TALLINN UNIVERSITY OF TECHNOLOGY

SCHOOL OF ENGINEERING

DEPARTMENT OF MATERIALS AND ENVIRONMENTAL TECHNOLOGY

ENCAPSULATION OF YTTRIUM OXYHYDRIDE WITH TIO_2 THIN FILMS DEPOSITED BY ULTRASONIC SPRAY PYROLYSIS.

ÜTRIUMOKSÜHÜDRIIDI KAPSELDAMINE TIO_2 ÕHUKESE KILEGA KASVATATUD ULTRAHELI PIHUSTUSPÜROLÜÜSI MEETODIL

MASTER THESIS

Student: … Oluwatobi Moses Olowosokedile.......

Student code: ... ...195436KAYM........

Supervisors: ...Dr. Merike Kriisa; Research Scientist....
; Dr. Smagul Karazhanov (IFE), Kjeller, Norway

Tallinn, 2021.

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Department of Materials and Environmental Technology

THESIS TASK

Student: … Oluwatobi Moses Olowosokedile, 195436KAYM ... (name, student code)

Study programme, …KAYM09/18 - Materials and Processes for Sustainable Energetics ... (code and title)

main speciality: Processes for sustainable energetics.

Supervisor (s)… Dr. Merike Kriisa; Research Scientist….

; Dr. Smagul Karazhanov (IFE), Kjeller, Norway. +4796517797………. (position, name, phone)

Consultants: ……………………………………………………………... (name, position)

……………………………………………………………………………… (company, phone, e-mail)

Thesis topic:

(in English): *Encapsulation of yttrium oxyhydride with TiO_2 thin films deposited by ultrasonic spray pyrolysis.*

(in Estonian): Ütriumoksühüdriidi kapseldamine TiO_2 õhukese kilega kasvatatud ultraheli pihustuspürolüüsi meetodil.

Thesis main objectives:

1. To deposit TiO_2 thin films on top of YHO films by ultrasonic spray pyrolysis at different substrate temperatures and study the optical properties of the films before and after they are illuminated by UV light.
2. To study the effect of growth temperature on the properties of the encapsulated YHO films with TiO_2 thin films.

Thesis tasks and time schedule:

No	Task description	Deadline
1.	To deposit TiO_2 thin films onto YHO films at different substrate temperatures of 100°C to 250°C	
2.	To analyse optical, structural, and morphological properties of the encapsulated YHO films	
3.	To write the maters thesis	

Language: English… **Deadline for submission of thesis:** “.......” ....201…. a

Student: …Oluwatobi Moses Olowosokedile…. ....…......... “.......” …........201…. a

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Supervisor: …Merike Kriisa………………. “.......” ....201…. a

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Consultant: Smagul Karazhanov ……………………… “.......” ....201…. a

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CONTENTS

Preface

This thesis forms part of an international research collaboration, led by Dr. Smagul. Karazhanov at the Institute for Energy Technology, (IFE) kjeller, Norway with Prof. Ilona Oja Acik being the lead collaborator at the laboratory of thin films chemical technologies, Department of Materials and Environmental Technology (TALTECH).

I would like to thank my supervisor, Dr. Merike Kriisa, research scientist at the laboratory of thin films chemical technologies for her support during the process. I wish to thank Associate Professor Arvo Mere who helped me during the XRD measurements, Dr. Erki Kärber who helped me during optical measurements, and Dr. Atanas Katerski who helped me during USP setup and cutting of samples at the glove box.

We have studied the optical, structural, and morphological properties of TiO_2 thin films on top of YHO films by ultrasonic spray pyrolysis at different substrates temperature for dynamic smart windows applications. The TiO_2 thin films were sprayed from solution of molar ratio of 1:4 containing titanium (IV)isopropoxide and acetylacetone in ethanol and deposited onto YHO films at substrate temperature of 100°C to 250°C for dynamic smart windows. The resulting encapsulated YHO films with TiO_2 thin films where first exposed to 30 minutes UV light and monitored for 6 hours bleaching time, afterwards, the samples were placed in 12 hours of UV lights, bleaching time were then monitored for 6 hours and up till an average of 50 days. UV-VIS Spectroscopy, XRD and SEM were used to characterize the encapsulated YHO films. The outcome showed that optical transmittance of the films decreased after illumination. The thickness of the TiO_2 thin films on top of YHO films decreases from 42.51 nm to 29.30 nm as the temperature increases from 100°C to 150°C, while the surface of the encapsulated YHO films rupture as the temperature increases from 200°C to 250°C. YHO films samples encapsulated with TiO_2 thin films at substrate temperatures of 100°C and 150°C showed a uniform, homogenous and continuous encapsulated surface while encapsulation seem to have failed as temperature increases from 200°C to 250°C.

Keywords: YHO, TiO_2, ultrasonic spray pyrolysis, encapsulation, thin films, UV light.

List of abbreviations and symbols.

AcacH- acetylacetone
ε – Absorption coefficient
H^- - Hydrogen ion
HVAC - Heating, ventilation, and air conditioning
TTIP - Titanium isopropoxide
TiO_2 – Titanium dioxide.
UV - Ultraviolet
USP – Ultrasonic spray pyrolysis
O^{2-} - Oxygen ion
$[O_2^-]$ – Oxygen concentration
WO_3 – Tungsten oxide
YHO – yttrium oxyhydride
YH_2 – yttrium di hydride
YH_3 – yttrium tri hydride
λ – Absorption band wavelength
XRD - X-ray diffraction
Y – yttrium
[Y] - yttrium concentration

1.0 INTRODUCTION

The need to save energy in building to efficiently improve the life of inhabitant has received relevance in recent technology trend. The design to minimize energy consumption in building is therefore essential by reducing illumination loads and Heating, ventilation, and air conditioning (HVAC) energy use. This has contributed 25-30% of primary energy consumption worldwide [1]. It was reported that roughly 40% emission of carbon dioxide in 2017 was from operations related to building [2].

Smart windows play an important role in the energy management of a building as it regulates transmission of light in both visible and near infrared light of electromagnetic radiation. To improve the visual comfort, heating, cooling, and lightning need of inhabitant of a building, smart windows technologies have received much attention to strive under varying climate and weather condition. The advancement in the design of window components such as frames, glazing, as well as the use of improved materials and techniques (insulation materials, glazing, coating, etc) is necessary for comforting window characteristics [3,4]. The insulating properties of windows, control heat gain/loss from electromagnetic radiation, transmission of light, surface treatments, coatings, tints, all affect the energy features of windows. Chromogenic materials and devices have brought about studies in nanotechnology [5]. However, electrochromic materials used in electrochromic windows attracted attention because of their interactive and dynamic control of spectral features applicable in the construction sector [6-9]. Based on this, smart windows, coupled with light and temperature sensor enable to save energy over conventional passive photochromic coatings by its lightning systems and climate control [10]. Smart windows aim to regulate heat and light that passes through its surface while maintaining a comfortable vision through the window.

In this thesis, research on ultrasonically sprayed TiO_2 thin films grown on YHO films for encapsulation of the substrate and characterization of obtained layer properties has been carried out. The YHO films encapsulated with TiO_2 at substrate temperature of 100ºC and 150ºC could be used to improve the strength of photochromic properties of the films which in turn could be used as energy saving windows in building.

2.0. BACKGROUND AND LITERATURE REVIEW

2.1. Photochromic Effects in Organic Materials and Its Applications

Photochromism has become a common coined name because of its use in photochromic spectacles. Photochromism has been defined as the property of a material to exhibit a reversible change of colour by induced light based on a chemical reaction and having a different absorption spectrum [11]. Photochromic systems can involve very different structural or bond changes. [12]

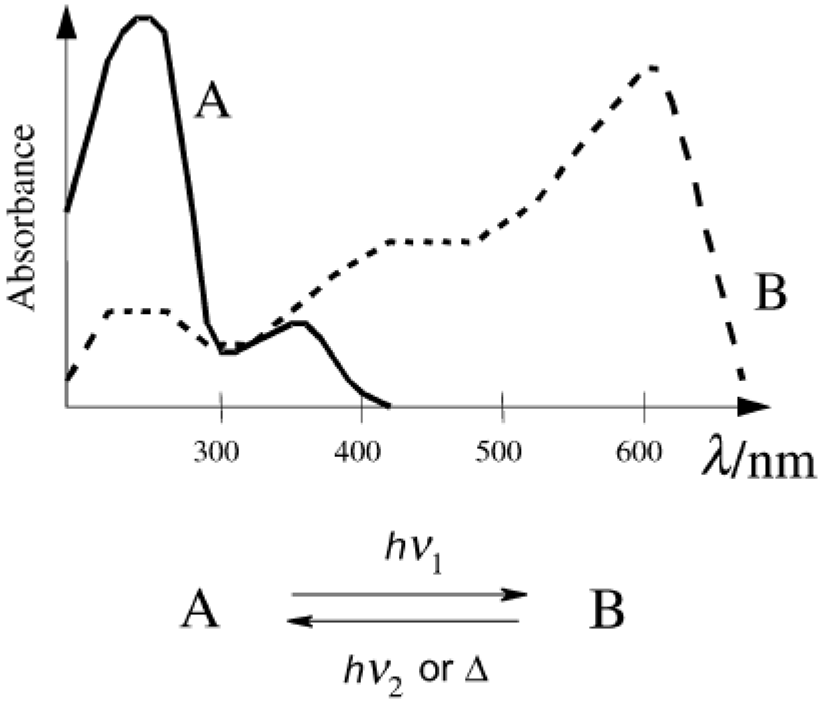
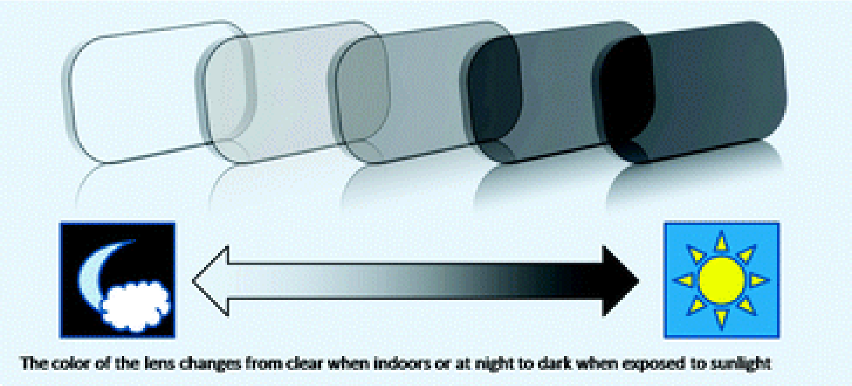


Figure 2.1. Photochromism of materials A and B [11].

Figure 2.1 explains how a thermodynamically stable material A is transformed into material B by electromagnetic irradiation. The reverse reaction can occur either thermally mostly called Photochromism of type T or photochemically (Photochromism of type P). Most photochromic molecules have been observed to show a pale yellow or colourless form A and a coloured form B, molecules that undergo such changes are Positive Photochromism. Bimolecular photochromic molecule such as photocycloaddition reactions is a negative photochromism or inverse [λ_{max} (A) > λ_{max} (B)] [11]. Materials that show photochromic effect are applicable in wide areas of studies and respond to absorption of light in the Ultraviolent to Infrared region of the electromagnetic radiation. Unimolecular reactions have been observed to be the most common organic photochromic system. [13].

The application of photochromic materials has been of tremendous use in everyday life ranging from buttons, hair colourant, knobs, lunch boxes for children, on and off levers, etc. Substances that show photochromism have been used for a long time and have played an important role in the digital age. The transmission, gating, and digital storage of data has become a recent trend of interest [14]. Most widely used storage device are CD and DVD uses light to write and erase information.



Photochromic organic–inorganic hybrid materials - Chemical Society Reviews (RSC Publishing)

Photochromic Ink - Photochromic Materials (smarol.com)

Figure 2.2 Photochromic lenses, shoes, and clothes: contributions to comfort and to fashion [15]

The reversibility feature of a photochromic material meets the requirement of rewritable media such as CD-RW, DVD-RW where bits travel in zeros (0) and one (1).

Photochromic materials are engineered in the sub-nano scale which makes it suitable for fast switching. The molecular nanometre is foundational to switching unit, compared to a bit. High resolution is the result of two-photon phenomenon as shown in literature (Figure 2.3. a and b) [16].

