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Study of the Structural Properties of $NiCl_2:Al_2O_3$ Thin Film Prepared by Chemical Thermal Spraying Technique

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دراسة الخصائص التركيبية للأغشية الرقيقة $NiCl_2:Al_2O_3$ المحضرة بطريقة الرش الكيميائي الحراري

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ABSTRACT

Background: The structural properties of films are studied by several techniques, and X-ray diffraction (XRD) is one of the most reliable methods that were adopted in this research, so will explain it in some detail, in addition to assays (SEM) that give information on surface morphology. Materials and Methods: The thermochemical spraying technique was used to prepare the thin films. Results: The results showed that increasing the temperature led to a decrease in the average particle size, in contrast to the doping process. Conclusion: The reason for the decrease in particle size is due to the increase in the Full Width at Half Maximum (FWHM) in addition to an increase in the crystallization of the thin films, and also the membrane becomes more regular and crystalline defects decrease and increase.

Keywords: thin film , thermal chemical spraying , doping , semiconductor , annealing

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1. INTRODUCTION

The study of thin films aroused the interest of many physicists for more than a century and a half, starting in omit (1838), the study of thin films has a wide range in many physical research in particular and in this research for other specialty in general, prepared thin films via the chemical reaction technique, other for preparing thin films were developed over the years to obtain films with new specifications that are suitable for use in practical applications [1].

A layer of material ranging from fractions of a nanometer to several micrometers in thickness is called the thin film. Thin film have very interesting properties that are quite different from those of their bulk form. Due to the fact that thin films properties are dependent on interrelated parameters and the deposition technique employed for their synthesis. Nanostructured materials are defined as clusters, crystallites or molecules which have dimensions in the 1 to 100 nm range. Both academic and industrial interest in these materials over the past decade comes from the distinguishable variations in fundamental electrical, optical and magnetic properties that occur as one progresses from an 'infinitely extended' solid to a particle of material consisting of several atoms[2,3], and because these layers are very thin, they are deposited on bases called the base (Substrate) and they are made of different materials such as glass[4], quartz, Aluminum and silicon[5].

In our research, the thermal chemical spray method is used. This technique is one of the chemical methods for thin-film deposition, and was developed during the 1960s due to the need for a lower-cost technology to prepare large-area devices in the photovoltaic industries. Thin films of inorganic sulfides and cyanides have been prepared by hydrolysis on a hot base. This method is distinguished from other methods of preparation by its simplicity and the low cost of the manufactured devices used in preparing the films compared to the costs of the devices used in other methods. Thin films are prepared under normal atmospheric conditions and at room temperature, so they do not give properties like those of films prepared under low pressure and thin films with good homogeneity and large surface areas can be prepared[6-8].In this technique only chemical solutions are used, i.e. the powder of the material cannot be deposited directly or by using alloys. This technology depends on spraying the material to be deposited in the form of a film on hot bases under certain omit depending on the type of material used, where a thermochemical reaction occurs between the atoms of the hot base material. Because of this interaction, a thin film is formed [9]. Nickel chloride is one of the nickel salts used in this research, and it is a chemical compound with the symbol ($NiCl_2$), its color is green. Nickel chloride in its various forms is considered the most important source of nickel in chemical synthesis it absorbs moisture from the air to form a solution [10]. Aluminium is the third element in the Earth's crust after oxygen and silicon. Aluminium turns into a medium or a catalyst to combine automatically with oxygen, forming the thin film of aluminium oxide that protects the aluminium or allows it to be colored after the anode oxidation, or the formation of aluminium minerals such as (bauxite), aluminium oxide (Al_2O_3), which ends with either aluminum metal and its alloys or in aluminuim

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oxide ceramics, which appear as a single crystalline segment in a (sapphire) gemstone, the first laser or unique polycrystalline ceramic material [11-13]. Actually it has many applications the importance of alumina stems from its use as a raw material for the extraction of aluminium metal, as a raw material for the manufacture of a wide range of advanced and traditional ceramic products, and as an active agent in various chemical industries. Its many applications are related to its abundance and low cost [14-17]. This study was conducted in order to know the effect of annealing on some structural properties and surface roughness of thin films and to know the possibility of doping nickel chloride ($NiCl_2$) with aluminium oxide(Al_2O_3).

2. MATERIALS AND METHODS

A Nickel chloride solution was prepared by dissolving 0.9 g in 30 ml of distilled water and heating it for 5 minutes at 70 °C. The mixture is mixed with a magnetic stirrer, and a solution of aluminium oxide is prepared, in which 5 g is dissolved in 100 ml of distilled water for 30 minutes at a temperature of 70 ºC, then filtered with filter paper. After that the pure and doped films were deposited on glass bases using a thermochemical spraying technique, then all films were annealed at 350 ºC.

