REPUBLIC OF TURKEY
MUĞLA SITKI KOÇMAN UNIVERSITY
GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

DEPARTMENT OF METALLURGICAL AND MATERIALS ENGINEERING

SYNTHESIS AND CHARACTERIZATION OF VANADIUM OXIDE-SILICON OXIDE COMPOSITE COATINGS BY SOL-GEL TECHNIQE

MASTER OF SCIENCE (M.Sc.)

MARWAH RAHMAN RAHI RAHI

JANUARY 2022
MUĞLA

REPUBLIC OF TURKEY
MUĞLA SITKI KOÇMAN UNIVERSITY

GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES

SYNTHESIS AND CHARACTERIZATION OF VANADIUM OXIDE-SILICON OXIDE COMPOSITE COATINGS BY SOL-GEL TECHNIQE

MASTER OF SCIENCE (M.Sc.)

MARWAH RAHMAN RAHI RAHI

JANUARY 2022
MUĞLA

MUGLA SITKI KOÇMAN UNIVERSITY

Graduate School of Natural and Applied Sciences

THESIS ACCEPTANCE APPROVAL

The thesis, submitted by **MARWAH RAHMAN RAHI RAHI,** entitled **"SYNTHESIS AND CHARACTERIZATION OF VANADIUM OXIDE-SILICON OXIDE COMPOSITE COATINGS BY SOL-GEL TECHNIQUE"** has been accepted on 12/01/2022 in fulfilment of Master's Degree in the Department of METALLURGICAL AND MATERIAL ENGINEERING by the thesis committee listed below.

THESIS DEFENCE COMMITTEE

Assist. Prof. Dr. Tolga Tavşanoğlu — Signature:
Muğla Sıtkı Koçman University, Muğla

Assoc. Prof. Dr. Aslı Çakır — Signature:
Muğla Sıtkı Koçman University, Muğla

Assoc. Prof. Dr. M. Şeref Sönmez — Signature:
Istanbul Technical University, İstanbul

DEPARTMENT APPROVAL

Prof. Dr. Ali Arslan Kaya (Head of Department) — Signature:
Muğla Sıtkı Koçman University, Muğla

Assist. Prof. Dr. Tolga Tavşanoğlu (Supervisor) — Signature:
Muğla Sıtkı Koçman University, Muğla

Date of Defence: 12/01/2022

I declare; all results, documents, information and files that I gained and presented as part of this thesis, were obtained by me within the study of this thesis and those are fines by scientific and academic ethic rules. Also, I declare; all original information and results belong to someone else that were not achieved within the study of this thesis were referenced by ethic rules of academy and science.

MARWAH RAHMAN RAHI RAHI

Day/Month/Year

To My Father Soul

ACKNOWLEDGEMENT

I would like to thank to Assist. Prof. Dr. Tolga Tavşanoğlu for the patience he showed during my studies, the facilities and opportunities he provided, and his unwavering support in all circumstances and difficulties. I am grateful to him for being my role model, which I will acquire at every step I take academically. Just like in this thesis, every success I have will have their signature under it. And I am honored and content that I have been among his students.

At the same time, I would like to thank Assoc. Prof. Dr. Aslı Çakır who did not spare her help and support in my thesis preparation process.

I am eternally thankful to my family for their love, Prayers, and care for me to educate and prepare me for my future.

To my brother (Mustafa) my soul mate, To my dear mother , The secret to my success ,To my sister(Maha) friend of my life , I will love you all to the last breath .Thanks you all for support. I dedicate this thesis to my dear father may god have mercy on you.

ÖZET

VANADYUM OKSİT – SİLİSYUM OKSİT KOMPOZİT KAPLAMALARIN SOL-JEL TEKNİĞİYLE ÜRETİMİ VE KARAKTERİZASYONU

Marwah Rahman Rahi Rahi

Yüksek Lisans Tezi

Fen Bilimleri Enstitüsü

Metalurji ve Malzeme Mühendisliği Anabilim Dalı

Danışman: Dr. Öğr. Üyesi Tolga Tavşanoğlu

Ocak 2022, 38 sayfa

Vanadyum oksit, kromik ve fotokatalitik özelliklerini veren, tersine çevrilebilir yarı iletken-metal geçiş yapma kabiliyeti nedeniyle geniş çapta çalışılmıştır. Vanadyum oksit ayrıca cam kaplama teknolojilerinde, yarı iletken endüstrisinde, pillerde ve süper kapasitörler, kataliz ve optik anahtarlama cihazları olarak çeşitli uygulamalarıyla bilinir. Öte yandan SiO_2 kaplamalar, ayarlanabilir kırılma indeksi, kontrol edilebilir mikroyapı ve görünür bölgede iyi geçirgenlik gibi benzersiz özelliklere sahiptir; bu nedenle mikroelektronik, optoelektronik, sensörler, fotokataliz, kendi kendini temizleme ve güneş enerjisi teknolojileri gibi birçok uygulama alanında kullanılmaktadırlar.

Bu çalışmada, öncül olarak metal alkoksitler, vanadyum oksit için vanadyum (V) oksitriizopropoksit ve silisyum dioksit için TEOS (tetraetil ortosilikat) kullanılarak bir vanadyum oksit-silisyum dioksit kompozit solü hazırlanmış ve V_2O_5-SiO_2 kaplamalar sol-jel daldırmalı kaplama tekniğiyle cam üzerine kaplanmıştır. SEM incelemeleri V_2O_5-SiO_2 kaplamaların nanoyapıda olduğunuı göstermiştir. XRD analizleri, kaplamaların 300 °C'de amorf olduğunu, 400 ve 500 °C'de V_2O_5 fazının elde edildiğini ve amorf SiO_2 matris çinde kristalin V_2O_5 fazlı nanokompozitler olduğunu ortaya koymuştur. Optik analizler, tavlama sıcaklıklarının artmasıyla kaplamaların şeffaflığının arttığını ve 500 °C'de tavlanmış kaplamada yaklaşık %70'lik en yüksek şeffaflığın elde edildiğini göstermiştir.

Anahtar Kelimeler: Vanadyum Oksit, Silisyum Oksit, Kompozit Kaplamalar, Sol-jel, Daldırmalı Kaplama, Mikroyapı Özellikleri, Kristal Yapı, Optik Özellikler

ABSTRACT

SYNTHESIS AND CHARACTERIZATION OF VANADIUM OXIDE-SILICON OXIDE COMPOSITE COATINGS BY SOL-GEL TECHNIQE

Marwah Rahman Rahi Rahi

Master of Science

Graduate School of Natural and Applied Sciences

Department of Metallurgy and Materials Engineering

Supervisor: Assist. Prof. Dr. Tolga Tavşanoğlu

January 2022, 38 pages

Vanadium oxide has been widely studied due to its ability to undergo a reversible semiconductor to metal transition which gives the material its unique chromic and photocatalytic properties. Vanadium oxide is also known for its various applications in glass coating technologies, semiconductor industry, in batteries and as supercapacitors, catalysis, and optical switching devices. On the other hand, SiO_2 coatings have unique properties such as adjustable refractive index, controllable microstructure, and good transmittance in the visible region; therefore they are used in many application areas such as microelectronics, optoelectronics, sensors, photo-catalysis, self-cleaning and solar energy technologies.

In this study, a composite sol of vanadium oxide-silicon dioxide was prepared by using metal alkoxides as precursor, namely vanadium (V) oxytriisopropoxide for vanadium oxide and TEOS (tetraethyl orthosilicate) for silicon dioxide and V_2O_5-SiO_2 coatings were successfully deposited on glass substrates by sol-gel dip coating technique. Microstructural analyses by SEM demonstrated the nanostructure of V_2O_5-SiO_2 coatings deposited. XRD analyses revealed that the coating is amorphous at 300 °C and V_2O_5 phase obtained at 400 and 500 °C demonstrating that coatings are nanocomposites with crystalline V_2O_5 phase in the amorphous SiO_2 matrix. Optical analyses showed that the transparency of the coatings increased with the increase in the annealing temperatures and the highest transparency of about 70% was obtained from the coating annealed at 500 °C.

Keywords: Vanadium Oxide, Silicon Oxide, Composite Coatings, Sol-gel, Dip Coating, Microstructural Properties, Crystallinity, Optical Properties

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

DC	Direct Crystallization
2Θ	Angle of X-Ray Beam
CVD	Chemical Vapor Deposition
°C	Temperature
G	Gibbs Free Energy
H	Enthalpy
IR	Infrared Radiation
LPC	Liquid Phase Crystallization
MIT	Metal Insulator Transition
MIC	Metal Induce Crystallization
M(OR)n	Metal Alkoxide
K	Transition Temperature (Kelvin)
PLD	Pulsed Laser Deposition
O	Oxygen
SPC	Solid Phase Crystallization
S	Entropy
SIO_2	Silicon Dioxide
SEM	Scanning Electron Microscope
TEOS	Tetra Ethyl Ortho Silicate
T°C	Critical Temperature
VO_2	Vanadium dioxide
V	Vanadium
XRD	X-Ray Diffraction

1. INTRODUCTION

Vanadium dioxide is a thermo-chromic material that undergoes a reversible semi-conductor-to-metal phase transition at a temperature of about 68°C (Beteille and Livage 1998). The phase transition is accompanied by changes in the electrical, optical and magnetic properties of the material (Burkhardt et al. 2002). Despite the fact that vanadium oxide has been studied extensively for over forty years, it is still not known in detail how the transition occurs (Eyert 2002). However, it is known that the transition is caused by a structural phase transformation from a low-temperature monoclinic structure to a tetragonal rutile structure at a high temperature (Eyert 2002). The monoclinic structure is non-metallic and transparent in the IR range at room temperature, but above the transition temperature (68°C) the material becomes metallic and reflects IR radiation (Livage 1999).Vanadium oxides have been extensively studied for their ability to undergo a reversible metal-insulator transition. It is also known for its various applications in glass coating technologies, semiconductor, batteries and super capacitors, catalysis, and optical switching devices (Lanwang 2018).

