Low-ε Coatings for Energy Efficiency

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This report is submitted as a Final Year Project (FYP) thesis in partial fulfillment of the requirement for the degree of

BE Materials Engineering

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CERTIFICATE

This is to certify that work in this thesis has been carried out by **Hamza Khalid, Muhammad Bilal and Tanzeel ur Rehman,** and completed under my supervision in School of Chemical and Materials Engineering, National University of Sciences and Technology, H-12, Islamabad, Pakistan.

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DEDICATION

WE DEDICATE OUR THESIS TO OUR PARENTS, SUPERVISORS AND INSTRUCTORS WHO HAVE INSPIRED US TO HIGHER IDEAS OF LIFE AND TAUGHT US THE UPS AND DOWNS OF LIFE.

"WE CANNOT HELP EVERYONE BUT EVERYONE CAN HELP SOMEONE"
BY RONALD REAGAN

IN ALL WE DEDICATE OUR EFFORTS IN THIS WORK TO ALL OF HUMANITY.

ACKNOWLEDGMENTS

All praises are to Almighty Allah, who is the Creator, the Maintainer and the Regulator of the worlds. He is the One, who gives the power to us to think, utilize our expertise in knowledge in achieving remarkable solutions for the mankind in every field. It is His blessing on us that we are able to use our technical knowledge in accomplishing our work in the form of this Final Year Project.

We pay our gratitude to our Last Prophet (S.A.W.S) who through his sayings and actions, inculcated in us the spirit to get and excel in education, which is the root of Islam, as the first revelation revolved around the word "Read".

In Surah Zumr, Allah says in the **Quran**,

"Are those equal, those who know and those who do not know?"

This Quranic verse sums up the entire importance of the education in the lives of humans.

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-Bilal, Hamza, Tanzeel

ABSTRACT

Low-emissive (Low-ε) coating creates a surface that reflects heat (UV and Infrared radiations), while allowing visible light to pass through. It provides a solution to heat losses from the inside during the winters as well as blocking heat from the outside during the summers making it an energy efficient glass. The low-ε coatings are thin coatings of a few nanometers and can be coated via sputtering, spray pyrolysis or electrochemical deposition. Metallic coatings of Silver, Aluminum, Tin, Zinc etc give a low-ε value of around 0.05-0.10. The uniformity, stability and durability of these films is an important aspect of successfully deposition thin films on a glass substrate. These films will be characterized using SEM and UV-Vis-NIR spectroscopy.

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CHAPTER 1

Introduction

Over the years utilization of glass has increased exponentially in architectural aspects as there is a trend for higher buildings that use glass for a glazed beautiful look. Many such buildings use glass exterior but due to global warming the weather has changed a lot. During summers when the temperature is high such buildings get hotter inside and they have to use air conditioners to overcome such issues. In winters they need to get warmer inside due to this the finances increase by installing air heaters and the cost of their maintenance as well.

To overcome this issue, low emissive coated glasses were developed and till now their global market is gradually increasing and it is estimated to reach 4.26 billion dollars by 2025. These low emissive coatings help to make the interior warmer in winters and cooler in summers. The development of these low-ε coatings is one of the most active research topics of material sciences nowadays [1, 2]. This is due to their commercialization and economical prospect with extensive practical applications such as window glass [3], moisture resistance [4], mobile communication [5], engines [6], semiconductors [7, 8], thermal applications [9, 10], medical [11] etc.

Emissivity is the ratio of heat emitted or radiated by an object as compared to a black body. It ranges between 0-1. And as for blackbody its value is 1 because it is a perfect absorber of heat and hence a perfect radiator too i.e. the more a body absorbs the more it radiates. It is also described as the property of a surface to emit or radiate energy. When any surface is exposed to energy, some of it is absorbed; some is reflected back while the rest is transmitted through. Every material shows this phenomenon so as glass which is our focus. The thermal emittance of normal glass is about 84% so the development of a coating that reflects most of heat it receives and absorbs the least possible amount for that we focused to produce efficient low emissivity coatings. Solar spectrum compromise of 3 parts, UV (3%), visible (45%) and IR rays (52%) [12]. IR rays (long wavelength rays) have the highest weightage in solar spectrum and they are the main cause of heat so the focus of our study is to block these rays, and allow maximum percentage of visible rays to transmit through so that they it can be utilized for window glass.

Low-ε coating works as reflector to heat and its efficiency depends upon the material used[13], in our case we are going to mainly use silver whose emissivity value is 0.02 means it will reflect 98% [14]of heat. In summers, sunlight strikes building's window coated with low-ε material (Ag) and it will reflect 98% of IR rays while allowing visible rays to pass and this will keep inside cooler. Where in winters, heat rays (long wavelength rays) from inside, radiated from everything present in interior of building, strike the window and is reflected back inside by low-ε coating keeping the interior warmer thus reducing our energy usage all around[15]

There are a number of materials, based upon their properties and applications, are being coated on windows[16]. We will be examining Al, Sn, Cu and Ag but our primarily focus is on Silver-Ag due to its effective properties and our requirements. The emittance of silver is 0.02 percent which is mentioned earlier and the properties we are going to interpret and examine with glass are like thermal emittance, total solar energy transmittance, visual transparence, solar heat gain coefficient SHGC, U and R factor so basically all properties which are related to energy and visual performance[17-19].

CHAPTER 2

Literature Review

2.1: LOW EMISSIVITY

The ability of any material to radiate energy through it is known as emissivity. Low emissivity materials are those which emit less amount of energy. Basically a material having low emissivity will reflect more and radiate less. Low-ε materials decrease the transfer of thermal heat that occurs by thermal radiation. Low-ε glass reduces the amount of infrared and ultraviolet light to radiate through the glass without reducing the visible light entering to the rooms.[8] Low emissivity glass windows have very thin coating which is transparent and which transmit less and reflects more. These coatings are even thinner than the human hair. As the coating is very much thin so it has no significant effect on the visibility and colour of the glass window and has significant impact on energy efficiency by reducing the heat losses. Whenever the light or heat is absorbed by the glass two things can happen, either the surface of glass reradiates the energy or the moving air causes it to shift away. For any window the important component for the heat transfer is radiant heat emission. Thus the insulating properties can be improved by reducing the emittance of windows[20]

As the low-ε glass has excellent heat preservation characteristics it is greatly used as car windows and glass walls for buildings. Low emissivity glass windows helps to keep the interior of the room cooler in summers and hot in winters. In summers hot air from outside is not allowed to enter the room through the windows and in winters warm air from inside is not allowed to transfer through the windows[21]

<u>How to measure Emissivity:</u>

By using Stephan's Boltzmann law emissivity is measured:

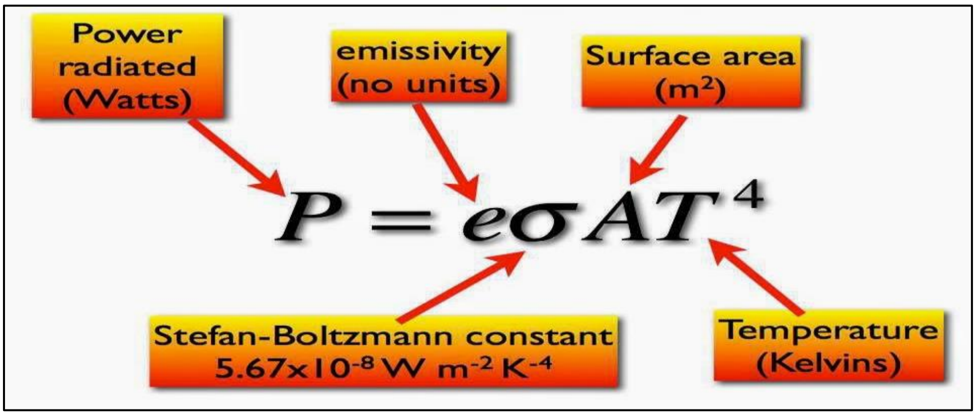

Fig 2-1: Stefan-Boltzmann formula for emissivity calculation

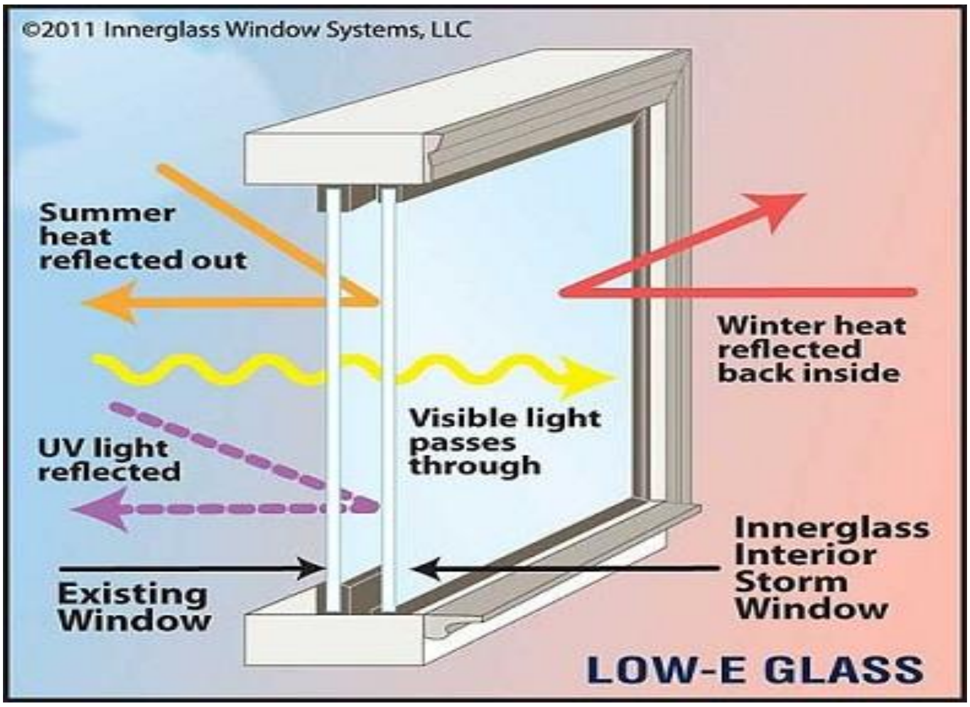

Fig 2-2: Low-E glass radiation reflectance and transmission

Different material shows different emissivity upon its characteristics:

Table 2-1: Low-ε values of metals and their oxides

Materials	Emissivity Values (no unit, dimensionless)
Carbon	0.85-0.95
Aluminum	0.11
Brass	0.61
Copper (oxidized)	0.60
Black gloss paint	0.90
Gold (polished)	0.020
Silver	0.02
Glass (uncoated)	0.91

Material	Temp (°C)	Emissivity
20-Ni, 24-Cr, 55-Fe, Oxidized	500	.97
Iron, Oxidized	499	.84
Molybdenum, Oxidized	371	.84
Nickel Oxide	538 - 1093	.59 - .86
Platinum, Black	260	.96
Titanium, Anodized onto SS	93 - 316	.96 - .82
Smooth Glass	0 - 93	.92 - .94
Fe_2O_3	24	.91
Al_2O_3	24	.94
ZnO	24	.95
$MgCO_3$	24	.91
ZrO_2	24	.95
MgO	24	.91
Glazed Silica	1000	.85

2.2 PROPERTY REQUIREMENT FOR LOW EMISSIVITY COATING

2.2.1 Low-ε Properties

For the low-ε coating high optical transmittance is required in the visible range and high reflectance is required in the UV and NIR range.

