INVESTIGATION OF SOLID STATE Nd:YAG LASER TEXTURING ON POLYIMIDE SUBSTRATE USING SPOT OVERLAP FOR EFFICIENT PHOTOVOLTAIC DEVICE APPLICATIONS

M.Tech. Thesis

By

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DISCIPLINE OF MECHANICAL ENGINEERING

INDIAN INSTITUTE OF TECHNOLOGY INDORE

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INVESTIGATION OF SOLID STATE Nd:YAG LASER TEXTURING ON POLYIMIDE SUBSTRATE USING SPOT OVERLAP FOR EFFICIENT PHOTOVOLTAIC DEVICE APPLICATIONS

A THESIS

Submitted in partial fulfillment of the requirements for the award of the degree

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Yadav Vinayak Manik

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CANDIDATE'S DECLARATION

I hereby certify that the work which is being presented in the thesis entitled **Investigation Of Solid State Nd:Yag Laser Texturing on Polyimide Substrate Using Spot Overlap for Efficient Photovoltaic Device Applications** in the partial fulfillment of the requirements for the award of the degree of **MASTER OF TECHNOLOGY** and submitted in the **DISCIPLINE OF MECHANICAL Indian Institute of Technology Indore**, is an authentic record of my own work carried out during the time period from July 2014 to July 2016. Thesis submission under the supervision of Dr.I.A.Palani associate professor Discipline of Mechanical Engineering and Dr.M.Anabarasu associate professor Discipline of Electrical Engineering.

The matter presented in this thesis has not been submitted by me for the award of any other degree of this or any other institute.

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Yadav Vinayak Manik

Dedicated to known or unknown peoples who work for betterment of people.

ABSTRACT

This dissertation presents study of texturing of polyimide used as substrate of flexible solar cell by using nanosecond Nd: YAG laser. Irradiation of intense nanosecond laser pulse at various experimental parameters alters surface morphology of polyimide. Laser induced periodic surface structure (LIPSS) was observed and its mechanism also elaborated. Variation of optical, structural properties with respect to variation of experimental parameter is investigated. It is observed that Laser texturing of polyimide changes optical properties drastically. Textured polyimide substrate can be used as a substrate of thin film solar cell to enhance light trapping which leads to improvement in efficiency.

LIST OF PUBLICATIONS

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CONTENTS

LIST OF FIGURES

List of Tables

Chapter 1 INTRODUCTION

1.1 Photo Voltaic Cell

Solar cell made from semiconductor which has properties of metal and some properties of insulator. Silicon is semiconductor which is widely used in solar cell fabrication. P-type semiconductor is obtained when semiconductor doped with trivalent impurities to create deficiency of electron called as 'holes'. If semiconductor is doped with penta-valent impurities to create excess electron then it will become N-type semiconductor. Generally N-type layer has smaller thickness than P-type layer. N-type layer is exposed to sun light by providing front contacts.

When sunlight incident on solar cell it may reflect, absorb or pass through. Direct conversion of light in to electricity is called as photovoltaics. Some materials exhibit a property known as the photoelectric effect that causes them to absorb photons of light and release electrons. When enough electrons are absorbed by negative layer of solar cell, electron becomes free from it. These electrons migrate towards positive layer creating voltage difference. Power produced by each cell depends upon material of solar cell, exposed area and method of fabrication etc. If multiple solar cells are used then it can fulfill desired voltage and current need.

Different solar cell technologies

Thick film Solar Cell: This technology covers major part of solar cell industry (85% to 90%). This technology uses different form silicon varying its purity. Efficiency and cost of solar cell increases as purity of silicon increases. It can use single crystal, polycrystalline silicon for manufacturing of solar cell.

- **Mono crystalline silicon**: This cell has highest practically achieved efficiency. It has better stability over long period of time. It can perform better under lowlight condition. It requires costly pure silicon.

- **Polycrystalline silicon**: It does not require pure silicon, it leads to lower cost. This cell is less stable than Mono crystalline silicon. It has lower cost.

1.2 Thin film solar technology:

Thin film technology is becoming popular as it is cheap. Thin film solar cell requires less amount of absorber material. Efficiency of thin film solar cell is around 10% to 12 %.There is huge scope to enhance efficiency of thin film solar cell to get efficient solar cell at lower cost. Flexible solar cell are developed by using thin layer of absorbing material. Research is going worldwide to enhance performance of thin film solar cell. Most popular material used for thin film technology are

- Amorphous Silicon(a-Si)
- Cadmium Telluride(CdTe)
- Copper Indium Gallium Selenide(CIS/CIGS)

There are some innovative technologies like organic solar cell, flexible solar cell, and ultra-high efficiency solar cell etc. gaining popularity for efficiency enhancement.

Flexible Solar Cell

Many time there is requirement of flexible solar cell to trap solar energy from curved surfaces like roof top of car. Flexible substrate like plastics has lower weight. Goal of cost reduction in cost is can be achieved by developing thin film solar cell using amorphous silicon as absorber material. Due to light weight and high specific power, flexible solar cell developed on plastic films is used to give power for satellite during its operation.

Light Loss in Flexible Thin Film Solar Cell: Optical loss is significant in thin film solar cell. Thickness of absorbing material is small so it cannot absorb all wavelength light incidents on it. Generally used material like silicon does not absorb large portion of light spectrum. Reflection loss is important in case of thin film solar cell as it can take place from front as well as rear side of the cell.

Thin film cell has small thickness of absorber material so long wavelength light can penetrate up to substrate and can reflect back. This loss can be reduced by various optical management techniques. Texturing is an effective technique which creates specific type of roughness pattern on surface in order to alter properties [2]. Texturing can also improve light trapping in thin film solar cell by improving scattering.

1.2 Need of texturing in Flexible solar cell

Texturing is a process of applying specific type of surface pattern useful for various applications. Type of pattern produced by texturing, its distribution etc. affects behavior of textured surface. Requirement of specific application may be fulfilled by dimple, groves, nanostructure or by random texturing.

Light loss is significant in thin film flexible solar cells. Texturing of substrate alter surface morphology which is helpful for light scattering. Thickness of thin film solar cell is small so texturing of substrate (substance on which cell is developed) is feasible option to enhance light absorption by active layer. Long wavelength light can easily reach to substrate; its trapping is possible when it travel multiple times inside solar cell. Back reflector deposited on textured substrate will help for light trapping in long wavelength radiation. Laser has capabilities of altering surface roughness of many materials. Laser texturing of polyimide can fulfill need of light trapping in case of flexible solar cell. Now days thin film solar cell developed on flexible films is showing new direction to the existing technology. The general cross-section of the flexible solar cells produced by Solarion is shown in figure 1.1

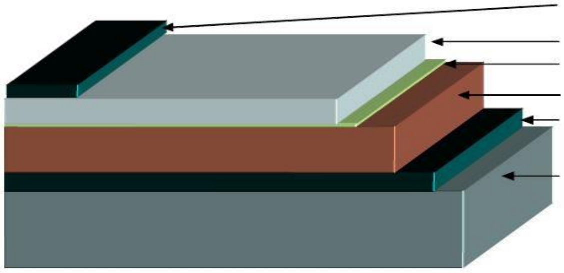


Figure 1.1 Schematic drawing of the flexible thin film solar cell

1.4 Organization of thesis

Chapter 1: This chapter explains importance of solar energy in human life. After brief information of working solar cell, major classification of solar cell technologies, need of texturing to reduce optical loss in case of thin film solar cell is explained.

Chapter 2: Presents importance of texturing for light trapping in case of thin film solar cell based on past work performed by many researchers. Research objective is obtainable from identified research gaps.

Chapter 3: This chapter explains detail experimental procedure followed during texturing of polyimide with Nd: YAG laser. Details of experimental setup and experimental parameters also presented.

Chapter 4: Investigation of laser textured polyimide with Microscopic images, Scanning Electron Microscope (SEM), Attenuated Total Reflection (ATR), X-ray Diffraction (XRD) and Ultraviolet and Visible Spectroscopy (UV-VIS) are presented with elaboration.

Chapter 5: Summarizes and concludes the thesis. Possible directions of future researches are also discussed.

Chapter 2 LITERATURE REVIEW

Conventional solar cell is stiff, heavy and brittle (in case of glass substrate) so it obstructs use of solar cell innovatively. Flexible solar cell can be used in attractive way's to get energy. Flexible solar cell is developed on flexible substrate (polymers or metal foil) [24]. It can be stick on uniform of soldier to make energy available for his /her energy even at remote places. Energy need of portable electronic gadgets can fulfill by same method. Due to light weight, flexibility it is preferred for terrestrial application. If thin film solar cell developed on flexible substrate it will give low cost solar cell that can be used efficiently to trap solar energy

If flexible solar cell developed on metal foil it may lead to some problems like corrosion, diffusion of impurities while deposition of cell. It get oxidized when it interact with chemicals or environment. Polymer films are attractive because of its low cost [3] Polyethylene teraphthalate (PET), polyethylene Naphthalate (PEN) Polyimide (PI) [4] [5] [6] are more interested because of their superior thermal stability, low cost etc. Many polymer performances may degrade with repetitive thermal load, exposure to different types of radiation, high vacuum etc. Many of these conditions are similar to space condition. Polymers can be classified in to elastomers, plastics and fibers. Plastic has only partially reversible deformability. Plastics can be further subdivided in to thermoplastics (whose deformation at elevated temperature is reversible) and thermosets (which undergo irreversible change when heated) [7]. Polyimide has attractive physical and chemical properties. Detail properties of polyimide are given in upcoming section.

2.1 Importance of thin film technology for flexible solar cell

Thin film technology is suitable for flexible solar cell due to following reasons

- Different types of chemical, physical methods are available for deposition. [6]
- Large area substrate can be deposited.[6]
- Optical and electrical properties of solar cell can be controlled[6].
- All deposition process can be integrated easily [6]
- It is mature technology and reliable with proven performance [9].
- It can be applied for innovative concepts like plasmonic solar cell[9].
- Light trapping can be controlled by controlling optical properties of cell or substrate.
- Flexible substrate gives freedom to roll to roll deposition technique giving enhanced speed of deposition which reduces cost of deposition.
- It requires less absorber material which leads to reduce cost of solar cell reducing pay-back period.
- Thin film technology is a green technology.

2.2 Different Light trapping methods

To enhance light absorption different approaches are followed by researcher out of which some methods are as follows.

- Dispersion of nano particles (Ag) on surface of solar cell. Surface Plasmon's resonance increase absorption of light for specific wavelength and angle of incidence. When nanoparticle present on surface it can absorb light of longer wavelength but reduces light absorption below resonance due to interference[4]

- Dispersion of different material's nanoparticle on solar cell then each material has different absorbing region of spectrum. This leads to absorb combine region of spectrum [4].
- If nanoparticles are present on rear side of thin absorber layer it can improve light absorption by resonance. It does not have adverse interference for longer wavelength. We can target different thin absorber layer with different absorber layer [4].
- Texturing of solar cell surface or substrate with periodic or random, enhance light trapping by modifying light path.
- Back reflector layer can be applied on substrate after or before texturing to reflect back long wavelength light.

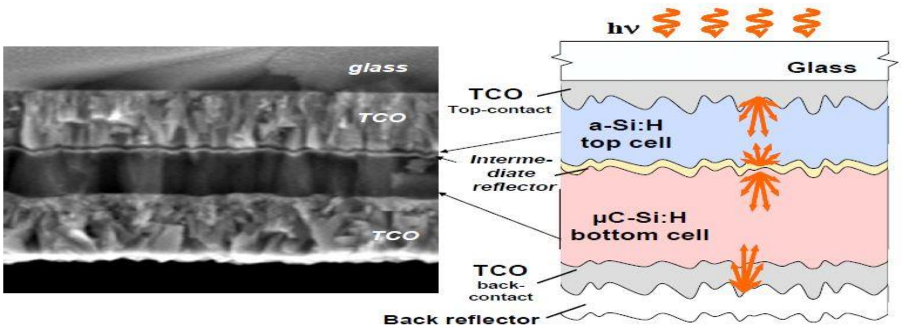


Figure 2.1 Left Scanning Electron Microscope image of the cross section of a micrograph solar cell in the p-i-n configuration with rough Zinc Oxide Transparent Conductive Oxide and intermediate reflector. Right: Corresponding schematic cross section of a micrograph [10]

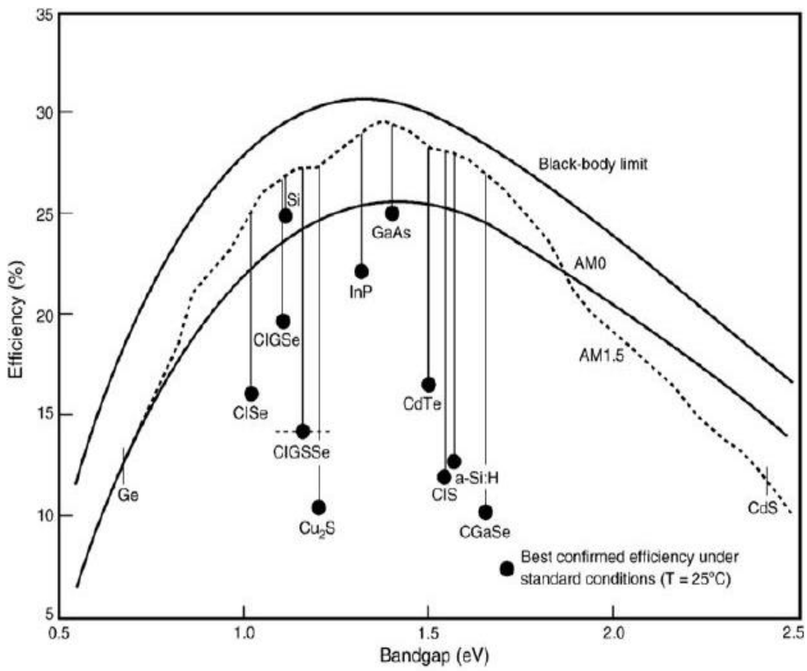


Figure 2.2 Maximum theoretical efficiencies dependence of the band gap energy for several solar cell technologies [12]

- Small antireflection layer on top of surface reduced reflection loss. This layer work efficiently only for normally incident light on it. For other angle of incident its performance degrades when light incident at other angle than 90˚ [11].
- We can engineer different material for better absorption of light [12].
- Texturing of front surface of solar cell enhance light trapping [10]
- Texturing of substrate increase light trapping from rear side of solar cell. Scattering is a phenomenon which increase light path due to multiple reflection [13].
- Texturing may deteriorate some electrical, physical, chemical properties of substrate. Trade off should be done between optical enhancement and other properties. Physically Flat substrate is better for deposition of cell and rough for enhancing light absorption. Solution for this problem was proposed by soderston et al. Substrate can be made optically rough by texturing then it is filled with material (filler)having higher refractive index. Substrate then polished to make

physically smooth as shown in Figure 2.3. Proper selection of filler material is important here to get desired result. [13]

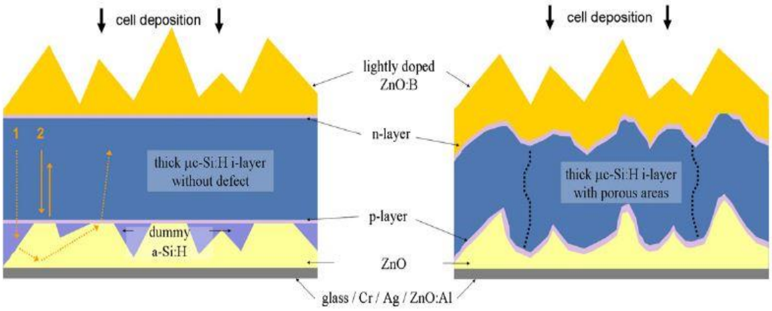


Figure 2.3 (Left) Solar cell deposited on optically rough and physically flat substrate (right) Solar cell deposited on textured substrate. [13]

2.3: Requirement of substrate properties for thin film solar cell development [14] [15]

Substrate properties are important while selecting solar cell deposition method. It takes decisive role in stability and quality of solar cell.