Since there are many stable phases of vanadium oxide that exists such as VO, V_2O_3, V_2O_5 and VO_2, various methods of preparation have been developed to produce high quality, high purity and phase specific vanadium oxides such as hydrothermal synthesis, sputtering deposition, chemical vapor deposition, pulsed laser deposition and sol-gel method (Ureña-Begara, Crunteanu, and Raskin 2017),(Öksüzoğlu et al. 2013). The sol-gel process is considered a recognized approach to the production of metal oxides, and it is a chemical route used to synthesize ceramic materials at low temperatures (usually at room temperature) based on a wet chemical. The process includes polycondensation reactions of monomers into a colloidal solution (sol), which turns into an integrated network (gel) of nanoparticles or bulk polymerized networks. The sol-gel process is widely used to make porous silica films. These films have many important technical properties, such as excellent thermal insulation, relatively low dielectric constant, continuously variable refractive index, high laser

damage threshold, good transmittance in the visible region, etc. (Dutta, Ellateif, and Maitra 2011).

On the other hand, SiO_2 coatings are increasingly used in optical, magnetic and thermal applications such as anti-reflective coatings, high reflectivity, and thermal insulations. In addition, their dielectric properties make SiO_2 coatings interesting for various microelectronic structures. Thus, it is becoming more and more attractive to use these silica films in various important fields, such as optical coatings, microelectronics, optoelectronics, photo catalysis, self-cleaning, and solar energy technologies, thermal insulation based on lightweight structural materials and other field of application (Moffatt and Wigstein 2005).

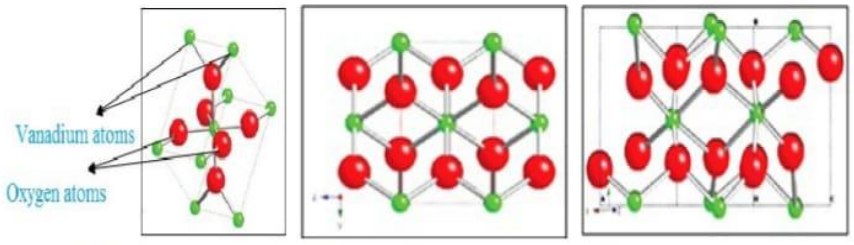
In this thesis, a composite sol of vanadium oxide-silicon dioxide was prepared by using metal alkoxides as precursor, namely vanadium (V) oxytriisopropoxide for vanadium oxide and TEOS (tetraethyl orthosilicate) for silicon dioxide. The microstructures of the coatings were analyzed by SEM and the crystallinity studies were realized by grazing-incidence XRD. Optical properties were revealed by spectrophotometer analyses.

2. THEORETICALBACKGROUND

2.1 Vanadium oxide

Vanadium (atomic number 23, elementary category transitional metal) exists in several valance (V^{+}, V^{2+}, V^{3+}, V^{4+}, V^{5+}) and mixed valance states [V_6O_{13} (V^{5+}, V^{4+}), V_8O_{15} (V^{4+}, V^{3+})]. Because of this various valance states it exhibits many compounds with oxygen like VO, VO_2, V_2O_3 and V_2O_5, etc. which have various structural, optical and chemical properties. Vanadium oxides are binary highly correlated metal oxides that change phase from semiconductor to metal in response to external influences such as temperature, electric field, or doping. The ratio of vanadium and oxygen significantly affects the structure and properties of the material (Darling and Iwanaga 2009).

Vanadium dioxide is an intelligent material that converts a semiconductor to metal. Transition (SMT) nearly at room temperature (~68 °C). It is tetragonal in the metallic state and monoclinic in the semiconducting state. Figure 2.1 and Figure 2.2 show the structural unit of VO_2 and its different phase structure (Darling and Iwanaga 2009).



(a) Unit cell of VO_2 at room temperature. (b) Tetragonal structure in metallic phase. (c) Monoclinic structure in semiconductor phase.

Figure 2.1. Unit cell structure of VO_2 in different phases. Red color indicates O_2 atoms and Green indicates vanadium atoms.

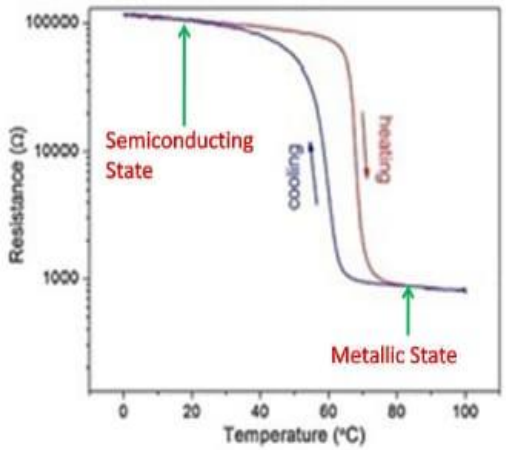


Figure 2.2. Hysteresis curves showing change in resistance as a function of temperature during phase transition in VO_2.

2.2 Vanadium Oxide Systems

Vanadium is a transition metal with the symbol V. There are more than fifteen stable vanadium oxide phases. The common oxidation states of vanadium are V^{2+} in VO, V^{3+} in V_2O_3, V^{4+} in VO_2, V^{5+} in V_2O_5.V_2O_5 is the highest oxygen state of vanadium-oxygen systems. There are V_4O_9 V_6O_{13}, and V_3O_7 intermediate states between V_2O_5 and VO_2. The intermediate phases called Magneli Phases are between the VO_2 and V_2O_3 as shown in Figure 2.3. (Park et al. 2001).

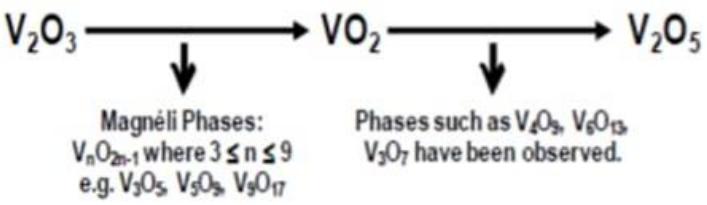


Figure 2.3. Major oxidation states and the other intermediate states of vanadium oxide.

The phase diagram of vanadium oxygen is shown in Figure 2.4. As shown in the diagram, there are three thermodynamically stable single phase fields of VO_x which are VO_2, V_6O_{13}, and V_2O_5 at about this rate.

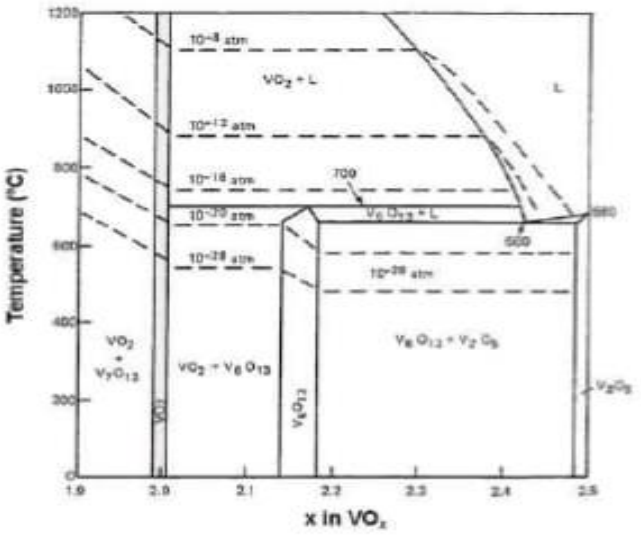


Figure 2.4. Phase diagram for the vanadium oxygen system.

Many oxygen phases of the vanadium display structural phase transition (Table 2.1).

Table 2.1.Metal – insulator transition temperatures of different vanadium oxide phases.

Oxide phase		**Transition temperature (K)**
Magneli Phases	V_2O_3	155
	V_3O_5	430
	V_4O_7	238
	V_5O_9	135
	V_6O_{11}	170
	V_7O_{13}	---
	V_8O_{15}	68
	V_9O_{17}	79
VO_2		340
V_6O_{13}		150
V_2O_5		530

There is interest in the literature in thin-film processes for processing vanadium oxides due to their widespread use. There are many possible ways to grow thin films of vanadium oxide. Chemical vapor deposition (CVD), sputtering, pulsed laser deposition, epitaxial growth and sol-gel method are the most common thin film preparation of vanadium oxides.

2.3 Properties of Vanadium Oxides

Like many transition metal oxides, vanadium (III, IV, and V) oxides have interesting properties as catalytic and magnetic materials, however, these properties and applications are not discussed here (Nag and Haglund Jr 2008). Instead, two properties of recent interest, with obvious commercial applications, will motivate the fabrication of functional devices from vanadium oxide: metal-insulator transition (MIT) and lithium intercalation properties of multiple vanadium oxide phases (Graham et al. 2003).

2.3.1 Vanadium oxide Metal-to-Insulator Transition

The MIT of V_2O_3 and VO_2 have been around for half a century. This transition is characterized by a subtle structural transformation and a change in conductivity that spans several orders of magnitude and occurs at a well-defined temperature. These and several intermediate vanadium oxides - and a number of other transition metal oxides - have this property (Pergament et al. 2013). But VO_2 is the most practical importance because its transition to 68 ° C is much closer to ambient temperature, which means that it is more readily available. This transition can be thermally, optically, or electrically induced, and the effectiveness of any device using such interactions is partially limited by the energy input required to induce the transition, which is minimized by transitions occurring near the operating temperature, it is desirable to work with any device at ambient temperature (Whittaker, Patridge, and Banerjee 2011).

One of the recent commercial endeavors that use MIT VO_2 is the development of energy efficient thermochromic window coatings (Wu et al. 2013). MIT escort -

predictable - significant change in VO_2 optical properties. In particular, the infrared (IR) reflectance of VO_2 increases significantly in the high-temperature metallic phase (Figure 2.5). Using this property, coated windows allow most of the solar energy to pass through when the temperature is below the junction temperature, and reflect most of the radiation when the temperature is above the junction temperature, creating a passive temperature mediation device. Because people prefer temperatures below 68 ° C, dopants (usually W) are used to lower the junction temperature.

The MIT in VO_2 can also be used in optical limiters and shutters. Optical limiters are used to protect sensors from saturation or damage at intensities that exceed the dynamic range of the devices. VO_2 limiters (IR) work by increasing their reflectivity - as opposed to absorption - when the incident radiation is intense. The speed of this transition (~ 0.5 PS) also makes VO_2 ideal for high speed optical shutters where a high intensity visible recording beam causes the transition and an infrared beam reads the state. These optical gates are of interest as elements in all-optical digital logic devices (Lysenko et al. 2006).