The emittance of low-ε glass coatings may be as low as 0.04. At its temperature such glazing will emit only 4% of the possible energy and so will reflect 96% of the incident infrared long wave radiation.

Visible light (400-700 nm) comprises 48% energy of the global solar spectrum. Infrared region IR, which is beyond 700 nm, makes 49% of spectrum and remaining 3% is of ultraviolet radiations (100-400 nm).

Coatings based on metals must be thick enough in order to obtain high IR reflection and enough thin to obtain high visible transmittance. These should be in the form of continuous film in order to prevent absorption. Ag, Al, and Au have high value of reflection of IR. So these are mostly used as heat-reflecting transparent coatings. But when exposed to the atmosphere thin metal films degrade easily and because of this reason multi-layer coatings are preferred more over single metal films.[22]

2.2.2 Optical & Thermal Properties:

(Reflectivity, Transmissivity, Refractive index, Absorption Coefficient)

Reflectivity:

Reflectivity or coefficient of reflection is defined as the ratio of the reflected power to the power incident on the surface.

Transmissivity:

Transmission or transmissivity (T) is the ratio of the transmitted radiation flux to the incident radiation flux. When all the phenomena of reflection, absorption and transmission are happening, then according to the conservation of energy:

$$\mathbf{R + T + A = 1} \qquad \text{(Eq. 2-1)}$$

Refractive Index:

It is the ratio of the speed of light in vacuum (c) to that in another medium (v). Mathematically

$$\boldsymbol{n = c/v} \qquad \text{(Eq. 2-2)}$$

Absorption Coefficient:

Absorption coefficient (α) is the fraction of radiant energy incident absorbed per unit length of the medium (in z direction)[23]. By Beer's law it is given as:

$$I(z) = I^{\circ} e^{\alpha Z} \quad \text{(Eq. 2-3)}$$

It is simply given as:

$$\alpha = \left(\frac{1}{w}\right) \ln\left(\frac{1}{t}\right) \quad \text{(Eq. 2-4)}$$

Where w is thickness and t is transmittance.[23]

R-Value:

R-Value is the capacity of any material to resist the flow of heat from it.

In our case, more the R-value of material the better it is. Formula for R-Value calculation is:

$$R = \frac{l}{\lambda} \quad \text{(Eq. 2-5)}$$

Where l is thickness of material and λ is thermal conductivity of it

U-Value:

U-Value is the insulation property of any material. It tells that how efficient a material is a heat insulator. Less the U-value of material more insulative it is. Formula for U-Vale is:

$$U = \frac{1}{R_{net}} \quad \text{(Eq. 2-6)}$$

Where; $R_{net}=R_1+R_2+\ldots..+R_n$

2.3 TYPES OF COATINGS:

There are two types of coat for low-ε glass which includes soft and hard coating.

2.3.1 Soft coating:

It is the coating that is done after the fabrication of glass by special equipment. It is termed as soft coating because it's easy to peel it off as compared to hard coating. It is also known as off line, sputtered or vacuum coating.

2.3.2 Hard coating:

The coating done at high temperature during the manufacturing of glass is hard coating. It is also known as on line or pyrolytic coating.

2.3.3 Comparison:

There are many environmental factors that effect soft coatings mostly humidity. Both of these coatings would reduce UV factor, same is case with SHGC in soft coatings but hard coatings would allow more value for SHGC. From hard coatings throughout the spectrum we can have about 90% transmittance but in case of soft coatings such results cannot be achieved which causes declined performance hard coating is a pyrolytic coat of metallic oxides along with some adhesives. They are deposited onto the glass surface when it is still hot generally scorched near UV region.

Table 2-2: Comparison of soft and hard coating

Attributes	*Soft Coating*	*Hard Coating*
Lifespan	1-5 Years	Unspecified under normal conditions
Manufacturing Repeatability	Low	High
Cost of Production	For a larger volume high	Automated and repeatability makes it lower

2.4 DEPOSITION METHODS:

Thin films can be deposited over substrates using different techniques which are listed below:

2.4.1 Sol Gel Coating:

In this kind of technique a glossy and hard film is obtained by applying a gel over the surface and drying it. Different kinds of solutions can be formed which basically gets deposited over the surface of substrate. The films could be obtained by allowing the substrate to dip into the solution or the other process is in which the substrate is mounted over the rotator which rotates the substrate and the drops of solutions are dropped over the substrate which by rotation force uniformly distributes over the surface.

Sol gel method is a simple and cost effective technique. By this technique complex and larger shaped substrates can be coated easily and larger material range depositions can be done by this process.

For not being a much cleaner process many unwanted atoms are present which ultimately effects the properties.[24]

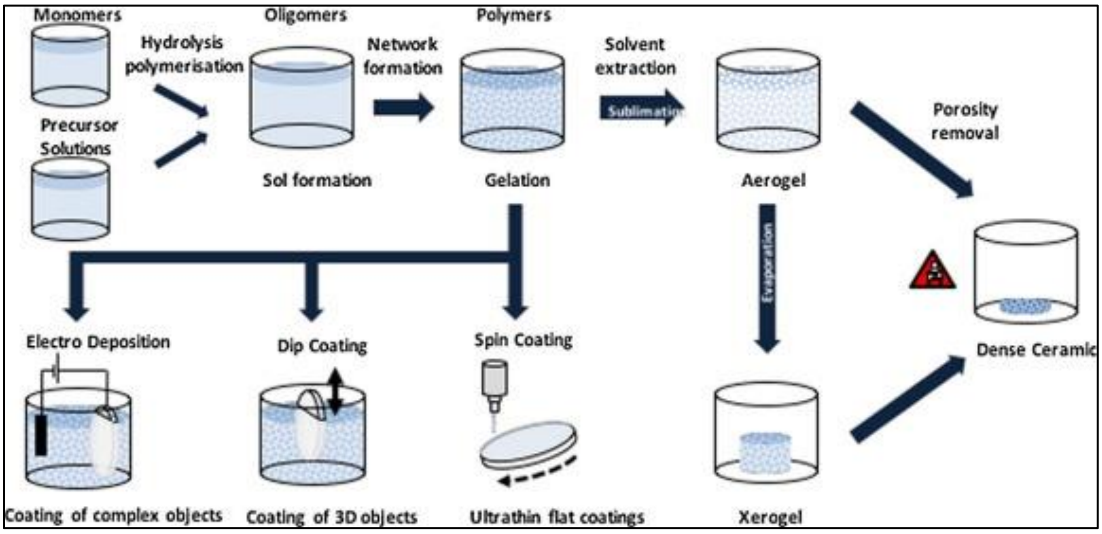

Fig 2-3: Schematic diagram of Sol-gel coating

2.4.2 Dip Coating:

It is considered to be easier as compared to others as the substrate is allowed to dip in a solution and after proper dipping the substrate is removed and allowed the solution to evaporate which leaves a thin are controlled by parameters vapor pressure and temperature.

Being a highly efficient method leaves minimal wastage of material. Substrates with complex geometries can have a complete coverage using dip coating.

For not being a perfect system the desired finish might not be achieved.[25]

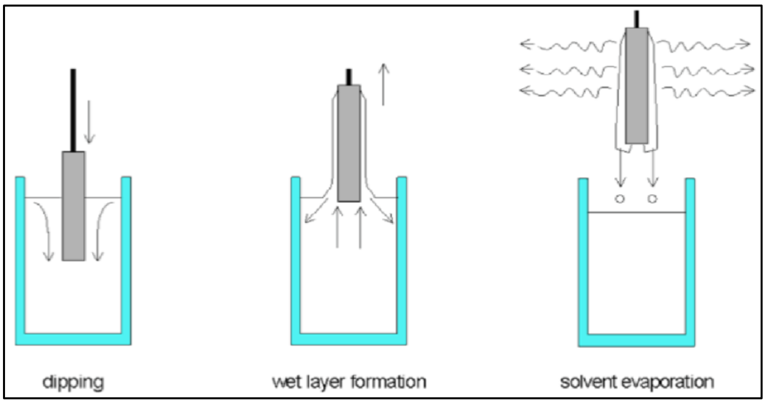

Fig 2-4: Schematic diagram of dip coating

2.4.3 Spin Coating:

Centrifugal force plays an important role for it as the drop deposited over the substrate, which rotates and due to centrifugal force and spin off uniformity is obtained. Due to this spin off the excess solution leaves in form of droplets. The solution evaporated from the film leaving a thin film behind.

Spin coating is relatively a simpler process through which coatings with uniform thickness can be acquired.

The extra material is removed in form of droplets which leads to wastage and the main disadvantage includes working over a single substrate at one time.[26]

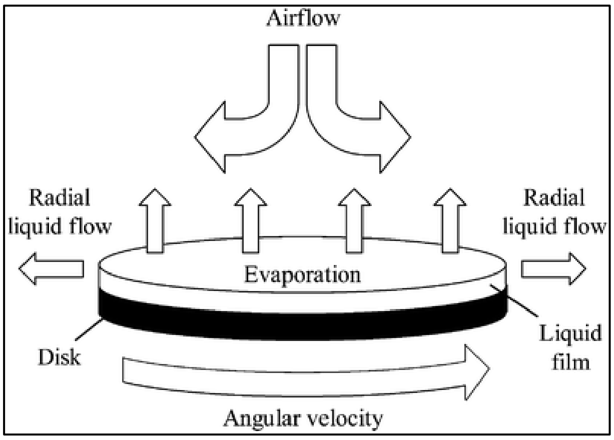

Fig 2-5: Schematic diagram of spin coating

2.4.4 Spray Pyrolysis:

In this kind of technique the solution is allowed to spray over the substrate by the help of an atomizer. The atomizer finally distributes the particles over the substrate placed and this can be done by using three techniques UV, Air blast and Electrostatic. The solution is evaporated to form micro sized droplets which afterwards are deposited over the film and sintered. It is one of the cheaper techniques and larger areas can be coated by this technique.

