

POLYCRYSTALLINE Si FILMS PREPARED BY Al- AND Ni- INDUCED CRYSTALLISATION

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Metal induced crystallization of amorphous Si (a-Si) has been studied for differently deposited structures and metal concentrations - glass/a-Si/Al, glass/Al/a-Si, glass/Al/a-Si+Al and glass/a-Si+Ni. The films were deposited at various substrate temperatures (T_s) by RF magnetron sputtering, and the structures were isothermally annealed at temperatures below that of the a-Si solid phase crystallization. The annealing was implemented in air, vacuum, N_2 , N_2+H_2 or H_2 . Raman spectroscopy, X-ray Diffraction Spectroscopy and Rutherford Back-Scattering were used to study the crystallinity of the resulting polycrystalline Si (poly-Si) films. The influence of the technological conditions on the structure of the films was studied. The results indicate that the structure of the poly-Si films is improved when the annealing is performed in an atmosphere containing H_2 . It is supposed that H_2 stimulates the crystalline grain growth during annealing, by increasing the diffusion rates of Al and Si. A mechanism for metal induced crystallization is suggested to explain the results.

(Received December 9, 2004; accepted January 26, 2005)

Keywords: Polycrystalline Si films, Raman spectroscopy, Metal Induced Crystallization

1. Introduction

Polycrystalline Si has been intensively investigated as a material for thin film transistors (TFT) and solar cells [1,2]. Usually, amorphous Si (a-Si) thin films can be transformed into poly-Si films by solid phase crystallization (SPC) or laser crystallisation [3,4]. However, these methods have disadvantages in large-scale manufacturing - expensive equipment, high temperature processing, expensive substrates for deposition of a-Si, etc. In comparison, the process of Metal Induced Crystallisation (MIC) of a-Si has attractive advantages – a low crystallization temperature, utilization of large scale cheap foreign substrates and simple equipment. It is well known that metal impurities in a-Si can induce its crystallization at annealing temperatures lower than the eutectic temperature of the corresponding metal-Si pair [5-13]. In reported studies, various metals (Al, Ag, Au, Cu, Ni, Pd, Pt) have been used to induce the low temperature crystallisation of a-Si and a-Si:H films by MIC [1,2,14,15]. The results of the reaction between the metal and the Si (crystalline or amorphous) can be a silicide or an amorphous alloy, depending on the metal-Si pair [5,9] After the isothermal annealing of a-Si in contact with different metals, large grain (micron scale) poly-Si can be obtained [6, 8]. Usually, the crystallisation of the structures is performed in air, vacuum or under nitrogen [14-18]. The properties of the resulting polycrystalline Si films have so far been investigated mainly in order to study the process of crystallization and the influence of the metal layer structure on it. No attention has been focused on the influence of the atmosphere, in which the crystallisation is performed [2,14-18].

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In this study, results on the Al- and Ni-induced crystallisation of a-Si deposited by RF magnetron sputtering under isothermal annealing in air, N₂, H₂+N₂, H₂ or vacuum are presented. The influences of different technological parameters - substrate temperature, metal concentration and annealing atmosphere - on the crystallinity of the poly-Si films are investigated. The results obtained are explained in terms of a MIC mechanism.

2. Experimental details

For the preparation of polycrystalline films, the structures glass/a-Si/Al, glass/Al/a-Si, glass/Al/a-Si+Al and glass/Al/a-Si+Ni were deposited. The technological parameters of the deposited structures are given in Table 1.

Table 1. Technological parameters of the investigated samples.

Notation	Structure	T_s , [°C]	Thickness [nm]	Annealing atmosphere	T_{an} , [°C]	t_{an} , [h]
A	gl/a-Si/Al	250	200	air	500	4
B	gl/a-Si+Al	350	300	vacuum	500	3
C	gl/Al/a-Si+Al	RT; 350; 500	200	vacuum	500	3
D	gl/a-Si+Ni	150; 250; 350	800	air	560	6
E	gl/Al/a-Si	250	150	N ₂ , N ₂ +H ₂ H ₂ ,	480, 500, 530	2-7