Flexibility: Flexible solar cells can be developed on flexible substrate. Roll to roll deposition is possible in flexible substrate.

Stability in vacuum: While deposition or during space operation substrate may expose to vacuum environment, it should not deteriorate in such condition.

Thermal stability: Substrate temperature may increase in many deposition processes. Low temperature deposition process processes are preferred for polymer as it has comparatively lower stability than metal foil or glass. Selection of deposition process mainly depends upon of substrate.

Coefficient of thermal expansion: Coefficient of thermal expansion (CTE) should be has value nearly equal to that of material of solar cell. If CTE of substrate and substrate has larger difference then it will develop stress at interface of substrate and solar cell when temperature changes.

Chemical stability: Substrate should not react with solar cell material during processing. It should not diffuse impurities in to solar cell otherwise it may hamper performance.

Environmental Stability: Substrate properties should not alter during actual operation. Adverse environmental conditions like moisture, heavy wind, long wavelength irradiation should not affect performance of substrate.

Surface properties: Substrate surface should be free from impurities. It should be optically rough and physically smooth.

Cost: If cost of substrate should be low to reduce cost of final product, hence increase acceptance of solar cell by large number of peoples.

Sustained mechanical properties: Sufficient mechanical strength is needed for substrate. Mechanical properties should sustain during when it expose to ultra violet (UV) radiation, atomic oxygen, thermal cycling etc. Mechanical properties should be stable irrespective of cell deposition method.

Adhesion of substrate with solar cell material: Adhesion of solar cell with substrate material should good. While bending of solar cell as it induces stress at interface of solar cell and substrate.

2.3.1 Texturing

Surface texturing is process of applying specific type of pattern of roughness on to the surface in order to change properties. Texturing may produces groves, dimples, channels, conical structure as per requirement. It has numerous application in field of tribology, micro fluidics etc. [11]

Physical texturing is deciding factor for accepting biological system and its response. In tribology it can improve performance by reducing friction. Short pockets produced by texturing act as collector of debris and lubricating oil. It avoids abrasive wear [16]

Textured surface can be used for aesthetic. In magnetic drives it was used to overcome friction problem. Texturing amend adhesion of mating surface or adhesion of coating. Texturing can be used to increase grip as in case of pen, micrometer. Texturing has application in self-cleaning surfaces, micro fluidics also. Texturing can be implemented in sensor and optical system to enhance light absorption.

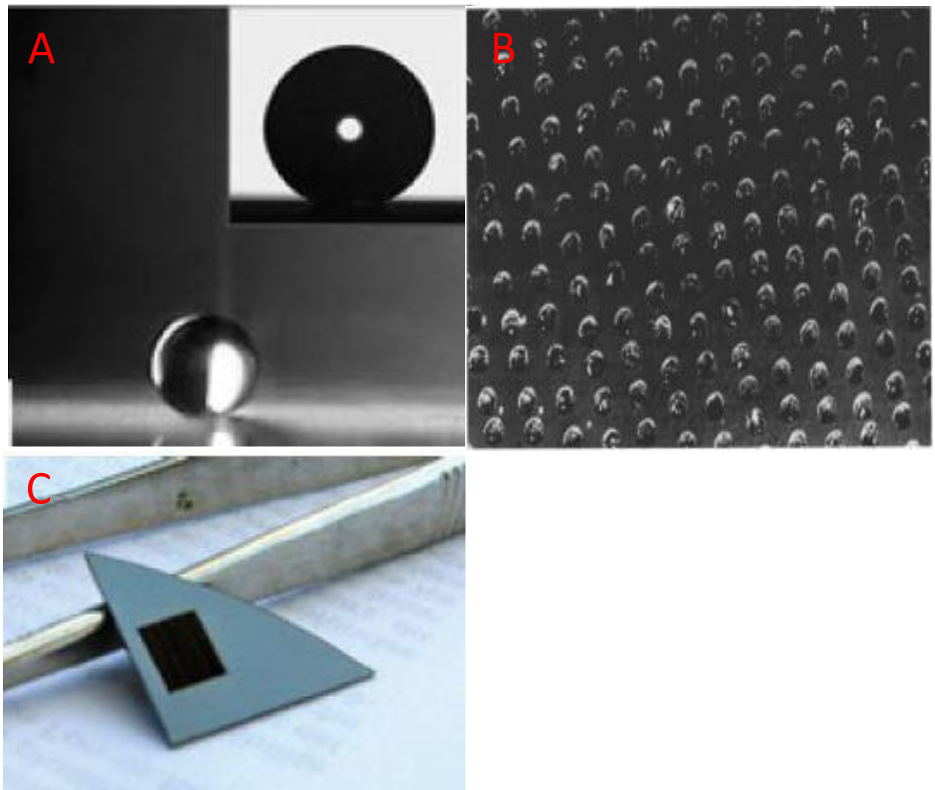


Figure 2.4.Use of texturing in various fields (A)Super Hydrophobic surface (B)Textured surface used in tribology to reduce friction (C)Textured square area appears black comparative to unprocessed region as it absorb nearly entire light incident on it. [11]

2.4Methods of Texturing:

Texturing can be performed by various methods like Chemical etching, Sand blasting, Laser processing or by ion etching. Chemical etching is simplest way to etch different materials. Chemical etching requires proper selection of etchant for specific material. This technique requires more control on environment condition such as temperature and time of etching. To get desired effect by optimizing parameter is difficult task. This process can produce random as well as periodic texture. Periodic texturing is possible only in case of isotropic etching. This type does not have precise control on shape, distribution of textured surface [11]

Lithographic technique has high control on textured features. This method is capable of producing repetitive results. It can be applied on different types of material. It is capable to generate large variety structure. This technique is costly so cannot be applied for mass scale production [17] [18]

Mechanical texturing method involves Blasting and rolling. Blasting uses hard ball to be strike on surface to be textured at high speed. Due to impact it produces dimple on surface to be textured. In rolling roller has exactly apposite shape that required on surface. Rolling can produce repetitive result rapidly. Mechanical method is contact method this may induce residual stress. These methods are not capable of producing nano scale textured features. Mechanical methods may damage workpiece by producing crack on workpiece which will act as stress concentrator during actual use. Texturing brittle material with mechanical method is difficult task [11] Methods like ion etching can be used for texturing. This method is time consuming and costly. It requires sophisticated setup so it is costly.

2.5 Importance of laser source for texturing:

Laser can process metal, semiconductor and polymer. As laser can concentrate energy on very small region it can process high melting point material. Laser processing method has great control on area to be processed. Laser processing can perform on selective area without disturbing remaining part. Laser processing is contact less method which gives more flexibility during processing. Various laser parameters such as wavelength, flounce, raster speed, overlap, processing environment can be varied to get different type of surface features. Laser is capable of producing texture ranging from nano level to macro level. Laser can be guided by optics which gives more freedom to establish experimental setup. Laser processing can be completely automated with process in case of solar cell development. Already solar cell industry is using laser for various process like cutting, joining, soldering etc. as shown in figure 2.5 due to its advantages. It is advantageous to use laser for solar cell for texturing due to above reasons. [12]

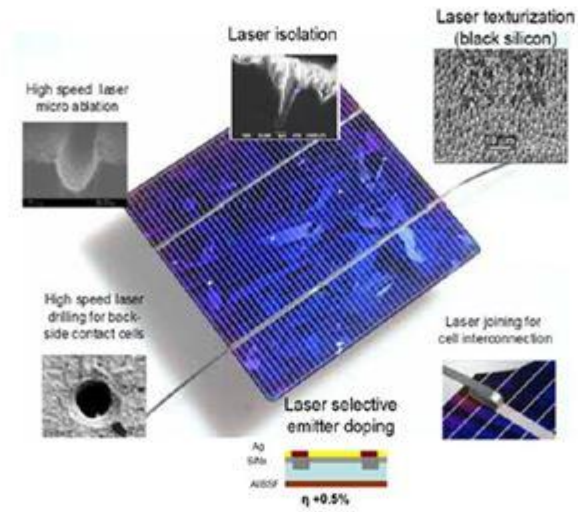


Figure 2.5 Laser application for solar cell and solar cell module fabrication

Various types of laser behave differently with same material. Following different laser phenomenon's can be used as per desired features.

- Laser induced periodic surface structure (LIPSS)
- Interference
- Ablation, Melting and evaporation

Laser produce textured surface without removing material or by removing material from workpiece. During processing if there is not sufficient fluence then it can texture without removing material.

Many researcher preferred pulsed laser source over continuous for texturing of solar cell having motive of enhanced light absorption. [19][12][20] [17]. Heating rate during interaction of pulsed laser with material is high which is capable of producing significant changes in processing material [11].Ablation is dominant phenomenon in pulsed laser. Laser pulse has important role in dynamics of ablation process. As pulse length shortened energy is given more rapidly to work piece material removing material more rapidly [22] Pulsed laser reduces thermally induced stress on workpiece [21]. Irradiation of intense shorter laser pulse in selective gaseous environment leads to dramatic change in morphology as well as optical properties of workpiece [19]. Effect of different environmental condition on absorbance of femto second micro structured silicon is shown in Figure 2.6

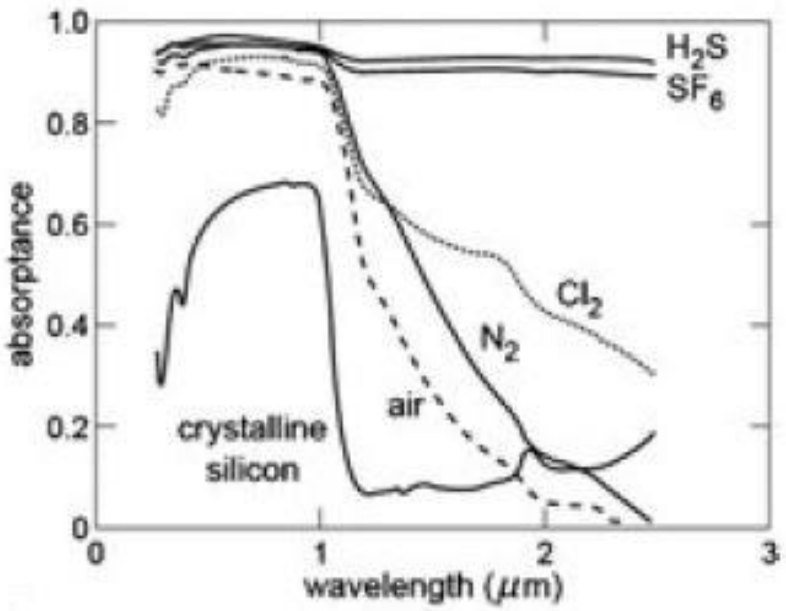


Figure 2.6. Dependence of absorption on different gaseous environment [11]

Laser is capable of producing changes in structure, composition, optical and mechanical properties of material.

2.5.1 Difficulties to use laser for texturing **[11] [23]**

Laser interaction with material is complex because many phenomenon's may occur simultaneously. Laser interaction with each material is unique so background research should be available for specific material and specific laser combination. Laser interaction with material depends upon following things

- **Optical Interaction:** Material may absorb, transmit or reflect light depending upon light and material properties. In case of material which has more transmittance multi-photon absorption takes place [23]
- **Thermal Interaction:** Different phenomenon like conduction, convection and radiation are important for deciding nature of texturing, heat affected zone (HAZ) and material removal rate.
- **Physical Phenomenon:** Depending upon experimental parameters and of material properties, processing material may undergo melting, ablation or vaporization when laser irradiated on it.

- **Experimental parameters:** Type of laser, fluence, wavelength, pulse width, percentage of overlap, raster speed, no of pulse etc. are some major influencing parameter. Selecting right experimental parameter to obtaining specific texture needed for specific application requires highly skilled person. Obtaining optimized parameter require large experimentation.
- **Processing environment:** Processing environment has influence on nature, performance characteristic of texture. Variation of nature of gas and pressure of gas led to change composition, shape, and distribution of texturing. Refractive index of environment, thermal conductivity, and heat transfer coefficient of surrounding environment are important deciding properties of processing environment.
- **Properties of workpiece material:** Physical properties (thermal conductivity, heat transfer coefficient, melting point) and optical properties (refractive index, coefficient of absorption, coefficient of reflectance, coefficient of transmittance) affect interaction of laser with workpiece.

Though huge research has been performed on laser texturing there is ambiguity within researcher community regarding some phenomenon.

2.5 Use of texturing in light trapping for solar cell:

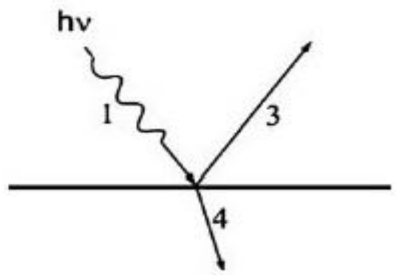


Figure 2.7 Interaction of light with planner surface

(1: Incident Light, 3: Reflected light, 4: Absorbed light)

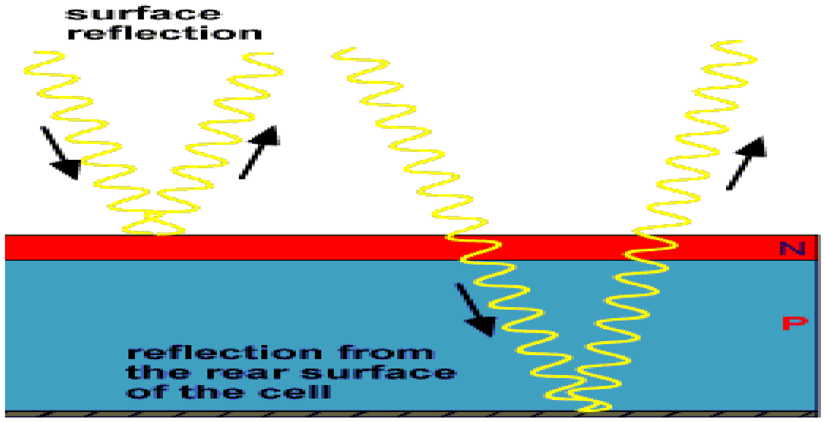


Figure 2.8 Reflection loss from thin film solar

When light incident on any surface it can be reflected, absorbed or transmitted completely or partially. These three processes occur simultaneously for many materials. In case of solar cell absorbed light is used for generation of electricity. If absorbed photon of light having energy greater than energy gap of semiconductor will generate electricity. Reflected and transmitted light is loss which depends upon material and surface properties.

