Effect of deposition time on the zinc nanowires growth on steel substrates

Efecto del tiempo de deposición en el crecimiento de nanocables de óxido de zinc sobre sustratos de acero

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DOI: 10.35429/JTD.2022.17.6.25.29 Received January 30, 2022; Accepted June 30, 2022

Abstract

Zinc oxide nanowires have gained great research interest for their unique and diverse properties, mainly due to their surface reaction and surface/volume ratio. The present work analyzes the effect of the deposition time of a zinc oxide seed by physical vapor deposition assisted by plasma and the growth time of zinc oxide nanowires by hydrothermal method. The samples were characterized by scanning electron microscopy, and the contact angle was measured to verify their hydrophobic or hydrophilic properties. With the characterization, a higher angle of 103.493° and a lower angle of 14.006° were obtained, due to the shape and size of the nanostructures and how encapsulates the air.

PVD, Zinc oxide, Hydrothermal method

Resumen

Los nanocables de óxido de zinc han ganado gran interés dentro del campo de investigación por sus propiedades únicas y diversas, debidas en su mayoría a su reacción superficial y su relación superficie/volumen. El presente trabajo analiza el efecto del tiempo de deposición de una semilla de óxido de zinc por la técnica de deposición física de vapor asistida por plasma y el tiempo de cultivo de los nanocables de óxido de zinc por el método hidrotermal. Las muestras se caracterizaron por microscopia electrónica de barrido y se midió el ángulo de contacto para verificar sus propiedades hidrofóbicas o hidrofílicas. Con la caracterización se obtuvo un ángulo mayor de 103.493° y un menor de 14.006°, esto debido a la forma y tamaño de las nanoestructuras y la manera en la que encapsula el aire.

PVD, Óxido de zinc, Método hidrotermal

Citation: ESTRADA-MARTÍNEZ, Fortino Fabián, MELO-MÁXIMO, Dulce Viridiana, VEGA-MORON, Roberto Carlos and HERNÁNDEZ-HERNÁNDEZ, Celia Massiel. Effect of deposition time on the zinc nanowires growth on steel substrates. Journal of Technological Development. 2022. 6-17:25-29.

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Introduction

One of the nanotechnology advantages is to generate solutions to current problems based on surfaces modifications. New properties in materials are achieved by the manipulation of atoms thus varying their shape, size, and/or functionalization [1].

On the other hand, physical vapor deposition (PVD) is a non-polluting technique used to produce coatings. This process involves the transfer of one (target) material onto another (substrate) from vaporization atom by atom, growing on the substrates a surface film [2].

This and similar techniques are mostly used for the synthesis of thin films used for materials protection; however, this technique can also be used for the generation of support (seed) for the growth or deposition of materials since it generates a homogeneous layer on the surfaces [3].

Additionally, among metal oxides, ZnO is relevant in various applications within the nanotechnology area due to its conductive and permeable properties [4]. The synthesis methods for these structures are simple and inexpensive, among them are inorganic reduction, chemical vapor deposition, green chemistry, and others [5].

The most stable structure of zinc oxide is *"wurtzite"*, this structure presents a singularity in its physicochemical properties, becoming hydrophobic or hydrophilic depending on their inclination due to the seed from which are generated [6].

The growth of zinc oxide nanostructures is affected by a first process known as seed. The result is a variety of shapes depending on the surface on which they are developing since their orientation and size depend on them [7].

Methodology to be developed

The growth of the zinc oxide nanowires was conducted in two stages: seed deposition and growth of the nanowires. The zinc oxide seed was deposited by PVD during 1 and 5 seconds with a constant oxygen flow (1 sccm) and power of 50 W, the substrates were 316L steel previously prepared up to mirror finish.

