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Original Research

Protective Coatings for Zirconium-Based Alloys: Improving their Mechanical and Biological Properties by YSZ Coating

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ABSTRACT

Zirconium is widely utilized in biomedical implants because of its exceptional properties, including corrosion resistance, wear behavior, and biocompatibility. However, these implants might have certain unfavorable effects since they lack surface features. Therefore, it is essential to establish the surface properties required for zirconium implants. Particularly, yttria-stabilized zirconia has demonstrated potential in controlling the biological and mechanical reactions to Zr. A zirconium 705 substrate was coated, ceramic coatings were developed using spray-grade yttria-stabilized zirconia (8YSZ), synthesized via the sol-gel method. The layer was seen using XRD, a scanning microscope and EDS mapping process which displays the spatial distribution of Zr, O, and Y in the 8YSZ layer; The zirconium alloy coated layer exhibited a 64% decrease in the coefficient of friction, a 41.3% improvement in hardness, and a 56% reduction in wear rate compared to the uncoated alloy.

INTRODUCTION

Zr and its alloys are well known for being biocompatible metal implants due to their self-regulating oxide covering, which limits ion release and shields the surface from corrosion. The integration of these essential components into the body requires surface modification (Salman et al., 2021). The choice of metal for an implant is mainly determined by its specific medical application. To ensure long-term performance and avoid rejection, the metal must exhibit key properties such as biocompatibility, durability, and resistance to corrosion. These qualities include exceptional biocompatibility, high wear and corrosion resistance, acceptable mechanical capabilities, ductility, and high hardness (Niinomi et al., 2015). Many thin film deposition techniques are used to improve the surface characteristics of biomaterials and to encourage their biocompatibility. There are studies on atoms condensing as thin layers on crystalline substrates from the vapor phase. The sputtering procedure produces dense, sticky films with the precise elemental composition among the several methods for creating thin layers . Despite the strong corrosion resistance of Zr and related alloys, its limited bioactivity has prevented them from being widely used in dental and surgical applications. One potential solution to this problem is to coat the surface of Zr-based products with a biocompatible layer, and Bioinspired surface modification on zirconia enhances osteoblastic cell responsiveness (Liu et al., 2013). Zr materials have attracted a lot of attention lately. Any biomaterial that is to be used in specific biomedical applications must have surface characteristics found in the environment of the human body (Salman et al., 2021), (Niinomi et al., 2015). Excellent surface performance is essential to prevent significant deterioration of Zr implants and to ensure their life in biological contexts. The concurrent growth of a thin layer on the surface of Zr is responsible for the surface behavior of Zr observed in a human body fluid. Therefore, one of the most important steps in obtaining outstanding surface qualities is to optimize and change the natural oxide layer on the surface (Salman et al., 2022). The complex process of choosing a coating technology is mostly dictated by the component's capabilities and budgetary restrictions. To properly balance the technical and financial viability of coating processes as well as the process's compatibility with the substrate, modern design procedures take the surface into consideration from the beginning. On the other

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hand, coating a component is still commonly utilized to enhance its properties after it has already happened. The constituents of the coating material might seep into the substrate, increasing wear resistance even after the coating-substrate contact has been broken. The coating should have a higher wear resistance than the substrate. Additionally, by lowering the friction coefficient and contact temperature, the coating lessens the chance of severe adhesive wear. Because the coating has a lower thermal conductivity than the substrate, it acts as a heat barrier. Cutting force affects cutting tool performance; cutting tool coated by multi-layer (TiO2/Al2O3/ZrO2) provide lower cutting forces than uncoated HSS tools. Unlike the conventional method, which uses a high temperature, the sol-gel coating method uses a moderate temperature. The materials produced by sol-gel synthesis are very homogeneous and pure when compared to other techniques. Zirconia stabilized with yttria, improving its tribological and thermal characteristics, and yields materials that are more homogeneous and pure than those generated with other methods (Asaad et al., 2022). Kareem et al. (2024) improvement tribological and thermal properties for cutting tool coated Zirconia stabilized with yttria nano coating using the sol–gel technique. The objective of this study is to improve the mechanical and biological properties of zirconium alloy surfaces by applying a nano coating using the sol-gel and plasma thermal spray processes.