3. RESULTS AND DISCUSSION

3.1. X-ray Diffraction Investigations

The importance of this measurement lies in knowing the crystal structure of the materials and showing the phases of the deposited materials and the nature of the arrangement of the atoms in them and their orientation. Figure (1) represents the X-ray diffraction diagram of the pure $NiCl_2$ film, and it is noticed that there are diffraction peaks corresponding to planes (101), (200), and (211), and when comparing these results with (ASTM) cards (American Standard of Testing Materials) numbered (03-065-0601) and as shown in Table(1),the results were somewhat consistent. program (x-pert) is used to extract assays with parameters (XRD).

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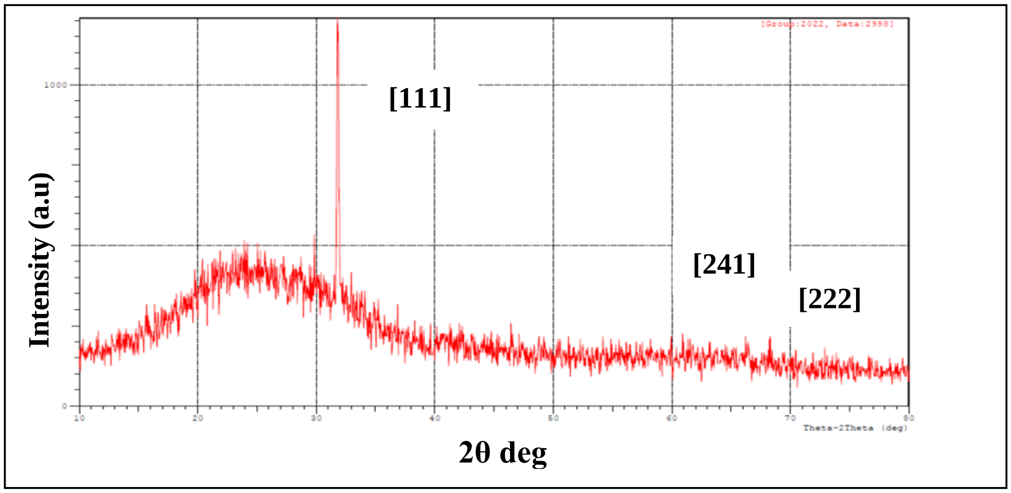


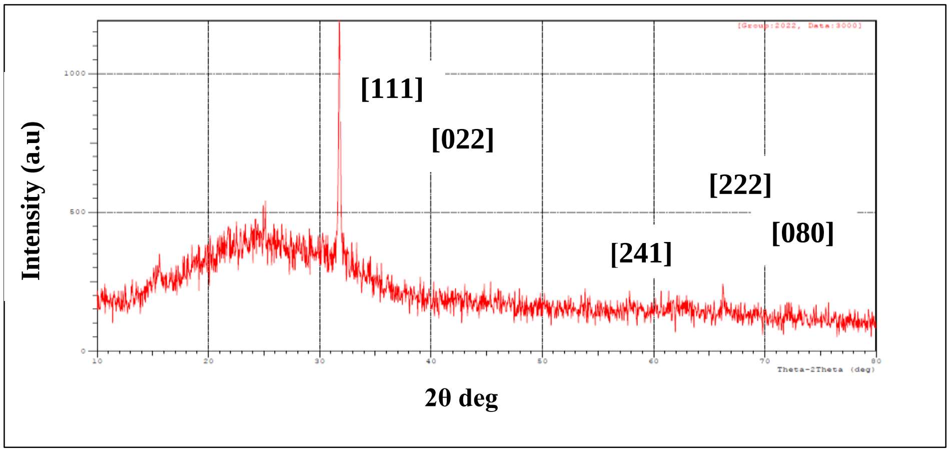
Fig(1): X-ray diffraction of $NiCl_2$ thin film pure.

Table1:shows the results obtained from the pure film.

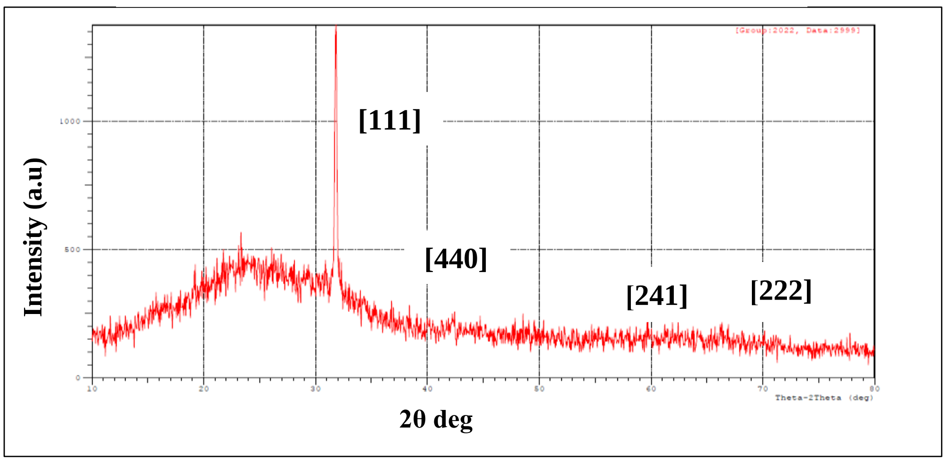
No	2θ	hkl	d(Å)	G.S (nm)	FWHM	I %	A	Sys.
1	32.944	111	2.716	33.7	0.254	99	a=3.778	orthorhombic
2	64.908	241	1.435	35.1	0.254	5.8	b=10.365	
3	69.123	222	1.357	61.4	0.165	2.1	c=4.221	
				Av.=43.4	Av.=0.231			

noticed through Figures (2),(3) and (4) which represent the results of X-ray diffraction of ($NiCl_2$) films doped with (Al_2O_3), with different inoculation percentages (1,2,3)% with the appearance of atomic growth in the dominant and characteristic crystallographic directions |