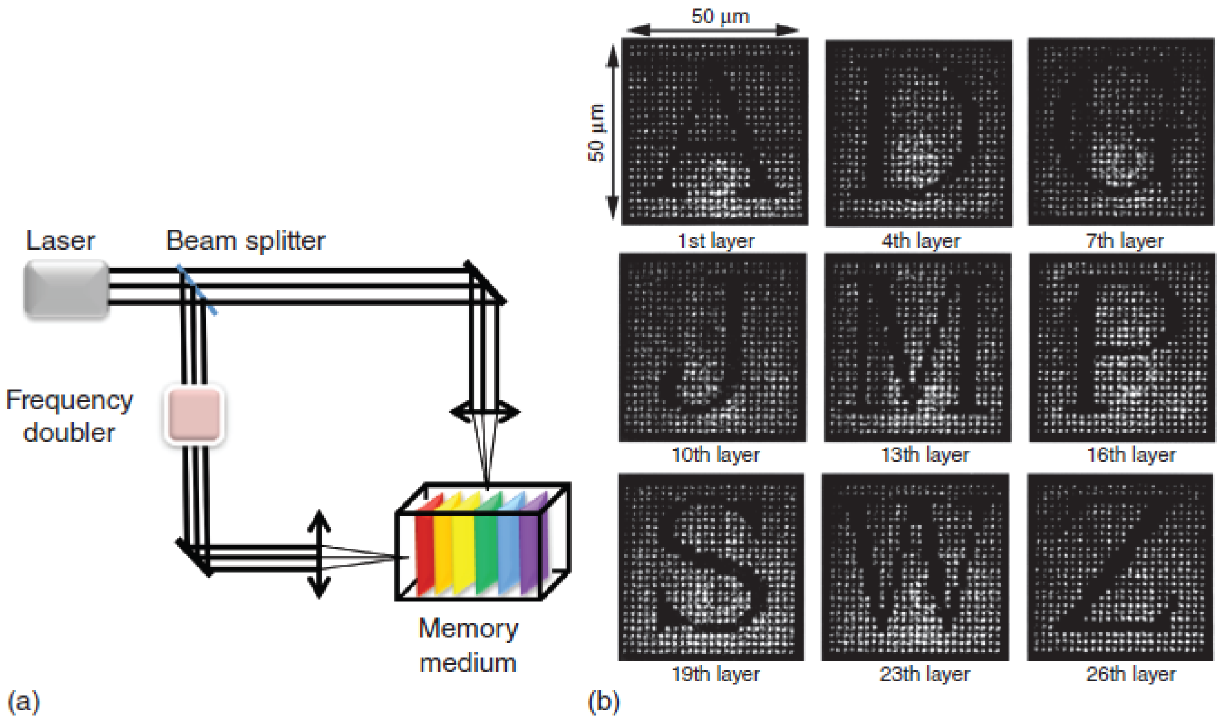
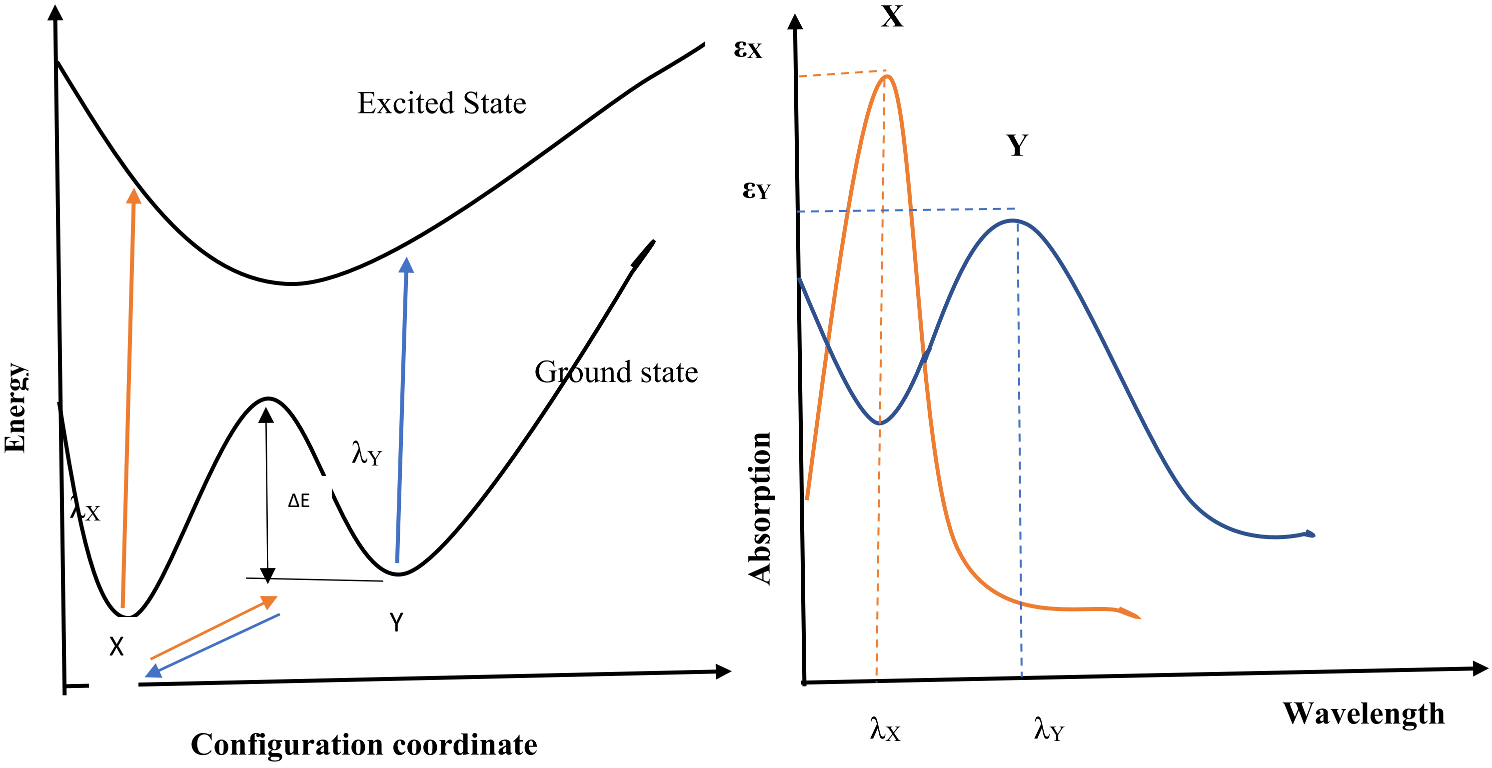


Figure 2.3. Rewritable optical memory medium based on photochromic compounds: (a) general structure of the recording medium [16d] and (b) alphabet letters recorded on the different layers [16b].

Another fast-treading application of photochromism is fluorescence in that excitation happens because of absorption of photons which have led to faster microscopes with better resolution with stable fluorescent probe [17].

2.2 Processes in Photochromism

In Figure 2.4, a simple reaction between two molecular substances X and Y has been introduced to explain the concept of photochromism [13]. Substances X and Y are separated by potential well called barrier (ΔE). In a situation where the barrier is low, Y is metastable and can revert spontaneously to X. As described earlier, photochromic spectacles followed this principle. This is referred to as a T-type which can thermally induced a reaction from Y to X. On the other way round, if the barrier is high, it denotes a bistable system, here, only photons can cause the reaction. This is called P-type, here changes can only occur in the presence of light. This feature is of great importance as it makes difference between photochromic bistable systems and other systems (ferroelectric or ferromagnetic). No concept of hysteresis is involved in the bistability of photochromic systems. Photochromic compounds are described using synonyms depending on the nature or the causes of the changes. Heliochromism, are activated by unfiltered sunlight and deactivated under diffused daylight conditions, hence they are suitable for sun lens applications [18], Electrochromism, reversible change of absorption spectra between two forms, A and B, resulting from oxidation/reduction reactions. Advantage of this type of devices is that the colour change is controlled by human, however, they are very expensive [19]., In thermochromism, thermally induced reversible colour change takes place [20,21], Piezochromism, a phenomenon when crystals undergo a change of colour due to mechanical grinding [22], Tribochromism, a piezochromism phenomenon where a colour change does not revert to the original prior to crystal fracture [23], Solvatochromism, is the reversible variation of the electronic spectroscopic properties [24,25], etc.



(a) **(b)**

Figure 2.4. Photochromism: Two-way light induced reaction between two molecules X and Y. (a) Potential energy diagram and (b) Absorption spectra of potential energy spectra

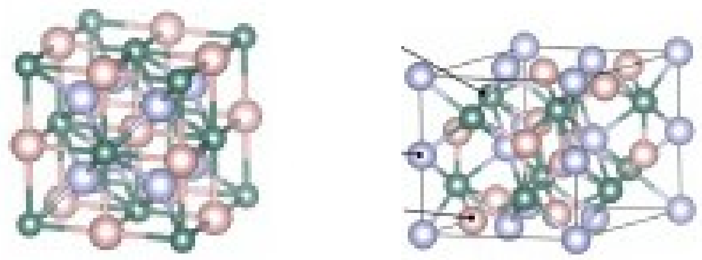
In a conventional photochromic effect on a material, molecule X absorbs electromagnetic radiation in the ultraviolet or near ultraviolet, with a characteristic absorption band wavelength (λ_X). Wavelength, ε_X is the absorption coefficient of X. Excitation of X from ground state occurs as photons is absorbed at characteristic band wavelength (λ_X). The excited X produces Y with a probability known as Quantum yield. In an analogous pattern Y revert to X, on the condition that Y is excited at characteristic band wavelength at λ_Y where the absorption of Y took place.

The colour of the molecules is revealed, and colour of light needed to induce the reactions are the results of the spectral position of the absorption band.

It should be noted that the ability of a material or chemical substance to undergo a reversible change of colour is non-destructive. Fatigue is the loss of expected performance over time due to chemical degradation. Oxidation has been observed to be the major cause of damage to photochromic substance [26]. However, some materials have been identified as fatigue-resistant substances, methyl-substituted dithienylperfluorocyclopentenes crystals are stable at 100°C but transmittance began to decrease after 10^4 cycles [27], Bacteriorhodopsin, undergo more than 10^5 photochemical cycles without damage [28].

2.3 Yttrium Oxyhydride And Its Photochromic Effect

The combination of hydrogen and yttrium to form Yttrium hydride compound is considered as a rare-earth metal hydride. Face-centred cubic (FCC) structure metallic compound, YH_2 changes its structure to a hexagonal closed packed (HCP) structure under high pressure in the presence of excess hydrogen to form YH_3 (See figure 2.5).



(a) FCC-YH_3
(high pressure phase)

(b) HCP – YH_3
(ambient pressure phase)

Figure 2.5. Face-centred cubic YH_3 (a) and hexagonal closed packed YH_3 (b) [29].

YH_3 has a band gap of 2.6eV, a transformational intermediate state is achieved under pressure of about 12GPa which results to further structural change at higher pressure [30,31]. The metallic-insulator transition of YH_2 to YH_3 is applicable in optical switch of windows from non-transparent to transparent [32]. At room temperature, yttrium hydride (YH_x) films, exhibit photo-switching optical properties under different Gpa pressures [33]. The continuous absorption of hydrogen in yttrium hydride results to the optical switch of colour from shiny material to yellow transparent material [34]. Yttrium hydrides films exhibits photochromism by absorbing electromagnetic radiation [35-38]. Ohmura et al. reported colour transition from orange transparent yttrium hydride film to opaque black when illuminated by visible laser light under ambient condition [39]. Annealing at room temperature, the black film returned to its initial orange colour. When yttrium hydride is exposed to air, it results to the incorporation of oxygen in the hydride and then form oxyhydride which has an interesting reversible photochromic feature. This switchable optical property enables a wider range of usage in many technological applications, such as smart windows, sensors, light modulator, and medical devices [40,41].

Mongstad et al reported a huge amount of oxygen of up to 1.67, [O]/[Y] in YHO films deposited by reactive sputtering [42]. The presence of oxygen in the films has been attributed to the exposure of the films to air [43]. Due to strong absorption of light in the visible range, it was reported that, as the band gap of YHO increases from 2.8eV to 3.7eV (because of increasing concentration of oxygen in the film), photochromism became very faint [44]. During the exposure of yttrium hydride to air, the lattice expands because of incorporation of oxygen. Under illumination, oxygen is extracted out of the lattice, leaving behind an oxygen deficient structure responsible for the optical darkening observed [45]. The film bleach back to its original state when YHO lattice expand in the presence of oxygen. The expansion/contraction of the film under illumination/dark because of inward/outward displacement of oxygen atom is refers to as breathing.

The sharing of similar crystal lattice in oxy hydrides is a chemical crystal classification of photochromic oxygen-containing hydrides films known as advanced multi anion materials [46-49]. Reduction of hydrogen content in oxyhydride system led to variation in structural properties has been attributed to one of the factors that may promote solid-state crystallization in yttrium oxyhydrides [50]. Mixed-anion compound offers flexibility in the regulation of hydrogen ion (H^-) – oxygen ion (O^{2-}) exchange capabilities through metal center valance charge state. This would form the basis for the formation of stable stoichiometric versions of oxy hydrides as described in figure 2.6 [51].