MATERIALS AND METHOD

Zircadyne 705 alloy samples measuring 13 diameter by 4 thickness mm2 were used in this study. The chemical composition of the samples was %C 0.05, %H 0.005, %O 0.17, %Hf4.5, %N0.025, Nb0.054, and %Fe+Cr 0.2. Following a 20-minute ultrasonic cleaning in a solution of acetone and distilled water, all zirconium samples were ground using silicon carbide abrasive sheets (400, 600, and 1000 grit) and allowed to dry in the cold air.

Plasma thermal spray- sol gel

Ceramic coatings were developed using spray-grade yttria-stabilized zirconia (8YSZ), which was synthesized using sol-gel. An 8YSZ layer was applied to the sample. Utilizing the sol-gel technique, YSZ was prepared by first creating a solution with zirconium n-propoxide (ZNP) , Yttria-stabilized zirconia (YSZ) coatings can be prepared by the sol-gel method through hydrolyzing and condensing the zirconium alkox¬ide (Zr(OC3H7)4) and yttrium alkoxide (yttrium II tris isopropoxide) using ethanol as a solvent, as the source precursor from SIGMA-ALORICH CHEMIE GmbH, product of USA Group . The solution was constantly agitated at 100 rpm in an ice bath while being ultrasonically sonicated. The molar ratios for the ethanol, isopropanol, and water in each of these solutions were 10:1, 6.6:1, and 7:1, respectively. After the sample coated with Y(OH)3 and Zr(OH)4 reached a pH of 4.43, it was submerged in this solution for seven minutes. Samples were then air-dried for 48 hours at ambient temperature and then calcined for three hours at 700°C, with a 3 °C/min rate (Asaad et al., 2023). The matching gel powder was calcined at 1000 °C to generate oxide particles. Plasma spraying was carried out using a semi-automated atmospheric plasma spraying (APS) system from Metco Inc., Westbury, L.I., New York. The coating powders nano yttria stabilized zirconia were mixed using a ball mill for 60 minutes, employing tungsten carbide balls with diameters ranging from 3 to 5 mm. A bond coat was first applied to the surface of the specimens. To achieve an optimal composite coating thickness using DC current, the process was optimized with an applied voltage of 35 V, which regulated spray speed and heat input. The powder feed rate was set at 5 g/min, and the spray distance was maintained at 150 mm. The ceramic materials and the substrate alloy were melted by the plasma produced by ionizing the argon that was present between the anode and the cathode.

Metallurgical Examination

X-ray diffraction XRD was used to identify the for the uncoated substrate ,sample coated with 8YSZ layer at a speed of 3 degrees per minute sample and 3 degrees per minute for thin layer. When compared to the diffraction data card (00 -005 - 0665), XRD of the substrate with the help of EDX analysis reveals the elemental distribution to estimate the phases. X-Ray instrument type (XRD), Shimadzu manufacturer, XRD dfractometer-XRD-7000, volt 40 kV, current 30mAmp, tube Cu, at Phi Nanoscience Center, Baghdad, Iraq was used. The detector was moved during an angles of 2θ = 20 to 80º degrees. As shown Fig.1.

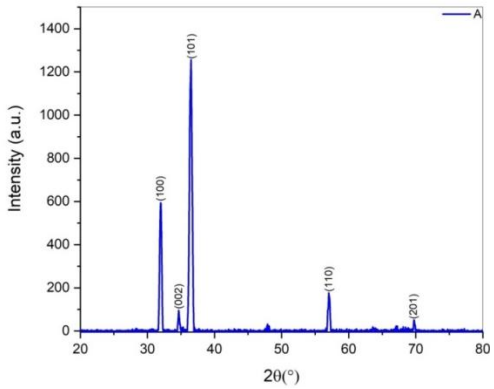


Fig. 1 X-Ray diffraction for zirconium

Coatings' microstructure analysis was measured. The F50 instrument, produced in the Netherlands, was utilized at Nano Lab using a field emission scanning electron microscope (FESEM) in Al-Khora Enterprise. An Electron Dispersive Spectroscopy (EDS) detector was installed in the FESEM to identify Y, Zr, and O elements in addition to other elements.