The a-Si for samples A (Fig. 1a) was deposited on glass substrates by RF magnetron sputtering of a c-Si (p-Si, 9-12 Ω.cm) target. The Al was deposited by sputtering on top of the a-Si film. The thickness of both layers was 200 nm. The substrate temperature (T_s) during the depositions of a-Si and Al was kept at 250 °C. These samples were isothermally annealed in air (sample A) and in N₂ (sample B) at 500 °C for 4 h. Samples C were prepared by evaporation of an Al layer on glass at room temperature (RT). On top of the Al, an a-Si+Al film was deposited at three different T_s - RT, 350 or 500 °C by RF magnetron co-sputtering of a c-Si (p-Si, 9-12 Ω.cm) target partially covered with Al chips. The Al content is defined as the ratio of the Si (A_{Si}) and the Al (A_{Al}) areas on the target - $\chi = A_{Si}/A_{Al} = 51$. The thickness of both layers was 200 nm. The samples C were annealed in vacuum at 500 °C for 3 h. The samples D were deposited on glass substrates by RF magnetron co-sputtering of a c-Si (p-Si, 9-12 Ω.cm) target partially covered with Ni chips. The ratio of the Si (A_{Si}) and Ni (A_{Ni}) areas on the target, $\eta = A_{Si}/A_{Ni}$, was varied between 34 and 109. Thus, different Ni concentrations in the films were achieved. The substrate temperature was kept at either 150, 250 or 350 °C. The thickness of the samples was 800 nm. The a-Si:H films for the samples E were deposited at $T_s = 250$ °C in an Ar atmosphere, at 0.5 Pa. The thicknesses of both layers, Al and the a-Si, were equal - 150 nm.

Different annealing atmospheres were used: air, vacuum, N₂, forming gas (N₂+H₂) and H₂. Annealing in different gas atmospheres under atmospheric pressure was done in a horizontal oven. The top Al layer, obtained after the aluminium induced crystallisation of the samples B, C and E was selectively etched before the experimental investigations were performed. The structures of the samples were studied by Raman or micro-Raman scattering and optical microscopy (OM). Raman spectra were excited by the 488 nm line of an Ar⁺ laser using a SPEX 1403 spectrophotometer. In the case of micro-Raman measurements, 1µm laser spots were applied, using RENISHAW equipment. The Raman spectra presented in each figure below were measured under the same conditions.

3. Results

3.1 Ni Induced Crystallisation (NIC)

The results of the Rutherford Back-Scattering (RBS) measurements show that upon changing η from 109 to 34, the Ni concentration (C_{Ni}) in the co-deposited a-Si+Ni films varied from

10.5 to 21 at.%. The elemental composition of the layers was found to be 77-80 at.% Si, 2-5.5 at.% O₂ and 3-4 at.% Ar₂.

Fig. 1 displays the Raman spectra of glass/a-Si+Ni samples with different Ni contents (C_{Ni} = 15 and 21 at.%) deposited at different T_s and annealed at 560°C for 6 h. The spectra have a Si-Si TO-like band at about 518-519 cm⁻¹, due to the poly-Si phase. The film stress was estimated to be about 500-250 MPa. The broad shoulder at about 470 cm⁻¹ indicates the presence of an a-Si phase together with the poly-Si one. The position of the Si-Si TO-like band and its full width at half maximum (FWHM) (12 cm⁻¹) do not change with increasing T_s . A comparison of the spectra

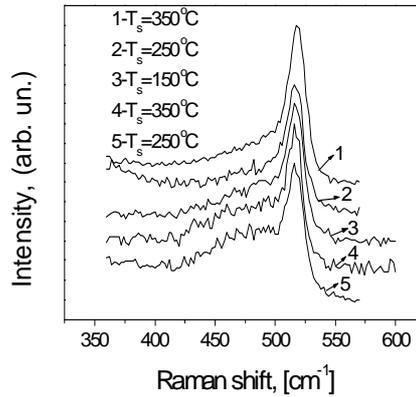


Fig. 1. Raman spectra of samples with different Ni concentrations - 21 at.% (1,2,3) and 15 at.% (4,5), at $T_{an}=560^\circ\text{C}$, $t_{an}=6\text{h}$. Raman intensities are in linear scale.

in Fig. 1 shows that the quantity of the amorphous phase in the films decreases with increasing Ni concentration. The Raman spectra can be fitted by two Gaussian bands at 518 and 470 cm⁻¹, corresponding to the crystalline and amorphous phases [19]. It is found that the degree of crystallinity increases from 0.17 to 0.56 when T_s increases from 150 to 350°C for the samples with $C_{Ni} = 21$ at.%. The value of r is lower for films with lower C_{Ni} (15 at.%), and also increases from 0.05 to 0.2 with increasing T_s . Films with lower C_{Ni} (< 15 at.%) do not crystallize under the same annealing conditions. Their Raman spectra (not shown here) correspond to those of a-Si. No crystallization is observed for a-Si+Ni films with $C_{Ni} > 15$ at.%, after annealing at lower temperatures.