Thin film solar cell has smaller absorber layer thickness due to which absorber layer is not capable of absorbing all wavelengths light that is incident on it. Crystalline silicon weakly absorbs longer wavelength light (above 1000 nm) [24].In case of µc-Si: H light above 800nm wavelength is absorb weakly [12]. Light can travel up to substrate and may be reflected back or transmitted depending upon substrate. Reflection of light may occur from top surface of thin film solar cell or top surface of substrate as shown in Figure 2.8.

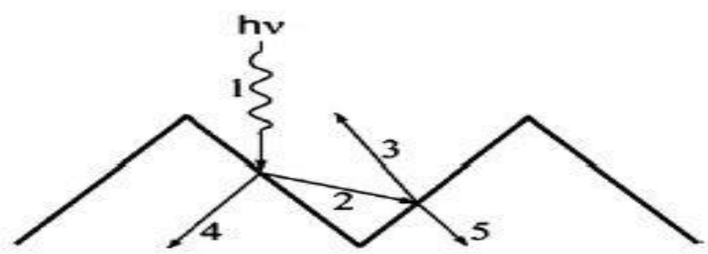


Figure 2.9 Effect of textured surface on interaction of light giving multiple reflections

(1: incident light,2 and 3: reflected light, 4and 5:absorbed light)

If texturing performed on any surface it alters its surface roughness. Texturing increases surface area. Textured surface has slope so reflected ray may incident on surface more than one time. Then it may again reflect, absorbed. Final effect of multiple reflections is increase light trapping. Degree of enhancement in light trapping depends upon geometry, dimension and density of textured feature on surface. If textured feature has very small size then light is not capable of resolving it. If sub wavelength structure is periodic then it can lead to optical response which is called as moth eye effect [11].This effect occur because medium make volumetric average of optical properties between processing material and surrounding medium [23]

Feature size	**Influence on reflectivity**
Far greater than **λ**	Light trapping due to multiple reflection enhance coupling in to the material .Light refracted at oblique angles increases effective optical path length.
Nearly equal to **λ**	Small features can successively scatter light, increasing the effective optical path length and enhancing absorption.
Lesser than **λ**	Sub-wavelength structure can reduce reflection through moth eye effect

Table 2.1 Multiple length scales over which reflectivity and absorption is determined by surface feature [11].

Above table shows texturing above and below or wavelength size dimension leads to increase light trapping. Post cleaning is required to remove debris produces during texturing. These post processing may also affect geometry and hence optical properties as shown in Figure 2.10

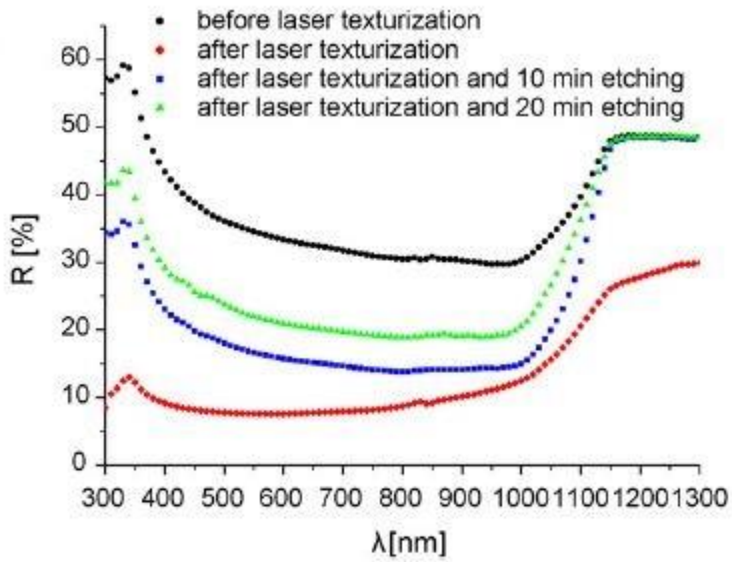


Figure 2.10 Reflection curves for wafers with texture in the form of parallel grooves

2.6 Requirements from good texturing for thin film solar cell

- Textured surface should scatter long wavelength (above 800nm) light efficiently.
- Solar cell deposition is sensitive to particle of size equal to thickness of cell. Textured surface should be clean. During texturing process may generate particles that should remove before deposition [24].
- Texturing should not have any abrupt changes as it will leads to discontinuity in cell [24]
- The question whether random or periodic photonic nanostructures lead to better light trapping in solar cells is currently hotly debated and remains controversial.

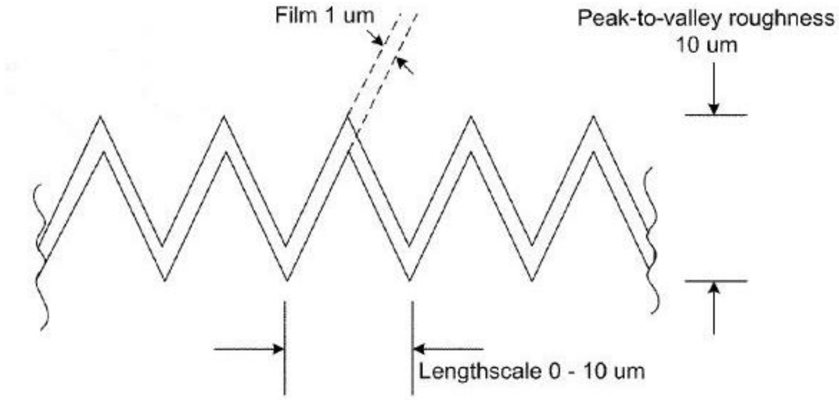


Figure 2.11 Maximum peak to height for good performance of thin film solar cell

[24]

Difficulties to use texturing for light trapping

- Obtaining exact shape, distribution of texture on surface is difficult. Variation with these may not improve performance of cell [24]
- Textured surface should be cleaned before deposition of solar cell[3].
- Laser texturing hamper some mechanical properties like strength of polyimide film as material removed from film due to ablation [24] [25]

2.7 Research Gap and Objective

Research Gap

- Previous researcher focused texturing of front side of thick solar cell.
- Very few people were concentrated on thin flexible solar cell substrate texturing.
- Little study has been performed on polyimide substrate texturing with Nd: YAG laser.

Objective of Research Work

- Obtain operating parameter in which texturing can be performed on polyimide substrate with Nd:YAG laser at 355nm,532nm and 1064 nm wavelength.
- Perform texturing on polyimide film with different percentage of overlap (10%,50% and 90%) and analysis changes occurred when percentage of overlap changes.
- Study changes in optical properties occurring as a consequence of Laser texturing.

Chapter 3 EXPERIMENTAL DETAILS

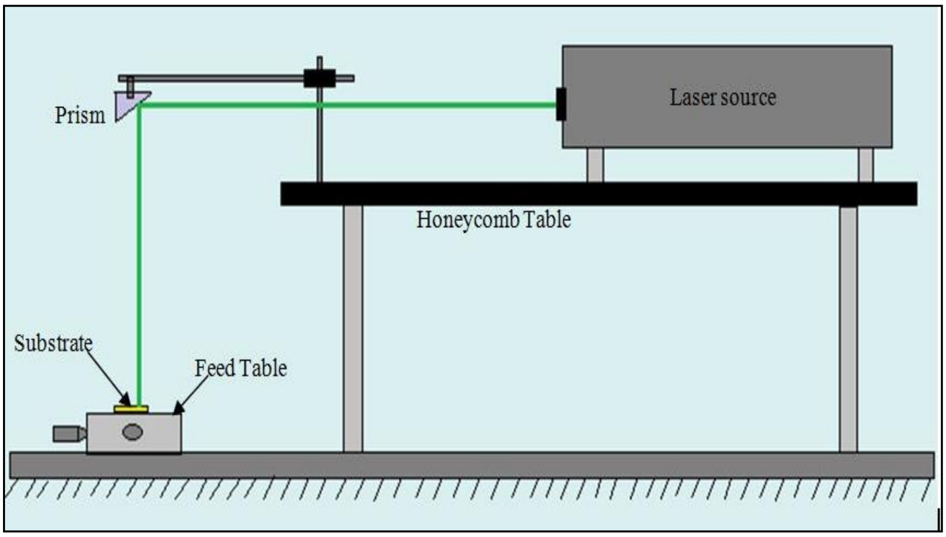


Figure 3.1 Schematic of the experimental setup of direct Laser Texturing.

3.1 Experimental Procedure

Figure 3.1 shows experimental setup used for direct texturing and texturing with overlap.Nanosecond pulsed Nd:YAG (Quanta Ray INDI) having pulse duration 5-8 ns and frequency 10Hz was used.It is capable of producing three wavelength(355nm, 532nm, 1064nm) usind KDP crystal.Laser beam has gaussian beam profile.It also can produce single shots.Laser beam was incident on prisam which bend it in 90 ˚ then it will incident on polyimide.Distance of prisam from head of laser was 80 cm and substrate was placed at 65cm from prisam.Angle of incident on polyimide is 90˚. Polyimide substrate was placed on feed table by using doubble sided sticky tape.Feed table can be move in X and Y direction by using micrometer having resolution of 10µm.

Laser diameter was calculated by allowing laser to fall on photographic sheet for sufficient time so that visible color change occure.This photographic sheet then observed under USB microscope. Laser spot diameter was found to be 5.4 mm at the place of substrate. Energy of laser was measured by keeping power meter (Newportc842-PE) at place of substrate for two minutes to obtain stedy state reading.All experimental were performed at room temperature(25˚C) in presence of air.Kapton HN polyimide film of thickness 25µm was used as substrate.

Table 3.1 Properties of Polyimide

Thermal Property	Typical Value
Thermal Coefficient of Linear Expansion	20 ppm/°C
Coefficient of Thermal Conductivity(W/mK)	0.12
Specific Heat, J/gK	1.09

Properties of polyimide film can be affected by moister. Polyimide was cleaned befor experiment ultrasonicaly by using Isopropiel Alchohol for 5 minutes.Then kept in clean room for drying.

3.2 Direct texturing

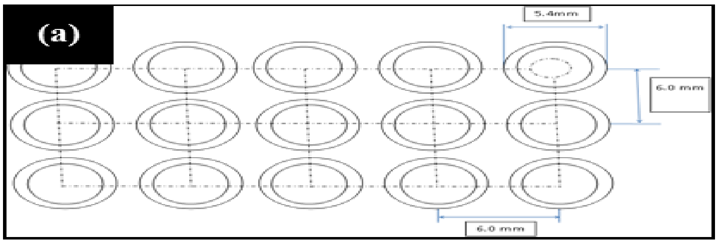


Figure 3.2 Laser Spot Pattern on Flexible Substrate matrix layout

Laser was incident upon substrate for given operating conditions.Required feed was given by micrometer when laser was not irradiating polyimide.In X and Y direction 6 mm feed was given so that laser should not irradiate same area more than once.15 spots were obtained for same parameter on same substrate.Layout of these spot is shown in Figure 3.2

Table 3.2 Experimental Parameters levels for Direct Texturing

Wavelength(nm)	Fluence(mJ/cm^2)	Time(sec)
355,532,1064	261.2,393,524	15,30,60

3.3Texturing with overlap of Laser spot:

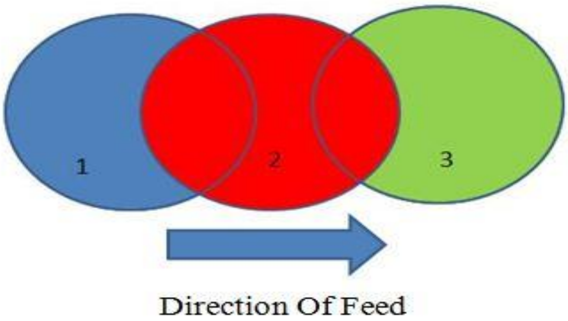


Figure 3.3 Steps followed during Texturing with overlap

During texturing with overlap sample has given feed lesser than diameter of laser spot. Table 3.3 show feed given to have required percentage overlap.Figure 3.3shows steps carried out during texturing.In Figure 3.3 region 1 shows spot which is already iradiated by laser.Region 3 will be irradiated by laser after irradiation of region 2 complits.Experimental parameters are shown in Table3.4. Fluence values are selected such that interferance ripple will occure.Time of interaction was kept constant (30 sec)for all experiments

Table 3.3 Feed required for different percentage of overlap

Percentage Overlap	Feed (mm)
10	5.0
50	4.79
90	2.4

Table 3.4 Experimental Parameters for Texturing with Overlap

Sample Number	Wavelength(nm)	Overlap(percentage)	Fluence(mJ/cm^2)
1	355	10	218.3
2	355	50	218.3
3	355	90	218.3
4	355	10	393
5	355	50	393
6	355	90	393
7	532	10	567.6
8	532	50	567.6
9	532	90	567.6
10	532	10	960.6
11	532	50	960.6
12	532	90	960.6
13	1064	10	873
14	1064	50	873
15	1064	90	873
16	1064	10	1244
17	1064	50	1244
18	1064	90	1244

Chapter 4 RESULT AND DISCUSSION

4.1 Microscopic Study

As shown in Figure 4.1(a,b,c) Nd:YAG laser interact differentaly at different wavelength with polyimide substrate. When laser of 532nm and 1064nm interact with polyimide it form blakish layer of carbonious product formed during decomposition [7]. It is represented in Figure 4.1 that higher wavelength has higher tendency to form blakish layer and wrinkls. Wrinkles gerenates on substrate at wavelength 532nm and 1064nm has tendency to from wrinkels.Due to formation of wrinkles R_{max} increases.

