Protective and Highly Reflective Multi-Layer Thin Films for Solar Thermal Reflectors

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MASTERS of SCIENCE in

THERMAL ENERGY ENGINEERING

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THESIS ACCEPTANCE CERTIFICATE

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For my Beloved Parents !

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Abstract

Energy from the sun light is being used to produce the electricity by solar PV and solar thermal power plants. Concentrators used in solar thermal power plants use coatings that have to survive in all environmental conditions. There are two kinds of reflectors 1st surface reflectors (reflect light from the front) and second surface reflectors (in which light is reflected from the back side). Reflectance of the 1st surface reflectors is more than 2nd surface reflectors but they need protection from the severe weathering conditions. In this regard, Multilayer reflective and protective thin films of the Chromium, Silver, Aluminum and Silica were prepared on the glass substrate. These thin films were coated by using Physical Vapor Deposition technique. Chromium (20nm) was used as the adhesive layer between the glass and reflective layers of the Aluminum (100 nm) and Silver (130nm) that were coated by using thermal evaporation method. The protective layer of Silica (200nm) for these reflecting thin films was coated by RF-sputtering. These thin films were then characterized by using X-ray diffraction, Scanning electron microscope, Atomic force microscope and UV-VIS-IR spectroscopy. The characterization results showed a compact and homogeneous surface morphology of the thin films and surface roughness measured by AFM also resulted roughness as low as 7 nm in case of Ag thin film. Prepared thin films showed very high reflectance upto 92%, 89% for the Ag and Al respectively even after performing the tape test for the adhesion the thin films showed about 85% and above reflectance that makes them suitable for the application in solar thermal power plants.

Keywords: *Thin-Films, Solar Reflectors, Physical Vapor Deposition, RF-Sputtering, Solar Power Plants*

List of Figures

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List of Publications

- **Muhammad Asad**, Muhammad Awais, Farhan Ahmad, Nadia Shahzad **"Development of Al Thin Films by Thermal Evaporation for Solar Thermal Reflectors Applications"** Proceedings of International Conference on Nano-Composites and Multifunctional Materials, NUST Islamabad, Pakistan, August 21-23,2017.

- Hamza Ahmed Raza, **Muhammad Asad**, Nadia Shahzad, Majid Ali, Sara Sultan **"Effect of Heliostats Reflectivity on the Cost of Solar Thermal Tower Power Plant"** Proceedings of International Conference on Energy Conservation and Efficiency, UET Lahore, Pakistan, November 22-23,2017.

List of Abbreviations

DC	Direct Current
RF	Radio Frequency
STPP	Solar Thermal Power Plants
Ag	Silver
Al	Aluminum
SiO_2	Silica

CHAPTER 1

Introduction

Energy is going to be one of the major challenges in the world as the demand for energy is increasing day by day. To meet the concerns regarding the energy security, environment and global climate change, renewable energy technologies are in focus worldwide. Solar energy applications are everywhere in residential to commercial and industrial to agricultural fields [1].

1.1 History of Solar Energy

Sun's diameter is 1.39×10^9 m and it has 5762K of black body temperature. The over-all energy coming from the sun is 3.8×10^{20} MW and that energy radiates in all directions, only 1.7×10^{14} KW of the entire radiations produced are received on Earth surface. The energy reaches earth surface in just eight min and twenty seconds that is 1.5×10^{11} m away from us [2]. Surprisingly, use of the solar energy was based on the concentrating collectors which are very hard to apply as they have to follow the Sun for which their construction and shape are a tough ask itself [3]. Back in 212 B.C. Archimedes (the Greek scientist) used concave mirrors to set the Roman navy ships on fire by focusing hundreds of the mirrors on the one ship. He also wrote a book "On burning mirrors'' but evidence of it is not available as no copy of this book lasted. In 18^{th} century solar furnaces were built which were made of glass lenses, mirrors and polished-iron. These were used to melt the Iron, copper and other different metals. In 19^{th} century several efforts were made to use solar energy into other systems of energy. Augustin Mouchot, who is a French inventor, invented the first engine that was driven by solar energy which produced mechanical power. In 2^{nd} half of 20^{th} century many projects were designed for using the solar heat to power the mechanical loads by concentrating the sun light using reflectors [3].

1.2 Pakistan's Potential for Solar Energy

Pakistan being an energy deficit country is facing a lot of hurdles in meeting the demand for its energy needs. Due to limited fossil fuel resources Pakistan has to import the fossil fuel which affects the country's poor economy [4]. In 2008, 71% oil needs of country were imported and the average annual increase in electricity demand was 8% in 2005-2010 and will continue to increase further [5]. Pakistan being under the solar belt receives high isolation of nearly 1KW for square meter of its landmass for about 7 hours on average daily basis. This shows it has a great potential for using any kind of solar driven system application. Potential of solar energy is that energy which can be provided by the sun annually. Till 2010 The percentage of renewable energy was less than 1% of total energy share in Pakistan [6]. Pakistan has built its 1^{st} PV based solar power plant which is producing 100 MWp in 1^{st} stage and has further capacity of producing 900 MWp in following two stages [7]. Pakistan has no solar thermal power plant yet but it is need of the hour to use its potential for the solar energy as the use of the fossil fuels only is not enough for narrowing the gap between country's energy demand and production. Energy mix and integration has become essential now.

1.3 Concentrating Solar Thermal Technologies

Solar is by far the most abundant renewable energy source out of all available renewable energy producing resources. Solar radiations can be used to produce electricity and heat that than produces electricity by two techniques Photovoltaic (PV) and Concentrating Solar Power (CSP) respectively. These both methods produce no Carbon but PV is factor 3 costly than the CSP technology. In a concentrated solar thermal power station the heat which is produced by concentrating solar radiations onto the working fluid like water to produce the heat, that is produced by burning the fuel in the conventional power plants, which produces steam to run the turbine [8]. There are four most common CSP systems that are being used to produce clean energy. Some of them are line focusing, which concentrate light on the tubes which carries the working fluid, systems. The other which focus solar radiations on to the point like in solar dish or in the solar tower these are known as point focusing systems [9].

1.3.1 **Linear Fresnel Reflector (LFR)**

This is a line focusing system. In LFR there are flat concentrating mirrors called Fresnel reflectors which concentrate light onto the receiver tubes. These tubes have the working fluid in it to transfer the heat. Water or synthetic oil is used as heat transfer fluid. The operating temperatures are $350^{o}C$ to $550^{o}C$ [10].

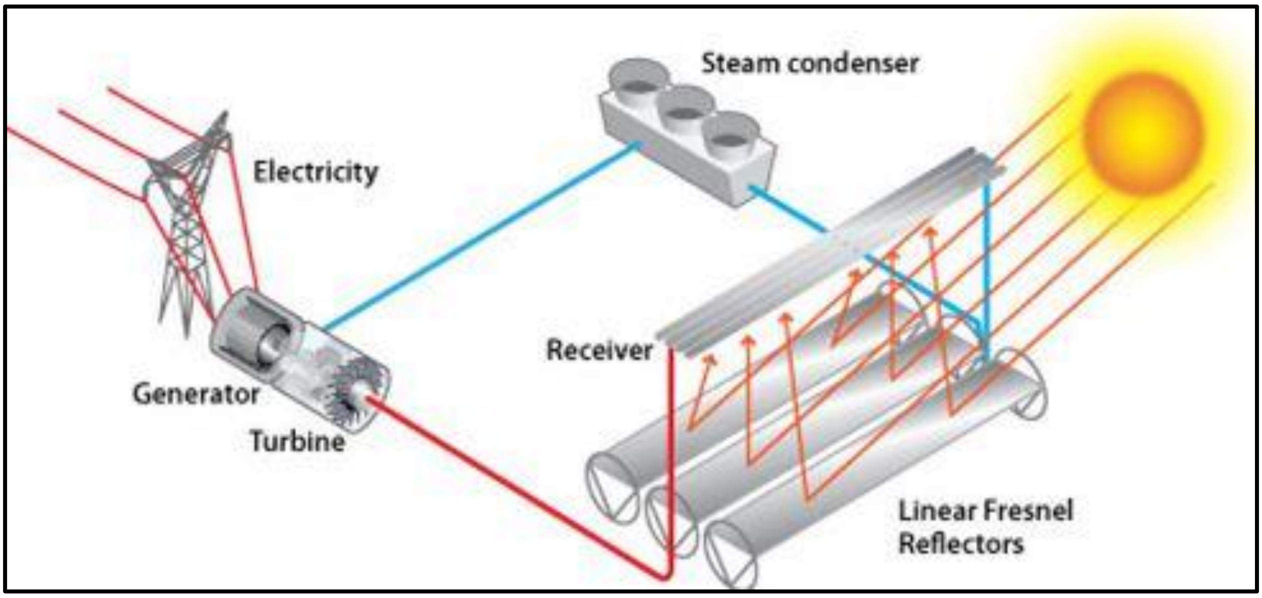

Figure 1: Linear Fresnel Concentrating Power Plant

1.3.2 Central Receiver or Solar Tower

In this solar power plant, there is a central receiver (also called solar tower) at which the solar concentrators (heliostats) concentrate the light on the top of the tower, which makes it a point focusing system. These heliostats track the sun continuously as the day passes by. Working fluid such as water, molten salts or air is heated well above 800^{o}C. That exchanges its heat in the heat exchanger where steam is produced which is used to run the steam turbine and thus generating the power [11].

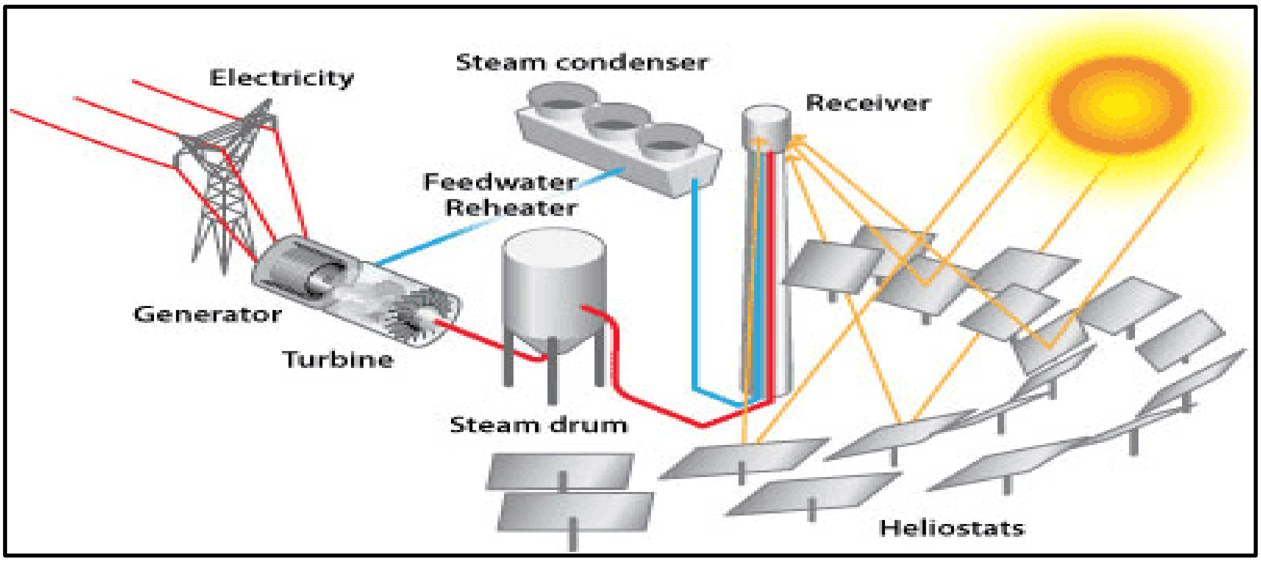

Figure 2: Solar Thermal Tower Power Plant

1.3.3 **Parabolic Dish**

This is also a point focus technology. The shape of the dish is almost parabolic through which the sun light is concentrated on to the absorber. This system has a motor generator that could be a Stirling engine or a smart gas turbine. Heat absorbed by the fluid like air in case of gas turbine is heated to very high temperatures which are then used to run the gas turbine and produce electricity [11]. As this system works on producing very high temperatures, when we compare it to the other CSP systems it holds the record 30% solar to electrical conversion efficiency but it is twice costly than the parabolic trough systems [12].

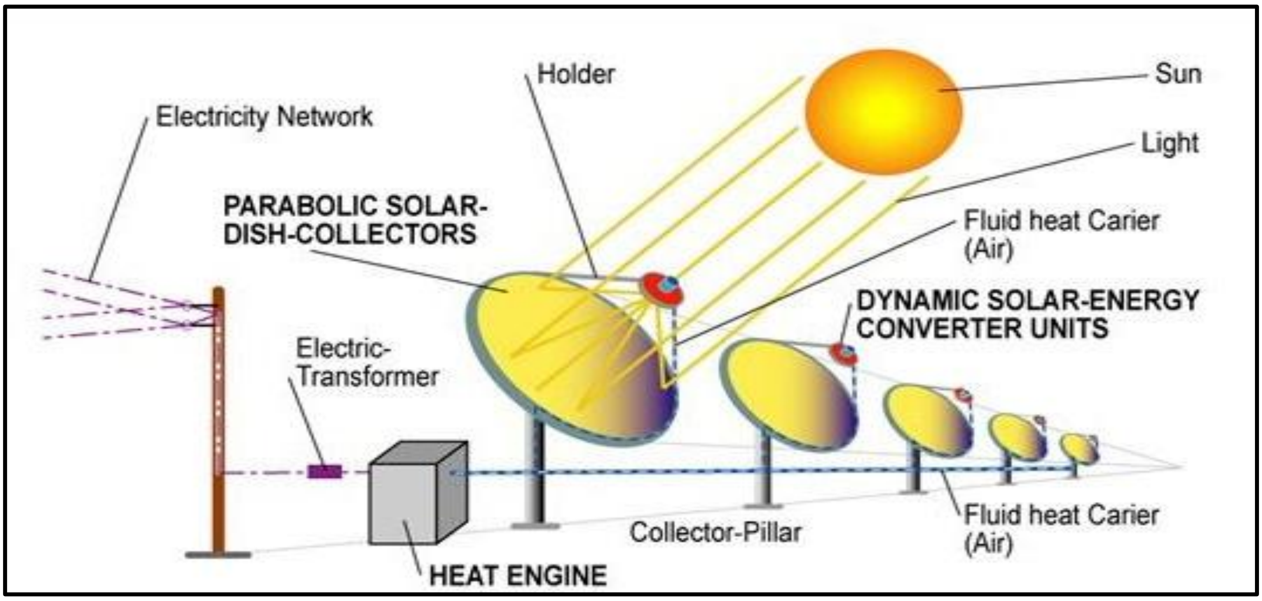

Figure 3: Parabolic Dishes

1.3.4 **Parabolic Troughs**

This is also a Line Focusing system like LFR. It is a single axis tracking technology. Reflectors track the position of the sun to get the maximum reflected radiations to heat up the heat transfer fluid (HTF) in the receiver tubes [10]. The reflectors are the parabolic shaped which concentrate the light on the receiver tube that is located at the focal point of the reflectors. In general, this power plant consists of 3 parts a solar reflectors and receiver tube, heat exchanger (for steam production) and power generating system (a generator and turbine) [9].

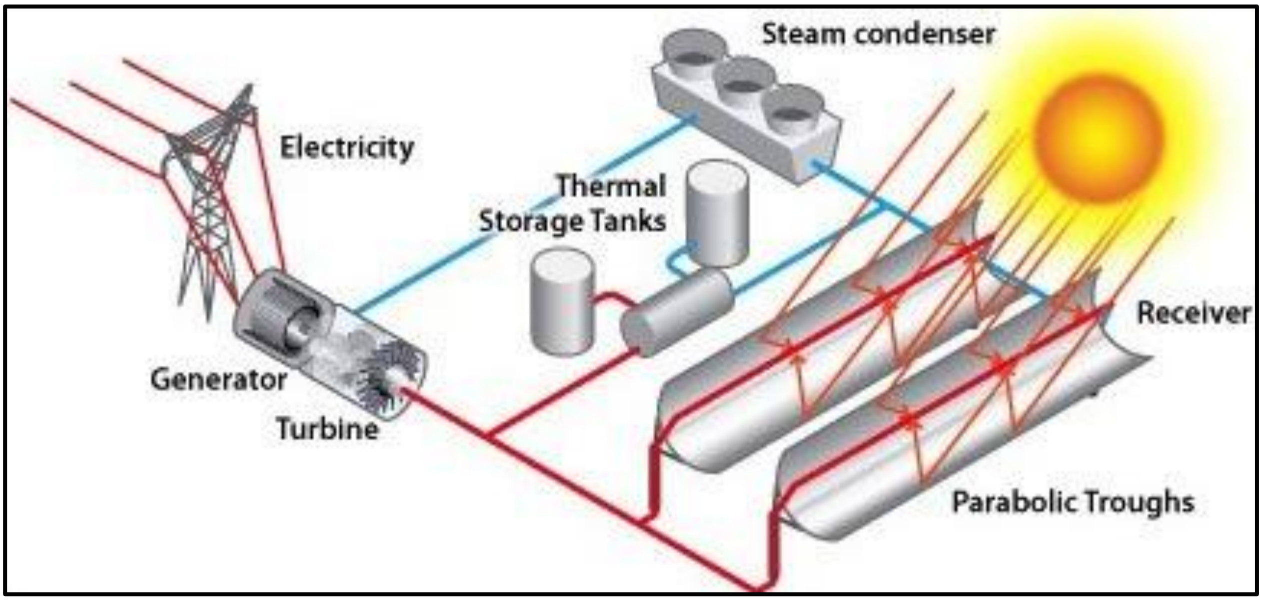

Figure 4: Parabolic Trough Power Plant

1.4 Types of Solar Reflectors

Solar reflectors come in different shapes (parabolic, flat, dishes) and sizes depending upon their application. The reflectors are also made of different material like glass, stainless steel, Aluminum and other different polymers. But these reflectors are divided in two types depending upon which side of the mirror is coated.

1.4.1 First Surface Mirrors

These are the mirrors in which the reflective and protective coatings are deposited on the front side of the mirrors. The light is reflected by the reflective coating before reaching to the mirror surface for example Ag or Al coated on the glass surface or on the stainless steel substrate [13].