3.2 Al Induced Crystallization (AIC)

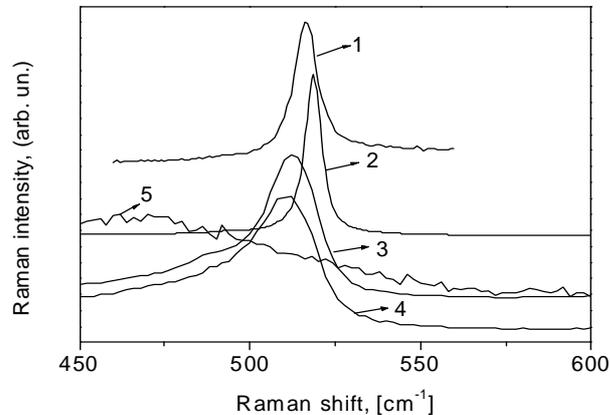


Fig. 2. Raman spectra after annealing of samples: glass/a-Si/Al annealed in air at $T_{an} = 500^\circ\text{C}$, $t_{an} = 4\text{h}$ (1), glass/a-Si+Al with $\chi=51$, annealed in vacuum at $T_{an}=500^\circ\text{C}$, $t_{an}=3\text{h}$ (2), glass/Al/a-Si+Al with $\chi=51$, deposited at different T_s - RT (3), 350°C (4) and 500°C (5), annealed in vacuum at $T_{an}=500^\circ\text{C}$, $t_{an}=3\text{h}$ (3,4,5). The Raman intensities are displayed on a linear scale.

The elemental composition of the glass/a-Si/Al samples, annealed in air, according to the RBS results is: Si - 55-60 at.%, Al - 10-15 at.%, O₂ - 25-30 at.%. Raman spectra of different samples obtained by AIC are shown in Fig. 2. The spectra of the samples glass/a-Si/Al annealed in air (curve 1) and in N₂ (curve 2) at 500 °C for 4 h. show Si-Si TO-like bands at 516-517 cm⁻¹ and indicate the presence of a crystalline phase. The shift of this band in comparison with the peak position in the Raman spectrum of c-Si (520 cm⁻¹), measured under the same conditions, can be explained by the tensile stress in the poly-Si. The film stress can be estimated to be 750-1000 MPa, from the wave number shift of the LO-TO peak compared to the Raman LO-TO peak of stress free c-Si, according to [20]. The FWHM of the Si-Si LO-TO-like band in the spectra of these samples (Fig. 2, curves 1 and 2) is 8 cm⁻¹.

Raman spectra of glass/Al/a-Si+Al samples with $\chi=51$ deposited at different T_s are shown in Fig. 2 curves 3-5. The samples in this set, deposited at lower T_s values (RT and 350 °C), have a Si-Si TO-like band together with a shoulder due to the amorphous phase. The position of the Si-Si TO Raman peak depends on T_s and shifts to lower wave numbers when compared to the layered structures (512 cm⁻¹ at RT and 510 cm⁻¹ at $T_s=350$ °C). The poly-Si film stress is estimated to be about 2000-2500 MPa. The FWHM of the Si-Si LO-TO-like band increases with increasing T_s (18 cm⁻¹ at RT and 20 cm⁻¹ at $T_s=350$ °C). For the sample co-deposited at $T_s=500$ °C, one observes only a broad amorphous band at about 470 cm⁻¹. The Raman spectra of the samples (Fig. 2, curves 4 and 5) can be fitted by three Gaussian bands at 512, 500 and 470 cm⁻¹. According to the procedure in [19], the crystalline volume fraction is determined from the ratio $r = I_{mc}/(I_{mc}+I_a)$, where $I_{mc} = I_{512}+I_{500}$ and $I_a=I_{470}$ denote the areas under the Gaussian peaks. These films have a crystalline volume fraction, r , of about 0.66.