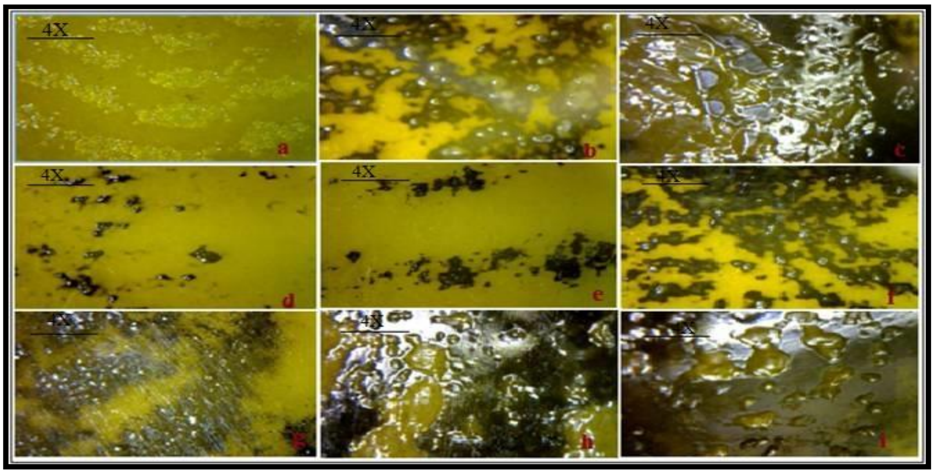


Figure 4.1Direct textured images of Polyimide samples using optical microscope

Table 4.1 Operating parameter for images in figure 4.1

Image Number	Constant Parameter	Varied Parameter
3a,3b,3c	Time =60sec Energy= 60 mJ	**Wavelength** **a**=355nm,**b**=532nm,**c**=1064nm
3d,3e,3f	Energy= 60mJ Wavelength=532nm	**Time** **d**=15sec,**e**=30sec,**f**=60sec
3g,3h,3i	Time =30sec Wavelength=1064nm	**Energy** **g**=60mJ,**h**=90 mJ,**i**=120mJ

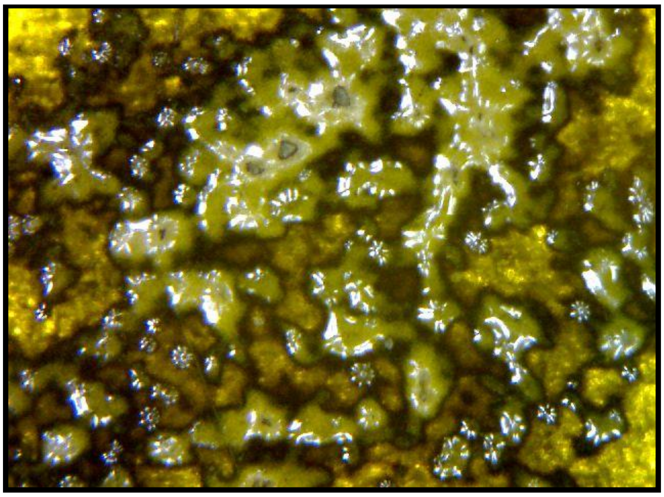


Figure 4.2 Formation of glassy layer at 1064nm at higher fluence

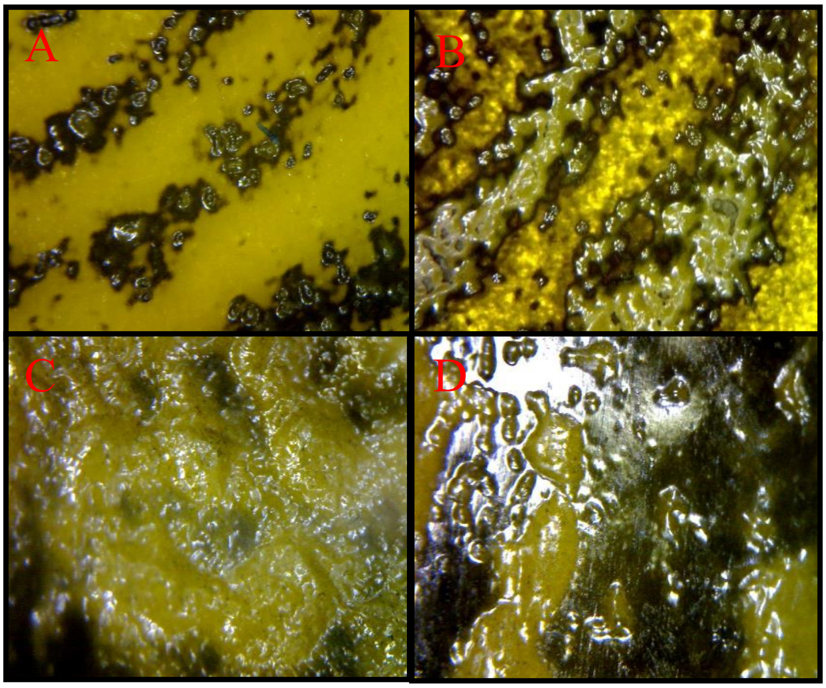


Figure 4.3 Difference between ripple ring and wrinkle produced (A and B): Interference ripple produced on polyimide surface when it is irradiated by 532nm and 1064nm respectively (C and D): Wrinkels produced on polyimide surface when it is irradiated by 532nm and 1064nm respectively.

4.1.1Wrinkles

Wrinkle are generated due to stimulation of polyimide by UV light and heat generated by laser irradiation. These wrinkles are different from laser induced periodic surface structure as shown in Figure 4.3. Wrinkles are randomly distributed in laser irradiated region. Laser is capable of providing UV light and heat. Capilary force is generated in this region. At threshold fluence and temperature wrinkles are generated. If fluence is below threshold value then after multiple pulses black color residue will be formed on polyimide surface at 532nm/1064nm wavelength. LIPSS takes place at comparitively higher fluence than fluence required for generation of wrinkles. When 355nm wavelength laser interact with polyimide, no blakish layer observed at any fluence. At 355nm wavelength tendency to form wrinkle is less.

Wrinkle surface can be used in modification of surface properties like wetting nature etc. In case of thin film solar cell substrate texturing wrinkles are not desired. Wrinkles will form discontunity during solar cell deposition. Use of 532nm or 1064nm wavelength for texturing lead to generation of carbonious product which increase absorption. Thickness of polyimide film is inversaly propotional to wrinkle formation.

4.1.2Remedy to control wrinkles generation during laser texturing with 532nm and 1064nm wavelength.

- If we use higher thickness polyimide film then wrinkle formation reduced [26].
- If we cover polyimide film with glass having more transmittance and applying pressure on glass at 532nm and 1064nm wavelength then glass can counter act with capilary forces [27]. By selecting proper wavelength wrinkle formation can be avoided. At lower fluence polyimide surface temperature is reducess which leads to reduction in wrinkle generation

4.2Thermogravimetric Anyalysis (TGA)and Differential Thermogravimetric Anyalysis(DTA)

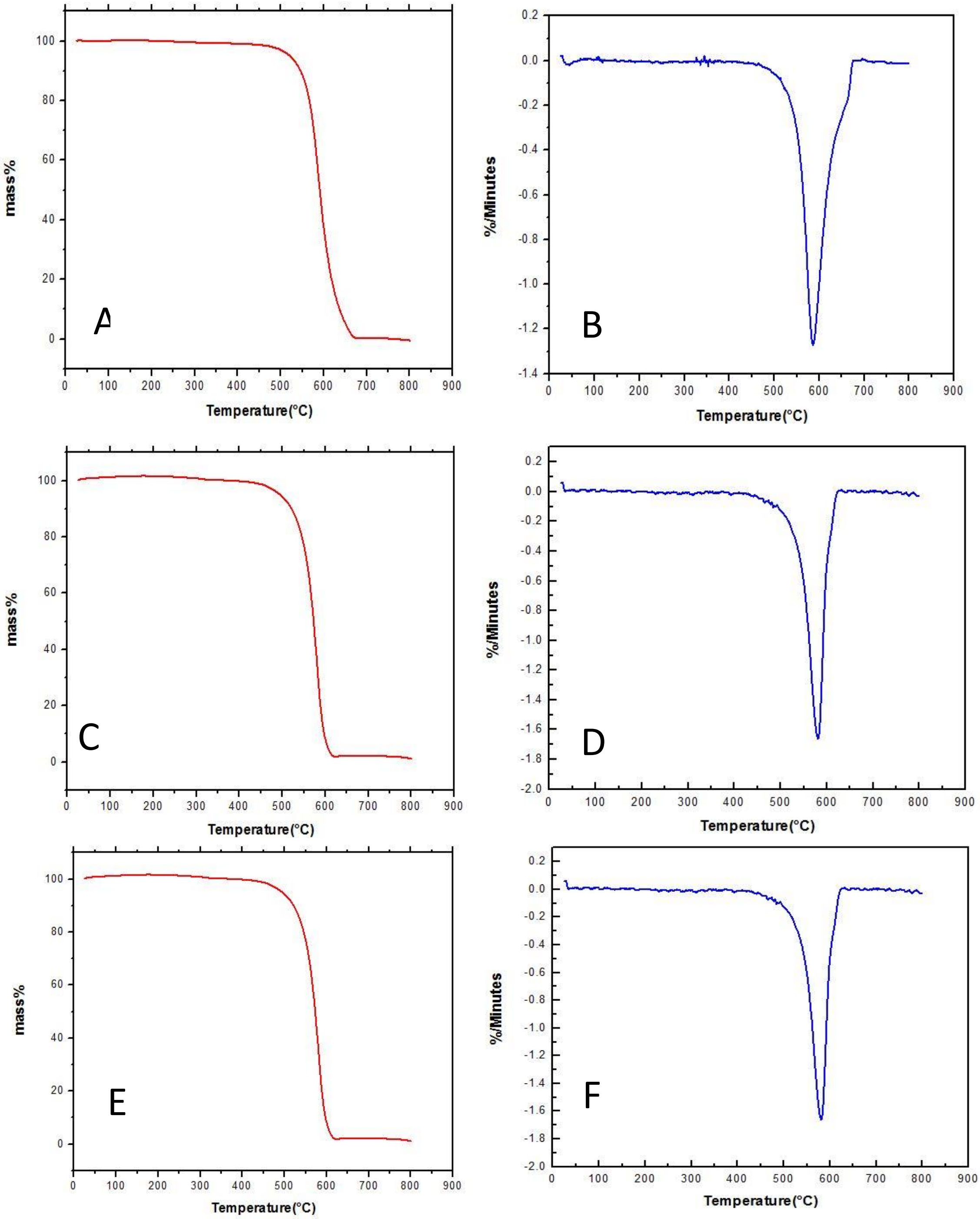


Figure 4.4 TGA and DTA results

Table 4.2 Processing parameter for TGA and DTA results

Image Name	Processing Parameter	Initial Weight of sample
A,B	Wavelength = 355nm, Overlap = 90%, Fluence = 393mJ/cm^2	4.039 mg
C,D	Wavelength = 532nm Overlap = 90% Fluence = 960.6 mJ/cm^2	2.4750 mg
E,F	Wavelength = 1064nm Overlap = 90% Fluence = 1244 mJ/cm^2	15.160 mg

TGA and DTA curves are obtained to understand behavior of polyimide film with respect to temperature. Laser irradiated portion of sample is cut and placed for investigation in nitrogen atmosphere. Heating of polyimide starts at temperature 25 ˚C and increases in a step of 10˚C/minute up to 900˚C.

TGA and DTA curve has sigmoidal and bell shape respectively as depicted in figure 4.4 and both curve obtained from same data.

$$dm/dt = \frac{M_i - M_{i-1}}{T_i - T_{i-1}} \quad (1)$$

Where, M = mass (either mg or %)

T= time in minute.

DTA graph was obtained by plotting *dm/dt* values against temperature as shown in figure 4.4 (B, D, F). As three samples have different mass, so that curves are plot by normalized mass of each sample.

$$M(\%) = \left(\frac{m_0 - m_t}{m_0}\right)100 \quad (2)$$

Where, m_0= Mass to which curve has to normalized

m_t= Mass which is to be normalized

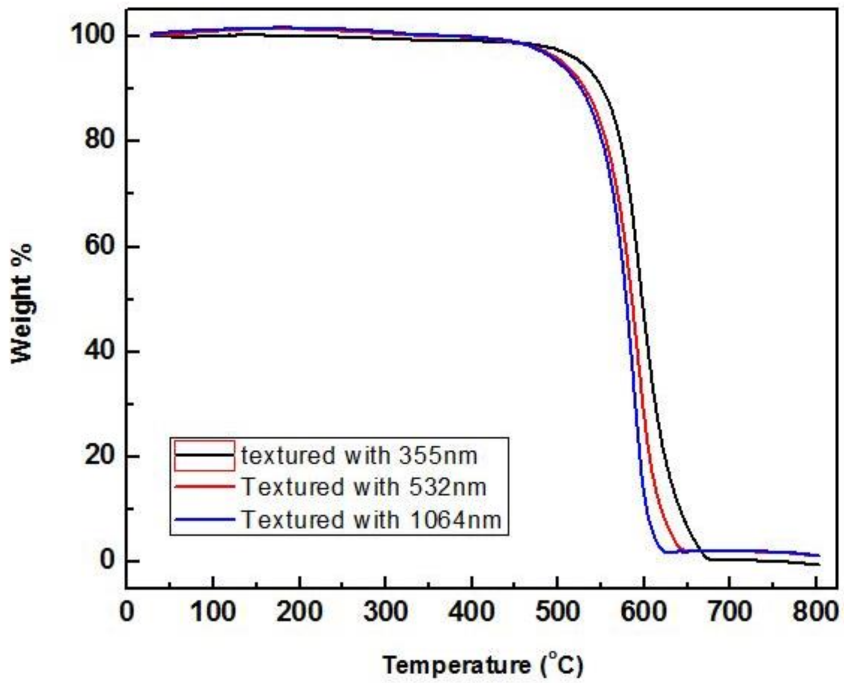


Figure 4.5 Normalized TGA results

When laser is irradiated on polyimide, heat is given by nanosecond pulse which rise temperature of it. This intense heat produces some changes in chemical and physical properties of polyimide. Due to intense heat thermal decomposition or thermal degradation of polyimide occurs. According to American society for testing of material thermal decomposition can be define as "process of excessive chemical species change cause by heat". It is a process where action of heat or elevated temperature of material, causes loss of physical, mechanical or electrical properties". Polyimide is thermostat, it does not show glass transition temperature [7]

Zones of TGA curve.

Lower temperature zone (T<400˚C): In this zone small mass reduction takes place due to evaporation of volatile surface impurities and moisture.

Temperature between 400˚C to 500˚C: In this region loss of additives having low molecular weight compound take place.

