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Investigation of Low Temperature Metal Induced Crystallization and Doping of Silicon Using Various Excitation Sources for Solar Cells and other Microelectronic Applications

Final Report

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Hameed A. Naseem
Husam H. Abu-Safe

University of Arkansas
Fayetteville, Arkansas 72701

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Executive Summary

The purpose of this project was to investigate metal-induced crystallization of amorphous silicon at low temperatures using excitation sources such as laser and rapid thermal annealing, as well as, electric field. Deposition of high quality crystalline silicon at low temperatures allows the use of low cost soda-lime glass and polymeric films for economically viable photovoltaic solar cells and low cost large area flat panel displays. In light of current and expected demands on Si supply due to expanding use of consumer electronic products throughout the world and the incessant demand for electric power the need for developing high grade Si thin films on low cost substrate becomes even more important. We used hydrogenated and un-hydrogenated amorphous silicon deposited by plasma enhanced chemical vapor deposition and sputtering techniques (both of which are extensively used in electronic and solar cell industries) to fabricate nano-crystalline, poly-crystalline (small as well as large grain), and single-crystalline (epitaxial) films at low temperatures. We demonstrated Si nanowires on flat surfaces that can be used for fabricating nanometer scale transistors. We also demonstrated lateral crystallization using Al with and without an applied electric field. These results are critical for high mobility thin film transistors (TFT) for large area display applications. Large grain silicon (~30-50 μm grain size for < 0.5 μm thick films) was demonstrated on glass substrates at low temperatures. We also demonstrated epitaxial growth of silicon on (100) Si substrates at temperatures as low as 450˚C. Thin film Si solar cells are being projected as the material of choice for low cost high efficiency solar cells when properly coupled with excellent light-trapping schemes. Ar ion laser (CW) was shown to produce dendritic nanowire structures at low power whereas at higher powers yielded continuous polycrystalline films. The power density required for films in contact with Al was demonstrated to be at least two orders of magnitude lower that that reported in the literature before. Polysilicon was successfully achieved on polyimide (Kapton©) films. Thin film Si solar cells on lightweight stoable polymer offer great advantage for terrestrial and space power applications. In summary we have demonstrated through this research the viability of producing low cost nano-, poly-, and epitaxial Si material on substrates of choice for applications in economically viable environmentally friendly sustainable solar power systems. This truly enabling technology has widespread applications in multibillion dollar electronic industry and consumer products.

Project Objectives:

The main goals for the project are outlined as follows:

1. Research: To carry out detailed investigation of low temperature metal induced crystallization and doping of silicon using various excitation sources- laser/lamp annealing, furnace/rapid thermal annealing, and voltage bias. Hydrogenated and un-hydrogenated amorphous films deposited using sputtering and PECVD were used. The objective is to develop a comprehensive model incorporating the basic mechanisms underpinning metal induced crystallization. This knowledge can be utilized in fabricating some silicon solar cells and thin film transistors.
2. National Lab Partnership: To develop strong partnership with National Renewable Energy Laboratory (NREL), and to use other DOE user facilities for research such as National Center for Electron Microscopy (NCEM) and Oak Ridge National Laboratory (ORNL).
3. Infrastructure: To train faculty, postdoctoral research associates, and graduate students in energy related fields and to graduate them. Also to improve equipment infrastructure.
4. Dissemination of results: To generate patents, publish refereed papers in journals and proceedings, make presentations at national/international and regional conferences and DOE review conferences.
5. Proposals: To submit new proposals for enhancing research infrastructure as well as for expanding research in related areas of interest to DOE.

Summary of Research Results:

- **The effect of the surface characteristics of the hydrogenated amorphous silicon (a-Si:H) on the metal-induced crystallization (MIC):**

Stage one

We have invented a technique that enables us to control the grain size of the poly-Si by putting down a thin insulating layer (such as SiO_2) between the metal (Al) and the amorphous silicon layer. The results of introducing this layer have been very exciting, where we noticed that grains as large as 10 μm could be achieved using this method. Figure 1 shows two SEM images of two poly-Si films fabricated using the MIC method. Figure 1-a shows the surface of a sample where no SiO_2 layer was introduced and Figure1-b shows a sample fabricated by the invented method. The image of sample b is taken at a lower magnification to show the size of the grain which is about 10 μm. TEM investigations have shown that the large features in Figure 1-b are made of a single crystal. Figure 2-a shows a Selected Area Diffraction (SAD) image taken for sample 1-b and Figure 2-b is a dark-field (DF) image of some part of that grain. The SAD image is taken for an area that is at least 3 μm large and the DF image shows a part of a grain and this part is at least 1 μm across. This analysis (done at NREL) clearly shows the great potential this technique holds for large area thin film solar cells and thin film transistors.

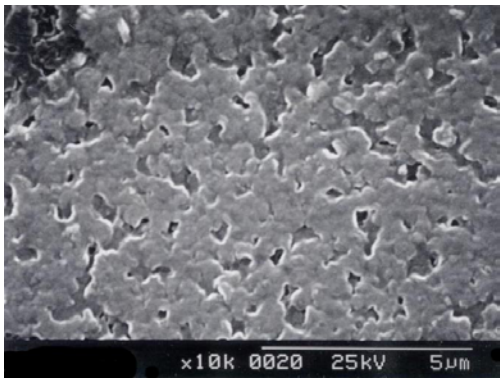


Figure 1-a

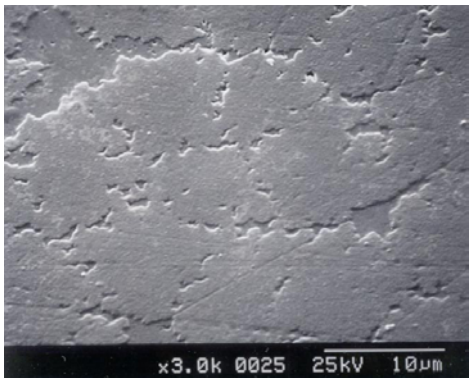


Figure 1-b

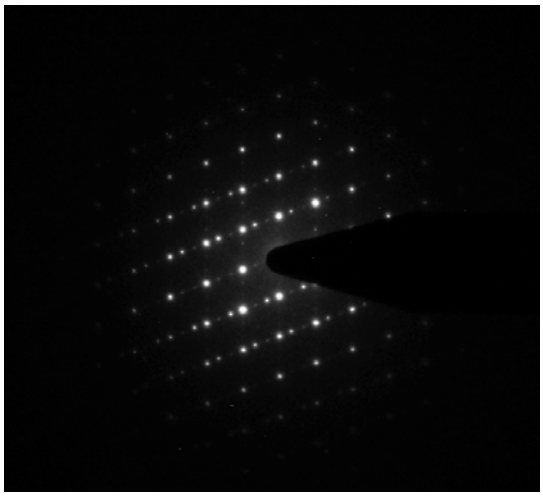


Figure 2-a

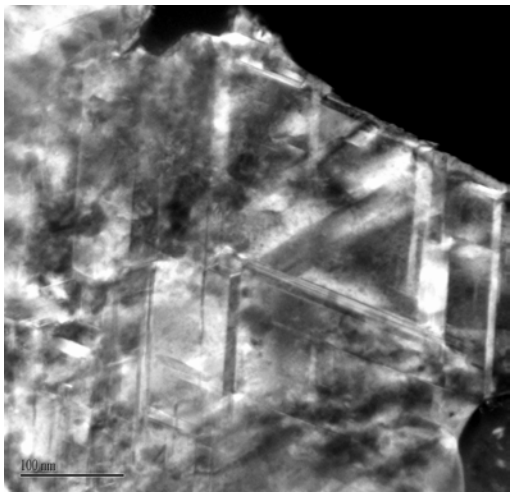


Figure 2-b

Stage two

The principal investigator, Professor Hameed Naseem and his graduate student, Marwan Barghouti, have submitted their invented technique to control the grain size of polysilicon thin films for patenting. This technique is the result of the major efforts made to grow large grains in the poly-Si films. The introduction of a thin oxide layer (such as SiO_2) at the interface between the aluminum and silicon in the metal induced crystallization process, produced large smooth-surface grains in the silicon films. More transmission electron microscopy (TEM) characterization was preformed on the films. The characterization was done in collaboration with the national center for electron microscopy (NCEM), Berkley, California. Figure 3 shows a bright-filed (BF) low magnification TEM image of one of the poly-Si islands. The image reveals that the island is actually made of one structure with no visible grain boundaries. The white areas surrounding the islands could be vacuum caused by the sample preparation for TEM or could be the regions in the film that remained amorphous. The inset shows the electron diffraction pattern (EDP) obtained from the area encircled by the white circle. The diffraction pattern is of very good quality Si single crystal. The diffraction pattern is an indication that the whole island is actually one single crystal of Si that is, at least, larger

than 6 μm. Figure 4 shows a dark-field TEM image of another sample annealed at 450°C for 20 minutes. The grain size in this sample is smaller than the sample shown in Figure 3. However, grains as large as 2 μm can be identified in this image. The electron diffraction pattern in the inset shows a single crystal diffraction pattern. The selected area aperture was 1 μm. The TEM and the AFM results support the idea that there is a process of coalescence between 100-200 nm grains to form large grains.