By the use of low cost spray pyrolysis technique indium tin oxide (ITO) thin films were prepared. The most common method for thin film preparation is the spray technique because of its simplicity; it also don't requires vacuum and hence comparatively cheaper method for large area coatings.[27]

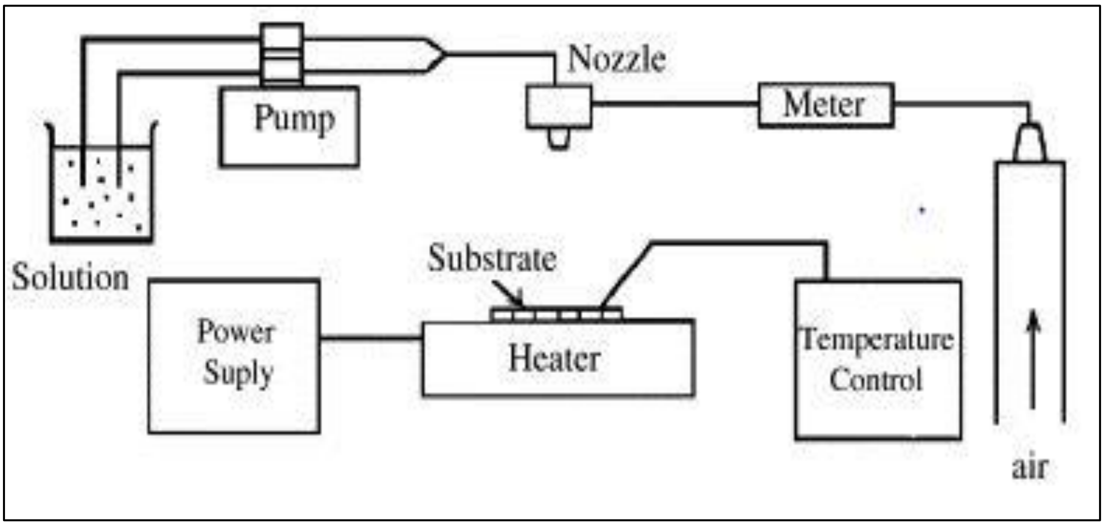

Fig 2-6: Schematic diagram of spray pyrolysis method

2.4.5 Sputter Coating:

This is done by acceleration of electrons originated from plasma and by their bombardment over a substrate. These highly charges particles are bombarded over a source material that actually gets eroded by these highly charged particles. The erosion results in origin of cluster of atoms or molecules which travel in some direction and by placing a substrate in their direction the target material can be coated over the substrate.[20]

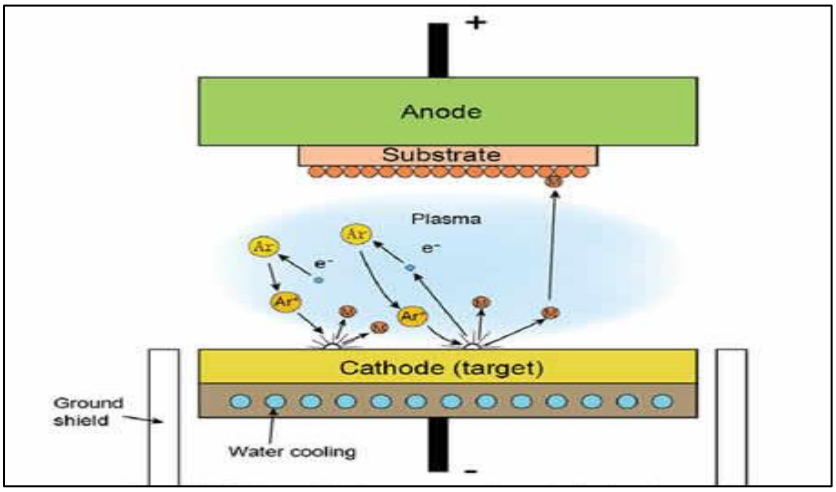

Fig 2-7: Schematic diagram of sputtering

2.4.6 Chemical Vapor Deposition:

This process involves a chamber having a heated substrate to be coated along with gases in it. These conditions results in chemical reactions over and near the substrate which results production of a thin layer over the substrate accompanied by unreacted gases and by products which are after that removed out of the chamber. This technique is widely used to get rid of defects that are caused by other processes.[28]

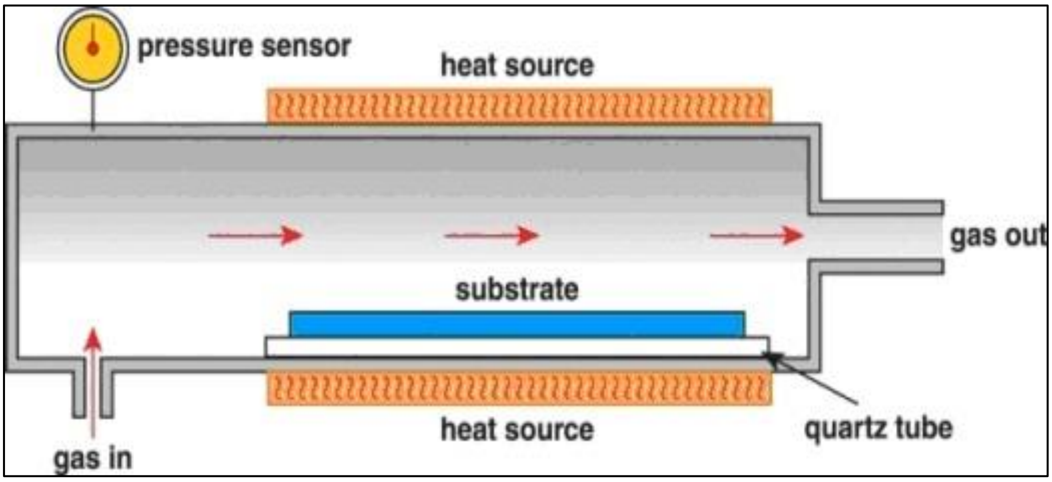

Fig 2-8: Schematic diagram of Chemical Vapor Deposition

2.4.7 Electro Chemical Deposition (ECD):

The other term known for it is electroplating. This process includes coating over a substrate that is due to the reduction of ions (metallic) present in the electrolyte. Electrolyte consists of both positive and negative ions in it so it makes it an ionic conductor. The cathode is made up of our working electrode which is to be dipped into the electrolyte in the designed cell along with anode which is referred as counter electrode. Both of these electrodes are connected to a battery source. The cathode is linked to negative terminal while anode to positive terminal, at anode reduction would occur which would reduce the metallic ions that are present in the ionic solution and these reduced metallic ions to atoms are deposited over the substrate. In the cell the positively charged ions carries the current from anode towards the cathode and this enables the ions to migrate and deposit as a thin layer over the substrate.

The longer the substrate would remain in the bath the thicker the coating would be. The advantages of this technique includes low consumption of energy, low wastage of material and lesser the capital investment.[29]

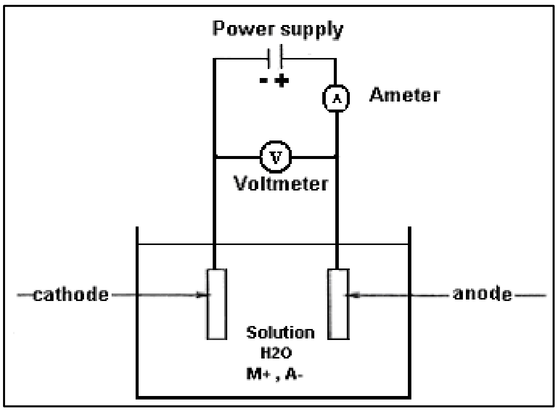

Fig 2-9: Schematic diagram of ECD

2.5 SUBSTRATE SELECTION:

Different processes can be used to coat different materials but it is necessary to have an appropriate substrate over which coating is to be done.

2.5.1 Fluorine doped Tin Oxide (FTO):

Being stable, mechanically harder, resistant to higher temperature, inert chemically FTO is considered as a promising material. Optical properties and electrical conductivity are increased due to wide thermal range properties. FTO features include color neutral glass which increases transmittance, electrical conductive and durable surfaces.

2.5.2 Indium dopes Tin Oxide (ITO):

The properties of this glass which makes it a choice as a substrate includes:

- Higher transparency from UV-IR region.
- Better conductivity.
- Lower micro roughness.
- Optical transmissivity of better homogeneity.

2.5.3 Soda Lime Glass:

Its composition consists of sodium oxide, SiO_2 and calcium oxide. The other name for it is crown glass. It is reasonably hard, chemically stable and inexpensive. The main component for is Silica. It is having higher glass transition temperature of 573 °C which can help us in annealing of samples for obtaining required phase.

They are used in everyday applications glass containers to window panes.

2.5.4 Polycarbonate Glass:

Polycarbonate glass offers higher impact resistance and is highly transparent to visible light. Such glasses includes carbonate group in their structures. They are having low resistance to scratches which can be overcome by doing harder coatings over them. It can hold longer than other glasses at higher temperature due to their melting point (155°C) and transition temperature (147°C). A proper surface treatment is required when material oxides are coated because they have no proper binder between sites on surface which results in weaker mechanical properties and lower adhesion.

Motivation:

The primary motivation was the worldwide energy crisis. Over the past the main energy source was fossil fuels which are depleting at very alarming rate. Due to which world is looking forward to renewable energy sources to meet future energy needs and solar radiation is the biggest renewable energy source. For that world started using large glass pans commercially and domestically to fulfill light demand through sunlight but with that the heating issue started arising caused by the IR region of solar spectrum, so to resolve this particular issue we carried out this project.