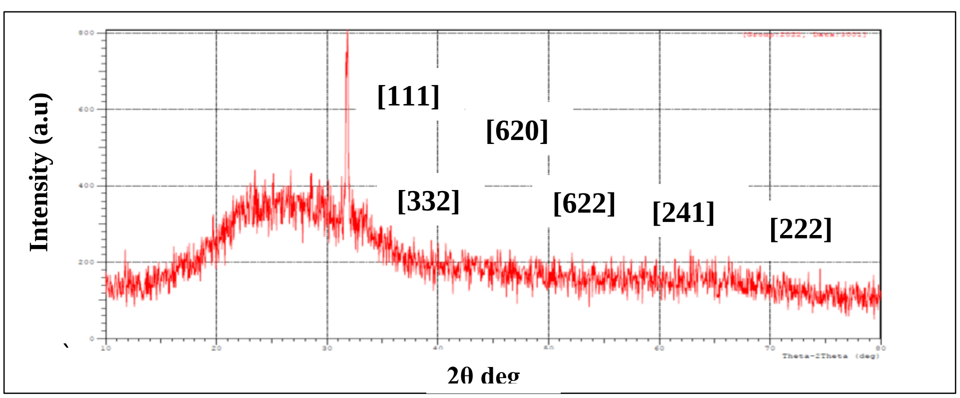
(111),(241),(222), with a slight displacement in the locations of the peaks (2θ) which indicates that the grafted films built polycrystalline structure and type (orthorhombic). Notice the appearance of the cubic system, which is specific to aluminum oxide, the material used in doping.



Fig(2): X-ray diffraction of $NiCl_2$:Al_2O_3 thin film at 1%.



Fig(3): X-ray diffraction of $NiCl_2$:Al_2O thin film at 2%.



Fig(4): X-ray diffraction of $NiCl_2$:Al_2O_3 thin film at 3%.

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It is noted that a noticeable change occurred in the intensity of the diffraction peaks for some levels after doping, so the intensity of the peaks decreased at the orientation (241), (222), and the intensity of the peaks increased at the orientation (111). The crystal structure, that is, some levels are preferred for the growth of crystals, and this indicates that the crystallization process is improved by adding aluminium oxide (Al_2O_3) to the films ($NiCl_2$), and the best level for crystal growth is (111), whose intensity is (ɪ=99)[18,19].

Table2: shows the results obtained for the doped film with (1%) Al_2O_3.

No	2θ	hkl	d(Å)	G.S (nm)	FWHM	I %	a	Sys.
1	44.924	022	2.016	18.5	0.472	0.2	a=3.77 b=10.46 c=4.37	orthorhombic
2	72.192	080	1.307	14.8	0.672	0.4		
3	32.944	111	2.716	43.8	0.197	99	a=3.778 b=10.365 c=4.221	orthorhombic
4	64.9.8	241	1.435	35.1	0.276	5.8		
5	69.123	222	1.357	113.5	0.093	2.1		
				Av.=45.14	**Av.=0.342**			

Table3: shows the results obtained for the doped film with (2%) Al_2O_3.

No.	2θ	hkl	d(Å)	G.S (nm)	FWHM	I%	a	Sys.
1	40.964	440	48.7	48.7	0.128	6.5	a=b=c= 12.433	Cubic
2	32.944	111	2.716	43.8	0.197	98	a=3.778 b=10.365 c=4.221	Orthorhombic
3	64.908	241	1.436	35.1	0.276	5.8		
4	69.123	222	1.315	113.5	0.093	2.2		
				Av.=60.275	**Av.=0.187**			

Table4: shows the results obtained for the doped film with (3%) Al_2O_3.