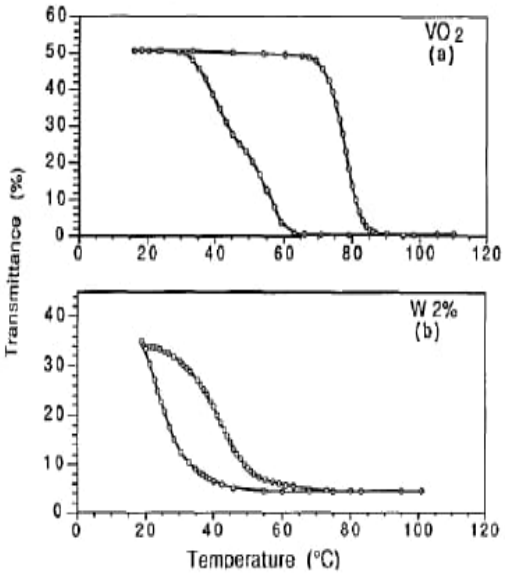


Figure 2.5. OpticalMIT in un-doped (a) and W-doped (b) VO_2 films.

Electronic logic gates are based on intermittent changes in conductivity. In modern transistor technology, this is done by changing the carrier density in the semiconductor contact using the gate voltage, but any material or device geometry that can reliably change its conductance by a significant amount is compatible with

existing logic architectures. Resistance switching in VO_2 (Figure 2.6) represents one possible mechanism for such devices (Koza et al. 2011).

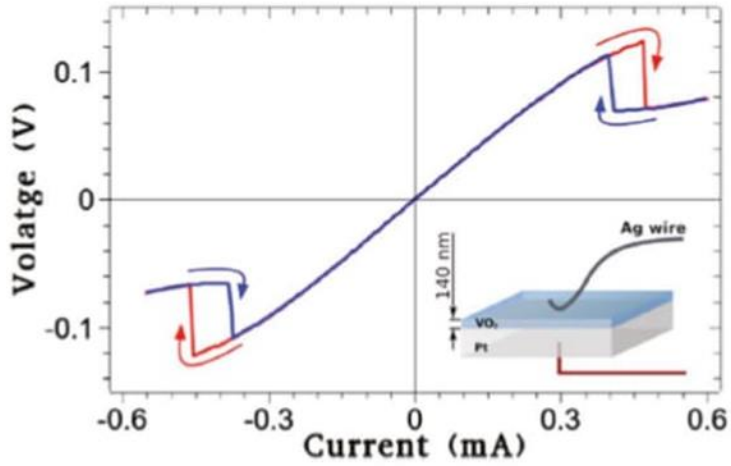


Figure 2.6. I-V curve for an electrodeposited VO_2 film showing resistance switching hysteresis (Koza et al. 2011).

2.3.2 Li Intercalation

Vanadium oxides have been studied as potential cathodes with lithium intercalation. They operate over a reasonable voltage range (1.5-3.5 V relative to Li) and have a high theoretical capacitance. Unfortunately, these materials generally exhibit unacceptably low cycle life (Pistola et al. 1985). Recent studies on nanostructured materials indicate that improvements in cyclic damping are possible, suggesting that vanadium oxide materials may become commercially viable.

Initial efforts were focused on $Li_2V_6O_{13}$, which has a random metallic conductivity property (most intercalation cathodes have poor conductivity, increasing the internal resistance at the cathode-collector interface). However, conductivity decreases with the addition of lithium, and the material does not have a satisfactory cycle life. Other tested vanadate's (LiV_3O_8, V_2O_5, VO_2) also have short cycle life, but studies with nanoparticles and amorphous vanadate show improved cycling performance (Scrosati and Garche 2010).

The use of nanostructured materials to improve the performance of electrode materials has proven effective in other systems, including commercially successful materials. $LiNi_\alpha Mn_\beta Co_\gamma O_2$ (NMC) cathode materials have different properties

depending on the ratio of transition metals: a composition with a high capacity tends to break down at the electrolyte interface, while a composition with a lower capacity has excellent stability with respect to the electrolyte. By covering a high-capacity material with a low-capacity cladding, silicon anodes have a much higher capacity than graphite anodes, but undergo significant expansion (about 300%), which leads to excessive deformation and destruction when lithium is introduced. Nanoparticles have internal resistance to deformational fracture, which makes them ideal for systems suffering from deformational fracture, which explains the interest in silicon nanowire anodes (Song et al. 2010).

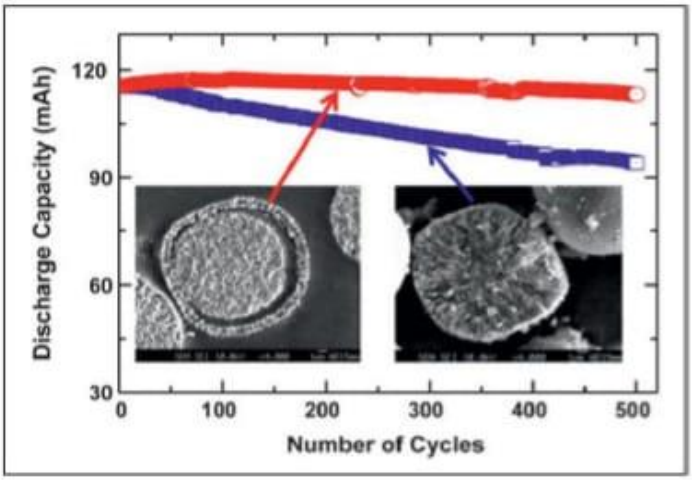


Figure 2.7.Thecycling characteristics of NMC (blue) and NMC core-shell (red) cathode materials with accompanying SEM images (Song et al. 2010).

Although vanadate cathode materials suffer from poor cycling characteristics, attributed primarily to strain during Li insertion and extraction, they compare favorably with commercial cathodes ($LiVO_2$-$LiCoO_2$) and the possibility of a very high charge capacity in some cases - make them remain a subject of interest as can be seen in Figure 2.7. Advances in nanostructured and composite electrode materials suggest that the reduction in vanadate capacity can be reduced.

2.3.3 Physical Properties

Vanadium oxide is a p-type material with a narrow band gap (0.7 eV) that exhibits a sharp change in resistance and a change in optical properties in the 0.8-2.2 μm region

due to phase transition the first kind, that is, from the semiconductor to the metallic phase transformation. The phase transition also affects its magnetic properties. As a transition metal oxide with narrow d-electron bands, this highly correlated electronic system is extremely sensitive to small changes in external parameters such as temperature, pressure, or doping. The VO_2 phase transition shows a change in resistance from 103 to 104 with a thermal hysteresis of the order of 3 to 10 ° C (Chakraborty 2014).

2.3.4 Parameters affecting phase transition

Phase transition of vanadium oxide depends upon lot of factors. Some of the parameters are

1. Vanadium to oxygen ratio: The ratio of oxygen to vanadium has been shown to affect the properties of the phase transition even with small deviations from the correct stoichiometry. It is also believed that this ratio is closely related to grain size. Therefore, good control of the deposition parameters is required to obtain a film with optimized properties.

2. Partial pressure of oxygen: When a reactive gas is introduced into the chamber during deposition, the oxygen partial pressure is a key factor to control stoichiometry. Electrical measurements show that this affects both the critical junction temperature and the amplitude of the resistivity change. Indeed, a change in the oxygen content leads to deformation of the unit cell and / or to the coexistence of various vanadium oxides.

3. Grain size: Since the individual grain size is smaller in oxygen-deficient films, void columnar boundaries are believed to be responsible for the increase in resistivity in the metal phase. In thin films, it has also been shown that the crystallite size affects the external properties of the material by changing the propagation of the transition front.

Many other parameters such as deposition rate, deposition temperature, substrate, etc. also affect the structure and stoichiometry of the film.

2.3.5 Applications

Smart windows, optical switches, high reflectivity surfaces, IR protecting glasses, thermal sensors, Li battery and many more where the application of vanadium oxide where the nonlinear phase changing properties is utilized (Reddy et al. 2009).

2.4 Sol-gel and Thin Film Coatings

A sol-gel process is a chemical pathway used to synthesize ceramic materials at low temperatures (usually room temperature) based on a wet chemical. The process includes poly condensation reactions of monomers into a colloidal solution (sol), which turns into an integrated network (gel) of nanoparticles or bulk polymerized networks. A sol, which is a liquid suspension of solid particles ranging in size from 1 nm to 1 micron, can be prepared by hydrolysis and partial condensation of a precursor such as an inorganic metal salt or alkoxide. Further condensation of sol particles into a three-dimensional network leads to the formation of a gel, which is a two-phase material with a solid encapsulating the solvent (Ertl, Knözinger, and Weitkamp 2008).

The two main advantages of the sol-gel process are the ability to incorporate various organic or inorganic functional additives into the sol-gel matrix, and the ability to obtain a universal final configuration such as monoliths / bulk glasses, thin films / coatings, nanoparticles and fibers

A sol is a stable dispersion of colloidal particles or polymers in a solvent. The particles can be amorphous or crystalline. Aerosol is particles in the gas phase and sol is particles in liquid (Brinker 2013). The gel consists of a three-dimensional continuous mesh, which contains a liquid phase. In a colloidal gel, the network is built from an agglomeration of colloidal particles. In a polymer gel, the particles have a polymer substructure consisting of aggregates of sub colloidal particles. Usually, sol particles can interact due to van der Waals forces or hydrogen bonds. The gel can also be formed from the binding of polymer chains. In most gel systems used to synthesize materials, interactions are covalent and gelation is irreversible. The gelation process can be reversible if other interactions are involved.

Materials obtained by the sol-gel method have found various applications in the fields of chemistry, biochemistry, engineering, and materials science. This particular attention is due to the simplicity of preparation and modification of the parameters of the sol-gel technology.

