



Study of the Topographic and Optical Characteristics of Cerium Oxide Thin Films Prepared by the Thermochemical Spraying Method

Zainab Emad khaled¹  , Amjad H. Jassim²  

^{1,2}Department of Physics, College of Science, University of Tikrit, Tikrit, Iraq

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ABSTRACT

In this work, cerium oxide thin films were prepared by thermochemical spraying. The material was deposited on different segments, including glass, crystalline silicon and porous silicon. An atomic force microscope (AFM) was used to analyze surface roughness and morphological homogeneity of the thin films, where its AFM images showed clear differences in roughness and surface homogeneity between samples. Nanoscale compositions appeared more clearly in the silicon wafers compared to glass and photoluminescence analysis (PL) technology was used for the study of optical emissions and locations of oriented lengths, where this technique revealed a major emission at 863.2 nm in the case of sedimentation on porous silicon, indicating the effect of the concentration on the optical properties, as well as the use of UV-visible (UV-Vis) spectrometry technology to determine the energy facets and absorption properties, where it showed that the energy gap differs by different substrates and was its highest value when deposition was on glass (3.46 eV). According to UV-Vis measurements, these results open the way for multiple applications in optical reagents and other optical devices.

Keywords: spray pyrolysis, rare earth element, cerium oxide (CeO₂), surface morphology

Name: Zainab Emad Khaled

E-mail: zenab.emmad@st.tu.edu.iq



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دراسة الخصائص الطبوغرافية والبصرية لأغشية أكسيد السيريوم الرقيقة المحضرة بطريقة الرش الكيميائي الحراري

زينب عماد خالد¹، أمجد حسين جاسم²

^{1,2} قسم الفيزياء، كلية العلوم، جامعة تكريت، تكريت، العراق

الملخص

في هذا العمل تم تحضير اغشية رقيقة من اوكسيد السيريوم بطريقة الرش الكيميائي الحراري، اذ تم ترسيب المادة على شرائح مختلفة، وهي كل من الزجاج والسيليكون البلوري والسيليكون المسامي. تمت دراسة الطبوغرافية "السطحية" والخصائص البصرية للأغشية المحضرة باستخدام تقنية مجهر القوة الذرية (AFM) لتحليل خشونة السطح والتجانس المورفولوجي للأغشية حيث أظهرت صور (AFM) الخاصة بها اختلافات واضحة في الخشونة وتجانس السطح بين العينات. بدت التراكيب النانوية بشكل أكثر وضوحاً في رقائق السيليكون مقارنة بالزجاج واستخدمت تقنية تحليل الفلورة الضوئية (PL) لدراسة الانبعاثات الضوئية ومواقع الأطوال الموجية حيث كشفت هذه التقنية عن انبعاث كبير عند الطول الموجي (863.2 nm) في حالة الترسيب على السيليكون المسامي يظهر تأثير التركيز على الخواص الضوئية، وكذلك استخدام تقنية مطياف الأشعة فوق البنفسجية - المرئية (UV-Vis) لتحديد فجوات الطاقة وخواص الامتصاص، حيث تبين أن فجوات الطاقة تختلف باختلاف قواعد الترسيب وكانت أعلى قيمة لها عند الترسيب على الزجاج (3.46 eV) وفقاً لقياسات المطياف (UV-Vis)، من هنا فان هذه النتائج تفتح الطريق لتطبيقات متعددة مثل الكواشف الضوئية والأجهزة البصرية الأخرى.

INTRODUCTION

Thin films are very thin physical layers, usually prepared with thicknesses ranging from nanometers to several micrometers. Research in this area has advanced significantly in recent decades, driven by increasing demand for these films across multiple applications, such as sensors, solar cells, batteries, and electronic transformers. (1) A common method of preparing these films is thermochemical spraying, a process that uses heat to accelerate chemical reactions that form thin films. Thermochemical spraying is one method for heating chemical compounds to high temperatures and spraying them onto a surface to form a thin film. This process depends on a chemical reaction that occurs when raw materials are heated, forming thin films. Multiple sources, such as direct flame or specialized heating ovens, provide heat. (2) Cerium is a rare-earth element belonging to the lanthanide series. Cerium is the most abundant element of this

MATERIALS AND METHODS

series, ranking 25th in terms of prevalence in the Earth's crust. (3, 4) Cerium oxide has significant chemical stability, allowing it to be used in harsh environments such as high temperatures or strong oxidizing conditions. (3). In addition, these thin films act as an effective antioxidant barrier, improving the stability of metal surfaces and prolonging their life. (5). The deposition of the atomic/molecular layer of the hybrid thin films of cerium (III) was studied using solid organic materials. This was further confirmed experimentally through X-ray photoelectron spectroscopy (XPS). Additionally, the thin films exhibited a strong ability to absorb ultraviolet (UV) radiation. These films show potential for future applications as effective oxidizing and reducing agents and as UV-absorbing materials. (6). The study aims to prepare thin films of cerium oxide and to study their optical and structural properties.