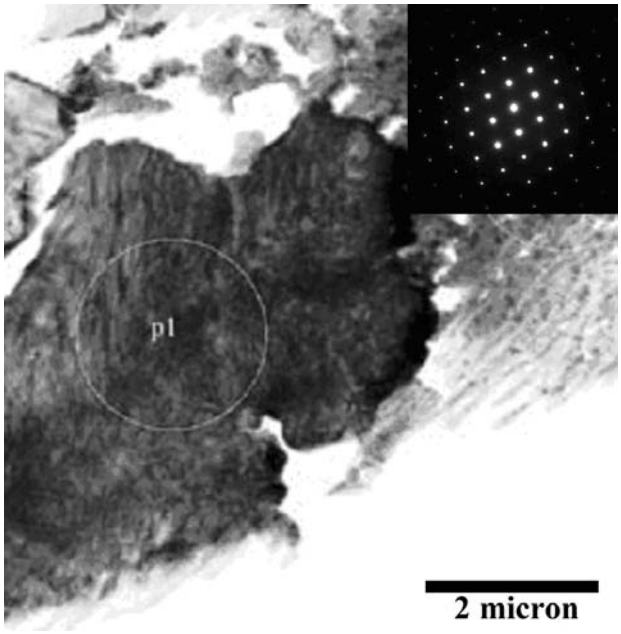


Figure 3: Low magnification. BF TEM image of one of the poly-Si islands. The Inset shows the EDP obtained from the area encircled

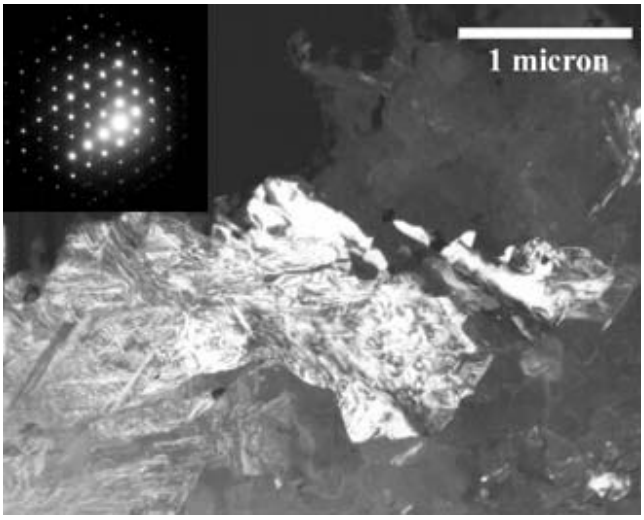


Figure 4: TEM DF image showing a single grain of irregular shape. The grain size is at least 2 μm.

- **CW Ar^+ Laser initiated Aluminum Induced Crystallization:**

Magnetron Sputtered α-Si thin films

Metal induced crystallization of amorphous silicon thin films using CW Ar^+ laser beam as an excitation source was investigated. The samples were prepared using Corning 7059 glass substrates. A 300 nm thick α-Si was deposited by magnetron sputtering on the glass substrate. A 200 nm thick Al film was subsequently sputtered on α-Si without breaking the vacuum. CW Ar^+ laser (λ=of 514.5nm) was used to irradiate the samples with various power densities and exposure times. A constant power of 3.2 W from the laser beam was used to irradiate the samples from the glass side. However, by changing the spot diameter, power densities were varied from 55 to 125 W/cm^2. For each power density, the samples were exposed to the laser beam for 10, 60 and 120 seconds.

Figure 5-a shows the X-Ray diffraction (XRD) pattern results obtained for the three power densities (P.D) 56.14W/cm^2, 79.85W/cm^2 and 124.75W/cm^2 exposed for 10 seconds to the laser beam. The crystalline peak for silicon Si (111) was observed at 28.5° showing the polycrystalline nature of the laser exposed samples. The peak intensity increased with increasing power density. Figure 5-b shows XRD patterns for samples exposed to the laser beam for 120 sec at different power densities. The silicon peaks are higher in this case. This leads us to conclude that crystallization starts and continues as long as the samples are irradiated until the entire sample is crystallized.

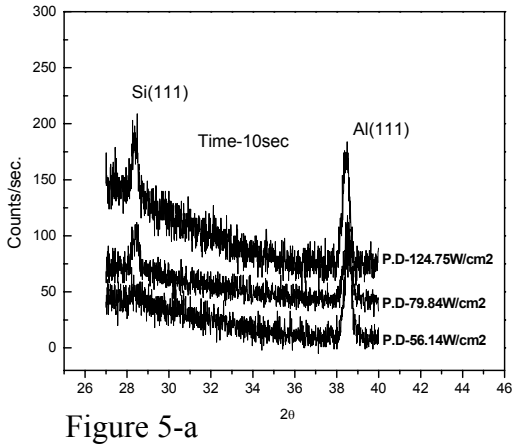


Figure 5-a

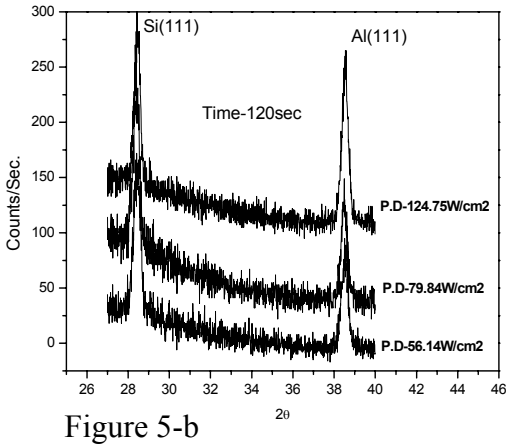


Figure 5-b

SEM analysis revealed changing surface features with increasing power density and exposure time. Figures 6-a, b, and c show the SEM pictures of three samples exposed to a constant power density of 56.14 W/cm^2 but three different exposure times of 10, 20, and 120 sec. Polysilicon grains start to nucleate and then increase in size to cover the whole area exposed by laser.

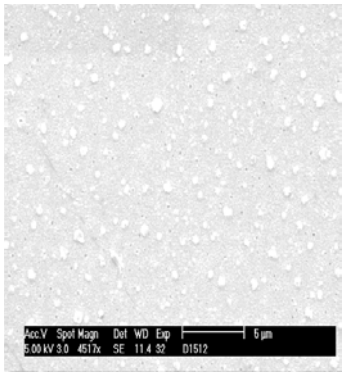


Figure 6-a (10 Sec.)

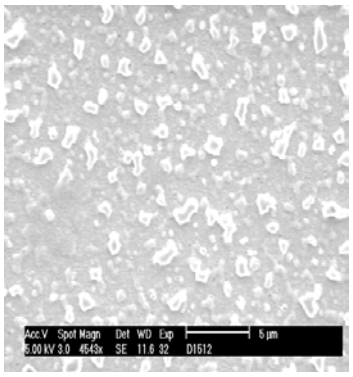


Figure 6-b (60 Sec.)

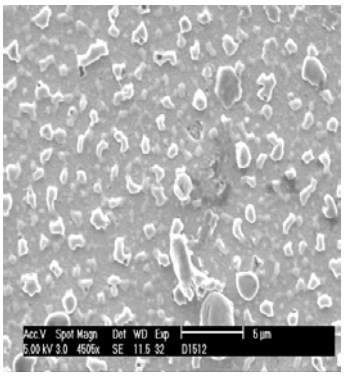


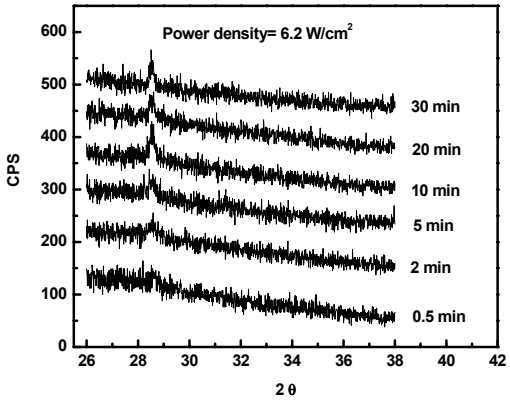
Figure 6-c (120 Sec.)

Hydrogenated α-Si thin films

As a continuation of the work initiated in the metal induced crystallization of amorphous silicon using CW Ar^+ laser beam as an excitation source, samples of hydrogenated amorphous silicon (a-Si:H) were prepared on corning 7059 glass and cover-glass substrates. A 300 nm thick α-Si:H was deposited by Plasma Enhanced Chemical Vapor deposition (PECVD) on the glass substrate. A 300 nm thick Al film was subsequently sputtered on α-Si without breaking the vacuum. CW Ar^+ laser (λ=of 514.5nm) was used to irradiate the samples with various power densities and exposure times. A constant power of 3.2 W from the laser beam was used to irradiate the samples from the glass side. However, by changing the spot diameter, power densities were varied from 3.6 to 6.2 W/cm^2. This is the lowest power density that has been recorded in laser crystallization of a-Si. For each power density, the samples were exposed to the laser beam for various time periods.

Figure 7 shows the X-Ray diffraction (XRD) pattern results obtained for the power density 6.2 W/cm^2 exposed for various time periods. The crystalline peak for silicon Si (111) was observed at 28.5° showing the polycrystalline nature of the laser exposed samples. The peak intensity increased with increasing exposure time.

Figure 7: The XRD pattern for the laser exposed samples at 6.2 W/cm^2 for various time periods. The (111) silicon peak at 28.5° indicate the crystalline nature of the irradiated samples. The peaks at 38.4° are for Al.



Laser spot size with 5.5 mm was obtained to get a 6.2 W/cm^2 laser power density. However, since the laser beam transverse profile was in the TM_{00} mode which is a Gaussian profile, the power density was at its highest peak at the center of the spot. Away from this center, the power density reduced in a logarithmic scale. This power changes influenced the crystallization process across the relatively large spot and therefore the morphology of the crystallized region. These changes are shown in Figure 8.

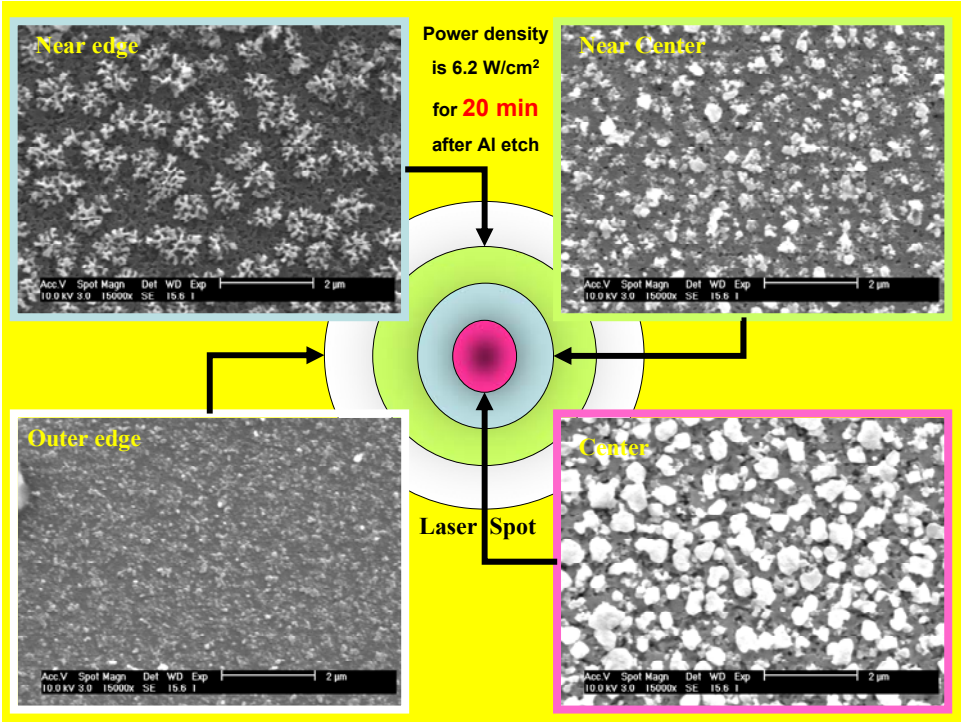


Figure 8: SEM images of the laser annealed samples indicating cross section areas outside, near edge, near center and center of the laser spot.

