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# An Innovative Method for Measuring the Optical Band Gap of Oxidized Surface Layer of Aluminum Tablets based on Absorption Spectra

Kamal Kayed*

*Department of physics - Faculty of Science - University of Damascus - Syria.*

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**ABSTRACT**

This research highlight an innovative method for calculating the optical band gap of thin layers of aluminum oxide resulting from the surface oxidation of non-transparent samples of aluminum. The aluminum samples were prepared using the compressing and annealing methods. Thermal heating in suitable oven was used for annealing procedures. The effects of annealing temperature on structural and optical properties of studied samples were investigated by measuring the optical band gaps and FT-IR spectra. The results show that the annealing temperature affects the structural and optical properties of the surface layer. In addition, it was found that in the case of annealing temperature at 1000°C, the optical band gap of alumina samples was found to be 4.99 and 4.19 eV at annealing times of 2 and 4 hours respectively. On the other hand, at the temperature of 500°C, the surface has metallic properties because the area of the oxide layer is very small. This indicates that the annealing temperature has great role in determining the properties of the surface layer.

**Keywords:** Aluminum oxide, annealing, optical band gap, plasma edge, reflectivity spectra.

## 1. INTRODUCTION

Aluminum is a chemical element with a symbol Al, and an atomic number 13. It is a silvery-white, soft, nonmagnetic element and it could be easily shaped [1-5]. Aluminum could be forged as thick plates for heavy industries or pulled as thin sheets for food packaging. It is also used in airplane's manufacturing due to its light weight [1]. It does not corrode and it is erosion resistance, especially under severe conditions. Aluminum is the third most abundant element in the Earth's crust (after oxygen and silicon) and it makes up about 8.3% of the crust by mass [1]. This metal is highly chemically reactive to oxygen that individual specimens could not be found. On the contrary, it is found combined with oxygen and silica in the rocks and the Earth's crust [1,2]. Aluminum oxide ($Al_2O_3$) is a chemical compound known as alumina. Aluminum oxide has two different forms (alpha and gamma) with different crystal structures [1-5]. Both forms differ in terms of chemical and physical properties and they are also used in different applications.

The procedure of powder metallurgy includes compressive forming of the metal powder using solid and highly resistant mold to get the required shapes. During this process, the spaces between the powder particles decrease and they partially solidify [1]. In the second stage, the

*Corresponding author information: Kamal Kayed, Department of physics, Faculty of Science, University of Damascus, Syria. Email address: khmk2000@gmail.com

compressed product is being sintered by heating the samples to relatively high temperatures, but less than the melting temperature of either the powder or the compounds. Sintering leads to higher solidification of the powder particles causing an increase in the density and the hardness of the product. Suitable sintering temperature and enough time should be chosen correctly to get a high quality product [1].

Valence electrons in any conductor act like free electrons, where the collision among the electrons is similar among gas molecules that are described in the kinetic theory of gases [6]. Optical properties of metals are produced due to the interaction between the fallen photons on the surface and the electronic cloud [5-7]. Optical reflectivity is considered the most important physical property of the metal layers, which generally relates to the interaction between the light and the free electrons which could be expressed by the equation of dispersion as follows [6]:

$$\varepsilon_f = 1 - \omega_p^2/(\omega^2 - i\omega/\tau) \tag{1}$$

where $\omega_p$ is the plasma edge. It is a unique feature of metals expressing plasma-resonance frequency of a free electron, $\tau$ is the relaxing time, $\omega$ is the light frequency and $\alpha$ is the absorption. Both electrons density and effective mass affect this plasma edge [6] and could be directly determined from the reflecting spectra where dramatic change of reflectivity happen at the plasma edge as a result of electrons drag difference [6]. Furthermore, shifting of plasma edge towards higher wavelengths means increasing the free charge holders [6].

In the present work, attempts have been made to study the optical properties of the oxidized surface layer of aluminum samples as a function of annealing temperature. The results of this study are of high importance because they include a method for measuring the optical band gap of the surface layers of non-transparent materials.

## 2. MATERIALS AND METHODS

Compressive forming of high purity aluminum powder (99.98%) was done using a hydraulic compressor. The applied pressure was 70 KN. Cylindrical samples were obtained with diameter 1.25 cm. The thickness is 2.1 mm, and the weight is 0.7 g.