The materials used in the thermochemical spraying process are hydrocerium chloride ($CeCl_3 \cdot 7H_2O$). It

is a solid, white-colored powder with a molecular weight of 372.58 g/mol. To prepare the solution of the substance in a concentration of 0.05 M, dissolve 1.7 gm of cerium chloride in 25 ml of distilled water for several minutes to ensure dissolution, so that we get the solution for the preparation of the thin films and then prepare the silicon strips of type N (111) orientation. They are cut in dimensions of 2 cm × 2 cm. The procedure involves preparing thin films on 0.1 mm-thick borosilicate glass substrates. At the same time, porous silicon wafers were prepared within the electrochemical cell consisting of cylindrical containers made of Teflon (an expression of polymeric material with high resistance to acidic solutions) as the negative electrode is made of platinum and the positive pole is the silicon substrate within the electrolytic solution and this electrolytic solution is hydrofluoric acid (HF) diluted by ethanol (C₂H₅OH) at a concentration of 40% and a relative ratio of 1:2, where it is characterized by electrical conductivity where we shed the density of a current (20 mA/cm²) to help the drilling process (porous) and thus get highly homogeneous porous silicon wafers where the spraying process has been done by choosing the right conditions and then stabilizing the distance between the spraying device and the base of the heater at 27 ± 1 cm, spraying the solution for the duration of 10 sec, and then stopping for the

duration of 3 min for the purpose of reaching the required base heat and then following another spray. This method is applied to the substrates of crystalline silicon, glass and porous silicon.

RESULTS AND DISCUSSION

X-Ray Diffraction (XRD) Results

X-rays are electromagnetic waves that lie between ultraviolet rays and gamma rays, characterized by relatively high energy and specific wavelengths ranging from 0.1 to 100 Å, comparable to interatomic distances. Therefore, they are used in crystal diffraction experiments to determine the shape and characteristics of the unit cell and the crystal structure of a given material, whether single-crystalline, polycrystalline, or amorphous. (7). The crystallographic interpretation of Figure (1) illustrates the following: the main peak appears more prominently in the crystalline silicon sample, indicating excellent crystalline growth; due to the well-ordered atomic arrangement of the substrate for the porous silicon sample, there is an increased distribution of random signals, which may suggest lower crystallinity or the presence of a dispersed nanostructure, leading to more complex and less sharp diffraction patterns. In the glass sample, a broad distribution of signals is observed, indicating weak crystallization and possibly a partially amorphous nature, due to its irregular surface and inability to orient crystals.

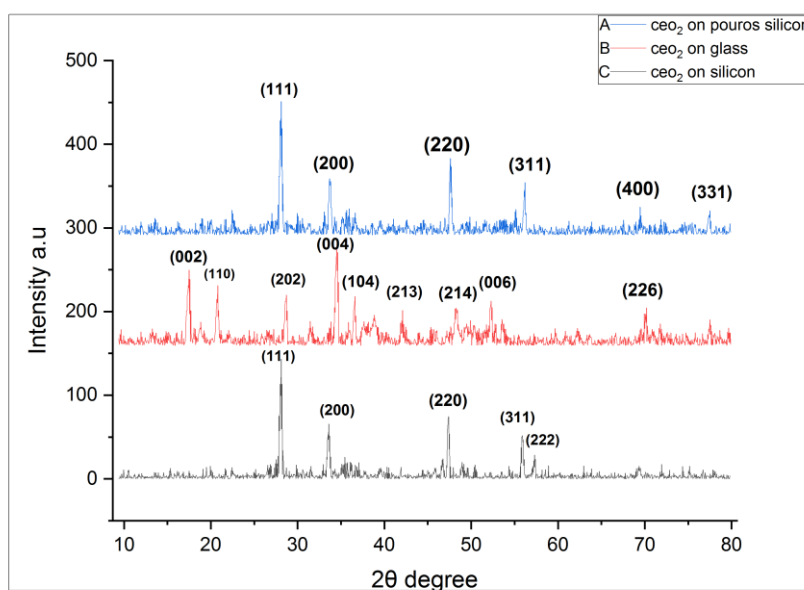


Fig. 1: XRD results for CeO₂/PS, CeO₂/Si, and CeO₂/glass.