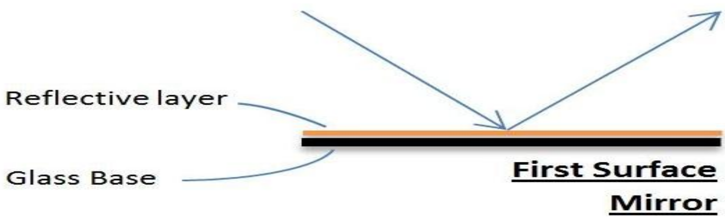

Figure 5: Front or 1st Surface Reflectors

1.4.2 Second Surface Mirrors

They use low iron glass or transparent glass on which the reflective and protective coatings are deposited from the back [13]. The sun radiations pass through the transparent glass and then these are reflected by the reflective coating.

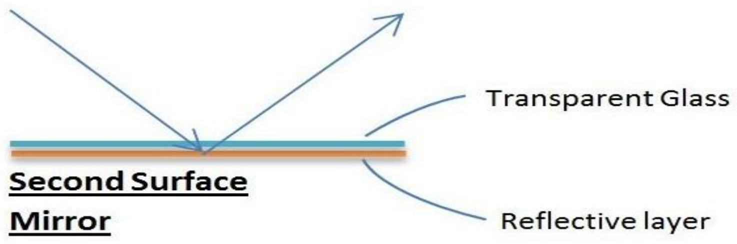

Figure 6: 2nd Surface Reflectors

1.5 Types of Coatings

There are varieties of the coatings that are being used for the development of the reflectors. These include reflective, protective and some base coatings as well.

1.5.1 Reflective Coatings

This is the reflective layer that is responsible for the reflection of the light from the reflectors to the targeted place. Mostly the metals show the reflective properties and among those Mo, Cu, Ag and Al are considered to be the best reflective coatings and therefore these are used in the reflectors extensively. Ag coatings are more durable and also have more reflection than the Al and other mentioned materials.

1.5.2 Protective Coatings

These coatings are the top layer coatings. They are used at the top of the reflective coatings to protect it from the environmental effects like rain, dust, storms etc. the oxidation of the metals can occur quickly which causes the degradation of the reflective films. So to protect the reflective film anti-soiling and self-cleaning materials such as SiO_2, TiO_2, WO_3, ZrO_2, ZnO, SnO_2 and CdS and corrosion resistant coatings are used [12].

1.6 Light Matter Interaction

Light falling on a surface can be absorbed, transmitted or reflected. As we know that the relation between absorbance, transmittance and reflectance is

$$A + T + R = 1$$

When light falls on a surface and it comes back this is called reflection. There are two types of reflection that can occur discussed below in detail.

1.6.1 **Specular Reflection**

It is a type of reflection in which the angle between the incident light and the reflected light remains same. That is

$$\theta i = \theta r$$

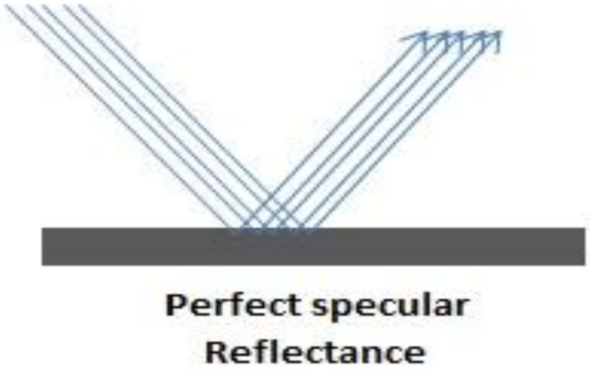

Figure 7: Specular reflection

1.6.2 **Diffuse Reflection**

Every surface cannot typically behave specular reflection due to the surface roughness light falling on the body reflects at different angles that type of reflection is called diffused reflection.

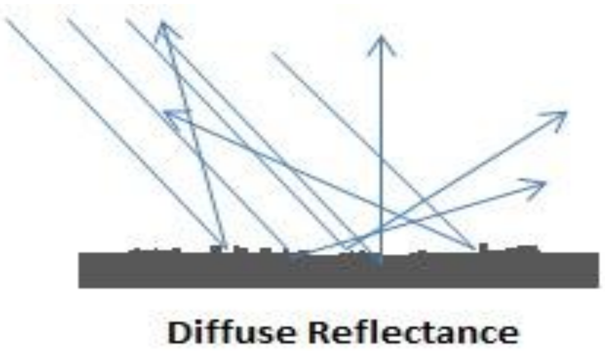

Figure 8: Diffused reflection

1.7 The New Concept

There are 1st and 2nd surface reflectors as discussed already in the earlier sections. The new idea is to prepare the 1st surface mirror with dual reflecting and protecting layers and they are called Compound and Integrated reflectors as shown in the figure below:

2nd Protective layer
2nd Reflective layer
1st Protective layer
1st Reflective layer
Adhesive layer
Glass

Figure 9: Double layers structure of the reflector

The idea is to deposit the pair of reflective and protective thin films on the top of the already present thin films of the same kind. What it will do is that it will increase the life of the reflectors if the top reflective thin films are affected by the abrasion or corrosion the thin films below them will take their place and in this way the reflectance is not affected and the efficiency of the power plant remains unchanged. It can be costly but it will surely increase the lifetime of the reflectors.

1.7.1 Integrated Reflector

The reflectors in which both the 1st and 2nd reflective layers are of same type i.e both are of silver or Aluminum but the protective layer remains the same are said to be Integrated reflectors.

1.7.2 Compound Reflector

The reflectors in which both the 1st and 2nd reflective layers are not of same type i.e 1st reflective layer is of Aluminum and second reflective layer is of Silver but the protective layer remains the same are said to be Compound reflectors.

1.8 Objectives

- Synthesis of multi-layer coatings for solar thermal reflectors with high reflectivity and self-cleaning properties.
- Determination of Structural properties of the thin films with X-Ray Diffraction.
- Surface morphology determination of the synthesized single and multi-layer thin films by Atomic Force Microscopy and Scanning Electron Microscope.
- Study of the Optical properties specifically measuring the Reflectance of solar light by reflectors by using UV-VIS-IR Spectrometer.

Outline of the Thesis

The preliminaries about the topic and the idea at which the work will be carried out have been discussed in the chapter 1. In the following chapters an extensive literature review has been presented. The working of the techniques used for the experimental work and for the characterization of the samples is described in chapter 3 and chapter 4 will discuss about the experimental details and procedures for the deposition of multilayer thin films. The chapter 5 will cover all the results and discussions about the outcomes of the characterize samples and conclusions and future recommendations have been added in the chapter 6.

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CHAPTER 2

Literature Review

Due to the availability of the high DNI (Direct normal irradiation) in the deserted areas the solar thermal power plants are built in these kinds of remote areas. The solar thermal power plants, having a major part of the solar reflectors, have to withstand in the all sorts of the environment and should maintain their reflective properties at their best. These reflectors can be affected by the wind storms, high irradiation flux, rain, sand and dust storms as they can be a cause of degrading the reflectors by corrosion, abrasion, soiling and oxidation [1]. The solar thermal power plants may be the solar dishes or solar tower that use heliostats to concentrate the light or the parabolic trough collectors represents about 30% collectors cost and if we include the structural and design costs of these collectors then it can go up to 50% for the solar dish technology and nearly 75% for the heliostats and the parabolic trough collectors [2]. Only the 5% decrease in the specular reflectance can cause nearly 5% increase in the levelized cost of the energy which shows the importance for the good reflectors in the solar thermal power plants.

So, in this regard a lot of work has been done on the protective and the reflective coatings of the reflectors. Different types of coatings on different types of the substrate materials have been prepared and one important factor for these reflectors is the technique used for the coatings of these reflectors. The testing of prepared reflectors is also considered to be important. So, all these different coatings on different materials prepared by using different techniques have shown dissimilar benefits, flaws, drawbacks and improvements with the passage of time.

2.1 History and Evaluation of Solar Thermal Reflectors

In the 1993-94 the National Renewable Energy Laboratory put emphasis on the requirements for improvement of the solar thermal reflector materials. Different types of the ideas were discussed for the improvement of the specular reflectivity of the reflectors and the degrading factors of the reflectors were discussed. The importance of accelerated testing of the reflectors was increased because the industry cannot wait for the a few years to predict the results for the reflectors performance [3].

Coating techniques used for the reflectors are Physical vapor deposition (PVD), thermal spray coating and Dip coating method. These techniques have different advantages and disadvantages. For example, Morales, A., and A. Duran in 1997 prepared the top protective coating of the SiO_2 by dip coating method. The films achieved were denser than when prepared with the PVD. The films prepared with PVD have low density of the film and therefore poor stability in outdoor conditions. Al front reflectors were coated with this technique which shows good stability at 400^oC in air. But these reflectors have a very low reflectance 85% which is a barrier for their use in solar thermal applications. Ag front reflectors were also tested after coating with SiO_2 by dip coating process on glass substrate. Their reflectivity was decreased by 1% after the Silica layer was coated. And they show stability up to 200^oC in air. These can be used as solar thermal reflectors application. Ag coated on stainless steel substrate also show good enough stability and mechanical strength for the solar reflectors applications [4].

In 2000 Martínez, Iván, et al presented the whole procedure of making the first surface solar mirror. They prepared the floated soda lime glass. Then they used magnetron sputtering technique to coat the aluminum which was failed due to the high temperatures so they used the thermal evaporation for the aluminum coating and also for the SiO_2 as top protective coating which showed potential application to be used as solar thermal reflectors [5].

In 2003 Fend, Thomas, et al worked on the study of different types of the reflector materials. The solar reflectors being used in the thermal power plants were tested in real outdoor conditions after the exposure of 4 to 6 years and in the accelerated weather conditions at the lab scale for their long term use the solar thermal technologies.

2.2 Development of Different Commercial and Laboratory Scale Reflectors

Below are the some details of different types of the reflectors that have been produced commercially.

2.2.1 **Solar Brite 95 Reflector**

Solar Brite 95 reflector has Ag as reflective layer also having a metallic protective layer on the back side. Just after its construction its measured reflectance was 92%.

But its reflectance decreased to below 90% after the testing at NREL and phoenix. After the accelerated exposure chamber test for 4 months its reflectance decreased below than 75%. These reflectors are no more available in the market.

2.2.2 Thin Glass Mirrors

Thin glass mirrors silver coated by the wet chemical process has longer durability but costlier considering the material and labor costs. The un-weathered reflectance is 93% to 95%. After a 3 years exposure to the outdoor conditions their reflectance decreased to 89% and some of the reflectors were degraded so badly after 5 years to exposure test and they were taken out of testing. Many of the reflectors were cracked as seen in different sites. These materials showed corrosion propagating through crack lines.

2.2.3 Alanod Miro2

Alanod miro2 are the front surface reflectors with aluminum as reflecting layer and with a top protective coating of alumina. However these reflectors corrode very rapidly by the outdoor pollutants. The reflectance was measured to be 90%. The reflectors protective coating could only withstand for 18 months of accelerated testing but after the changing to the fluoro-polymer as the protective coating the results were better and they are being used by two companies in parabolic trough power plants.

2.2.4 Flabeg Thick Glass Mirrors

Flabeg thick glass mirrors are silvered coated on the glass having more than 1mm thickness. Copper as the adhesive layer was used between glass and silver and painted from back for further protection. Their reflectance was as good as any other reflector and they showed excellent durability. They mentioned ‘’In almost all cases, corrosion is the most severe lifetime limiting factor for the solar thermal concentrators’’ [6].

Kennedy, C. E., and K. Terwilliger in 2005 tested Different kinds of reflectors for their development, durability and performance measurement. These include the following types of the reflectors:

2.2.5 Thin Glass Reflectors

Thin glass reflectors, which are 1mm thick and silver is the reflective layer produced by wet coating process. These are light weight but also fragile and difficult to handle. Corrosion has been observed after 2 years of outdoor exposure. The manufacturers switched to cu and Pb free process by using the copper-less and lead free paints in their commercial mirror lines.

2.2.6 **Thick Glass Mirrors**

The other type of reflector they tested is called Thick glass mirrors. They are normally of thickness greater than 1mm and they have cu at their back as protective coating for the silver layer and cu also provides the adhesive surface to the back protective paints which keeps the cu-silver mirrors protected from abrasion and corrosion. They show excellent durability but are heavy and fragile and also curved shapes of these thick glass mirrors are difficult to produce for that slumped glass is required thus making them costly but they are being commercially deployed.

2.2.7 **Aluminized Reflectors**

Aluminized reflectors are the front surface reflectors with Al as reflective coating and the alumina as the top protective coating on the polished aluminum substrate. Due to low cost and flexibility of these reflectors they are of great interest but they show poor durability in urban and industrial locations. Some companies do not sell these reflectors because of the delamination of the protective coating.

2.2.8 **Silvered Polymer Reflectors**

Silvered polymer reflectors are an alternative mirrors. These are silvered polymer mirrors that are light weight and more flexible. These acrylics showed some admirable optical properties and the silver were well protected from the environment. But it had delamination problems in the field when there were severe weather conditions due to weak adhesion between polymer and silver layer. ‘’ More aggressive goals of reflectivity above 90% and 15-30 years of lifetime have been pursued’’ [2].

2.3 Work Done on Protective Layers

As the importance of the solar reflectors coatings was realized then researchers put a great effort to study the reflective and top protective coatings and their behavior under different circumstances.

Therefore Dreesen, Laurent et al studied the behavior of the TiO_2 particles in year 2009, as Titania is used as top protective coating due to its anti-soiling properties. Thus they used DC magnetron sputtering to produce the Titania film onto the glass substrate. The effect of pressure was studied, which shows that after increasing the pressure from 20 to 40×10^{-3} mbar the nano particle size of the film increases from 7 to 11 nm. Titania showed better hydrophilicity when it was illuminated by the UV light and the hydrophilicity was lost when the samples were placed in the dark for more than 20 hours. It was observed that increase in annealing temperature increased the reflectivity but temperature increase did not always increase the reflectivity of the reflector as reflectivity starts decreasing when film was annealed above 400^oC [7].

In the same year Almanza, Rafael, et al came up with the concept of compound mirror, it is front reflector, and the only difference is that they had two layers of Aluminum and SiO_2 as top protective layer. The idea behind this concept is that if the corrosion, abrasion or holes are created in the top layers of the films then the lower layers replaces it so the ability to reflect the light by the reflector does not decrease and it will work for longer time [8].

So the research on the aluminum reflectors was carried on by the Ling, Xioming et al prepared the Al reflectors by thermal evaporation in 2010. Different types of the substrate materials were coated with Aluminum and then their roughness, morphology and the reflection was also tested. A compact and uniform morphology of the Al films was observed on the polished aluminum alloy it showed maximum reflectivity of 90% but when Al was coated on galvanized iron, reflectivity was observed to be 84% which was low and not acceptable to be used as reflector. The ABS sheets, when coated with Al, had an average reflectivity of around 90% but it was lesser on the edge area this indicates that the edge effect would harm optical uniformity of the mirrors [9].

In 2012, Xu, Y. J., et al studied the effect of the temperature and the film thickness of the Ag and SiO_2 when coated on the glass substrate. They tested a multiple combinations of thickness for the Ag and SiO_2 and came up with finding an optimized value that could resist the corrosion and abrasion well and that could reflect up to 96% of solar light. The optimum values of the Ag and SiO_2 were found to be 120nm and 320nm respectively. And the annealing temperature of 300 ^oC was considered to be best for the SiO_2 film structure. The film thicknesses lower than 30nm of the silver showed reflectivity below 85% and maximum at 130nm [10].

In the year 2013 Xu, Y. J., et al prepared the $Cu/Ag/SiO_2/TiO_2$ coated on glass substrate reflector. The main aim of the article was to find the optimum values of the combination of the above mentioned films and the effect of the annealing temperature on the TiO_2 film. The use of the TiO_2 was to behave as the anti-soiling agent as it has the hydrophilic properties when it is in anatase structure. TiO_2 when annealed at

500^oC has the highest reflectivity. The optimum structure was glass/Cu(30nm)/Ag(130nm)/ SiO_2(400nm)/TiO_2(300nm)/Air [11].

So use of TiO_2 as anti-soiling or dust repellent agent was more done by the Lorenz et al in year 2014. All the components of concentrating solar power plants are open to outdoor conditions, especially the glazing; have to resist dirt deposition and many degradation factors such as high UV irradiation, high temperatures and mechanical loads. Those causes that increase the effects of the dirt deposition, called soiling. Due to the dirt deposition in regions where we have more dust in the environment, soiling occurs on the reflectors and 60% decrease in the efficiency for the six month period has been noted for the PV modules due to dust accumulation.

SiO_2 and TiO_2 were used as the anti-soiling oxide metals and it was found that at contact angle less than 10^o the dirt particles can go off the surface under the certain inclination of the surface. The films were tested under the all types of weathering conditions like dew, humidity, dirt tests. The anti-soiling coatings proved to be very effective as only 1.6% transmission loss on the coated glass was measured after the dew and dirt accumulation test which had 6.8% loss when uncoated glass was tested [12].

In year 2015, Girard, R., et al studied the effects of the various degrading factors of the solar mirrors like rain, humidity, solar irradiation and temperature on three kinds of Solar mirrors with the silver and paints as the reflective and protective layers respectively.

The results show that the edge corrosion of the silver under the presence of water and high temperature was occurred and delamination of the paint coat was also observed under these circumstances. The high temperature alone does not affect the silver and the pigments layers but UV exposure is observed to be most dangerous constraint to the paint binder. Pitting causes the homogenous corrosion that was observed in these as well [13].

So Atkinson, Carol, et al also pointed out some major factors to be dealt with by the protective coatings of the reflectors. These problems are given below:

- **Protection from corrosion** normally the reflectors are made of the silver coatings on the back of the highly transparent glass. Cu is normally used on the back of the silver layer as it is oxidized and corroded by the environment very rapidly. Same thing happens with the Al for that alumina is coated on the top of the aluminum layer. Silica is being used for these purposes now a day in most the reflectors. These coatings help preventing from corrosion but corrosion occurs quickly where moisture and air can penetrate the protective layers.