Temperature between 500˚C to 700˚C: In this region decomposition of polyimide take place with maximum rate for all investigated samples. Maximum decomposition rate can be clearly seen in DTA curve peek as depicted in figure 4.4

Temperature above 700˚C: In this region no major change was observed in TGA and DTA curve. Residue of carbonaceous product generated during decomposition and stable nonvolatile by product remain stable irrespective of temperature in this region [7]

4.3 Attenuated Total Reflectance (ATR) Study

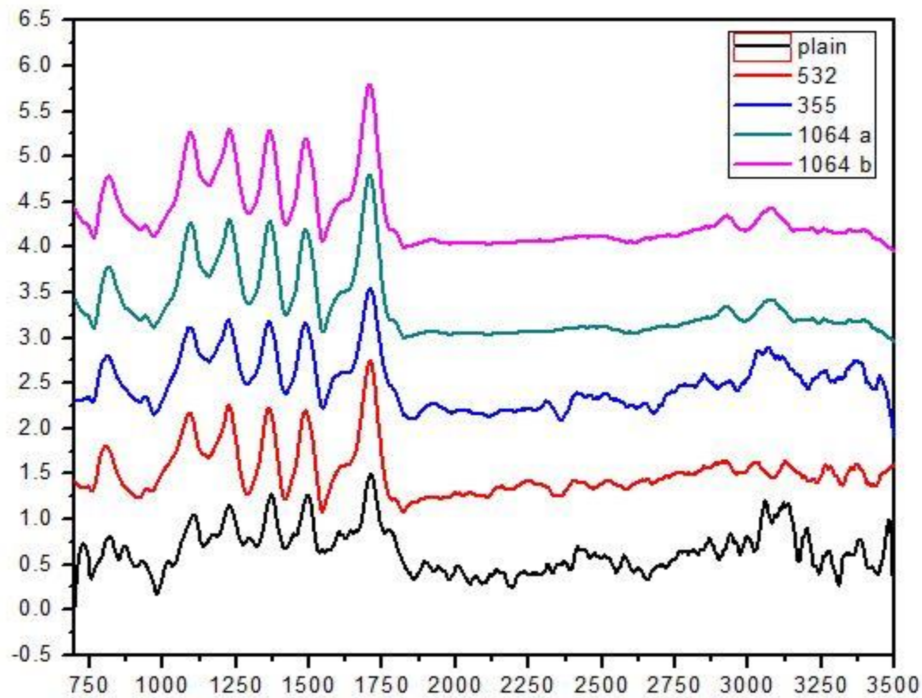


Figure 4.6 ATR –FTIR results of Sample textured with overlap

Figure 4.6 shows ATR results of polyimide textured at three samples texture with overlap of laser spot [355nm(sample no.5), 532nm(sample no.11), 1064nm(sample no.17)] as given in Table 3.4. At 1064 nm higher fluence leads to form glassy layer on polyimide. Investigate this glassy layer sample B in 1064 also tested. Peak at 1500 cm^{-1} is assigned to C=C stretching. [29]. Range of peak between 1500-1700 cm^{-1} is dealt with C=C aromatic [30].C-N stretching was observed at peaks 1367 cm^{-1} and 1230 cm^{-1}. Peak found at 1080 cm^{-1} is related with C-O [31]Wide absorption in the range 3000 $cm^{-1}$to 3200 cm^{-1} observed which is due to –OH. This absorption is strong in un-textured sample and reduced in textured samples [31] Peak at 1220 cm^{-1} shifted slightly to lesser wavenumber for laser textured sample. Peak at 1220 cm^{-1} is assigned to C-O [31]

4.4 XRD Analysis

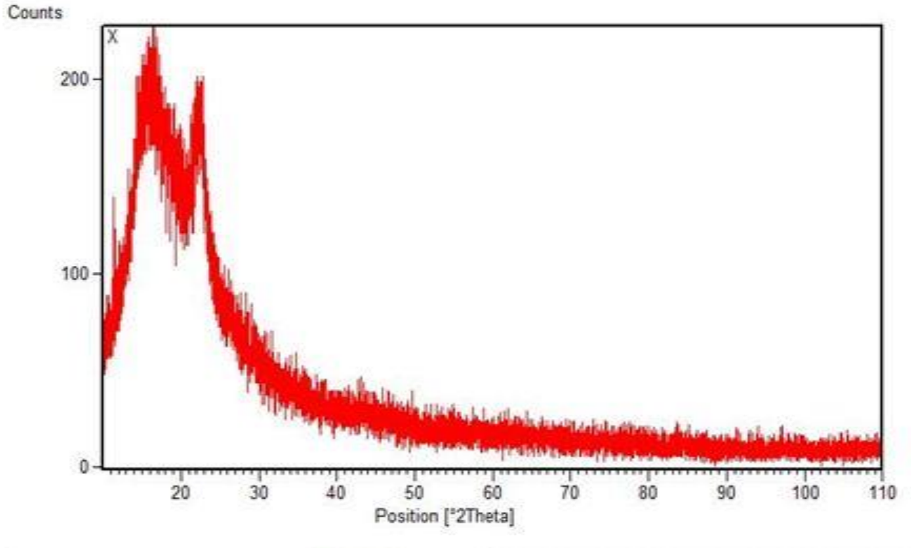


Figure: 4.7 Structural analysis by XRD of un-textured polyimidefilm

Figure: 4.7 shows XRD (Rigaku Smart Lab) of un-textured polyimide. As polyimide is amorphous material it produces a hump instead of a sharp peak. There was no distinguished peak observed. At any wavelength texturing [355nm (sample no. 4), 532nm (sample no 10) and 1064nm (sample no 16)] there is no change observed in XRD hump position.

4.5 Laser Induced Periodic Surface Structure (LIPSS)

Figure 4.8 Image of ripple ring formed during direct texturing of polyimide at (A) 355nm (B) 532nm(C) 1064nm

4.5.1 LIPSS at 355nm

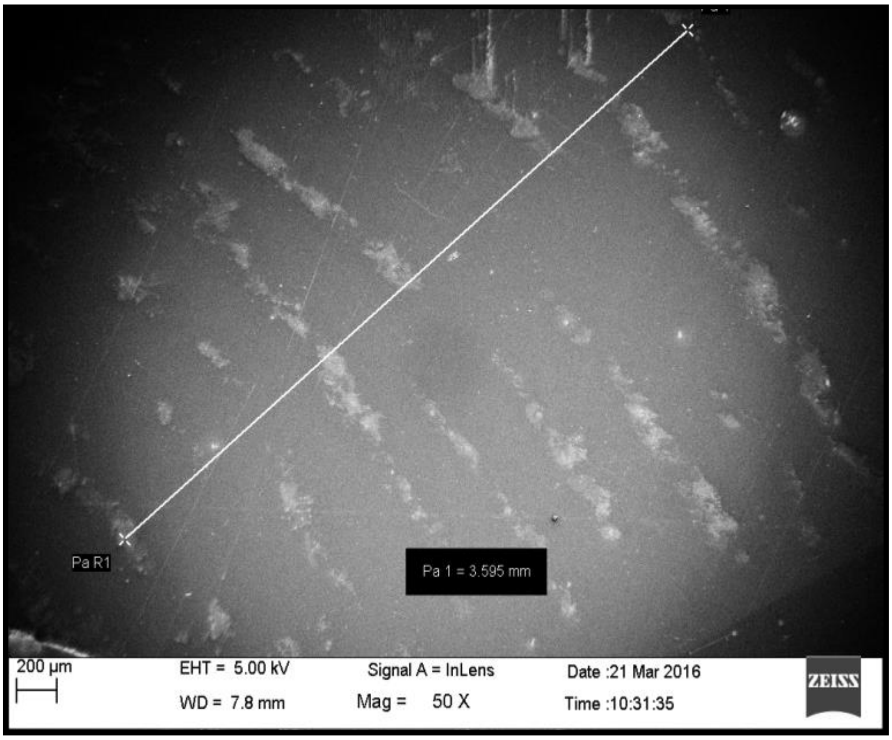


Figure 4.9 SEM image of ripple ring formed in 355 nm laser irradiated area

Figure 4.9 image shows ripple rings formed at 355nm. It is just beginning of periodic structure formation at 218.3mJ/cm^2 .Ripple are concentric having same center same as that of laser spot. There is discontinuity in ripple along periphery.

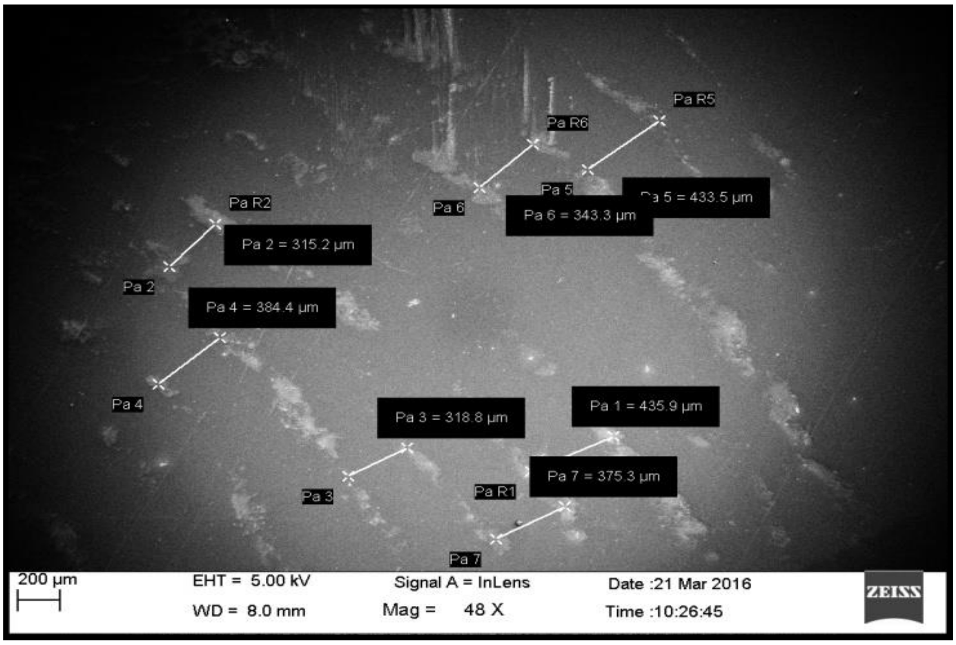


Figure 4.10 SEM image of distance between consecutive ripple rings.

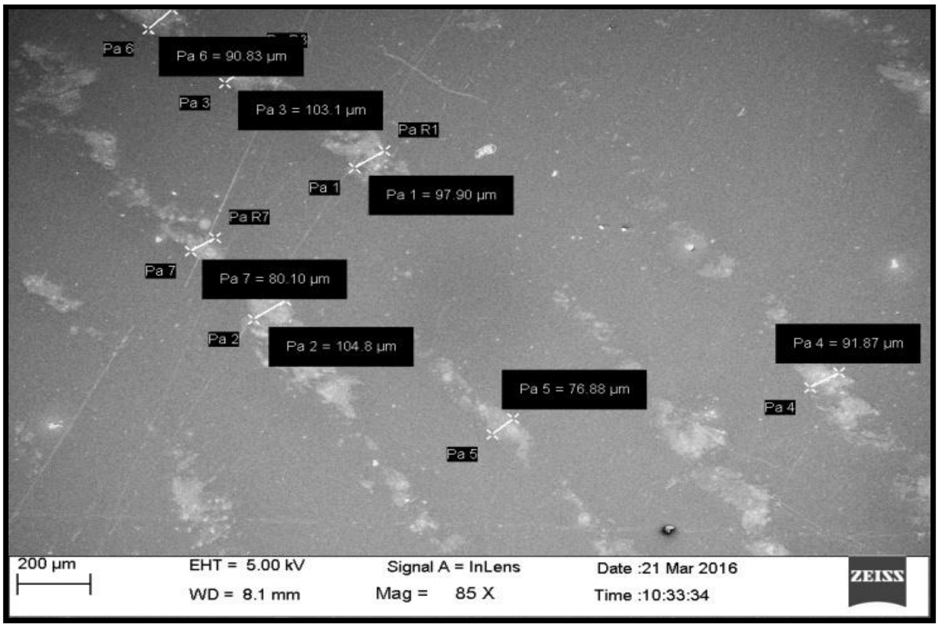


Figure 4.11 SEM image showing interference ripple width

Figure 4.11shows approximate ripple width is in range of 76.88µm to 104.8µm.It is also varying along periphery of ripple and increases as number of pulse increases.

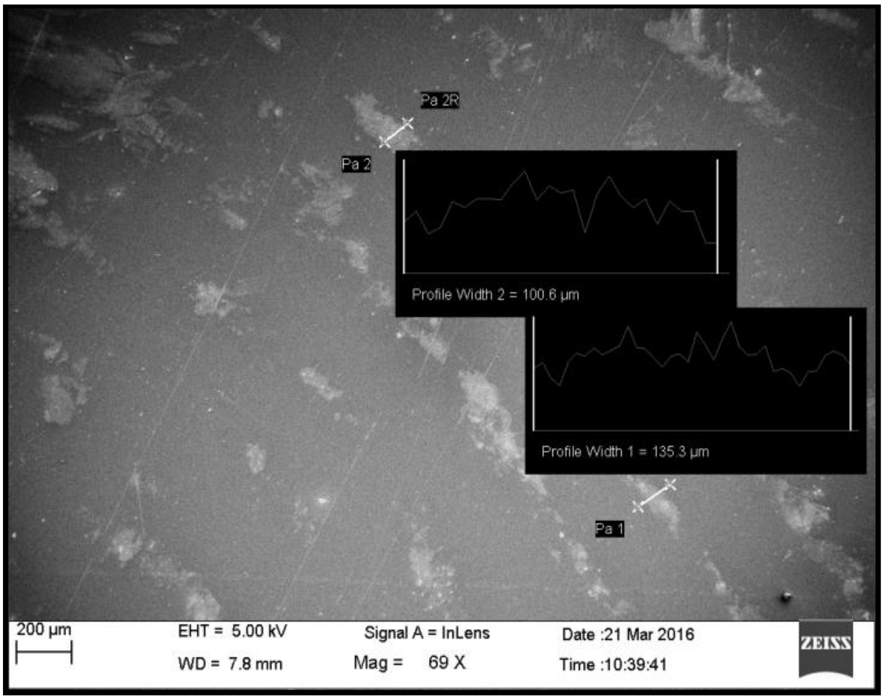


Figure 4.12 SEM image of approximate topography of ripple ring region

Figure 4.12 image shows surface profile obtained from SEM. This will give rough idea about surface. It can be seen that ring formed due to ripple structure rise above nearby area. It does not show any periodic cross section.

