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Article

Surface roughness assessment of zinc oxide nanostructures formed on biomedical steel

Evaluación de la rugosidad superficial de nanoestructuras de óxido de zinc formadas en acero biomédico

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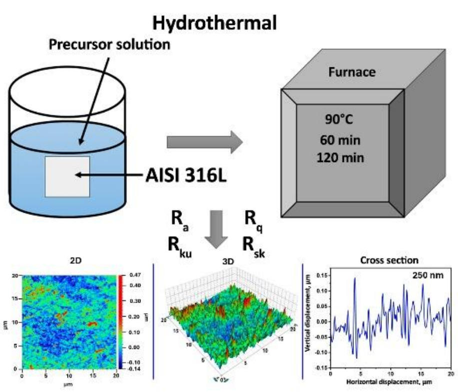
Abstract

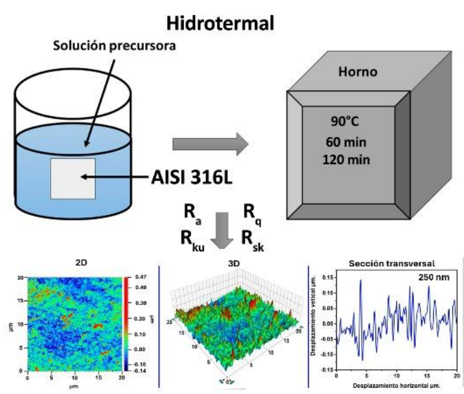
This investigation explores the effect of hydrothermal time on the topographical characteristics of zinc oxide nanostructures-based coating formed on AISI 316L steel. First, a Zn adhesion layer was deposited and later a ZnO film. Next, the ZnO nanostructures were grown by the hydrothermal method at a temperature of 90 °C for 60 and 120 minutes. Elemental analysis was conducted at the surface and its topographic characteristics were assessed by atomic force microscopy. After images acquisition of the surface of the samples, arithmetic roughness (Ra), root mean square roughness (Rq), kurtosis (Rk) and skewness (Rsk) were determined. The arithmetic roughness Ra varied from ~26 nm to ~41 nm when hydrothermal time increased from 60 to 120 min, whereas Rq increased from ~35 nm to ~53 nm. Regarding the kurtosis and skewness parameters, when varying the hydrothermal time, a decrease in their values was found.



Nanostructures, Atomic force microscopy, Topographical analysis

Resumen

Este trabajo explora el efecto del tiempo de hidrotermal en las características superficiales de nanoestructuras de ZnO formadas en un acero AISI 316L. Primero, se depositó una película de adhesión de zinc, y posteriormente una película de óxido de zinc. Después se formaron nanoestructuras de ZnO mediante el método hidrotermal a una temperatura de 90 °C con tiempos de 60 y 120 minutos. Se realizó un análisis elemental en la superficie y sus características se evaluaron mediante microscopía de fuerza atómica. Luego se determinaron la rugosidad aritmética (Ra), rugosidad media cuadrática (Rq), curtosis (Rku) y sesgo (Rsk). La rugosidad Ra varió de ~26 nm a ~41 nm al incrementar el tiempo de 60 a 120 min; mientras que Rq aumentó de ~35 nm a ~53 nm. En cuanto a los parámetros de curtosis y el sesgo, cuando se varió el tiempo de hidrotermal, se encontró una disminución en sus valores.



Nanoestructuras, Microscopio de fuerza atómica, Análisis topográfico

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Introduction

The AISI 316L biomedical steel is employed for different implant devices, its low cost and good properties make it suitable for this purpose. Among different devices, this steel can be used for dental implants (Saha & Roy, 2023).

The surface properties of dental implants play a crucial role in osseointegration and adhesion of bacteria. Although bacterial adhesion is influenced by multiple factors, surface roughness properties are a significant characteristic regarding biofilm formation. While there is no consensus on whether bacterial adhesion has a positive, negative, or neutral effect, it is widely recognized that rougher surfaces facilitate bacterial attachment due to the increased surface area. One strategy to mitigate bacterial infections involves enhancing the metal surface properties with antibacterial properties. For this purpose, different surface engineering methods have been proposed, including using zinc oxide nanostructures growth by the hydrothermal method (Tam et al., 2008).