- **Protection from dirt** due to the dirt and soiling of the reflectors 8 to 10% performance of the plant is reduced as the reflectivity losses increase. To avoid this type of loss anti soiling coatings of the certain transition metal oxides are used. Titania for example has these characteristics of performing photo catalytic activity which produces the electron hole pair which in presence of the air can catalyze the conversion of organic matter into carbon dioxide and water [14].

Recently in year 2016, Belasri, Djawed, et al published a research on the SiC mirrors. They tested the new SiC coated mirrors for their reflectance and durability and then they compared the results with the high quality concentrating mirrors with the help of different applied cleaning methods. The silicon carbide (SiC) reflectors basically use the fiberglass reinforced plastics (FRP) as the back support, silver for high reflectance and then the thin layer of the SiC as a protective layer on the top surface. SiC mirrors have three advantages one is they reduce the facet weight by 40% compared to the current glass facets. Second advantage is that SiC protects the mirror from scratches which can be caused by the sand abrasion in real environment as SiC is one of the hardest known materials and last is that it has the electrical resistivity lower than the glass so no static current produces on the surface which generates anti-soling effect that helps in removing dust easily. There were two different cleaning methods used:

(a) Three brush passages with a polyethylene brush were carried out for brushing with water and

(b) seven brush passages with a nylon brush were performed for brushing without water.

The portable reflectometer, 15R-RGB, was used for measurement of specular reflectance.

SiC mirrors have nearly the same reflectance as the high quality CSP glass mirrors currently being used in the CSP plants. The plus points of the SiC mirrors are that they have light weight which reduces the cost and saves the installation time. Further the cleaning without water shows that SiC is a good candidate for the places with high water scarcity issues as the cleaning without water will save maintenance cost [15].

So until now it is observed that the reflecting and protective coatings are mostly the same and are being used widely on different types of substrates but the problem with them is to find a technique which would be cheaper and easy to process. Also the quality of the films should not be compromised as the aim of these thin films is to make the life of the reflectors long approximately up to 20 to 25 years.

In this regard Chen, Dexin, et al has presented their findings. Wet-coating method was used to coat the Ag on the ABS sheets. The fabrication process mainly involves two steps:

(a) polymer improvement

(b) non-galvanic plating of silver

This method is said to be a preferable way for the thin films coating over the electron beam evaporation and magnetron sputtering as they both require expensive materials which limits their practical application.

To examine the elemental, morphological, electrical and optical properties of the Ag film XRD, XPS, AFM, field emission scanning electron microscopy, four point probe measurement and UV/VIS/NIR spectrometer were used. A thorough study for resistance and reflectance approves Ag thin films prepared by this simple method are suitable for solar energy applications. Comparing to the physical vapor deposition techniques like sputtering, the wet coating method is simple, inexpensive and easy [16].

Ennaceri, Houda, et al worked on the protective coatings of reflectors recently. In this paper transparent multifunctional anatase TiO_2 and wurtzite ZnO top-protective coatings were investigated for the purpose of the self-cleaning properties applications in the solar concentrators' application. These protective films were coated on thick glass silvered mirrors. The front surface mirrors were prepared by magnetron sputtering. The used substrate was tempered float glass with the dimensions of 25x25x2 mm. Both the silver and copper layers were deposited by magnetron sputtering as the reflective and metal back layer respectively where the top-protective layers of TiO_2 and ZnO were deposited by the chemical method called spray pyrolysis which aims to provide protection to the specular reflection of the mirrors against the environmental conditions.

Prepared coatings exhibit the properties like transparency, photo-catalytic, self-cleaning and switchable wettability. Hence every quality of these films make them convenient for the solar thermal reflectors applications [1].

Also Mishra, S. K., et al have coated multi-layers thin films of reflective and protective properties on the Aluminum sheet. In the article they prepared thin films of $Ni/Ag/SiO_2$, $Al/SiO_2/TiO_2$, $Al/Ni/Ag/SiO_2/TiO_2$ by using magnetron sputtering and the sol-gel coating process on the Aluminum sheet. Ag and Ni were deposited through sputtering where the titanium oxide and silica were deposited by sol-gel coating process.

SiO_2 and SiO_2-TiO_2 coating endured 1800 hours of aging test and more than 400 hours in speeded salt spray test. Reflectance was not decreased more than 1-2%. The Ag coating on the aluminum sheet increased the reflectivity to 95% when coated after Ni and between crystal clear and protecting layers of SiO_2 and SiO_2/TiO_2. Reflectivity decrease was observed to decrease up to 80% after 500 hours of the exposure.

These coatings showed 80% reflectivity after the 96 hours of the testing under the accelerated salt spray. These thin films exhibited high reflection and durability after the accelerated aging test conditions and salt spray tests hence they are capable coatings for solar thermal reflectors applications [17].

Alex, Sherine, et al Using the electro-deposition on the mild steel substrate they developed a reflecting Cu-Sn intermetallic alloy of predominantly single phase $Cu_{41}Sn_{11}$ based mirror material with 80% specular reflectance. The reflector consists of intermetallic δ-phase that was obtained by the electro-deposition and this was confirmed by the XRD and EDS analysis. AFM studies showed the relatively smooth film with roughness in nano range The effect of humidity (90% RH) and dust were also reported to determine the optical requirements of this new material as a reflector for solar concentrators [18].

2.4 Developing the New Concept

So the problem with the reflectors is mainly their lifetime as they are affected by abrasion and corrosion easily. In this thesis the work will focus preparing reflective layers and the study of their morphological behaviour. And also on the preparation and study of the protective layer SiO_2,to increase the life of the reflective layer. A new concept of integrated and compound reflectors will be developed that is to deposit the additional reflective and protective thin films on the top of the already present thin films of the same kind. It will increase the life of the reflectors. Let's assume that if the top reflective thin films are affected by the abrasion or corrosion the thin films below them will take their place and in this way the reflectance of the reflectors will not be compromised because the layer beneath the top reflective layer will start reflecting the sunlight and hence the efficiency of the power plant remains unchanged.

Summary

The types of reflectors thin films being used in the reflectors applications for the STPP has been studied with covering the all sorts of techniques that are used for the deposition of the thin films. Their pros and cons are discussed. Similarly benefits and harms of using different types of reflectors and different sorts of reflective thin films have also been discussed in detail.

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CHAPTER 3

Growth and Characterization Techniques for Solar Reflectors

In this chapter, techniques used for the deposition and characterization of the solar reflectors are discussed.

3.1 Methods of Deposition

As discussed in previous chapter, there are different types of methods by which thin films can be prepared. For example spin coating, electro chemical, chemical vapor deposition. But the films coated by these mentioned process includes preparation of the solutions for example for spin coating, the solution is prepared by the sol-gel process first and then that solution is used for the spin coating. Also the film prepared by these methods is less homogenous and rougher. So considering all these facts Physical vapor deposition method was chosen for coating which provides more controlled environment for deposition. Thermal evaporation was used for the deposition of the Cr, Al and Ag whereas the deposition of the SiO_2 was done by RF-sputtering method.

Working of both the thermal evaporation and RF-sputtering is discussed below in detail.

3.1.1 Thermal Evaporation

Basically it works on evaporation and condensation principle. It is a process in which the charge is placed in a basket or wire crucible and when current passes through that crucible the charge melts by getting the resistive heat energy from the current and then atoms with higher energy evaporates under the high vacuum conditions ultimately depositing themselves on the target substrate where they condense and stick to the substrate of relatively low temperature.

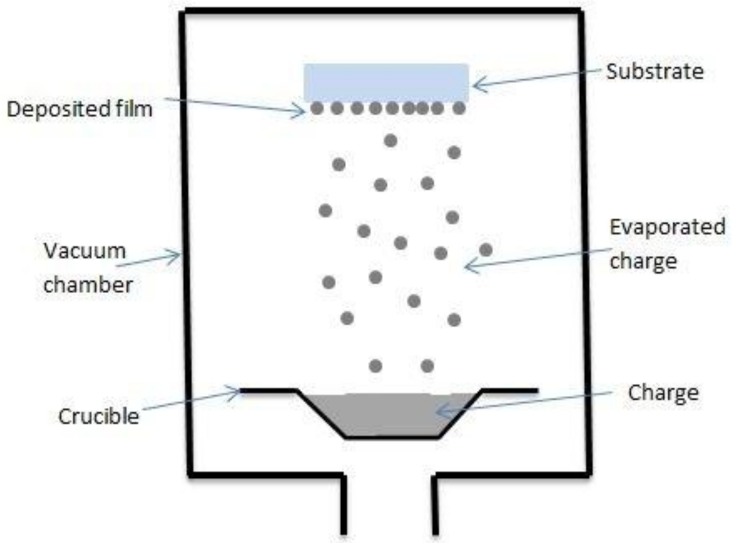

Figure 10: Thermal Evaporation Chamber

There are three main components of the thermal evaporation system.

3.1.1.1 Charge/Material

The material to be deposited on the substrate is called charge. It can be any metallic or non-metallic or dielectric material of certain types like Cr, Ni, Ag, Al, thin films are deposited widely by using thermal evaporation technique.

3.1.1.2 Crucible

The basket or the carrier in which charge is placed and through which current passes to melt the charge is called crucible. There are different types of crucibles made of different shapes and materials for different types of charge. For example wire crucible is preferred for Cr as Cr is a sublime material and doesn't melt but directly evaporates from solid to gas phase whereas basket crucible which is made of tin is preferred for Al and Ag. They melt from solid to liquid phase when current passes through the basket and afterwards it evaporates due to high vacuum inside the chamber.

3.1.1.3 Substrate

The wafer at which the film is deposited is known as substrate. It can be anything like glass, steel, Aluminum sheets, ABS, Si etc.

3.1.1.4 Deposition Control

Deposition rates are a strong function of the melting temperature. When the charge melts it evaporates, under the vacuum conditions, in all the directions but the deposition rate is determined by the quartz crystals placed inside the chamber parallel to the substrates. The charge depositing on to the crystals is measured in terms of the how much vibrational amplitude reduces due to the increase in the mass of the quartz crystal.

3.1.2 Sputtering

It is also a physical vapor deposition method. in this process the solid target is physically bombarded by the heavier ions which eject the atoms from the solid surface that under the vacuum get deposit on the substrate surface.

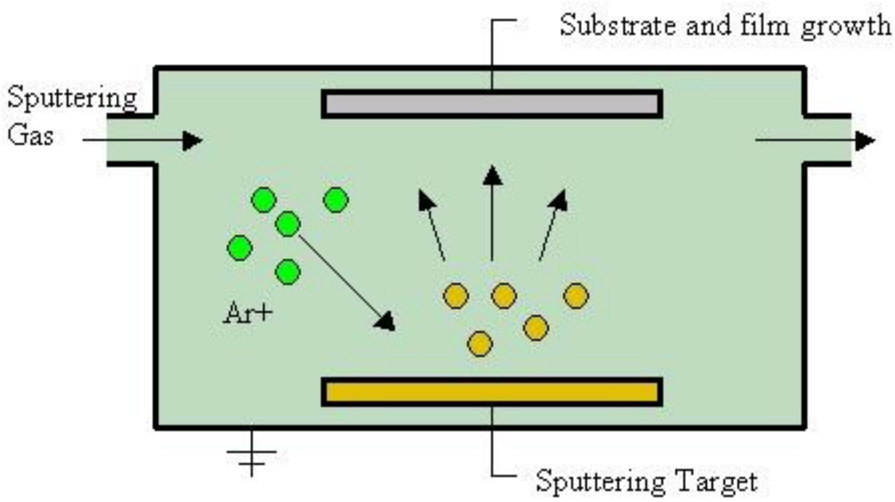

Figure 11: Sputtering Deposition Process [1]

It is basically of two types

3.1.2.1 DC-Sputtering

In this method the source of power is direct current rest of the procedure remains the same. And this is used for the deposition of the conductive materials. It is cheap and also has high deposition rates due to which it is cost effective.

3.1.2.2 RF-Sputtering

The difference between the DC and RF sputtering is that the power source in RF sputtering is RF-source instead of direct current source.

There are two steps in the RF-sputtering in the 1st step the target material gets negatively charged and attracts the positively charged ions which attach to the surface of the target material due to polarization. In 2nd step during the positive cycle of the power source the target is positively charged which releases the charge collected on the surface of the target material and the atoms of the target that then under the vacuum gets deposit on the substrate.

This method is mostly used for dielectrics and has low deposition rates as the sputtering yield of the dielectric materials is low that makes it costly as well.

3.2 Characterization Techniques

For the characterization of the thin films X-Ray Diffraction, Atomic Force Microscope, UV-VIS-IR Spectroscopy and Scanning Electron Microscope are used.

The working of these techniques is given below in detail.

3.2.1 Scanning Electron Microscope

The scanning electron microscope (SEM) is used for surface study of the specimen. It can magnify the sample surface up to 200 thousand times. SEM is equipped with an electron gun, a condense lens and an objective lens to produce electronic probe.

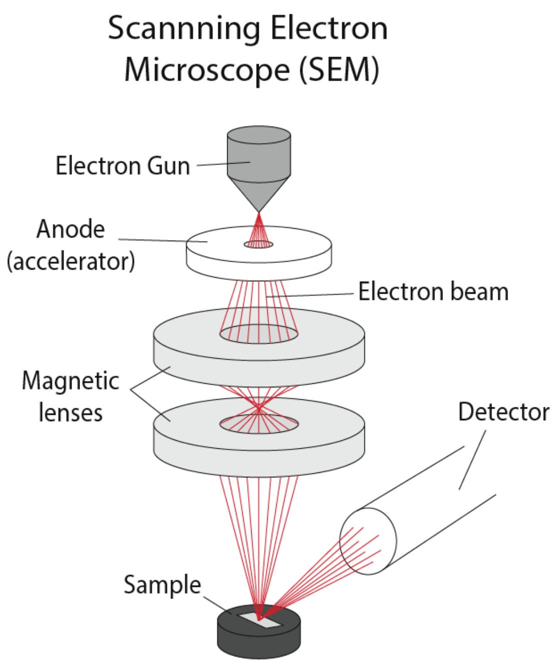

Figure 12: Illustration of Major Components of a Typical Scanning Electron Microscope for Image Formation [2]

SEM is used for topographic analysis while topography property is how a material looks like. Similarly, SEM is also used for Morphological (the shape and size of the particle) analysis of the material. Composition of material which means elements present in the specimen under study can be determine through EDX. Crystallographic information which means how atoms are arranged in material are derived from SEM results. It is preferred over ordinary microscope due to its higher magnification, higher depth of field and high resolution. Through SEM, clear images can be obtained at nano-meter resolution. Resolution is an ability to study two closely spaced points. Sample needs to be prepared for SEM testing. Sample should be conductive otherwise it starts sparking and there will be no visible image. Nonconductive samples are coated with either carbon or gold to make them conductive. There are back scattered electrons, beam electron and secondary electron which determine the properties of the specimen. SEM electron "kicks off" the electron from shells of the specimen under testing.

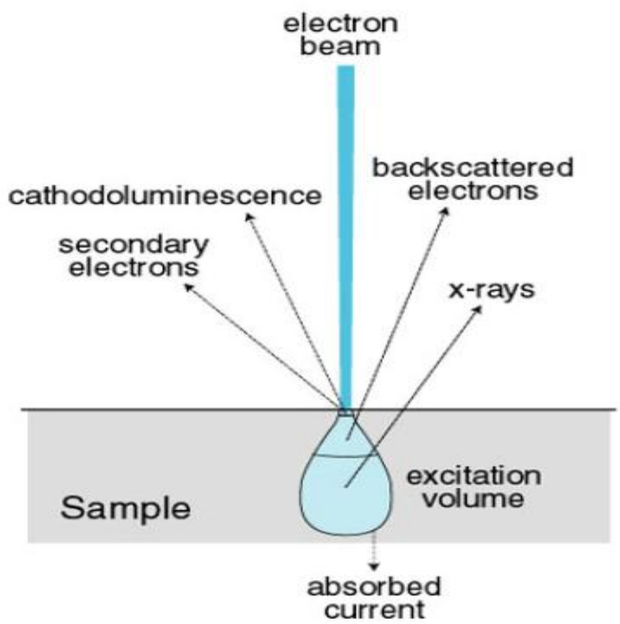

Figure 13: Emissions from the Sample [3]

This figure 13 shows that electron beam collides with sample and then few electrons back scatter and few are secondary electron. High vacuum is created in chamber to avoid collision with electron [4][5].

3.2.2 **Energy Dispersive Spectroscopy**

Energy dispersive spectroscopy is also attached to SEM and through EDS compositional analysis of the sample at a particular point are done. EDS actually uses X-rays emitting from sample while sample is being bombarded with the electron beam at a specific point. It provides localized elemental composition of the sample. It can identify all elements from atomic number 4 to 92. EDS is not a surface technique [6][7].

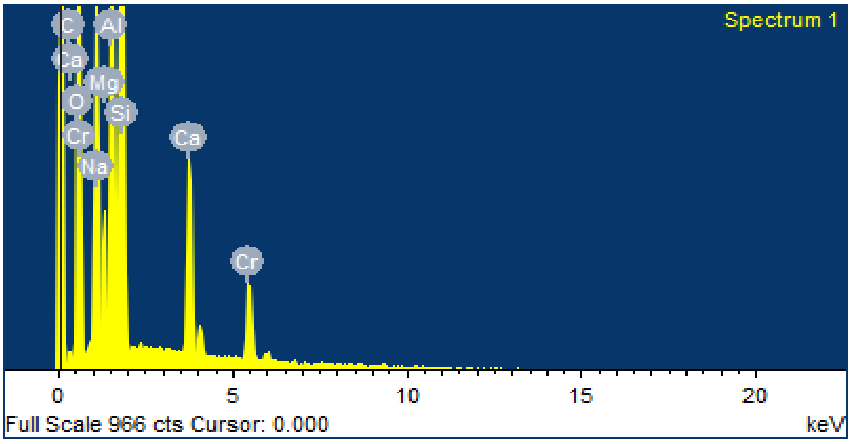

Figure 14: EDS Analysis (graph) of Glass/Cr/Al Thin Films

EDS results are in form of graph and in form of table as well. Above figure 14 shows composition of material and the table below shows detail of the compositional analysis that how much an element is present in the sample by wieght percentage.