The other motivation was the industrial aspect of this project. In a under developing country like Pakistan entrepreneurship boost up the country's economy at high extent so this project was a good go by looking at the gap between demand and supply of energy efficient glass, as there is no considerable mass production of these glasses by any local glass industry.

Objective:

The main objective was to develop transparent thin low- ε metal coatings over ITO glass surfaces which have transmission in visible region and reflectance in IR region up-to 90% and to use them as energy efficient coatings at domestic and commercial level.

CHAPTER 3

Experimental Methodology

LOW-EMISSIVITY THIN FILMS

3.1 EXPERIMENTAL DETAILS

3.1.1 Materials

For the preparation of metal solutions for the deposition of thin films the following chemicals were used: Aluminum Chloride Hexahydrate (ACH) $AlCl_3 \cdot 6H_2O$, ethanol CH_3CH_2OH, Tin (II) Chloride (TCD) $SnCl_2$. All the reagents used were procured from Sigma-Aldrich of analytical grade purity and used as received without any further treatment.

3.1.2 Substrate pre-treatment

To deposit metallic thin films, 20x20 mm sized samples of soda-lime glass coated with a transparent conducting oxide (TCO) and ITO (for optimization) were used as substrates. The ITO substrates were sonicated in ethanol, followed by thorough rinsing in deionized water and finally drying in a laboratory oven at 100°C.

3.1.3 Solution preparation

The Aluminum metal ion solution was prepared by dissolving ACH in ethanol to prepare a solution of 0.1M. Similarly, 0.1M Tin metal ion solution was prepared. The solutions were magnetically stirred till a clear solution was obtained.

3.1.4 Film deposition

The metallic thin films were deposited using electrochemical deposition. The reference electrode used was Saturated Calomel Electrode (SCE), the counter electrode was of platinum and the working electrode was the substrate on which the film had to be deposited. The substrate was dipped in the solution of the respective metal. The time of deposition was varied between 30, 60, 90 & 120 minutes. For the deposition of tin metal thin film, a voltage of -0.8V was used and for Aluminum metal thin film -1.8V was used.

The figure summarizes the complete procedure to obtain energy efficient coatings on soda lime glass.

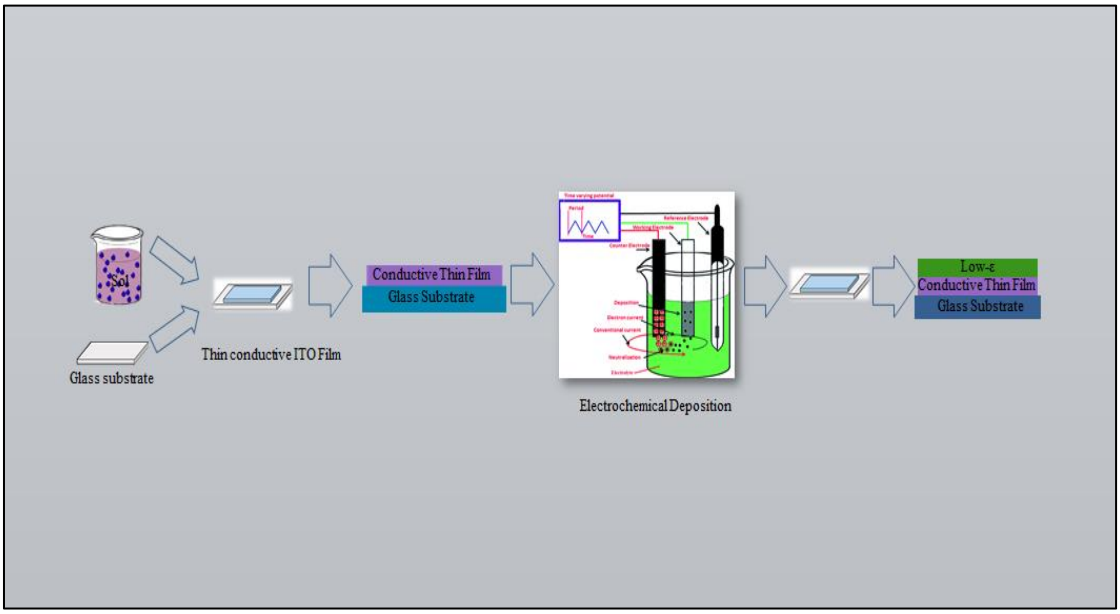

Fig. 3-1: Schematic representation of the methodology to make thin coatings of low-ε metals on soda lime glass.

3.2 CHARACTERIZATION TOOLS

3.2.1 Imaging Techniques

3.2.1.1 Scanning Electron Microscope (SEM)

In SEM, a fine probe of electron beam is focused and scans over the specimen's surface. Interactions of these electrons with the surface results in the electron or photon emission and a substantial portion of these emitted electrons/photons are collected by the detector. The output from detector modulates the brightness of cathode ray tube (CRT). For every point the electron beam strikes on the specimen's surface is plotted onto a corresponding point on the CRT and an image is produced. Secondary electrons (SE), backscattered electrons (BSE), and X-rays are produced due to the interaction of incoming beam and surface. SEs are low energy electrons emitted from resulting from inelastic scattering of primary electron beams scanning over the surface of specimen. BSEs are the high energy elastically scattered electrons of primary electron beam which are reflected out of the specimen's surface. X-rays can also be generated when high accelerating electrons impinge upon the specimen's surface. Using appropriate detectors information about phase contrast, chemical composition and surface topography (in elemental form) can be found.

The SEM provides a magnified image of specimen’s surface. Magnification of SEM can be as high as 300,000X with resolution approaching a few nanometers. SEM not only provides with surface topographical information but the composition of regions near the surface of material as well.

SEM analysis was performed using scanning electron microscope (JEOL-JSM-6490LA) with operating voltage of 10-20 kV, spot size of 35-60, and working distance of 10mm. The images were recorded in SE mode at low and high magnifications.

Figure 3-3(a) shows the JSM-6490LA SEM present in School of Chemical and Materials Engineering, National University of Science and Technology, Islamabad.

Figure 3-3(b) shows schematic of a typical SEM.

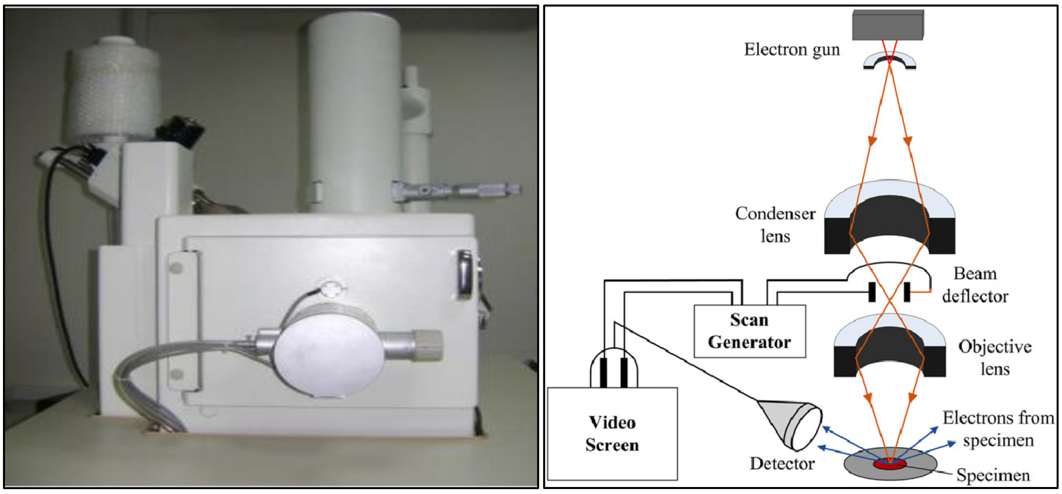

Fig. 3-2: (a) JOEL JSM-6490LA present at SCME; (b) SEM Schematic

3.2.1.2 Atomic Force Microscope (AFM)

AFM use a pointed tip on a flexible cantilever. The sample is scanned over by the tip, whenever the tip comes within a few angstroms of the surface of specimen repulsive van der Waals forces are generated between the atoms over the sample surface and the tip which results in deflection of cantilever. For the scanning of tip over the sample a piezoelectric sensor is employed over AFM and for maintaining constant separation between the sample and the tip over the scanner feedback control operates. To monitor the deflection in which light beam from a laser diode is reflected from the cantilevers’ back on a position-sensitive photodiode we use an optical lever. Due to extremely sensitive position sensor photodiode the vertical resolution of AFM is sub-Å whereas 1 nm is AFM lateral resolution. Figure 3-4 (a) and (b) show images of SPM available in SCME.

JEOL JSPM 5200 was used in tapping mode on an area of 2 × 2 μm2 for measuring of topography of deposited films and their roughness. The AFM studies were performed in air through operation in a tapping mode to maximize the tip–sample interactions. The probes used were micro-fabricated cantilevers (NSC35; μmasch) with respective values of length 130nm, nominal tip radius 10nm, spring constant 4.5 N/m, and resonance frequency to be 150 kHz.

Fig. 3-3: (a-b) Scanning Probe Microscope at SCME, NUST

3.2.2 Optical Characterization

3.2.2.1 UV-Vis-NIR Spectrophotometer

The working principle of the UV-Vis-NIR spectrophotometer is same as the UV-Vis spectrophotometer. The only difference is the range of wavelength that is used. Shimadzu UV-3600 Plus was used to measure the absorption, transmission and reflection over a range of 200 nm to 3300nm.

Fig. 3-4: UV-Vis-NIR spectrophotometer in CASEN, NUST

3.2.2.2 U-Value calculation Device:

We manufactured our own apparatus to calculate light transmission and heat blockage efficiency of our prepared samples and on the basis of obtained results we calculated U value of our samples.

Device is manufactured on proper standards. Height of box is 24 in and cross-sectional area is 8*8 in^2. Box is installed with a tungsten wired bulb which is used as light and heat source. Two slides are placed at optimum height difference with square opening at their centers. The openings are made to place samples there which need to be tested. A lux meter and infrared temperature detector device is used to measure the flow of heat and light through samples.

