Structural study of micro-crystalline silicon thin films deposited on flexible substrates with different thicknesses

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Microcrystalline silicon(μc-Si) thin films were deposited on the AZO/TPT flexible substrates by RF-PECVD. Effects of film thickness on the structure of thin films were investigated by XRD, SEM and Raman.The results indicated that uniform dense microcrystalline silicon thin films can be prepared by RF-PECVD. As thickness increases, the silicon thin film transitions from amorphous structure to microcrystalline structure. When the thickness reaches 493nm, the film begins to crystallize. However, the surface morphology deteriorates when the thickness reaches 882nm, which is reflects by poor surface smoothness and significant differences in grain size. XRD spectrum occurred (111), (220) and (331) peak, grain size and crystalline volume fraction increased along with film thickness increased, arrived at 82%.

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1. Introduction

Microcrystalline silicon (μc-Si) is a complex multiphase material composed of amorphous silicon and discrete micro crystalline phases. It has been widely studied and applied in the field of photovoltaics and other semiconductor devices due to its excellent optoelectronic properties and stability [1-5]. Flexible opto-electronic devices are suitable for various shapes and sizes of application scenarios. For instance, solar cells prepared on flexible substrates are more likely to achieve a perfect combination of photovoltaics and buildings, so research on microcrystalline silicon thin film materials deposited on flexible substrates is important and has practical value [6-10]. However, due to the fact that most flexible substrates are not resistant to high temperatures, and their thermal expansion coefficients do not match those of microcrystalline silicon, as well as their poor adhesion to thin films, the quality of thin films prepared on them needs to be improved.

RF-PECVD is the main method for preparing high-quality thin film materials in industry, and it is also the most common preparation method for microcrystalline silicon thin films. The deposition process conditions have a significant impact on the quality of thin films. At present, a large amount of research is focused on the effects of substrate temperature, RF power, deposition pressure, silane concentration, and other parameters on microcrystalline silicon thin films [11-13]. However, the thickness of the film also has a significant impact on the structure and properties of the film material. While many studies focus on process parameters, the effect of thickness evolution on flexible substrates is less studied, which is the focus of this work. This article focuses on studying the structure of μc-Si films deposited on AZO/TPT flexible substrates with different thicknesses. TPT films are a kind of composite materials and are usually used as encapsulation material for solar modules.

2. Experiment

The μc-Si thin films were prepared on AZO/TPT substrates by PECVD method, with reaction gases of SiH_4 and H_2. TPT material is a kind of composite materials and AZO/TPT is prepared in our former paper [14]. The experimental conditions are as follows:120℃ (substrate temperature),133Pa (pressure), 2.3×10^{-4}Pa (vacuum degree, base pressure), 30, 60, 90, 120 and 150min (deposition time), 2% Silane concentration (SiH_4/SiH_4+H_2), 50w (power). The film thickness was measured using a grating spectrometer (omn-λ300); The structure and average grain size of the thin film were analyzed using an X-ray diffractometer (X'Pert PRO, CuK α, λ=0.154 nm); Scanning electron microscopy (Sirion 200) was used to analyze the surface morphology of the thin film, with a magnification of 80000 times, and EDAX was used to analyze the composition of some particles; Analyze the

crystallinity of microcrystalline silicon thin films using a Raman spectrometer (model FRA 106/s).

3. Results and discussion

3.1. Thickness of μc-Si thin films

The thickness of μc-Si thin films was measured using a grating spectrometer and calibrated using a step meter. The film thickness was calculated based on the adjacent peak (or valley) wavelengths of the transmittance curve by the equation [15]: $d = \frac{\lambda_1 \lambda_2}{2n(\lambda_1 - \lambda_2)}$, n=3.3 (refractive index of the material). The film thickness and average growth rate obtained through calculation for different deposition times are shown in Table 1. It can be seen that the growth rate of the thin film is relatively low, around 0.1nm/s.