Advantages of the sol-gel process

- ✓ High purity homogeneous materials are obtained;
- ✓ The reaction control is done by using chemical techniques;
- ✓ It allows the formation of a pre-inorganic network in solution;
- ✓ The densification to inorganic solid is allowed at comparatively low temperatures due to the pre-inorganic network;
- ✓ It allows the synthesis of special materials such as films, reactive ceramic powders, fibers;
- ✓ Formation of new crystalline phases from the non-crystalline solids is obtained (Milea, Bogatu, and Duta 2011).
- ✓ Energy saving and minimizing the evaporation losses are provided thanks to low processing temperatures
- ✓ Provides cleanliness by avoiding contamination of containers during heat treatment, prevents unwanted separation and crystallization of micro phases (Milea et al. 2011).

On the other hand, the disadvantages of the sol-gel process;

- ✓ Cost of the precursors is high;
- ✓ Process duration is long;
- ✓ The synthesis of monoliths is difficult;
- ✓ The process chemistry with respect to properties control and reproducibility is difficult.
- ✓ A thick coat is created to provide corrosion protection and a thin tie coat is created to provide excellent adhesion between the metal substrate and the top coat (Pathak and Khanna 2012).

Sol-gel process of thin films includes the following steps:

- ✓ A sol is prepared,
- ✓ The sol become gel on a substrate,
- ✓ The gel dries and densifies by sintering.

These steps are illustrated in Figure 2.8.

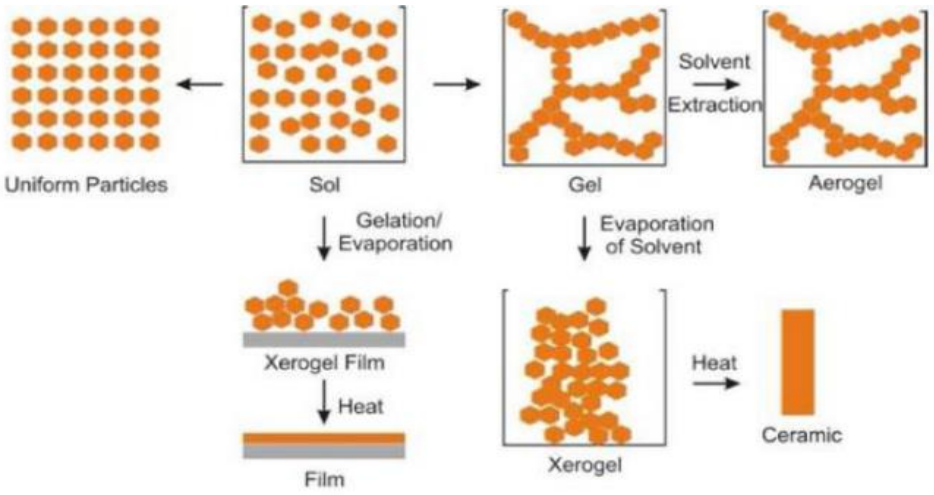


Figure 2.8. Sol-Gel Technology Scheme.

2.4.1 Components in Sol-Gel Method

2.4.1.1 Precursors

Typically in the sol-gel process, inorganic (salt) or organometallic precursors are used. Since the type of precursor solutions affects the structure of the condensed phase during the coating process The main formula of metal alkoxide is M(OR)n

M; to be coated metal

R; alkyl group like CH_3 (methyl), C_2H_5 (ethyl)

n; valence number of metal (Pathak and Khanna 2012).

2.4.1.2 Alcohol

Alcohols are molecules that are formed by adding an OH group to another molecule or alkyl. They are used as starting material and react with metal oxide. The most

common alcohols: CH_3OH (methanol), C_2H_5OH (ethanol), C_3H_7OH (propanol), C_4H_9OH (butanol) (TOYGUN, KÖNEÇOĞLU, and KALPAKLI 2013).

2.4.1.3 Catalyzer

These materials accelerate the reaction without reacting. They are divided into two categories that are acid and base) (TOYGUN et al. 2013).

2.4.2 Sol-Gel Chemistry

The sol–gel process can be summarized in the following key steps:

- ✓ Synthesis of the 'sol' from hydrolysis and partial condensation of alkoxides.
- ✓ Formation of the gel via poly condensation to form metal–oxo–metal or metal– hydroxy–metal bonds.
- ✓ Synthesis or 'aging' where condensation continues within the gel network, often shrinking it and resulting in expulsion of solvent
- ✓ Drying the gel either to form a dense "xerogel" via collapse of the porous network or an aerogel for example through supercritical drying.
- ✓ Removal of surface M-OH groups through calcination at high temperature
- ✓ $M(OR)n + nH_2O \rightarrow M(OH)n + nROH$ (hydrolysis).

  $M(OH)n \rightarrow MOn/2 + (n/2)H_2O$ (condensation).

  $M(OR)n + (n/2)H_2O \rightarrow MOn/2 + nROH$ (overall).

After preparing sol, thin films can be obtained by spin coating, dip coating and spray-up techniques (Tech n.d.).

For sols, various thin-film coating methods are applicable. Spin coating, immersion and spray are the main coating methods. Spin coating and spraying are used to produce coatings on only one side of the substrate material. Because of its simplicity and controllability, spin coating technique is mostly used for one sided coating deposition.

The spin coating starts with the deposition of the sol on to the wafer. The spin coater starts to spin up and the solution covers the whole surface of the wafer. In spin off

phase, maximum spinning speed is achieved and the solution starts to get thinner like a sheet on a table. At the last phase of the spin coating evaporation is occurred. Figure 2.9. shows the illustration of spin coating steps (Sakka 2005).

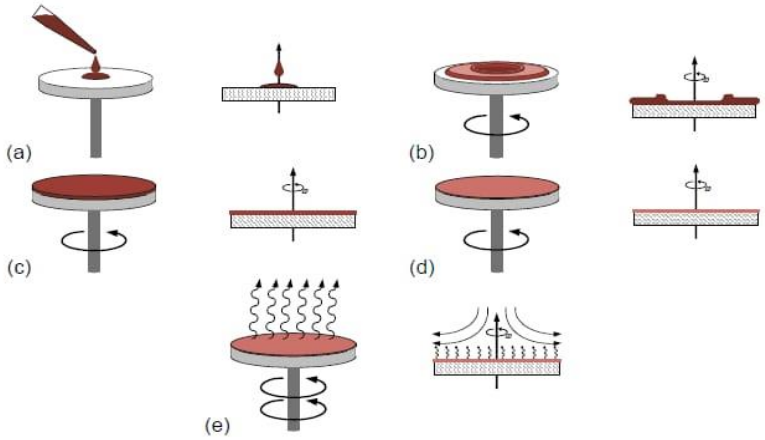


Figure 2.9. (a) deposition (b) spin up (c) spin off phase-1 (d) spin off phase-2 (e) Evaporation.

Dipping is a two sided coating method for sol applications. The substrate is dipped in to the sol and then removed from the sol to outside of the sol containing cup. The excess of the sol is dropped and evaporation occurs. Figure 2.10. shows the dip coating process steps.

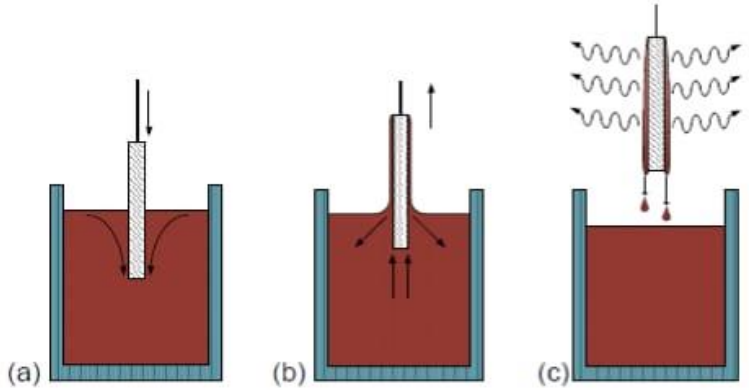


Figure 2.10. Dip coating processes steps (a) dipping (b) wet layer formation (c) Solvent evaporation.

2.5 Vanadium oxide thin film coatings

The structure and morphology of vanadium oxide films are intimately related to the deposition method and the operating conditions. Where noted that the choice of a

deposition method depends of the thin film application. For instance, electrodeposition (Vernardou et al. 2012) for electrochromic V_2O_5 films, reactive sputtering, a sol-gel, hydrothermal method, requiring large surface coatings, was performed. During the deposition process in vacuum or in a reducing atmosphere, oxygen atoms are removed from the film network when V_2O_5 is heated above its melting point, which causes the formation of defects or reduced VO_2 phases. Therefore, structural and morphological disturbances and phase instability can be controlled by appropriate deposition parameters (Mjejri et al. 2016).

While mainly thin films of vanadium dioxide are used for smart windows. VO_2 films can affect the flow of solar heat into buildings under construction, thereby reducing the need for heating in winter and cooling in summer. For example, VO_2 thin films can be recycled as an energy-saving material in aircraft, home and vehicle construction by altering infrared light, where VO_2 is an inorganic impurity with a dark blue solid and is usually a blue color variant. VO_2 is amphoteric, dissolves in alkali to impart brown color $[V_4O_9]^{2-}$ ion, or at high pH $[VO_4]^{4-}$ and in non-oxidising acids to accord the blue vanadyl ion, $[VO]^{2+}$. VO_2 has a phase passage extremely near to room temperature (陳劭恩 2017).

VO_2 can act as extremely fast optical shutters, infrared modulators, and optical modulators for cameras, data storage, missile guidance systems, and other applications.

VO_2 is a fully interacting metal oxide with a metal-to-insulator passage at 67°C, which can also operate quickly due to the ultra-short laser pulse, making it highly suitable for a variety of switching applications including power saving applications. VO_2 films can modulate the increase in solar heat onto the glass surface to reduce cooling requirements. The thin film thickness parameter is used to measure the VO_2 transition temperature. Previous studies show that the materials used and the thin film thickness parameter influencing the transition temperature result either increase or decrease (Xie et al. 2018).

For dip-coating techniques, the withdrawal speed influences the formation of the resulting thin films a low deposition rate produces thin films with a low sample throughput, by increasing the deposition rate, sample throughput is improved but the resultant thin films are often thicker. While the advantages of spin coating are the

ability to produce uniform films quickly and easily, ranging from several Nano meters to a few micron thicknesses. Spin coating is one of the most common techniques for changing the related parameter between thickness and rpm. As the value of rpm increasing, the thickness is either decreasing or not depending on the material used (Liu et al. 2016).