The term hydrothermal originated from the discovery of changes in the minerals and rocks of the Earth's crust due to the action of water, appropriate temperature, and pressure. There are three hydrothermal synthesis methods: isothermal, used in processes where high transport efficiency is not required, so there is no change in temperature; temperature gradient, used for crystal growth, in this method, the reaction products are transported from higher solubility to lower solubility; and finally, the temperature decrease method, where the desired phase is saturated at a high temperature and precipitated by cooling (Zhang et al., 2012).

Different variables, such as temperature, reaction time, and seed layer properties, influence the characteristics of the nanostructures formed by the hydrothermal method. Therefore, the motivation of this work was to investigate the influence of the reaction time on the surface characteristics of the nanostructures formed, constantly maintaining seed layer and temperature. The assessment of the surface features was conducted by atomic force microscopy.

This work contributes to enhancing the knowledge about zinc oxide synthesis methods and how hydrothermal time influences surface roughness, which is relevant for the potential use of a zinc oxide-based coating for dental implants.

Methodology

The preparation of the 316L biomedical stainless-steel substrates began by cutting circular sections (25.4 mm in diameter and 5 mm in thickness), then using SiC abrasive paper of different grit sizes up to 2000, and finally polishing them to mirror finish with 1 micron diamond paste.

The deposition of the ZnO seed layer was obtained by a physical vapor deposition process, using the reactor chamber depicted in Fig. 1. In the first stage, an adhesion layer of zinc was deposited using a 99.99% pure Zn target, and argon as the working gas with a flow rate of 20 sccm and working pressure of 2 bar. The ZnO seed layer was then deposited on top of the adhesion layer using oxygen with a flow rate of 5 sccm, a deposition time of 50 seconds, and a power of 30 watts.

Box 1

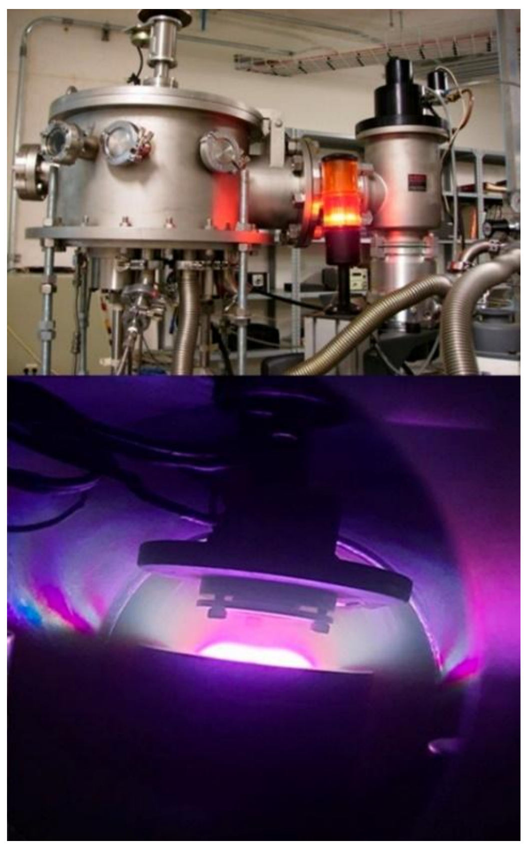

Figure 1
Reactor chamber used for seed layer deposition.

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The synthesis of the ZnO nanostructures was performed using the technique employed by Melo-Máximo & Velázquez-Jiménez (2020), in which hexamethylenetetramine dissolved in deionized water in a 1:1 solution and zinc nitrate diluted in deionized water in a 1:1 solution. These were mixed, and the substrates previously coated with the ZnO seed by PVD were immersed in the solution and later placed into the furnace at 90 °C and maintained for 60 and 120 minutes (Fig. 2). With this setup, two conditions were obtained, with the identification given in Table 1.