3.3 Influence of the annealing atmosphere

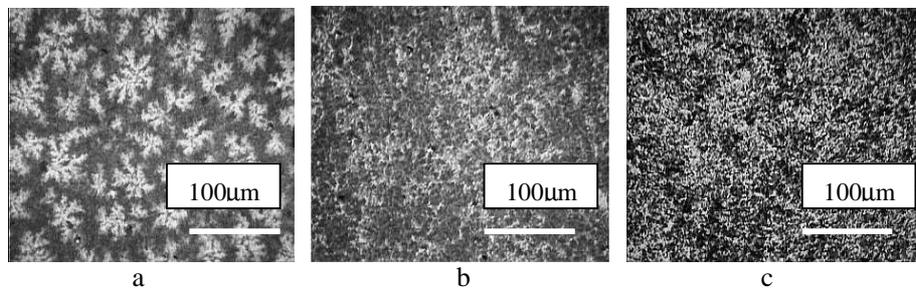


Fig. 3 Optical images of poly-Si films after annealing of the structure glass/Al/a-Si at 500 °C in: N₂ for 6h (a); N₂+H₂ for 5h (b); H₂ for 3h (c) atmospheres.

The optical micrographs of poly-Si films annealed in different ambiances are shown in Fig. 3. The poly-Si, obtained by annealing in N₂ (Fig. 3a) have large grains (dendrites), with 20-30 µm size, separated by inter-crystalline spaces of about 10-20 µm. When annealing is performed in an atmosphere containing H₂ (Fig. 3b and c), the surface of the obtained poly-Si is very smooth. The grains are very closely packed and continuous poly-Si films are obtained. The size of the inter-grain space decreases to about 2-3µm. The polycrystalline Si surface in these cases is more uniform. In order to analyse the grain orientations shown in the OM-images, X-ray Diffraction Spectroscopy (XRD) measurements have been performed. In Fig. 4, the XRD-pattern of a poly-silicon film is shown. It is evident that the crystallographic peak of the silicon is (111).

The crystallinity of the samples after the annealing was investigated by Micro Raman spectroscopy. The Micro Raman spectra of poly-Si layers prepared by isothermal annealing in different atmospheres at 500 °C are shown in Fig.5. Two spectra were measured for each sample – on the grain surface and between the grains. All of the spectra of the annealed samples have a Si-Si TO-like phonon band centered in the range 520-521.3 cm⁻¹, showing the presence only of the Si crystalline phase. The Micro Raman spectrum taken from the grain (Fig. 5, curve1a) of the poly-Si film, prepared by annealing of the structure Al/a-Si in N₂ for 6 hours has a band centered at 520 cm⁻¹, with a FWHM of 10 cm⁻¹. When the annealing is performed in an atmosphere of forming gas

(N_2+H_2) for 5h, the positions of both Raman peaks, from the grain surface and from the inter grain material, (Fig.5, curves 2a and 2b) are centered at 521cm^{-1} , with a decreased FWHM of 7cm^{-1} and 7.5cm^{-1} respectively. The most narrow Si-Si TO-like band centered at 521.3cm^{-1} with a FWHM of 7cm^{-1} (Fig. 5 curve 3a, 3b) is demonstrated by the sample annealed in H_2 for both grain and

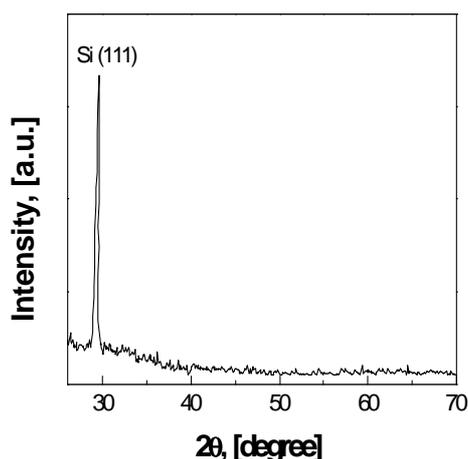


Fig.4. X-ray diffraction spectrum of a poly-Si film.

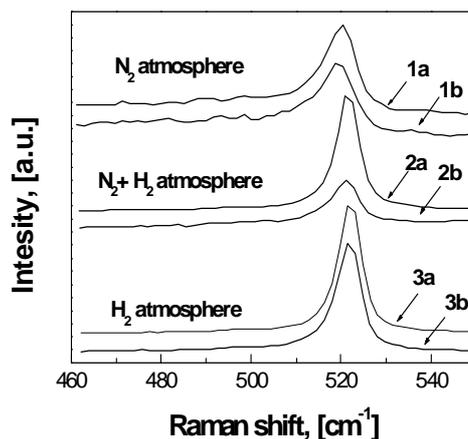


Fig. 5. Micro Raman spectra of samples of glass/a-Si/Al, measured at intra-grain (a) and inter-grain (b) positions, annealed in different atmospheres.

inter-grain material. The results demonstrate that annealing in the presence of H_2 results in an improvement of the structure of both the grain and inter-grain materials.

