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Plasma characterization and morphological study for zirconium thin films deposited by DC magnetron sputtering at different powers

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Abstract

The electrical properties of zirconium thin films has been studied by Langmuir probe technique. The electron energy, electronic density, and ionic density of the samples were measured at low pressure with a varying power (60 to 160 W). Our results show that the energy of electrons decreases with increasing power, while the densities of electrons and ions have increased. Scanning electron microscope (SEM) images were used to determine the thickness as well as surface morphology of zirconium thin films, where the thickness was increased with the increasing of the power of the plasma DC generator. The energy dispersive X-ray spectroscopy (EDX) analysis method was employed to reveal information about the composition of the films. Plasma characterization and the quality of prepared films (roughness and thickness) allows us using these films in several potential applications.

Keywords: Zn thin films, magnetron sputtering, Langmuir probe, morphology, SEM.

1. Introduction

Metal of groups (IV-VI) have been attracting much attention due to their distinctive chemical and physical properties. They are refractory compounds with high melting points, and they exhibit high resistance against corrosion [1], which make them potential substitute for noble metals in various applications of material science [2,3]. In recent work, we have found the corrosion resistance of TiAlV films decreases with increasing the current density, where the grain size increased due to current density [4]. Surface coatings include the expansion of a layer of molten or semi-molten material on the substrate. One of the main requirements of surface coating is to improve and enhance the surface properties of the material rather than completely reshaping the composition of the material. Some examples of these processes are plasma and thermal spraying, sol-gel, cladding, electroplating, and Physical Vapor Deposition (PVD) [5], and Chemical Vapor Deposition (CVD) [6]. The basic principle of the CVD process is a chemical reaction between the target and gases in order to form solid materials that condense on the substrate surfaces inside the deposition chamber [7], while the film a vapours are generated in the PVD process either by evaporating a molten target or by ejection of atoms from a solid target that is subject to bombardment by an ionized gas. Steam remains as neutral atoms in the vacuum or becomes ionized, reacting with the ionizing gas and then placed on the substrate. PVD and CVD techniques are used to deposit metals as well as nitride films [1]. CVD technique is not common, because all materials available are solid and ineffective due to processing difficulties and flow control. In Magnetron Sputtering method, the advantages are high deposition rate, good uniformity, and low temperature of the substrate during the deposition. Magnetron sputtering [1,8] is widely used in the manufacturing technology, because of producing high-quality films with thickness and morphology controlled [8,10]. Plasma and deposition parameters such as substrate temperature, power [1], and oxygen partial pressure [11] influence the physical properties of the deposited films including structural, corrosion, and mechanical behaviors.

The results will allow us to determine the influence sputtering power on the plasma parameters and morphology (surface and thickness) properties and growth behavior of the deposited Zr thin films. We have performed two characterizations, the first is plasma

(a)

(b)

Figure 1. Experimental set-up of DC magnetron sputtering, (a) with plasma and (b) without plasma.

properties and the second is films quality (morphology and thickness) using Langmuir probe as well as Scanning electron microscope (SEM).

2. Experimental details

DC magnetron sputtering system was used to prepare the films by a starting from high pure (99.99 percent) Zr target (50 mm diameter and 6 mm thickness) in plasma discharge argon (20 sccm Ar flux) for deposition of Zr film. The working pressure was fixed at 9 mTorr for 10 min deposition time. The DC power was varied from 60 to 160 W. Figure 1 shows our DC magnetron sputtering set-up with and without plasma. The power influences on morphological properties, electron flux, ion and electron densities behavior of these films have been studied in the present work.

3. Results and discussion

3. 1 Langmuir probe measurements

Using Langmuir probe technique, we have characterized the composition of Zirconium plasma at low pressures with varying DC power. At the first, we have measured the energy of the electrons with variation of the DC power as shown in figure 2. It is seen that the energy of electrons decreases gradually with increasing the power. This phenomena is due to an increase of the ion bombardment with the higher power, as it was found in the recent work [5], where they studied the effects of the power and pressure on aluminum doped ZnO films via RF magnetron sputtered properties.