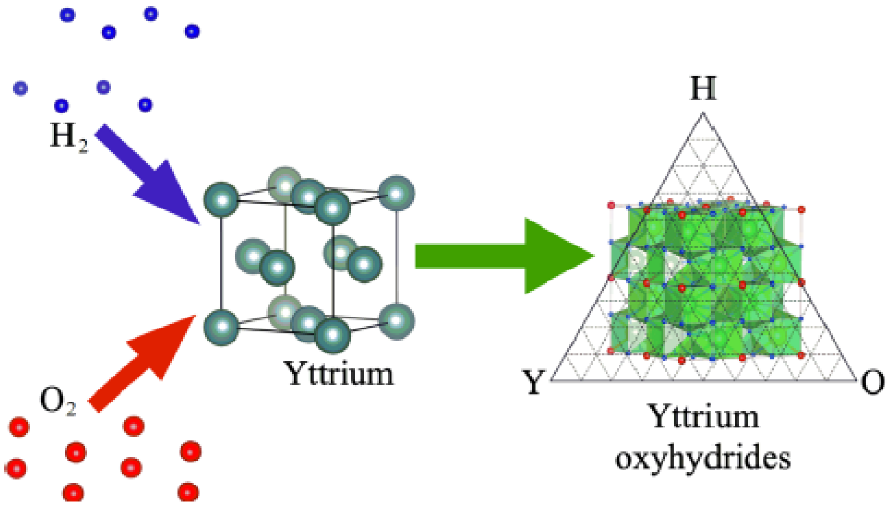


Figure 2.6 Cluster of Oxygen and Hydrogen with Yttrium to form Yttrium oxyhydride [51]

2.4 The Role of Encapsulation

In organic photochromism, light-responsive molecular containers are designed in such a way to shield the system to execute a specific function when such molecules are encapsulated in a molecular container [104]. This kind of encapsulated molecules have their application in drug delivery, use as a catalyst, etc. Because of the reversibility of colour that took place in photochromic materials when illuminated, this property has made it an interesting field of study in recent year with its wide varieties of applications such as ophthalmic glasses, optical memories, and switches, etc. Photochromic dye used in optical lenses is mostly common where the optical lens returned to its original colour in the dark. Ambient conditions and molecular structure of the photochromic materials are major factors that influences the rate of optical switching. The time taken for a photochromic material to switch from dark (when illuminated) to its original colour in a cyclic manner is of great importance to the stability of such material. In recent year, a stable photochromic material with a faster switching rate is of great concern. Different methods have been employed to improve optical switching in photochromic dye. One of such method, Photochromic dye mixed with lens monomer followed by curing solution resulted to a slow faded photochromic lens from dark to colourless. Photochromic lens was produced by adding dyes into hard coating material. Such lens exhibited slow fading rate with uneven dispersion of the dyes. Other methods like dissolution of dye in polymerizable monomers, still resulted to slow fading as detailed in Encapsulated Photochromic Dyes U.S Pat. No. 61, 005.755 at column 1, line 003 to line 014. (US2011/0189462 A1).

The need for rapid switching rate of photochromic compounds is therefore essential for photochromic lens. Photochromic dyes have been encapsulated using dendritic polymer [52,53]. The encapsulated photochromic dyes have an increased fading rate relative to the ones encapsulated by non-dendritic polymers.

Yttrium oxyhydride is found to exhibit a reversible photochromic property when a substantial amount of oxygen is present in the lattice [54]. This interesting property made it useful in many technological applications, such as photochromic coating in smart windows and sensors [55,56]. Previous studies showed that the strength of the photochromic effect or behaviour of this materials became unstable under long exposure to air, thereby increasing the oxygen concentration of the film accompanied by an optical bandgap widening [57]. Titanium oxide as an encapsulant was used to stabilize the film under harsh conditions. In this study, yttrium oxyhydride is encapsulated with ultrasonically sprayed titanium dioxide to stabilize the YHO films and keep the films photochromic property active through longer period of time.

2.5 Titanium dioxide

Titanium dioxide (titanium (IV)oxide) or titania with chemical formula TiO_2, commonly found in ores, occurring as a mineral in nature and belong to the family of transition metal oxides [58]. Titanium dioxide has become one of the most important materials used in thin films technology for the past decades because of its wide applications such as gas sensors [59], various optical coating (because of its excellent transmittance in the visible region, chemical stability, non-toxicity, chemical stability, and optical refractive capability) [60-62], solar cells [63], self-cleaning materials [64], window layer in photovoltaic solar cells such as Grätzel cell [65] and photocatalysis [66].

Three crystalline phases exist for titanium dioxide: brookite, anatase and rutile (Figure 2.7). Anatase and rutile are stable phases, and both have a tetragonal crystalline structure with densities $3.84 g/cm^3$ and $4.26 g/cm^3$, respectively [67], the brookite is of low interest because of its unstable orthorhombic crystalline structure. At high temperature of about 800°C, anatase and brookite phases convert to rutile irreversibly [68].

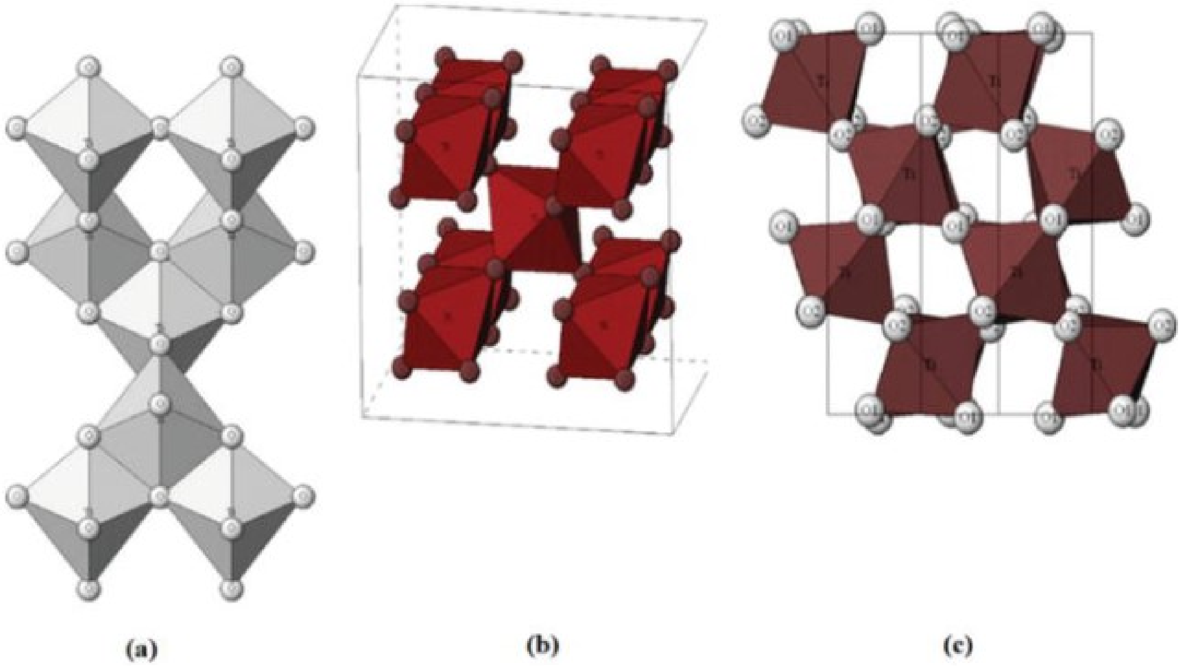


Figure 2.7 Crystalline structures of titanium dioxide (a) anatase, (b) rutile, and (c) brookite [69].

Several techniques have been reported to prepare titanium dioxide thin films, e.g., plasma enhanced chemical vapour deposition [70], ion beam sputtering [71], anodization [72], molecular beam epitaxy (MBE), atomic layer deposition (ALD) and ultrasonic spray pyrolysis [73-75].

2.5.1 Ultrasonic Spray Pyrolysis (USP)

Ultrasonic spray pyrolysis technique is suitable to prepare metallic oxide films over large area. It has been used to deposit insulating and active device layers [76-77]. Homogeneity over a large area have been observed with metallic oxide films prepared by spray pyrolysis for different applications. USP was employed as a technique used to encapsulate YHO with TiO_2 in this study.

USP techniques is considered the cheapest, easiest process to grow thin and thick films. The setup is simple and easy to operate. This has made it an attractive and versatile process for mass production, rapid growth rate, ease of doping and reproducibility. The films are grown in the presence of air, hence, there is no need of vacuum systems.

2.5.2 Ultrasonic Spray Pyrolysis Process

In the experimental setup of USP, an atomizing ultrasonic generator with chemical reagent is required with a gas carrier, and a heated substrate. The vaporized fine droplets are deposited to the heated substrate through the gas carrier. The air entrainment controls the transportation of droplets to the substrate. The diffusion of the entrained droplets forms either thin or thick films on the heated substrate. In the preparation of thin films from titanium dioxide, deposition temperature plays an important role. Effects of temperature on film qualities, including mineralogy, transmission and optical band have been reviewed [78-81]. Figure 2.8 shows basic setup of ultrasonic spray pyrolysis.

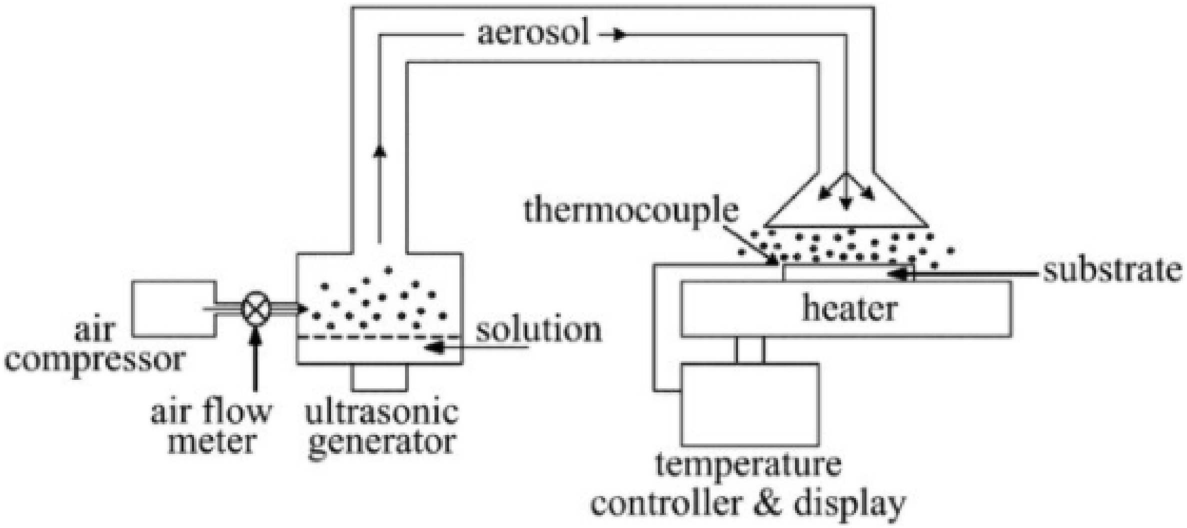


Figure 2.8 Basic setup for ultrasonic spray pyrolysis [82]

2.6. Summary of literature overview

Yttrium oxyhydride (YHO), are inorganic nanomaterials that belong to emerging class of materials called oxyhydrides with oxides and hydride anions sharing the same sites in the lattice. [96-99]. The material was discovered by Trygve Mongstad from the Department for Solar Energy of the Institute for Energy Technology (IFE), Kjeller, Norway. The material exhibits photochromic properties at ambient conditions. While the nature of the effect is not clear, a reduction in transmittance from 90% to 50% for a light -induced tuning have been reported [100]. The results of the materials ever since its discovery have been documented in many journal articles and patents [40,101-103]. The encapsulation of yttrium oxyhydride is a recent tread that attracted attention to strengthen the photochromic properties of the material. Deposition of TiO_2 thin films on top of YHO films by ultrasonic spray pyrolysis at different substrate temperatures have been employed to encapsulate the YHO films.

- ✓ Titanium dioxide is well known optical coatings with good transmittance in the visible region, desirable qualities with high refractive index and chemical stability.
- ✓ Different techniques have been used to deposit TiO_2 thin films. The widely used techniques have been the chemical spray pyrolysis and sol-gel spin coating methods.
- ✓ Ultrasonic spray pyrolysis is a fast, simple, cheap, and convenient method to grow uniform and fine homogeneous droplets of TiO_2 thin films on YHO films; to our best knowledge, the deposition of TiO_2 thin films on top of YHO films by ultrasonic spray pyrolysis is not studied.
- ✓ Titanium dioxide has shown exceptional properties suitable for various applications, for example, air purification, medical treatment, photocatalysis, etc.

2.7. Aim of the thesis.

The aim of the thesis is to deposit TiO_2 thin films on top of YHO films by ultrasonic spray pyrolysis at different substrate temperatures and characterize the films before and after UV illumination.

To study the effect of growth temperature on the properties of the YHO films encapsulated with TiO_2 thin films.

3.0. METHODOLOGY

3.1. Chemicals

The chemicals used for the encapsulation of YHO films with TiO_2 films are listed in table 3.1 below.

Table 3.1. Precursors used for the encapsulation of YHO films with TiO_2.

Material Deposited	**Precursor**	**Formula**	**Company**	**Purity**
	Titanium (IV)-isopropoxide	$C_{12}H_{28}O_4Ti$	Merck Schuchardt OHG, Germany	≥ 98%
TiO_2	acetylacetone	$C_5H_8O_2$	Merck Schuchardt OHG, Germany	≥ 98%
	ethanol	C_2H_5OH	OU Estonian spirit, Estonia	≥ 96.6%

3.2. The preparation of spray solution: TiO_2

The chemicals used for the preparation of the spray solution are titanium (IV)isopropoxide (precursor), acetylacetone (stabilizer) and ethanol (solvent).