No.	2θ	hkl	d(Å)	G.S (nm)	FWHM	I %	a	Sys.
1	46.107	620	1.967	59.5	0.135	0.1	a=b=c= 12.441	Cubic
2	48.498	622	1.875	9.3	0.945	0.3		
3	33.765	332	2.652	14.8	0.578	22		
4	32.944	111	2.726	43.8	0.197			
5	64.908	241	1.432	35.1	0.276			
6	69.123	222	1.354	113.5	0.093			
				Av.=46	**Av.=0.370**			

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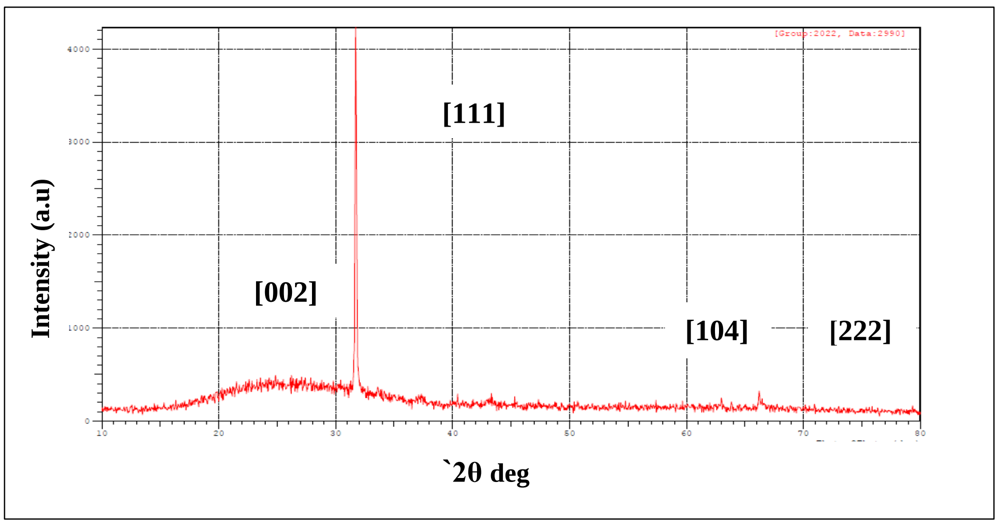
When annealing, noticed that an increase in the width of the middle, and this leads to a decrease in the particle size with an increase in temperature, which, as shown in the figures, as an increase in the base temperature leads to a decrease in the secondary levels, in addition to an increase in the crystallization of the film, and also the membrane becomes more regular and crystalline defects decrease and increase. Also the sharpness of the peaks, and this is due to the increase in crystallinity and regularity that occurs in thin film, and the increase in the height of the peaks when the base temperature is increased and attributed to the fact that the heat works to reduce crystal defects by giving the atoms of the material potential energy to rearrange themselves in the crystal lattice [20,21]. Figures (5),(6),(7) and (8) show the X-ray diffraction of pure ($NiCl_2$) films deposited on a substrate-(glass) bases at a temperature of (350), and thin films doped with (Al_2O_3) where the crystalline structure of all films appeared polycrystalline, and at a temperature of (350) the pure ($NiCl_2$) film was It has three peaks (three crystal levels): level (111) at ($2\theta = 33.653$), level (104) at ($2\theta = 65.099$), and level (222) at ($2\theta = 70.742$).



Fig(5): X-ray diffraction of $NiCl_2$ thin film (pure) at 350^0 C.