Box 2

Table 1

Nomenclature of the samples

Sample	Zn adhesion	ZnO film	Hydrothermal time
S60	20 s	50 s	1 h
S120			2 h

Box 3

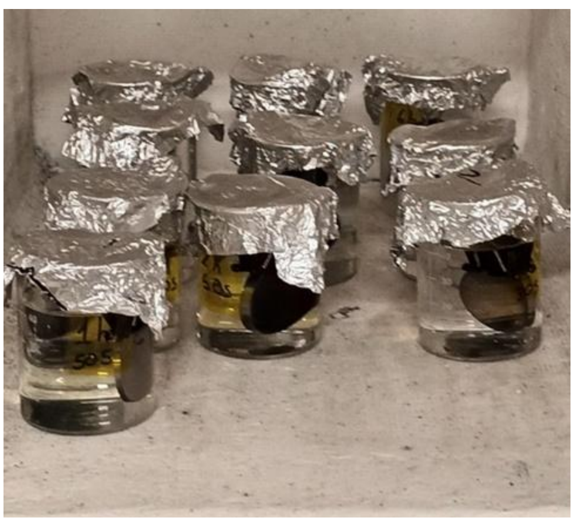

Figure 2

Samples placed inside the furnace for the hydrothermal method

Energy dispersive spectroscopy was used to analyze the surface of the samples and investigate the composition of the nanostructured coating. Later, topographic characteristics were studied by atomic force microscopy (AFM), conducting scanning in tapping mode. Each sample was scanned in 5 different areas with a scan size of 20×20 µm². Next, Gwyddion software was used to obtain the following amplitude parameters: arithmetic roughness (R_a), root mean square roughness (R_q), skewness (R_k), and kurtosis (R_{ku}).

Results

Fig. 3 presents the elemental composition of both S60 and S120 samples. It can be seen that the presence of zinc and oxygen was confirmed, along with iron from the substrate steel. Similar intensities were registered for both conditions.

Box 4

a)

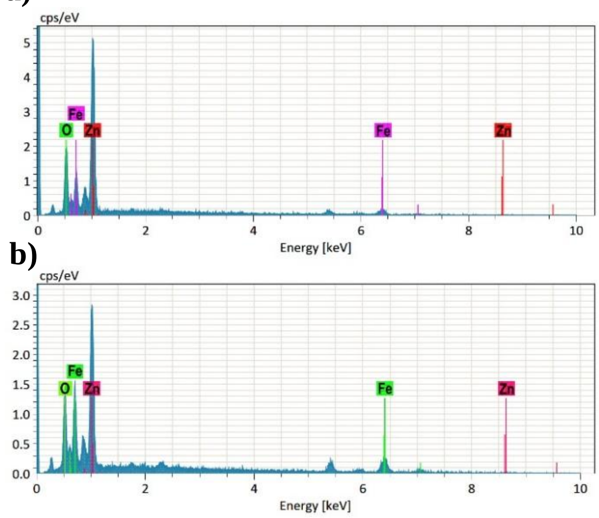

Figure 3

Elemental composition on the surface a) S60 sample; b) S120 sample

Fig. 4 and Fig. 5 show the 2D and 3D AFM images of the S60 and S120 samples, respectively. The 2D images show a height variation between 0.11 and 0.45 µm for S60, whereas this variation goes from 0.14 to 0.47 µm in S120. In the height side palettes, red means a higher area was observed, which, in this particular case, could mean the height of nanostructures formed by the hydrothermal method. In both samples, there are areas where these heights are registered, concluding then that the synthesis procedure allowed the formation of nanostructures. Regarding the darkest blue areas, corresponding to the lowest areas, the S120 sample presents large amounts of it.

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Box 5

a)