100ml of titanium (IV)isopropoxide solution (0.1M) were used for the deposition of all samples. To prepare 100ml of spray solution, 50ml of ethanol was placed in a conical flask followed by 4.0ml of acetylacetone and then stirred for one minute, 3ml of TTIP precursor solution was quickly added and shake thoroughly for 2 minutes at room temperature. Ethanol is then added to the bottle to reach 100ml of solution and stirred again to make homogeneous solution. The molar ratio of titanium (IV)isopropoxide to acetylacetone was 1:4. The encapsulation of YHO films by depositing TiO_2 thin film was started immediately after the solution was ready. The schematic of preparing this solution is shown in Figure 3.1.

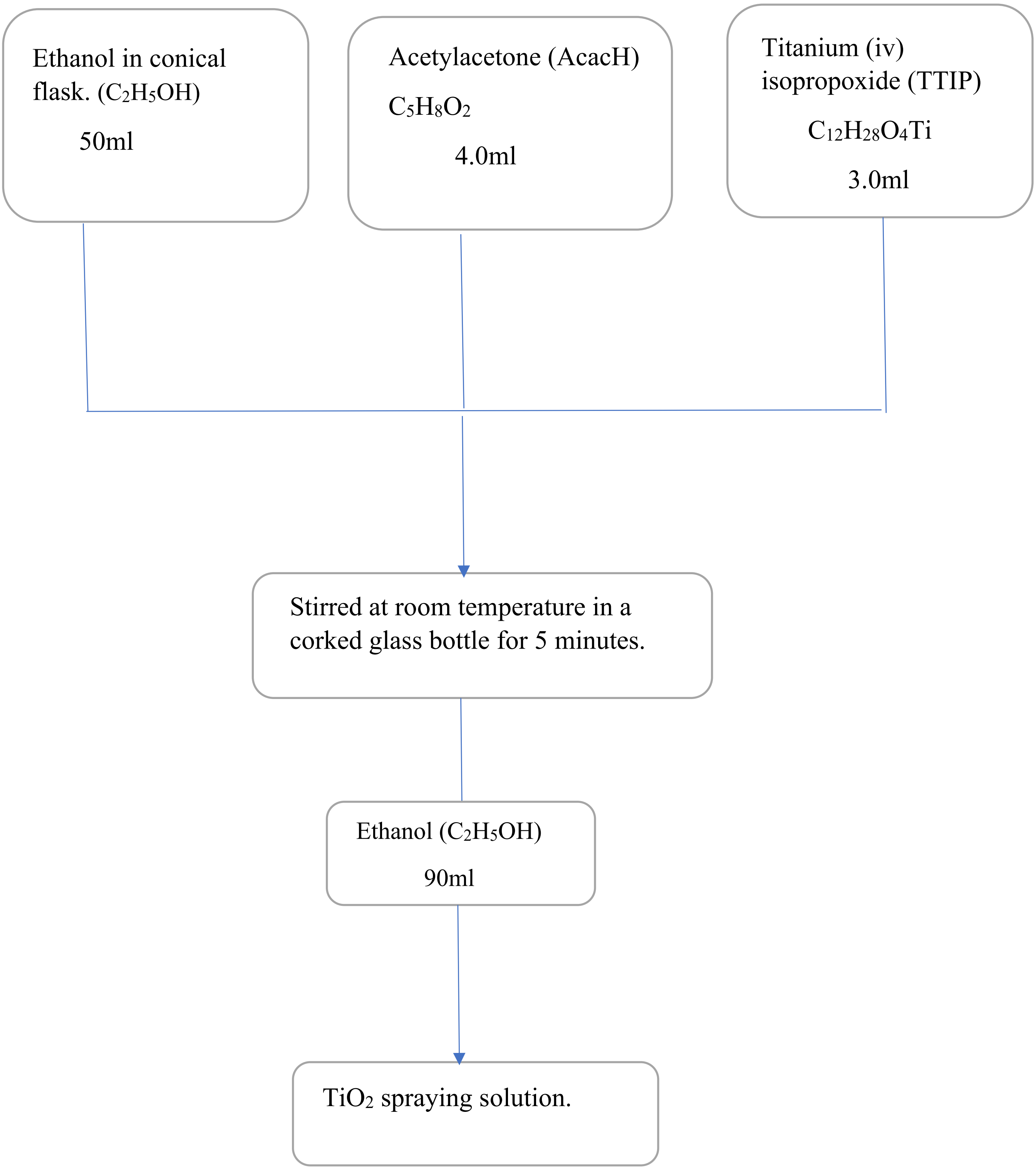


Fig. 3.1. The schematic diagram for preparation of TiO_2 film spray solution.

3.3. Substrates

YHO samples deposited by reactive magnetron sputtering under varying deposition conditions, such as different H_2/Ar ratio (0.081-0.25) deposition pressures, chamber pressures (0.54-1.0. Pa) with thickness varying from 200nm – 600nm having different oxygen concentration were grown in Institute for Energy Technology (IFE), Kjeller, Norway. (See table 3.2) The delivered samples were kept in SalvisLAB vacuum box (15.0mbar/70.4mbar) to prevent further oxidation or exposure to air and were cut into 1.5cm by 1.0cm dimension inside an Innovative Technology IL-2GB glove box (1000ppm O_2, 1.8 ppm H_2O). Thereafter, the samples were ready for encapsulation by USP.

Table 3.2. YHO films of different thicknesses and deposited at different H2/Ar deposition pressures.

S/N	**Sample Names**	**H_2/Ar ratio**	**Chamber pressure (Pa)**	**Thickness of samples (nm)**	**Oxygen Concentration (wt.%)**	
1	NL	0.125	0.59	200 - 600	8-32	>> O_2
2	ND	0.25	0.59			>> O_2
3	Y02	0.081	0.54			> O_2
4	Y03	0.25	0.6			>>> O_2
5	Y04	0.125	1.0			>>>> O_2

3.4. USP apparatus and parameters

Figure 3.2 shows a schematic ultrasonic spray pyrolysis system synonymous to the one in the Laboratory of Thin Films Chemical Technologies, TALTECH. The main components of the spraying system are ultrasonic generator, air compressor, temperature controller, heater, aerosol transportation pipe, air flow meter. The temperature controller is vital in this setup as it control the thermostat heating of the substrate temperature.

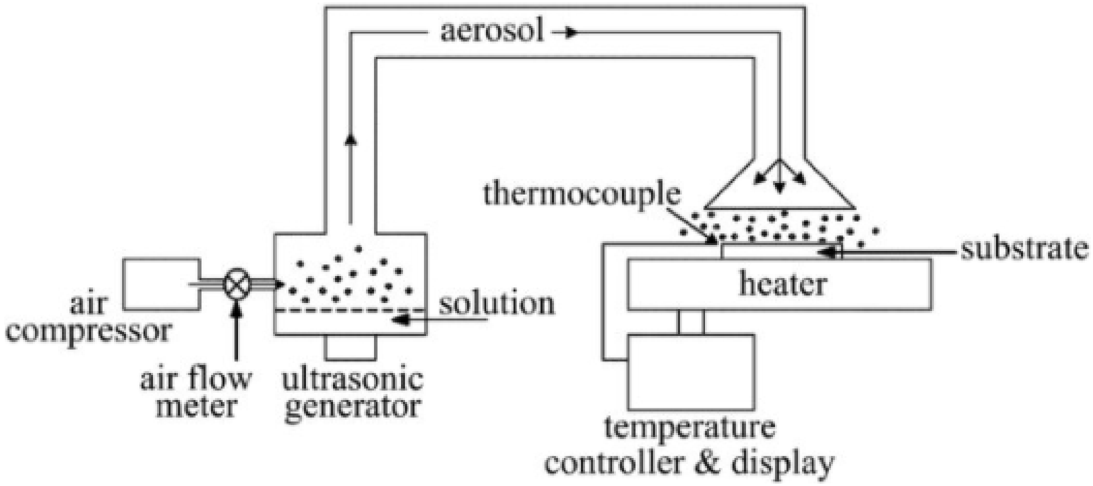


Fig 3.2. Schematic set-up for ultrasonic spray pyrolysis [82]

3.5. Deposition of TiO_2 thin films to encapsulate YHO films.

Deposition of TiO_2 thin films to encapsulate YHO films by ultrasonic spray pyrolysis method is described in Table 3.3. The YHO films were placed at the centre of the heater, then the deposition temperature was set using the temperature controller which in turn controls the heater. The prepared 100ml solution of TiO_2 was poured into ultrasonic generator by using funnel. The system deposition spray parameters were set to 4 steps and 12 cycles.

The correction of spray direction and the transportation of the generated aerosol were regulated by the director gas flow rate and the carrier gas flow rate and were set at 1.0L/min and 5.0L/min.

The moment the heater reaches the required temperature, it was left for 3 minutes to stabilized. The ultrasonic generator was switched on to generate aerosol for 1 minutes to ensure enough aerosol for spraying.

The spray deposition commenced after the air controller was switched on.

Table 3.3. Deposition parameters for growing TiO_2 on YHO by Ultrasonic spray pyrolysis

Sample Name	**Substrate**	**Condition**	**Substrate temperature (°C)**	**Carrier Gas flow rate (L/min)**	**Director Gas flow rate (L/min)**
Y02_100 **Y03_100** **Y04_100** **NL_100** **ND_100**	**YHO films**	**0.1M TTIP Solution. 4 steps, 12 Cycles**	**100**	**5.0**	**1.0**
Y02_150 **Y03_150** **Y04_150** **NL_150** **ND_150**	**YHO films**	**0.1M TTIP Solution. 4 steps, 12 Cycles**	**150**	**5.0**	**1.0**
Y02_200 **Y03_200** **Y04_200** **NL_200** **ND_200**	**YHO films**	**0.1M TTIP Solution. 4 steps, 12 Cycles**	**200**	**5.0**	**1.0**
Y02_250 **Y03_250** **Y04_250** **NL_250** **ND_250**	**YHO films**	**0.1M TTIP Solution. 4 steps, 12 Cycles**	**250**	**5.0**	**1.0**

The carrier and director gas flow rates were kept constant within all depositions at 5.0L/min and 1.0L/min, respectively. The number of steps and cycles were kept constant at 4 steps and 12 cycles respectively for the varied substrate temperatures of 100°C to 250°C.

3.6. Illumination of encapsulated YHO films with UV Light.

The TiO_2 thin films grown on YHO films deposited at different substrate temperature were exposed to ultraviolet light in two phases using PHILIPS TL-D 15W lamb. A group of 5 different samples were deposited at temperature range 100°C to 250°C and exposed to UV light. At first, the encapsulated samples were exposed to 30 minutes UV light and then monitored the switching time (bleaching) after 6 hours. The second phase of the exposure was overnight (12hours), thereafter bleaching was monitored for 6 hours and up to 51days. The illustration of the exposed encapsulated samples to UV light is described in Table 3.4.

Table 3.4. Illustration of exposed samples to UV light with bleaching time.

Sample Name	**Substrate**	**Substrate temperature (°C)**	**UV-Light Exposure (hours)**	**Bleaching Time (Days)**				**Group**
Y02_100 **Y03_100** **Y04_100** **NL_100** **ND_100**	**YHO films**	**100**	**1/2**	**1/4**	**-**	**-**	**-**	**A**
Y02_150 **Y03_150** **Y04_150** **NL_150** **ND_150**	**YHO films**	**150**	**1/2**	**1/4**	**-**	**-**	**-**	**B**
Y02_200 **Y03_200** **Y04_200** **NL_200** **ND_200**	**YHO films**	**200**	**1/2**	**1/4**	**-**	**-**	**-**	**C**
Y02_250 **Y03_250** **Y04_250** **NL_250** **ND_250**	**YHO films**	**250**	**1/2**	**1/4**	**-**	**-**	**-**	**D**

Y02_100_ovgt **Y03_100_ovgt** **Y04_100_ovgt** **NL_100_ovgt** **ND_100_ovgt**	**YHO films**	**100**	**12**	**1/4**	**1**	**5**	**51**	**Ao**
Y02_150_ovgt **Y03_150_ovgt** **Y04_150_ovgt** **NL_150_ovgt** **ND_150_ovgt**	**YHO films**	**150**	**12**	**1/4**	**1**	**4**	**50**	**Bo**
Y02_200_ovgt **Y03_200_ovgt** **Y04_200_ovgt** **NL_200_ovgt** **ND_200_ovgt**	**YHO films**	**200**	**12**	**1/4**	**1**	**3**	**49**	**Co**
Y02_250_ovgt **Y03_250_ovgt** **Y04_250_ovgt** **NL_250_ovgt** **ND_250_ovgt**	**YHO films**	**250**	**12**	**1/4**	**1**	**2**	**48**	**Do**

3.7 Characterization methods

3.7.1 Ultraviolet-visible Spectroscopy

The total transmittance and total reflectance of the encapsulated YHO samples were measured using Jasco V-670 UV-VIS-NIR spectrophotometer. The measurements were done in the wavelength range of 300nm to 1800nm throughout the study.