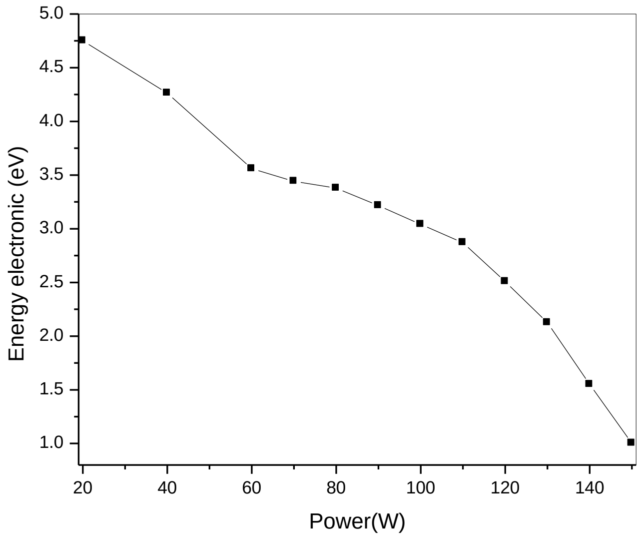


Figure 2. Energy electronic versus the power.

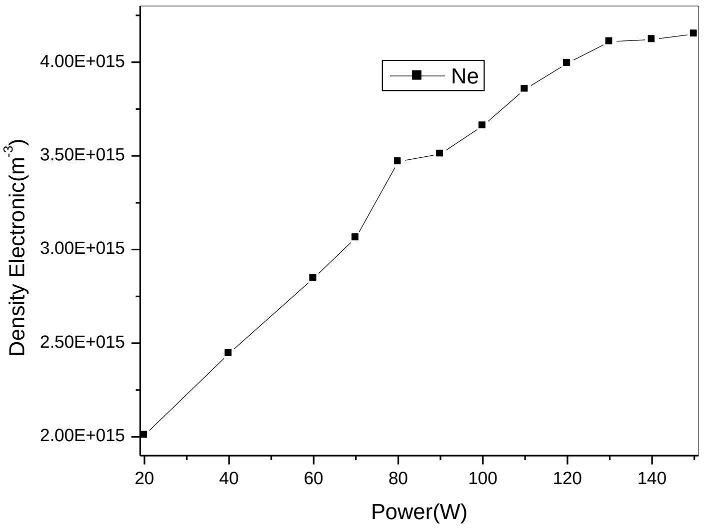


Figure 3. Electron density versus the power.

With respect to measure the electrons density with increasing DC power using the Langmuir probe, we have found that the density of electrons increases rapidly at first, after a certain value it increases slowly, and this is shown in figure 3. This behavior of the electrons density can be explained by increasing the ionization rate during the variation range (20-150 W) of the power [12].

Figure 4 shows the density of zirconium ions, which measured using the Langmuir probe technique. In the fact, the energy provided by the generator accelerates the electrons inside the plasma. The accelerated electrons provided more collisions from the argon atoms inside the chamber, and these processes led for producing more and more of electrons. The increase in electrons density is accompanied by an increase in the density of ions, as well [13]. The increase of the ion density leads to the increase of thickness with power (it will be shown in next paragraph) which was observed in our previous works using DC power of generator [13,14].

3. 2 Morphology characterization (SEM) and (AFM)

3. 2. 1. Surface morphology (top view SEM)

General view (aspect) of the surface of deposited films at different powers are shown in figure 5 using SEM. It is seen that the prepared films at low power are less rough than the prepared films at high power (greater thickness). The increasing in grain size is mainly due to increase of the number of collisions energy of electron with power. The sputtered Zr atoms also undergo more collisions leading to a high probability of agglomeration and growth even before arriving at the substrate.