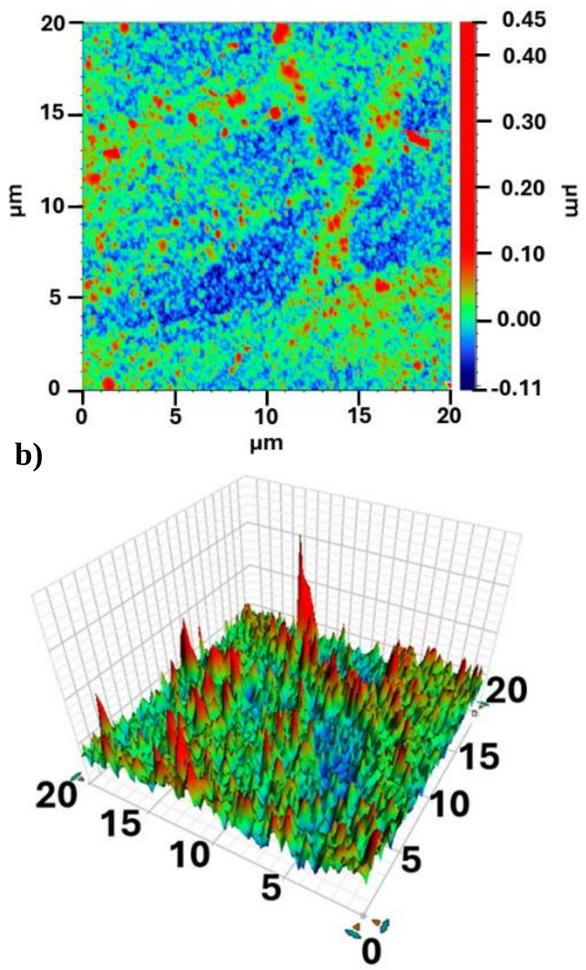

Figure 4
AFM images of S60 sample a) 2D view; b) 3D view.

Box 6

a)

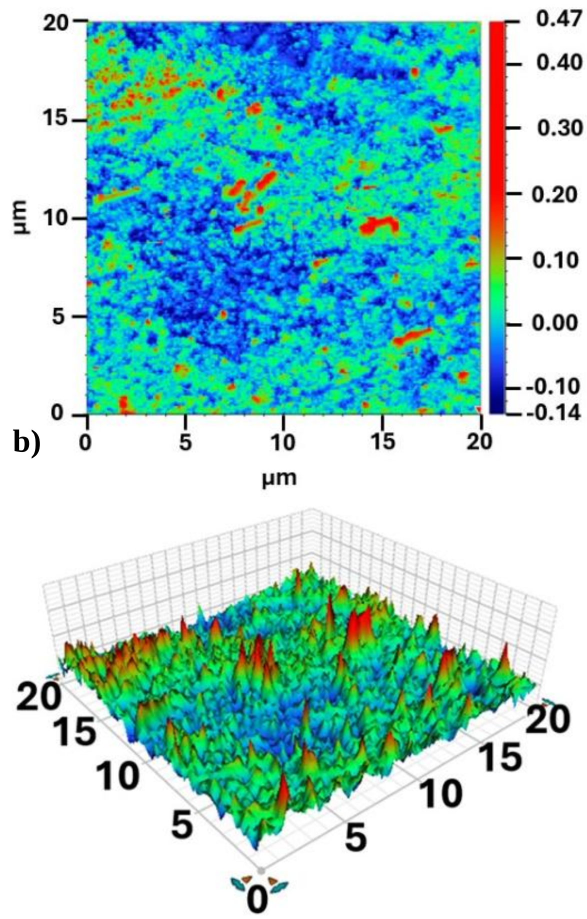

Figure 5
AFM images of S120 sample a) 2D view; b) 3D view.

In the representative cross-section plots depicted in Figure 6, it can be seen that the surface of the samples presents clear peaks and valleys along the depth of the analyzed line. A similar range of values was registered for both samples, reaching a peak height of around 0.15 µm, although S120 reached a slightly lower value of around -0.12 µm. From both AFM images and cross-section plots, it can be noticed that an increase in reaction time from 60 to 120 minutes affected the surface roughness. This can be associated with the more extended growth period; during the procedure, various processes occur, such as nucleation, growth, and aggregation. Therefore, a more extended period allows for more accumulation of particles and larger formation of structures; all these processes could impact the surface characteristics changes.