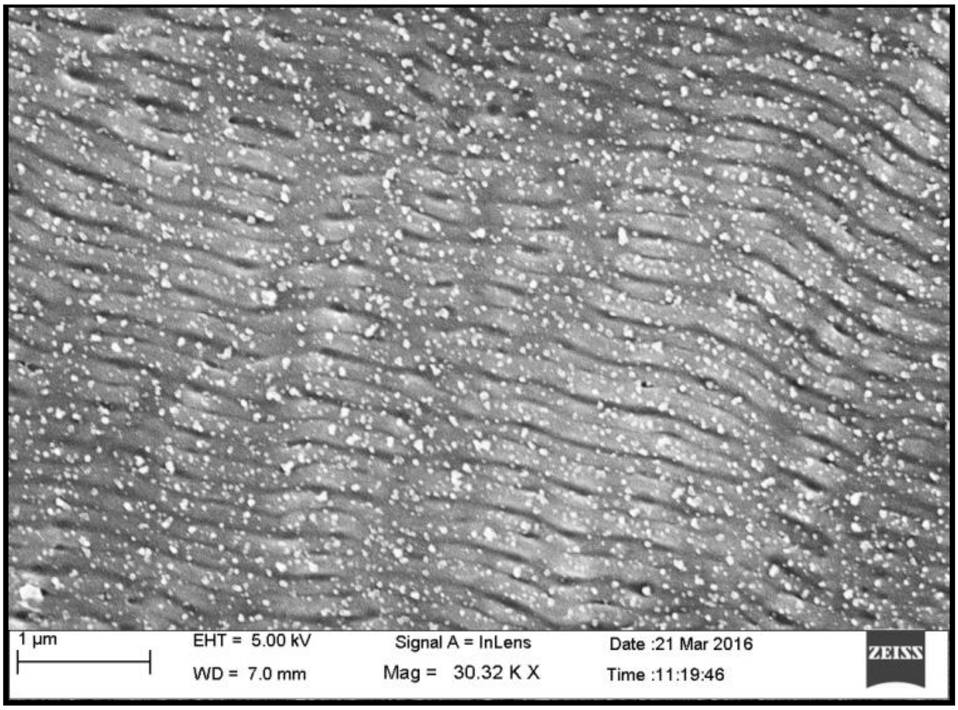


Figure 4.13SEM image of nanoparticle distributed in laser nanostructure

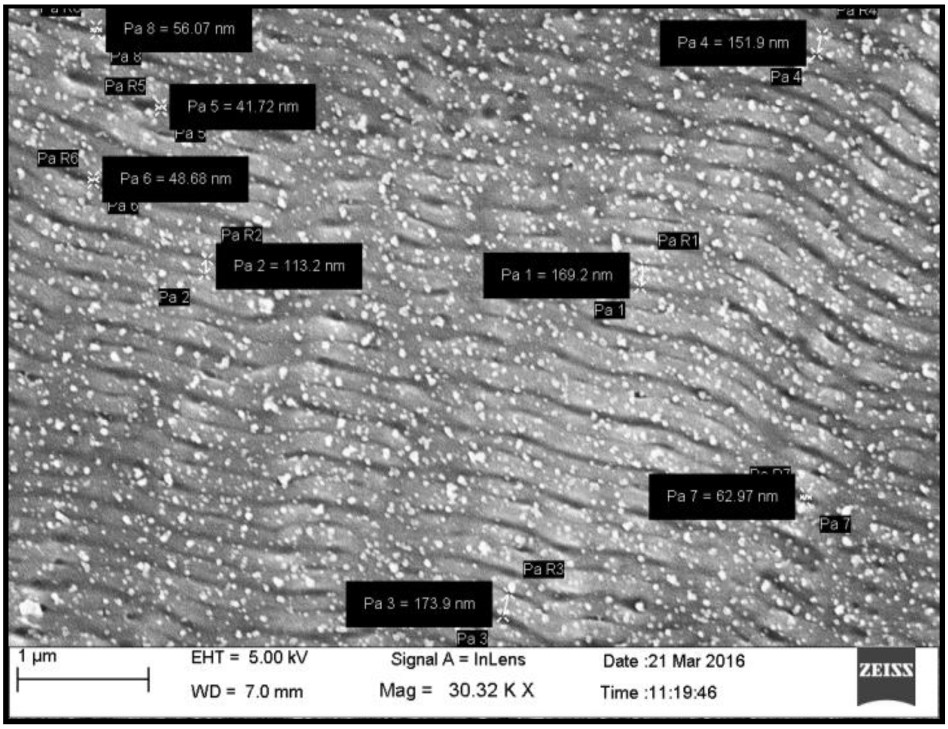


Figure 4.14 SEM image of period of nanostructure produced in ripple ring

Figure 4.13 and 4.14 shows ripple ring periodic nano structure was obtained at 218.3mJ/cm^2 at 5 pulse. This nanostructure is periodic and looks like ripple produced in water when we drop a small stone. In nanostructure region dispersed nanoparticles were observed.

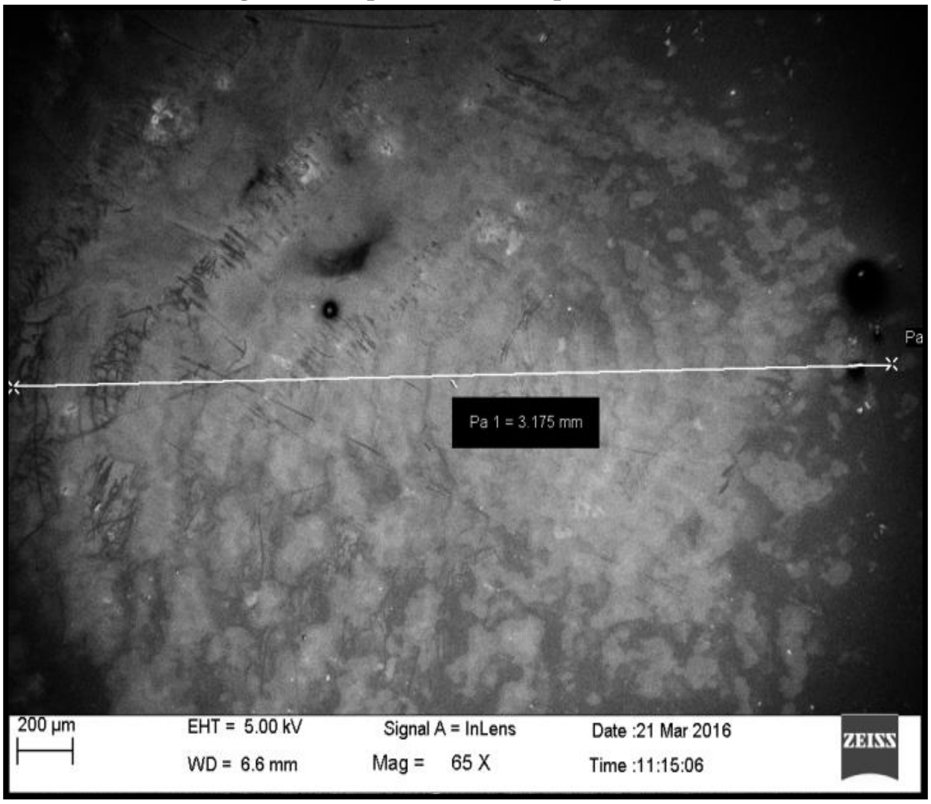


Figure 4.15 SEM image of ripple ring after 100 pulse

Figure 4.15showing ripple ring width at 218.3mJ/cm^2 and 100 pulses. This rings nearly covering entire laser irradiated area. It is observed that width of ripple ring varies along radius. As we go away from center width of ripple ring is increasing.

4.5.2 LIPSS at 1064 nm

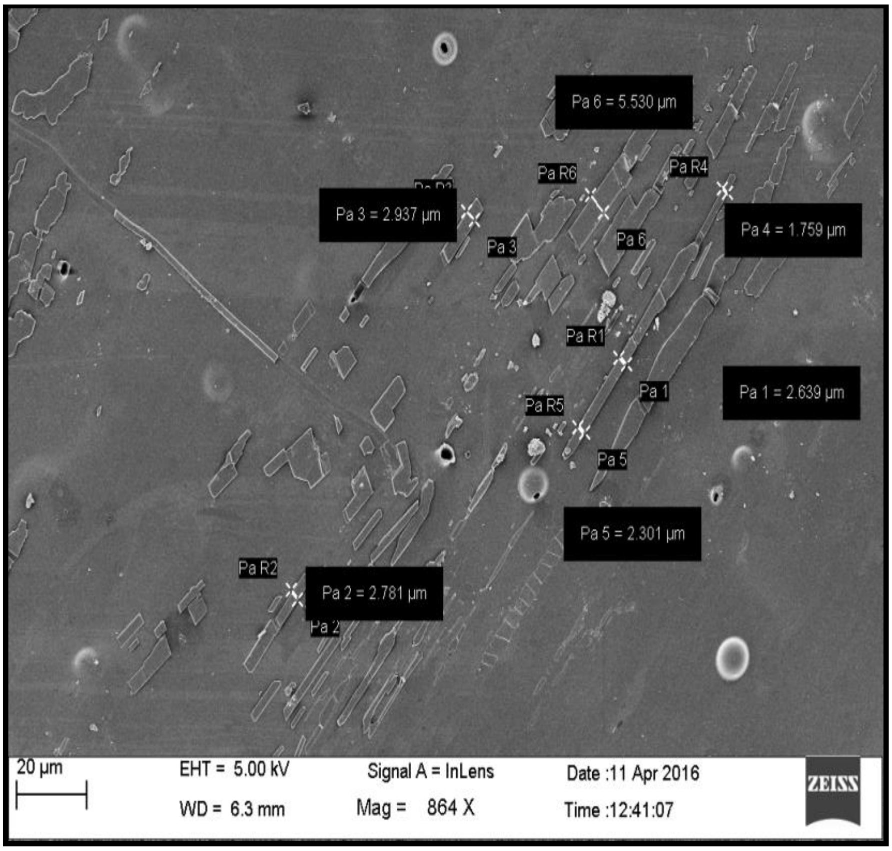


Figure 4.16 SEM image of ripple ring formed in 1064 nm laser irradiated

Figure 4.16 this is ripple ring area at 1064nm for 873mJ/cm^2 interacts with polyimide for 100 pulses. It has nearly rectangular shape having longer side parallel to perimeter of ripple ring. Randomly distributed holes were observed in laser irradiated area. These rectangular projections are not equidistant. Width of projection is around 3µm.

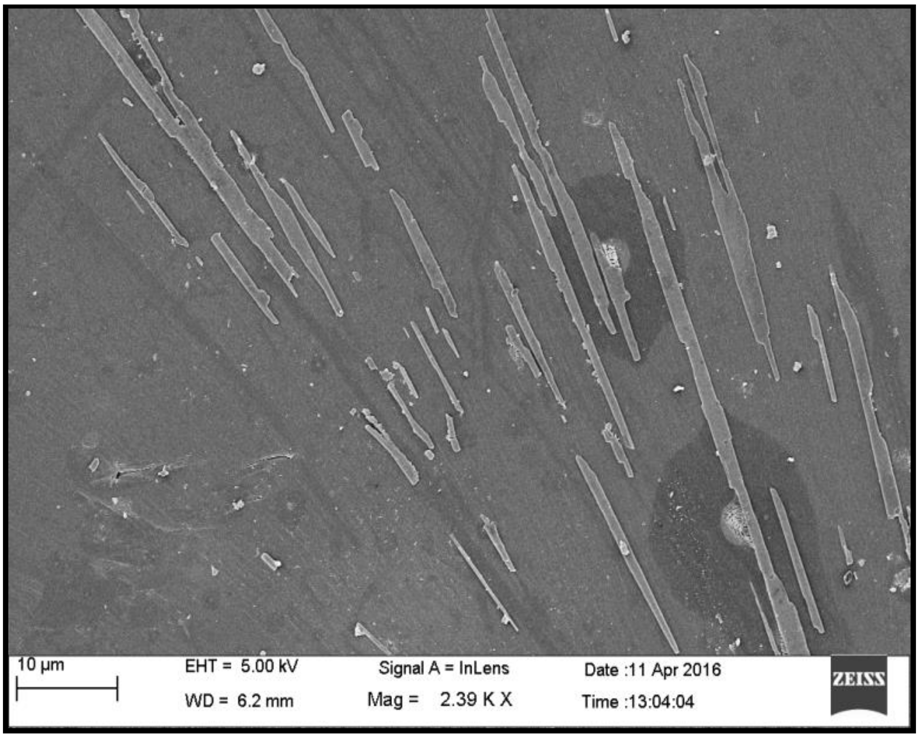


Figure 4.17 SEM image of ripple ring formed in 1064 nm laser irradiated area after 100 pulses

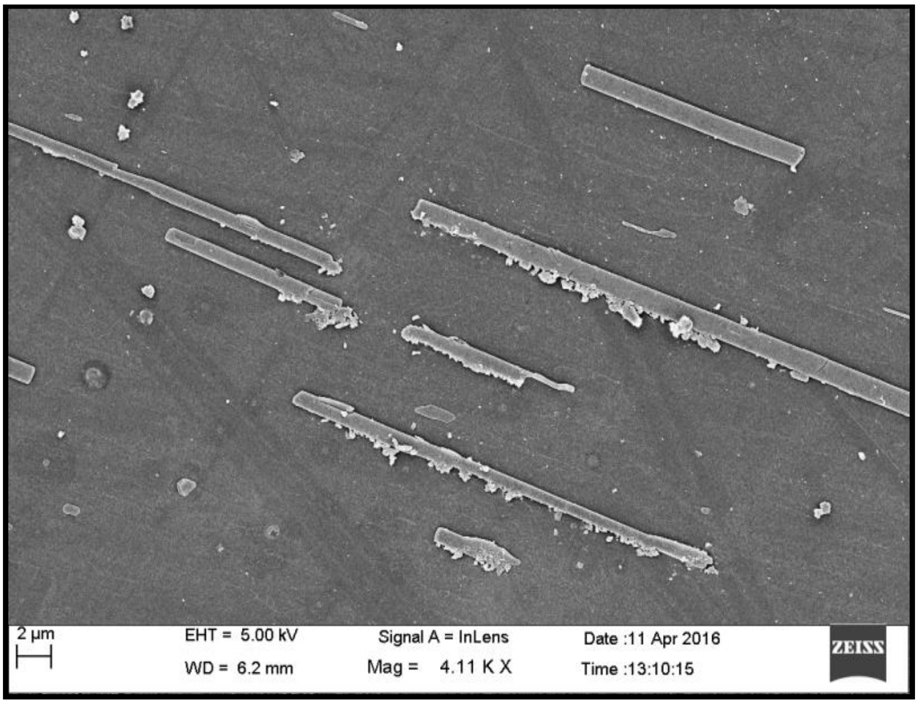


Figure 4.18 SEM image showing lose particles near longer side of structure after 100 pulses

Figure 4.18 shows ripple ring area at 1064nm for 873 cm^2 interacts with polyimide for 100 pulses. It has nearly rectangular shape having longer side parallel to perimeter of ripple ring.

Randomly distributed holes were observed in laser irradiated area. These rectangular projections are not equidistant. Width of projection is around 3µm.