Table 1. Thickness and of thin films with various deposition time

Deposition time (min)	150	120	90	60	30
Film thickness (nm)	882	655	493	364	218
Deposition rate (nm/s)	0.098	0.091	0.091	0.10	0.12

3.2. Structural and morphological analysis

Fig. 1 shows the XRD patterns of thin films with different thicknesses. Crystallization peaks appear when the film thickness is above 493nm. As the thickness increases, the crystallization peaks become more pronounced, and the (111), (220), and (331) peaks of silicon appear. And when the film thickness is below 364nm (no crystallization peak appears in the XRD pattern of the sample at deposition times of 30min and 60min), the deposited film is amorphous silicon. According to the Debye Scherrer formula [16]: $D = \kappa\lambda / (\beta \cos\theta)$, the average grain size of thin films was calculated, the grain sizes were 148.2nm, 102.5nm and 63.6nm for μc-Si thin films with thickness of 882nm, 655nm and 493nm, respectively.

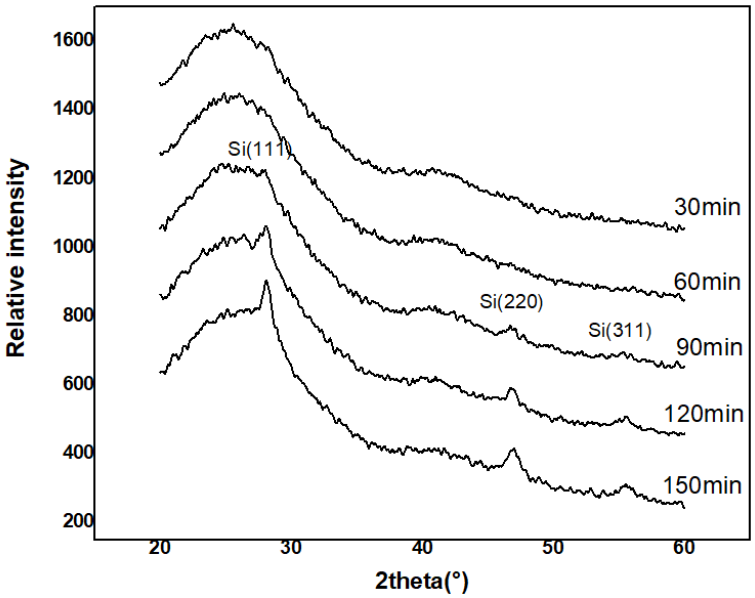
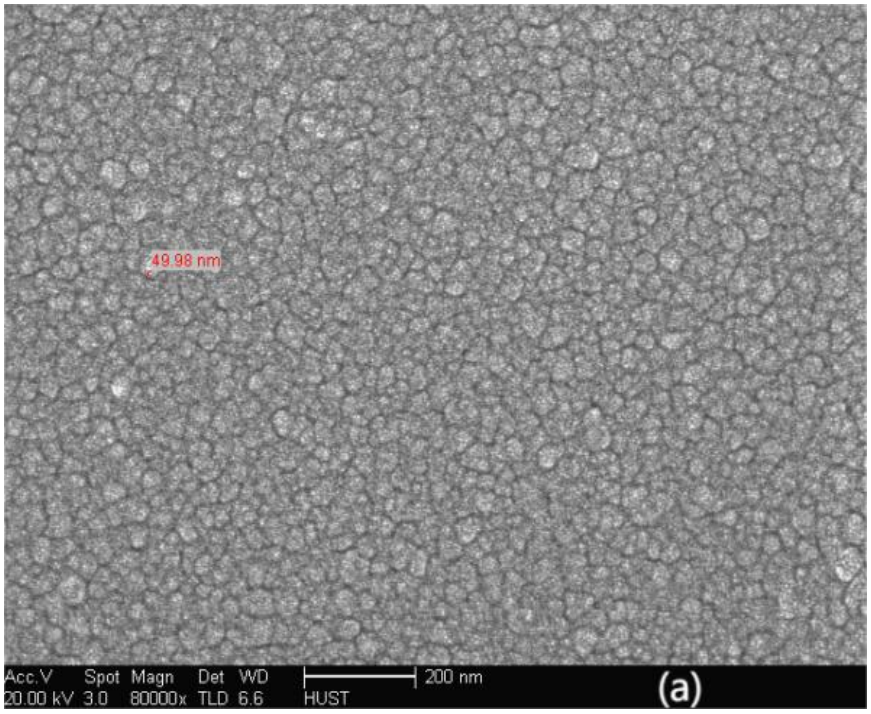