In Figure (1)(A), we observe that the horizontal axis (2θ) represents the angles at which XRD was measured. The main diffraction peaks appear at (111), (200), (220), (311), (400), and (331), which correspond to the fluorite cubic structure of cerium oxide (CeO₂). The peak at (111) indicates that the thin film tends to have a preferred crystalline orientation in this direction, meaning that the crystals grew with a strong alignment along this plane. The presence of sharp and strong peaks suggests that the film has a satisfactory crystalline structure. The high crystallinity of cerium oxide can be beneficial for optical and electronic applications, such as photodetectors and photocatalysis.

Figure (1)(B) presents the XRD pattern of cerium oxide deposited on glass. Based on the annotations in the figure, the crystallographic properties can be analyzed as follows: the recorded diffraction peaks appear at different 2θ angles with specific crystallographic planes such as (002), (110), (004), (202), (104), (213), (214), (006), and (226). The strongest peak is at (004), indicating a preferred growth orientation in this direction. The overall distribution of peaks suggests the presence of small-sized crystals with a relatively ordered structure. The presence of crystallographic planes (002),

(004), and (006) suggests that the material follows a tetragonal or hexagonal system. Other planes, such as (110) and (104), may indicate a polycrystalline nature. Given that glass typically leads to amorphous or polycrystalline growth, the presence of relatively sharp peaks suggests that the material has acquired some degree of crystallinity. However, an amorphous fraction may still be present. The crystal structure may be less ordered than that of the sample deposited on crystalline silicon.

Figure (1)(C) represents the analysis of the XRD pattern, which helps in determining the crystal structure and phase composition of the material. The main diffraction peaks appear at (111), (200), (220), (311), and (222); these peaks indicate a face-centered cubic (FCC) crystal structure or a mixture of different crystalline phases. The most intense peak at (111) suggests a preferred growth orientation in this direction. Cerium oxide can crystallize in multiple phases, with the hexagonal phase being the most common. Regarding the effect of the substrate (crystalline silicon), the absence of strong crystalline silicon peaks (e.g., at 28.4°) indicates that the deposited film fully covers the substrate.

Table 1: XRD Parameters for the Prepared Thin Films

	Pos. [2θ .]	(hkl)	FWHM [2θ .]	d-spacing [\AA]	Strain %	Grain Size (nm)	Matched By
CeO ₂ /Glass	16.718	(002)	0.5196	5.3031	0.002243	14.95874	00-044-1001
	21.8304	(110)	0.2598	4.07137	0.001113	29.69166	00-044-1001
	28.56	(202)	0.2598	3.12549	0.001099	29.3044	00-044-1001
	34.2618	(004)	0.2165	2.61729	0.000903	34.67659	00-044-1001
	36.9182	(104)	0.433	2.43484	0.001792	17.20976	00-044-1001
	42.1663	(213)	0.5196	2.14315	0.002115	14.10726	00-044-1001
	48.6081	(214)	0.2598	1.87312	0.001033	27.55879	00-044-1001
	53.2133	(006)	0.433	1.72137	0.001689	16.2219	00-044-1001
	70.3811	(226)	0.6927	1.23193	0.00247	9.268446	00-044-1001
Average					0.001485	24.6516	

	Pos. [2θ .]	(hkl)	FWHM [2θ .]	d-spacing [\AA]	Strain %	Grain Size (nm)	Matched By
CeO ₂ /Si	28.8469	(111)	0.3168	3.0925	0.001339	24.01644	01-075-0076
	33.9396	(200)	0.8448	2.43146	0.003526	8.894363	01-075-0076

	46.2016	(220)	0.3168	1.9633	0.001271	22.80963	01-075-0076
	55.8407	(311)	0.6336	1.64508	0.002443	10.95577	01-075-0076
	59.2136	(222)	0.6336	1.55917	0.002404	10.78016	01-075-0076
Average					0.002196	15.49127	

	Pos. [°2Th.]	(hkl)	FWHM [°2Th.]	d-spacing [Å]	Strain %	Grain Size (nm)	Matched By
CeO₂/PS	28.6821	(111)	0.3696	3.10989	0.001562	20.59311	01-075-0076
	34.1042	(200)	0.7392	2.62684	0.003084	10.16052	01-075-0076
	47.5892	(220)	0.4224	1.90924	0.001686	17.01761	01-075-0076
	56.3705	(311)	0.3168	1.63087	0.001218	21.85761	01-075-0076
	69.2265	(400)	0.264	1.30696	0.000948	24.49069	01-075-0076
	78.0479	(331)	0.264	1.2104	0.000895	23.11821	01-075-0076
Average					0.002323	15.37682	