- **The effect of substrate temperature and Al-oxide interface layer on aluminum induced crystallization of sputtered amorphous silicon:**

The effect of substrate temperature on aluminum-induced crystallization (AIC) of amorphous silicon deposited by sputtering was studied by changing the deposition temperate of a-Si from 200 to 300ºC in a Al/a-Si/glass configuration. After deposition, the samples were annealed in the temperature range from 300ºC to 525ºC for different periods of time. The X-ray diffraction (XRD) patterns confirmed the crystallization of the a-Si films. From the analysis, we report that the crystallization of a-Si initiate at 350ºC annealing temperature. Crystallization saturated within 20 minutes of annealing time at 400ºC. On the other hand, when Al-oxide is present at the interface in the a-Si/Al/glass structure, higher annealing temperatures and longer annealing times were required to complete the crystallization of a-Si. Environmental Scanning Electron Microscope

(ESEM) and Energy Dispersive X-Ray (EDX) mapping were used to study the surface morphology as well as the layer sequence after crystallization. This analysis revealed that, Si-Al layer-exchange occurs regardless of the deposited configuration.

- **Investigation of aluminum induced crystallization of amorphous silicon using lateral crystallization.**

The mechanism of metal induced crystallization was studied with the aid of lateral crystallization. Some more experiments were performed on lateral crystallization in addition to the experiments done previously. A model was developed to predict the lateral crystallization velocity on the basis of resistance measurement of the film at 400^{o}C. This model was developed at 300^{o}C, previously. The results came out to be consistent with the previous results. The lateral crystallization velocity at 400^{o}C came out to be 0.44 microns/hour whereas at 300^{o}C, it was 0.35 microns/hour. Figure 9 shows the plot between laterally crystallized lengths versus annealing time as determined from the model.

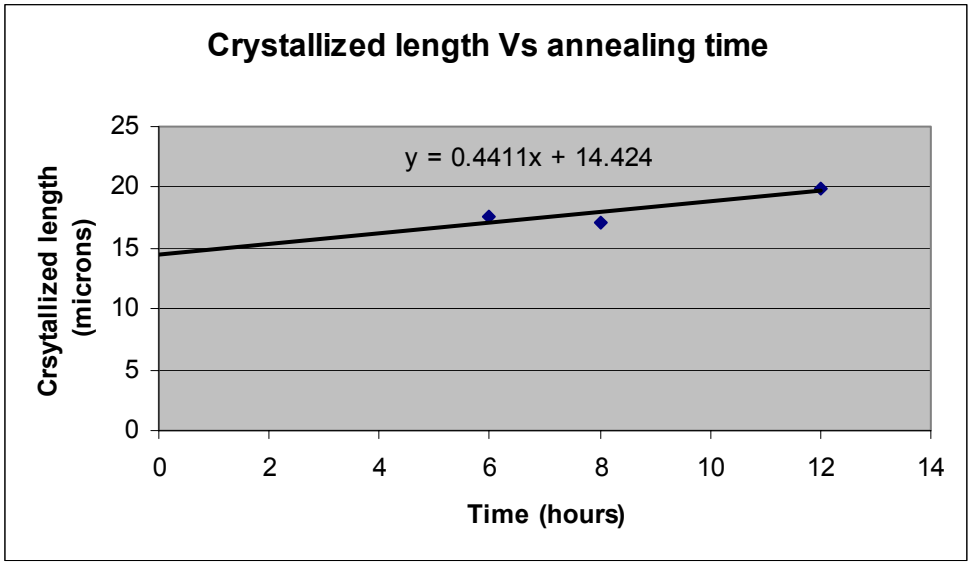


Figure 9

- **Characterization of silicon nitride films for thin film transistor applications**

Optical and electrical properties of silicon nitride thin films were studied to check their viability as gate dielectric for thin film transistors. Hydrogenated amorphous silicon nitride films were deposited using plasma enhanced chemical vapor deposition (PECVD). Silane (SiH_4) and nitrogen (N_2) were used for the deposition. The flow rate of silane was kept at 2 sccm and the N_2 flow rate was varied from 100 sccm to 200 sccm. The power was kept at 20 Watts. The band-gap of the films was measured using spectrophotometer. The study revealed that the films having more amount of nitrogen have larger band-gap as compared to the ones having lesser amount of nitrogen. Figure 10 is a plot between N_2 flow rate and the Tauc band-gap. It can be seen that bad-gap first increases with increase in the flow rate and then becomes almost constant. The band-gap of as much as 4.67 eV has been obtained. Fourier transform infrared (FTIR) spectroscopy was used to determine

the hydrogen content of the films. Figure 11 shows an FTIR plot for the sample with a SiH_4 flow rate of 2 sccm and N_2 flow rate of 200 sccm. From the plot it can be seen that there is no hydrogen peak at 2200 cm^{-1} for Si-H; hydrogen is present in the form of N-H at 3400 cm^{-1}.

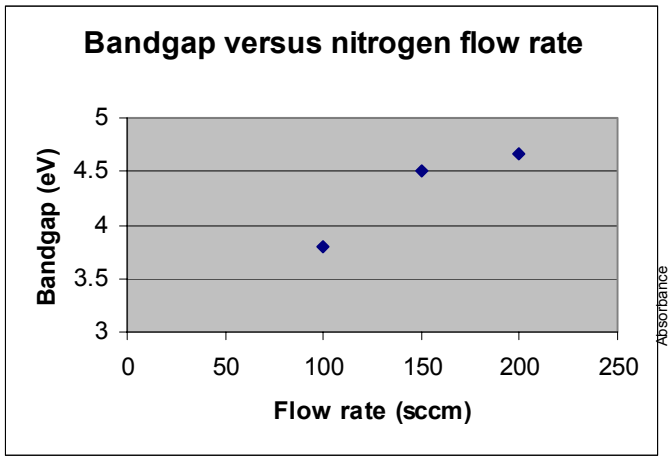


Figure 10

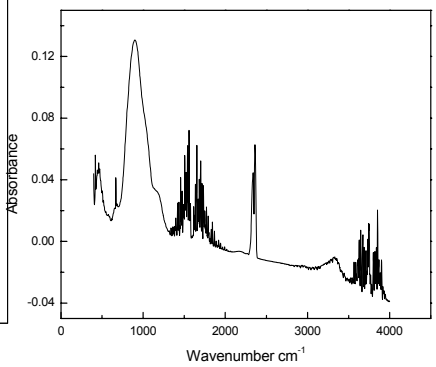


Figure 11

- **Aluminum induced crystallization of sputtered hydrogenated amorphous silicon:**

As part of our objectives, we investigated the role of hydrogen in metal induced crystallization of amorphous silicon. In order to have control over the amount of hydrogen incorporated inside the amorphous silicon films, we modified the silicon sputtering chamber in our multi-chamber deposition system. A hydrogen gas line was added to this chamber. We begin our investigation by depositing hydrogenated amorphous silicon (a-Si:H) thin films at 250ºC on glass substrate in hydrogen and argon ambient. The hydrogen flow rate was varied to obtain different hydrogen content (at.%) inside the films. The deposition rate increased more than two folds with the introduction of the hydrogen during deposition. The films were coated with a thin layer of sputtered aluminum deposited at 40ºC. To crystallize the a-Si:H films, all of the samples were annealed in a temperature range from 225ºC to 400ºC for 20 and 40 minutes. The area under the X-Ray diffraction (XRD) peaks of crystalline silicon increased with hydrogen content. To further characterize these films, scanning auger microscopy (SAM) was used at Oak Ridge National Laboratory (ORNL). Depth profiles as well as auger mapping at different depth from the surface were collected. Figure 12 shows the Auger depth profiles of samples with deposition sequence of Al/a-Si:H/glass under un-annealed, and annealed at 275 and 350°C conditions. In the unanealed sample Al is at the top whereas as at 350°C si appeared on top. Profile at 275°C shows an intermediate stage.

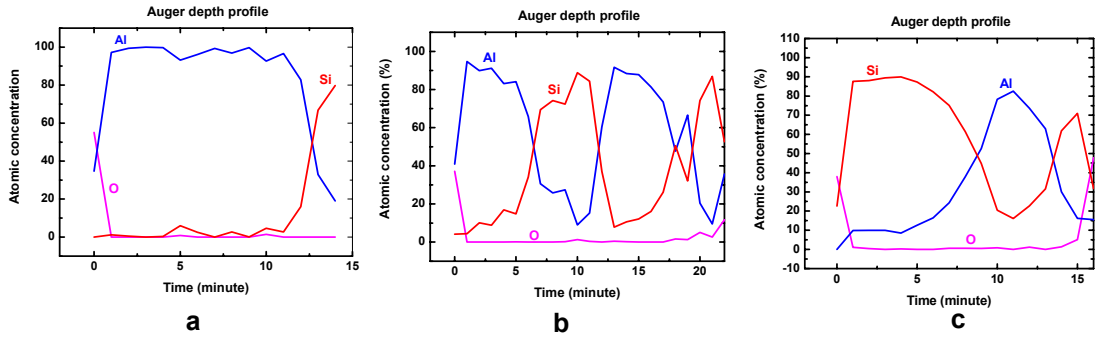


Figure 12: shows the Auger depth profile of the sample with deposition sequence of Al/a-Si:H/glass. a) Un-annealed, b): annealed at 275°C and c) annealed at 350°C.

Figure 13 shows Auger maps of the hydrogenated sample (14.2 at.%) annealed at 225°C at a depth of 60 and 400 nm from the surface. It is observed that the grain size of the poly-Si layer is about 10µm.