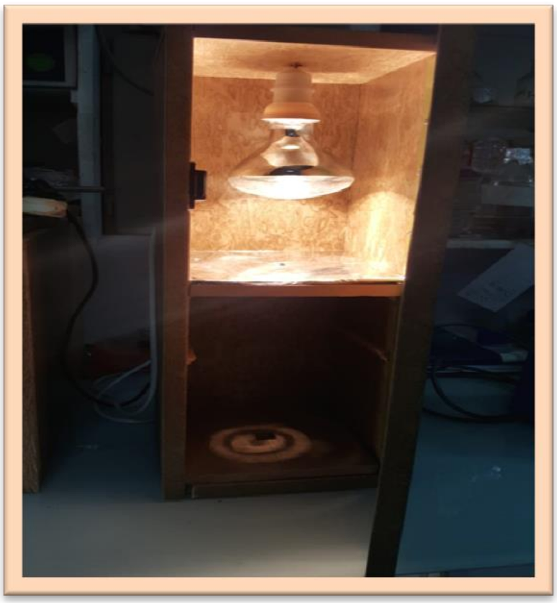

Fig. 3-5: Setup for the measurement of U-Value.

Fig. 3-6: Lux Meter (a) & Infrared Meter (b)

CHAPTER 4

Results and Discussion

LOW- ε COATINGS:

ITO glass was used for the electrochemical deposition process as ITO glass has a conductive layer of indium tin oxide over it. Through this process thin layers of Aluminum (Al) and Tin (Sn) were deposited over the substrates. Both of these metals have low emissive values; 0.04 and 0.05 respectively[16]. Due to such properties their surfaces reflect back the radiations (IR) that are responsible for cause of increase in temperature and therefore increasing the energy efficiency as their surfaces do not allow such radiations to pass.

4.1 Optical Properties:

Absorption is the transfer of radiant energy in to another form of energy which is by the interaction with matter. Reflectance of the surface of a material is its effectiveness in reflecting radiant energy. The absorbance of a medium is defined by the ratio of absorbed radiant power to incident radiant power. The reflectance is defined by the ratio of reflected radiant power to incident radiant power. Greater the absorption and reflectance of a material for certain wavelength fraction of incident solar spectrum i.e IR, lesser is that part transmitted through to produce heat and therefore it will be energy efficient. The contribution of UV, visible and Infra-red radiations in solar spectrum are 3%, 48% and 49% respectively.

4.1.1 UV-Vis-NIR Analysis:

To study the optical properties of our samples UV-Vis-NIR analysis was carried out. The samples were made from electrochemical deposition technique. Metallic salts; aluminum chloride hexahydrate ($AlCl_3.6H_2O$) and tin chloride ($SnCl_2$) were used as the source of Al and Sn metals respectively. Changes were also observed by varying deposition time.

By using this analysis, graphs of transmittance, reflectance and absorption versus wavelength were plotted. From these graphs the transmittance, reflectance and absorption of coated samples were analyzed over the long wavelength range (200-3000 nm) of the solar spectrum. This technique gave us a detailed analysis over UV, Visible and IR regions; whereas our main focus was visible region for optical transparency and IR region for heat causes.

Generally UV region range from 100nm to 400nm while visible from 400nm to 700nm and infrared region from 700nm to 3300nm.

4.1.1.1 Prepared Samples:

ALUMINUM (Al):

TRANSMISSION:

A graph of transmission vs wavelength is plotted and the result is analyzed.

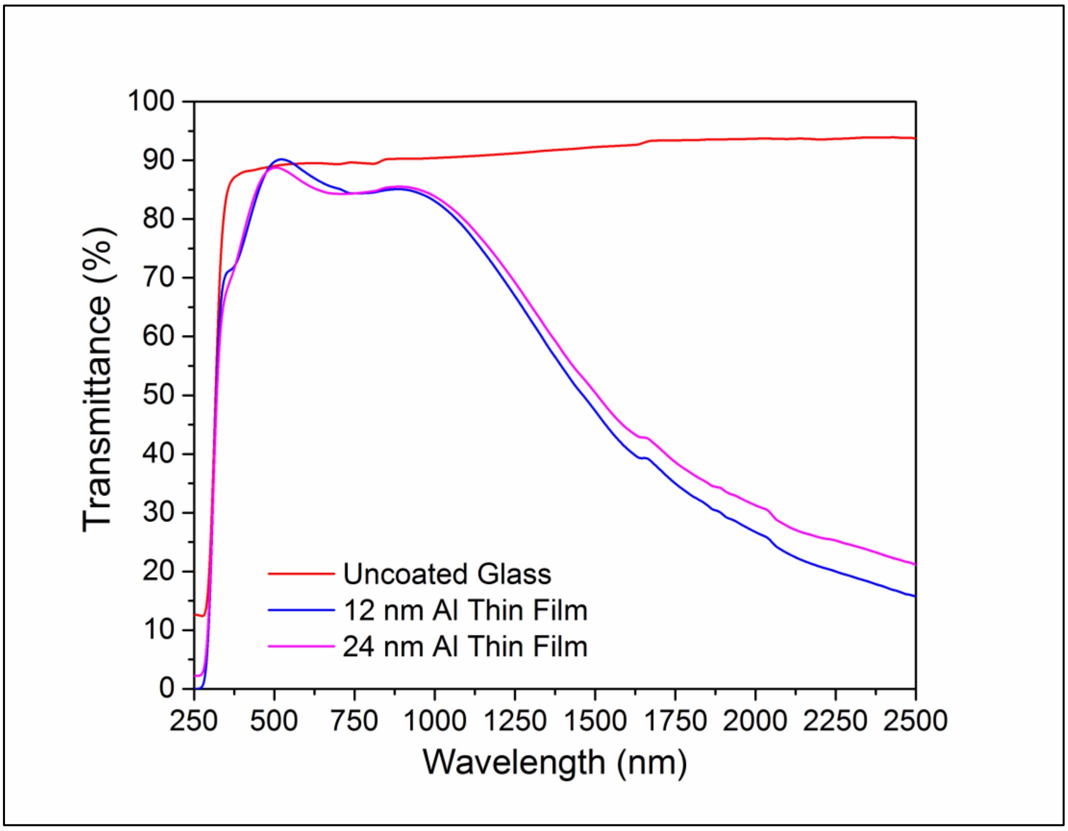

Fig. 4-1: Optical transmission spectra of Al in comparison to uncoated ITO glass

In case of uncoated glass it was observed from the results that about 90% transmittance of light in visible (350nm-700nm) and infrared region (700nm-3300nm).

For aluminum the time parameter used was 1 & 2 hr. 12nm thick layer was deposited over the period of 1 hr while 24nm thickness indicates deposition time of 2 hr. In case of 12nm it was observed that in visible region the transmission was around 90% which was the required result and in case of IR region its transmission dropped to even 20%. So the results showed that there was more transmission of visible light while the coatings did not allowed the infrared radiations to transmit. Same was the case for 24nm thickness but in this case the visible light transmission was somewhat more than that of 12nm making it much efficient in this case.

ABSORBANCE:

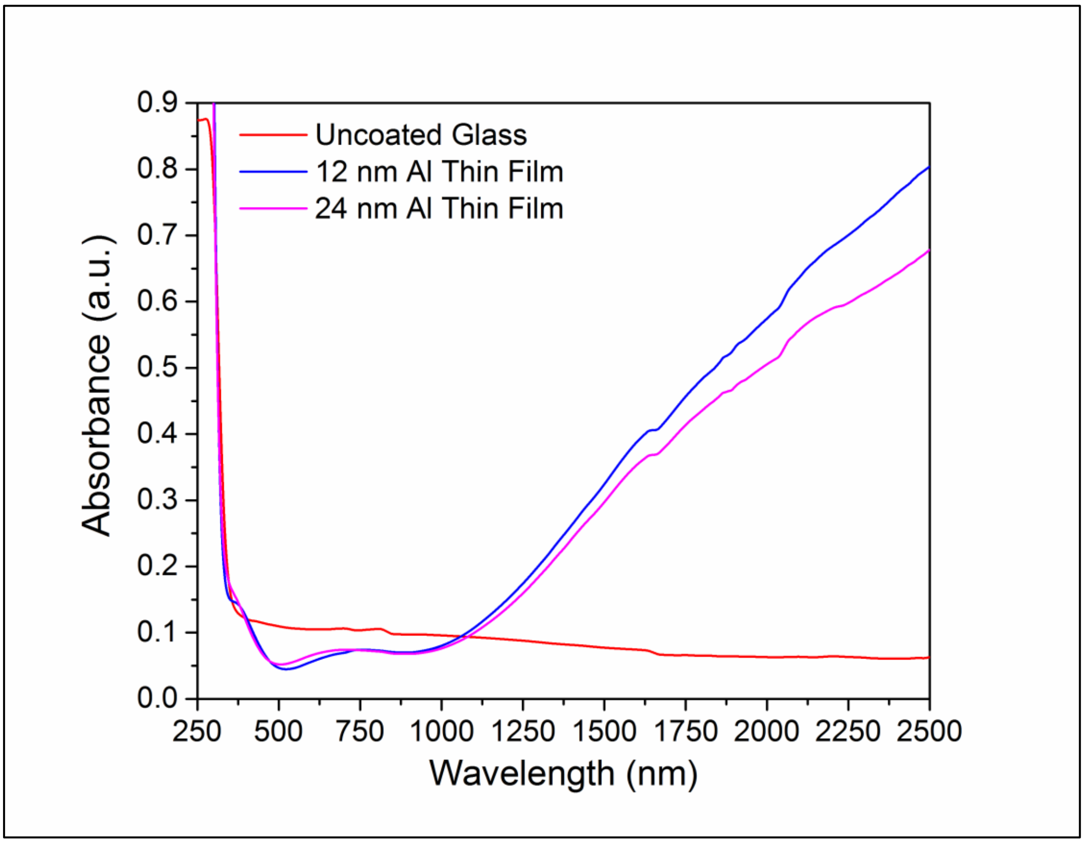

Fig. 4-2: Optical absorption spectra of Al coatings in comparison to uncoated substrates

For an uncoated glass the absorbance was very less in all regions of spectrum passing through it.

In case for 12nm thickness the absorbance in visible region is significantly low which matches our requirements as it needs to be lesser in amount so that visible light can pass through the coated glass and about less then 20% absorbance was observed, for inrared region the absorbance needed to be higher which is clearly seen in results.

In case of 24nm coated layers of Al the results were somehow same but the absorbance slighlty droped in IR region.