AFM Results

The atomic force microscope is a device that studies surfaces at the nanoscale by passing a very sharp needle over the sample's surface; morphological analysis reveals how atoms are distributed and arranged on the surface and illustrates the heterogeneity and characteristics of each granular composition.⁽⁸⁾ It analyzes surface roughness as in Figure 2. It was noted that the surface has moderate coarseness, with sharp, centrally located peaks that produce variations in altitude and lows in thin-film areas, and that its average value is high (29.8689 nm). It was noted a relative regularity in the distribution of granular agglomerations, where the coarseness of the root mean square (RMS) (4.39127 nm), which is an indication of the thin film's surface variability, is suitable for optical or photoelectric applications and that agreed with^(8, 9).

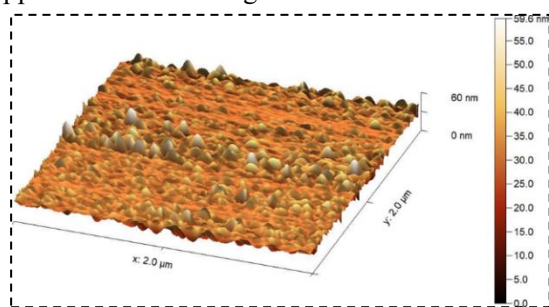


Fig. 2: AFM result for CeO₂/Glass thin film prepared by thermochemical spraying process.

Figure 3 shows that the roughness is lower than in the previous sample when deposited on crystalline silicon, and that low altitudes are useful in photovoltaic and electronic applications, with an

average value of 14.2166 nm and a root-mean-square (RMS) of 3.41268 nm.

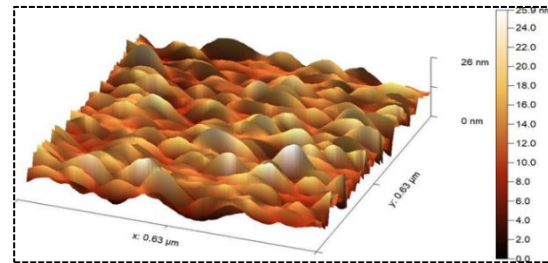


Fig. 3: AFM result for CeO₂/Si thin film prepared by thermochemical spraying process.

Figure 4 shows that the deposited material's surface is not perfectly smooth, exhibiting variations and irregularities that contribute to an overall surface coarseness. The mean roughness was measured to be 3.33848 nm, and the root mean square (RMS) roughness was 4.58519 nm. This level of surface roughness can significantly influence the material's properties, including its ability to absorb light, interact with surrounding substances, and contribute to a moderately coarse texture.^(9, 10)

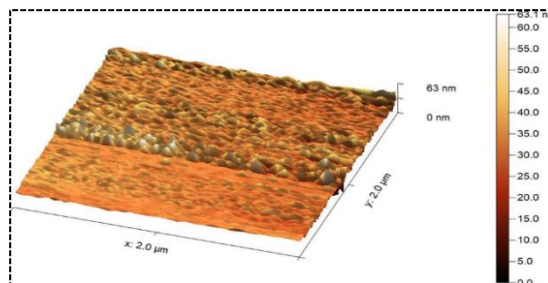


Fig. 4: AFM result for a CeO₂/PSi thin film prepared by a thermochemical spraying process.

PL Results

Photoluminescence is a non-corrosive optical technique that characterizes and verifies defects at

the point level and also measures the material's energy surface by irradiating the crystal with photons above the material's energy bandgap. (10). In Figure 5, light emission, we observe the issuance of photons from the glass as a result of being absorbed by photons. Its top is observed at 581.13 nm and provides information on the two types of light emitted, as this wavelength lies in the yellow part of the visible spectrum and corresponds to the energy gap (2.13 eV). It refers to the energy required to excite electrons in the material and is an important characteristic that influences light absorption and emission.

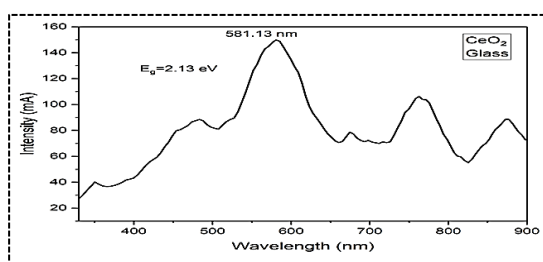


Fig. 5: PL spectrum of CeO₂ / glass thin film prepared by thermochemical spraying process.