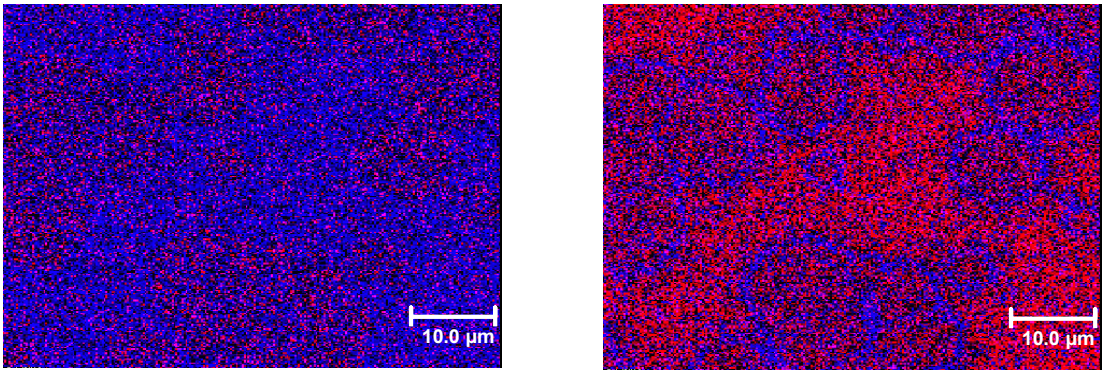


Figure 13: Auger map of the 14.2 % hydrogenated sample annealed at 225°C at a depth (a) about 60 nm and (b) about 400 nm from the surface.

- **The effect of substrate temperature and interfacial oxide layer on aluminum induced crystallization of sputtered amorphous silicon.**

The effect of substrate temperature and interface oxide layer on aluminum induced crystallization of amorphous silicon is investigated. The effect of substrate temperature on the process was studied by changing the deposition temperate of a-Si from 200 to 300ºC in a Al/a-Si/glass configuration. Figure 14-a shows the XRD peak area of (111) silicon as a function of substrate temperature. Within the investigated temperatures, the substrate temperature had a minor effect on the crystallization process as a whole. Figure 14-b shows the effect of interfacial oxide on the crystallization process. The presence of an oxide layer did indeed increase the crystallization temperature especially in the case of aluminum oxide.

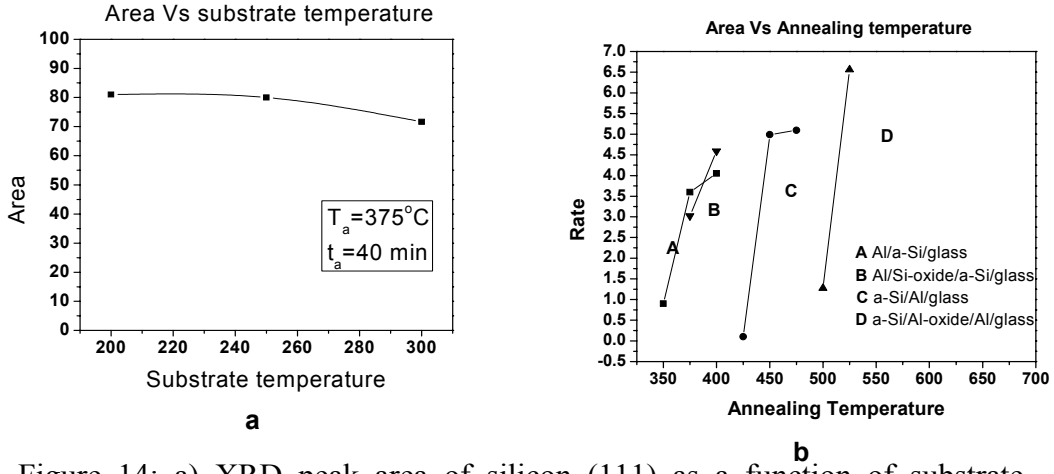


Figure 14: a) XRD peak area of silicon (111) as a function of substrate temperature. A changing the substrate temperature above between 200-300°C had a minor effect on the overall process. b) Crystallization rate versus annealing temperature.

- **Investigation of the impact of Stress on Aluminum-Induced Crystallization of Amorphous Silicon**

Although crystallization of amorphous silicon has been extensively studied, the effect of stress on the crystallization is not well understood. The film stress along with many other factors, such as, amorphous silicon and metal deposition parameters will affect the crystallization temperature. We conducted a systematic study of the effect of stress on aluminum-induced crystallization of PECVD a-Si:H. For the first time, stress is decoupled from other parameters, which improves the understanding of the physics behind the effect.

In this study, a set of experiments was designed to vary the stress of a-Si:H films by adjusting the substrate curvatures. To decouple the impact on the crystallization from other factors, identical thin film structures, Al (200 nm)/a-Si:H (300 nm), were deposited on the front surfaces of all substrates. a-Si:H films of various thicknesses were then deposited on the back of the substrates to adjust the samples' curvature. After thin film deposition, all samples were annealed at 325°C for 30 minutes. One sample had thin films on the front surface only (sample b), while the other had identical thin films on both sides of the substrate (sample a). Figure 15 shows XRD spectra from the two samples. The peak corresponds to Si (111). It is clear that after annealing, the sample without thin films on the back surface was crystallized, while no crystallization was observed on the other sample. Since the only difference between the two samples is the back surface structure, this experiment clearly reveals the impact of stress on the crystallization of a-Si:H.

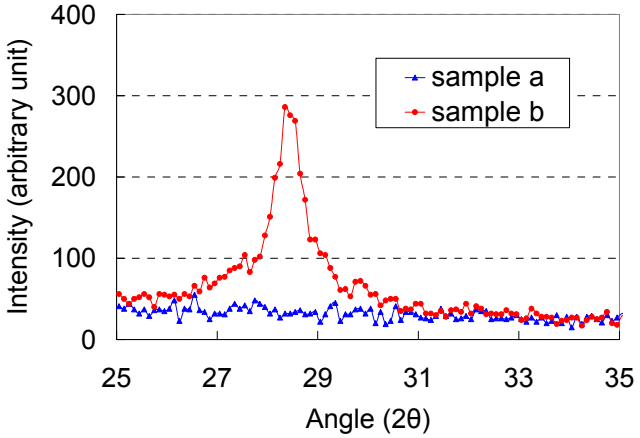


Figure15: The effect of stress on the crystallization process of PECVD films. Sample 'a' had a thin film deposited only on one side of the substrate whereas sample 'b' had thin films deposited on both sides of the substrate. Sample 'a' had lower tensile stress and lower crystallization temperature.

The effect of stress on crystallization of sputtered a-Si was found to be opposite to that of PECVD a-Si film discussed above (see figure 16). The samples which had films on both sides (sample b) crystallized faster than the ones with only one film on the side (sample a). This could be related to the type of stress involved- compressive or tensile. More investigations have to be conducted to explain this contradiction.

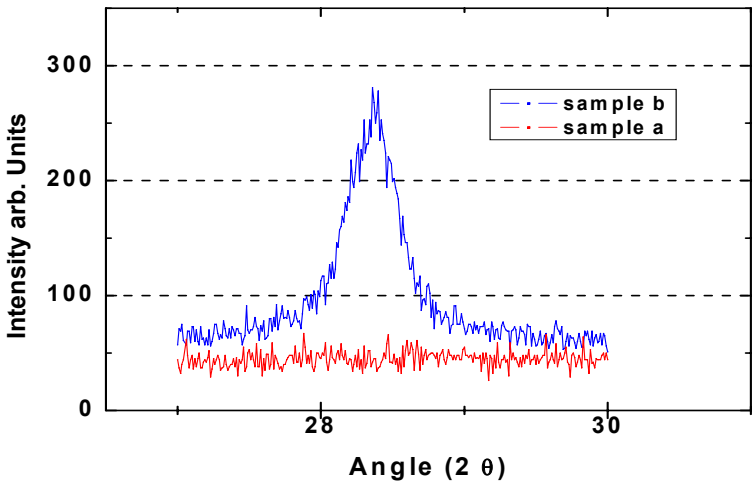


Figure 16: The effect of stress on the crystallization process of sputtered films. The effect is opposite to the one in the PECVD case. This contradictory behavior could be related to the type of stress imparted on the films during deposition.

- **Silicon nanowire technology: Fabrication of silicon nanowire network in aluminum thin films**

The formation of isolated silicon nanowires and silicon nanowire networks using aluminum thin film is investigated. The formation mechanism of the network mainly depends on the diffusion of silicon in the aluminum thin film. The silicon stops at the film grain boundaries. The continuous accumulations of silicon at these boundaries give raise to a continuous network of silicon nanowires. Characterization of the nanowires has been done using scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDS). Figure 17 Show SEM silicon-alumium nanowires These results are unique in the fact that the nanowires found are grown in a horizontal fashion instead of the more common vertical direction. Most of the nanowires have a diameter of about 60 nm and a length of over 5 μm.

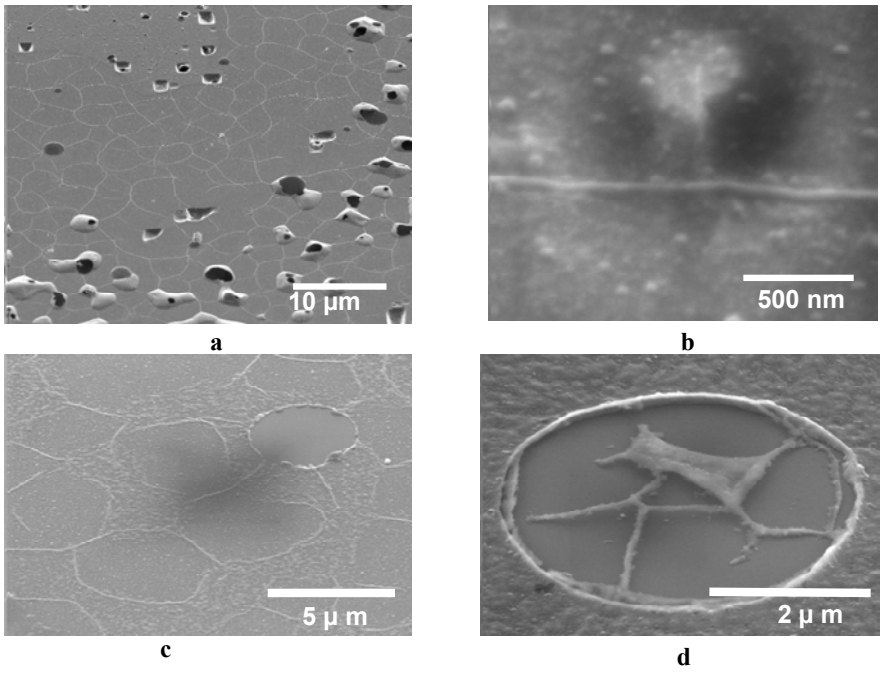


Figure 17**:** SEM images of silicon nanowire network formation. a) The white lines are the accumulation silicon and aluminum at the aluminum grain boundaries. b) A close-up SEM image at one of these wires. c) The aluminum between the wires dissolves in the top layer of the silicon substrate and surfaced up at the grain boundary forming an alloy of aluminum and silicon nanowire. d) After a long annealing time the network begins to separate it self from the silicon substrate.