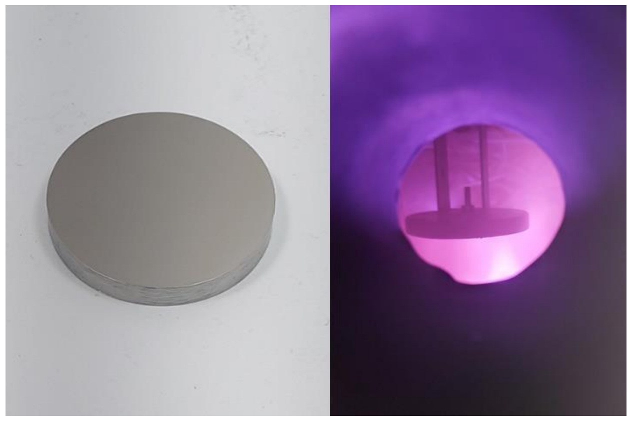


Figure 1 Mirror-polished 316L substrate. Plasma from the process of seed deposition by PVD

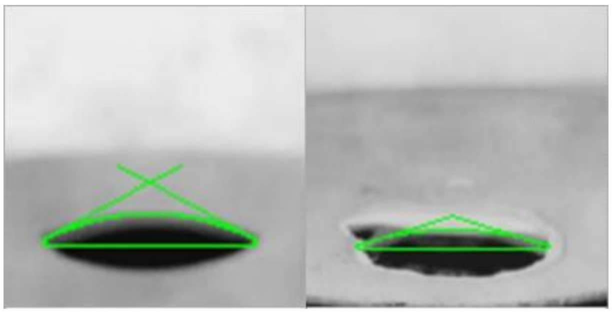
Once the seeds were deposited and characterized by contact angle test, the samples were immersed in 1:1 solution of zinc nitrate and hexamethylenetetramine, then placed inside an furnace for 1, 2 and 4 hours at a temperature of 90 °C.

Figure 2 Samples inside the furnace

Each of the samples was carefully washed with distilled water and left to dry at room temperature in a desiccator, then characterized by contact angle with open-source ImageJ software, scanning electron microscopy and energy dispersion analysis were also performed.

Results

The characterization of the samples showed a change in the size of the structures as time passed, in addition to the change in their wettability. Figure 3 represents the sample with a seed of 5 seconds and 4 hours in the furnace. The contact angle decreased to approximately half the size compared to the seed only.



Seed 28.402 ° Nanowires 14.336°

Figure 3 Contact angles of the sample obtained with 5 seconds of seed deposition and 4 hours of the hydrothermal method

The SEM image figure 4 exhibits long structures and with a non-uniform arrangement so it is possible that the contact angle decreased since there is no encapsulated air between the structures. The peaks present in the EDS indicate the presence of zinc and oxygen on the surface.

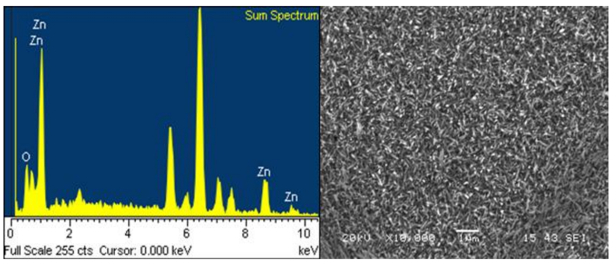
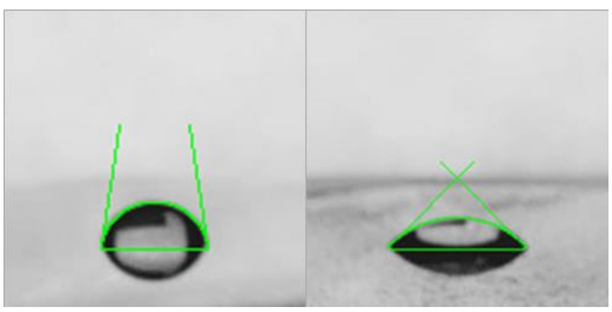


Figure 4 EDS and SEM of the sample obtained with 5 seconds of seed and 4 hours of hydrothermal method

In figure 5 can be observed the behavior of the sample obtained with 5 seconds of seed deposition and 2 hours of growth; the contact angle of the seed is higher than the obtained on the surface with nanowires.



Seed 81.011 ° Nanowires 45.979°

Figure 5 Contact angles of the sample obtained with 5 seconds of seed deposition and 2 hours of the hydrothermal method

The SEM image of Figure 6 shows small agglomerations with linear structures on the surface. Like the previous sample, EDS shows the presence of zinc and oxygen with a smaller intensity.