Figure (6) exhibited the effect of the presence of rare earth elements such as cerium on the absorption and emission of light, as the peaks that appear indicate the areas of light resonance at which those emissions and absorption of light occur on the crystal silicon wafer of that material.

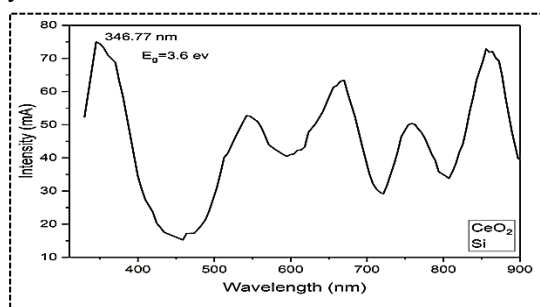


Fig. 6: PL spectrum of CeO₂ / Si thin film prepared by thermochemical spraying process.

By analyzing the spectrum in Figure 7, we observe a major emission at 863.2 nm, where the equivalent (1.44 eV) is that the irregular distribution in this spectrum reflects the existence of defect centers or quantum overlaps resulting from the nanoscale composition of porous silicon.

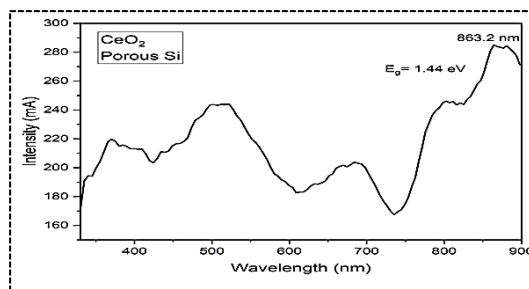


Fig. 7: PL spectrum of CeO₂ / PSi thin film prepared by thermochemical spraying process.

The best photoluminescence in porous silicon has been achieved due to quantum-confinement effects resulting from high porosity, which narrows the energy gap and increases the efficiency of light emission by trapping electrons and holes for longer, thereby enhancing emission at longer steering lengths. In crystalline silicon, scintillation is weak due to the radioactive recombination of electrons and holes. When the material is deposited, there is a slight improvement, but it will not be as efficient as the porous silicon; this agrees with (11).

Optical Properties

UV-Vis spectroscopy is one of the most commonly used techniques for determining energy gaps in insulating materials and semiconductors. Using glass as a concentrator allows us to study photosynthesis more accurately because it is transparent in the ultraviolet range, facilitating visualization. If the light interacts with the material, the beam loses part of its energy, and the photons interact with the material's electrons, resulting in the material's optical properties, which are associated with the electromagnetic field of the radiation. (12), and when we calculate the radiation in 10 (Tauc plot) from the optical absorption data, it allows us to determine the nature of the electronic transition (direct or indirect) (13). As in Figure (8) where the curve shows us a sharp increase at a certain energy value where the intersection point with the horizontal axis represents the energy gap of the material its value (3.86 eV), it makes cerium oxide has a wide energy gap that means it shouts an electrical insulator and not a semiconductor in the visual field where energy facets are used in many applications such as photocatalysis in the field of ultraviolet radiation and in environmental

applications such as oxidation and stimulation of chemical reactions and used as insulating thin films in the field of electronics such as metal oxide transistors (MOSFETs) and in optical anti-reflective coatings (for not absorbing light in the visual field)⁽¹¹⁾.

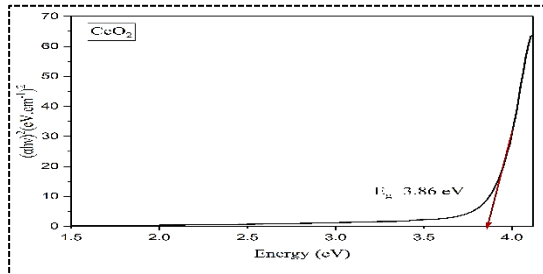


Fig. 8: Energy gap photons and absorption intensity for CeO₂ / Glass.

In Figure 9, the optical indirect band gaps of the thin films were calculated from UV-Vis data. E_g is at (3.46 eV), the band gap of the substance, where the energy of the electron is affected by the equivalent package to the delivery package, which represents that this value is of great importance in understanding the electronic and optical properties of the material.