The optical behaviours of the sprayed TiO_2 films on YHO films were in reference to air in order to determine their total optical transparency near the infra-red region. Origin pro software and spectra manager on the spectrophotometer was used to execute all analysis and plot of graphs.

The Jasco V-670 UV-VIS-NIR spectrophotometer is designed to measure the absorption spectrum of a sample over a wavelength range of 190 to 2700 nm (extensible to 3200 nm using the optical wavelength extension accessory). The light sources used are a deuterium (D_2) lamp (187 – 350 nm) for use in the UV region and halogen (WI) lamp (330 to 2700 nm) for use in the VIS/NIR region. The light from the light source is focused and enters the monochromator. It is dispersed by the grating in the monochromator and focused on to the exit slit. The light that passes through the exit slit is monochromated. This light is split into two beams by a sector mirror, one going to the sample to be measured and the other to the reference sample. The beams that have passed through the sample and reference sample are alternatively incident upon the detector (that is a photomultiplier tube or a PbS photoconductive cell). The light incident upon the photomultiplier tube is converted into an electrical signal and, after being subjected synchronous rectification, it is converted into a digital signal. The signal processed by the computer is displayed on the monitor as digital data or a spectrum [105].

3.7.2. X-ray Diffraction

The structural pattern, phase, and crystallinity of TiO_2 films grown on YHO films was investigated by X-ray Diffraction (XRD). The XRD patterns were measured using Rigaku IV Ultimate XRD diffractometer with monochromatic Cu Kα radiation (λ=1.5406 Å, 40KV at 40mA).

The diffracting angle (2θ) measurement were varied between start angle of 20^o and closed angle of 80^o with scan step of 0.01^o and scan speed of 5^o/min. X-rays are generated by accelerating electrons to a very high speeds in a vacuum and directing them against the anode (target). The X-ray spectra generated by electrons colliding against the target, can be divided into continuous spectrum indicating continuous X-rays (white X-rays) and a discrete spectrum for characteristic X-ray. X-ray diffractometry uses Kα X-ray generated by the metal targets [106]. X-ray diffraction peaks are produced by constructive interference of a monochromatic beam of X-rays scattered at specific angles from each set of lattice planes in a sample. The peak intensities are determined by the distribution of atoms within the lattice. Consequently, the X-ray diffraction pattern is the fingerprint of periodic atomic arrangements in a given material. X-ray diffraction is a high-tech, non-destructive technique for analysing a wide range of materials including fluids, metals, minerals, polymers, catalysts, plastics, pharmaceuticals, thin-film coatings, ceramics, solar cells, and semiconductors. The technique finds innumerable practical applications in various industries, including microelectronics, power generation, aerospace, and many more. XRD analysis can easily detect the existence of defects in a particular crystal, its resistance level to stress, its texture, its size, and degree of crystallinity, and virtually any other variable relating to the sample's basic structure. [106].

3.7.3. Scanning Electron Microscopy

The morphological properties of the encapsulated samples of YHO films with TiO_2 were determined using scanning electron microscopy. In this type of electron scanning, electrons interact with atoms of the samples to produce various signals detailing surface topography and composition of the samples [83]. The encapsulated samples were cut into appropriate sizes to fit in specimen chamber. Two different SEM machines were used to characterize the encapsulated samples: EVO MA 15 Zeiss (Center of Materials Research, Tallinn University of Technology) and Zeiss Ultra 55 (Department of Material Science, Tallinn University of Technology).

4.0 Results and discussion

4.1. Optical properties of encapsulated YHO films

4.1.1. Total transmittance of TiO_2 thin films deposited onto YHO films.

A group of different five (5) YHO films samples (Y02, Y03, Y04, NL and ND) were encapsulated with TiO_2 thin films at 100ºC, 150ºC, 200ºC and 250ºC substrate temperature. Encapsulated samples from Group A-D were exposed to 30 minutes UV light while samples from Ao-Do were exposed to 12 hours UV light as stated in Table 3.3. For group A-D, optical transmittance spectra of TiO_2 thin films deposited onto YHO films at temperatures from 100ºC to 250ºC and followed by 30 minutes of UV light exposure are presented in Fig. 4.1.

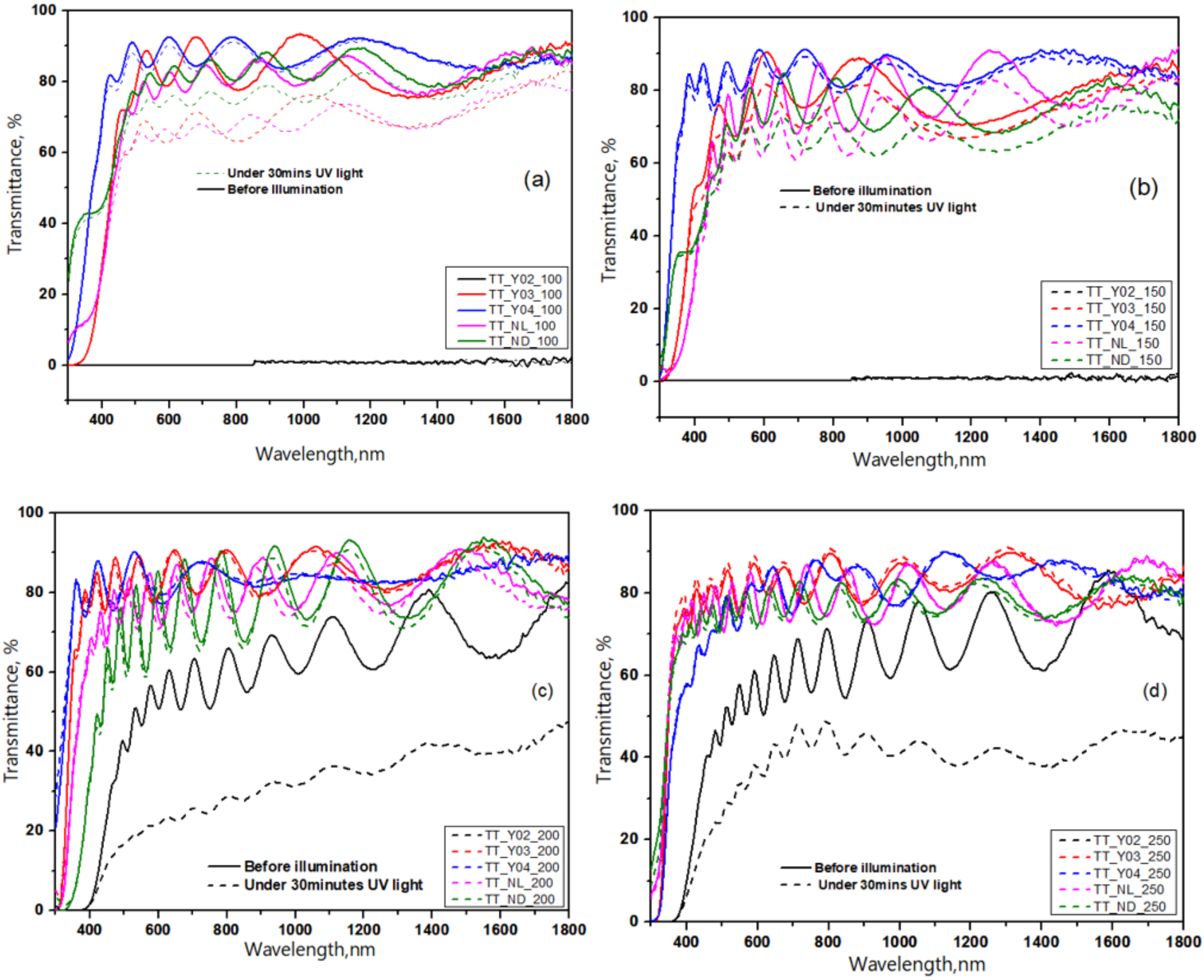


Figure 4.1. Specular transmittance spectra of encapsulated YHO films with TiO_2 measured before illumination and after 30 minutes of illumination deposited at substrate temperatures from 100ºC to 250ºC. (a) encapsulated at 100ºC (b) encapsulated at 150ºC (c) encapsulated at 200ºC and (d) encapsulated at 250ºC.

The average transmittance of all encapsulated YHO films with TiO_2 thin films was measured over the wavelengths between 300nm and 1800nm.

The transmittance of group A samples grown at 100ºC is high throughout the measured wavelength. It has an average transmittance of 88% and 75% under 30minutes illumination (Fig 4.1(a)) with exception of sample Y02_100 having no transmittance as this is darkened sample of YHO film.

The average transmittance of group B samples deposited at 150ºC and illuminated for 30 minutes, is ca 86% and 77%, respectively. Sample Y02_150 is not transparent as it is darkened YHO film.

The non-transparency of the darkened YHO films is applicable everywhere unless otherwise stated. (Fig 4.1(b)) with exception of sample Y02_150 having no transmittance as this is a darkened sample of YHO film.

The transmittance of group C samples at 200ºC has an average transmittance of 87% and 86% under 30 minutes illumination (Fig 4.1(c)). It is worth noting that as the substrate temperature increases, the darkened YHO samples (i.e., Y02_200 and Y02_250) began to become transparent, and the characteristics of the samples began to change. This shows the effect of temperature on the samples, the growth temperature had greater influence on the transmittance of YHO films encapsulated with TiO_2 thin films. With an increase in growth temperature, the bleaching rate was faster. T. Mongstad et al [84] and Elbruz et al [85] have showed that the photochromic performance of YHO thin films is drastically influenced by temperature. The effect of transmittance changes was negligible as the temperature increases and when illuminated. The illumination effect under UV light was also observed by J. N. Yao et al [86] and C. Bechinger et al [87].

The same effect of temperature increase was observed in Group D for samples grown at 250ºC has an average transmittance of 85% and 84% when illuminated under UV light for 30 minutes (Fig 4.1(d)). The only exception is sample, Y02_250 became more transparent. The effect of illumination became prominent in the darkened YHO films as temperature increases from 200ºC to 250ºC.

The specular transmittance spectra of Group Ao samples grown at 100ºC after the 12 hours of exposure to UV light is presented in Figure 4.2(a), Group Bo samples grown at 150ºC presented in 4.2 (b), Group Co samples grown at 200ºC is presented in 4.2 (c) and Group Do samples grown at 250ºC is presented in Figure 4.2 (d).

The average transmittance of Group Ao samples grown at 100ºC decreased from 88% to 33% under illumination. The darkened YHO film sample, Y02_100_ovgt, has no transmittance as this is a darkened sample of YHO film. The transmittance of Group Bo samples grown at 150ºC has an average transmittance decreased to 46%.

Similar effect of temperature changes was observed in Group Co samples and Do samples grown at 200°C and 250°C respectively, with little change in the decreased average transmittance as the temperature increases. The average transmittance of Group Co samples grown at 200°C decreased to 83% with exception of sample ND_200_ovgt (59%). The average transmittance of Group Do samples decreased to 80%, except for darkened sample, Y02_250_ovgt (39%).