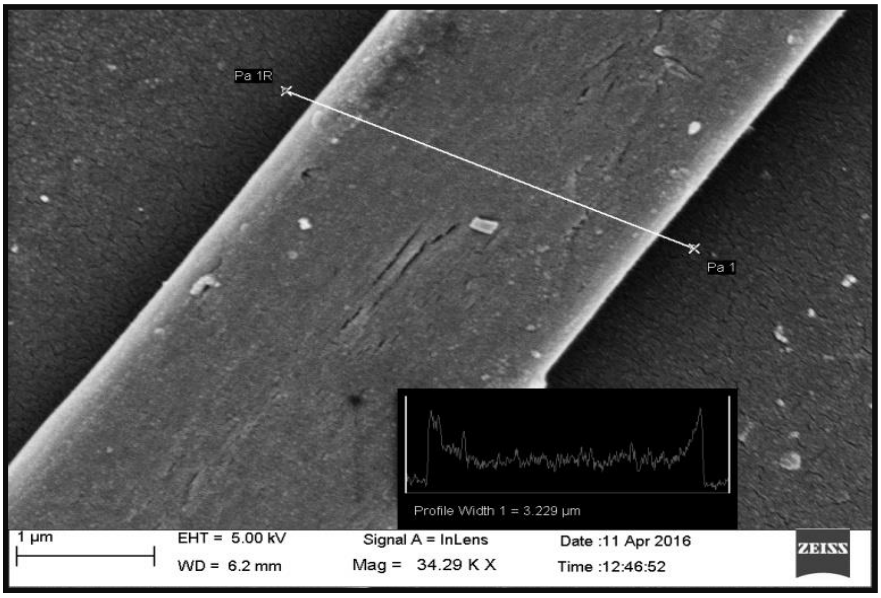


Figure 4.19 SEM image showing approximate topography of rectangular structure produced

Figure 4.19 shows profile of rectangular projection. It has width nearly 3.229µm. Profile taken along width of projection. Its edges lifted up from surface more sharply from both side. Middle portion lifted lesser as compared to edges.

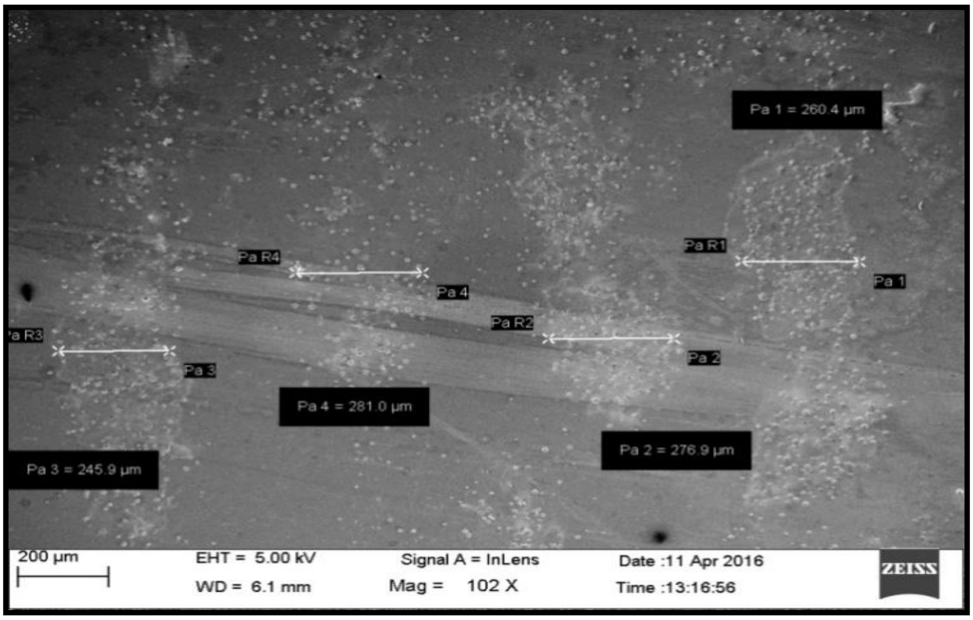


Figure 4.20 SEM image of Holes produced in ripple ring region at higher fluence

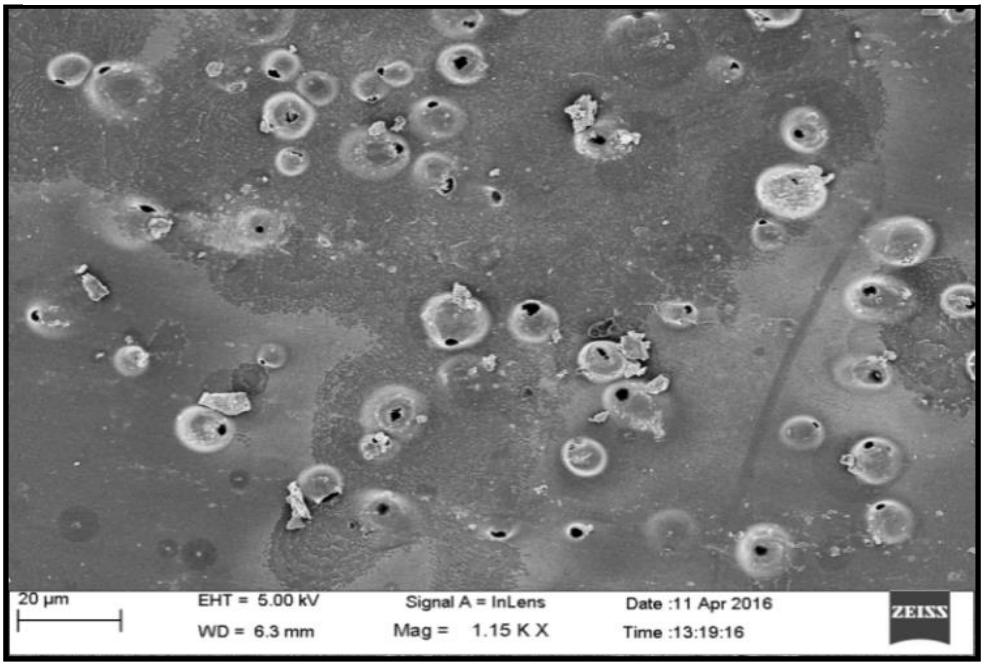
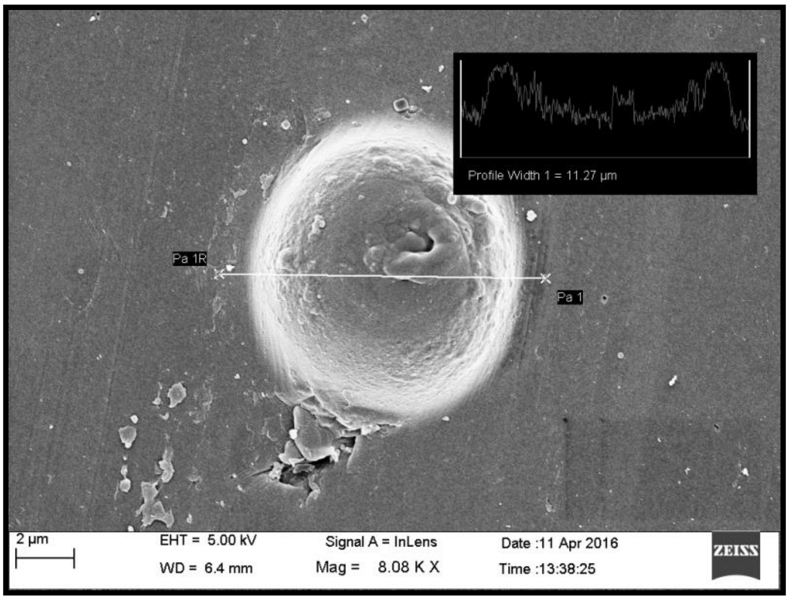


Figure 4.21 SEM image of enlarge view of holes produced in ripple ring region at higher fluence

Figure 4.20 and 4.21 shows when laser fluence is higher (1244mJ/cm2) for 20 pulses it was observed that ripple ring of nearly 270µm has more holes. Hole has size nearly 2 to 4 µm and random distribution. Near to each hole color change can be seen in circular region. This hole seems to be opening of spherical cavity (diameter nearly 10µm) produced inside polyimide surface.



FFigure 4.22 SEM image of circular region undergone color change

Figure 4.22 shows enlarged view of single circular region undergone color change. Its edges from perimeter lifted by larger distance and as we go towards center this projection decreases.

4.5.3 Mechanism of LIPSS

When polyimide expose to laser at 90˚ then ripple were observed as shown in Figure 4.8. Ripple were formed above a critical value of fluence and required minimum number of pulse.

Table 4.3 Minimum number of Pulse and Fluence required to produce ripple structure

Wavelength(nm)	Fluence(mJ/cm2)	Number of pulse
355	218.3	5
532	567	4
1064	873	7

LIPSS was observed at macroscopic level in all three wavelengths. Micro, Nano scale periodic structure was observed in case of 355nm and 1064nm.

LIPSS is phenomenon in which periodic surface ripples, small undulation will generate on laser irradiated surface due to material laser interaction [19]. This structure has ordering and distinct surface morphology. LIPSS was first observed by Birnbaum [32] on semiconductor surface. Further research shown that LIPS can be formed on many polymer like polyimide (PI), Polyethylene Terephthalate (PET) and Polystyrene (PS) [32]

Interference of Laser with surface Plasmon may be possible reason for ripple and periodic structure formation [33][32].Micro /nano structure was observed on sample above a particular value of fluence. Generation of micro/nano structure observed after some minimum number of pulses which is dependent on laser wavelength. Angle of incidence of laser also influence micro/nano structure produced. Many mechanisms were proposed by researchers and constant debate is going to explain exact phenomenon.

Interference of laser with surface Plasmon has been proposed to explain ripple formed on metal, dielectric and semiconductor [(33].Surface Plasmon's are launched by excited material. Ripple originate from interference of incident/refracted laser light with scattered or diffracted light near surface [32]but physical mechanism of their occurrence depends upon material and laser irradiation parameters.

Period of ripple formed by interference of laser with surface Plasmon can be expressed as

$$\Lambda = \frac{\lambda}{\lambda/\lambda_{s} + \sin\theta} \qquad [32], [33] \ldots\ldots\ldots\ldots \qquad (3)$$

Where, λ=Wavelength of incident laser

λ_{s} = Wavelength of surface plasmons

θ=Angle of incidence of Laser

λ_x Can be obtained by the dispersion relation on metal dielectric interface for assuming ε"<|ε'| and real ε_d

$$\lambda_x = \frac{\lambda\sqrt{(\varepsilon' + \varepsilon d)}}{\varepsilon' \varepsilon d} \quad ....(4)$$

Where ε" is imaginary part of ε, ε_d is dielectric constant of material. When laser incident normally in air Θ = 0 and $\varepsilon_{d=1}$ therefore

$$\Lambda = \lambda_x = \lambda(1 + \frac{1}{\varepsilon'}) \quad ....(5)$$

Polyimide has $\boldsymbol{\varepsilon}'$=3.4

At 355 nm from Equation 1 ripple period comes 459.4 nm and at 1064nm it comes around 1376.94 nm from calculation. Experimental result vary with theoretical result, variation is more as in case of 1064 nm. Above theory can give period of micro/nano structure formed in ripple ring region.

Nature of structure formed in ripple region or ripple rings formed is dependent on many laser parameters like Wavelength, Fluence, Number of Pulse, Angle of incidence etc. [32] [33] [19]

Effect of Wavelength: Periodic macro ripples were observed in all three wavelengths. Periodic micro structure was observed at 1064nm and periodic nano structure was seen in case of 355nm.This shows laser wavelength has deciding factor in type of surface structure obtained. As shown in $\Lambda = \lambda_x = \lambda(1 + \frac{1}{\varepsilon'})$ period of micro/nano structure increases as laser wavelength increases. Absorption of laser also depends upon wavelength as shown in equation 3.so wavelength goes on increasing higher fluence required to generate ripple structure. As absorption is low at higher wavelength texturing require higher fluence at higher wavelength.

Effect of Fluence: When laser fluence is above ablation threshold of material then LIPSS is obtained [23]. If large number of pulse irradiated below this threshold pulse then no ripples were observed. At lower fluence region central part of laser spot get textured without any ripples. At higher fluence nano/micro structure formed in ripple ring region gets damaged.

Effect of Number of Pulse: Above a critical value there required minimum number of pulses to generate ripples structure. If fluence is above minimum threshold required to generate ripple then with increase of pulse nano/micro structure gets damage. After large number of pulse this structure formed in ripple ring gets vanish.

As pulse number increases width of ripple ring goes on increasing. After large number consecutive ripples completely mixed as shown in Figure 4.15

In case of 1064nm holes were observed in ripple ring region. Interference is taking place so constructive and destructive circular rings were formed. At constructive region temperature goes higher which starts thermal decomposition of polyimide. During decomposition some gaseous byproducts will be generated these come out and form a hole [7].

4.6 Optical Absorption by UV-VIS

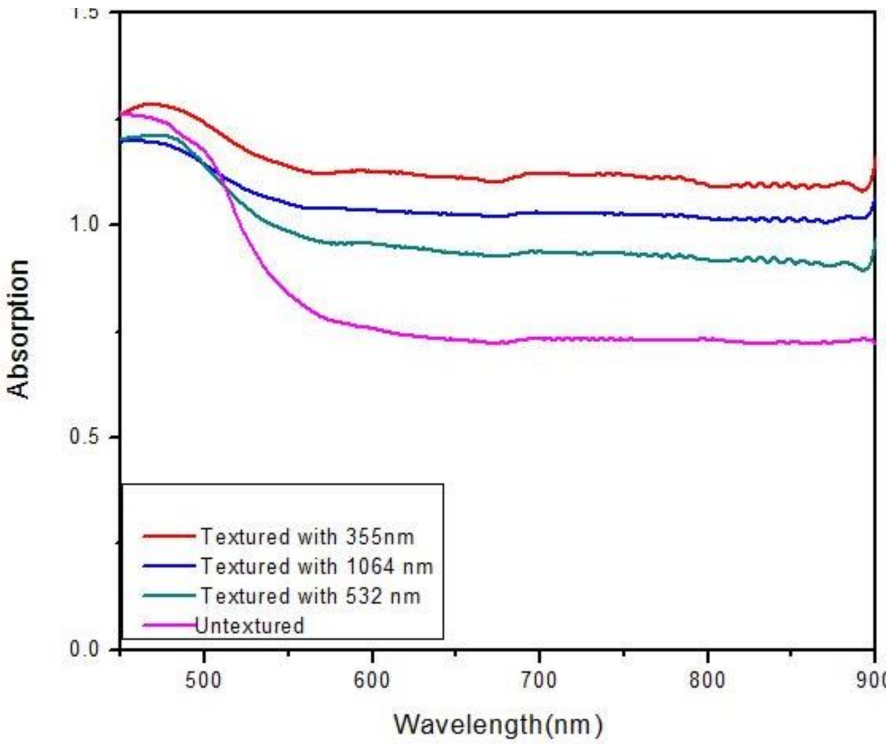


Figure 4.23 Absorption textured side of polyimide processed at various wavelengths

Figure 4.23 shows optical absorption of polyimide before texturing by UV-VIS spectrum (Varian Cary 100).This shows that at higher wavelength absorption decreases by nearly 25 percent. Previous study shows that at 355 nm polyimide has highest absorption at lower wavelength and it decreases as wavelength increases. At 1064 nm has lowest absorption coefficient out of three working wavelengths of laser [35]. As 355nm wavelength has higher photon energy and higher absorption coefficient lower fluence is required for texturing.