Figure 18 shows the EDS profile for the sample taken at a spot on top of the wires. In (a) the higher signal coming from the oxygen indicate that most of the aluminum has turn into aluminum oxide. In (b) aluminum was etched, the small signal of the aluminum comes from the atoms incorporated inside the silicon nanowires.

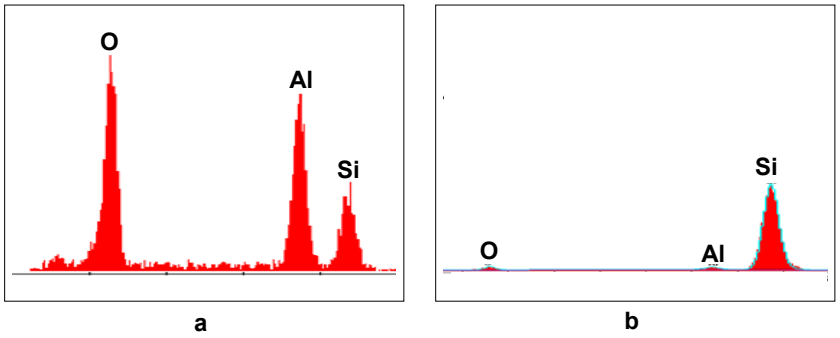


Figure 18**:** EDS profile. a) before aluminum etch and b) after aluminum etch. The small signal of aluminum indicate the incorporation of aluminum inside the silicon nanowires

- **Epitaxial silicon thin films by low temperature aluminum induced crystallization of amorphous silicon:**

As part of our objectives, we have implemented the process of metal induced crystallization into growing eptiaxial films. (100) oriented crystalline silicon (c-Si) substrate were used to grow epitaxial silicon films having the same orientation as that of the substrate. c-Si/Al/a-Si configuration is annealed at temperatures below 550°C. The Al and the a-Si layer exchange order in a process known as layer inversion. The process occurs through the diffusion of silicon into Al. The silicon resides on the substrate and keeps growing until it forms a crystalline layer. The resulting crystalline silicon thin film was examined. Epitaxial growth of the crystalline silicon thin film is evident based on x-ray diffraction (XRD), Scanning Electron Microscope (SEM) and Transmission Electron Microscope (TEM) studies. Auger depth profiling made it possible to develop a model describing the growth mechanism.

The microstructure of a plane view SEM image of a 300 nm thick silicon film produced by layer inversion on (100) oriented crystalline silicon substrate is shown in figure 19. The image was taken after annealing at 525°C for 60 minutes and etching of aluminum. The image is a representative area of the film over the entire substrate. The morphology of the film shows that it is continuous and uniform. The microstructure also reveals that there are areas where crystallization did not occur. Nevertheless, the film is very uniform.

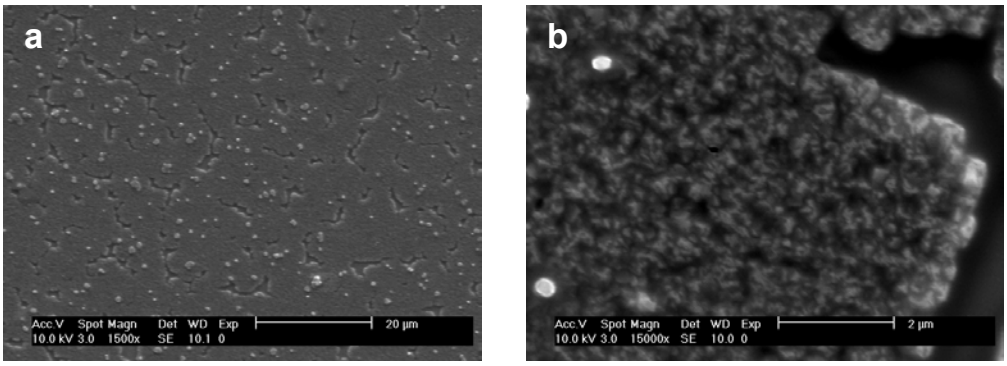


Figure 19: SEM image of the 300 nm Si thick film. The image scale is at: a) 20µm b) 2µm

To check for epitaxial nature of the film x-ray diffraction analysis was performed. The x-ray diffraction spectrum in figure 20 (a) shows the (100) silicon peak. This result indicated that the crystal structure of the crystallized film is similar to the crystal structure of the underlying substrate. The peak at 2θ ~69.4 corresponds to the (100) peak. On the contrary, a polycrystalline film with no preferred orientation would show multiple peaks at different 2θ values when the samples are grown on glass substrates as shown in figure 20 (b).

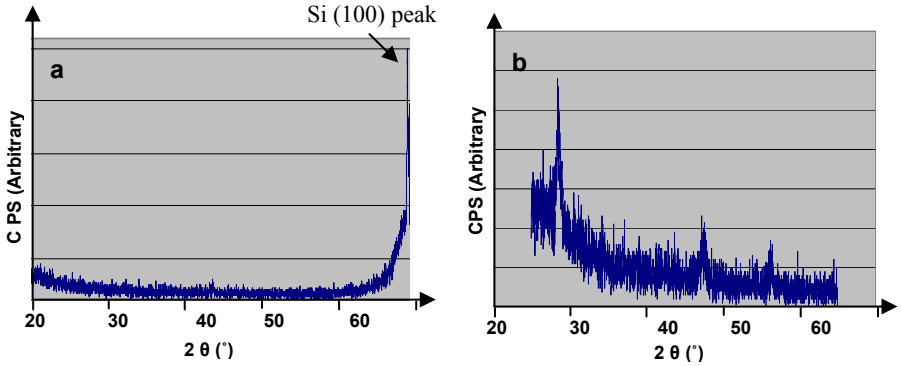


Figure 20: a) XRD results for layer inverted 300 nm thick films on (100) oriented crystalline silicon substrate. b) XRD results for the resulting films on glass.

TEM analysis of the specimen annealed at 525 for 1 hour was performed in the cross sectional mode. High resolution cross section images are shown in figure 21. Figure 21 (a) represents the interface between the film and the substrate. It was not easy to distinguish the substrate from the film at the interface. The sample was moved until a flaw at the interface was found and that's where the image was taken. Our inability to distinguish the film from the substrate at the interface was a good proof of epitaxy. The rest of the images in figure 21 clearly show the crystal fringes to be growing in the same direction starting from the interface with the substrate, through the center of the epitaxial film and towards the interface with the aluminum film. This has led to our assertion that the film is entirely epitaxial throughout its thickness.

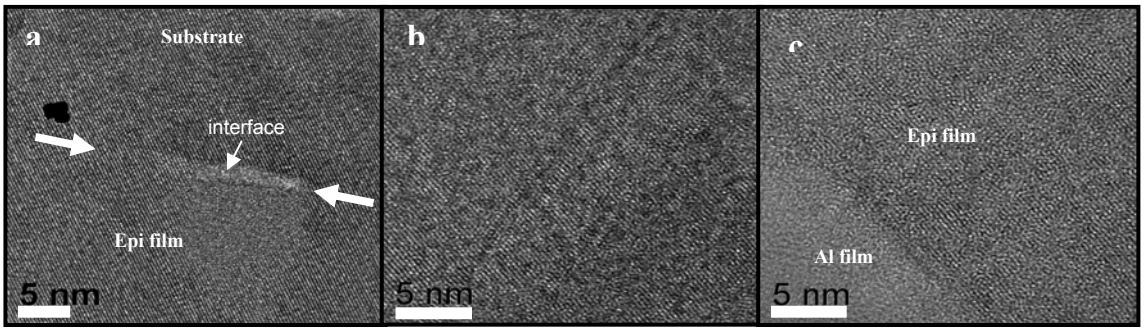


Figure 21: High resolution TEM images of the cross section at (a) The interface between the epitaxial film and the (100) silicon substrate, (b) The center of the epi film and (c) The interface between the epi film and the aluminum film showing the epitaxial nature of the film through its entire thickness.

Auger depth profiling of the configuration was studied as a function of anneal time. Figure 22 shows the depth profile at consecutive lengths of anneal time. Figure 22 (a) shows the profile of the unannealed sample. It can be noted that after 10 minutes of annealing at 525°C (Figure 22 (b)), a mixture of silicon and aluminum was formed within the entire thickness of the films. Silicon started building up at the surface of the substrate while aluminum moved towards the top. Silicon had more concentration than aluminum through the thickness. After 15 minutes of annealing, as shown in Figure 22 (c), silicon formed a film on the substrate and aluminum was being rejected towards the top. The top part of the film was still a mixture of Al and Si, but now Al has more concentration than silicon on the top part. It can be noticed in the depth profile after 30 minutes of annealing at 525°C (Figure 22(d)) that the entire bottom part of the film was only silicon and the top part was only aluminum. This means that the entire process of layer inversion in that part of the film had completed in only 30 minutes.

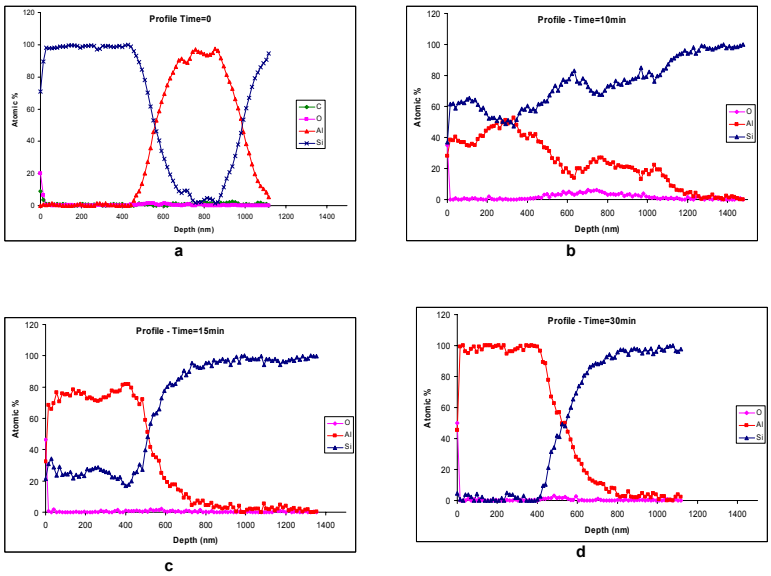


Figure 22: Auger depth profiles of the layer inversion process as a function of anneal time when 300 nm thick film of a-Si:H is deposited on top of a 300 nm thick film of aluminum and annealed at 525°C for (in minutes). a) as deposited, b) 10, c) 15 and, d) 30.