An estimate of the grain size can be deduced from both the red shift and the FWHM of the Raman peak [21]. It should be noted that accurate values for the grain size cannot be determined from the relationships described previously in the literature [21,22], as they depend on the structures of the grains and their boundaries. Despite this, comparisons between spectra obtained from similar materials are valid. The grain size is inversely proportional to the FWHM of the peak [23], so it can be deduced that the grain size increases when the annealing has been performed in the presence of H_2 , as confirmed by the OM pictures.

3. Discussion

The results obtained can be explained on the basis of the model of MIC [3, 6, 11]. The process of MIC starts with an interaction between the metal atoms and the Si atoms. The difference in the electronegativity between the metal and the Si leads to redistribution of the electric charge of the metal-Si bonds closer to the metal atoms, and this changes the position of the electrons around the neighbouring Si-Si bonds. The electron pairs from electrons belonging to two neighbouring Si atoms transform to electrons belonging to more than two neighbouring Si atoms, i.e., the electrons in the a-Si are no longer localized; they occupy delocalized orbitals. This changes the distance between the Si atoms, because the Si-Si bonds weaken. The metal phase, being responsible for loosening the covalent bonding in Si, makes the amorphous phase much less metastable or even unstable. The interface boundary between the metal and the a-Si layer is appropriate for a metal-Si interaction leading to the process of a-Si crystallization, because of amorphous Si imperfections - vacancies, dislocations and dangling bonds. During the isothermal heat treatment, the semiconductor film is dissolved into the metal film, where it diffuses and precipitates.

The main difference between AIC and NIC is due to the different reaction behaviours of Al and Ni with Si. Ni is known to create silicides, whereas Al forms a eutectic.

The initial step during the annealing of the Al/a-Si interface is dissolution of a-Si in Al, followed by diffusion of the Si solute through the metal. The metal film is saturated with Si: the dissolved semiconductor becomes supersaturated and the supersaturation can be relieved by

crystallite growth. The mechanism of crystallization involves intermixing of Al and Si atoms and the formation of an alloy of high metal concentration in the amorphous/crystalline interface [24]. The Al layer with its crystal structure induces, in the newly-formed Si phase, a structural conformity with the crystalline phase. As a result, poly-crystalline Si grains are formed. The Al atoms diffuse along the grain boundaries and segregate between them and outside of the poly-Si layer. The process of crystallization will stop when all of the Al becomes “repulsed” at the a-Si/poly-Si interfaces. This process of AIC is known as the aluminium-induced layer exchange process in the case of an Al and Si layered structure [15]. Another case of AIC is when the Al atoms are dispersed in an a-Si film, as in the case of co-sputtered a-Si+Al layers. The process of AIC in the presence of dispersed Al clusters in an a-Si matrix is realized by nucleation in the volume of the film after rearrangement of metallic or metal-rich clusters, before crystallization of the a-Si occurs [24]. Once crystallization has started, there will always be enough Al on the growth surface because of the Al repulsion from the polycrystalline Si. Therefore, in co-sputtered films, the formation of small globular polycrystallites of Si will take place, in contrast to the column-like structure of the poly Si with larger grains crystallized from a-Si /Al layer structures. The shift of the LO-TO-like band to lower energies is greater for co-sputtered samples, indicating the presence of smaller grain sizes in the poly-Si films.

It has been reported [24,25] that oxidation of the Al layer causes a slowing down of the process of AIC. In the case of co-sputtered samples deposited at $T_s = 500^\circ\text{C}$, the dispersed Al atoms and clusters are probably oxidized during the deposition, and the crystallization cannot start because of the Al deficiency. The Raman spectra of this sample show only the presence of an amorphous Si phase. Even if the Al/a-Si interface should oxidize during the annealing in air of layer structured samples, Si diffusion could take place through the thinner spots of the Al_2O_3 , as considered in [25]. The comparison between the samples obtained by annealing in air and in vacuum shows that oxidation in air does not stop the crystallization, as can be seen from the Raman spectra in Fig. 2, curves 1 and 2. The Raman spectra demonstrate that poly-Si films obtained by annealing in air have the same crystalline quality as those annealed in N_2 . Probably, this is due to the greater affinity of Al to O_2 than to Si. However, it is possible that the Si grains have an oxidized surface.