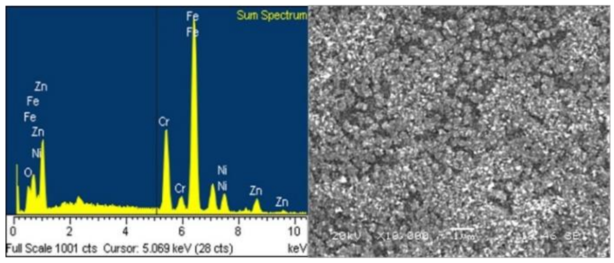
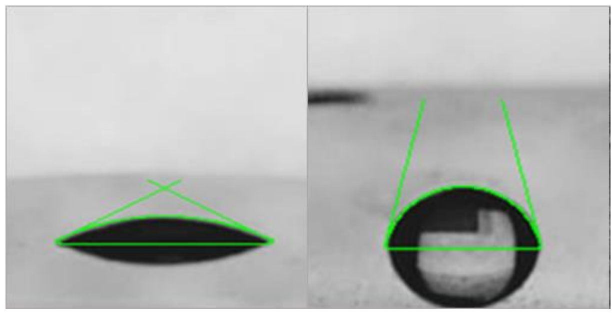


Figure 6 EDS and SEM of the sample obtained with 5 seconds of seed and 2 hours of hydrothermal method

The sample obtained with 5 seconds of seed deposition and one hour growth can be seen in Figure 7. The contact angle of the surface with nanowires shows an increase compared to the measured in the seed.



Seed 32.204 ° Nanowires 75.143°

Figure 7 Contact angles of the sample obtained with 5 seconds of seed deposition and 1 hour of hydrothermal method

In the SEM image (figure 8), similarly to previous sample, can be observed more dispersed agglomerations on the surface. EDS analysis exhibits zinc and oxygen peaks at a lower intensity than those of previous samples.

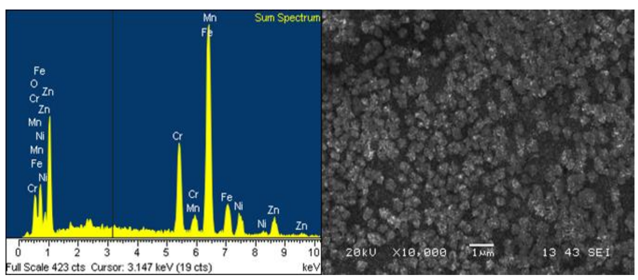
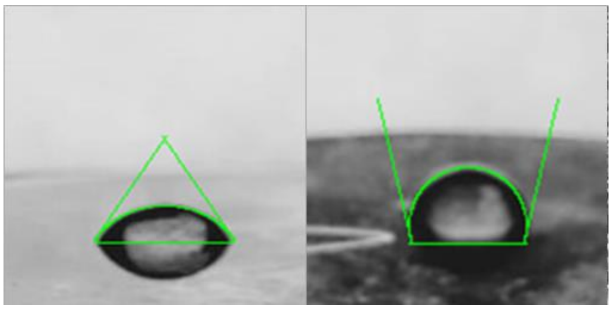


Figure 8 EDS and SEM of the sample obtained with 5 seconds of seed and 1 hour of hydrothermal method

The samples with 1 second of deposition had a different behavior to those of 5 seconds since, in all of them, the angle increased with respect to that of the seed.

ISSN 2531-2960

 Journal of Technological Development
June 2022, Vol.6 No.17 25-29



Seed 59.228 ° **Nanowires 103.493°**

Figure 9 Contact angles of the sample 1 second of seed deposition and 4 hours of the hydrothermal method

Figure 9 presented the sample with 1 second of seed and 4 hours of growth, the SEM image (Figure 10) shows long and disordered structures where the air is encapsulated; therefore it has a contact angle higher than 100°.

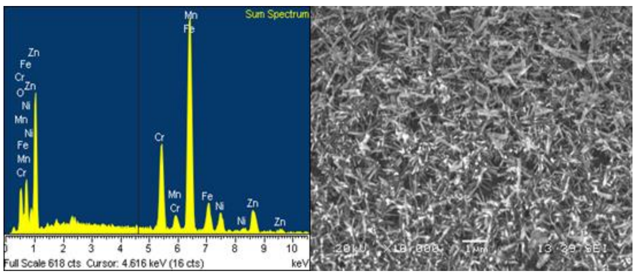
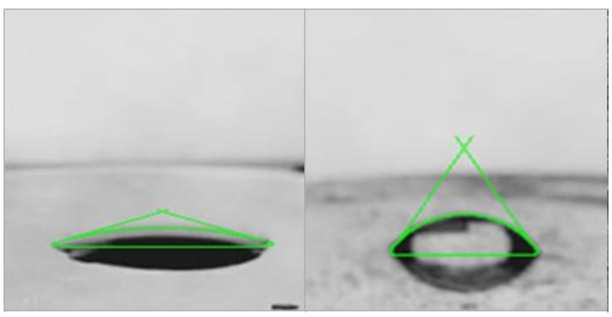


Figure 10 EDS and SEM of the sample obtained with 1 second of seed and 4 hours of hydrothermal method

Similarly, to previous samples, in the surface were observed zinc and oxygen.