In conclusion we found that epitaxial nature of the crystalline film was obtained using hydrogenated amorphous silicon as the starting film. The process of layer inversion is observed in a-Si:H when the films are annealed at temperatures >475 °C and a complete layer inversion happens when annealing for at least 1 hour at 475°C.

- **Analytical studies of the capping layer effect on aluminum induced crystallization of amorphous silicon.**

The capping layer effect on metal induced crystallization of amorphous silicon was studied. Three sets of samples were prepared for this study. All samples had the basic layer structure of amorphous silicon layer deposited on a glass substrate. This deposition was followed by the deposition of a thin aluminum layer. In the second and third sets, however, a third layer of amorphous silicon was deposited on top of the aluminum layer. The thickness of this layer was 10% and 25% of that of the aluminum in the second and third sets, respectively. The samples were annealed at 400°C for 15, 30 and 45 minutes. The crystallization fraction in the resultant films was analyzed using XRD patterns. The surface morphology was examined using SEM and atomic force microscope (AFM). The composition analysis of the crystallized films was conducted using energy dispersive x-ray spectroscopy (EDX). TEM was used to examine the grain size in the fabricated films.

XRD measurements were performed on the samples after annealing. Figure 23 shows the X-ray diffraction patterns of the 0%, 10% and 25% samples. The samples were annealed at 400°C for 15, 30 and 45 minutes. A noticeable change in the Si (111) peak height is observed going from 15 minutes to 30 minutes in the XRD pattern in the first set. The change from 30 to 45 minutes is not as much. However, we can say in general that the crystallization is increasing with the increase of annealing time in all samples.

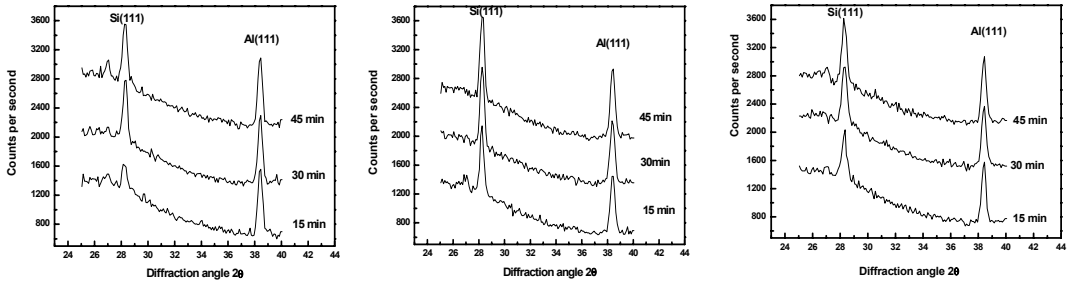


Figure 23: XRD patterns for a) 0% samples b) 10% samples c) 25% samples. All samples were annealed at 400 °C.

The 0% sample surface morphology was observed by SEM before and after annealing. SEM shows the surface features of the sample. Figure 24 shows the SEM image of the sample before annealing. It is clear from the figure that the un-annealed sample is quite smooth. This is because no interaction occurs when a-Si is in contact with any metal unless there is some excitation source such as thermal annealing.

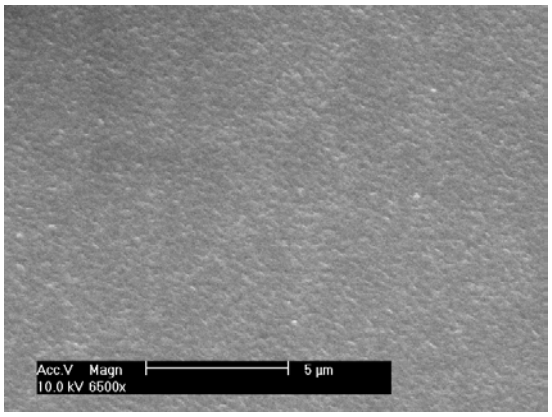


Figure 24: SEM images for 0% sample before annealing

Figure 25 shows the annealed SEM images of the 0% sample at 400°C for 15, 30 and 45 minute respectively. The white spots are silicon protrusions on the Al surface. The clue that these protrusions are composed of silicon comes from the EDX patterns taken at the two spots (The EDX patterns are not included). It is observed that the number of these protrusions is increasing with annealing time. These protrusions make the aluminum surface rough. The protrusions come from the Si layer underneath the Al film.

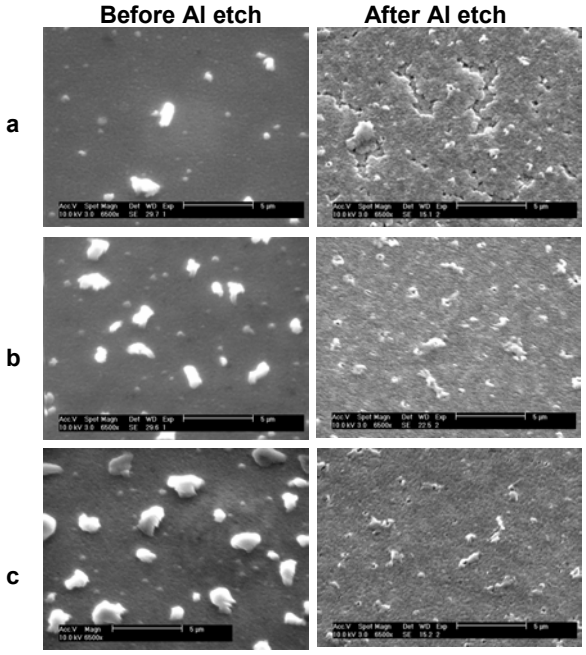


Figure 25: SEM images for 0% sample annealed at 400°C for a) 15, b) 30, and c) 45 minutes

Figure 26 Shows the SEM for the 10% sample before and after annealing. Again the Si protrusions make the aluminum surface rough.

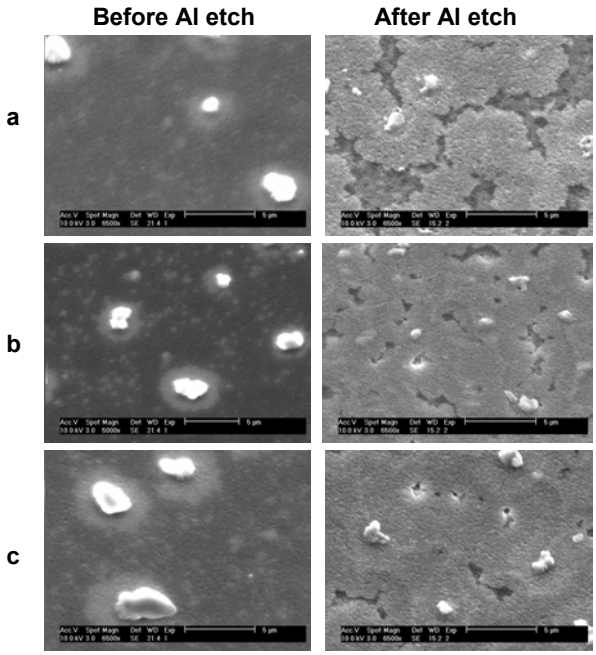


Figure 26 SEM images for 10% sample annealed at 400°C for a) 15, b) 30, and c) 45 minutes

Figure 27 Shows the SEM for the 25% sample before and after annealing. Here the silicon surface is much smoother than the silicon films in the other two sets.

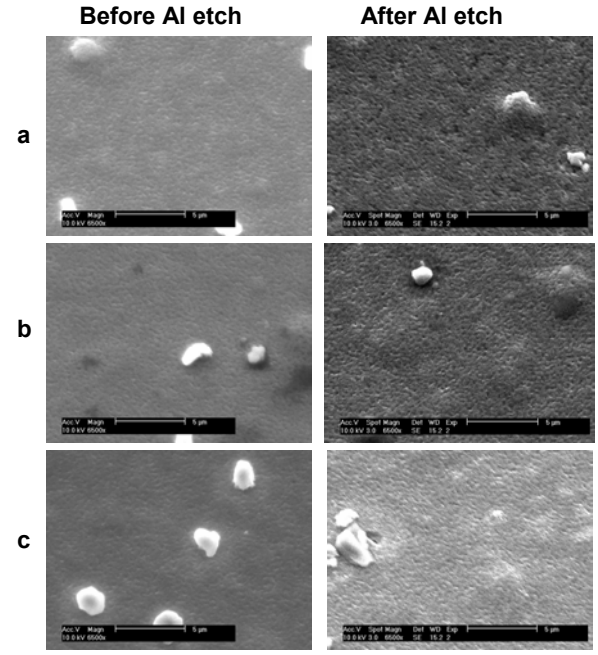


Figure 27: SEM images for 25% sample annealed at 400°C for a) 15, b) 30, and c) 45 minutes

The TEM images taken at the University of Arkansas are shown in figure 28 The grains and their boundaries can be identified from these images. The grain size of the 10% sample is higher (1.5μm) than the 0%. Also the grain size of the 25% sample is higher than 0% sample. The selected area diffraction (SAD) pattern indicating silicon rings, such as, Si(111), Si(220) , Si(311), Si(400) etc.

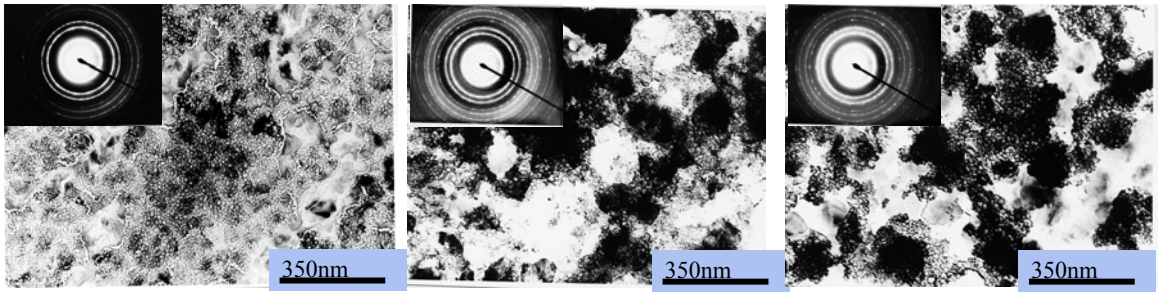


Figure 28: Comparison of the TEM images of the three samples annealed at 400°C for 30 minutes.

