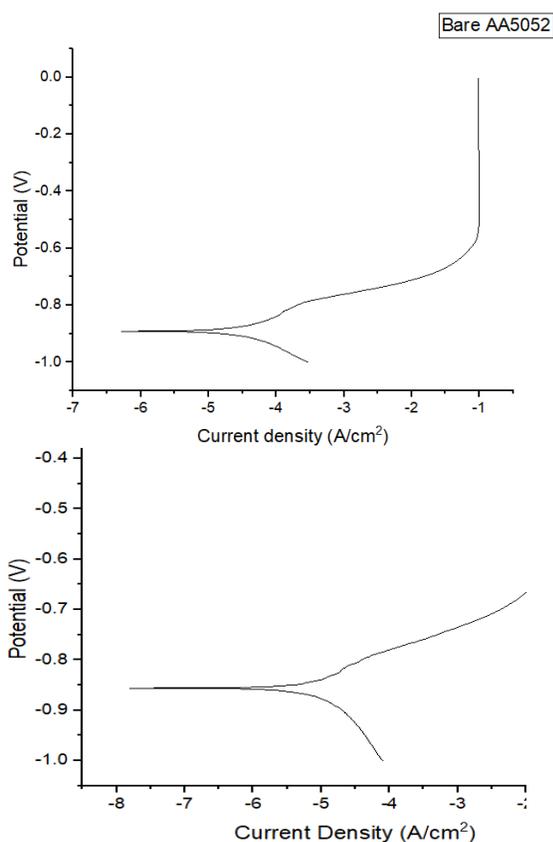


Fig. 4 Electrochemical analysis of the (a) bare and (b) ZrO₂-TiO₂ coated AA5052 Substrate

The electrochemical examination of uncoated and coated aluminum substrates is shown in the abovementioned figures. Using the potentiodynamic polarization approach, the corrosion resistance behavior of uncoated and coated AA5052 substrates was investigated. The electrolyte was a solution of 3.5% sodium chloride. Measurements of potentiodynamic polarization were made at the open circuit potential. At a potential scan rate of 100 mV/min, potentiodynamic polarization measurements were performed. Tafel extrapolation was used to calculate the corrosion potential (E_{corr}) and corrosion current density (i_{corr}) using a potential scan rate of -250mv to 250mv.

In a solution of 3.5% sodium chloride, the bare AA5052 substrate's E_{corr} and i_{corr} values were discovered to be 891 mV and 0.0038 mA/cm², respectively. The E_{corr} and i_{corr} values for the coated AA5052 substrate were -856 mV and 0.0030 mA/cm², respectively. Indicating an increase in the corrosion resistance property, the E_{corr} and i_{corr} values for bare and coated AA5052 substrates reduced from 891 mV to 856 mV and from 0.0038 mA/cm² to 0.0030 mA/cm², respectively.

4. Conclusion

On the AA5052 Substrate, spin coating and chemical deposition techniques successfully created multi-layered ZrO₂-TiO₂ nanostructured coatings. The many peaks demonstrated the polycrystalline nature of the coating at various intensities in the XRD results. The highest peak that could be measured was 40.5° with 29327.72 intensity. According to SEM images, the coatings were non-uniform, non-homogeneous, and contained some particle agglomerations. The elemental composition of the coated and

bare substrate was identified by EDX analysis. Aluminium, titanium, zirconium, and oxygen were all present in the coated substrate, with corresponding weight percentages of 17.1, 61.8, 7.8, and 13.3.

Ecorr and Icorr values for bare and coated AA5052 substrates reduced from 891 mV to 856 mV and from 0.0038 mA/cm² to 0.0030 mA/cm², respectively, electrochemical analysis, demonstrating a minor improvement in the substrate's corrosion resistance capability.