Table 1: EDX Analysis Showing Percentage Presence of Each Element

Element	**Weight %**	**Atomic %**
O	41.81	56.04
Na	6.74	6.29
Mg	2.10	1.85
Al	11.70	9.30
Si	29.72	22.70
Ca	4.49	2.40
Cr	3.44	1.42
Totals	100.00	

3.2.3 **X-Ray Diffraction**

XRD works on the principle of Bragg's law. Bragg's law was derived by the English physicists Sir W.H. Bragg and his son Sir W.L. Bragg in 1913. XRD is used to measure the average spacing between the layers of the atoms of the specimen. XRD is used to determine crystal structure of materials and determine the orientation of crystal atoms.

3.2.3.1 *Bragg's Law of Diffraction*

When a beam of X-rays collides with the lattice plane of structure of the material under testing, every atom of the material emits a secondary wave. These emitted secondary waves then interface with each other and finally produce diffracted beam.

$$n\lambda = 2dSin\theta$$

Where d is the spacing between the atoms.

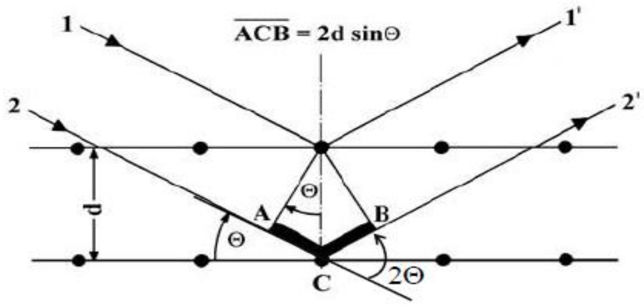

Figure 15: Bragg's Law as Working Principle of XRD [8]

XRD equipment work on principle of Bragg's law derived from law of conservation of energy and law of conservation of momentum [9] [10].

The distance between the two planes is "d". When secondary waves interface they produce constructive interference that results in a peak on the graph. Sample should be in powdered form for XRD or it should be coated on a substrate as thin film. XRD is used for phase identification, radial distribution function, crystal distribution function, percentage of material as crystalline or amorphous and determine the crystal structure of the sample [11][12].

There are two arms like structure in XRD machine. X-rays source is on one side while detector is on another side of the machine. We can scan the sample from 0^{o}-90^{o} but to be on safe side XRD is operated from 10^{o} to 80^{o}. The result of XRD is in form of peaks against the angle. More intense the peaks the more crystalline material is.

3.2.4 **Atomic Force Microscope**

Atomic force microscope (AFM) is basically a scanning probe microscope technique in which a tip (micro meter) scans surface of the specimen and image is formed by the force acting on the tip.

A typical AFM has a sharp tip cantilever (micrometer size), sample stage, laser source and a detector as shown in the figure below.

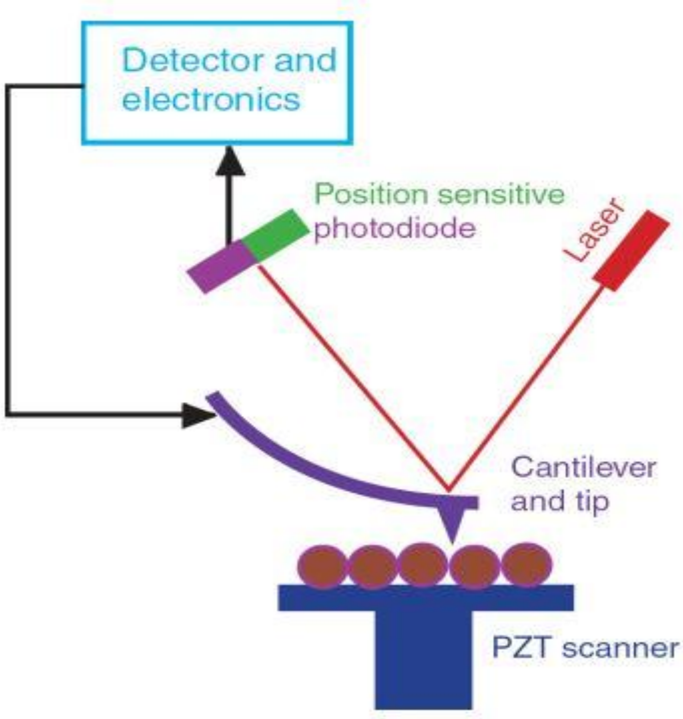

Figure 16: Parts of AFM [13]

3.2.4.1 Working of AFM

When the tip is brought near to the sample surface, the deflections are formed in the cantilever. These deflections are detected by the laser light falling on the cantilever which reflects at different angles to the detector. The topographical micrograph obtained by the AFM is shown below.

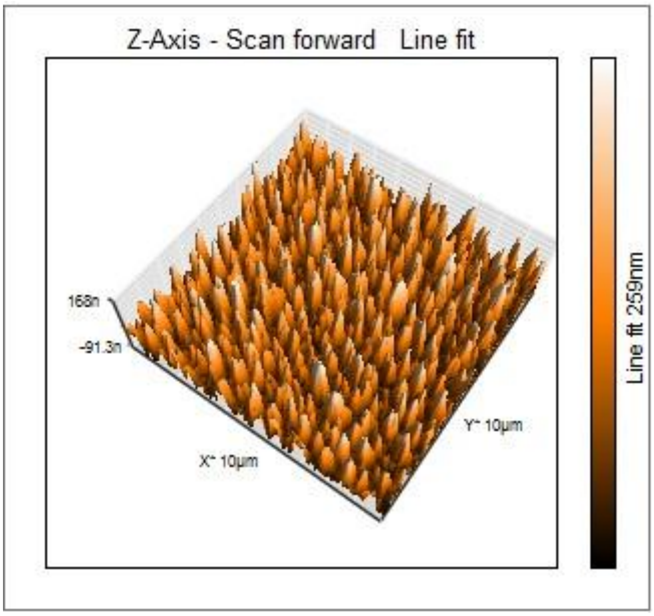

Figure 17: AFM Micrograph of Al Coated Glass at 10 µm

AFM can work in no. of modes like static mode or dynamic mode. Dynamic mode can further be divided into contact mode and non-contact mode. We can use the AFM in any of these modes depending on the fact for what we are using it for.

It is safe to use it in the dynamic and non-contact mode because the tip being in nm range size is very sensitive. When used in the static mode the risk of the tip to touch the surface of the sample increases which can lead to the breakage of the cantilever tip [14] .

When measuring the roughness by the AFM, the results are shown in form of the parameters as shown in the figure below:

Name	Value
Area	100.8pm^2
Sa	41.902nm
Sq	50.645nm
Sy	363.28nm
Sp	257.14nm
Sv	-106.15nm
Sm	-18.338fm

Figure 18: Roughness Analysis Result of the Aluminum Coated Glass At 10 µm

In the above diagram the term "Area" represents the selected area at which roughness analysis are performed.

Sa is the average roughness of the area. **Sq** is the root mean square roughness of the area. These are both normally considered as the surface roughness parameters. The **Sv** and **Sp** are evaluated from the lowest and highest points on the surface respectively.

3.2.5 UV-VIS-IR Spectroscope

Spectrophometers are used to measure the absorption and reflectance of the gasses, liquids and solids.

The diffused reflectance is measured with the help of an integrating sphere that detects all the light reflected from a surface from all the directions.

Below is the process diagram of a UV-VIS-IR Spectroscope:

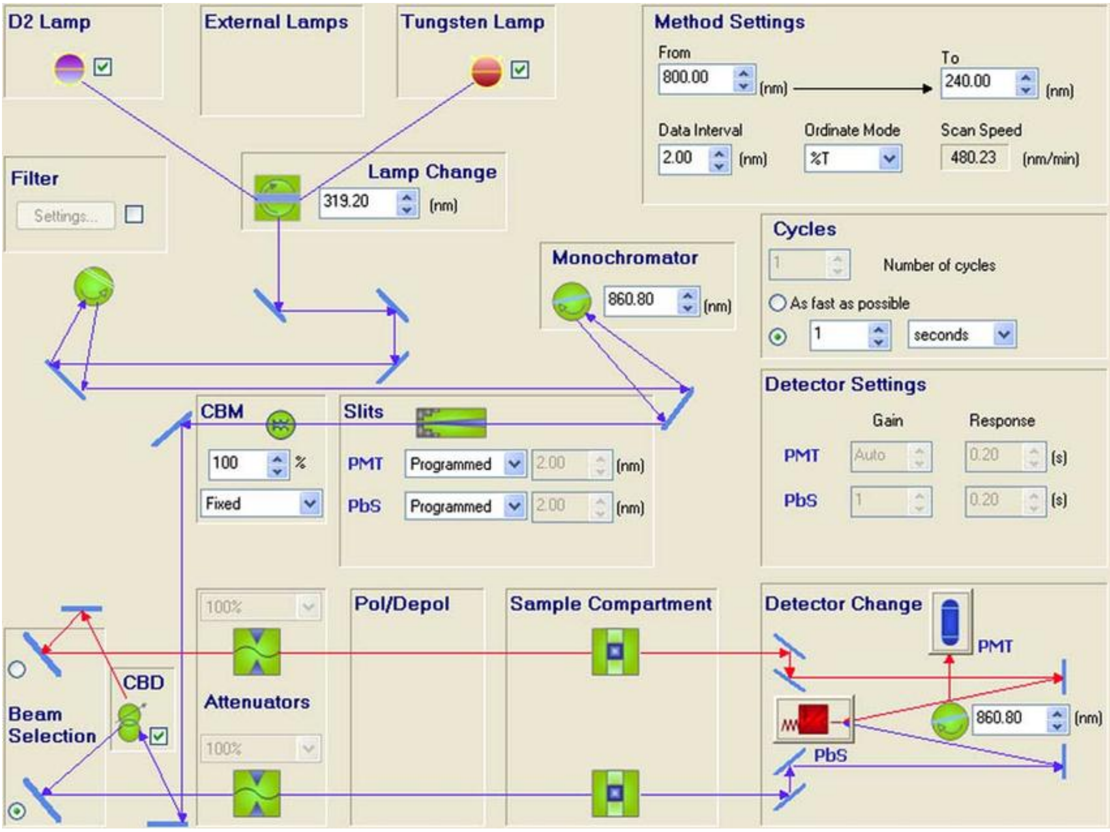

Figure 19: Flow Diagram of the Working of a Spectrophotometer [15]

The main components of UV-VIS-IR spectroscope are shown in the figure above and discussed below:

3.2.5.1 Choice of lamps

There are two types of lamps used in the apparatus to produce the required light.

D2 Lamps: Deuterium lamps are used to produce the Ultraviolet radiation ranging from 160 nm to 390 nm.

Tungsten Lamp: These lamps are source of the visible and infrared radiations.

3.2.5.2 Monochromater

It is a device which is used to select the radiation of a single wavelength of our choice.

3.2.5.3 Detectors

Two kinds of detectors are used in the system one are **PMTs** that is Photomultiplier Tube which are used for the ultraviolet and visible regions of light and the others are cooled leadsulfide **PbS** detectors which are normally used to detect the light of the infrared region.

3.2.5.4 CBD and CBM

CBD stands for Common Beam Depolarizer. As the name depicts that CBD depolarizes the radiations coming from the monochromater.

Whereas the CBM stands for Common Beam Mask. It is basically used to stop and give passage to the beam of light from 0 to 100 percent.

3.2.6 Scratch Tester

One of the methods to get the maximum force required to damage the thin film coatings and bring failures in them is by scratch testing. Different types of coating methods are used on different types of substrates like glass, polymers and Teflon.

The thin films that are used in the solar reflectors applications should have high wear resistance. Therefore the use of the scratch tester is a good way to test these parameters of the thin films.

3.2.6.1 Working Principle of Scratch Test

A diamond tipped stylus usually of 2 µm is used at which a constant or a progressive load is applied to measure the minimum load required to create the damage in the thin film. The point where crack occurs in the coating or when the delamination starts is known as the point of failure.

The outcome of the test is in the form of force that has been recorded to disturb the coating which is useful to determine adhesion of them with the substrates.

Summary

The working of characterization techniques for the reflectors like XRD, AFM, UV-VIS-IR spectroscope, SEM and scratch tester has been discussed in the chapter. Their importance has been discussed according to the given application and examples have been given of their results that how they are explained and what they determine.

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CHAPTER 4

Experimental Work

In this chapter the preparation of the thin films deposition has been discussed in detail.

The deposition of Chromium (Cr), Aluminum (Al) and Silver (Ag) was done by the thermal evaporation technique and the Silica (SiO_2) was deposited by RF-Sputtering method by using NVTS400-22-07, NANOVAK, Turkey.

4.1 Selection of Material

The materials used for the preparation of the reflective and protective thin films include some metals and metal oxides. Both silver and aluminum were selected to be used as reflective thin films. They have excellent reflection properties showing above 90% light reflectance.

The metal oxides like ZnO, TiO_2 and Al_2O_3 are used as protective and self-cleaning thin films. We have selected the silica (SiO_2) due to its high transparency for light and great abrasion resistance and it can withstand against all the outdoor weathering conditions that can cause damage to the reflective layers if exposed to the environment directly.

The substrate used was float glass that is very cheap and easily available. Glass has this priority over other base materials that it is hard and has more mechanical strength when compared with the aluminum sheets.

4.2 Cleaning Methods for the Substrate

Float glass was used as the substrate of dimensions (25mm×25mm×3mm). Before the deposition of the thin films the glass slides were cleaned by two methods.

4.2.1 Sonication and Solution Method

The glass slides were ultra-sonicated for 15 minutes in acetone and after washing with deionized water they were soaked in piranha solution, which was prepared by adding H_2SO_4 and H_2O_2 in molar ratios of 3:1, for 10 minutes to remove any dust particles from the surface. At the end, the substrates were again washed with deionized water and then dried by air dryer. Then these slides were directly taken into the evaporation chamber and placed carefully for the experiment to be performed.

4.2.2 **Sputter Cleaning Method**

Basically it is cleaning of the solid substrates under vacuum by sputtering. RF – sputter cleaning method was used to clean the substrate.

The procedure is described for the RF-sputter cleaning below in detail.

First of all the chamber is pumped to achieve the vacuum level and after the desired vacuum level i.e. 10^{-7} Torr is achieved then following steps are taken to perform the sputter cleaning:

- Ar and N_2 cylinders in accordance with their limited pressure values (For Ar max. value is 2 bar and for N_2 it is between 3-6 bar) were opened to their limited values.
- Then Gas Control was turned on from the Equipment Console.
- Then Ar pressure value was set as 30 mTorr from Pressure Control Window.
- To allow the Ar gas leak inside the chamber for the plasma formation, “SET” button from Pressure Control Window was pressed. It automatically turned the throttle on.
- The pressure inside the chamber rapidly increases and approaches the value around 10^{-2} Torr.
- Before Turning the RF Source on from the Equipment Console wait until the Ar pressure reaches 30mTorr.
- After that in TH-DC-RF Source Control Window the RF Sputter Source parameters were defined as shown in the figure:

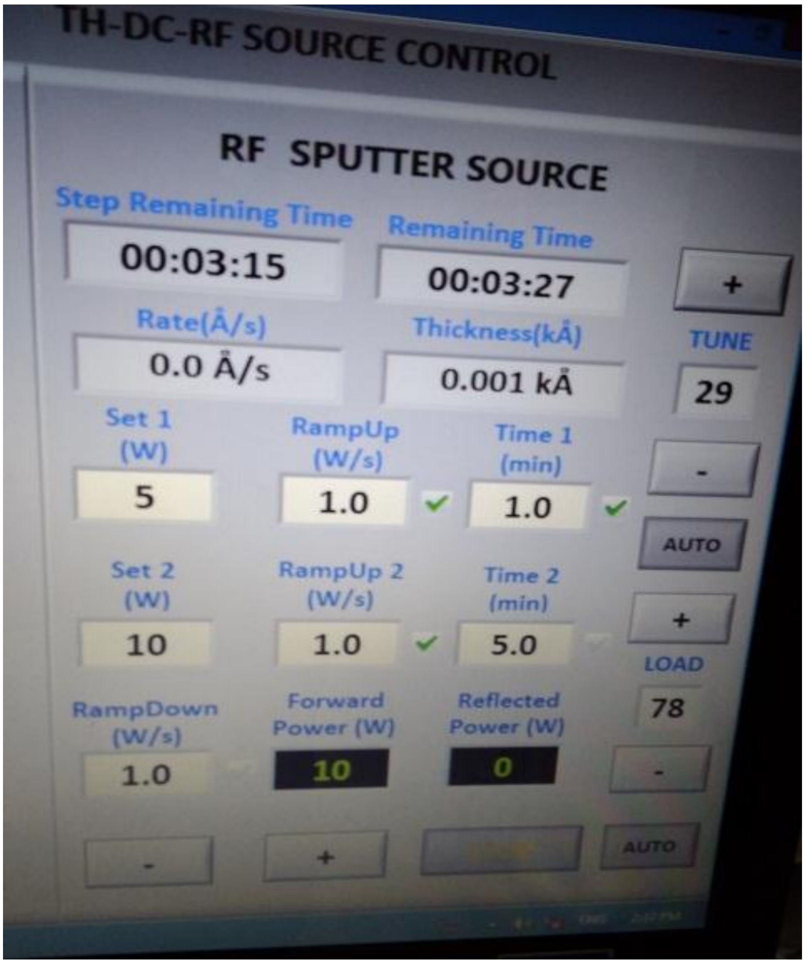

Figure 20: RF-Sputtering Parameters

- Shutter was removed and RF Sputtering source was turned on to start sputtering.
- After a minute or so plasma is generated of violet color (as can be seen in the picture below).