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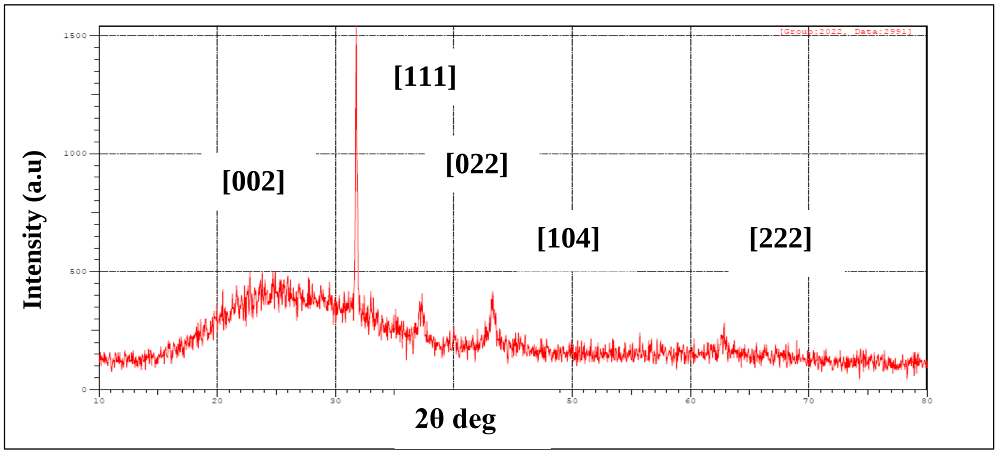
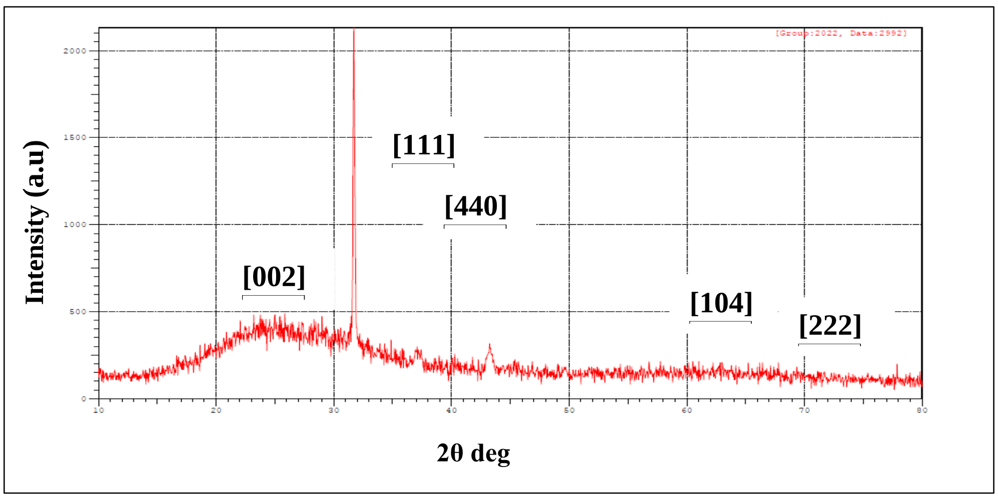
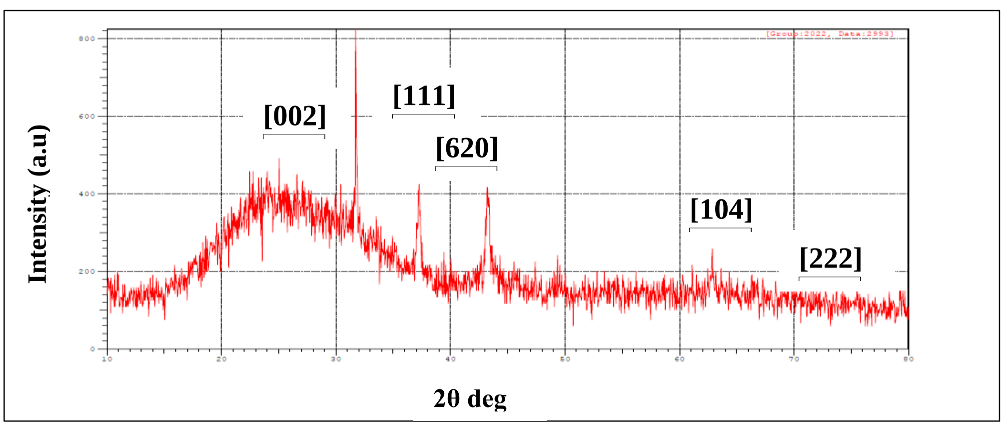


Fig(6): X- ray diffraction of $NiCl_2:Al_2O_3$ thin film (1%) at 350 ^oC.



Fig(7): X- ray diffraction of $NiCl_2:Al_2O_3$ thin film (2%).



Fig(8): X- ray diffraction of $NiCl_2:Al_2O_3$ thin film (3%) at 350 ^oC.

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Table5: Results of the effect of annealing on the pure film.

No	2θ	hkl	d(Å)	G.S(nm)	FWHM	I %	A	Sys.
1	33.653	111	2.661	36.4	0.236	98	a=3.875 b=4.552 c=6.162	Orthorhombic
2	65.099	104	1.431	50.4	0.195	6		
3	70.743	222	1.33	46.6	0.217	7		
4	28.957	002	3.081	37.1	0.229	20		
				Ave=42.652	**Ave=0.273**			

Table6: Results of the effect of annealing on doped film (1%).

No	2θ	hkl	d(Å)	G.S(nm)	FWHM	I %	A	Sys
1	46.418	022	1.954	55.8	0.472	0.613	a=3.77 b=10.365 c=4.221	Orthorhombic
2	72.96	080	1.295	42.6	0.678	0.24		
3	33.653	111	2.661	36.4	0.236	98	a=3.875 b=4.552 c=6.162	
4	65.099	104	1.431	50.4	0.195	6		
5	70.742	222	1.33	46.6	0.217	7		
6	28.957	002	3.081	37.1	0.229	20		
				Av.=44.816	**Av.=0.350**			

Table7: Results of the effect of annealing on doped film (2%).

No	2θ	hkl	d(Å)	G.S(nm	FWHM	I %	a	Sys
1	41.061	440	2.196	49.6	0.182	9.3	a=b=c=12.425	Cubic
2	33.653	111	2.661	36.4	0.236	97	a=3.875 b=4.552 c=6.162	orthorhombic
3	65.099	104	1.431	50.4	0.195	8		
4	70.742	222	1.33	46.6	0.217	5		
5	28.957	002	3.081	37.1	0.229	19		
				Av.=44.02	**Av.=0.211**			

Table8: Results of the effect of annealing on doped film (3%).