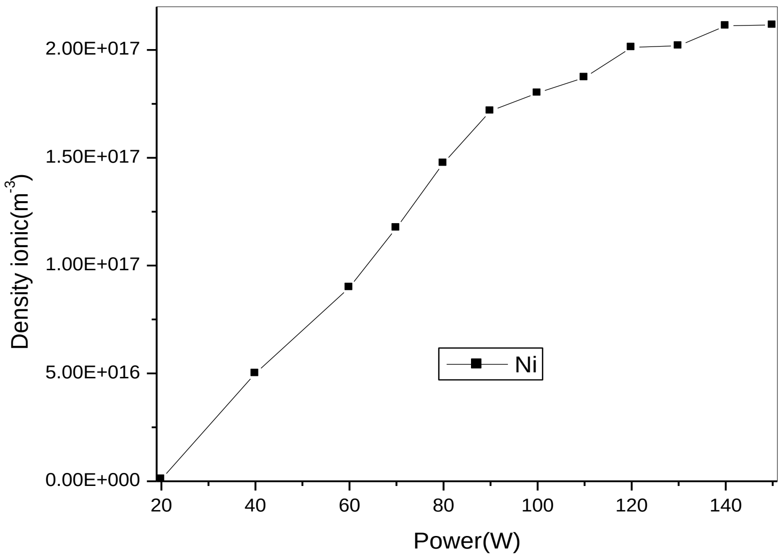
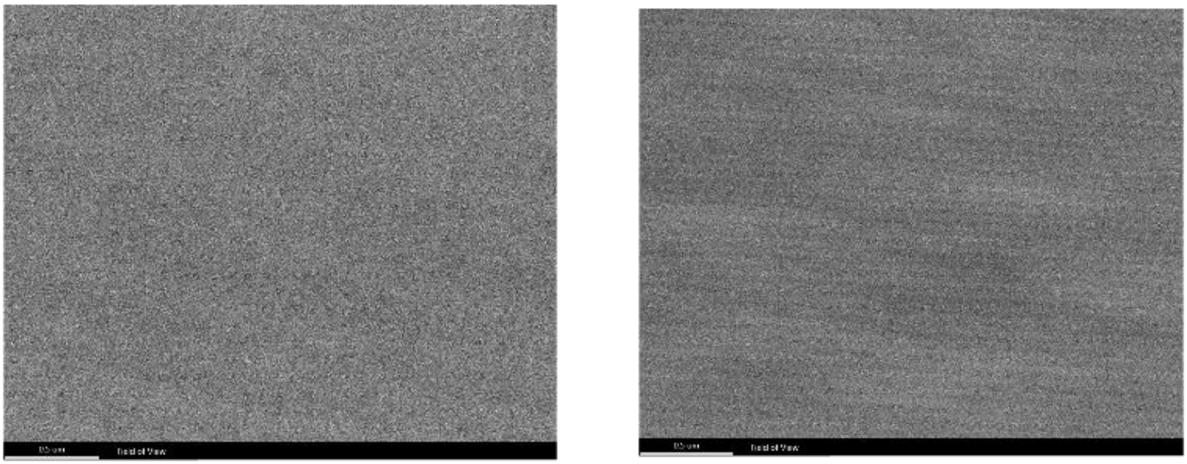
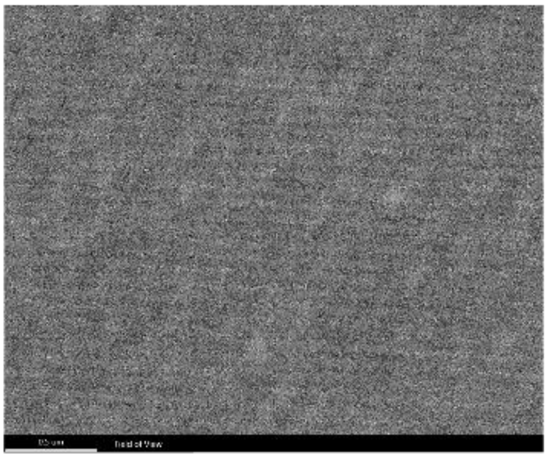


Figure 4. Ion density versus the power.