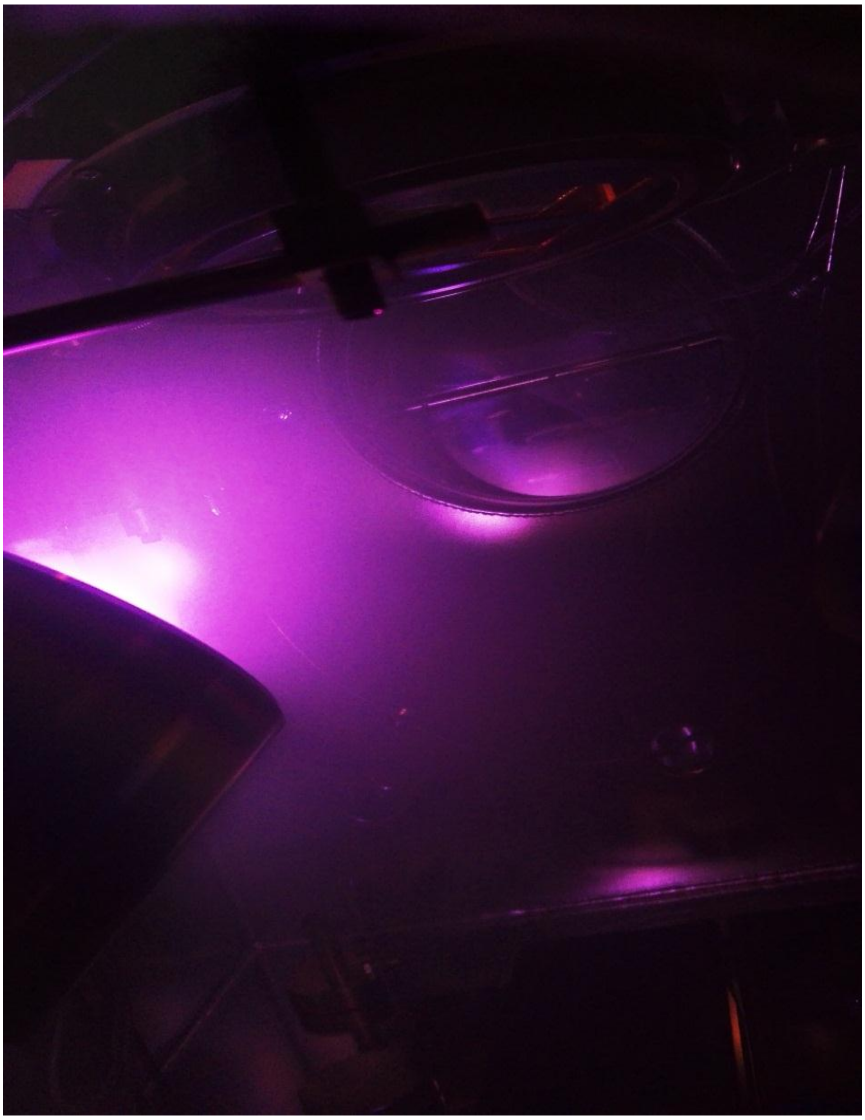

Figure 21: Violet Plasma Formation

- Finally, when the cleaning was finished the shutter closed automatically and sputtering was stopped.
- When we press “THROTTLE”, the vacuum level re-establishes abruptly, i.e. 10^{-2} Torr to 10^{-6} Torr.
- Then RF Source was turned off from the Equipment Console.

4.3 Thermal Evaporation of Chromium

Chromium due to its greater size and reflective properties was chosen as the adhesive layer for the silver and Aluminum to be deposited on the glass. The silver and Aluminum does not stick well with the glass and can be scrapped away easily. The atomic size of the Al is 143 pm and of Ag atom is 172 pm whereas the size of Cr atom is 200 pm. So to increase the adhesion between the glass and the both reflective thin films Cr was chosen as the smaller size atoms fill in well on the relatively larger atoms therefore Cr was chosen.

Figure 22: Cr Pellets (99.999% pure) Used in Thermal Evaporation

The deposition process of 18nm thin Cr layer is described below.

Firstly the chamber is evacuated and after the desired vacuum level i.e. 10^{-7} Torr is achieved then:

- First of all Thermal Source from the Equipment Console is turned on.
- Then charge source 1 was chosen by twisting the knob from the thermal source panel on Equipment Console.
- Cr pellets were placed in the wired basket in source 1. Wired baskets are mostly used for the charge which sublimates and deposited afterwards.
- After that from TH-DC-RF Source Control Window Thermal Source parameters were defined as [1,2] (here for Cr source) as shown in the figure below.

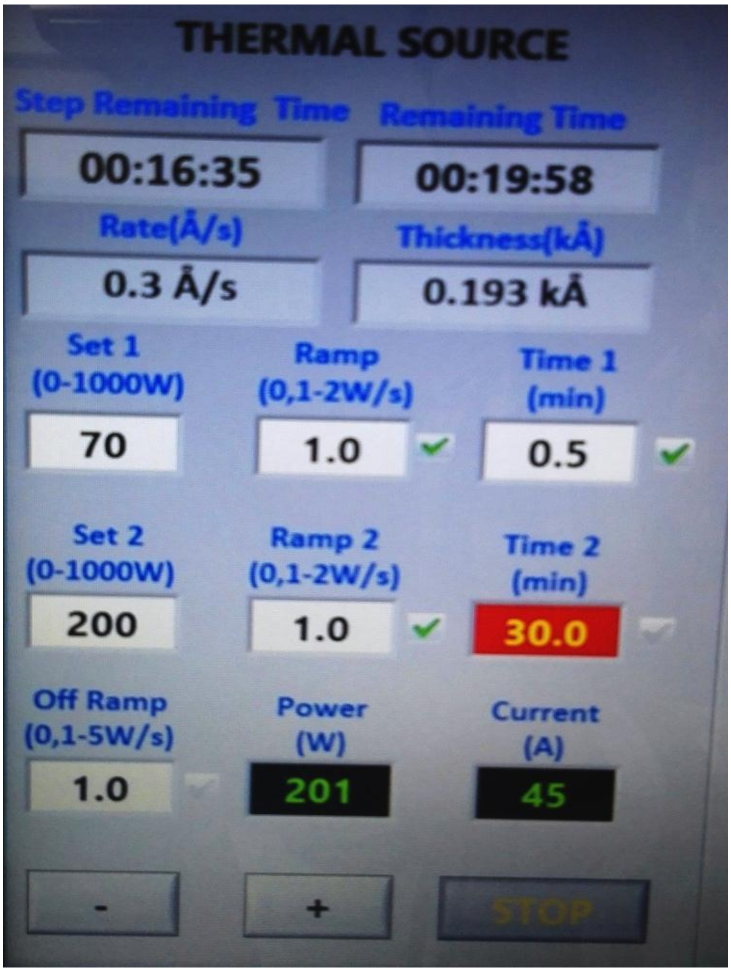

Figure 23: Parameters for Thermal Evaporation of the Cr

- “start” button from the Thermal source window begins the evaporation process.
- In a few seconds, the crucible turns red hot.
- Shutter opens automatically when the deposition starts but it is better to keep it closed manually until a justifiable and constant deposition rate is attained i.e. 0.3 Å/s or more (Deposition rate can be increased/decreased via increasing/decreasing the power).
- Once the constant deposition rate is attained, press “zero” and then “update” on the thickness control window and open the shutter.
- Finally, when the deposition is finished the shutter closes automatically.
- The deposition process is stopped then from Thermal Source window by pressing “STOP”.
- Then Thermal source was turned off from the Equipment Console.

4.4 Thermal Evaporation of Aluminum

Aluminum being cheaper than the other reflective metals is a good option to be used as the reflective thin films in solar thermal reflectors applications [3]. The properties like low weight, high solar reflectance, high strength and high corrosive resistance (as the oxide of the Al repel water and hence fights against the corrosion and protects the film at the surface) makes aluminum durable and effective to be used in the mentioned application.

The process to deposit the Al by thermal evaporation is the same as the Cr but there are only a few things and steps involve that differ. Al pellets of high purity (99.999%) were used to deposit thin film of only 100 nm thickness.

Thermal evaporation process of the Aluminum is given below:

First of all the chamber is evacuated and after the desired vacuum level i.e. 10^{-7} Torr is reached then:

- Thermal Source from the Equipment Console is turned on.
- Then charge of source 2 was chosen by twisting the knob from the thermal source panel on Equipment Console.
- As Al pellets were placed in the boat in source 2. Tungsten boats are mostly used for the charge that melts due to high melting point of tungsten.
- After that from TH-DC-RF Source Control Window Thermal Source parameters were defined (here for Al source) as shown in the figure 24.
- Initially, as the aluminum melts the deposition rate increase abruptly even at the low power applied. As it can be seen in the picture below that at only 70 watts applied power the deposition rate reaches 1.3 Ǻ/sec.

Figure 24: Parameters for Thermal Evaporation of the Al

- But it was observed that the deposition rate decreases after a few minutes. And the applied power has to be increased slowly to maintain the deposition rate [4][5].
- “start” button from the Thermal source window begins the evaporation process.
- In a few seconds, the crucible turns red hot.
- Shutter opens automatically when the deposition starts but it is better to keep it closed manually until a justifiable and constant deposition rate is attained i.e. 0.2 Å/s or more. (Deposition rate can be increased/decreased via increasing/decreasing the power)
- Once the constant deposition rate is attained, press “zero” and then “update” on the thickness control window and open the shutter.
- Finally, when the deposition is finished the shutter closes automatically.
- The deposition process is stopped then from Thermal Source window by pressing “STOP”.
- And then the Thermal Source is turned off from the Equipment Console.

4.5 Thermal Evaporation of Silver

Silver has the highest thermal and electrical conductivity among all the materials and also it can stand high temperatures. Ag also has very high solar reflectance and it has small particle size as well [6]. Therefore this makes it best candidate for the solar thermal reflectors applications.

The process to deposit the Ag by thermal evaporation is the same as the Cr and Al but there are only a few things and steps involve that differ. Ag thin film of only 130 nm thickness was deposited.

The parameters used are as [7,8] Thermal evaporation process of the Silver is given below:

First of all the chamber is evacuated and after the desired vacuum level i.e. 10^{-7} Torr is reached then:

- Thermal Source from the Equipment Console is turned on.
- Then charge of source 2 was chosen by twisting the knob from the thermal source panel on Equipment Console.
- As Ag pellets were placed in the boat in source 2. Tungsten boats are mostly used for the charge that melts due to high melting point of tungsten.
- After that from TH-DC-RF Source Control Window Thermal Source parameters were defined (here for Ag source) as shown in the figure below.

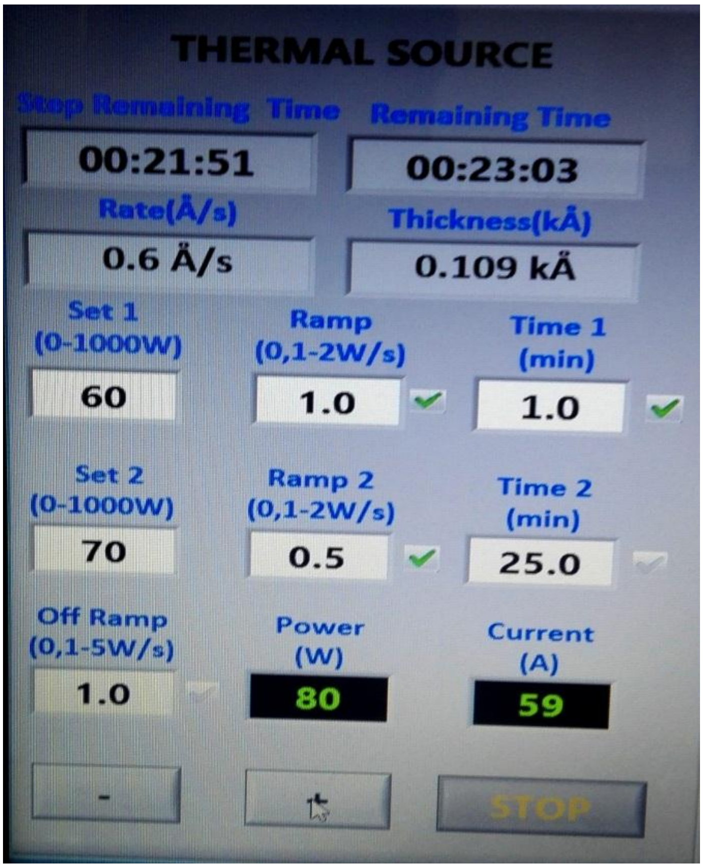

Figure 25: Parameters for Thermal Evaporation of the Ag

- “Start” button from the Thermal source window begins the evaporation process.
- In a few seconds, the crucible turns red hot.
- Shutter opens automatically when the deposition starts but it is better to keep it closed manually until a justifiable and constant deposition rate is attained i.e. 0.2 Å/s or more. (Deposition rate can be increased/decreased via increasing/decreasing the power)
- Once the constant deposition rate is attained, press “zero” and then “update” on the thickness control window and open the shutter.
- Initially, as the aluminum melts the deposition rate increases abruptly even when the low power was applied and then for a few minutes it maintains a constant deposition rate i.e. it remains at the 0.6 Å/s for most of the time during the deposition of the Ag.
- But it was observed that the deposition rate increases as the time passes. And the applied power has to be decreased slowly to maintain the deposition rate.

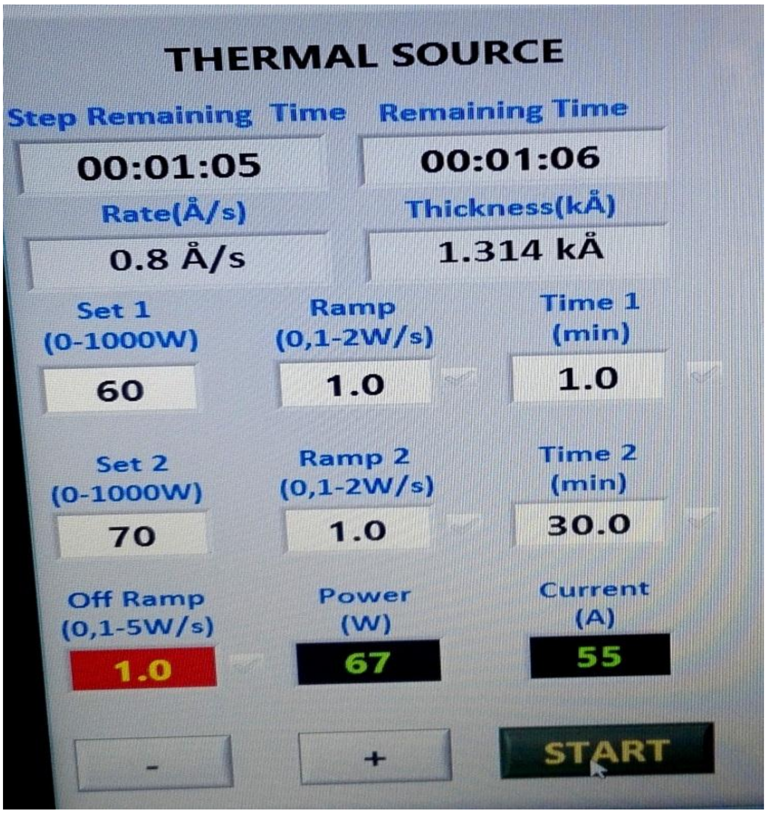

Figure 26: Deposition Rate Increased at the Same Power After Few Minutes

- This could be due to the fact that the charge melts more and more with the time and the evaporation process speeds up so does the deposition rate increases.
- Finally, when the deposition is finished the shutter closes automatically.
- The deposition process is stopped then from Thermal Source window by pressing "STOP".
- Then Thermal source was turned off from the Equipment Console.

4.6 Coating SiO_2 with RF-Sputtering

SiO_2 is a highly transparent, hard and durable. It can work as the protective layer for both silver and aluminum as they tarnish in the outdoor harsh weather conditions. Therefore for the safety of the reflecting layers SiO_2 has been selected [9,10].

SiO_2 was coated by using RF-sputtering process. The details of the experiment are given below with the key insights.

First of all the chamber is pumped to achieve the vacuum level and after the desired vacuum level i.e. 10^{-7} Torr is achieved then following steps are taken to perform the sputter cleaning:

- Ar and N_2 cylinders in accordance with their limited pressure values (For Ar max. value is 2 bar and for N_2 it is between 3-6 bar) were opened to their limited values.
- Then Gas Control was turned on from the Equipment Console.
- Then Ar pressure value was set as 30 mTorr from Pressure Control Window.
- To allow the Ar gas leak inside the chamber for the plasma formation, "SET" button from Pressure Control Window was pressed. It automatically turned the throttle on.
- The pressure inside the chamber rapidly increases and approaches the value around 10^{-2} Torr.
- Before Turning the RF Source on from the Equipment Console wait until the Ar pressure reaches 30mTorr.
- After that in TH-DC-RF Source Control Window the RF Sputter Source parameters were defined. Below is the picture of the vacuum control window:

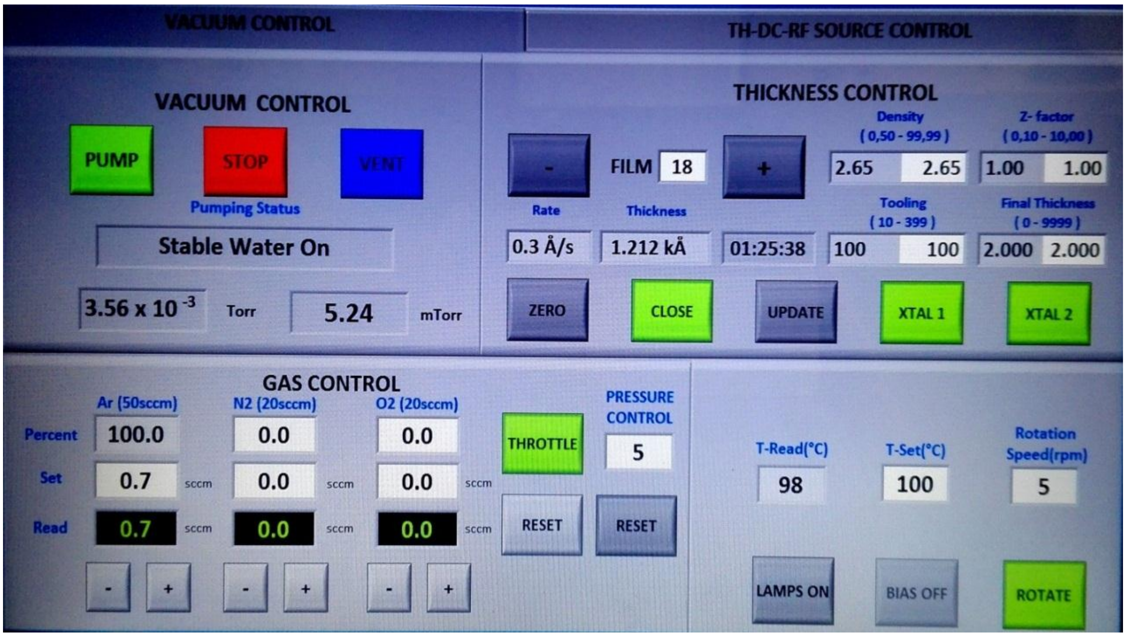

Figure 27: Vacuum Control, Thickness Control, Gas Control and Temperature Control Window for RF-Sputtering of SiO_2

- Shutter was removed and RF Sputtering source was turned on to start sputtering.
- After a minute or so plasma is generated of violet color (as can be seen in the picture below).