REFLECTANCE:

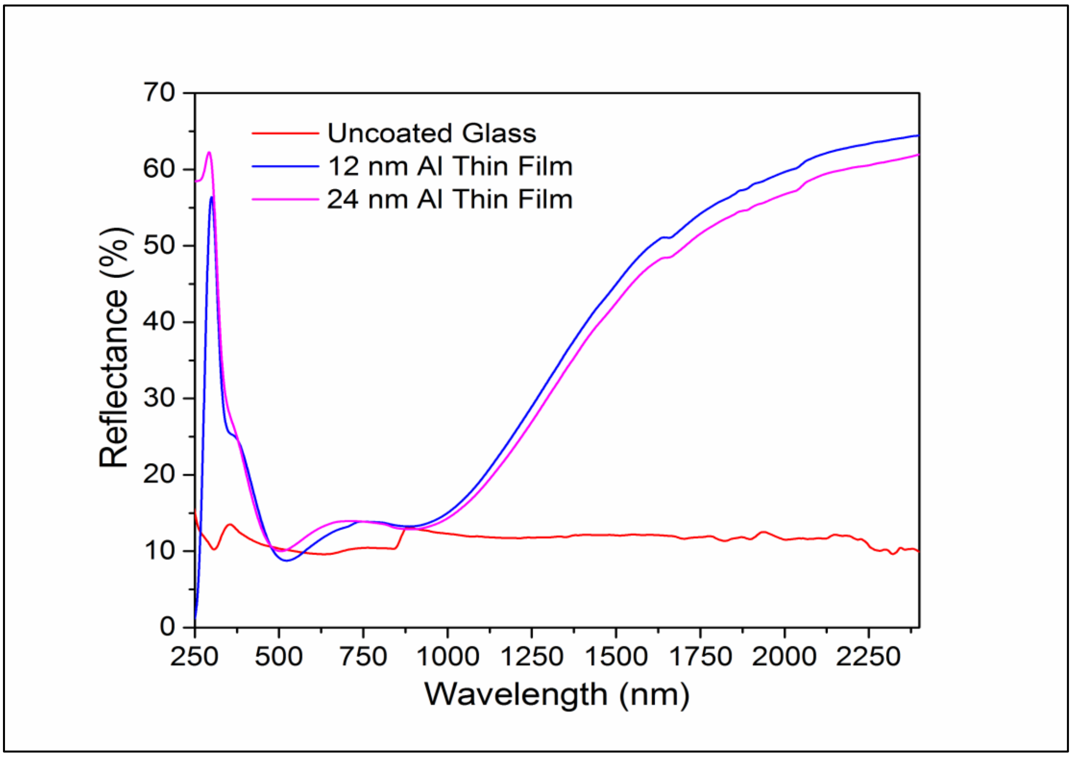

Fig. 4-3: Optical reflectance spectra of Al coatings in comparison to uncoated substartes.

For uncoated glass there was less than 12% reflectance of in any of the regions. As the IR region is responsible for cause of increase in temperature so from the results it was observed that reflectance gradually increased to more than 60% by doing coatings making the glass enery efficient. For visible light to pass through there should be less reflectance which can be seen in results that there was less then 15% reflectance for visible region in both cases (12nm& 24nm).

Tin (Sn):

TRANSMISSION:

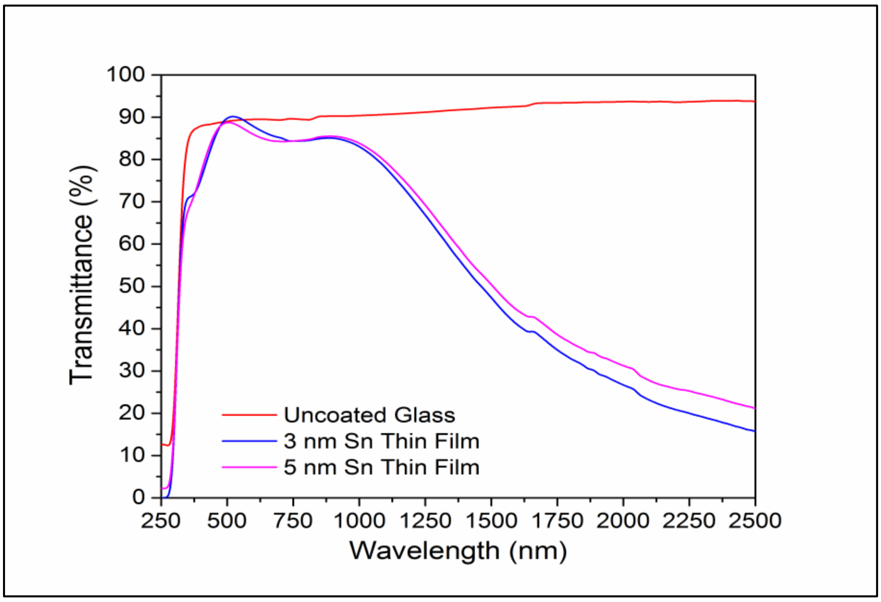

Fig. 4-4: Optical tranmission spectra of Sn coatings in comparison to uncoated substrates.

3nm thin film represents coating for 1 hr while 5nm for 2hr. In both of the cases it was observed from the results that there was more 85% transmittance of visible region and the transmittance of IR region gradually droped to 20% which were the desired results.

ABSORBANCE:

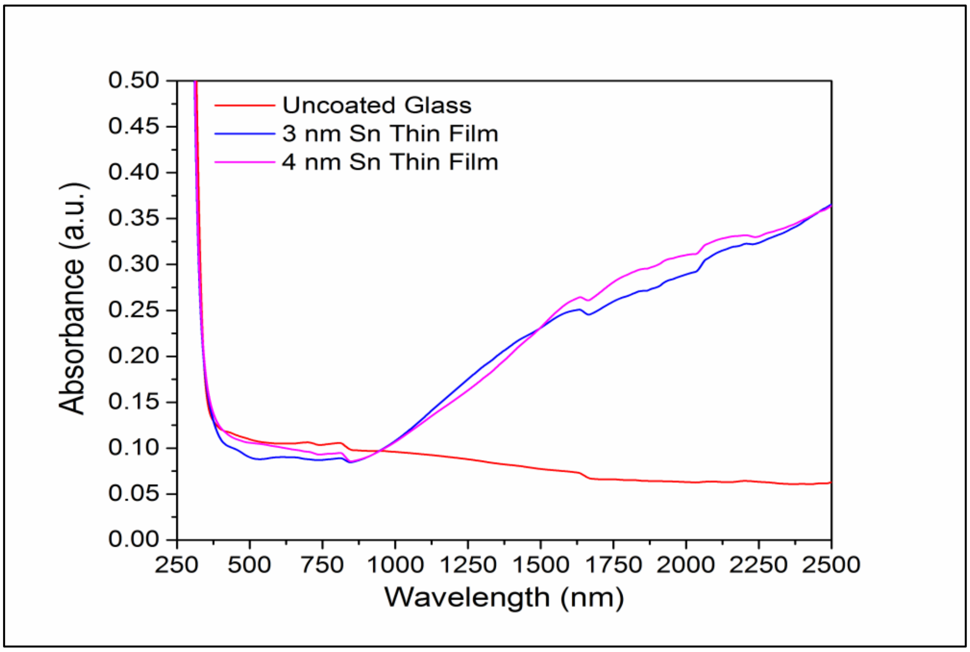

Fig. 4-5: Optical absorbance spectra of Sn coatings in comparison to uncoated substrates.

As already mentioned, IR being the main cause for increase in temperature so it needs to be restricted from passing through. There was a little absorbace of visible region of light observed while there was more than 60% absorbance of IR region. From the results it can be seen that 4nm thick coatings were slightly more efficient. As the UV region is not responsible of causing heat and it makes a few percent of spectrum of light so it was not under considerstion and it did not effect the results as well.

REFLECTANCE:

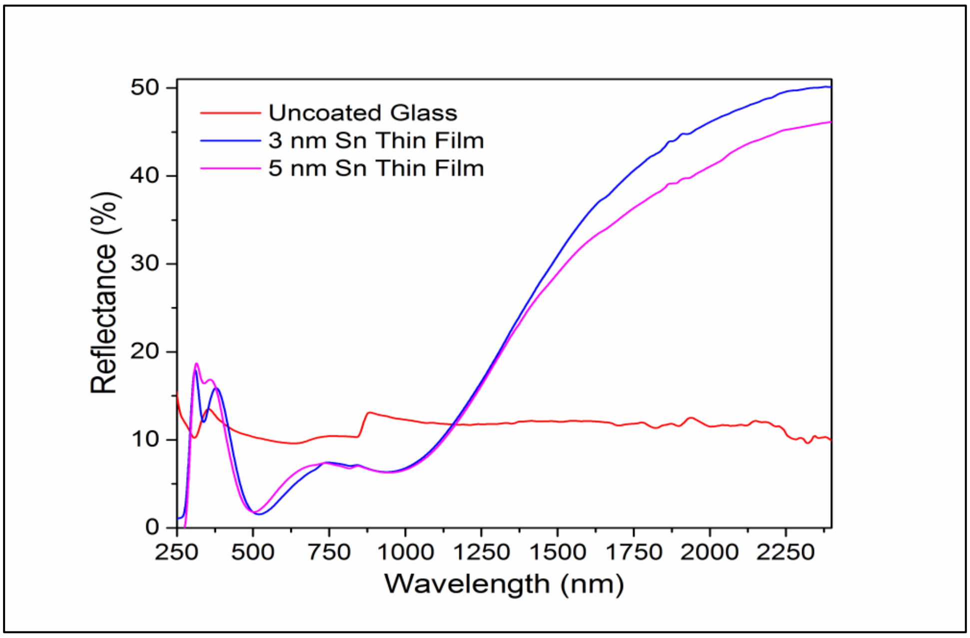

Fig. 4-6: Optical reflectance spectra of Sn coatings in comparison to uncoated substrates.