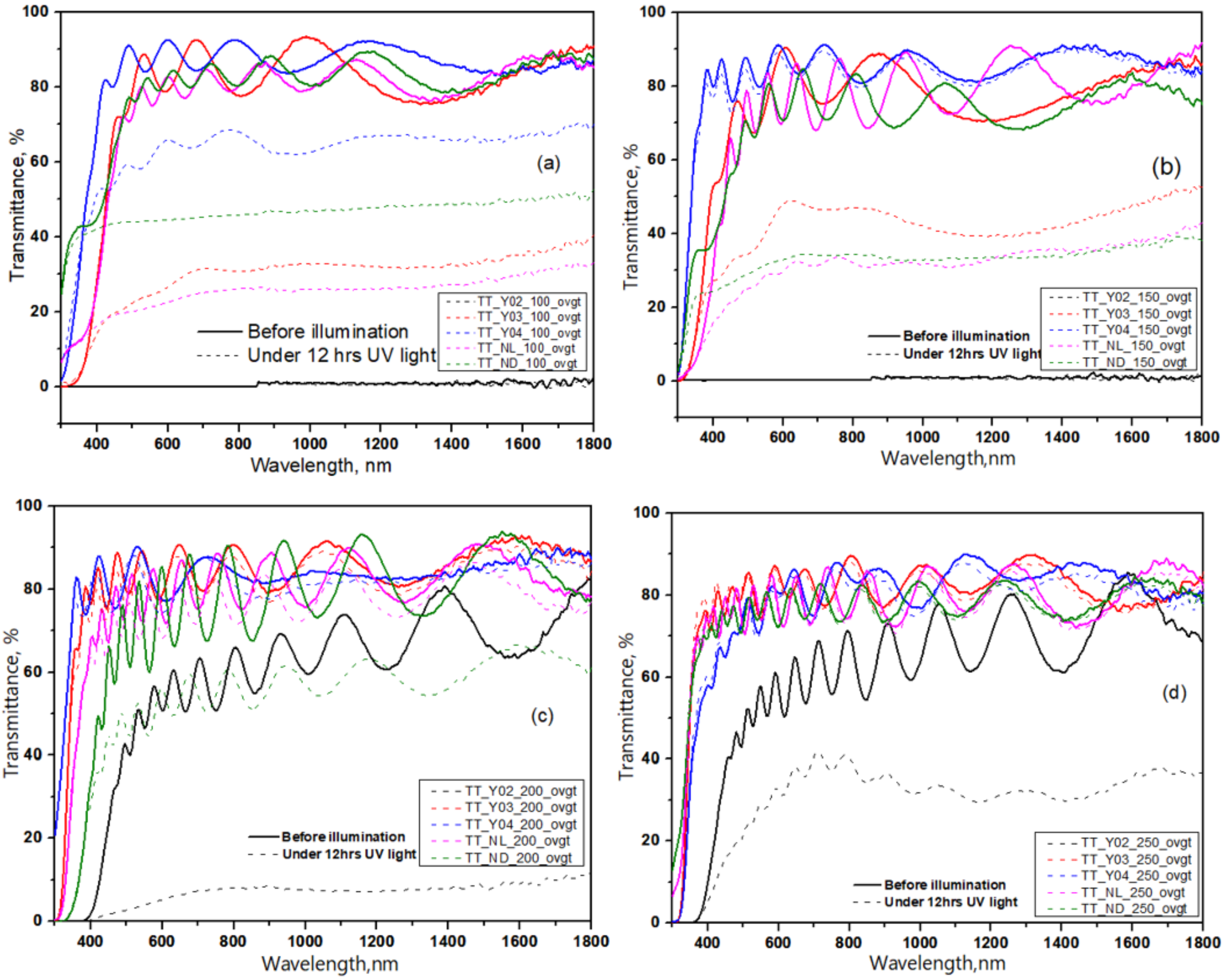


Figure 4.2. Specular transmittance spectra of encapsulated YHO films with TiO_2 measured before illumination and after 12 hours of illumination deposited at substrate temperatures from 100°C to 250°C. (a) encapsulated at 100°C (b) encapsulated at 150°C (c) encapsulated at 200°C and (d) encapsulated at 250°C.

4.1.2. Bleaching properties of TiO_2 thin films deposited onto YHO films.

The bleaching properties of the encapsulated YHO films with TiO_2 were monitored in two phases. The first phase was exposed to 30 minutes of UV light and then monitored for 6 hours in the dark. The second phase were exposed to 12 hours of UV light, then monitored for the first 6 hours up till 51days. The illumination time was increased to 12 hours in order to determine the maximum bleaching time of the samples. Figure 4.3 (a) shows how the 100°C samples relaxed back after 6 hours, Figure 4.3 (b) shows the bleaching for samples deposited at 150°C, Figures 4.3 (c) for samples deposited at 200°C and Figure 4.3 (d) presented samples deposited at 250°C

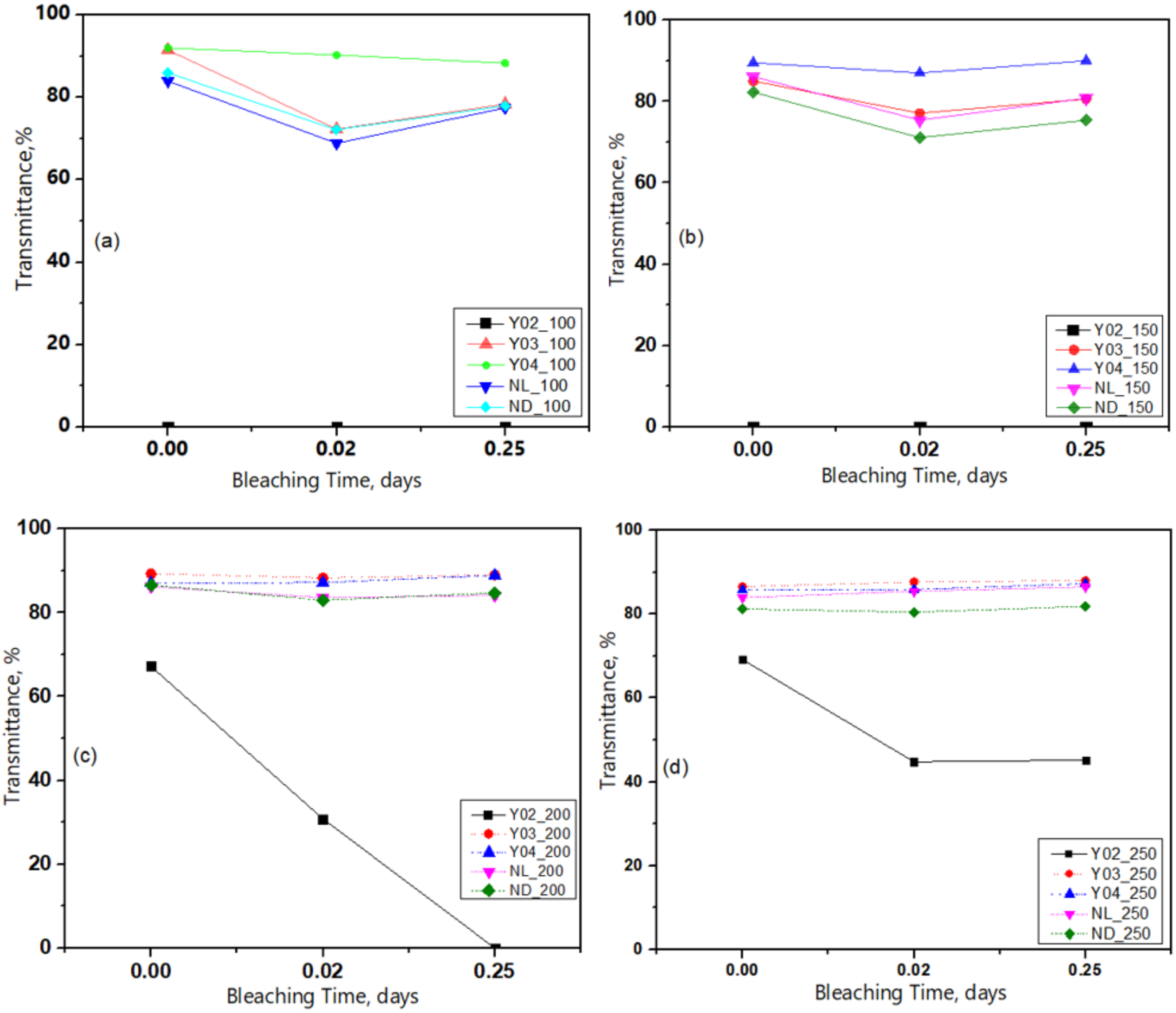


Figure 4.3. Transmittance spectra of encapsulated YHO films with TiO_2 over 6 hours bleaching time after 30 minutes illumination. (a) encapsulated at 100°C (b) encapsulated at 150°C (c) encapsulated at 200°C and (d) encapsulated at 250°C.

For all encapsulated samples of YHO films that are exposed to 30 minutes of UV light, they all showed a rapid recovery within 6 hours with average transmittance of 7% for 100°C, 6% for 150°C, 1% for 200ºC and 0% for 250ºC samples closed to As-deposited samples. However, samples, Y04_100 at 100ºC and Y04_150 at 150ºC showed the highest recovery rate with transmittance of 90%. This is probably due to the oxygen content of the samples. Similar postulation was made by T. Mongstad et al [84]. The effect of photodarkening became less noticeable as the temperature increases from 200ºC to 250ºC. The illumination effect under UV light was also observed by J. N. Yao et al [86] and C. Bechinger et al [87]. The average transmittance of recovered samples over the wavelength of 300nm – 1800nm for samples encapsulated at 100ºC was 81%, 150ºC was 82%, 200ºC was 87% and 250ºC was 86%. It is worth noting that the recovery rate for darkened YHO films (i.e., Y02_250) was very slow, with transmittance of 45% for samples at 250ºC. As at the time this sample was monitored for bleaching, it was not fully recovered to its initial transmittance state. One could deduce that the rate at which the lattice expand because of oxygen intake was slow.

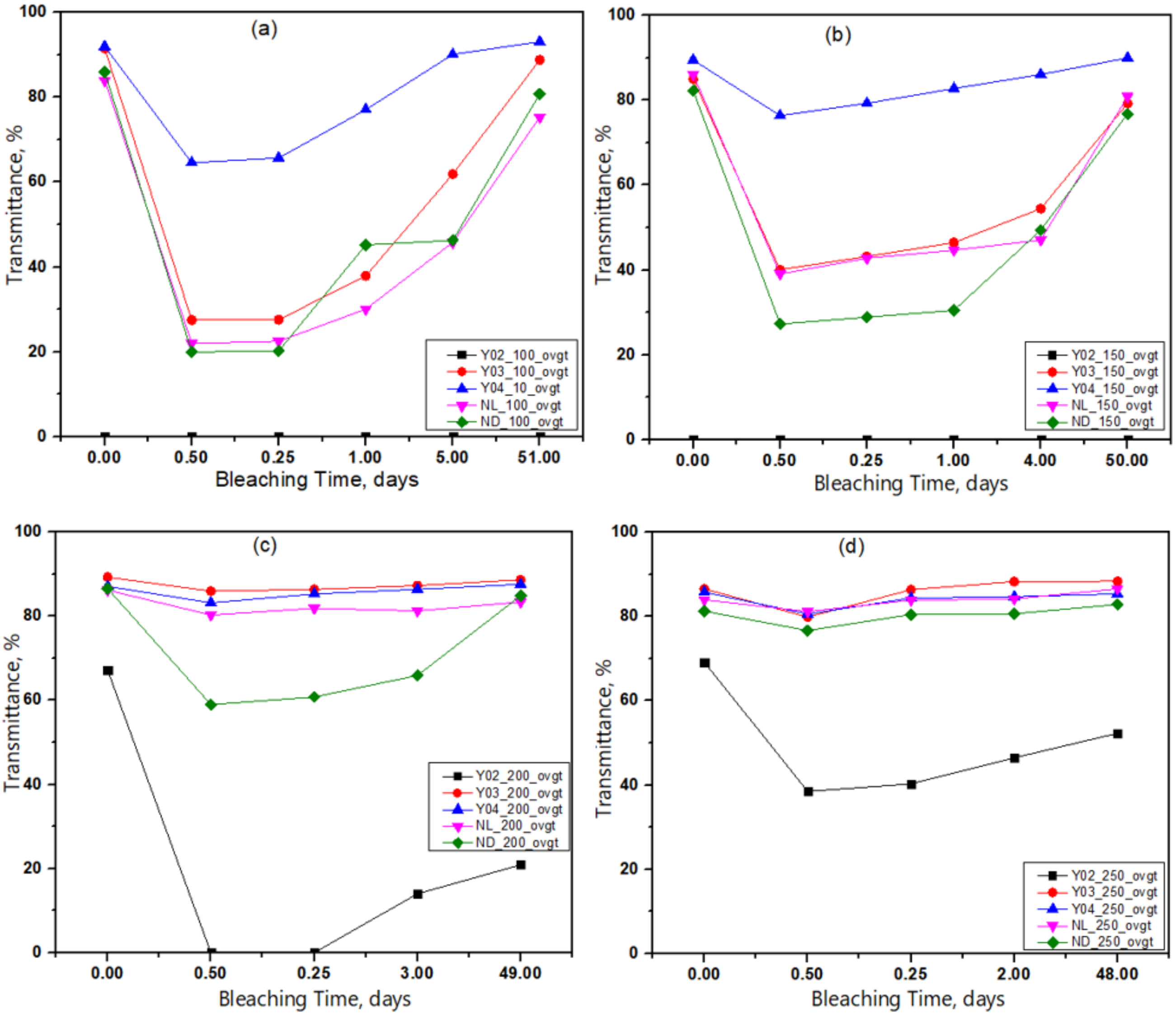


Figure 4.4. Transmittance spectra of encapsulated YHO films with TiO_2 over an average of 50 days bleaching time after 12hours illumination. (a) encapsulated at 100ºC (b) encapsulated at 150ºC (c) encapsulated at 200ºC and (d) encapsulated at 250ºC.

For the second phase, when the samples were exposed to 12 hours of UV light, it took all the samples in Group Ao-Do an average of 50 days to switch back to their initial state of yellowish colour. The average transmittance of recovered samples at 100ºC and 150ºC was 85% and 82% respectively. (See figure 4.4 (a)-(d)).

However, samples in group Co grown at 200ºC and 250ºC do not appear photo darkened when viewed with naked eye after 12 hours of exposure to UV light and have a consistent bleaching rate with slightly higher transmittance, averaged at 86%. An exception to this tread was the darkened YHO films where switching was slow with 21% transmittance for Y02_200_ovgt and 52% for Y02_250_ovgt.

4.2. Structural properties of TiO_2 thin films deposited onto YHO films.

4.2.1. X-ray diffraction, (XRD) results of TiO_2 thin films deposited onto YHO films.

Figure 4.5 shows the XRD result of TiO_2 thin films grown on YHO films deposited at different deposition temperature from 100ºC to 250ºC. Figure 4.5 presents the samples illuminated for 30 minutes and using the illumination time of 12 hours was the same (not shown).