(a) 100 W

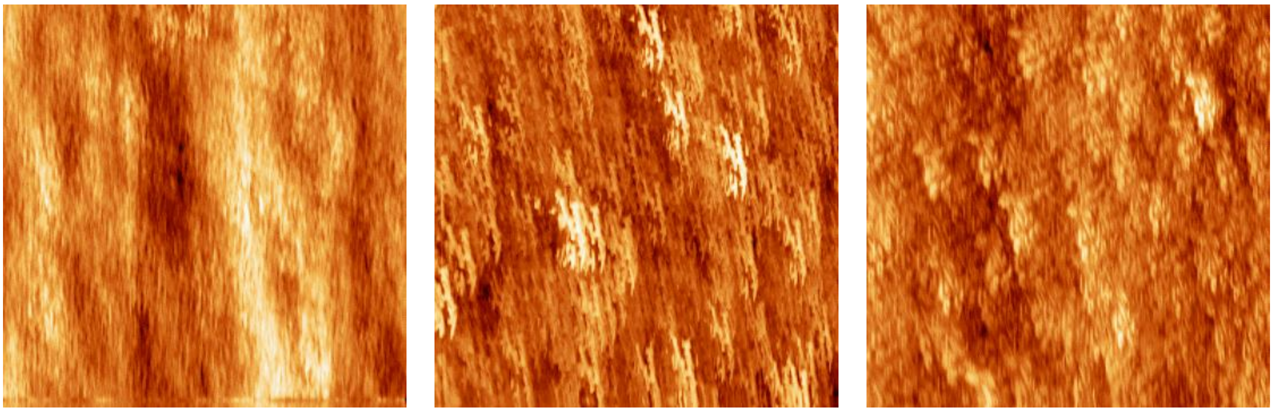
(b) 140 W



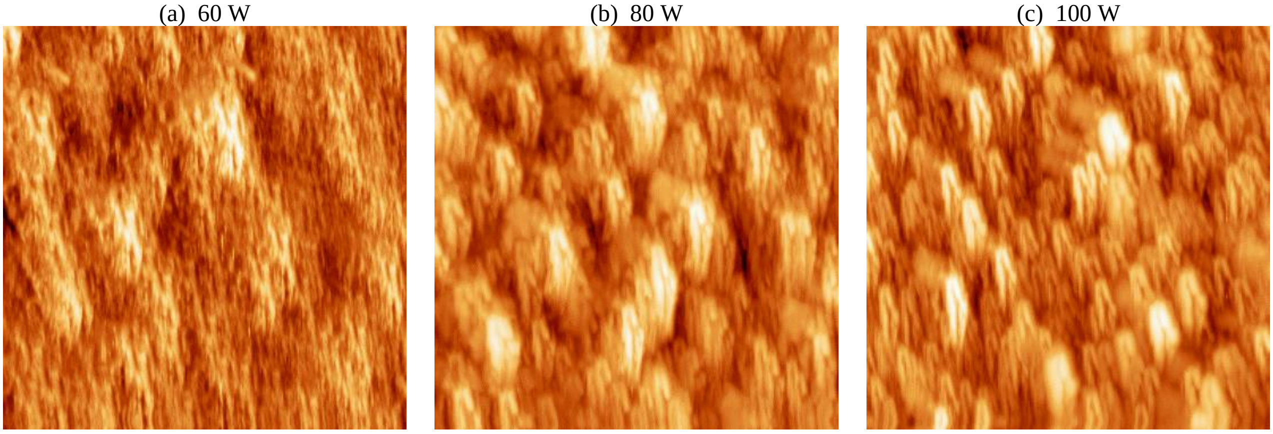
(c) 160 W

Figure 5. SEM Surface morphology of Zr films deposited on Si substrate at different power (from 100 to 160 W).

The higher grain size of the Zr film at higher power may be due to increased diffusivity of atoms facilitating the grain growth. It is consistent with published literature [13,15]. Consequently, the roughness stays low while the thickness increases with power increase, which allows for using these films in potential application in microelectronics.



(a) 60 W (b) 80 W (c) 100 W



(d) 120 W (e) 140 W (f) 160 W

Figure 6. AFM Surface morphology (3×3µm) of Zr films deposited on Si substrate at different power (from 60 to 160 W).

3. 2. 2. Surface morphology (top view AFM)

AFM Surface morphology (top view) of thin films deposited at powers of 60 W and 80 W within a 3×3µm area, are presented in figures 6a and 6b, respectively. The films are smooth with a low roughness varied from 1 to 1.5 nm. Similarly, figures 6c, 6d, 6e, and 6f show AFM surface morphology for films deposited at powers of 100, 120, 140, and 160 W, respectively. These films exhibit more roughness, varying from 2 to 7 nm. These results are in a good agreement with the SEM morphology (figure 5), increasing the roughness is attributed to the increase of the grain size as well as the thickness of the samples [16], in accordance with the increase of the ion bombardment and DC power [14,17].