The characterization processes were made after the sample surface had been cleaned using alcohol. Two samples were treated thermally by annealing them to 500°C and 1000°C using thermal oven for 2 or 4 hours. All samples were characterized using Fourier Transform Infrared (FTIR) (Model: IR prestige-21 Shimadzu). The reflectivity measurement was performed by using Cary 5000 UV/VIS/NIR spectrometer. The conditions of the samples preparation are shown in Table 1.

**Table 1** Samples preparation conditions

| | Annealing temperature(c°) | Annealing time(hr) |
|---|---|---|
| Sample1 | - | - |
| Sample2 | 500 | 2 |
| Sample3 | 1000 | 2 |
| Sample4 | 1000 | 4 |

## 3. RESULTS AND DISCUSSION

Fourier Transform-Infrared spectroscopy has been used to determine the chemical composition of the samples. An example of FTIR spectrum is that of the sample 4, shown in Figure 1. The broad bands around 3400 – 3700 cm$^{-1}$ reveal the presence of O-H stretching vibrations, and the additional peaks appeared in the region of 1100-1700 cm$^{-1}$ represent the presence of physisorbed water, while the line at 2350 cm$^{-1}$ is due to $CO_2$ from atmospheric contamination. The intensity of these lines decreases with the annealing. The bending vibration of terminal Al=O and Al–O appears in the range 670 cm−1-1300 cm$^{-1}$. These results were in a good agreement with the earlier literature [8].

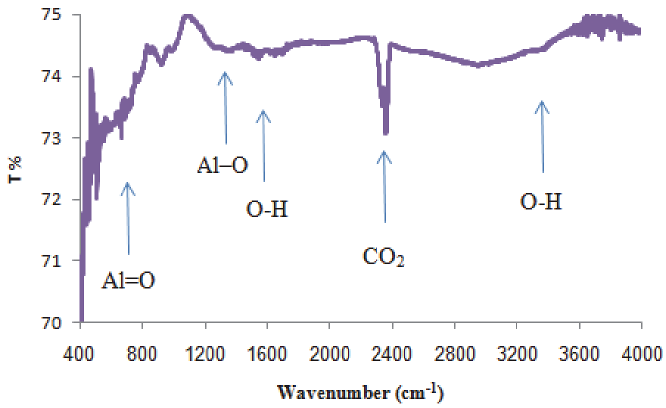


**Figure 1.** FT-IR spectra for sample 4

The non-treatment sample has a higher transition than the rest of the samples. This indicates that the surface oxidation of the samples leads to a decrease in the IR transition. Figure 2 illustrates the optical reflectivity as function of wavelength. The reflection values decreased for the annealed samples as compared to the non-annealed sample. The decreasing range includes all wavelength values with one exception which is sample 3, where reflectivity increases in the visible range. The reason for this behavior is the decreasing of the non-oxidized metal ratio in the surface (the aluminum atoms are localized in the bulk rather than near the surface) which decreases the concentration of the charge carriers. Nevertheless, there is a very slight shift to the plasma edge toward the low wavelengths due to the radical change in the mechanism of the interaction mechanism between the light and the matter at the surface because the surface layer has oxidized [6].

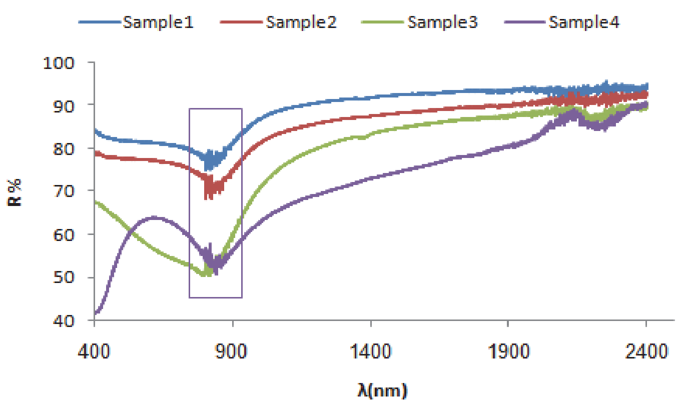


**Figure 2.** Reflectivity spectra for prepared samples (The rectangle contains the plasma edges)

The reflectivity declines due to the formation of the oxide layer on the surface which absorbs a portion of the incoming light. Therefore, it can be assumed that the amount of the shortfall in the reflectivity for samples 2, 3 and 4 is equal to the amount that is absorbed by the oxide layer. Accordingly, the absorbance of the oxide layer in the case of the samples 2 until sample 4 can be obtained by subtracting the reflectivity curves for these samples by the reflectivity curve of the sample 1. Figure 3 illustrates the optical absorption (Δ) for the samples 2, 3 and 4. For sample 4, there is a radical change noticed in the absorption mechanism in the visible region of the spectrum. This can be explained by the fact that the surface acquires the properties of a semi-conductor material because of the formation of the oxide layer [6,7].