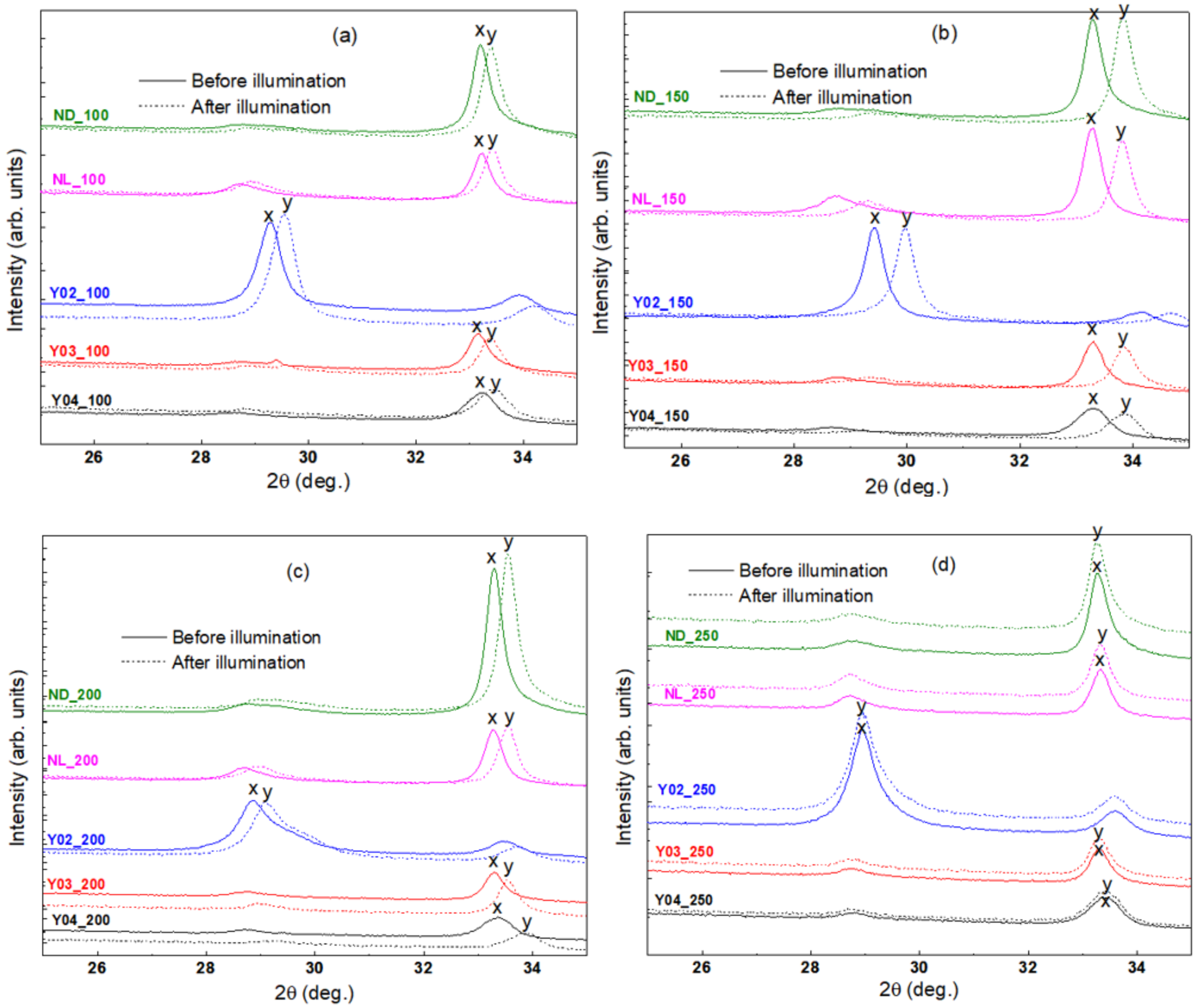


Figure 4.5. XRD patterns of encapsulated YHO films before and after illumination. (a) encapsulated at 100ºC (b) encapsulated at 150ºC (c) encapsulated at 200ºC and (d) encapsulated at 250ºC. “x” and “y” represent peaks before and after illumination of the encapsulated YHO films, respectively.

XRD studies indicated the grown TiO_2 thin films are amorphous. Crystalline TiO_2-anatase films have been observed in substrate temperature range of 300ºC-500ºC [88-95]. On the XRD pattern, for every samples grown at 100ºC to 200ºC, the peak “x” changed to peak “y”. According to Figure 4.5 (a), Figure 4.5 (b) and Figure 4.5 (c), a peak shift from plane (200) to plane (111) was visible for all the samples deposited at 100ºC, 150ºC and 200ºC after sample illumination. The peak shift diminished for illuminated samples that were grown at 250ºC. As shown in Figure 4.5 (d), there was no more peak shift for illuminated samples indicating that the samples were totally oxidized, and the encapsulation failed. The similar tendency was observed by Elbruz et al [96], and J. Montero et al [97]. Interestingly, sample named Y02 had a different preferred orientation peak irrespective of growth temperature. The different preferred orientation for sample Y02 is most probably caused using different deposition pressure to grow YHO films which affects the oxygen content of the compound. The peak shift in the samples is explained by the encapsulated YHO films lattice's ability to contract and expand when subjected to illumination and darkness.

4.3. Scanning Electron Microscopy (SEM)

4.3.1. The morphological properties of TiO_2 thin films deposited on top of YHO films.

According to the SEM images presented in figure 4.6 – 4.9. The surface of the YHO films with TiO_2 was rough and dependent of the growth temperature. According to SEM, the grain size of YHO films was further distorted as the growth temperature increases. The TiO_2 thin films were deposited on top of YHO films with columnar grains. The thickness of TiO_2 thin films deposited on top of YHO films at 100°C was 42.51nm. To study the surface morphology that was responsible for the encapsulated YHO films transmittance, SEM was carried out. According to SEM study, the TiO_2 thin films deposited at 100°C on top of YHO films showed a well encapsulated surface without any destructive effect. However, as the deposition temperature increases to 150°C, the surface of the encapsulated began to show a trace of crack. The encapsulation of YHO films with TiO_2 thin films failed as the temperature was increased from 200°C to 250°C. The surface look rougher and damaged as cracks was more prominent. This could explain why there were no physical change in the appearance of the samples when viewed with the bare eye. The samples could be said to continuously oxidized as the protective layer of TiO_2 thin films was already damaged. This effect of temperature on YHO films are in good accordance with the results obtained by Elbruz et al [85].

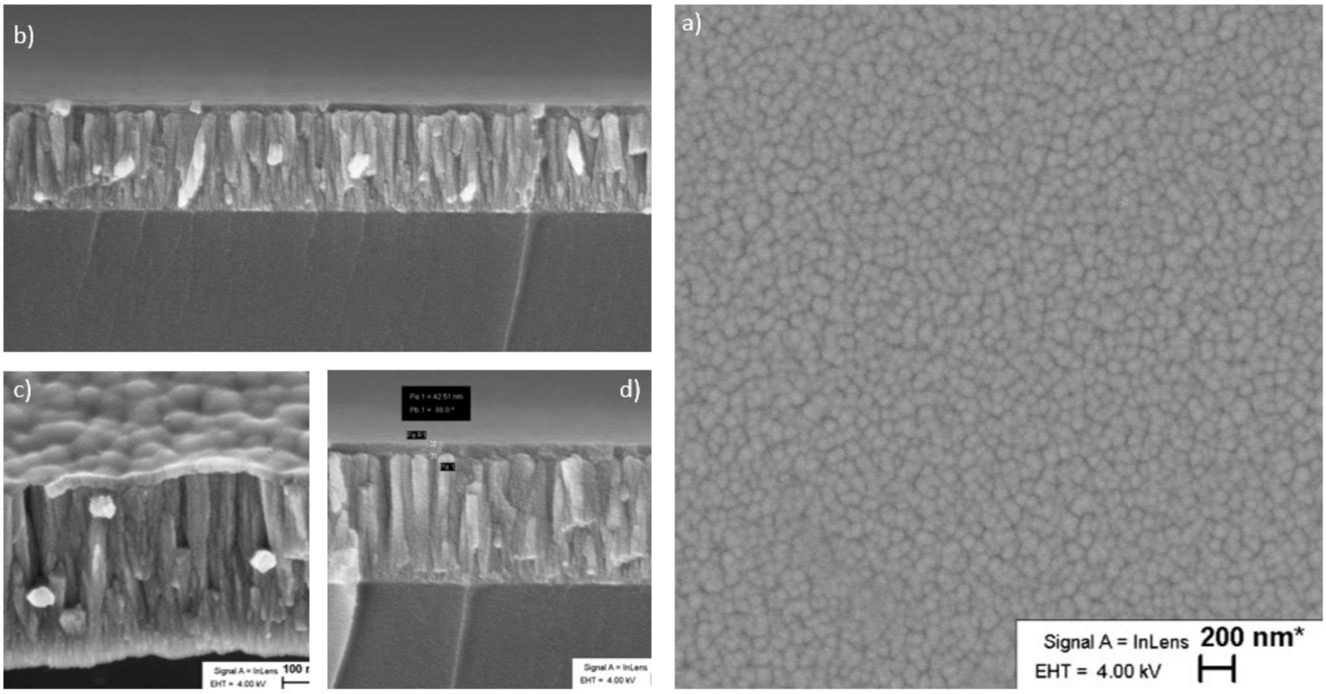


Figure 4.6. Planar (a) and cross-sectional (b, c, d) SEM images of TiO_2 thin films grown on YHO films by ultrasonic spray pyrolysis deposited at substrate temperature of 100°C.

As the deposition temperature increases to 150°C, the SEM showed that the top layer of the YHO films was encapsulated with TiO_2 thin films but with a reduction in thickness of the TiO_2 thin film layer. The thickness of deposited TiO_2 layer was 29.30nm. At 150ºC, the surface began to show a sign of crack. This crack could be as result of the fact that, the substrate temperature began to exceed the tolerant temperature of YHO films. Similar postulation has been postulated by Elbruz et al [85] (see figure 4.7(b)).

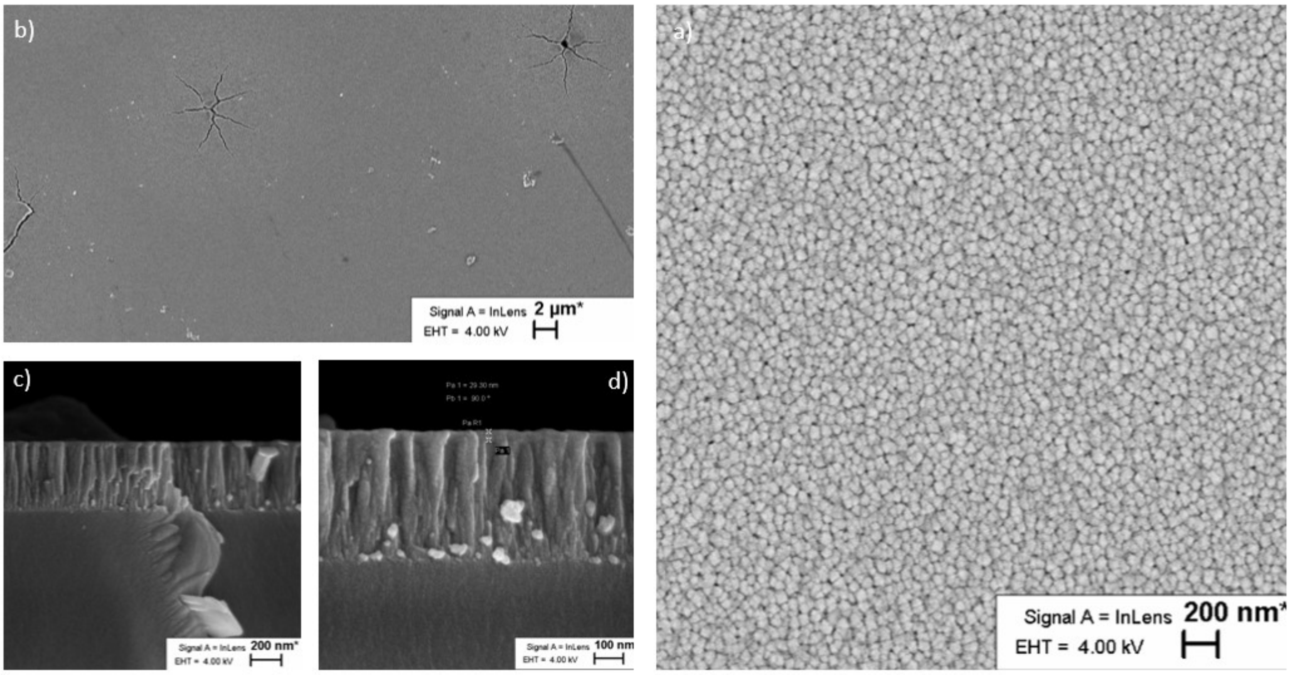


Figure 4.7. SEM image of TiO_2 thin films grown on YHO films by ultrasonic spray pyrolysis deposited at substrate temperature of 150ºC.