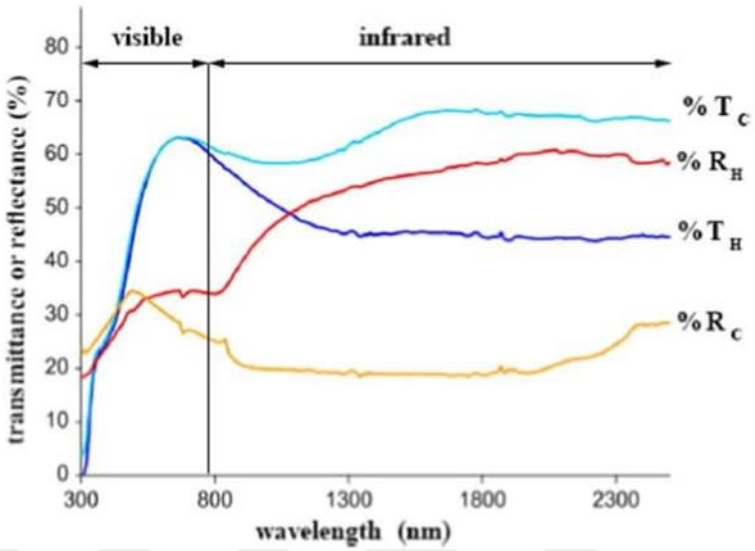


Figure 2.11. Spectral reflectance (R) and transmittance (T) forVO_2 film above (H) and below (C) T_t.

2.6 Silicon Dioxide (SiO_2)

Silica occurs in nature mainly in two forms: crystalline and amorphous, and the crystalline form has three subclasses. The three main forms of crystalline silica - quartz, tridymite, and cristobalite - are stable at various temperatures and have subdivisions.

Silica is most often found in nature in the form of quartz, as well as in various living organisms. In many parts of the world, silica is the main component of sand. Silica is one of the most complex and widespread families of materials, which exists both in the form of several minerals and is produced synthetically. Known examples include fused silica, crystals, fumed silica, silica gel, and aerogels. Applications range from materials of construction to microelectronics and components used in the food industry. There are three crystalline forms of silica; quartz, tridymite, and cristobalite. The first industrial applications of crystalline silica were probably

associated with metallurgical activities and the production of glass in three to five thousand years BC. Throughout history, it has continued to support human progress, being a key raw material in the industrial development of the world, especially in the glass, foundry and ceramic industries. Silica is contributing to today's information technology (IT) revolution by being used in plastics for computer mousses and providing raw materials for silicon chips (Ye et al. 2013).

Silica occurs in nature mainly in two forms: crystalline and amorphous, and the crystalline form has three subclasses. The three main forms of crystalline silica — quartz, tridymite, and cristobalite — are stable at various temperatures and have subdivisions. For example, geologists distinguish between alpha and beta quartz. When low temperature alpha quartz is heated at atmospheric pressure, it converts to beta quartz at 573 ° C. At 870 ° C, tridymite is formed, and cristobalite at 1470 ° C. The melting point of silica is 1610 ° C, which is higher than that of iron. , copper and aluminum, and this are one of the reasons it is used to produce molds and cores for the production of metal castings.

The crystal structure of quartz is based on four oxygen atoms bonded together to form a three-dimensional shape called a tetrahedron, with one silicon atom in the center. The myriads of these tetrahedra are connected to each other by angular oxygen atoms to form a quartz crystal. Quartz is usually colorless or white, but is often colored by impurities such as iron, and then it can be of any color. Quartz can be transparent or translucent, so it is used in glass production and has a vitreous luster. Quartz is a hard mineral due to the strength of the bonds between atoms and it scratches glass. It is also relatively inert and does not react with dilute acid. These are valuable qualities in a variety of industrial applications. Depending on how the silica precipitate was formed, quartz grains can be sharp and angular, semi-angular, semicircular, or round. Foundry and filtration require partially rounded or rounded grains for best performance (Bhattacharyya et al. 2014).

- Crystalline silicon dioxide dissolves very slowly in hot alkaline solutions, while its amorphous form reacts with alkalis at room temperature.

- Quartz does not react with other acids but it dissolves in hydrofluoric acid, and the reaction proceeds at room temperature..

-Silicon dioxide reacts with molten sodium and potassium carbonates.

-At very high temperatures above 600 °C, quartz reacts with alkaline substances such as limestone or calcium carbonate.

- Natural silicon dioxide or quartz reacts with carbon at about 2000 ° C to form silicon. This reaction is used to extract silicon from ore...

- Under high temperature and pressure conditions, silica is hydrolyzed by water to form hydroxide of silicon, which is highly unstable.

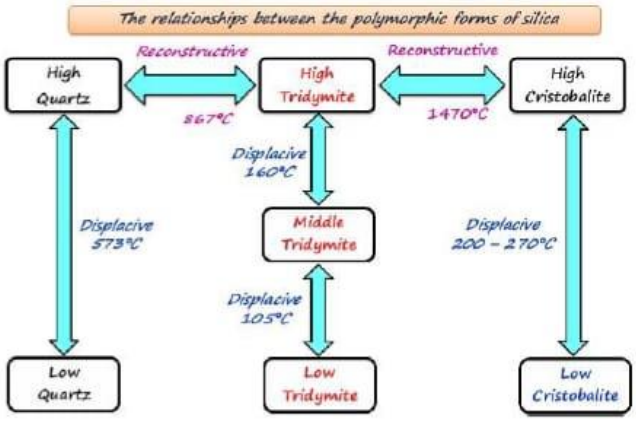


Figure 2.12. The Phase Transition of Silica.

2.6.1 Structure of Silicon Dioxide (SiO_2)

Tetrahedral arrangement with one silicon bonded to four oxygen atoms. Most of the oxygen atoms will be bonded to two silicon atoms, so that the two tetrahedra will join at the corner (bridging atoms).

The orientation can be random, resulting in an amorphous structure. Some oxygen atoms will only bond to one silicon atom (not crossed atoms). The relative number of bridges to the absence of bridges determines the "quality" of the oxide. If all oxygen atoms are connected by a bridge, a regular crystal structure is obtained – quartz (Qin et al. 2019).

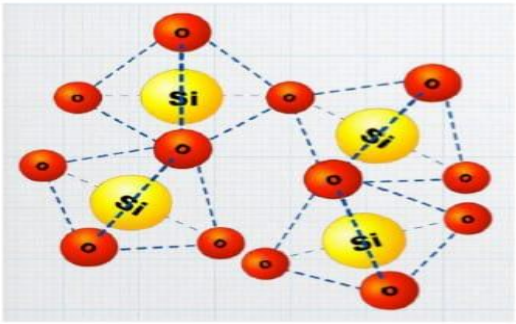


Figure 2.13. Structure of SiO_2

2.6.2 Uses of Silica

Silicon dioxide is used in many fields such as; engineering, electronics, glass for Windows, metal alloys, sandblasting etc.

- ✓ Quartz is used in the glass industry as a raw material for glass production.
- ✓ Silica is used as a raw material for the production of concrete..
- ✓ Silica is added to varnishes due to their hardness and resistance to scratch.
- ✓ Amorphous silica is added as fillers to rubber as a filler in tire manufacturing. This helps to reduce the fuel consumption of the vehicle
- ✓ Silica is used in the production of silicon.
- ✓ Since silicon dioxide is a good insulator, it is used as a filler in electronic circuits.
- ✓ Because of its piezoelectric properties, quartz is used in transducers.
- ✓ It is a substance used for making optical fibers.
- ✓ Due to its ability to absorb moisture, silica is used as a desiccant (Basu and Kanungo 2011).

2.6.3 SiO_2 Thin Film Coatings

Despite great advances and expectations raised by other materials, silicon is still the material of choice for further research in critical technologies. Si/SiO_2 thin films,

which have selective optical properties, show promising potential in semiconductors, optoelectronics and telecommunications (Dubey, Jhansirani, and Singh 2017). Various synthesis methods are used such as thermal oxidation, thermal evaporation, ion-assisted deposition, ion beam sputtering, magnetron sputtering, plasma-enhanced chemical vapor deposition, sol-gel and pulsed-laser deposition (Fardad 2000). The films with high thermal stability, amorphous structure, and corrosion resistance are used in the structure of optical multilayer films, optical filters, and other optical components. Specifically, this reflection study is of importance to develop anti-reflection coatings and to improve the efficiency of solar cells. Porous silicon has excellent broadband anti-reflective properties (Priolo et al. 2014).

Silicon is the most suitable semiconductor, attracting a lot of attention due to its band gap of 1.12 eV at room temperature, high chemical stability and high availability. Reducing the material use and manufacturing costs of silicon solar cells, along with efficiency gains, are major challenges in reducing the cost of solar energy to make it competitive with conventional energy sources. The existing technologies for fabricating Si thin films are Plasma enhanced CVD, Hotwire CVD, Catalytic CVD and sputtering (Murmu et al. 2019). However, production cost is still very high due to the large capital investment for these fabrication setups. Beside solar application of Si thin films, new findings such as luminescence from Si nanoparticles, engineered Si nanoparticles have increase hope to fabricate other electronic devices such as LED, LASERs etc.

Also the fabrication of specially engineered nanoparticles for these applications required an additional method of fabricating devices from them, such as solution processing methods. Most of the solution processing methods, such as sol-gel chemical bath deposition, centrifugation, etc., have the disadvantages of agglomeration, problems with poor film quality (Becker et al. 2009). Along with this, the volume fraction of crystals is another problem that reduces the performance of devices consisting of these thin films. Solid phase crystallization (SPC), direct crystallization (DC), metal induced crystallization (MIC) and liquid phase crystallization (LPC). However, attempts to make Si thin films by this method remain in effect (Patients et al. 2012).

And tunable optical properties can help fulfill the dream of inexpensive Si-based thin-film devices. Spraying based solution process method can be used to fabricate

the thin films for further advancement in this direction to fabricate colloidal quantum dot solar cells using spray coating and traditional spin coating methods (Becker et al. 2013).