For both UV and visible region it was observed that there was less than 5% reflectance and as the region progressed the reflectance got incresed to more than 50% which was the desired result for any low emissive coated material

4.1.1.2 Reference Samples:

We brought two types of Pilkington glass, which is imported in Pakistan for commercial purposes as low-ε glass, as reference to compare the effectiveness and efficiency of our low-ε samples. Following are the results of those reference glass samples:

ABSORPTION:

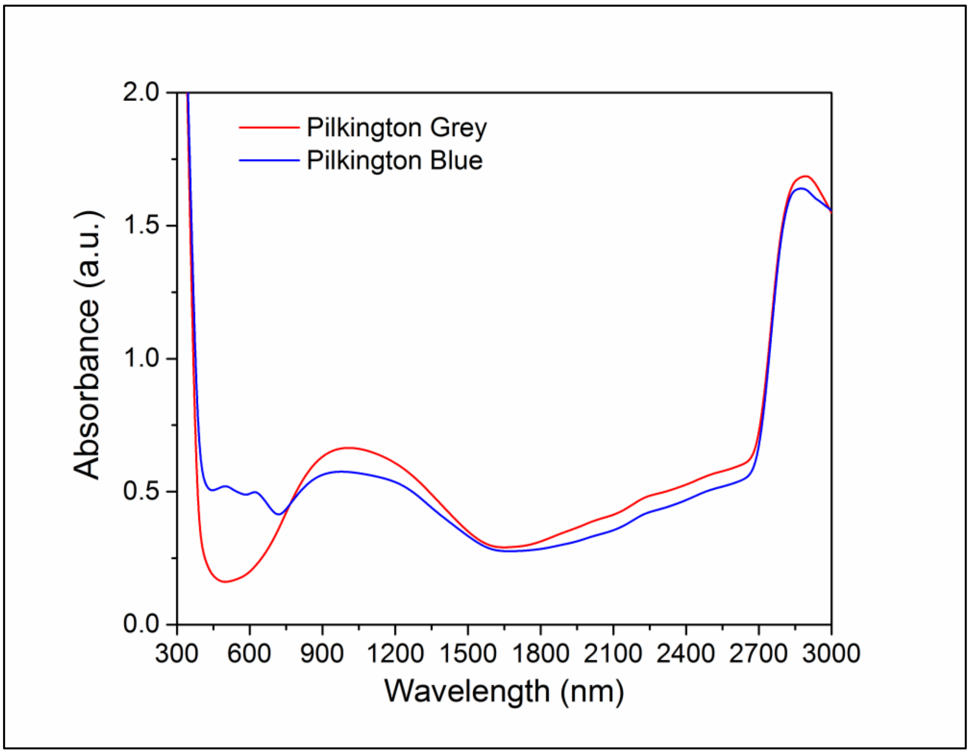

Fig. 4-7: Optical absorbance spectra of Pilkington glass samples

TRANSMISSION:

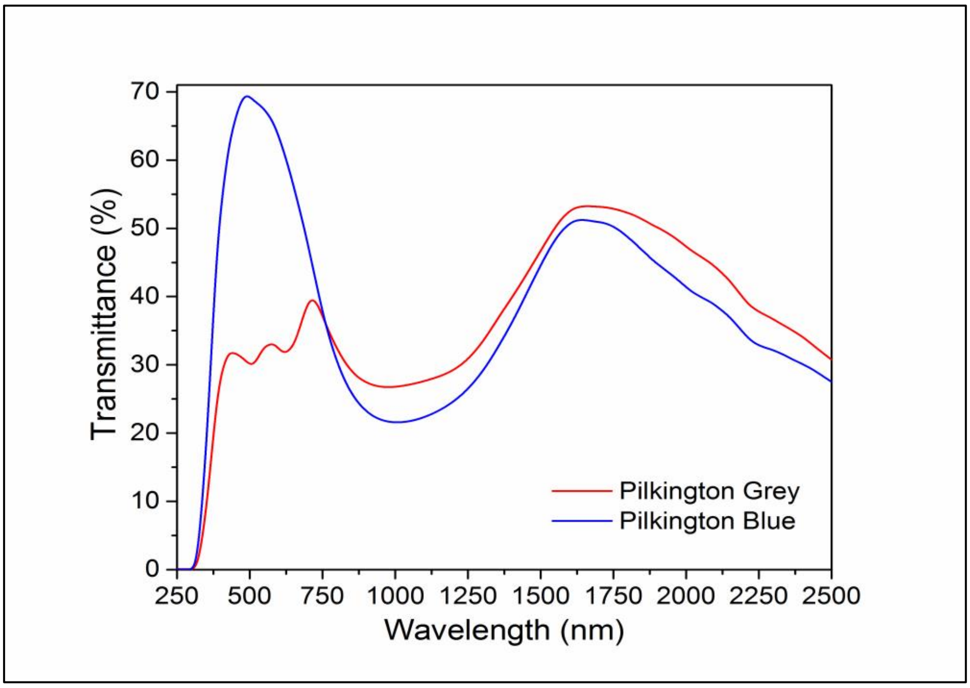

Fig. 4-8: Optical absorbance spectra of Pilkington glass samples

Pilkington glass samples show high reflectance in IR region but in visible region the transmission is bit low which is due to induced colour in them. So by comparing results of our prepared samples with that of Pilkington samples we clearly observe that the efficiency of our prepared samples is better than that of Pilkington samples.

4.2 Surface Analysis:

4.2.1 Scanning Electron Microscopy (SEM):

To study the morphology of the prepared samples surfaces SEM analysis was carried out. SEM analysis tells us about the surface smoothness, particles distribution and average particle size of deposited species. The results of SEM are:

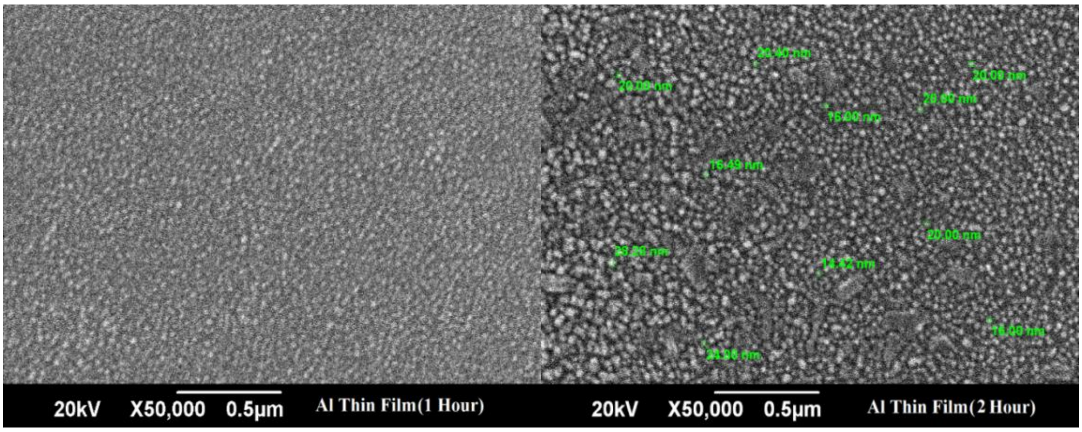

Fig. 4-9: SEM images of thin films of Al

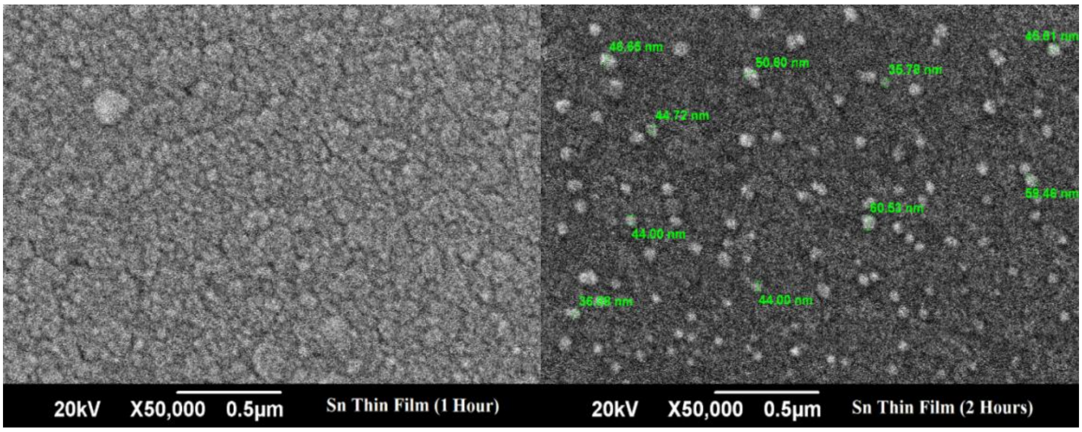

Fig. 4-10: SEM images of thin films of Sn

Samples coated for 1hr & 2hr of both Al & Sn were analysed. The results indicates the uniformity of surfaces. It also shows the uniform size distribution of particles and the average particle sizes for Al & Sn coatings are 18nm & 48nm respectively. Hence of requirement of uniform distribution of particles was achieved.

4.2.2 Atomic Force Microscopy (AFM):

To further study the tropology and analyse the roughness distribution, AFM characterization was carried out. The 2D & 3D images of film are shown in figures:

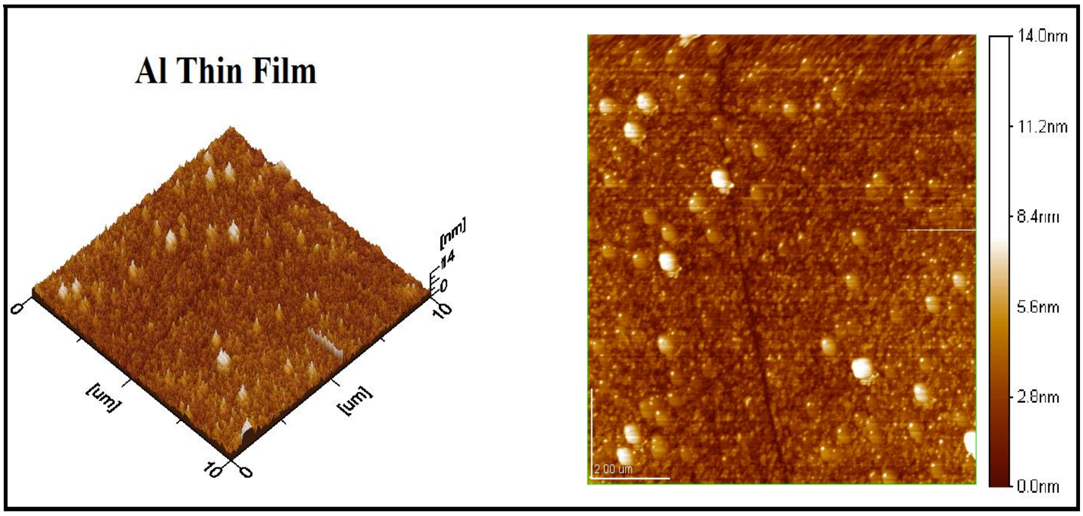

Fig. 4-11: AFM images of thin films of Al

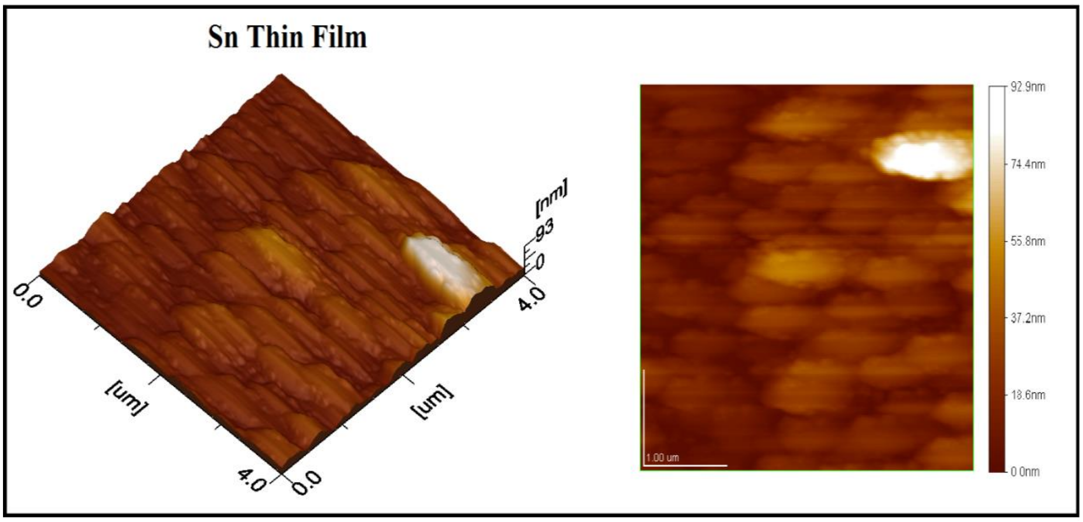

Fig. 4-12: AFM images of thin films of Sn

The results for both the coatings of Al & Sn indicate that the surfaces are uniform and smooth which was required. And they also show that surfaces have uniform roughness and uniform particles size distribution.

We require that most of the incoming IR region to be get reflected back and when the surface is bit rough it provides more sides to disperse the incoming beam and reflect it back to the first medium, hence these results support the required results.

Table 4-1: Root mean square roughness of Al and Sn metal thin film coatings

Sample Name	Rq (Root Mean Square) Roughness
Al metal coating	0.571 nm
Sn metal coating	1.353 nm

4.3 Phase analysis:

4.3.1 X-Ray Diffraction (XRD):

To identify and analyze the present phases of coatings XRD characterization techniques was used. Following are the graphs obtained from XRD in which angle is plotted against the intensity peaks of present phases:

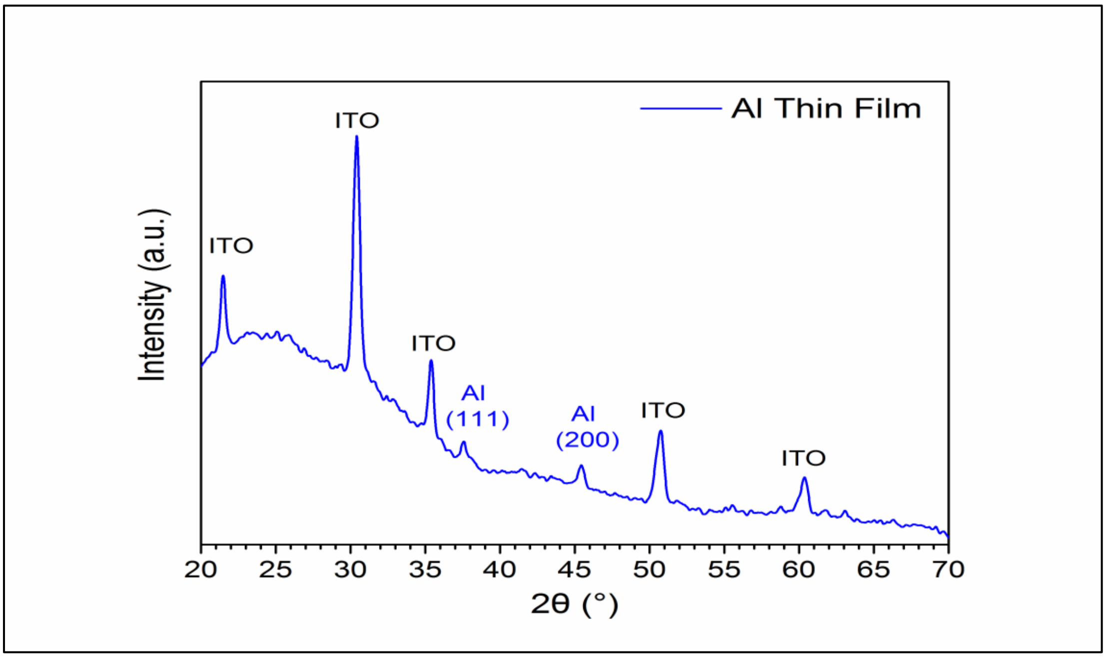

Fig. 4-13: XRD graphs of thin films of Al

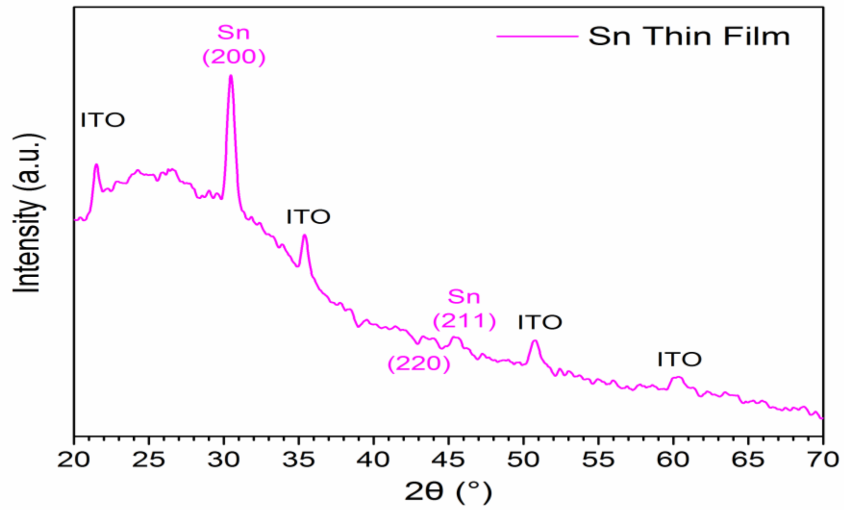

Fig. 4-14: XRD graphs of thin films of Sn

These graphs clearly show the peaks of ITO which is the conductive coating done for the deposition of metals over glass by ECD and beside that the peaks of Al & Sn phases which are Al(111), Al(200) & Sn(200), Sn(211) respectively. These results indicate that the glass substrates are coated with Al and Sn metals. The peaks are not that much sharp which is due to the reason that the thickness of coatings is very less.

4.4 APPARATUS

4.4.1 U-Value calculation Device:

The value percentage of visible light transmission and IR blockage was measured using our own manufactured apparatus and by that U-Value was calculated.

Light transmission:

Light passed through the opening of slides and below the slide lux meter detected the incoming light value in lux. The samples were placed turn by turn on slides to calculate the percentage of light transmitted through them.

Heat blockage:

Heat from the bulb passed through the opening of slides. Samples were placed turn by turn on slides. The temperature on the upper and lower sides of glass samples was calculated using the infrared gun meter and on basis of these obtained values heat blockage efficiency of samples was calculated.

U-Value:

By using the above obtained values we calculated the U-Values of our samples. Following are the calculated results of our samples and reference samples. These results clearly show that the efficiency of our prepared samples is higher than that of reference samples.

Table 4-2: U-Value of low-ε glass samples

SPECIMEN	LIGHT TRANSMISSION %	HEAT TRANSMISSION %	U-VALUE
Pilkington	58	84	3.8
Simple Glass	93	55	5.6
Al Coated	86	45	2.9
Sn Coated	64	50	3.6

CHAPTER 5

Conclusion

5.1 CONCLUSION

Single layer thin film coating was deposited on soda lime glass to make it energy efficient. Low-emissive metallic thin films were deposited using electrochemical deposition. Thin films of Aluminum and Tin were deposited at room temperature on glass substrates and the effect of incoming solar beam was thoroughly investigated by varying time period (15min, 30min, 45min, 60min, 90min, 120min), which increased the thickness with the increase in time period. The effect of the metal thin films on the reflectance in the Near Infrared (NIR) range (700-3300nm) was compared with that of uncoated ITO. Both thin films show an increasing trend in the absorption and reflection of NIR. Aluminum reflected more NIR than Tin where both films showed a constant transmission (>85%) in the visible range which was the basic requirement of whole work. The overall thickness of the multilayer coating is 6nm & 24nm for Al & Sn respectively for the deposition time period of 2hr. The reflection in IR region, which cause the blockage of heat, increased with the increase in thickness of metal coatings but with the increase in the thickness the transparency in visible region started decreasing. In summary, a highly transparent and energy efficient thin film coating was deposited using a low cost solution based method.

5.2 FUTURE RECOMMENDATIONS

Following future work needs to be done for improvement and further development of present results.

5.2.1 Low emissive Properties of other metals

Other metals show promising emissive ability such as Silver, Gold which need to be deposited for better comparison and more effective results as they are expensive but have low emissivity than Al & Sn.

5.2.2 Mechanical Testing of Thin Film

The thin films need to be tested for their mechanical strength against abrasiveness. This can be done by the following tests:

1. Scotch tape peel-off test
2. Weather resistance tests

5.2.3 Deposition Using Other Methods

These films can be deposited via other methods like sputtering, spray coating and physical vapor deposition. A comparative study between different methods of deposition would show which method gives better property for commercialization purposes.

5.2.4 Large Area Coating

For the commercialization of these coatings, they have to be deposited on large area samples using sputtering, dip-coating or spray pyrolysis method. Dip-coating and spray pyrolysis are both low cost, solution based methods where sputtering is expensive but very efficient with great control.

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