No	2θ	hkl	d(Å)	G.S(nm)	FWHM	I%	a	Sys
1	46.06	620	1.968	28.1	0.153	0.1	a=b=c=12.425	Cubic
2	48.449	622	1.877	51.2	0.945	22	a=3.875 b=4.552 c=6.162	orthorhombic
3	33.732	332	2.654	55	0.578	22		
4	65.099	104	1.431	50.4	0.195	9		
5	70.742	222	1.33	46.6	0.217	6		
6	28.957	022	3.081	37.1	0.229	5		
				Av.=43.54	**Av.=0.386**			

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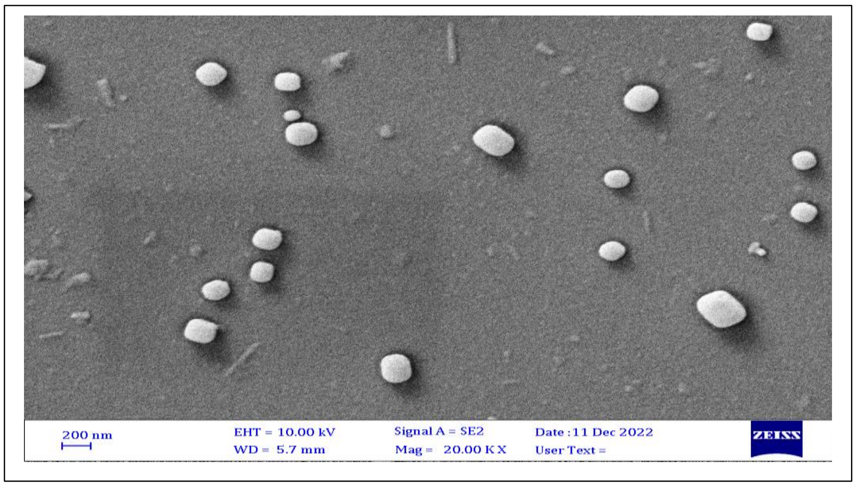
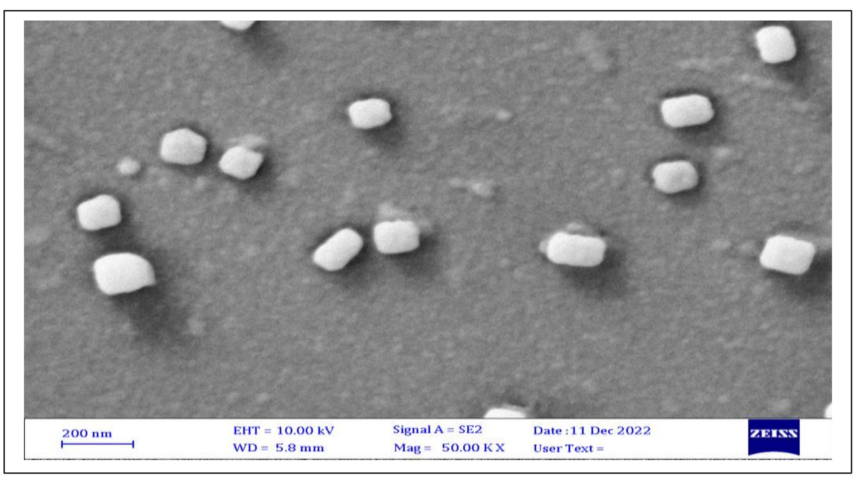
3.2. Scanning Electron Microscopy (SEM)

Scanning microscopy was used to investigate the surface shape of the samples and the dispersion of aluminium oxide (Al_2O_3) in the thin film ($NiCl_2$). Figures (10),(11),(12) and (13) show the increase in doping rates has a clear effect. Effect on surface topography. When the inoculation rates is noticed an increase in the density and grain size of the membrane. It should also be noted that the composition of the membrane is polycrystalline, where the grain boundaries can be distinguished, such as aluminium oxide when grafted. Its atoms are also present in the film with atoms of the substance ($NiCl_2$), and this increases the collisions between the molecules, which lead to the loss of the energy of the molecules in an adequate amount. to form molecular clusters where they align with each other. This agrees with refer [22-24].



Fig(9): SEM image of $NiCl_2$ thin film pure.



Fig(10): SEM image of $NiCl_2$:Al_2O_3 thin film at 1%.