The following figures (Figure 2.13, Figure 2.14) show transmission curves for comparing thin films of SiO_2 coated by centrifugation and applied by dipping from the same gels. It can be found that a thin film of SiO_2 spun from Gel A with a high precursor concentration has a better transmittance than that applied by immersion, but the opposite is true for thin SiO_2 films deposited from the other two gels with a low precursor concentration. In addition, it can be seen that all of the SiO_2 thin spun films have a thicker film than the dip coating for each spin coating or dip coating (Kramer et al. 2015).

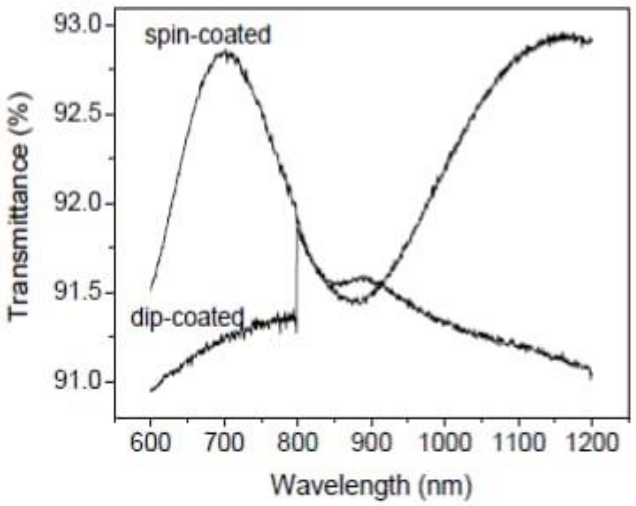


Figure 2.14. Transmittance spectra of spin-coated and dip-coated SiO_2 thin films from gel A (1:7:7).

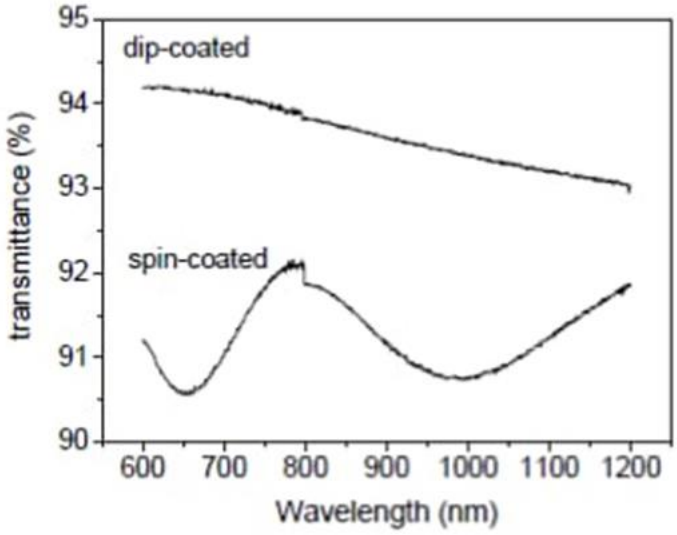


Figure 2.15. Transmittance spectra of spin-coated and dip-coated SiO_2 thin films from gel B (1:15:15).

3. EXPERIMENTAL STUDIES

In this chapter, the details related to the solution preparation, specimen preparation, coating deposition and post-treatments are given. The details of the characterization methods used are also explained.

3.1 Solution Preparation

Vanadium (V) oxytripropoxide and tetraethyl orthosilicate (TEOS) were used as precursors for the preparation of vanadium oxide – silicon oxide composite sol. Ethanol was used as solvent and HCl as catalyst for this solution preparation. All the chemicals were received from Sigma-Aldrich and were used as received. Molar ratios of 1:1:40 were used for Vanadium (V) oxytripropoxide: TEOS: Ethanol and HCl was added dropwise until the pH of the solution reached 0.1. In Figure 3.1 Daihan Scientific MaXtir™ 500S magnetic stirrer and Metler Toledo S210-U model benchtop pH meter used in the preparation of vanadium oxide – silicon oxide composite sol can be seen.

Figure 3.1. (a) magnetic stirrer (b) benchtop pH meter.

Step by step solution preparation was realized in the following way; first ethanol was put into a beaker that was on the magnetic stirrer, TEOS was added while stirring at low speeds (300 rpm), and then vanadium (V) oxytripropoxide was added and stirred during 15 minutes. After the homogenization, HCl was added dropwise until the pH reaches 0.1. The final solution having a pH value of 0.1 was stirred during 3 hours at increased speeds (700 rpm) and aged 7 days before the deposition.

3.2 Substrate Preparation

Soda-lime glasses were used as substrates in this study. Cleaning of the substrates is very important to get reliable results. Uncleaned samples result with inhomogeneous coatings and/or the cracking of the coatings. For that reason, all substrates were cleaned in an ultrasonic bath, in acetone, ethanol and distilled water respectively. Each cleaning stages took 10 minutes. In Figure 3.2 ultrasonic cleaning bath can be seen.

(a) **(b)**

Figure 3.2. Cleaning of the substrates: (a) substrates, (b) soda_lime glasses.

3.3 Coating process

Teknosem TDC-10 model dip coating machine was used to deposit coatings from the composite vanadium oxide – silicon oxide sol. Dipping speed of 60 mm/min was used for the depositions. 1 dipping-rising cycle were realized for the coatings. The substrates were dried in ambient atmosphere for 24 hours without using any external heating. Dip coating machine used in this study can be seen in Figure 3.3.

Figure 3.3. Dip coating machine.

3.4 Post-treatment of the coatings

Substrates were annealed at 300, 400 and 500 °C during 1 hour at atmospheric conditions to understand the effects of the temperature on the structural, optical and crystalline properties of the coatings. According to the annealing temperatures, deposited coatings were labeled as VS_1 for vanadium oxide – silicon oxide coating annealed at 300 °C, VS_2 for vanadium oxide – silicon oxide coating annealed at 400 °C and VS_3 for vanadium oxide – silicon oxide coating annealed at 500 °C.

3.5 Characterization of the coatings

Microstructural features of the coatings were revealed by Zeiss LEO 1530 model SEM observations. Grazing-incidence XRD (Panalytical, X'Pert MPD) analyses were realized for phase analyses, in $2\theta/\Omega$ scan mode where 2θ is the scanning angle selected between 10-80 and Ω is the x-rays coming angle and selected as 0.5, a very

low angle to get signals only from the coating. Optical properties of the coatings were investigated by Jasco V-530 UV/VIS spectrophotometer measurements.

4. RESULTS & DISCUSSION

In this chapter, microstructural, optical and crystallinity properties of the coatings deposited will be presented and discussed.

4.1 SEM Observations

Microstructural features of the coatings were observed by SEM analyses and the results are presented in Figure 4.1.

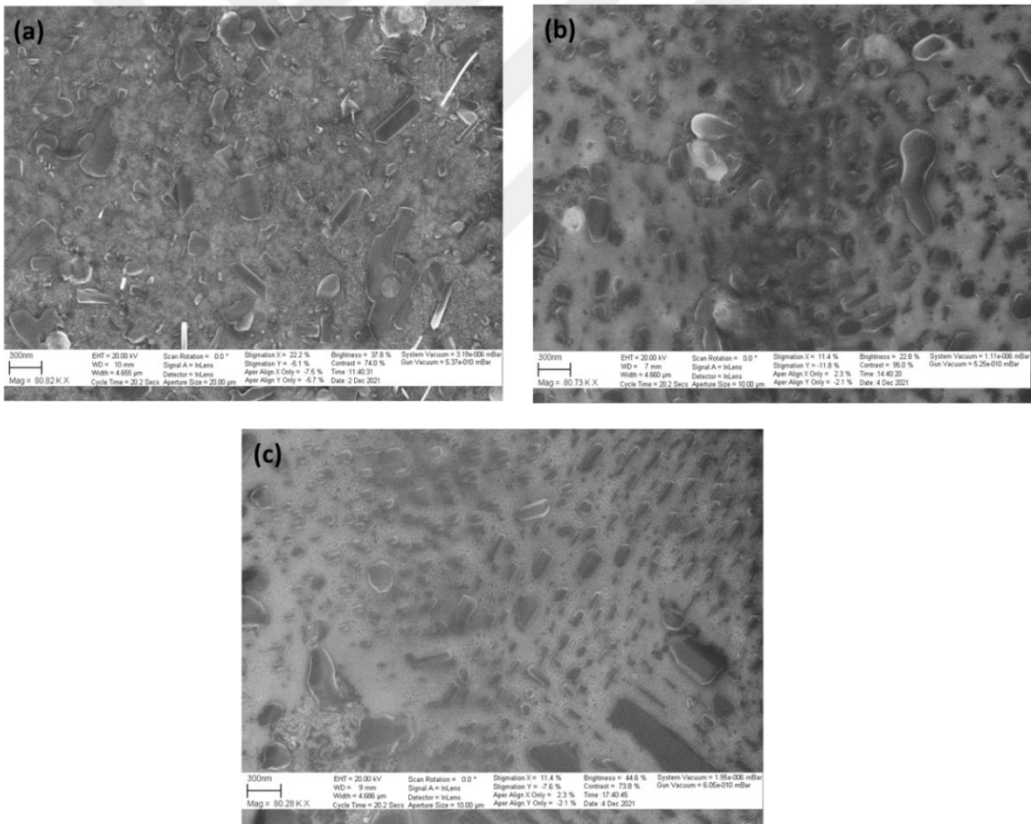


Figure 4.1. SEM microstructures of (a) VS_1 (b) VS_2 and (c) VS_3.

Nanostructured nature of the composite vanadium oxide – silicon oxide coatings can be seen from the figure. According to the figure, the annealing temperatures of 300, 400 and 500 °C did not cause any changes on the microstructures of the coatings.

The film thicknesses were also observed and are around 100 nm for each coating, however the pictures are not presented because of the clarity reasons due to the charging problems encountered during the observations.

4.2 XRD Analyses

Grazing-incidence XRD analyses were realized to detect the crystalline properties of vanadium oxide-silicon oxide composite coatings and the results are presented in Figure 4.2.