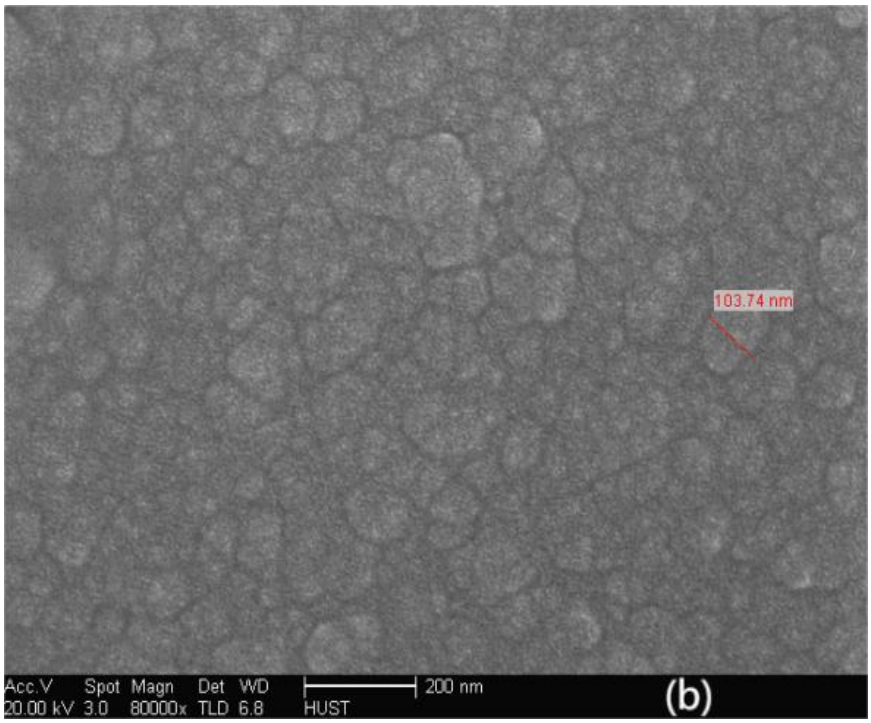
Fig. 1. XRD of the samples with various deposition time

In order to further understand the surface morphology and composition of the prepared thin films, SEM tests were conducted on the relatively thin (deposition time of 90 minutes) sample with just appeared crystallization peak and the microcrystalline silicon sample with good crystallinity (deposition time of 150 minutes). As shown in Fig. 2a, the thin films with a deposition time of 90 min were a structurally dense and flat microcrystalline silicon

material with a uniform particle size of approximately 50nm, which is consistent with the results obtained by XRD.

However, the thicker sample with deposition time of 150 min have poor surface smoothness, significant differences in grain size, with larger ones exceeding 100nm, and obvious particle accumulation on the surface (Fig. 2 b and c), which may be due to surface contamination. EDAX energy spectrum analysis was performed on the accumulated particles (Fig. 2d). It can be seen that the particles contain C, O, Al, and Ba except for Si element. C and O elements may be caused by insufficient vacuum during the deposition process or contamination during sample storage, while the presence of aluminum and barium elements is more puzzling, perhaps due to contamination of the electrodes or chambers.