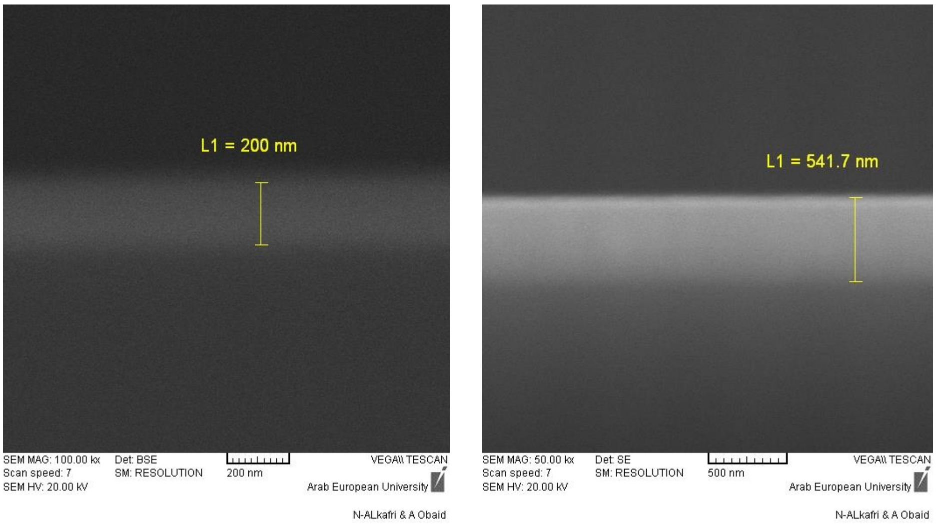
3. 2. 3. Cross section (thickness)

Figure 7 shows the obtained SEM cross section of Zn films deposited on Si substrate as a function of DC power. The obtained thicknesses from SEM cross sections are displayed in figure 8. We observe that the thickness increases from 200 to 1050 nm with increasing the power from 100 to 160 W, in agreement with pervious works [13,18]. The increase of the thickness with power is due to the enhancement of the ionic bombardment which is related to the power of the DC generator or decrease of the pressure [18].

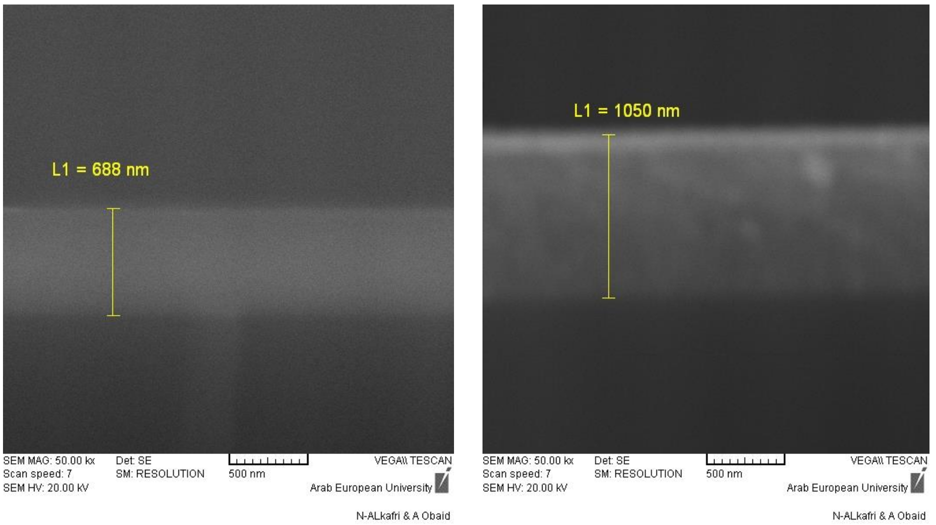
Figure 9 present EDX spectrum of film deposited at 160 W, where the principal peak is due to Zr elements and the other peaks are assigned to C and O contamination. Table 1 presents weight percentage for C, O and Zr elements. It shows the sample has Zr as maxima percentage (88.41%) which confirms the creation and growth of zirconium film, while C and O have contaminated the films due to the exposure to atmospheric environment and/or small amount of humidity adsorbed in the chamber.

4. Conclusions

Zirconium films have been successfully prepared by DC magnetron sputtering technique at different DC power (100-160 W). The effects of power on the structure and morphology of the deposited Zr films were investigated. The Langmuir probe technique was helped to understand the mechanism of thin films formation through the electrical measurements of electron energy, ion density, and electron energy. These measurements are important to know which variable controls the composition of these films. The EDX analysis showed that the composition of the films has good quality and low contamination. SEM technique was used to measure the thickness of the films and assess their roughness and morphology. It was found that the thickness increases with power increase. However, the roughness stays low and thus permits potential of these films for microelectronics applications.



(a) 100 W (b) 120 W



(c) 140 W (d) 160 W

Figure 7. SEM cross section of Zn films deposited on Si substrate at different of DC powers.