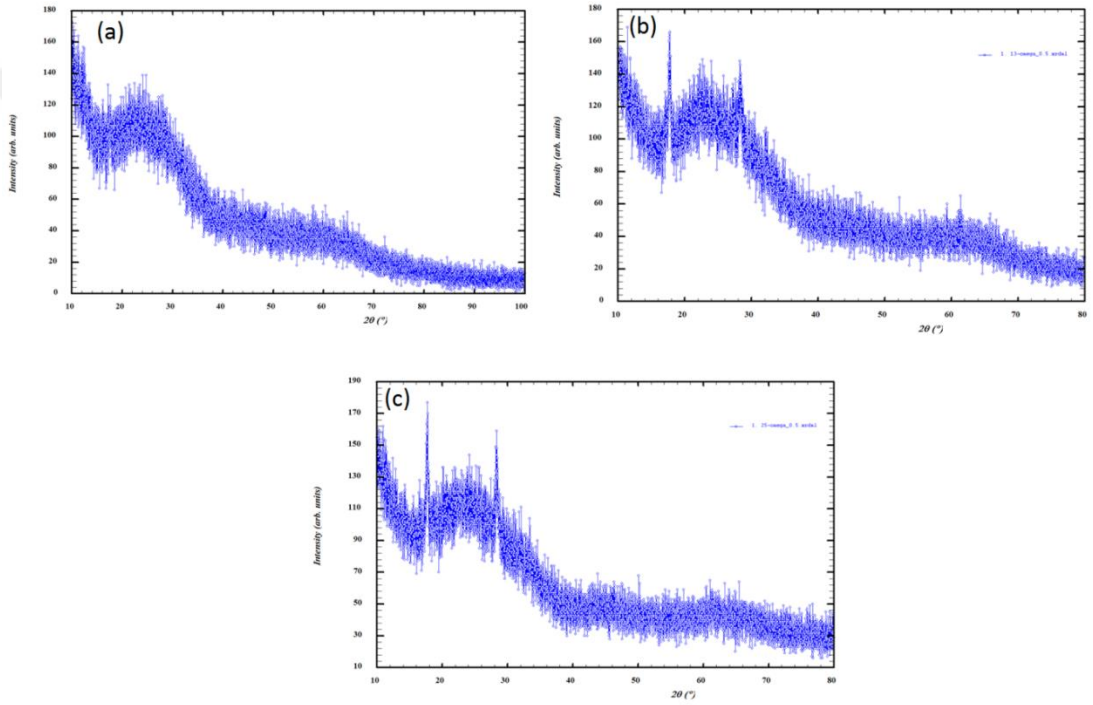


Figure 4.2. XRD spectra of (a) VS_1 (b) VS_2 and (c) VS_3.

As can be seen from the figure, the coating annealed at 300 °C is amorphous whereas there are crystalline patterns at 400 °C and 500 °C. To clearly detect and identify the patterns present in the figure (b) and (c) Rietveld refinement was realized on the raw XRD data and the results are presented in Figure 4.3. According to the figure, the peaks belong to V_2O_5 demonstrating that coatings annealed at 400 and 500 °C are nanocomposites with crystalline V_2O_5 phase in the amorphous SiO_2 matrix.

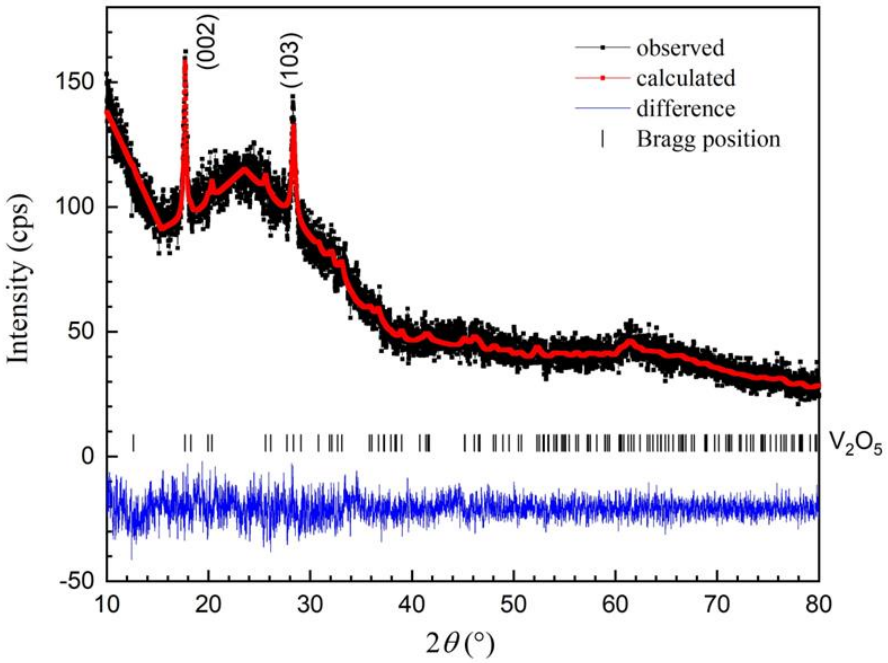


Figure 4.3. Rietveld refinement of XRD data.

4.3 Optical Analyses

The optical properties of the coatings were measured by spectrophotometer analyses over the spectral range of 280-900 nm. Results are shown in Figure 4.4.

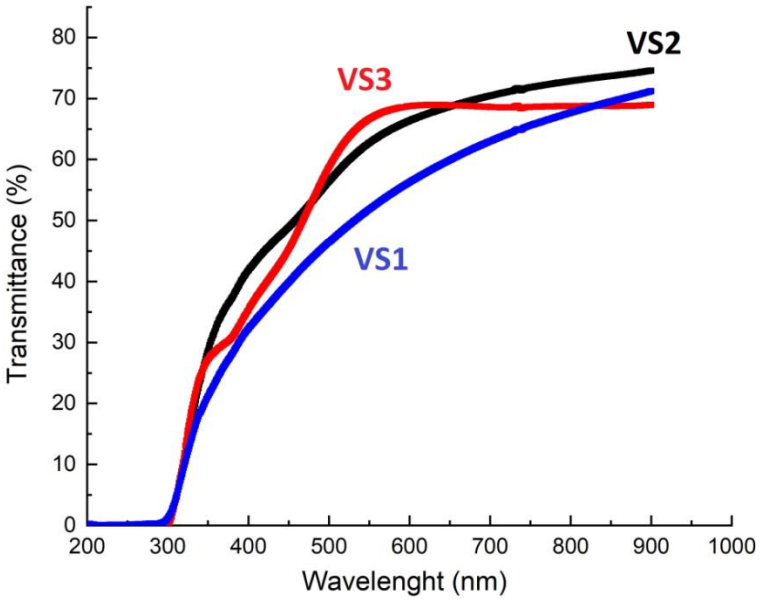


Figure 4.4. Transmittance values of the coatings.

As can be seen from the figure, the transmittance values thus the transparency of the coatings increased with the increase in the annealing temperatures. Specifically for the visible light range (390-700 nm), VS_1 annealed at 300 °C has the lowest transmittance, the highest transmittance obtained from VS_3 annealed at 500 °C is about 70% and most importantly is quite stable.

5. CONCLUSIONS

In the present work, V_2O_5-SiO_2 coatings were successfully deposited on glass substrates by sol-gel dip coating technique. Following conclusions were drawn from our study;

- Microstructural analyses by SEM demonstrated the nanostructure of V_2O_5-SiO_2 coatings deposited.
- XRD analyses revealed that the coating is amorphous at 300 °C and V_2O_5 phase obtained at 400 and 500 °C demonstrating that coatings are nanocomposites with crystalline V_2O_5 phase in the amorphous SiO_2 matrix.
- Optical analyses showed that the transparency of the coatings increased with the increase in the annealing temperatures and the highest transparency of about 70% was obtained from the coating annealed at 500 °C.
- Synthesis and characterization of the coatings with increased vanadium oxide molar ratios can be suggested as further studies.

REFERENCES

Basu, Sukumar, and Jayita Kanungo. 2011. "Nanocrystalline Porous Silicon." *Crystalline Silicon - Properties and Uses*. doi: 10.5772/23355.

Becker, C., D. Amkreutz, T. Sontheimer, V. Preidel, D. Lockau, J. Haschke, L. Jogschies, C. Klimm, J. J. Merkel, P. Plocica, S. Steffens, and B. Rech. 2013. "Polycrystalline Silicon Thin-Film Solar Cells: Status and Perspectives." *Solar Energy Materials and Solar Cells* 119:112–23. doi: 10.1016/j.solmat.2013.05.043.

Becker, C., F. Ruske, T. Sontheimer, B. Gorka, U. Bloeck, S. Gall, and B. Rech. 2009. "Microstructure and Photovoltaic Performance of Polycrystalline Silicon Thin Films on Temperature-Stable ZnO:Al Layers." *Journal of Applied Physics* 106(8). doi: 10.1063/1.3240343.

Beteille, F., and J. Livage. 1998. "Optical Switching in VO2 Thin Films." *Journal of Sol-Gel Science and Technology* 13(1–3):915–21.

Bhattacharyya, Sayan, Yitzhak Mastai, Rabi Narayan Panda, Sun-Hwa Yeon, and Michael Z. Hu. 2014. "Advanced Nanoporous Materials: Synthesis, Properties, and Applications."

Brinker, C. Jeffrey. 2013. "Dip Coating." Pp. 233–61 in *Chemical Solution Deposition of Functional Oxide Thin Films*. Springer.

Burkhardt, W., T. Christmann, S. Franke, W. Kriegseis, D. Meister, B. K. Meyer, W. Niessner, D. Schalch, and A. Scharmann. 2002. "Tungsten and Fluorine Co-Doping of VO2 Films." *Thin Solid Films* 402(1–2):226–31.

Chakraborty, Supriya. 2014. "GAS SENSING OF NANOSTRUCTURED SEMICONDUCTING OXIDES (VO2 AND TiO2) AND NITRIDE (GaN)."

Darling, Robert B., and Shiho Iwanaga. 2009. "Structure, Properties, and MEMS and Microelectronic Applications of Vanadium Oxides." *Sadhana* 34(4):531.

Dubey, R. S., K. Jhansirani, and Shyam Singh. 2017. "Investigation of Solar Cell Performance Using Multilayer Thin Film Structure (SiO2/Si3N4) and Grating." *Results in Physics*. doi: 10.1016/j.rinp.2016.11.065.