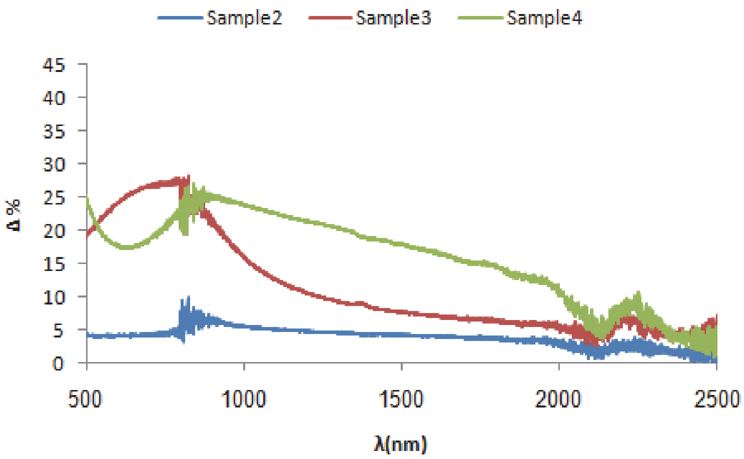


**Figure 3.** The optical absorption (Δ) for the samples 2,3 and 4

The curves in Figure 3 can be used to calculate the absorption coefficient and the optical energy gap. The optical gap was calculated for all samples using the relation [9]:

$$\alpha h\nu = A(h\nu - E_g)^2 \quad (2)$$

where E is the energy of the incident light, $E_g$ is the estimate of the optical band gap and **A** is a constant. Therefore, $E_g$ can be found by plotting the variation of $(\alpha E)^{1/2}$ against E, where the extrapolation of the linear region of the curve with x-axis gives the value of the optical band gap of a thin film. Figure 4 shows the method of calculating of the energy band gap in the case of sample 3.

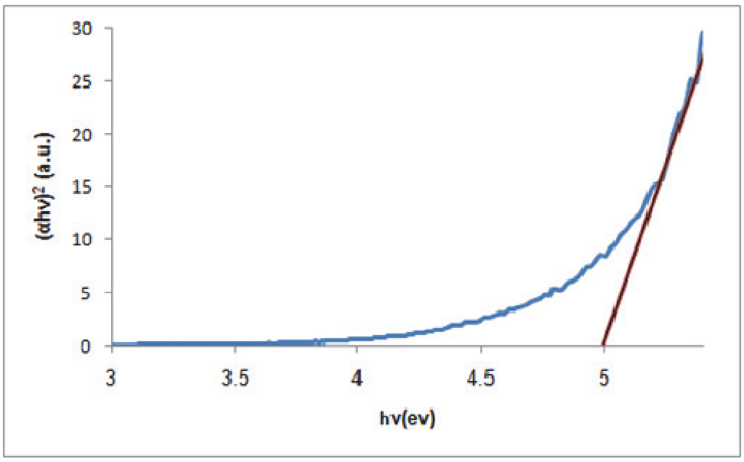


**Figure 4.** variation of $(\alpha h\nu)^2$ against hv for sample 3

In the case of the samples 3 and 4, the linear part of the curves intersects with the x-axis at a point 4.99 and 4.19 eV respectively. These values are consistent with the calculated values of the optical energy gap of aluminum oxide [10]. These results indicate that a large area of the surface has oxidized and has the characteristics of a semi-conductor. The decrease in the band gap value when applying extreme annealing conditions could be attributed to the phase transitions.

In the case of the samples 2, no linear part is observed in the curve because there is a large non-oxidized area of the surface, so the surface layer still has a metallic structure, and the optical properties depend on the interaction between photons and free electrons. Consequently, it can be concluded that the absorption curve shape is related to the degree of surface oxidation.

## 4. CONCLUSION

The effects of annealing of aluminum samples for different temperature, which were prepared using the compressing and annealing methods, were investigated through FTIR spectroscopy and UV/VIS/NIR spectrometer. Based on the results, the changes in reflectivity spectra due to the thermal treatment determine the optical absorption properties and the optical band gap of the oxidized surface layer. The optical band gap energy decreased with the increasing of annealing temperature.

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