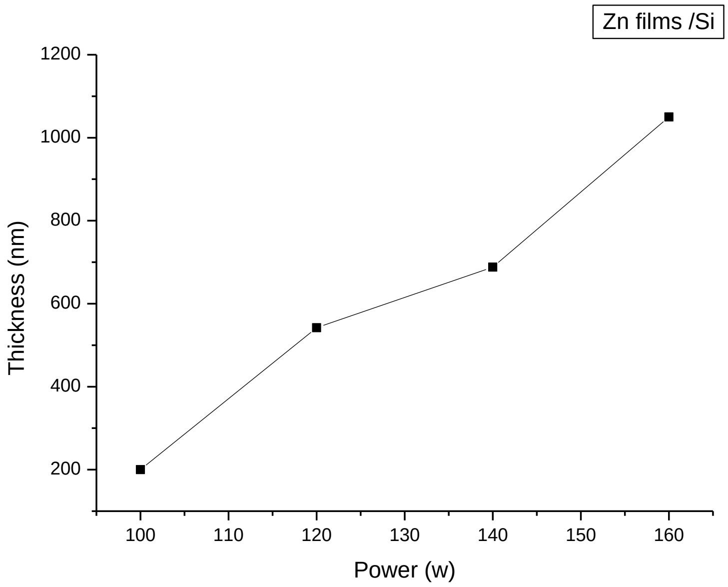


Figure 8. Thickness of Zn films deposited on Si substrate as a function of DC power.

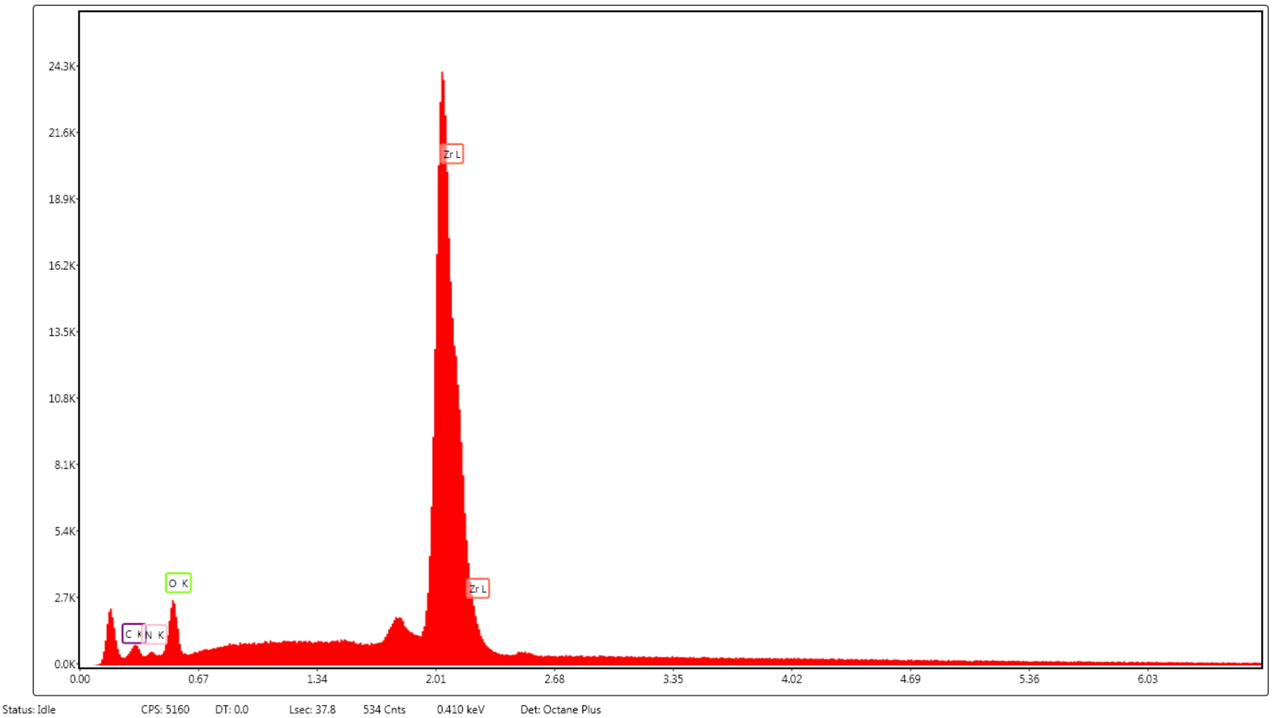


Figure 9. EDX spectrum of film deposited at 160 W.

Table 1. EDX results of Zr/Si film deposited at 160 W.

Element	C K	O K	Zr L
Weight (%)	3.15	8.44	88.41

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