Dutta, Binay K., T. M. A. Ellateif, and Saikat Maitra. 2011. "Development of a Porous Silica Film by Sol–Gel Process." *World Acad. Sci. Eng. Technol* 73:308–12.

Ertl, Gerhard, Helmut Knözinger, and Jens Weitkamp. 2008. *Preparation of Solid Catalysts*. John Wiley & Sons.

Eyert, Volker. 2002. "The Metal-insulator Transitions of VO2: A Band Theoretical Approach." *Annalen Der Physik* 11(9):650–704.

Fardad, M. A. 2000. "Catalysts and the Structure of SiO2 Sol-Gel Films." *Journal of Materials Science* 35(7):1835–41. doi: 10.1023/A:1004749107134.

Graham, John L., Catherine Bothe Almquist, Sachin Kumar, and Sukh Sidhu. 2003. "An Investigation of Nanostructured Vanadia/Titania Catalysts for the

Oxidation of Monochlorobenzene." *Catalysis Today* 88(1–2):73–82.

Koza, Jakub A., Zhen He, Andrew S. Miller, and Jay A. Switzer. 2011. "Resistance Switching in Electrodeposited VO2 Thin Films." *Chemistry of Materials* 23(18):4105–8.

Kramer, Illan J., James C. Minor, Gabriel Moreno-Bautista, Lisa Rollny, Pongsakorn Kanjanaboos, Damir Kopilovic, Susanna M. Thon, Graham H. Carey, Kang Wei Chou, David Zhitomirsky, Aram Amassian, and Edward H. Sargent. 2015. "Efficient Spray-Coated Colloidal Quantum Dot Solar Cells." *Advanced Materials* 27(1):116–21. doi: 10.1002/adma.201403281.

Lanwang, Mary Grace. 2018. *Comparison of Commercial and Sol Gel Prepared Vanadium Oxide*.

Liu, Yueyan, Juncheng Liu, Yuanbao Li, Danping Wang, Lin Ren, and Kaishun Zou. 2016. "Effect of Annealing Temperature on the Structure and Properties of Vanadium Oxide Films." *Optical Materials Express* 6(5):1552–60.

Livage, Jacques. 1999. "Optical and Electrical Properties of Vanadium Oxides Synthesized from Alkoxides." *Coordination Chemistry Reviews* 190:391–403.

Lysenko, S., A. J. Rua, V. Vikhnin, J. Jimenez, F. Fernandez, and H. Liu. 2006. "Light-Induced Ultrafast Phase Transitions in VO2 Thin Film." *Applied Surface Science* 252(15):5512–15.

Milea, C. A., C. Bogatu, and A. Duta. 2011. "The Influence of Parameters in Silica Sol-Gel Process." *Bulletin of the Transilvania University of Brasov. Engineering Sciences. Series I* 4(1):59.

Mjejri, I., Laura Maria Manceriu, Manuel Gaudon, Aline Rougier, and Faouzi Sediri. 2016. "Nano-Vanadium Pentoxide Films for Electrochromic Displays." *Solid State Ionics* 292:8–14.

Moffatt, Claire, and Anders Wigstein. 2005. *Sol-Gel Synthesis of VO2 Thin Films and the Effects of W and Re Doping*. Swedish defence research agency.

Murmu, Peter P., John Kennedy, Sidharth Suman, Shen V Chong, Jérôme Leveneur, James Storey, Sergey Rubanov, and Ganpati Ramanath. 2019. "Multifold Improvement of Thermoelectric Power Factor by Tuning Bismuth and Antimony in Nanostructured N-Type Bismuth Antimony Telluride Thin Films." *Materials & Design* 163:107549.

Nag, Joyeeta, and R. F. Haglund Jr. 2008. "Synthesis of Vanadium Dioxide Thin Films and Nanoparticles." *Journal of Physics: Condensed Matter* 20(26):264016.

Öksüzoğlu, Ramis Mustafa, Pınar Bilgiç, Mustafa Yıldırım, and Okan Deniz. 2013. "Influence of Post-Annealing on Electrical, Structural and Optical Properties of Vanadium Oxide Thin Films." *Optics & Laser Technology* 48:102–9.

Park, K. M., S. Yi, S. Moon, and S. Im. 2001. "Optimum Oxygen Concentration for the Optoelectronic Properties of IR Sensitive VOx Thin Films." *Optical Materials* 17(1–2):311–14.

Pathak, S. S., and A. S. Khanna. 2012. "Sol–Gel Nanocoatings for Corrosion Protection." Pp. 304–29 in *Corrosion Protection and Control Using Nanomaterials*. Elsevier.

Patients, Supportive, Palliative Care in Solid Cancer, Bassam Abdul Rasool Hassan, Zuraidah Binti Mohd Yusoff, Mohamed Azmi Hassali Othman, Saad Bin, Additional information is available at the end of the Chapter, and Http://dx.doi.org/10.5772/55358. 2012. "We Are IntechOpen , the World ' s Leading Publisher of Open Access Books Built by Scientists , for Scientists TOP 1 %." *Intech* 13.

Pergament, A. L., G. B. Stefanovich, N. A. Kuldin, and A. A. Velichko. 2013. "On the Problem of Metal-Insulator Transitions in Vanadium Oxides." *International Scholarly Research Notices* 2013.

Pistola, G., M. Pasquali, M. Tocci, V. Manev, and R. V Moshtev. 1985. "Lithium/Lithium Vanadium Oxide Secondary Batteries: IV. Evaluation of Factors Affecting the Performance of Test Cells." *Journal of Power Sources* 15(1):13–25.

Priolo, Francesco, Tom Gregorkiewicz, Matteo Galli, and Thomas F. Krauss. 2014. "Silicon Nanostructures for Photonics and Photovoltaics." *Nature Nanotechnology* 9(1):19.

Qin, Getong, Xin Wu, Jianwu Wen, Jing Li, and Min Zeng. 2019. "A Core-Shell NiFe2O4@ SiO2 Structure as a High-Performance Anode Material for Lithium-Ion Batteries." *ChemElectroChem* 6(3):911–16.

Reddy, Ch V. Subba, Edwin H. Walker Jr, S. A. Wicker Sr, Quinton L. Williams, and Rajamohan R. Kalluru. 2009. "Synthesis of VO2 (B) Nanorods for Li Battery Application." *Current Applied Physics* 9(6):1195–98.

Sakka, Sumio. 2005. *Handbook of Sol-Gel Science and Technology. 3. Applications of Sol-Gel Technology*. Vol. 3. Springer Science & Business Media.

Scrosati, Bruno, and Jürgen Garche. 2010. "Lithium Batteries: Status, Prospects and Future." *Journal of Power Sources* 195(9):2419–30.

Song, Hyun-Kon, Kyu Tae Lee, Min Gyu Kim, Linda F. Nazar, and Jaephil Cho. 2010. "Recent Progress in Nanostructured Cathode Materials for Lithium Secondary Batteries." *Advanced Functional Materials* 20(22):3818–34.

Tech, M. n.d. "FACULTY OF ENGINEERING AND TECHNOLOGY SRM UNIVERSITY SRM NAGAR, KATTANKULATHUR–603 203." *PRACTICE* 2(1):3–4.

TOYGUN, Şafak, Gülhan KÖNEÇOĞLU, and Yasemen KALPAKLI. 2013. "GENERAL PRINCIPLES OF SOL-GEL." *Sigma: Journal of Engineering & Natural Sciences/Mühendislik ve Fen Bilimleri Dergisi* 31(4).

Ureña-Begara, Ferran, Aurelian Crunteanu, and Jean-Pierre Raskin. 2017. "Raman and XPS Characterization of Vanadium Oxide Thin Films with Temperature." *Applied Surface Science* 403:717–27.

Vernardou, D., A. Sapountzis, E. Spanakis, G. Kenanakis, E. Koudoumas, and N. Katsarakis. 2012. "Electrochemical Activity of Electrodeposited V2O5 Coatings." *Journal of The Electrochemical Society* 160(1):D6.

Whittaker, Luisa, Christopher J. Patridge, and Sarbajit Banerjee. 2011. "Microscopic and Nanoscale Perspective of the Metal− Insulator Phase Transitions of VO2: Some New Twists to an Old Tale." *The Journal of Physical Chemistry Letters* 2(7):745–58.

Wu, Jing, Wanxia Huang, Qiwu Shi, Jinghan Cai, Dong Zhao, Yubo Zhang, and Jiazhen Yan. 2013. “Effect of Annealing Temperature on Thermochromic Properties of Vanadium Dioxide Thin Films Deposited by Organic Sol–Gel Method.” *Applied Surface Science* 268:556–60.

Xie, Bing He, Wen Biao Fu, Guang Tao Fei, Shao Hui Xu, Xu Dong Gao, and Li De Zhang. 2018. “Preparation and Enhanced Infrared Response Properties of Ordered W-Doped VO2 Nanowire Array.” *Applied Surface Science* 436:1061–66.

Ye, Longqiang, Yulu Zhang, Xinxiang Zhang, Teng Hu, Rui Ji, Bin Ding, and Bo Jiang. 2013. “Sol–Gel Preparation of SiO2/TiO2/SiO2–TiO2 Broadband Antireflective Coating for Solar Cell Cover Glass.” *Solar Energy Materials and Solar Cells* 111:160–64.

陳劭恩. 2017. “Effects of Annealing on Thermochromic Properties of W-Doped Vanadium Dioxide Thin Films Deposited by Electron Beam Evaporation.”

CURRICULUM VITAE

Personal Information

Name Surname : M****h R**i
Nationality : I**q
Birth place and date : **/*/1***
Marital Status : S****e
Phone : 055*********
E-mail : m****d @gmail.com

Education

Degree	Institution Name	Graduation Year
High-School	Adnanıye Secondary School-Iraq	2013
Bachelor Degree	Almustansırıya university/ materıal engıneerıng	2018
Master Degree	Muğla Sıtkı Koçman University/Metallurgıcal and Materıal engıneerıng	2022

Language

1. English - V.Good
2. Arabic – Native

Training Programs

Establishment	Position	Year
Project management training program	Iraq- Baghadad	06.2015-09.2015 06.2016-09.2016
A laboratory traınıng program-Eskan company	Iraq-Erbıl	09.2017-10.2017

Hobbies

1. Swimming
2. Reading
3. Cooking