Figure 28: Violet Plasma Formation for SiO_2

- Finally, when the desired thickness of thin film was achieved, the shutter closed automatically and sputtering was stopped.
- then we press “THROTTLE”, the vacuum level re-establishes abruptly, i.e. 10^{-2} Torr to 10^{-6} Torr.
- Then RF Source was turned off from the Equipment Console.

Summary

In this chapter the experimental procedure to produce the thin films of the Al, Ag and SiO_2, by using Combined Thermal and Sputter System, has been discussed in detail. The parameters used for each of the thin film has been shown and deliberated.

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CHAPTER 5

Results and Discussion

In this chapter, the characterization results through Scanning electron microscopy (SEM), Atomic force microscope (AFM), X-Ray Diffraction (XRD) have been discussed. Optical characteristics by UV-VIS-IR Spectrophotometer for reflectance measurement of the all prepared samples before and after the tape test of glass/Cr/Al, glass/Cr/Ag, glass/Cr/Al/SiO_2 and glass/Cr/Ag/SiO_2 and of Integrated (glass/Cr/Ag/SiO_2/Ag/SiO_2) and Compound (glass/Cr/Al/SiO_2/Ag/SiO_2) have been discussed in detail.

5.1 Crystallinity Test

X-Ray Diffraction was performed for all the prepared samples. Below are the Diffractograms of each sample.

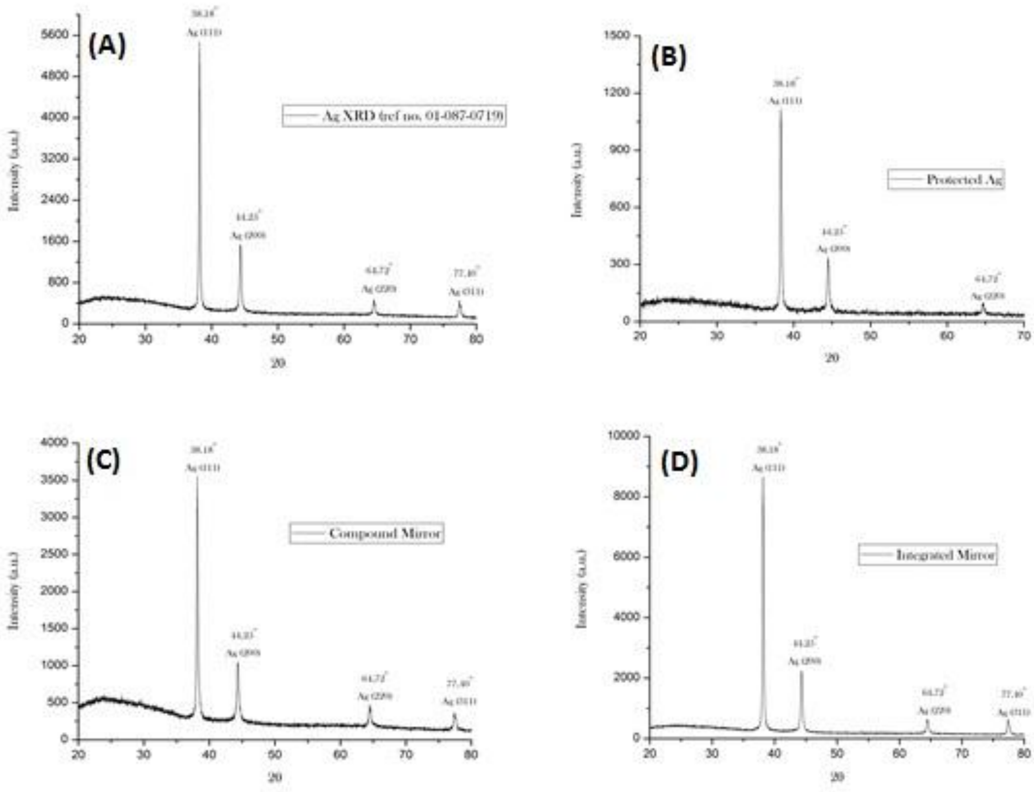

Figure 29: Ag Based Mirrors XRD Diffractograms (A) Ag Based Mirror (B) Protected Ag Based Mirror (C) Compound Mirror (D) Integrated Mirror

Only Ag has shown a crystalline behavior in the multilayer thin films. Aluminum and silica has totally amorphous nature.

Below Diffractograms show the amorphous behaviour of the Al based mirrors. Even small peaks of the Al_2O_3 were found in the Al based mirrors because the outdoor exposure of the samples Al, being very reactive, starts to convert into Al_2O_3 which degrades the mirrors optical characteristics[1]

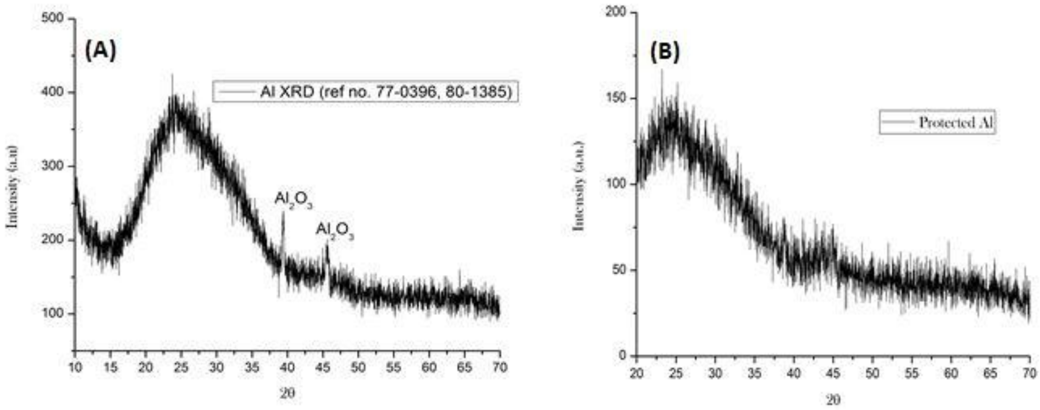

Figure 30: Aluminum Based Mirrors Diffractograms (A) Unprotected Al Mirrors (B) Protected Al Mirrors

5.2 Morphological Analysis

All the prepared samples glass/Cr/Al, glass/Cr/Ag, glass/Cr/Al/SiO_2 and glass/Cr/Ag/SiO_2 thin films were analyzed by the AFM and SEM. AFM is basically used to measure the surface roughness of the samples with only reflecting films and with the protected reflecting thin films to compare and observe if the surface roughness increases or decreases due to the SiO_2 protective layer.

The AFM results of the prepared samples are given below:

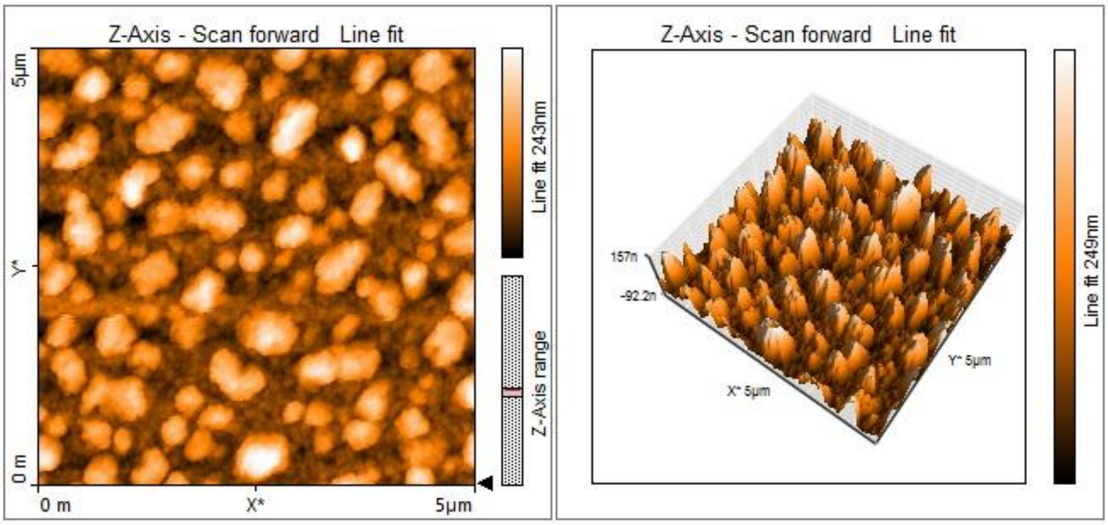

Figure 31: AFM Micrograph of Glass/Cr/Al Sample at 5 µm

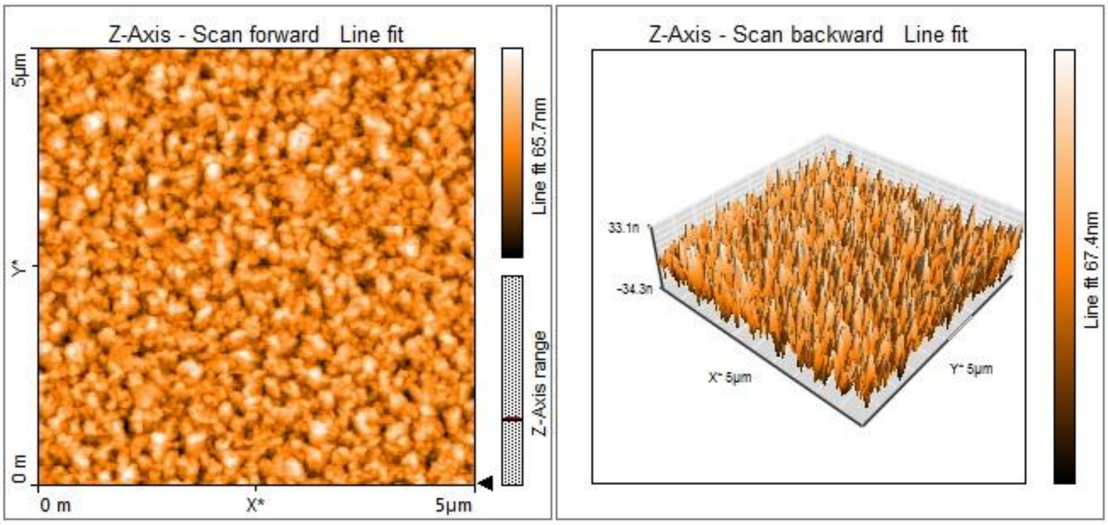

Figure 32: AFM Micrograph of Glass/Cr/Ag Sample at 5 µm

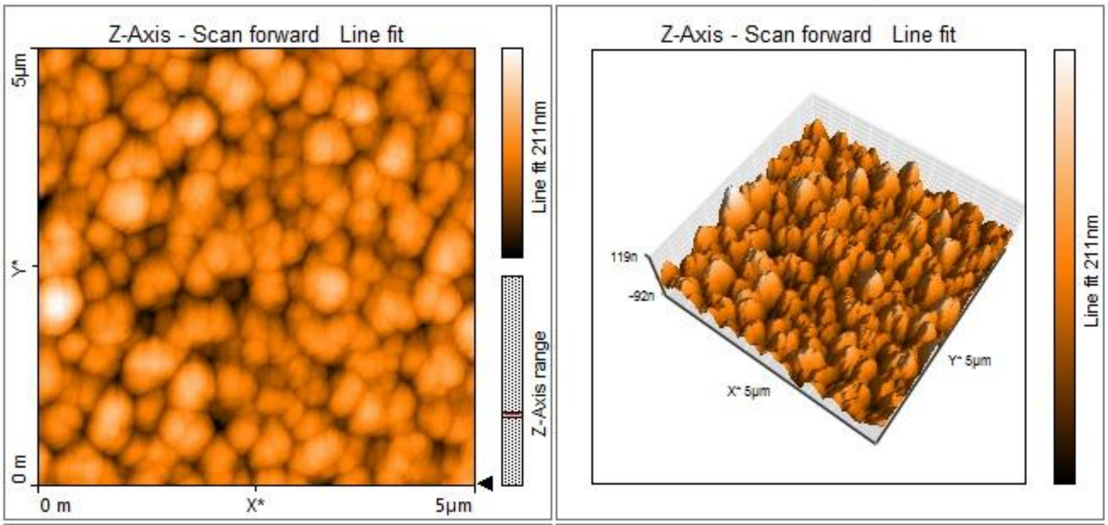

Figure 33: AFM Micrograph of Glass/Cr/Al/SiO_2 Sample at 5 µm

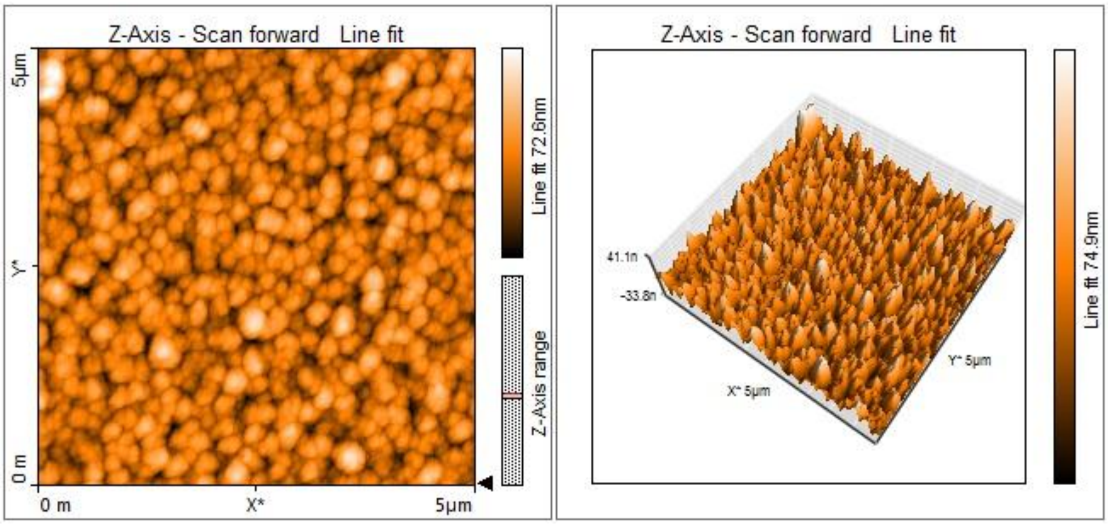

Figure 34: AFM Micrograph of Glass/Cr/Ag/SiO_2 Sample at 5 µm

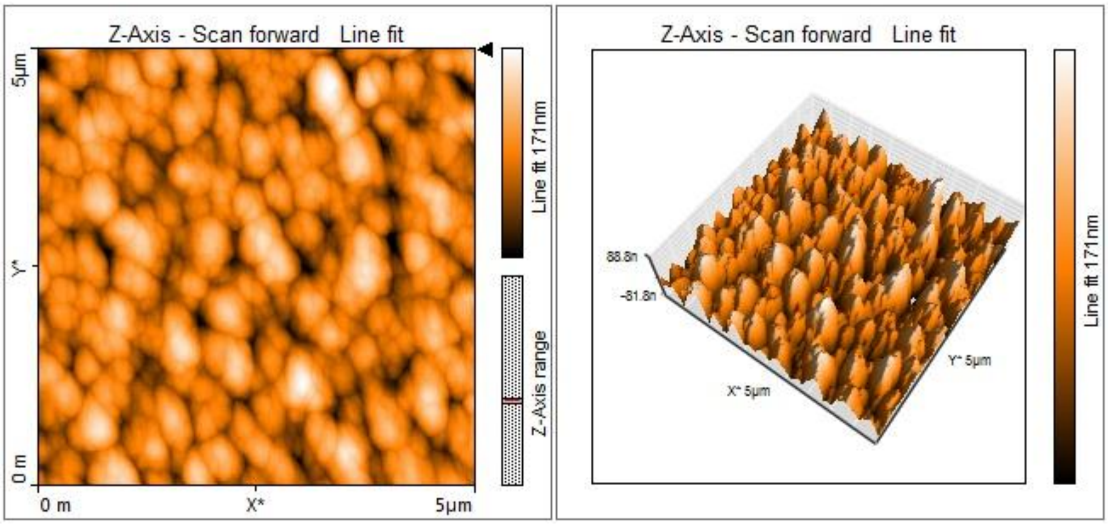

Figure 35: AFM Micrograph of Compound Mirror at 5 µm

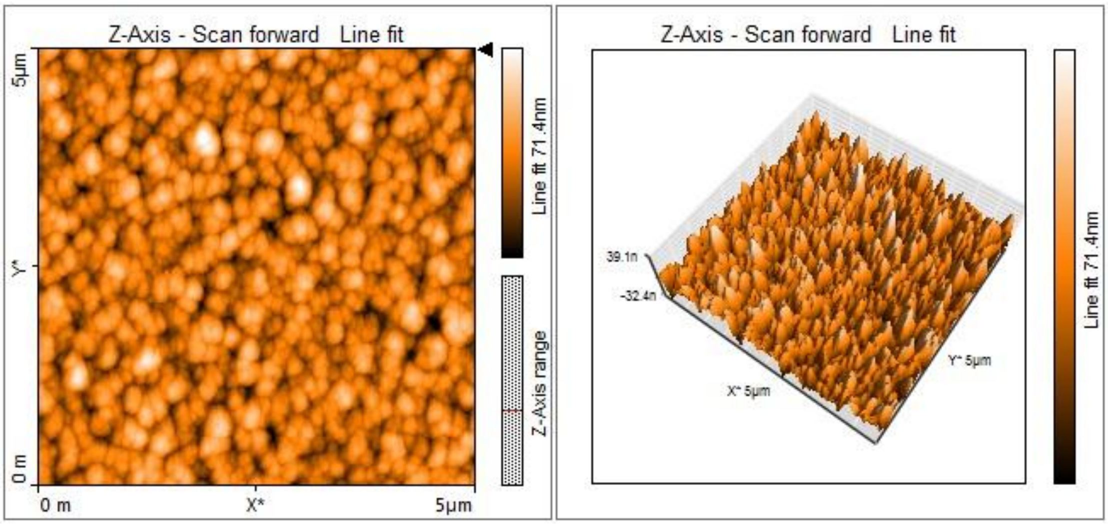

Figure 36: AFM Micrograph of Integrated Mirror at 5 µm

The table below shows the thickness and average roughness of the all the prepared samples.