As the temperature further increases to 200ºC, according to the SEM images, the surface of the encapsulated YHO films became more roughen and cracks became prominent, (See figure 4.8 (a) and (b)), the number of cracks increased even further as the temperature increased to 250ºC. (See figure 4.9 (a) and (b)).

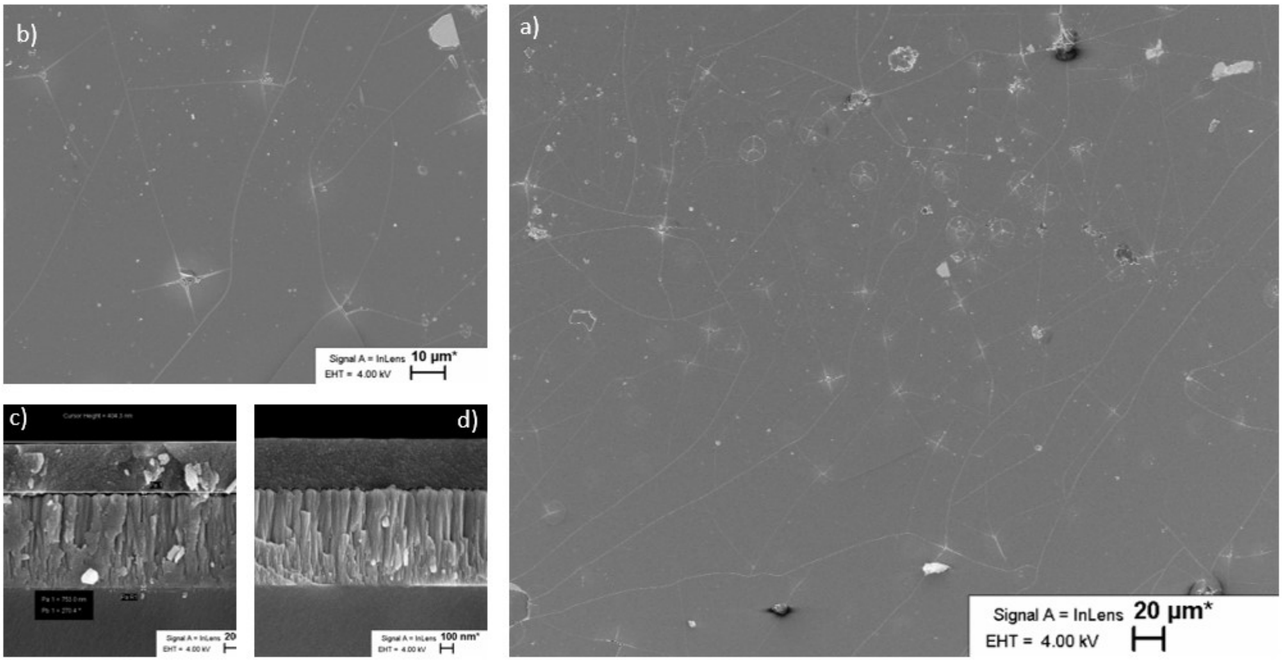


Figure 4.8. SEM image of TiO_2 thin films grown on YHO films by ultrasonic spray pyrolysis deposited at substrate temperature of 200ºC.

The growth temperature at 200ºC and 250ºC is not suitable to encapsulate of YHO films with TiO_2 thin films. The morphology of encapsulated YHO films with TiO_2 is greatly influenced by the growth temperature.

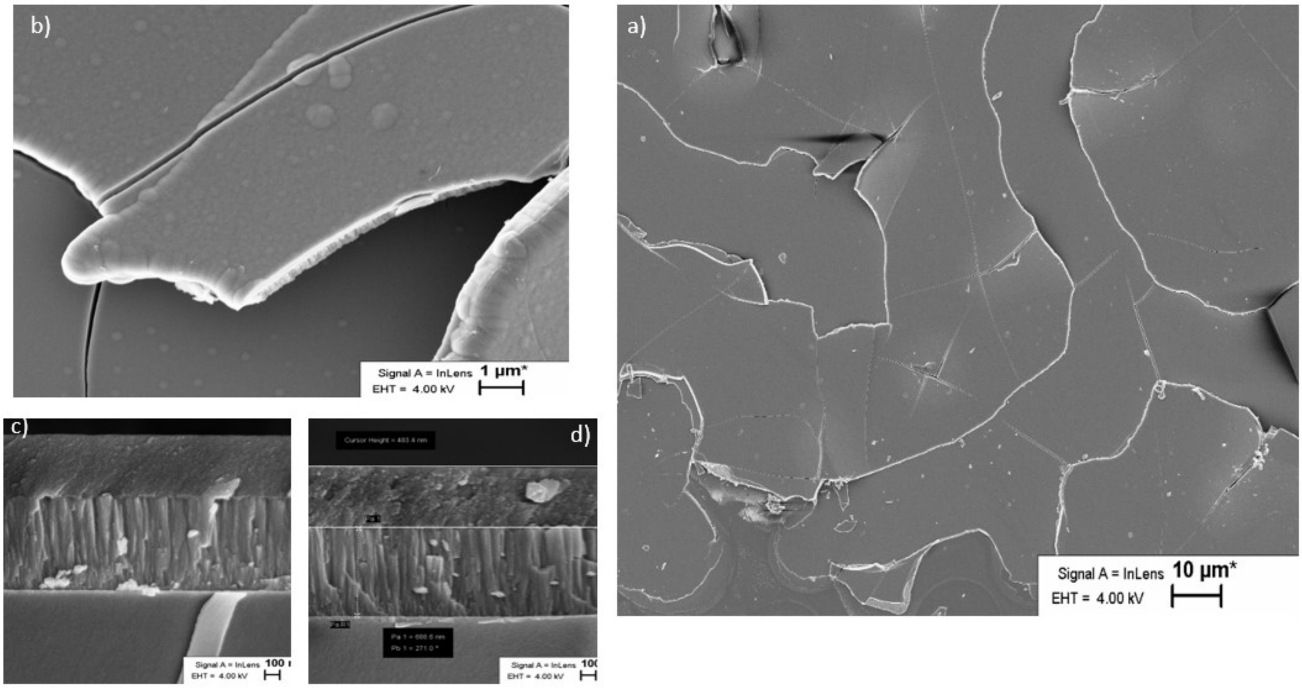


Figure 4.9. SEM image of TiO_2 thin films grown on YHO films by ultrasonic spray pyrolysis deposited at substrate temperature of 250ºC.

5.0. Conclusions

TiO_2 thin films were grown on YHO films by ultrasonic spray pyrolysis method using 0.1M TTIP. The growth temperature to properly encapsulate YHO films with TiO_2 thin films was determined. It was concluded that growth temperature has influence on the YHO films. The growth temperature of 100°C showed a uniform and well encapsulated YHO films with TiO_2 thin films.

The structural properties of deposited TiO_2 thin films on top of YHO films depend on growth temperature. Before illumination, all YHO films exhibited preferred orientation along plane (200) irrespective of growth temperature but sample Y02 has different preferred orientation. In the films exhibiting photochromic properties shift of the (111) peak toward larger angles is observed. The shift is reduced with increasing the temperature and disappears at 250°C when the films do not exhibit photochromic properties.

The optical properties of the encapsulated YHO films with TiO_2 thin films are dependent of illumination time. The optical transmittance of the encapsulated YHO films decreases as the films are illuminated. The decrease in transmittance is proportional to the illumination time.

The morphology of deposited TiO_2 thin films on top of YHO films is affected by growth temperature. TiO_2 thin films grown at substrate temperature of 100°C showed a uniform, smoother, and homogeneous thin films of the encapsulated YHO films. TiO_2 thin films grown at substrate temperature of 150°C indicated a uniform thin film but with emergence of cracks. Whilst TiO_2 thin films grown at higher temperatures of 200°C and 250°C showed a rougher damaged surface with cracks.

6.0. Summary

According to literature, YHO has its main application in smart windows which has led to the start-up of SunPhade in 2018. The commercial production of this material was limited due to the ceramic nature of YHO, dependence of photochromic effect on angle of incidence of sunlight, degradation and worsening of the film adhesion on the substrate. The encapsulation of YHO films with TiO_2 thin films was carried out to improve the strength and the photochromic properties of the material.

This thesis focuses on the deposition of TiO_2 thin films onto YHO films by ultrasonic spray pyrolysis method. The effect of growth temperature on the structural, optical, and morphological properties before and after illumination by UV light were investigated. Ultrasonic spray pyrolysis was used due to its simplicity and ability to cover large areas with homogenous droplets of thin films. The steps and cycles, and deposition temperature was determined before the study. The number of steps and cycles remained constant while the substrate temperature varied.

YHO films were successfully encapsulated with ultrasonically sprayed TiO_2 thin film. It was concluded that the optical, structural, and morphological properties of TiO_2 thin films deposited onto YHO films depend on growth temperature.

According to this study, using the steps number of 4 and a cycle number of 12 and growth temperature of 100°C leads to a compact crack-free TiO_2 layer that encapsulates YHO without affecting its photochromatic property.

XRD pattern reveals how the encapsulated YHO films lattice contracts under illumination and expand in the dark/air. This explains the decrease in optical transmittance of the films when illuminated.

The morphology of the encapsulated YHO films with TiO_2 thin films is dependent on the growth temperature. Encapsulated YHO films with TiO_2 thin films grown at substrate temperature of 100°C showed a smooth, homogenous, and well encapsulated YHO films without any damage, Increasing the growth temperature resulted in the increased number of cracks on TiO_2 layer on top of YHO films. YHO layers with TiO_2 films grown at 250°C resulted in a rough, cracked, and damaged surface. The thickness of the deposited TiO_2 thin films onto YHO films decreases as the temperature increases.

To fine-tune the properties of YHO films encapsulated with ultrasonically sprayed TiO_2 thin film, the effect of other USP deposition parameters, such as steps number and cycle number, should be optimised in detail.

7.0. Kokkuvõte

Kirjanduse andmetel on YHO põhiliselt kasutust leidnud nutiaknana aka *smart window*, mis on viinud idufirma SunPhade loomisele 2018 aastal. YHO keraamilise omaduse, päikesevalguse nurgast sõltuva fotokromaatilise efekti ja filmi degradeerumise ning puuduliku aluse külge adhesiooni tõttu on antud materjali masstootmine olnud limiteeritud. Antud töös kapseldati YHO kilede TiO2 kiledega, et muuta kile vastupidavamaks ja parendada materjali fotokromaatilisi omadusi.

Antud töö keskendub YHO alustele kasvatavatele TiO2 õhukeste kilede sadestamisele ultraheli pihustuspürolüüsi meetodiga. Uuriti sadestustemperatuuri mõju kilede struktuurilistele, optilistele ja morfoloogilistele omadustele enne ja pärast UV valgusega töötlemist. Ultraheli pihustuspürolüüs on kilede kasvatamise meetodina äärmiselt lihtne ning on võimeline katma ligilähedaste suuruste tilkadega suuri alasid. Astmete ja tsüklite arv ning sadestustemperatuur määrati kindlaks enne uurimistööd. Astmete ja tsüklite arv oli konstantne samal ajal, kui kasvutemperatuuri muudeti.

YHO alusete kapseldamine ultraheli pihustuspürolüüsi meetodil kasvatatud TiO2 kiledega oli edukas. Tööst järeldus, et YHO alustele kasvatatud TiO2 kilede optilised, struktuursed ja morfoloogilised omadused sõltusid kasvutemperatuurist. Töö tulemuste põhjal saab fotokromaatsete omadustega YHO kilesid kapseldada ühtlase ja mõravaba TiO2 kihiga kui TiO2 kasvatatakse järgnevatel tingimustel: aste 4, tsüklite arv 12 ja kasvutemperatuur 100C.

XRD tulemustest on näha, kuidas kapseldatud YHO kilede võreparameetrid kahanevad ja laienevad vastavalt UV kiirguse olemasolule või puudumisele. See seletab ka optilise läbilaskvuse vähenemise valgustatud objektidel.

YHO pind sõltub kapseldamiseks mõeldud TiO2 kasvutemperatuurist. 100C juures kasvatatud TiO2 kile on YHO kapseldamisel ühtlane, pidev ja sobilik YHO kapseldamiseks, kuna YHO kilel puuduvad vigastused. YHO peale kasvatatud TiO2 kasvutemperatuuri tõstmise tulemusena suurenes kile pinnal olevate mõrade hulk. 250C juures kasvatatud TiO2 kiledega kapseldatud YHO kile oli ebatasane, mõradega ning kahjustunud pinnaga. TiO2 kasvutemperatuuri tõustes väheneb YHO alustele kasvatatud TiO2 kilede paksus.

Ultraheli pihustuspürolüüsi meetodil kasvatatud TiO2 kiledega kapseldatud YHO omaduste täpsemaks töötlemiseks peaks optimiseerima ka teisi ultraheli pihustuspürolüüsi pihustusparameetreid nagu näiteks astmete arv ja tsüklite arv.

8.0. References

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Dedication

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