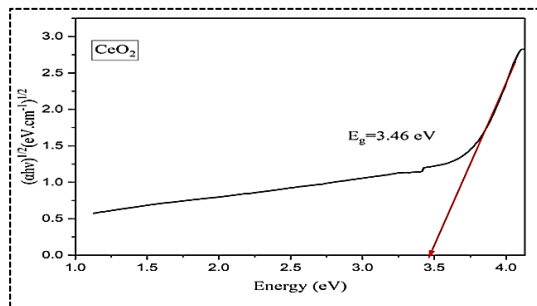


Fig. 9: Touc's plot of the CeO₂ / glass.

The results demonstrate the optical absorption spectrum of cerium dioxide (CeO₂) deposited on the glass substrate in a thermochemical spraying way, where Figure 10 shows a sharp drop in absorption at (400-360 nm), indicating the edge of the optical absorption. At this wavelength, the absorption is weak, meaning the material is transparent in the visible range and below the red. From previous results, we found that the energy gap is 3.46 eV. These values are in line with CeO₂ properties, where the thermochemical spraying route provides better control over homogeneity and thickness than other methods, such as Physical Vapor Deposition (PVD).

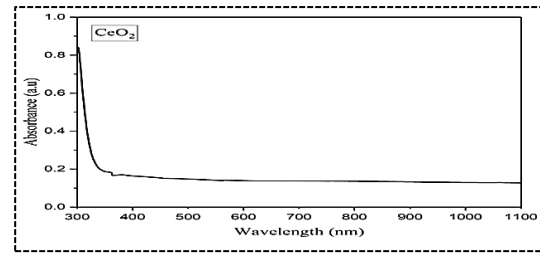


Fig. 10: The relationship between wavelength and Absorbance for CeO₂ / Glass.

When analyzing permeability behavior, it was noted that the curve rises at a certain wavelength, then settles to higher values in the visible and infrared regions, and that this behavior is common in insulating materials or semiconductors with a wide energy gap, where absorption is high in the ultraviolet range. Then the material becomes highly permeable at large lengths. When examined under high transparency in the visible and infrared fields, this metabolite is suitable for several applications, the most important of which are solar cells.^(8, 11) It is considered a powerful material and is applied as a barrier, as well as in light-sensing devices, optical coatings and thin films resistant to oxidation and corrosion.

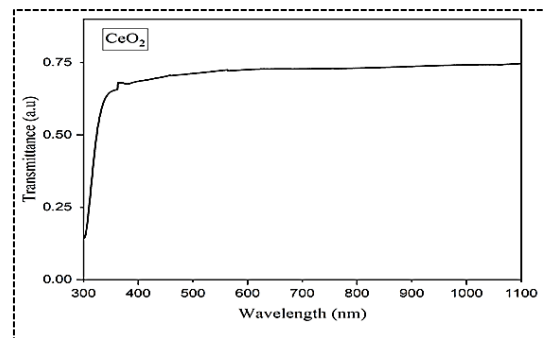


Fig. 11: The relationship between wavelength nm and transmittance for CeO₂ / glass.

By qualitative analysis of the comparison between the absorption and permeability spectra, we find that the relationship is inverse, as shown in Figures (10) and (11), where we see that absorption is very high at wavelengths below 400 nm. This indicates that the material absorbs these wavelengths. Therefore, we get an explanation for the sharp decrease in permeability in that range, after which (400 nm) becomes almost transparent. (Permeability approaches 1 and absorption approaches 0). This

means it does not absorb light significantly in this field, as its high permeability in the visible field makes it transparent beyond the critical energy limit, which agrees with ⁽¹⁵⁾.

CONCLUSIONS

In this research, we got thin films of (CeO₂) deposited on several substrates of both crystalline silicon, porous silicon and glass, where we used a lot of analyses for each of the AFM, UV – Vis and PL. The AFM analysis provides information on surface roughness and homogeneity. This helps us understand the relationship between surface structure and optical properties. The high roughness rate affects optical dispersion, thereby affecting emission efficiency and absorption. We infer through the results of PL a major emission at (863nm). The energy gap value of cerium Oxide can be obtained on the glass substrate, where it was found to be equal to (3.46 eV), as well as (1.44 eV) on porous silicon, indicating the effect of its concentration on the optical properties of thin films. In general, these results showed that cerium chloride interacts with each substrate in different forms, suggesting that the selection of the concentrate can be adjusted.

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Author contributions: The authors contributed equally to the study.

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