Finally, AFM analysis of all samples was done at the UA analytical lab. AFM was used in the tapping mode to perform surface topography measurements. A representative area of each sample was chosen to acquire an image and then analysis was performed to extract detailed information about the surface roughness. Figure 29 shows comparison of the AFM images of the three samples at 400°C for 15, 30 and 45 minutes. The average roughness (R_a) vales were found to be approximately 55, 53 and 43 nm for 0%, 10% and 25% sample respectively. The average surface roughness values decreases with the capping layer of a-Si on top of the Al.

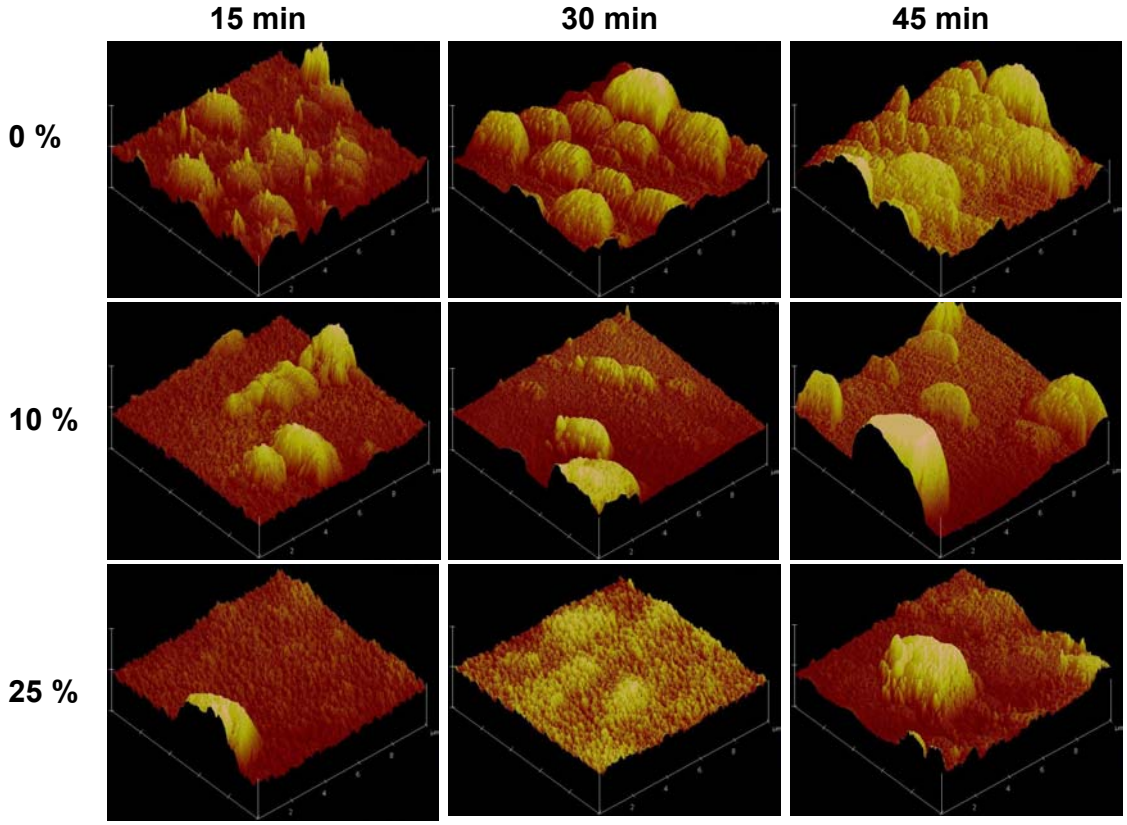


Figure 29: Comparison of the AFM images of the three samples at 400°C for various times.

- **Fabrication of poly-silicon thin films on glass and flexible substrates using laser initiated metal induced crystallization of amorphous silicon.**

Poly-silicon thin films on glass and Kapton® substrates are fabricated using laser initiated metal induced crystallization of hydrogenated amorphous silicon films. The process starts by depositing 200 nm amorphous silicon films on the substrates. Following this, 200 nm of sputtered aluminum films were deposited on top of the silicon layers. The samples are irradiated with an argon ion cw laser beam for annealing. Laser power densities ranging from 4 to 9 W/cm^2 were used in the annealing process. Each area on the sample is irradiated for a different exposure time. Optical microscopy was used to examine any cracks in the films and loss of adhesion to the substrates. XRD patterns from the initial results indicated the crystallization in the films. SEM images showed dendritic growth in the films. The composition analysis of the crystallized films was conducted by EDX.

Figure 30 shows the optical images of silicon films deposited on the Kapton substrates. These images are taken after Al is etched off. The power density was changed by changing the laser output power. The laser beam spot size was taken to be 1 mm. It can be seen form the figure that as the laser power density increase, more of the silicon grains appear. At 9 W/cm^2 the silicon film looks continuous without any cracks or peeling.

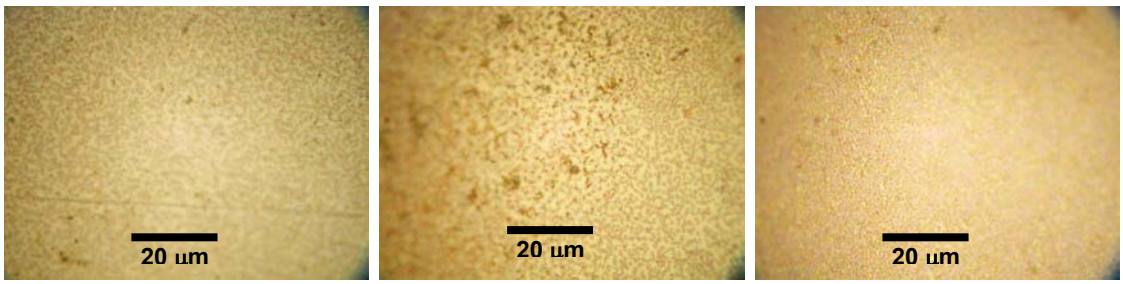


Figure 30: Optical images of silicon film on the Kapton substrates. The samples were scanned with a laser beam at: a) 4W/cm2 b) 6W/cm2 c) 9W/cm2. The scanning speed was 2.4 mm/sec.

Figure 31 shows the SEM images of the Kapton samples. The dendritic growth in the silicon films increase with increasing power density. The images show larger black spots at the Al surface compared to those in the first set. The images also indicate much more violent changes in the surface morphology of these films before Al is etched. This could be explained in terms of the higher temperature increase in the films which lead to fast formation of a thick alloy layer underneath the Al layer.

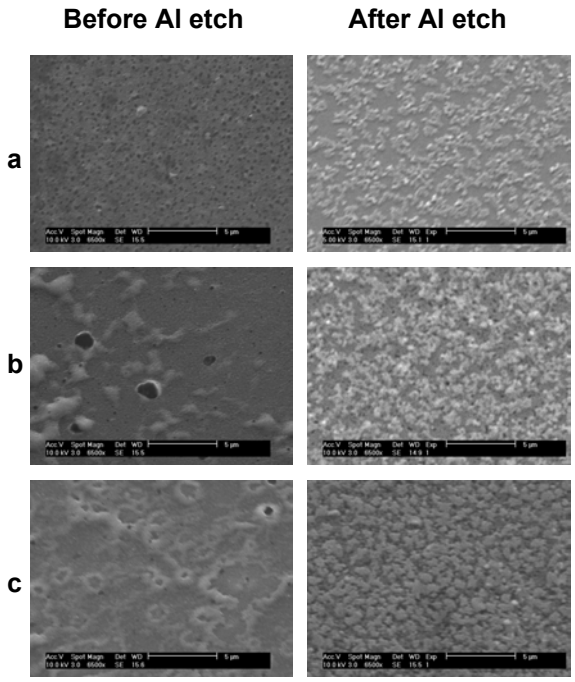


Figure 31: SEM images of the samples in the second set scanned at laser power density of a) 4 W/cm^2 b) 6 W/cm^2 c) 9W/cm^2

Schematics of the growth mechanism are shown in Figure 32. In these schematics, it is showing (Figure 32 (b)) that after prolonged laser exposure time an alloy layer will form underneath the Al layer. From this alloy layer silicon will start to nucleate and form crystalline silicon.

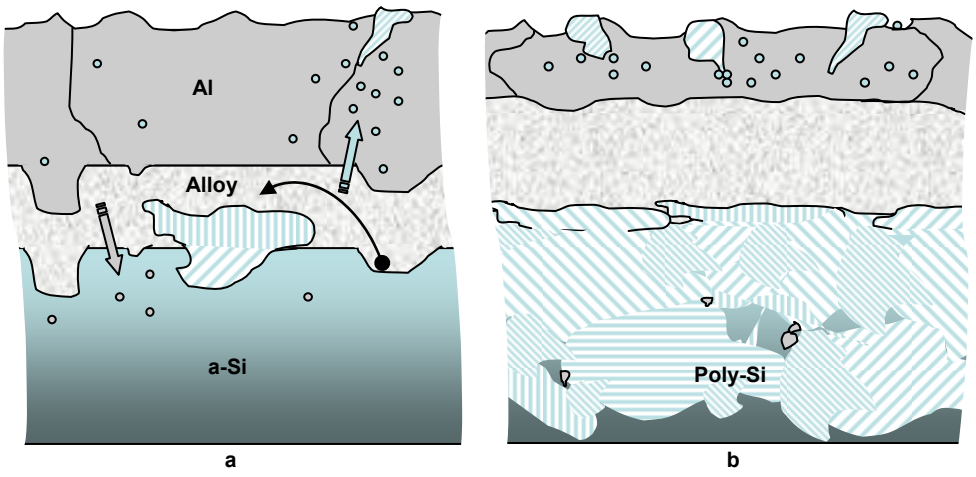


Figure 32: Schematics of the laser crystallization mechanism. The small circles indicate the aluminum in the a-Si film and the silicon in the aluminum layer. a) Just after the sample is irradiated with the laser beam, a thin alloy layer of Al and Si is formed at the interface. In this layer silicon deposits on the crystallized regions in the film. b) After prolonged periods of time continuous films are created on the substrate.