References

- [1] Fatma Aydin Unal, Semih Ok, Murat Unal, Sebahat Topal, Kemal Cellat, Fatih Şen, *Journal of Molecular Liquids*, no. 19, pp. 36108-2, 2019
- [2] Veanuga Ganapathy, Tedi Kurniawan, H Mas Ayu, Yuli Panca Asmara Rosdi Daud, Niki Prastomo and Asep Bayu Dani Nandiyanto, *Journal of Engineering Science and Technology*, vol. 13, no. 6, pp. 1713-1721, 2018
- [3] Y Joshua Dua, Matt Damrona, Grace Tang a, Haixing Zheng a, C J Chua and Joseph H. Osborne B, *Progress in Organic Coatings*, vol. 41, pp. 226–232, 2001
- [4] R Di Maggio, L Fedrizzi, S Rossi and P Scardi, *Thin Solid Films*, vol. 286, pp. 127-35, 1996
- [5] Ivana Bacic, Helena Otmacic Curkovic, Lidija Curkovic, Vilko Mandic and Zrinka, *International Journal of Electrochemical Science*, vol. 11, pp. 9192 – 9205, 2016.
- [6] V R Capelossi, M Poelman, I Recloux, R P B Hernandez, H G de Melo and M G Olivier, *Electrochimica Acta*, vol. 124, pp. 69- 79, 2014
- [7] Duhua Wang and Gordon P. Bierwagen, *Progress in Organic Coatings*, vol. 64, pp. 327-338, 2009.
- [8] A N Khramova, N N Voevodinb, V N Balbysheva and R A Mantzc, *Thin Solid Films*, vol. 483, pp. 191-196, 2005.
- [9] M L Zheludkevich, R Serraa, M F Montemor, I M Miranda Salvado, and M G S Ferreira, *Surface and Coatings Technology*, vol. 200, pp. 3084-3094, 2006.
- [10] Gilmar P Thim, Maria A S Oliveira, Evandro D A Oliveira and Francisco C L Melo, *Journal of Non-Crystalline Solids*, vol. 273, pp. 124-128, 2000.
- [11] Hehong Zhang, Xiaofeng Zhang, Xuhui Zhao, Yuming Tang and Yu Zuo, *Coatings*, vol. 8, no. 11, pp. 397, 2018.
- [12] F Andreatt, L Paussa, A Lanzuttia, N C Rosero Navarro, M Aparicio, Y Castro A.Duran, D Ondratschek and L Fedrizzia, *Progress in Organic Coatings*, vol. 72, pp. 3-14, 2011.
- [13] Armaghan Naderi, Abolghasem Dolati, Abdollah Afshar and Genevieve Palardy, *Materials Chemistry and Physics*, vol. 122696, 2006.
- [14] M. A. M. Halmi, M. A. Harimon, A. E. Ismail, D. Chue, "Fatigue Behaviour of High-Velocity Oxy-Fuel Coatings on Medium Carbon Steel," *International Journal of Engineering Trends and Technology*, vol. 69, no. 6, pp. 56-70, 2021. Crossref, <https://doi.org/10.14445/22315381/IJETT-V69I6P209>
- [15] F Andreatta, L Paussa, P Aldighieri, A Lanzutti, D Ondratschek and L Fedrizzi, *Aluminium Surface Science & Technology*, vol. 42, pp. 293-298, 2009.
- [16] E A G Shillington and D R Clarke, *Acta Materialia*, vol. 47, pp. 1297-1305, 1999.
- [17] Xiaolong Chen, Yanfei Zhang, Xinhua Zhong, Zhenhua Xu, Jiangfeng Zhang, Yongliang Cheng, Yu Zhao, Yangjia Liu, Xizhi Fan, Ying Wang, Hongmei Ma, Xueqiang Cao, *Journal of the European Ceramic Society*, vol. 30, 1649–1657, 2010.
- [18] E.P. Busso, H.E. Evans b, Z.Q. Qian C, M.P. Taylor, *Acta Materialia*, vol. 58, pp. 1242– 1251, 2010.
- [19] Seema , Sunil Rohilla , Sonia Arora , Saneh Lata , Anita Yadav , Preeti kaushik, "To Study the Structural Characterization of the Polyaniline/CoFe₂O₄ Nanocomposites," *SSRG International Journal of Applied Physics*, vol. 9, no. 2, pp. 1-6, 2022. Crossref, <https://doi.org/10.14445/23500301/IJAP-V9I2P101>
- [20] D. D. Hass, Y. Y. Yang, H. N. G. Wadley, *Journal of Porous Materials*, vol. 17, pp. 27–38, 2010.
- [21] SUN Jie, ZHANG Lili, ZHAO Dan, *Journal of Rare Earths*, Special Issue, vol. 28, pp. 198, 2010.
- [22] Matthew R. Begley and Haydn N.G. Wadley, *The American Ceramic Society [SI]*, pp. S96–S103, 2011.
- [23] Sorina Iliina, Gheorghe Ionescu, Victor Manoliu, Radu Robert Piticescu, *Incas Bulletin*, vol. 3, no. 3, 2011.
- [24] Zhuo Yu, Hengbei Zhao, and Haydn N. G. Wadley, *Journal of The American Ceramic Society*, vol. 94, no. 8, 2011.
- [25] Cody H. Nguyena, K. Chandrashekhara B, Victor Birman, *Mechanics Research Communications*, vol. 39, pp. 35–43, 2012.
- [26] Renata Batista Rivero Garcia, Fábio Santos da Silva and Elizabete Yoshie Kawachi, *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, vol. 436, pp. 484-488, 2013.
- [27] F Rovere, D Music, JM Schneider, PH Mayrhofer, *Acta Materialia*, vol. 58, no. 7, pp. 2708-2715, 2010.