Hardness and wear test

Vickers hardness was used to test the coatings' hardness sample with 13 diameter by 4 thickness mm2 using a load of 50 g for ten seconds, in accordance with ASTM E384 standard. The hardness test was carried out using a digital Micro Vickers hardness tester (TH717), a micro Vickers device featuring a square base and a diamond pyramid. The University of Babylon/College of Materials Engineering hosted the test. The average of three measurements was used to record the hardness value for each. According to ASTMG9, this test was performed using the pin-on-disc technique using Type MT4003 version 10.0 control and data collection software for friction and wear testing. The experiment was run with a 15N load at room temperature (32°C) at 30-minute intervals. The sliding distance was 264 mm, and the sliding speed was 350 rpm. A ball that was

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pressed up against the rotating disc served as the pin. A ball of martensitic steel with a hardness of 990 HV was utilized to assess the cutting insert's wear. The wear tests were conducted on both coated and uncoated inserts. To determine the weight loss, the specimens were accurately weighed before and after the test using a sensitive scale.

Biological Test

Muller-Hinton (M-H) prepared by adding 20 mL of the powder into 1 L distilled water and then heated on a burner with shaking. M-H must be autoclaved for 15 minutes at 121°C to be sterilized. Then it was allowed to cool to 50 °C before pouring into a petri dish and leaving for about 15 minutes for solidification before flipping upside down and storing in the refrigerator at 4 °C.

The antibacterial potential of the prepared Samples (Zr, YSZ NPs) was investigated against Gram's negative and Gram's positive bacterial strains using agar well diffusion assay (Bahjat et al., 2021; Kashan et al., 2020). About 20mL of on Muller–Hinton (MH) agar was aseptically poured into sterile Petri dishes. The bacterial species were collected from their stock cultures using a sterile wire loop (Kashan et al., 2021). After culturing the organisms, 6 mm-diameter wells were bored on the agar plates using of a sterile tip. Into the bored wells, different concentrations of the Samples (Zr, YSZ NPs) were used. The cultured plates containing the Samples (Zr, YSZ NPs) and the test organisms were incubated overnight at 37°C before measuring and recording the average the zones of inhibition diameter (Jihad et al., 2021; Mohammed et al., 2020). Data were statically analysis using Graphpad prism program (Ali et al., 2018). Data are represented as mean ± SD of three experiments. Indicate statistically significant difference at $p<0.05$ (Younus et al., 2019) and (Jabir et al., 2022).

RESULT AND DISCUSSION

The results indicate that multi diffraction peaks of YO1.458 and ZrO2 which matches with the standard JCPDS No. of 00-039-1063 and 01-088-1007, as shown Fig. 2.

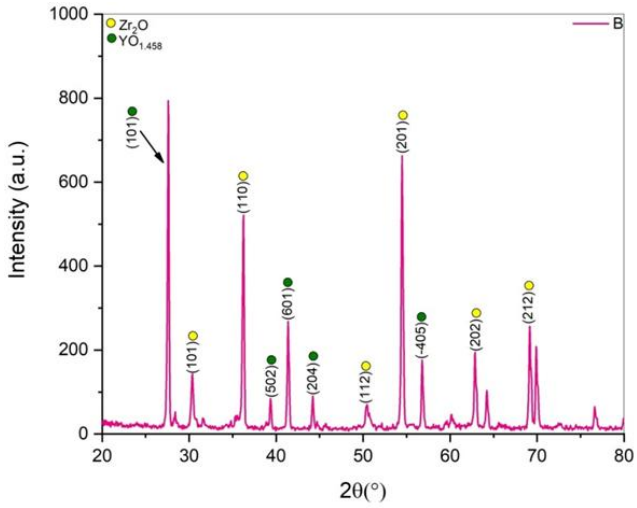


Fig. 2 X-ray Diffraction for thin layer 8YSZ

Electron Scanning The morphology of the coated sample 8YSZ surfaces is depicted in Fig.3, where in agglomeration circular particles are growing. The results of the EDS mapping process are seen in Fig.4, which displays the spatial distribution of Zr, O, and Y in the 8YSZ layer. The values of each element confirmation through Table 1.