Table 2: Surface Roughness Measured by the Atomic Force Microscopy

Name	Thickness (nm)	Average Roughness (nm)
Unprotected Al Mirror	120	40.9
Protected Al Mirror	320	21.7
Compound Mirror	550	22.1
Unprotected Ag Mirror	150	8.2
Protected Ag Mirror	350	7.4
Integrated Mirror	580	8.3

As the sharp cones in the structures of both Al and Ag thin films can be seen in the figure 29 and figure 30. The roughness of the aluminum based mirrors was more as the grain size of the Al is greater than the Ag. Therefore its roughness is also greater. But the deposition of the SiO_2 helped to not only protect the mirrors but also made the surface smoother. As it can be observed that the surface roughness of the protected Al mirrors is less than the unprotected aluminum mirror. Such decrease in the surface roughness does not increase reflectance but it helps to resist the possible abrasiondue to sand and hence it resists reduction in reflectance [2].

The surface roughness of the thin films plays important role in the efficiency of the solar thermal power plant. This is due to the fact that as the surface roughness increases the scattering off the surface of the solar light becomes more and more dominant and we get diffused reflectance instead of the specular reflectance which then increases therefore it affects the performance of the power plant because the concentrated light does not get there where it was supposed to be reached. Only a 5% decrease in the reflection of the light can drastically decrease the overall efficiency of the power plant [3].

Scanning electron microscopic analysis is performed to see the thin films at the nano scale. But in our case we only needed to observe if the thin films are uniform and homogenous and that there are no impurities or holes present in the thin films. For this confirmation, SEM images at different scales of magnification and work distances are captured and discussed.

The SEM images are given below:

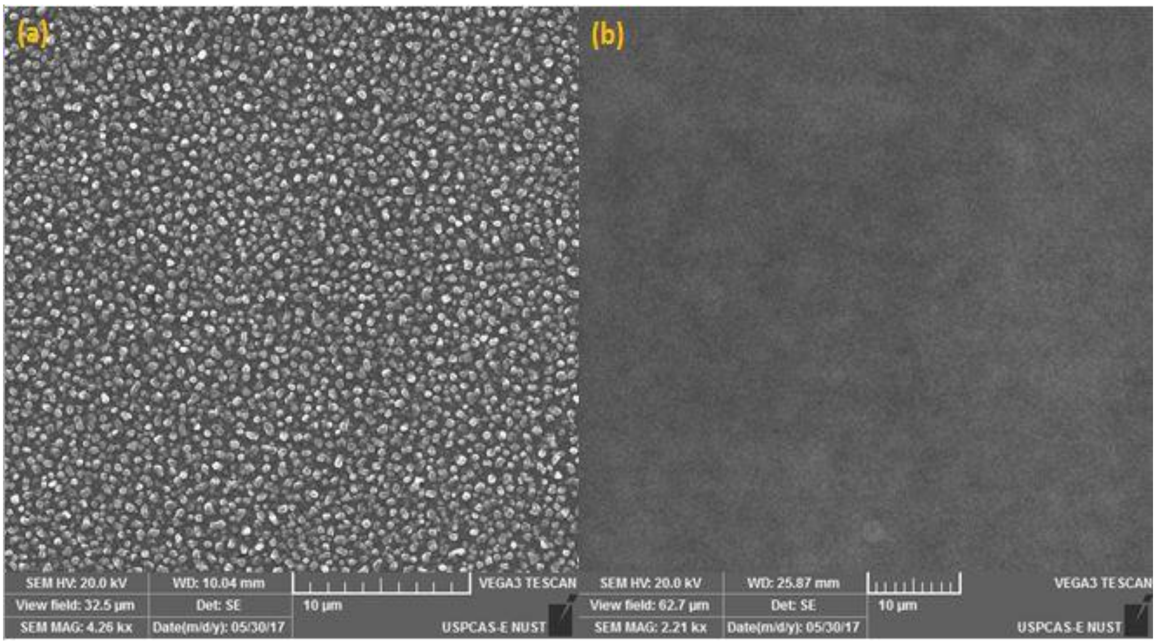

Figure 37: SEM Micrographs of Glass/Cr/Al at 10 µm (a) When Work Distance is 10 µm (b) When Work Distance is 25 mm

In figure 37 (a) we can see that the film is smooth and the film material has similar behavior in whole area under consideration. We can observe that when the work distance is increased, as in the figure 37 (b), the film has compact morphology.

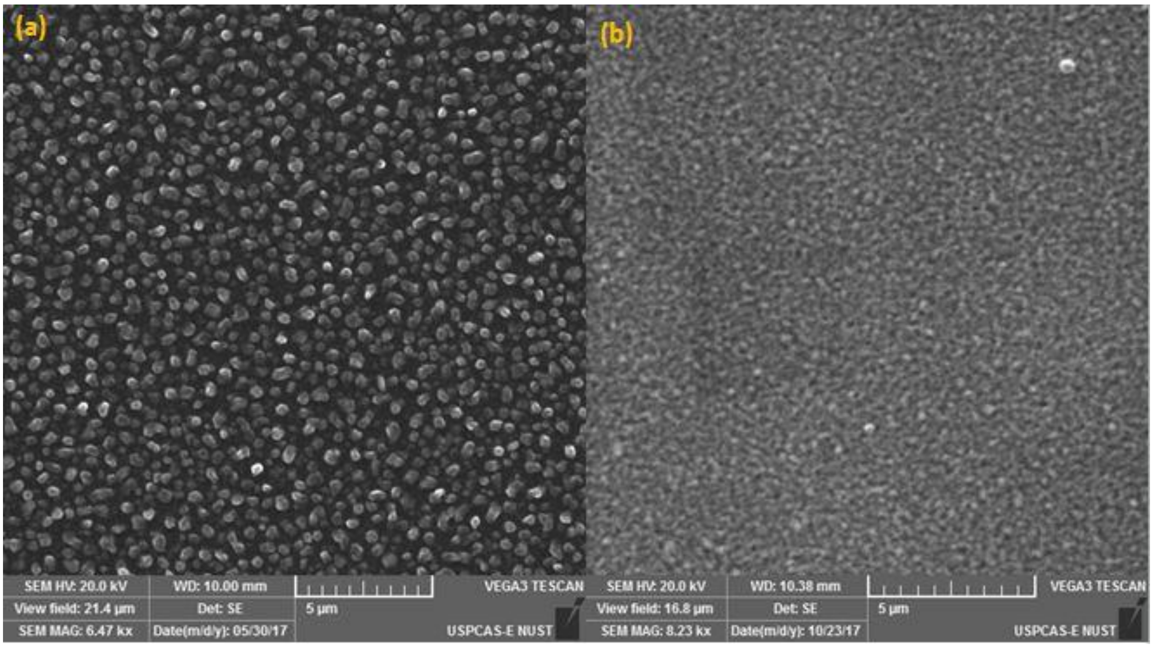

Figure 38: SEM Micrographs of (a) Glass/Cr/Al at 5 µm (b) Glass/Cr/Ag at 5 µm

We can see that at the same magnification the Aluminum thin films has greater grain size, in figure 38 (a), than Silver thin films but thin films are homogenous and smooth and has the same morphological behavior throughout the area under the observation. This reason makes the fact clear that why Al thin films has greater surface roughness as measured by the AFM and that was mentioned in the section above.

Below are the SEM images of the protected Al and Ag mirrors.

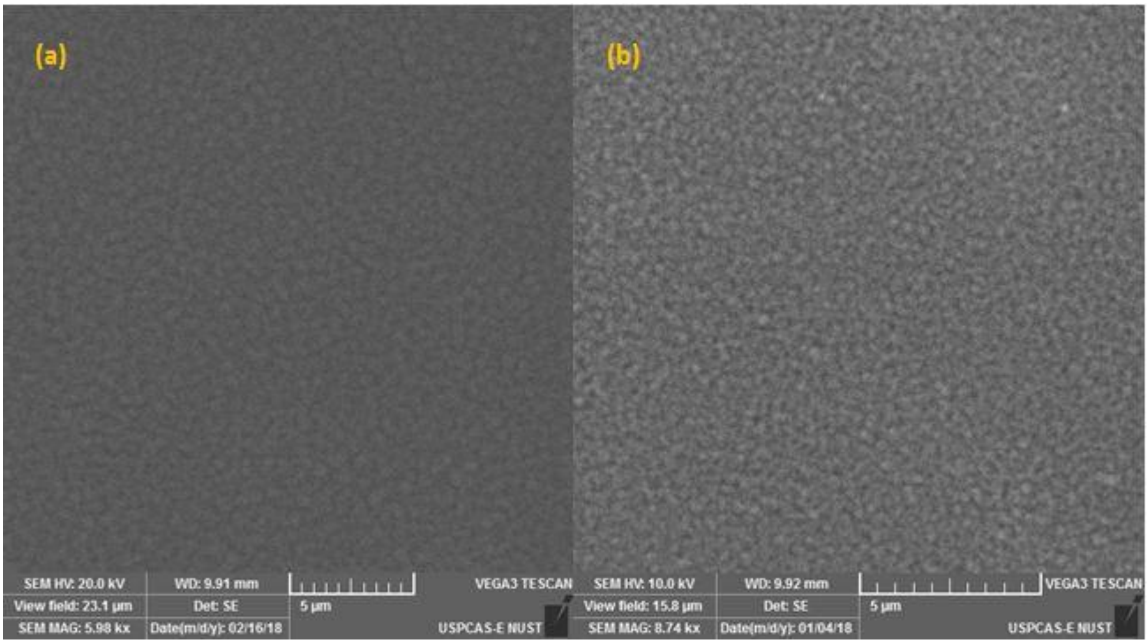

Figure 39: (a) SEM Micrograph of Al Protected Mirror at 5 µm (b) SEM Micrograph of Ag Protected Mirror at 5 µm

Figure 39 (a) above shows that after the deposition of the SiO_2 the grain type structure of the Aluminum thin film turned into more compact morphology and same happened with the silver thin films surface.

But in the case of Compound mirrors there were many small white dots present on the surface that refers to the some impurities when it was observed at 20 µm as shown in the figure 40 (b) given below:

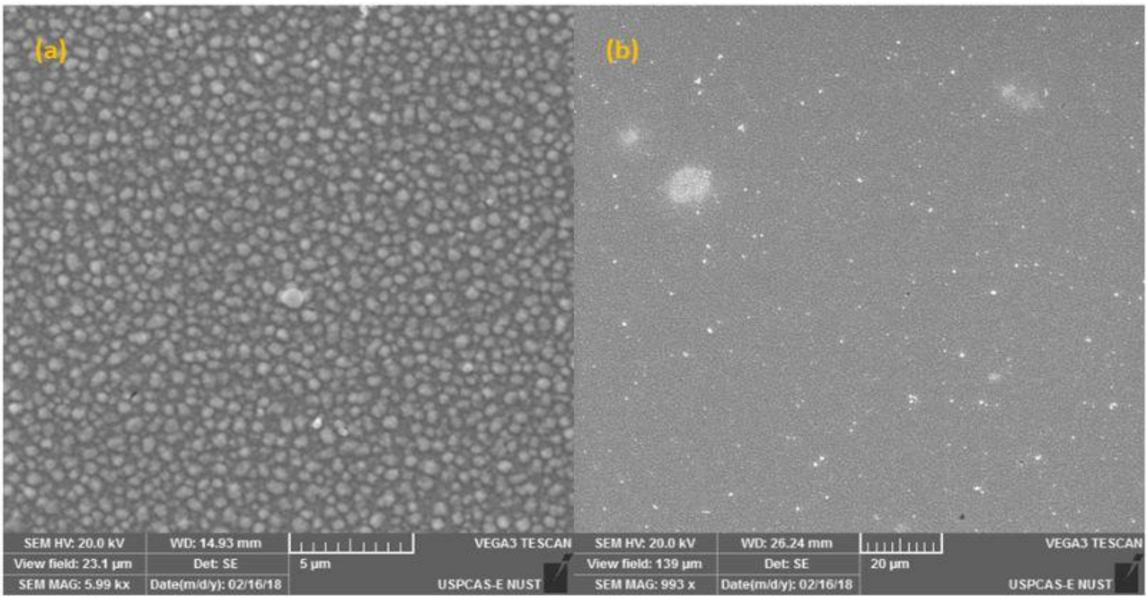

Figure 40: (a) SEM Micrograph at 5 µm (b) SEM Micrograph at 20 µm

But the partial enlargement of SEM micrograph in figure 40 (a) shows that the film has compact morphology. But there is still some room for the improvement of these coatings under the vacuum to make them less pollutant.

Below are the images of the Integrated mirrors. Figure 41 (b) shows a hole on the surface of the thin films. This again could be due to the fact that the surface was not already cleaned well. Or the scratch while handling the samples in the chamber could have caused this type or hole in the surface.

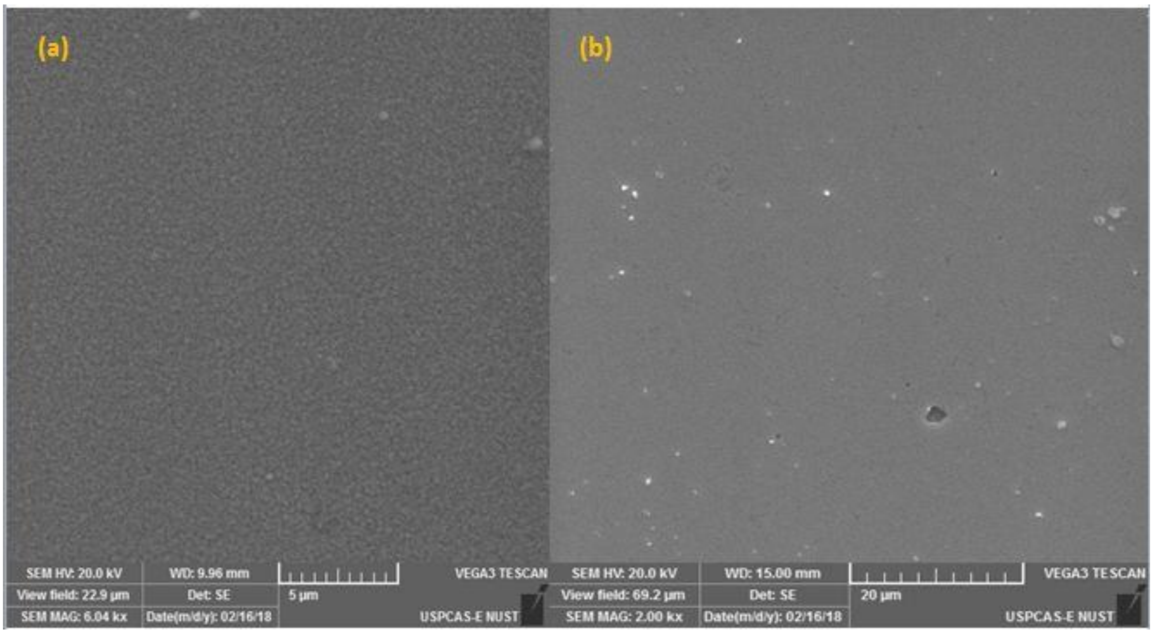

Figure 41: SEM Micrographs of Integrated Mirror (a) at 5 µm (b) at 20 µm

But the partial enlargement of SEM micrograph shown in figure 41 (a) depicts that the film has compact morphology.

5.3 Optical Characterization

Optical analysis is basically the main testing criterion for the prepared samples. As the application of these films is in the solar thermal concentrators. And higher the reflectance higher will be the efficiency of the power plant as more and more radiant flux would be gained by the receivers. The optical analysis includes the solar reflectance, transmittance and absorption measurement typically from 250 nm to 2500 nm by using UV-VIS-IR spectroscopy before and after the tape test of the samples.

5.3.1 **Reflectance Spectrum of Samples before Tape Test**

Reflectance spectrum of the Aluminum based with protected layer and without protected layer samples and of silver based with protected layer and without protected layer samples was obtained by using Lambda 950 UV-VIS-IR spectroscopy.

The reflectance spectrum of each sample is given below:

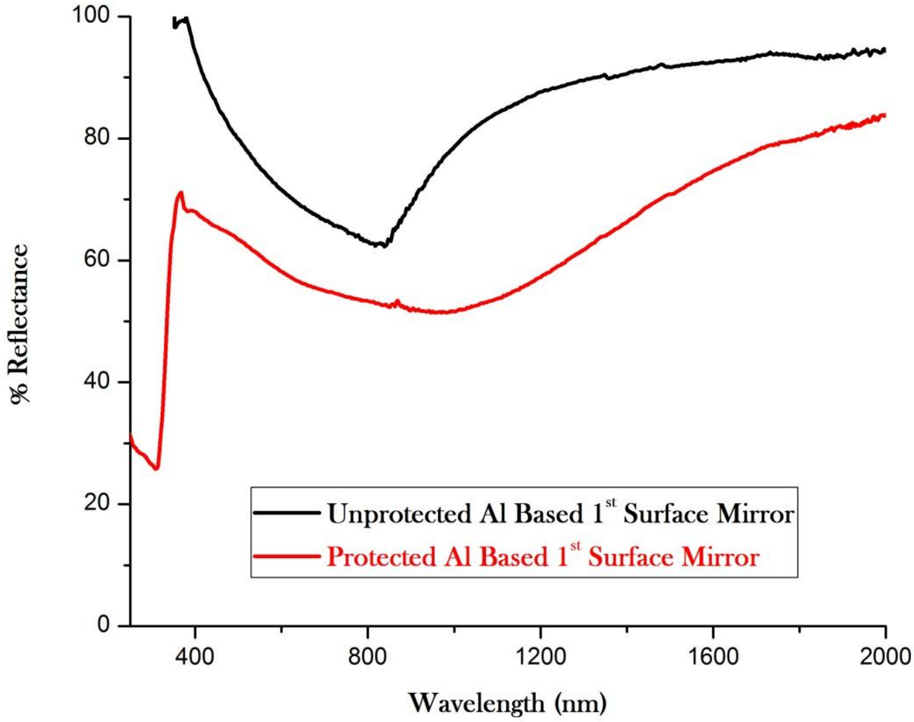

Figure 42: Aluminum Based Mirrors Reflectance Spectrum

The average reflectance of the Aluminum based mirror is 89%. that is 4% more than already achieved reflectance mentioned in the literature[4][1].