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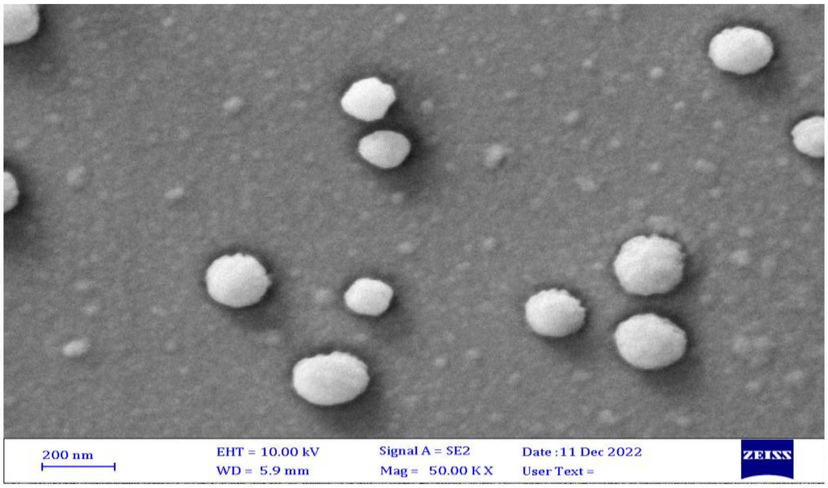
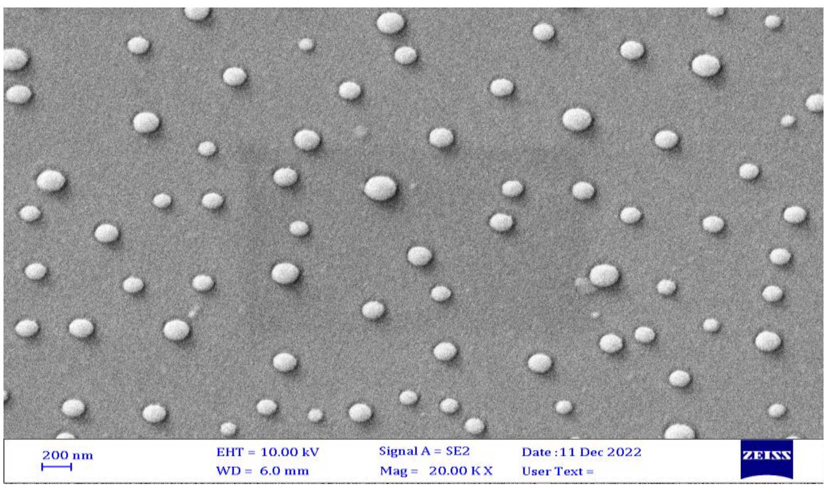


Fig(11): SEM image of $NiCl_2$:Al_2O_3 thin film at 2%.



Fig(12): SEM image of $NiCl_2$:Al_2O_3 thin film at 3%.

an annealing degree of 350 ^{0}C, where we note that the average particle size decreases with increasing temperature and (FWHM) increases, and this is consistent with the results of (XRD).

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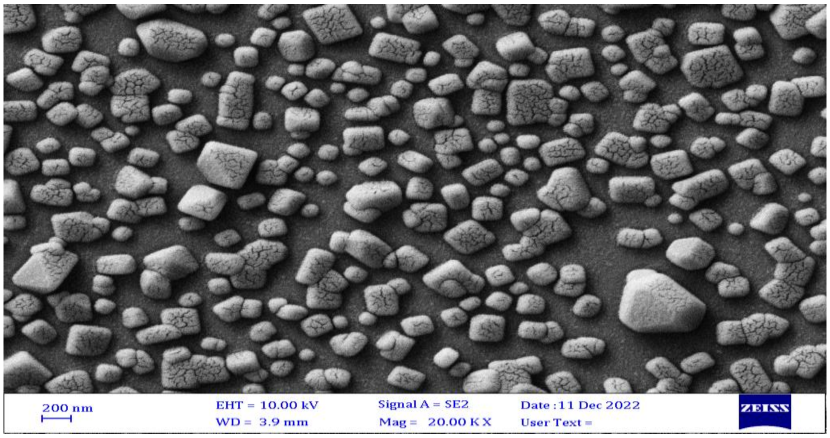
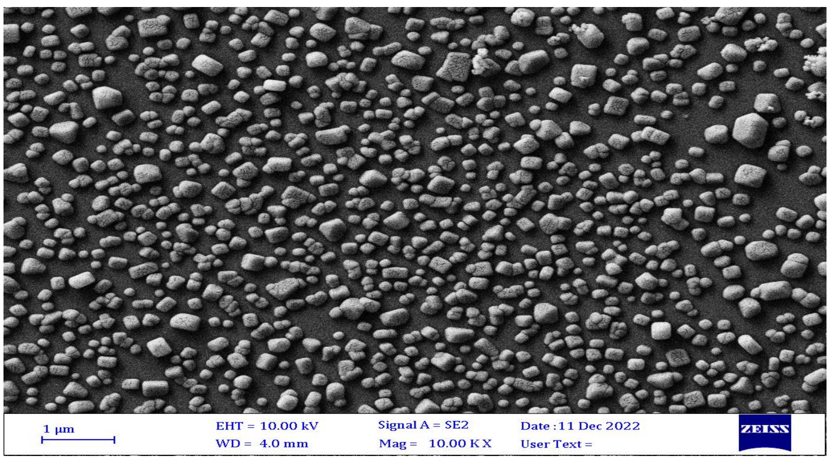
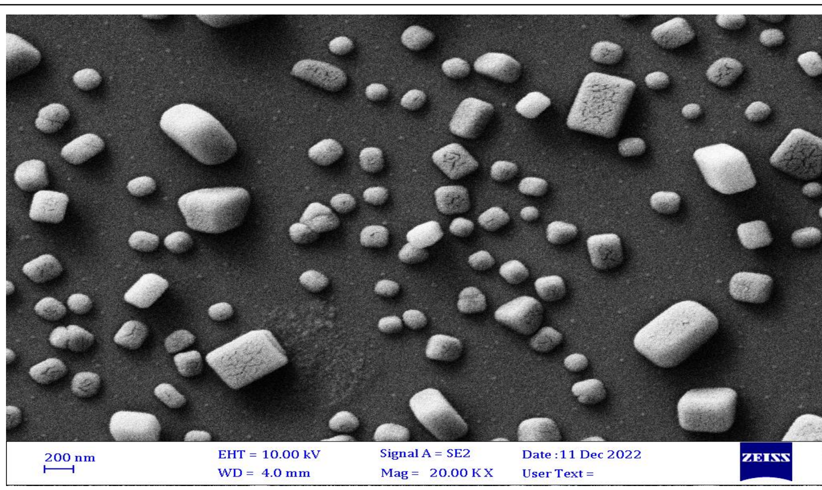