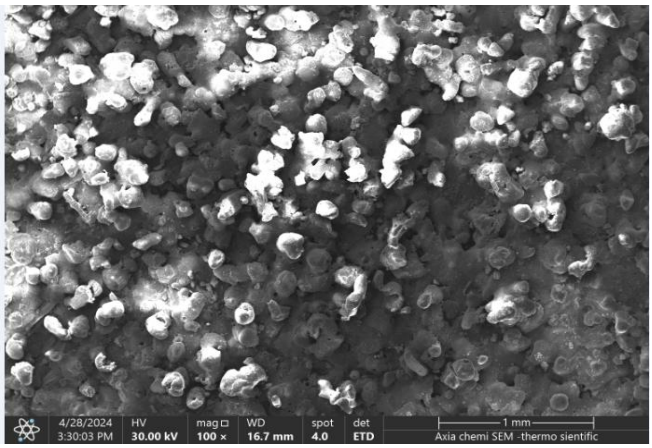
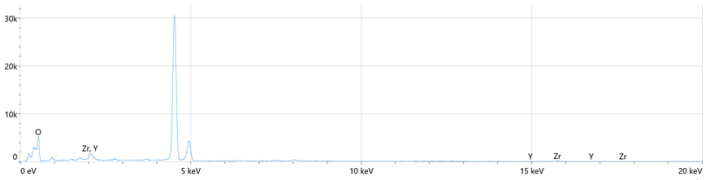


Fig. 3 FESEM micrograph of 8YSZ based coatings

Table 1 Weight % for each element of coating layer

Element	Atomic %	Atomic % Error	Weight %	Weight % Error
O	94.4	0.9	74.8	0.7
Y	0.4	0.2	1.7	1.1
Zr	5.2	0.3	23.6	1.5



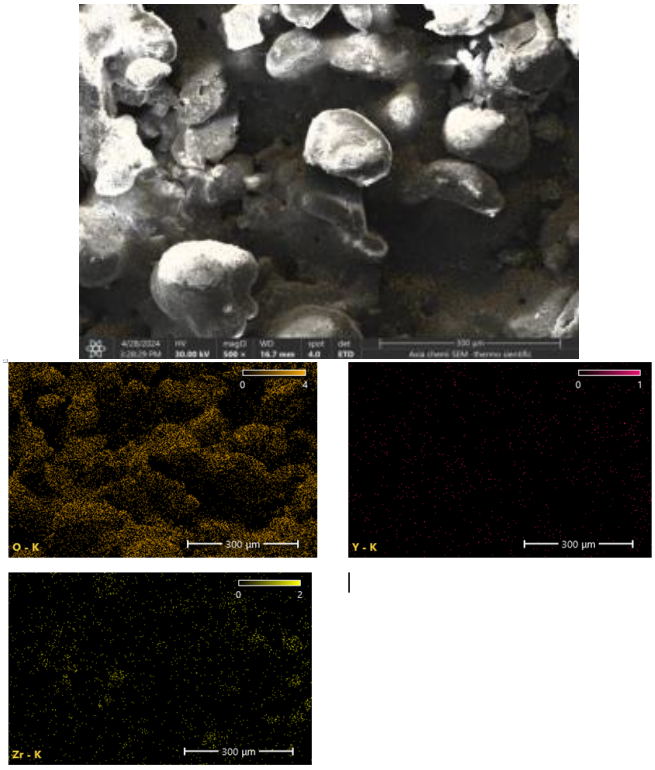


Fig. 4 EDS mapping of prepared cross sectional coated sample

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The results from surface micro hardness of the substrate and coated sample are shown in Fig. 5. Coated alloy have the ability to improve a surface's micro hardness. These results can be attributed to the good adherence and uniform distribution of ceramic oxides YSZ deposited on the substrate.

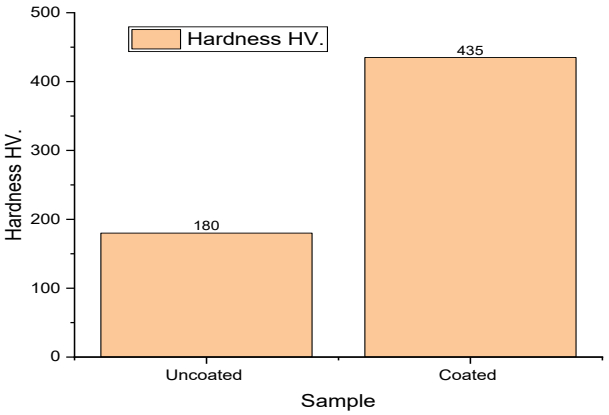


Fig. 5 Hardness test for coated and non-coated samples

For 30 minutes, the impact of the YSZ coating on wear rate was investigated at a load of 15N. As illustrated in Fig.6, coated samples with YSZ layer essentially have the lower rate of wear compared to uncoated sample due to high hardness of zirconia stabilized yettira coatings. As seen in Fig. 7, the added coefficient of friction for each sample that is coated and uncoated indicates which pertains to an improvement in friction properties.