Seed 18.321 ° **Nanowires 56.21°**

Figure 11 Contact angles of the sample obtained with 1 second seed deposition and 2 hours hydrothermal method

For the sample with 1 second of seed deposition and two hours of culture the contact angle also increased.

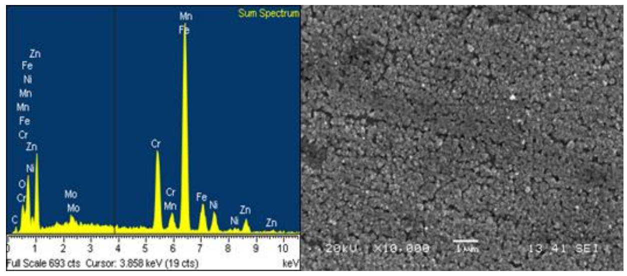
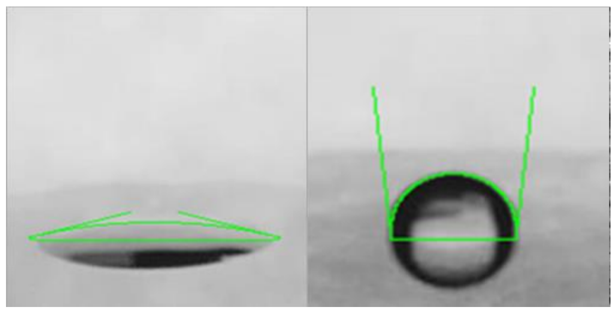


Figure 12 EDS and SEM of the sample obtained with 1 second of seed and 2 hours of hydrothermal method

From SEM inspection of the surface can be observed agglomerations, however, they are so close together that appear to be a single one. The presence of zinc oxide is observed in the EDS according to the peaks obtained.



Seed 14.006 ° **Nanowires 96.151°**

Figure 13 Contact angles of the sample obtained with 1 second of seed deposition and 1 hour of the hydrothermal method

The 1-second seed presented in Figure 13 has an angle of 14° that, after the growth of nanowires, increases to 96°.

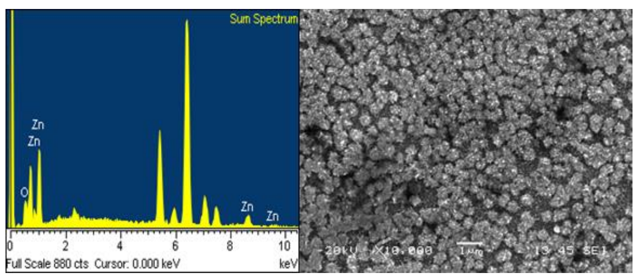


Figure 14 EDS and SEM of the sample obtained with 1 second of seed and 1 hour of hydrothermal method

Characterization by SEM shows agglomerations with linear structures. The signal of both zinc and oxygen in this sample is low compared to the others, althought it corroborates oxide formation.

ISSN 2531-2960

Acknowledgements

To the Graduate Studies and Research Division of TECNM-ITTLA, to the surface engineering laboratory of the Technological Institute of Monterrey campus Estado de México, as well as to CONACYT for the national scholarship granted.

Conclusions

Zinc oxide nanowire arrays were successfully synthesized using the hydrothermal method from a seed deposited by PVD. The relationship of seed deposition time and hydrothermal growth time is observed in the change of structures above the surface.

The characteristic hexagonal wurtzite structure of zinc oxide is not very noticeable in the MEB images since the structures are small.

The interaction of the surface with the liquids had evident changes, in some of the samples (seed of 1 second) increased compared to the seed deposited, however, the other samples (seed of 5 seconds) have unusual variations since only one of them had an increase in angle while the others decreased.

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