Figure 4.23 shows comparison of front side absorption of sample textured with three wavelengths with un-textured polyimide. Above 520 nm all textured samples shows higher absorption than un-textured polyimide. Highest absorption was observed at 355nm. Absorption at 1064 nm varies drastically depending upon glassy layer formation. Glassy layer reduces absorption due to its higher reflectivity.

Table 4.4 Details of UV-VIS samples in Figure 4.23 to 4.28

Wavelength	Sample Number
355	3
532	9
1064(a)	15
1064(b)	18

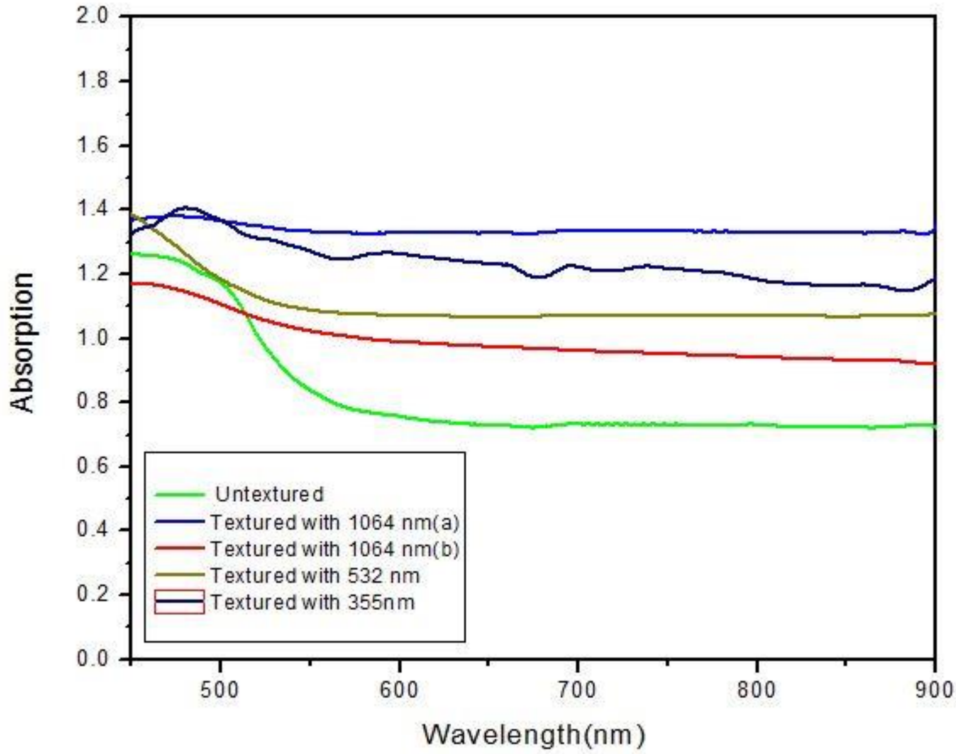


Figure 4.24 Back side absorption of polyimide processed at various wavelengths

Back side of textured polyimide was studied in figure 4.24.Sample textured with 532nm wavelength has higher absorption than un-textured sample. Polyimide textured with 355nm has higher absorption than polyimide textured by 532nm. Absorption of polyimide textured with 1064 nm wavelength depends upon glassy layer formation. Glassy layer formed at higher fluence and large time of interaction of laser with polyimide. Glassy layer is product of decomposition of polyimide during laser irradiation and has reflecting nature. When glassy layer formed on polyimide textured with 1064nm, it has lower absorption [sample 1064(b)] otherwise it show higher absorption[sample 1064(a)]

4.7 Optical Transmission and Reflectance by UV-VIS

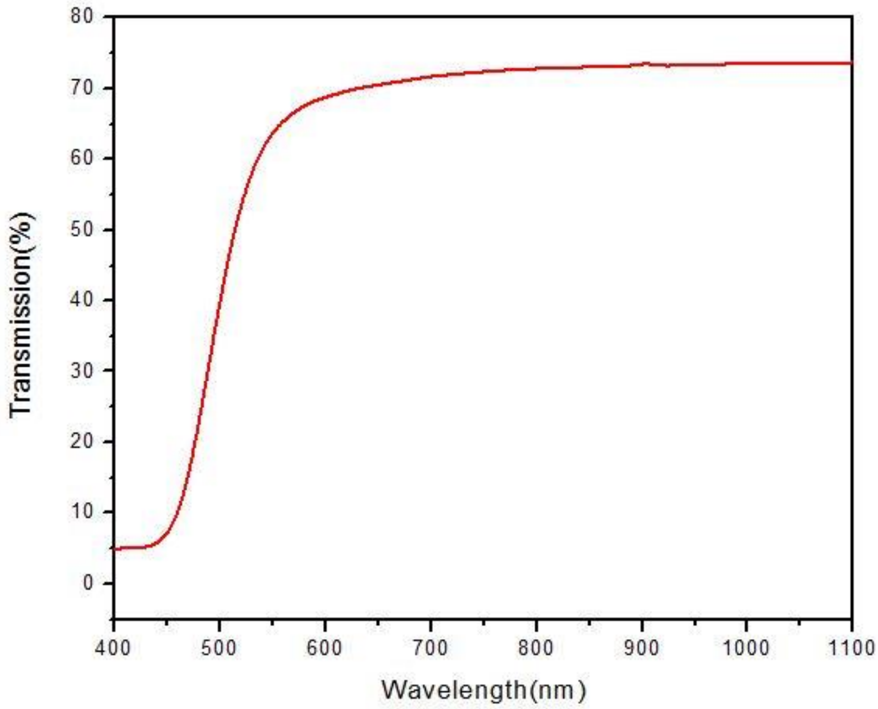


Figure 4.25 Transmission of polyimide textured by 355nm wavelength

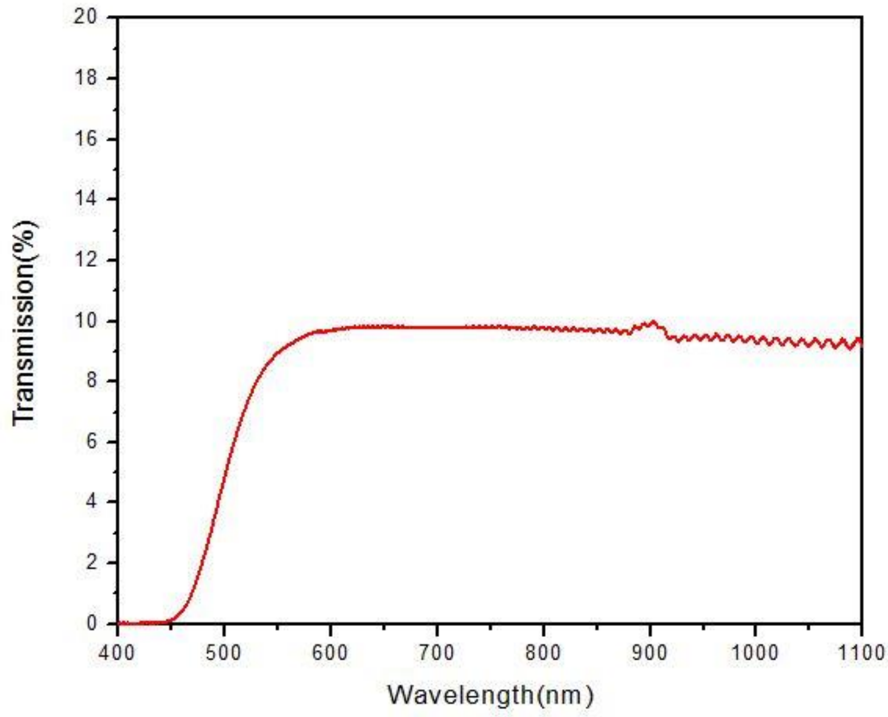


Figure 4.26 Transmission of polyimide textured by 532nm wavelength

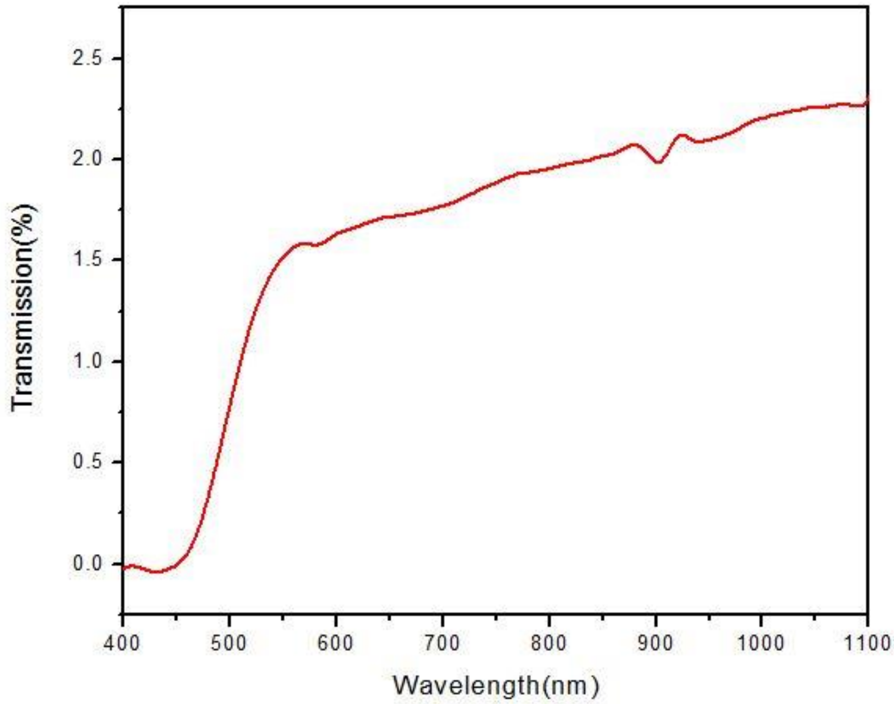


Figure 4.27 Transmission of polyimide textured by 1064nm wavelength

It is found by UV-VIS study that polyimide film has high transmittance from 400nm to 1200nm (nearly constant around 92%).Laser texturing reduces transmittance of polyimide. Reduction in transmittance is more when polyimide is textured by 1064nm and less when we use 355nm wavelength as shown in figure 4.25and Figure 4.27. Transmittance of textured polyimide is varying with respect to wavelength. Reduction in transmittance is high for wavelength of light is below 600nm for all investigated samples as shown in figure 4.24, 4.25 and 4.27.Graph shows, if wavelength of light is below 450nm transmittance becomes nearly zero when 532nm and 1064nm used for texturing.

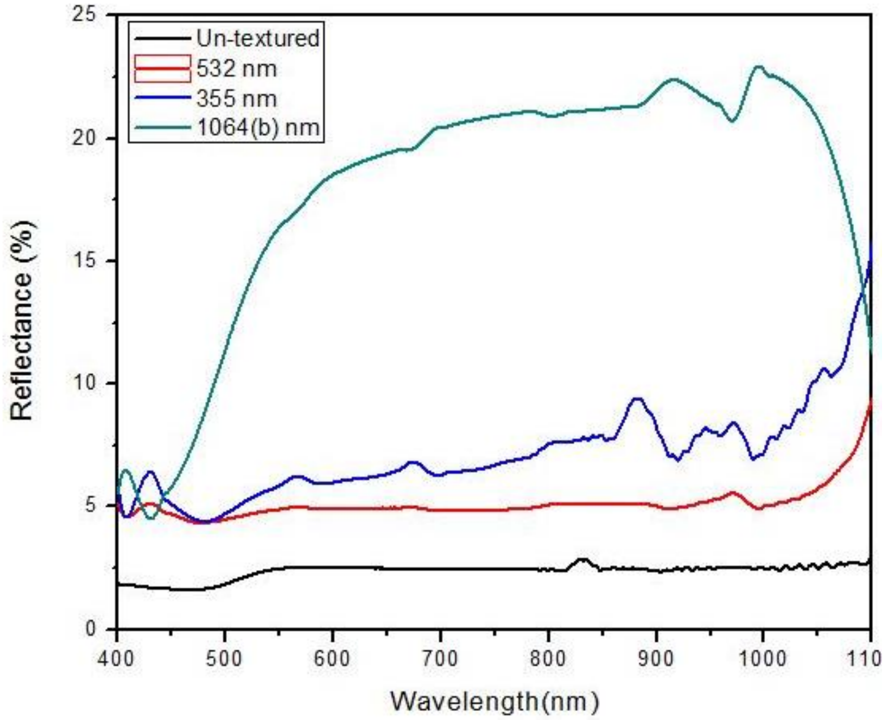


Figure 4.28 Variation of reflectance of textured polyimide with respect to processing wavelength

It is observed that when texturing performed on polyimide it increases its reflectivity as shown in figure 4.28. Un-textured polyimide has reflectance around 2 to 3 %. Increase in reflectance is lower when polyimide textured with 532 nm wavelength laser. Increase in reflectance is more when 355nm laser was used for texturing. If texturing is performed with 355nm or 532nm laser wavelength there is slight increase in reflectance. Increase in reflectance is more when 1064nm wavelength is used. This may be happening due to shiny/glassy layer of decomposition products formed during laser irradiation. As transmittance of polyimide is decreasing drastically after laser texturing this may be reason to increase in reflectance.

Chapter 5 CONCLUSIONS AND SCOPE FOR FUTURE WORK

5.1 Conclusions

- Wrinkles are observed on polyimide surface at 532nm and 1064nm wavelength of laser.
- Interference rings are observed at all three wavelengths and periodic structure observed inside the interference ring when laser wavelength is 355nm or 1064nm.
- Polyimide can be textured rapidly at lower fluence when laser wavelength 355nm. Generation of carbonious product on surface of polyimide is absent at 355nm.
- Optical properties of polyimide change drastically when it is textured at 1064nm wavelength. Variation of transmission, absorption and reflectance is affected by glassy layer formed at 1064nm.

5.2 Scope for Future Work

- Simulation study can give better inside of laser interacting with polyimide when experimental parameter is varied. Optimized parameter should be obtained from simulation to get optimum performance of solar cell developed on polyimide.
- Actual performance of solar cell deposited on textured polyimide need to be investigated.
- Laser texturing many properties like surface wetting, electrical resistance, mechanical strength etc. Adverse effects of laser texturing on polyimide need to be studied thoroughly.

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CONTENTS

LIST OF FIGURES

List of Tables