In the case of Ni induced crystallization, the Ni atoms interact with broken Si-Si bonds and nickel silicides are formed (at annealing temperatures above 400°C , NiSi_2 is formed [8,9]). During the annealing, NiSi_2 nuclei diffuse, and along their paths poly-Si grains are formed. The needle-like Si crystallites grow by silicide-mediated crystallization of the a-Si [9]. When the density of Ni atoms in the a-Si+Ni films is lower than a certain value, no NiSi_2 precipitates can be formed and so there are no crystallisation nuclei. This can explain the observed threshold Ni concentration of about 15 at.% Ni for crystallization of co-deposited a-Si+Ni samples in our case. A critical density of Ni atoms is reported in [8] for NIC using a Ni film deposited on an a-Si surface. The results show that both the Ni concentration and the substrate temperature are important parameters for the NIC of a-Si+Ni films. Higher concentrations of Ni, C_{Ni} and T_s stimulate the creation of crystallization nuclei in the a-Si+Ni films: their density increases and thus the degree of crystallinity increases.

The observed differences in the calculated tensile stress in the poly-Si films can be explained on the basis of the difference between AIC and NIC. In the case of AIC, some of the Al atoms are bonded to Si atoms in the grains, giving p-type conduction. As mentioned above, during AIC, because of the low solubility of Al in Si, a large fraction of the Al is located in the grain boundaries of the poly-Si, thus creating tensile stress. In the case of NIC, Ni and Si form NiSi_2 at the growth front, which acts as a template for the formation of crystalline Si. No Ni is detected in the poly-Si area after NIC [9]. NiSi_2 and c-Si have a small difference in the lattice constants, of 0.4%. Thus, less tensile stress could be induced in poly-Si obtained by NIC. The higher value of the stress in samples obtained by annealing of co-sputtered a-Si+Al films can be related to the smaller size of the grains in comparison with layered samples.

The structural improvement of the poly-Si films which is obtained when the annealing is performed in the presence of H_2 can be explained by supposing that H_2 stimulates the crystalline grain growth, probably by increasing the diffusion rates of Al and Si, which exchange their places during the AIC. Probably it is due to the reducing properties of H_2 - it reduces the Al_2O_3 film formed during the storage of the Al films in air for 24 hours before the a-Si film deposition. As a result, aluminium hydroxide is formed, leading to an enhancement of material transport, as reported in [26]. This results in an acceleration of the process of AIC and an increase in the grain size in poly-Si films

when annealing is performed under the same conditions, as was observed and deduced from the OM and Micro Raman studies. The significant reduction in the grain boundary volume results in a decrease of the quantity of Al left in between the grains, and a decrease in the tensile stresses in the polycrystalline Si films.

4. Conclusions

Poly-Si layers were grown by isothermal annealing of samples of glass/a-Si/Al, glass/Al/a-Si, glass/a-Si+Ni and glass/Al/a-Si+Al in vacuum, air, N₂, N₂+H₂ and H₂ at temperatures below that of the a-Si SPC. In the case of NIC, the Raman spectra of the annealed samples showed that the crystallinity of the layers depends on the Ni concentration. It was demonstrated that a threshold value of the Ni concentration exists for NIC of co-deposited a-Si+Ni. It was also shown that the T_s for co-sputtered a-Si+Ni and a-Si+Al films is an important parameter for the crystallization process. Poly-Si films prepared from co-sputtered a-Si+Al films had a higher tensile stress than those from a-Si ones, probably due to the smaller size of the poly-Si grains in the films. Poly-Si films obtained by NIC had a lower stress than films obtained by AIC.

The results indicate that the structure of the poly-Si films is improved when the annealing is performed in a gas atmosphere. When AIC is performed in the presence of hydrogen at the same annealing temperature, full crystallisation of the structures is achieved for a shorter time than in the case of annealing in air and N₂. A reduction in the value of the tensile stress is obtained, which is related to a significant reduction in the grain boundary volume and the Al presence there.

It is suggested that H₂ stimulates crystalline grain growth during annealing, by increasing the diffusion rates of Al and Si during the AIC. Probably, this is due to the reduction properties of H₂ - it can reduce the Al₂O₃ film formed during the storage of the Al films in air. As a result, aluminium hydroxide can be formed, leading to an enhancement of the material transport. This leads to an acceleration of the process of AIC and an increase in the grain size in poly-Si films when annealing is performed at the same temperature.

Acknowledgements

The author thanks Dr. O. Angelov for assistance in the preparation of the samples and Dr. M. Sendova-Vassileva for the Raman measurements performed using a SPEX 1403.

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