The reflectance of the thin films depends on the roughness, base layer and the environment in which they are prepared. So the use of chromium and in a very high vacuum conditions the coating of the Al thin film depicts the clear increase in the reflectance upto 89% as compared to the reflectance of the polished Aluminum alloy (86%) and galvanized iron (84%) as found in the literature [5] .

The reflectance of the aluminum based mirror protected with the SiO_2 thin layer is observed to be decreased. The average reflectance of this mirror is 64% that is very low as compared to the reflectance of the aluminum mirror without protected layer.

The reason behind the low reflectance of this mirror could be that the SiO_2 layer's thickness is too high (i.e 200 nm) that needs to be optimized.

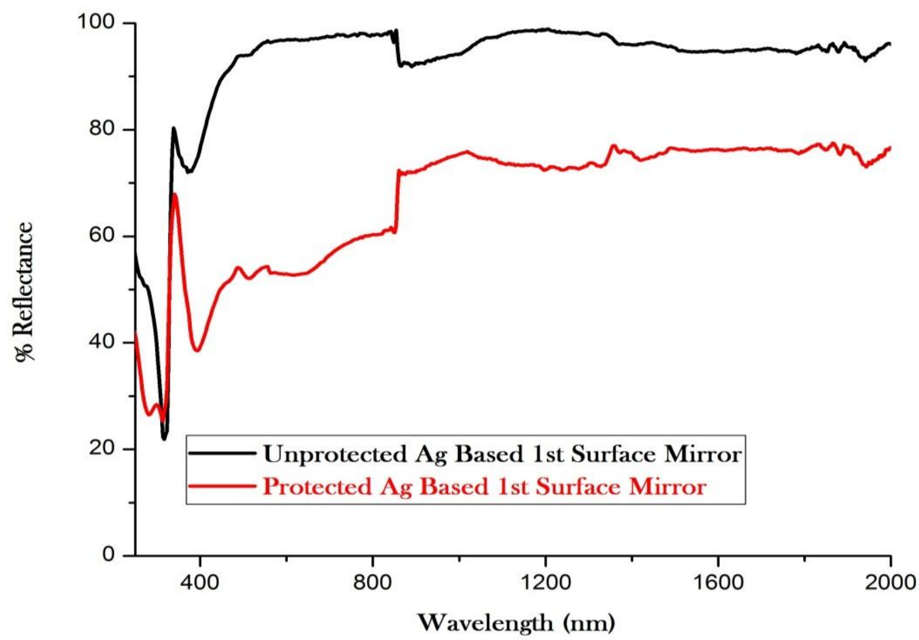

Figure 43: Silver Based Mirrors Reflectance Spectrum

Ag based mirrors has the highest average reflectance than all of the samples prepared in our work. The average solar reflectance of these samples is 93% which is higher than the aluminum based reflectors as expected. The reason it has more reflectance than the Aluminum is that the reflectance in the visible and near infrared region of the

Ag based mirrors is greater than the Al based mirrors. Like in the range of 400 nm to 1000 nm the average reflectance of Ag based mirrors is 95% but in the same mentioned range the reflectance of the Al based mirror is only 72% that is 23% lower than the Ag based mirrors.

As in the case of Al based mirrors when protecting layer of the SiO_2 was coated on the Ag based mirrors, the decrease in the reflectance was observed. The average reflectance of protected Ag based mirrors is about 67% that is 25% less than the reflectance of the unprotected mirrors.

5.3.1.1 Integrated and Compound Mirrors

Integrated mirrors have composition as (glass/Cr/Ag/SiO_2/Ag/SiO_2) which includes Ag and SiO_2 additional layers. As the reflective layers are the same i.e Ag, therefore we call it Integrated mirror and the Compound mirror (glass/Cr/Al/SiO_2/ Ag/SiO_2) is the one in which Al and SiO_2 layers are then covered by the Ag and SiO_2 layers. As the reflective layers are different i.e Al and Ag, so we call it compound mirror.

The reflectance graphs of both are given below:

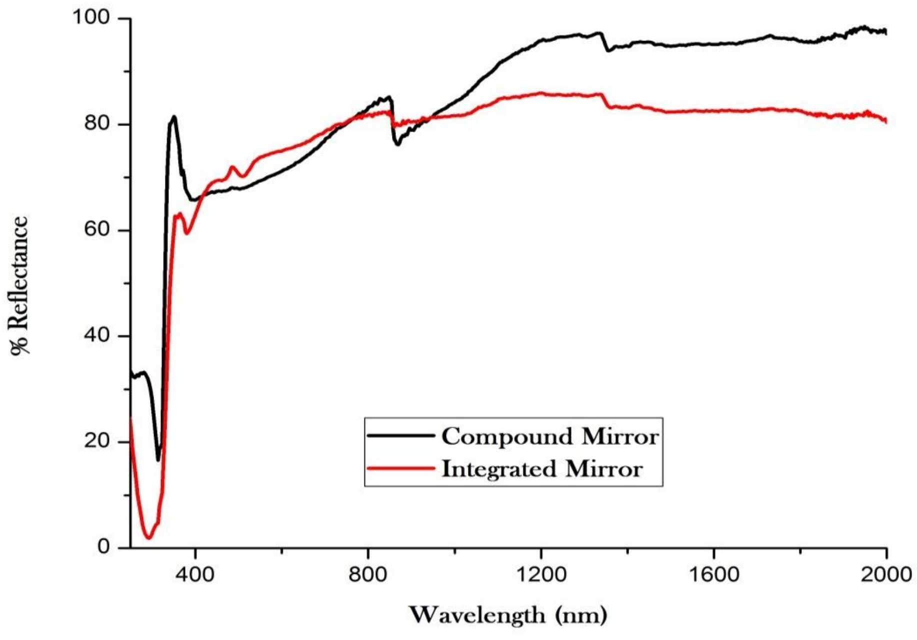

Figure 44: Reflectance Spectrum of Compound and Integrated Mirror

The average reflectance of the integrated mirror is about 76%. As we coated the same silver based mirror with protective layer whose reflectance was 67%. The other extra two layers of the same material helped an increase in reflectance of 9%. Which is a lot of improvement in reflectance considering the loss of reflectance by protective layer about 25% in the before mentioned case.

The average reflectance of the Compound mirrors is more than the integrated mirror it is about 84% which is a lot of improvement. We can also say that the combination of Al and Ag coated with SiO_2 in between them works better than the Ag and Ag combination having SiO_2 between them. This was obviously increased by having the Ag on the top that has added up an improved reflectance in the visible region for the Al protected mirrors.

5.3.2 Reflectance Spectrum of Samples after Tape Test

The reflectance spectrum of the samples after they were tape tested which was basically a test to check the adhesion of the thin films with the glass [6]. The reflectance spectrum obtained, provide a proof of very strong adhesion of the Aluminum with the glass coated with a very thin layer base layer of Cr.

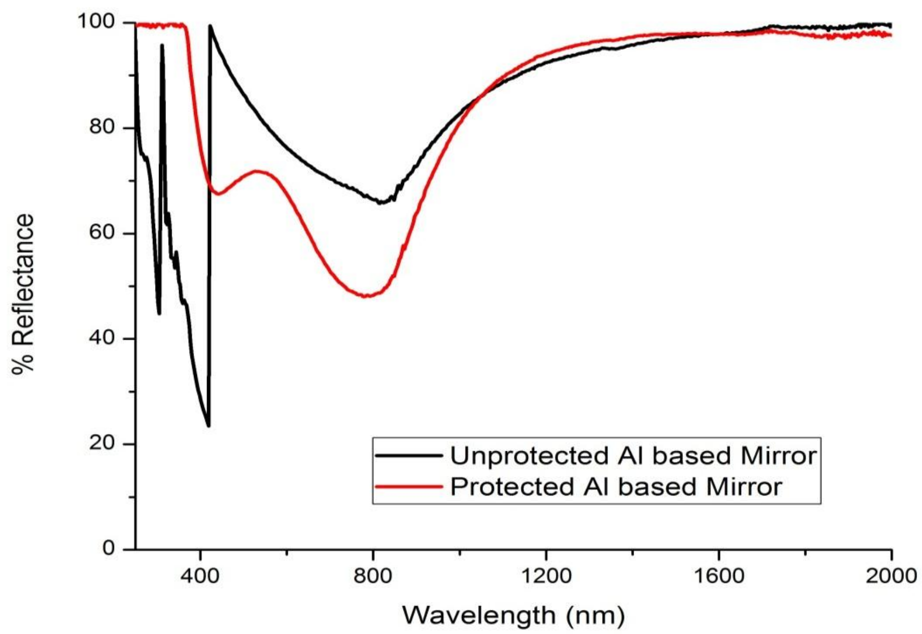

Figure 45: Aluminum Based Mirrors Reflectance Spectrum after Tape Test

The average reflectance of the Al based unprotected and protected mirrors after the tape test is observed to be 85%. It can be witnessed that the Al thin film has shown a very strong adhesion with the glass that even after a vigorous tape test the film has shown a reflectance of 85% but on the other hand the thin film of SiO_2 is observed to be removed from the surface as the reflectance of the protected Al based mirrors is increased to 85% from 64% that was when the protective coating was on the Al based mirror. It can also be said that the SiO_2 was effective and protective that due to which reflectance of the mirror didn't decrease.

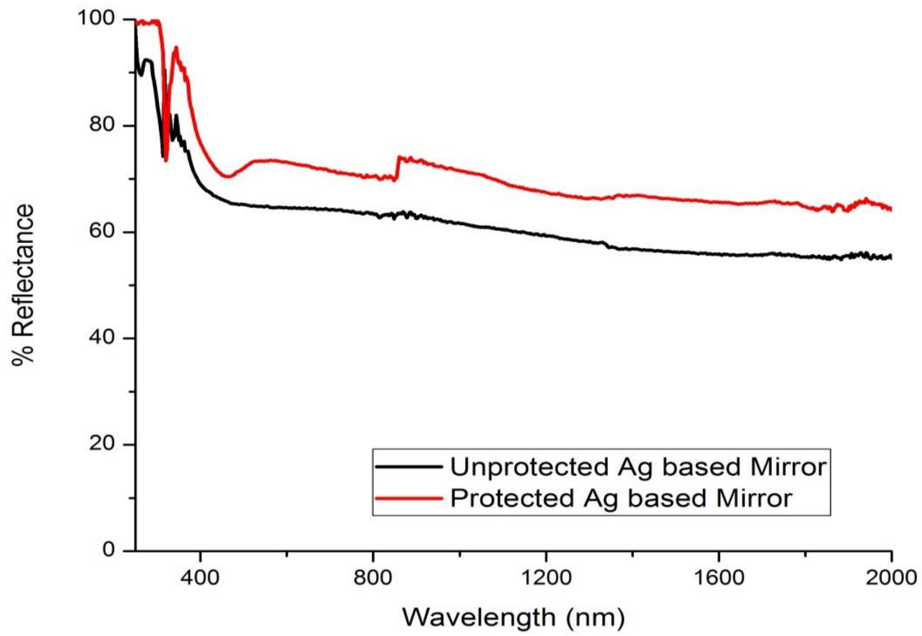

Figure 46: Silver Based Mirrors Reflectance Spectrum after Tape Test

In the case of Ag based mirrors the unprotected and protected mirrors have shown the reflectance of 62% and 71%. As the adhesion of the Ag with the glass is not good therefore most of the Ag thin film was peeled off the glass surface and stuck with the tape but still some of the silver remained there showing 62% reflectance. On the other hand in the protective Ag based glass the thin film of SiO_2 protected Ag layer that SiO_2 layer must have been removed by the tape exposing more silver and as a result it has shown 71% reflectance after the tape test.

Similarly considering the behaviours of the thin films of Al, Ag and SiO_2 the Integrated mirror has the reflectance increase as the top protective coat was removed due to the tape test exposing more Ag thus showing a high reflectance percentage of 89.

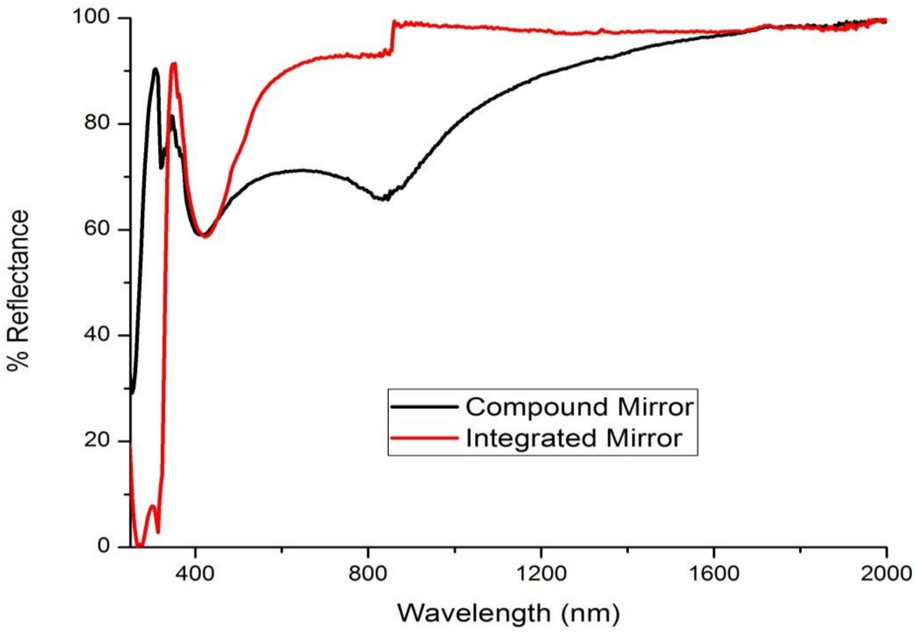

Figure 47: Reflectance Spectrum of Compound and Integrated Mirrors after Tape Test

On the other side the reflectance of the Compound mirror is 83% again similar to the value of the reflectance of the samples without tape test. As the Al thin film was covered by the additional layers of the Ag and SiO_2 which were removed by the tape but Aluminum below wasn't affected hence it showed a very high reflectance.

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CHAPTER 6

Conclusions and Future Recommendations

6.1 Summary

The thin films of Silver and Aluminum for front surface reflector applications were successfully prepared. These films were produced by the Thermal Evaporation process. Silica was used as the protective thin film that was coated by RF-Sputtering method. Six types of combinations of multi-layer thin films were prepared for the application of solar reflectors that are glass/Cr/Al, glass/Cr/Ag, glass/Cr/Al/SiO_2, glass/Cr/Ag/SiO_2, Integrated (glass/Cr/Ag/SiO_2/Ag/SiO_2) and Compound (glass/Cr/Al/SiO_2/Ag/SiO_2) mirrors. All these samples have surface roughness 40.9 nm, 8.2 nm, 21.7 nm, 7.4 nm, 8.3 nm and 22.1 nm respectively. SEM micrographs showed a compact and homogeneous surface morphology of the all combination of thin films. A few holes and small white dots were observed in the case of integrated and compound mirrors that refer to the some impurities presence in the thin films from the environment. The reflectance of unprotected Al and Ag based front surface thin films was measured to be 89% and 93% respectively. The reflectance of the protected mirrors was 67% in the case of Ag and 65% in the case of Al. The compound mirror showed reflectance upto 84% and the reflectance of the integrated mirror was 76%. Even after the tape test that was used to check the adhesion of the thin films, the films have shown a very high reflectance upto 85% mainly of Aluminum based mirrors. So considering all these results and performances of the thin films these coatings show great potential to be used as front surface reflectors application for solar thermal reflectors due to high reflectance and dense surface morphology.

6.2 Deduction

- The Silver coated mirrors have more compact morphology, low roughness and crystalline structure than the Aluminum coated reflectors. They also have showed higher reflectance than the Aluminum based mirrors.

- But the adhesion of the Ag thin films was very poor when tape test was performed.
- The roughness of the silver based mirrors was observed to be 8 nm and the roughness of the Al mirrors was observed to be 40.9 nm which is obviously very high than Ag based mirrors.
- The coating of SiO_2 helped to improve the surface roughness of the Al and as well as Ag based mirrors.
- The tape test showed that SiO_2 showed resistance and provided protection to the reflective layers from peeling off the glass surface.
- The SEM images showed that there were no holes present in the thin films and EDS analysis detected no impurities in the prepared thin films of all the prepared six types of samples.
- The light reflectance of the Ag based mirrors was about 94% but it was 77% only for the Al coated mirrors.
- The solar reflectance of the Ag based mirrors was 93% and of Al based mirrors it was 89%.
- Al thin film showed great adhesion with the glass and remained unchanged even after they went under tape test.
- When these mirrors were coated with the protective layer of SiO_2 the reflectance of both these mirrors decreased to about 20% in the Al based and 25% in the Ag based mirrors.
- The solar reflectance of the integrated mirrors is 76% and of the compound mirrors was measured to be 84%.
- The reflectance after the tape test was 85% of the Aluminum based mirrors that shows great adhesion bonding of Al with glass.
- Reflectance of Ag based mirrors was observed to be less than the Al based mirrors after the tape test.
- Both aluminum and silver based mirrors due to having high reflectance specially in the IR region of solar radiations and very low surface roughness shows great potential to be used as concentrators in solar thermal power plants.

6.3 Future Recommendations

- The optimum value of reflective thin film thickness should be experimentally determined.
- Similarly the optimum thickness for the top protective coating should also be experimentally determined.
- The effect of different vacuum levels on these thin films can also be studied.
- The economic analysis for these thin films for different kinds and shapes of the reflectors can be performed.