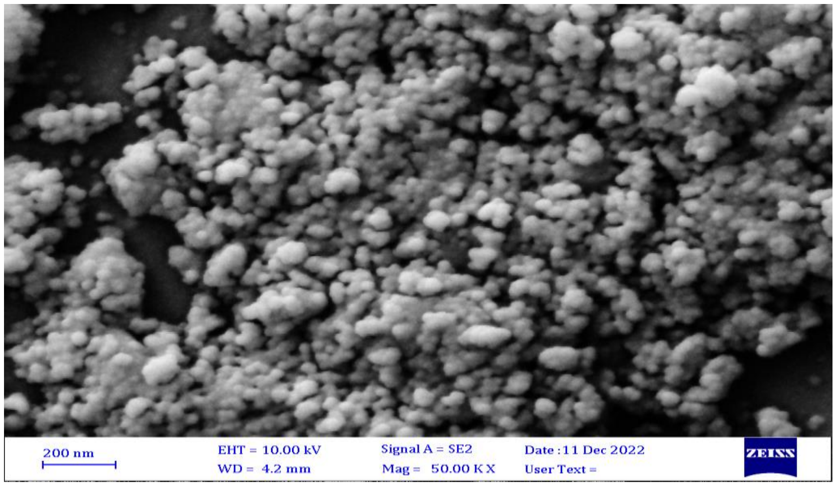
Fig(13): SEM image of $NiCl_2$ thin film (pure) at 350 ºC.



Fig(14): SEM image of $NiCl_2$:Al_2O_3 thin film (1%) at 350^0C.



Fig(15): SEM image of $NiCl_2$:Al_2O_3 thin film (2%) at 350^0C.



Fig(16): SEM image of $NiCl_2$:Al_2O_3 thin film (3%) at 350^0C.

Table9: showing the grain size of (XRD) and (SEM) measurements.

Sample	G.S (nm) (XRD)	G.S(nm)(SEM)
Pure NiCl2	43.4	35.50
1%	45.14	39.96
2%	46	72.57
3%	60.275	42.43

Table10: showing the effect of annealing at(350 ^{0}C) on the grain size of (XRD) and (SEM) measurements.

Sample	G.S (nm) (XRD)	G.S(nm)(SEM)
Pure NiCl2	42.625	33.5
1%	44.816	37.96
2%	43.542	58.23
3%	55.025	29.3

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4. CONCLUSIONS

With the addition of an impurity (Al_2O_3) results that is no change the crystalline structure of the polycrystalline ($NiCl_2$) films, but rather reduce the intensity of the peaks in both directions (241),(222) and increase the intensity of the peaks in the dominant direction and effect appears in the locations of the peaks in different proportions in a limited manner, and this will lead to the improvement of the crystal structure. The average particle size increased with the increase in the doping percentage, and it reached the largest particle size at the percentage of doping (3)%, where the particle size reached (46 nm), but when the films were annealed at a degree (350 ^{0}C), we noticed that the particle size decreased due to the slight widening of the Full Width at Half Maximum (FWHM), as the average particle size at the percentage of impregnation reached (3)% (43.542 nm).

Conflict of interests.

There are non-conflicts of interest.

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الخلاصة

تمت دراسة الخصائص التركيبية للأغشية من خلال عدة تقنيات ، ويعتبر حيود الأشعة السينية (XRD) من أكثر الطرق الموثوقة التي تم اعتمادها في هذا البحث ، لذلك سنشرحها بشيء من التفصيل ، بالإضافة إلى قياسات (SEM) التي تعطي معلومات عن مورفولوجيا السطح. المواد والطرق: تم استخدام تقنية الرش الحراري الكيميائي لتحضير الأغشية الرقيقة. النتائج: أظهرت النتائج أن زيادة درجة الحرارة أدت إلى انخفاض متوسط حجم الجسيمات على عكس عملية التشويب يعود سبب الانخفاض في حجم الجسيمات إلى زيادة العرض الكامل عند نصف الحد الأقصى (FWHM) بالإضافة إلى زيادة التبلور ، يصبح الغشاء أكثر انتظامًا وتناقص العيوب البلورية و يزيد. أيضا الحدة من القمم.

الكلمات المفتاحية : غشاء رقيق , الرش الكيميائي الحراري , التشويب , شبه موصل , التلدين

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