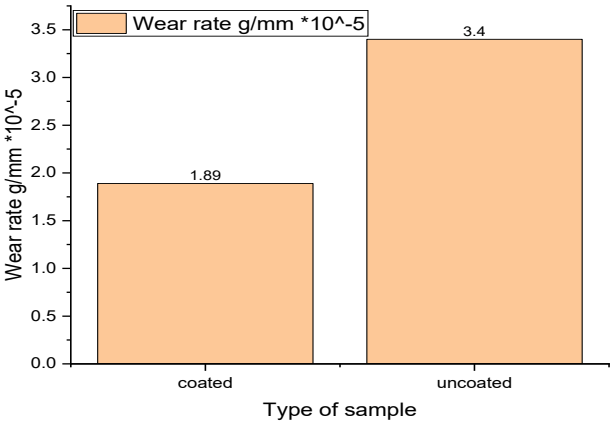


Fig. 6 Test of wear rate for both coated and untreated samples

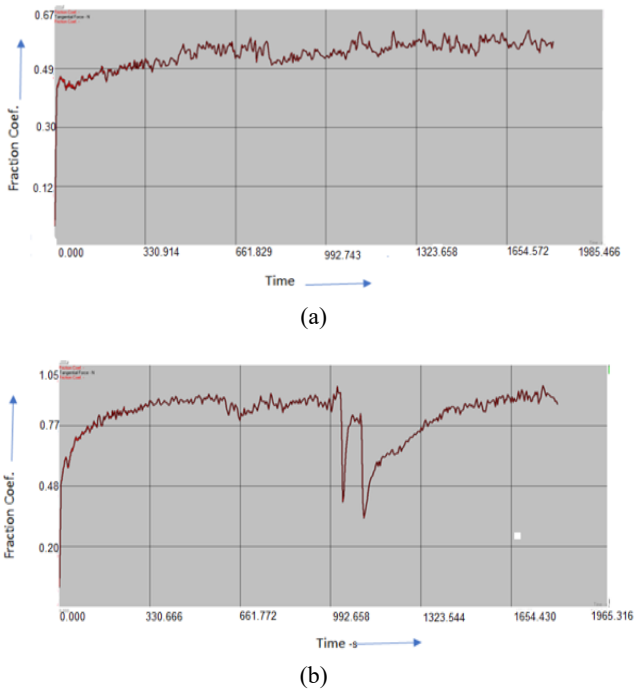


Fig. 7 Coefficient of friction for (a) coated sample, (b) uncoated sample

All result of antibacterial activity with different concentration with 250gm from bacteria shown by the figures below all details explained by table 2, and Fig. 8. Can be noticed the ability of the YSZ coating to stop the growth of bacteria E. coli can be improved.

Table 2 Explain the antibacterial activity of nanoparticles.

Antibacterial analysis (Zone of inhibition (mm))			
Sample	A	B	C
E.coli	6	8	15

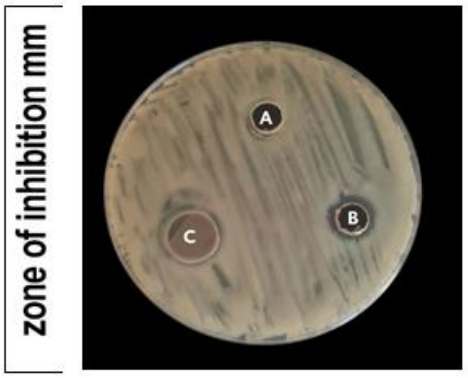


Fig. 8 Antibacterial activity of (test material) activity against E.coli. A, Control. B, Zr. C,YSZ.

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CONCLUSION

In conclusion, the zirconium alloy was coated with nano coating YSZ via a plasma thermal spray technique. Applying a YSZ layer gave the Zr alloy antimicrobial characteristics. The coated sample's biological test analysis showed that the coating YSZ has excellent antibacterial activity against E-coli. coli. The layer coated zirconium alloy showed 64% reduction in friction coefficient, 41.3% increase in hardness, and 56% reduction in wear rate when compared to the uncoated alloy.

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