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Box 7

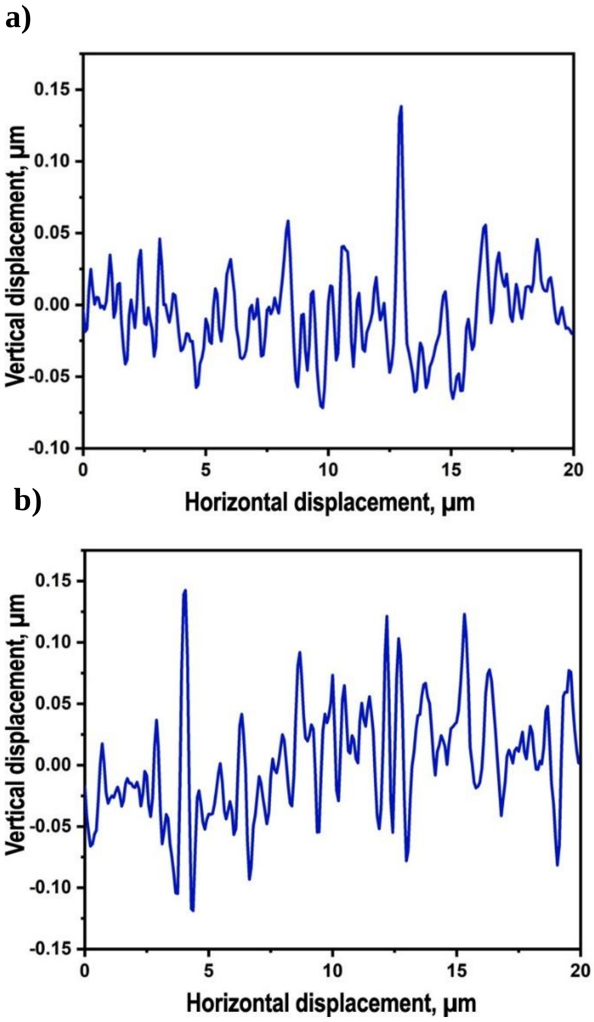

Figure 6

Cross-section plots along horizontal axis a) S60 sample; b) S120 sample.

Table 2 gathers all the surface parameters extracted from the AFM images. It can be seen that an increase in surface roughness was obtained when extending the reaction time in the hydrothermal method. When the time was 60 minutes, arithmetic and root mean square roughness were 26.811 and 35.188, respectively. However, when two-fold time was used, these values increased to R_a = 41.237 nm and R_q = 53.945 nm.

Furthermore, the amplitude parameters skewness (R_{sk}) and kurtosis (R_{ku}) are also presented in Table 2. Skewness indicates the symmetry of the surface profile relative to the mean line. Profiles with removed peaks or deep scratches exhibit negative R_{sk}, while those with filled valleys or prominent peaks show positive R_{sk}.

On the other hand, Kurtosis represents the sharpness of the probability density distribution of the profile. Profiles with a few high peaks and low valleys have R_{ku} values less than 3, whereas profiles with numerous high peaks and deep valleys have R_{ku} values greater than 3. A balanced profile features an R_{sk} of zero and an R_{ku} of 3 (Gadelmawla et al., 2002). Considering this, S60 presents a R_{ku} of 7.552, corresponding to a surface with numerous peaks and low valleys, whereas its positive R_{sk} value means that the surface is comprised of filled or extended valleys and high peaks. Moreover, for the S120 sample, both amplitude parameters R_{ku} and R_{sk} describe similar information since its R_{ku} > 3 and has a positive R_{sk}.

Box 8

Table 2

Surface roughness results

Parameter	S60	S120
R_a, nm	26.811	41.237
R_q, nm	35.188	53.945
R_{ku}	7.552	4.819
R_{sk}	1.005	0.706

Conclusions

In this work, the influence of the hydrothermal time was investigated. First, a seed zinc oxide layer was deposited by sputtering. Later, the hydrothermal method was used for growing zing oxide nanostructures, and two reaction times were established: 60 and 120 minutes. Through elemental analysis, the presence of zinc and oxygen elements was confirmed. The AFM results revealed that arithmetic and root mean square roughness increased around 50 % when hydrothermal time changed from 60 to 120 minutes.

Conflict of interest

The authors declare no interest conflict. They have no known competing financial interests or personal relationships that could have appeared to influence the article reported in this article.

Authors' Contribution

The contribution of each researcher in each of the points developed in this research, was defined based on:

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Estrada Martínez Fortino Fabián: He conducted the synthesis of ZnO nanostructures (PVD and hydrothermal method) and worked on data formatting and writing original draft.

Melo Máximo Dulce Viridiana: Contributed to the project idea and methodology of ZnO nanostructures synthesis.

Arzate Vázquez Israel: Conducted the AFM tests and guided the formal analysis.

Vega Morón Roberto Carlos: He contributed with the project idea, resources and writing, review & editing of the manuscript.

Availability of data and materials

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Abbreviations

AFM	Atomic Force Microscopy
AISI	American Iron and Steel Institute
PVD	Physical Vapor Deposition

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