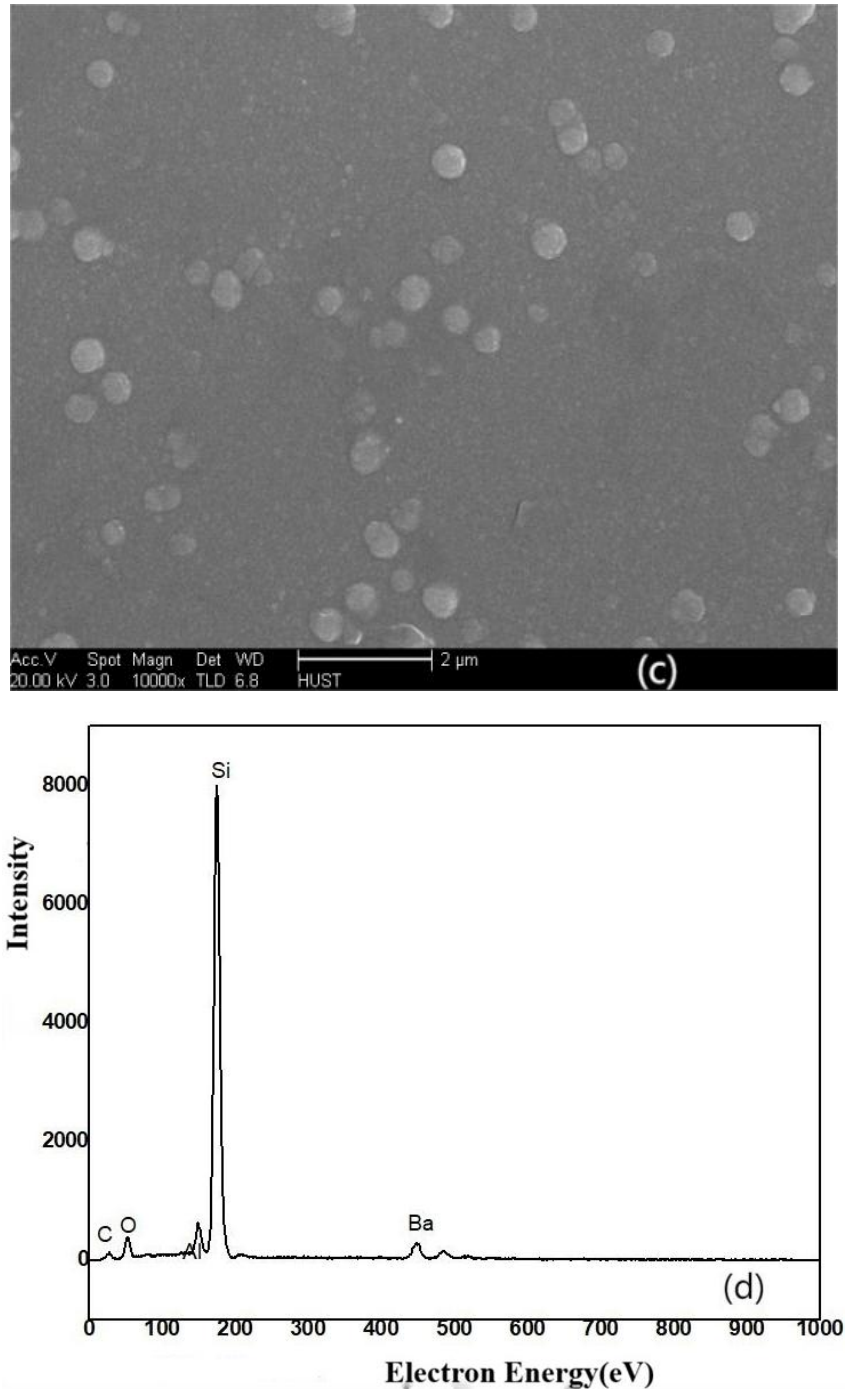


Fig. 2. SEM images of thin films with 90min deposition time(a) and 150min deposition time(b) and(c) and EDAX of accumulated grain(d) (colour online)

Raman crystallization ratio Xc of the material can be obtained based on the Raman spectrum of silicon thin films. The peak near 520 cm^{-1} corresponds to the Raman scattering component of crystal particles. The peak near 510 cm-1 corresponds to the Raman scattering component caused by the bond angle expansion of grains with a size of only a few nanometers and grain boundaries. The peak near 480 cm^{-1} corresponds to the Raman scattering component of amorphous silicon [17]. Fig. 3a shows the Raman spectrum of microcrystalline silicon with a film thickness of 882 nm (deposition time 150 min). The film exhibits a distinct and sharp peak near 520 cm^{-1}. By performing Gaussian fitting on the Raman spectrum (Fig. 3b), the crystallization rate of the film is determined to be 82% according to the formula $Xc=I_C/(I_C+I_A)=(I_{520}+I_{510})/(I_{520}+I_{510}+I_{480})$ [18] (where Ic and Ia refer to the integrated intensities, respectively). However, when conducting Raman testing on a microcrystalline silicon film with a thickness of 218 nm (deposition time 30 min), no peak was observed near 510 cm-1, indicating that the film is amorphous, which is consistent with the observation results from XRD.