National Laboratories Partnerships:

- We have developed a strong partnership with the National Renewable Energy Laboratory (NREL). We conducted our communications with them via telephones and e-mails as preliminary planning for sending samples to them in order to be characterized using their state of the art facilities. We worked closely with M. A. Al-Jassem from NREL on TEM, AFM and SEM.

- One trip was made by Marwan Brgouthi from our group to the National Center for Electron Microscopy (NCEM) labs at Lawrence-Berkeley National Labs twice to get hands on training in using their state-of-the-art TEM. Sample preparation as well as characterization was conducted by the student under the supervision of Dr. Christian Kisielowski.

- Several visits to Oak Ridge National Laboratory (ORNL) were made by our graduate students (Maruf and Khalil) to get hands on training using Scanning Auger Microscopy (SAM) Transmission Electron Microscopy (TEM). Harry Meyer at the Microscopy, Microanalysis, and Microstructures Group, Metals and Ceramics was our point of contact.

Human Resource Development:

Undergraduate Trainees:
- Mutee Al-Rahman, Junior, U of A, worked during Spring 2002;
- Daniel Hotz: Senior, U of A
- Jon Hockenburger, REU, Bob John University worked during summer 2003.
- Patrick A. Grandt, REU, Southeast Missouri State University, worked during summer 2005 fabrication of silicon nanowires using gold droplets.
- Vincent Liu, REU, University of California, Berkeley, worked during summer 2004.

Masters in Electrical Engineering:
- Saad Abbasi
- Sampath Paduru
- Jamal Quraishi
- Sajad Al-Islam

Masters and Ph. D. Students:
- Marwan Barghouti
- Maruf Hossain

Ph. D. Students
- Khalil Sharif

Post Doctoral Fellow:

- Dr. Husam H. Abu-Safe was supported partially by the electrical engineering department and a grant from the Arkansas Center for Space and Planetary Sciences.
- The Department of Electrical Engineering provided funds to hire Dr. Li Cai as a post doctoral Research Associate (half-time).

Visiting professors:

- Dr. Ram Kishore joined our group for two months (May through June, 2005) to do TEM work on metal induced crystallization. His stay was funded through a national science foundation grant under US-India Cooperative Research: Investigating Metal Induced Crystallization on Thin Films of Amorphous Silicon.
- Dr. Adnan Shariah of Jordan University of Science and Technology spent his Sabbatical leave as a Fulbright Visiting Professor working with our group on the project.

Patents

Hameed A. Naseem, Marwan Barghoti, "Formation of Large-grain Silicon by Addition of Oxide Layer (SiO_2) in Low-temperature, Rapid Aluminum-induced Crystallization," filed with the US Patents Office, October 20, 2003.

Equipments and Instruments

- A single chamber PECVD system was commissioned for nitride deposition.
- Two turbo molecular pumps in the cluster tool that is used for thin film deposition were replaced by new and more efficient pumps.
- The silicon sputtering system was upgraded to deposit hydrogenated amorphous silicon as well.
- The turbo molecular pump of the thermal evaporator system was also replaced.
- A thermal evaporator to deposit gold was installed and maintained in our laboratory The Cluster Tool that is used for thin film deposition was equipped with laser/optical/thermal annealing chamber this summer.

Theses/Dissertations

Masters

- "**Investigation of a Si/Al/a-Si Configuration in the Aluminum-induced Crystallization of Sputtered Amorphous Silicon**", Sajad Al-Islam, Master Thesis, University of Arkansas, Fayetteville, AR, July 2005.

- "**CW laser assisted aluminum induced crystallization of magnetron sputtered amorphous silicon thin films**", Sampath K. Paduru, Masters Thesis, University of Arkansas, Fayetteville, AR, July 2004

- "**Analysis of Aluminum Enhanced Lateral Crystallization of Hydrogenated Amorphous Silicon**," M. Saad Abbasi, Master Thesis, University of Arkansas, Fayetteville, AR, May 2003.

- "**Effect of Native Oxide on Metal Induced Crystallization of Amorphous Silicon**," Marwan Al-Bargouti, Master Thesis, University of Arkansas, Fayetteville, AR, August 2003.

Ph. D.

- "**Large Grain Poly-Silicon Thin Films by Metal Induced Crystallization of a-Si:H**" Marwan Al-Bargouti, Ph.D. Dissertation, University of Arkansas, Fayetteville, AR, July 2004.

- "**The effects of hydrogen on aluminum induced crystallization of sputtered hydrogenated amorphous silicon**", Maruf Hossain, Ph.D. Dissertation, University of Arkansas, Fayetteville, AR, December 2004.

- "**Epitaxial Silicon Thin Films by Low Temperature Aluminum Induced Crystallization of Amorphous Silicon**", Khalil sharif, Ph.D. Dissertation, University of Arkansas, Fayetteville, AR, November 2005.

Refereed Journal and Proceedings Papers

Journal Papers

- Husam H. Abu-Safe, Maruf Hossain, Hameed Naseem, William Brown, and Abdullah Al-Dhafiri, "**Chlorine-doped CdS thin films from $CdCl_2$-mixed CdS powder**", Journal of Electronic Materials, Vol. 33 (2), (2004).

- Marwan Al-Barghouti, Husam H. Abu-Safe, Hameed A. Naseem, William D. Brown, and Mowafak Al-Jassim, "**The effect of an oxide layer on the kinetics of metal induced crystallization of a-Si:H**", Journal of the Electrochemical society, v **152**, n 5 G354 (2005)

- Maruf Hossain, Husam H. Abu-Safe, Hameed Naseem and William D. Brown**, "Characterization of hydrogenated amorphous silicon thin films prepared by magnetron sputtering**", Journal of Non-Crystalline Solids, **352** (1), 18-23 (2006)

- Maruf Hossain*, Husam H. Abu-Safe, Hameed Naseem, and William D. Brown, "**The Effects of Hydrogen on Aluminum-Induced Crystallization of Sputtered Hydrogenated Amorphous Silicon**", Journal of Electronic Materials, **35** (1), 113-117, (2006)

- Maruf Hosssain, Harry M. Meyer, III, Husam H. Abu-Safe, Hameed Naseem and W. D. Brown, "**Large Grain Poly-Crystalline Silicon Thin Films Prepared by**

Aluminum-Induced Crystallization of Sputter-Deposited Hydrogenated Amorphous Silicon", Journal of Material Research, **21** (3), (2006)

- Maruf Hossain, Husam H. Abu-Safe, Hameed Naseem, William D. Brown, **"Effect of stress on the aluminum-induced crystallization of hydrogenated amorphous silicon films",** Journal of Materials Research, 21(10), 2582-2586. (2006)

- Marwan Albarghouti, Hameed Naseem, Mowafak Al-Jassim, "**TEM Investigation of the Role of a Nano-oxide Layer in Aluminum-Induced Crystallization of a-Si:H",** Electrochemical and Solid-State Letters, 9(7), G225-G227. (2006)

- K. Srivastava, K. N. Sood, R. Kishore, H. A. Naseem, "**Interfacial diffusion effect on metal induced crystallization of an amorphous silicon. A microstructural pathway",** Electrochemical and Solid-State Letters, 9(7), G219-G221. (2006)

- Maruf Hosssain, Harry M. Meyer III, Husam H. Abu-Safe, Hameed A. Naseem, William D. Brown, "**The effect of hydrogen in the mechanism of aluminum-induced crystallization of sputtered amorphous silicon using scanning Auger microanalysis"**, Thin Solid Films, 510(1-2), 184-190. (2006)

- Husam H. Abu-Safe, "**Investigation of the multi-conversion processes in periodically poled $LiNbO_3$ based optical parametric oscillators**", Applied Optics. 44 (34), 7458 (2005)

- Husam H. Abu-Safe, "**Difference frequency mixing of strongly-focused Gaussian beams in periodically poled $LiNbO_3$**", Appl. Phys. Lett v 86, n 23, 231105 (2005)

<u>Proceeding Papers</u>

- Sampath K. Paduru, Husam H. Abu-safe, Hameed A. Naseem, Adnan Al-Shariah, and William D. Brown, "**CW Argon-Ion Laser Initiated Aluminum Induced Crystallization of Amorphous Silicon Thin Films**", Mat. Res. Soc. Symp. Proc. Vol. 808, A4.5.1 (2004).

- Maruf Hossain, Husam Abu-Safe, Marwan Barghouti, Hameed Naseem, and William D. Brown, "**The Effect of Substrate Temperature and Interface Oxide Layer on Aluminum Induced Crystallization of Sputtered Amorphous Silicon**", Mat. Res. Soc. Symp. Proc. Vol. 808, A4.22.1 (2004).

- Mohammad Saad Abbasi, Husam Abu-Safe, Hameed Naseem, and W.D. Brown, "**Modeling of Aluminum Induced Lateral Crystallization of Hydrogenated Amorphous Silicon**", Mat. Res. Soc. Symp. Proc. Vol. 808, A4.26.1 (2004).

- Vincent H. Liu, Husam H. Abu-Safe, Hameed A. Naseem, and William D. Brown, "**Fabrication of silicon nanowire network in aluminum thin films**", Materials Research Society Symposium Proceedings, **862**, 363-368 (2005)

- Marwan A. Albarghouti, Husam H. Abu-Safe, Hameed A. Naseem,William D. Brown, Mowafak M. Al-Jassim, and Kim M. Jones, "**Large grain poly-Si thin films by metal induced crystallization of a-Si:H**", **31st** IEEE Photovoltaic Specialists Conference proceedings, 1070-1073 (2005)

- Maruf Hossain, Husam H. Abu-Safe, Hameed Naseem, William D. Brown and Harry Meyer. "**Aluminum induced crystallization of sputtered hydrogenated amorphous silicon for economically viable thin film silicon solar cells"**, **31st** IEEE Photovoltaic Specialists Conference proceedings, 1088-1091 (2005)

- Hameed A. Naseem, Husam H. Abu-Safe, Khalil Sharif, Marwan A. Albarghouti, Maruf Hossain, William D. Brown, and Ram Kishore, "**Comprehensive study of the mechanism of metal induced crystallization of hydrogenated amorphous silicon thin films**", IWPSD-2005 conference proceedings, **IWPSD-2005**, 1279-1286, (2005)

- R. Kishore, S. Hata, N. Kuwano, Y. Tomokiyo, H. A. Naseemand W.D. Brown, "**In-situ TEM Investigation of Aluminum Inducred Crystallization of Sputtered Amorphous Silicon**", IWPSD-2005 conference proceedings, **IWPSD-2005**, (2005)

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