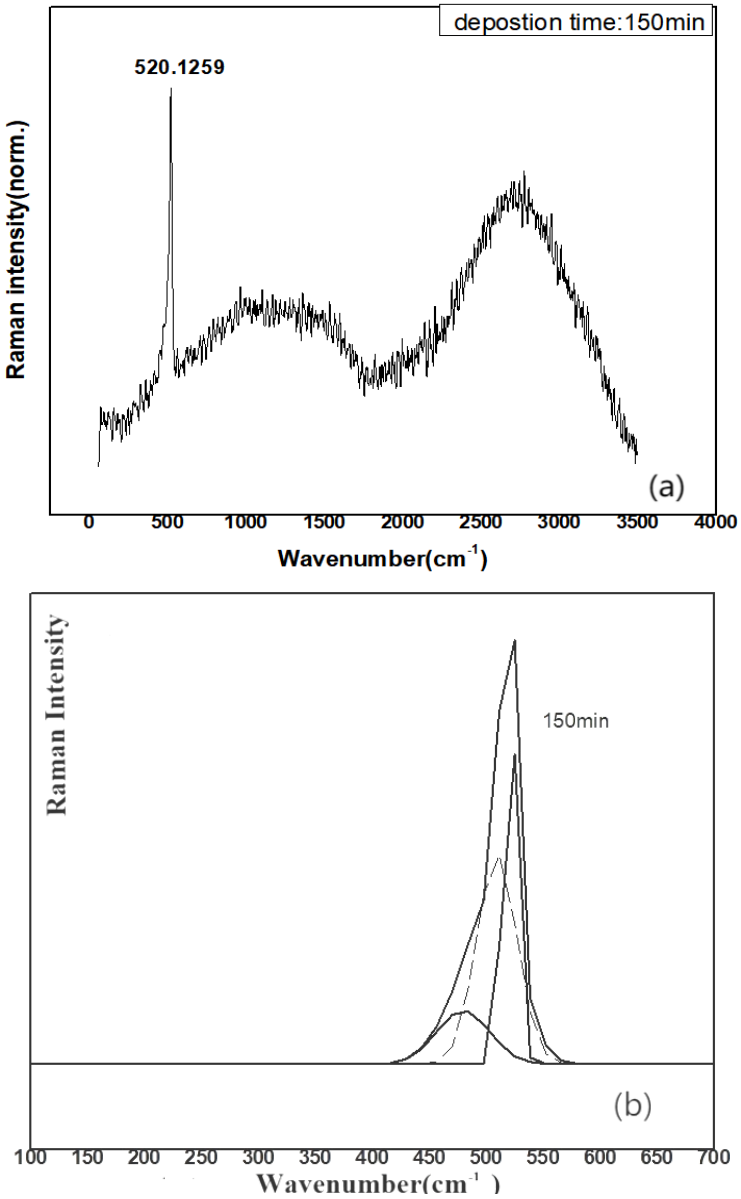


Fig. 3. Raman spectrum of 882 nm μc-Si thin film (a) and Gaussian fitting curve (b)

4. Conclusions

In this paper, Uniform and dense microcrystalline silicon thin films were prepared by RF-PECVD on AZO/TPT flexible substrates. As the thin film thickness increases, the thin film transitions from amorphous structure to microcrystalline structure, showing crystallization peaks at (111), (220), and (331), and the intensity of these crystallization peaks gradually increases. The grain size and crystallization rate of microcrystalline silicon increase significantly with increase of film thickness, and a maximum crystallization rate of 82% are obtained. This study validates the feasibility of using RF-PECVD to deposit uniform and dense microcrystalline silicon thin films on flexible AZO/TPT substrates, and provides essential insights into the phase transition from amorphous to microcrystalline structures, offering a crucial process reference for the development of flexible optoelectronic devices.

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