Metal induced crystallization of amorphous silicon using metal solution

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Abstracts

Amorphous silicon (a-Si) was crystallized by metal induced crystallization using metal solution. The a-Si films spin coated with a 5,000 ppm Ni solution were crystallized at as low as 500 °C. Needlelike morphology, developed as a result of the migration of $NiSi_2$ precipitates, appears in the MIC poly-Si. The growth of the needlelike crystallites proceeds to a direction parallel to <111>. The crystallization temperature can be lowered to 450 °C by Au addition. The enhancement of crystallization results from the decrease of interfacial energy at the $NiSi_2$/Si interface by Au addition.

I. INTRODUCTION

Polycrystalline silicon (poly-Si) has been attracted much attention because of its wide range of applicability in large-area electronics, such as thin film transistor (TFT), solar cell, image sensor and so on. In recent years, there have been number of experimental and theoretical studies on the crystallization of amorphous silicon (a-Si) at low temperature on glass substrates. Solid phase crystallization (SPC) is a typical method obtaining crystalline silicon from a-Si. SPC has many advantages over excimer laser annealing (ELA), such as simplicity, low cost, uniformity and large area applicability. But its crystallization temperature of SPC is too high for large area glass substrates to be used. For the poly-Si thin film transistors (TFTs) on glass, all thermal steps in the TFT process must remain below the strain temperature of glass substrate. Therefore there has been studied to decrease the crystallization temperature and to shorten the crystallization time.

It is well known that SPC temperature of a-Si can be lowered by the addition of some metals. When a certain metal, for example, Al[1], Cu[2], Au[3], Ag[4] or Ni[5,6], is deposited on a-Si, the a-Si crystallizes to poly-Si at a lower temperature than its SPC temperature. The metal induced crystallization (MIC) is a well-known technology. According to previous reports[5,6], the reaction between a metal and an a-Si occurs at an interlayer by diffusion and it lowers the crystallization temperature. Such enhancement of crystallization is due to an interaction of the free electrons from the metal with covalent Si bonds near the growing interface.[7] Considering the metal-Si eutectic temperature, an a-Si film can be crystallized at below 500 °C. Russel et al.[8] reported that the crystallization of a-Si using a-Si/Cu layers occurs at about 485 °C. Bean et al.[4] reported the crystallization of hydrogenated amorphous silicon (a-

Si:H) in a-Si:H/Ag/a-Si:H structure above 350 °C. Cammarata et al.[6] has demonstrated the formation of $NiSi_2$ precipitates by ion implantation of Ni into a-Si and then heating. The $NiSi_2$ precipitate acts as a nucleation site and can migrate through a-Si. The onset temperature for crystallization of a-Si is significantly reduced in the presence of $NiSi_2$ precipitates and occurred at 500 °C. The metals used as contact layers in metal/a-Si structures can be classified in two groups: silicide-forming metals (Ni, Cr, Pd, Pt, etc.) and metals that do not form silicides (Al, Au, Ag). Table 1 summarizes the crystallization temperature of pure a-Si when in contact with various metals. In our previous experiments[9], we showed that crystallization of a-Si could be achieved at temperature as low as ~ 450 °C by Ni silicide mediated growth.

In spite of the low temperature crystallization of the above cases, there is a serious problem of metal contamination into the crystallized Si. The recent investigations[10,11] on the metal induced crystallization of a-Si films using a metal solution seem to reduce the metal contamination in the crystallized Si. In this work, we studied a metal induced crystallization of a-Si:H using a metal solution instead of metal deposition on a-Si to reduce metal contamination in poly-Si.

Table I. Metal induced crystallization of a-Si

Material	Eutectic temperature (°C)	Crystallization temperature (°C)	Measurement*	Reference
a-Si		560 ~ 720	OA	[21]
		550 ~ 750	P-TEM	[22]
a-Si/Al	577	180	ED	[23]
		100 ~ 300	Raman	[24]
		325 ~ 350	RBS	[25]
		300	P-TEM	[26]
		250 ~ 500	C-TEM, HREM	[27]
		157	P-TEM, AES	[1, 28]
a-Si/Au	363	100	ED	[23]
		R.T. ~ 660	P-TEM	[14]
a-Si/Ag	845	300	ED	[23]
		540	P-TEM	[29]
		400	RBS	[30]
		410	C-TEM, DSC	[31]
a-Si/Cu	802	530	SEM	[10]
a-Si/Ni	966	500 ~ 600	P-TEM	[6, 13]
		450 ~ 550	SEM, XRD	[32]
		470 ~ 500	P-TEM, XRD	[11]
		400 ~ 660	HREM	[12]
		500	P-TEM[1]	[33]
a-Si/Ni/Au		450 ~ 530	P-TEM	[9]
a-Si/Pd		500	P-TEM	[34]
		600	P-TEM	[35]

*AES, Auger electron spectroscopy, DTA, differential thermal analysis, ED, electron diffraction, HREM, high resolution electron microscopy, OA, optical absorption, C-TEM, cross-section TEM, P-TEM, plan-view TEM, RBS, Rutherford back-scattering spectroscopy, XRD, X-ray diffraction

[1]Metal induced lateral crystallization

Ⅱ. EXPERIMENTAL

Hydrogenated amorphous silicon films of 200nm thickness were deposited by plasma enhanced chemical vapor deposition (PECVD) using SiH_4/H_2 mixture at a substrate temperature of 275 °C. Two kinds of metal (Ni and Au) are used for silicide mediated crystallization of a-Si film. The first group was prepared by spin-coating of Ni standard absorption solutions with various Ni concentrations on a-Si:H. The Ni concentration in HNO_3 and the speed of spin-coater were 500 ~ 10,000 ppm and 500 rpm, respectively. The second group of a-Si films was coated by (Ni+Au) mixing solution. (Ni+Au) were coated on a-Si:H by spin-coating of a mixture of Ni in HNO_3 and Au in HCl with various Ni/Au concentration ratios. The Ni concentration in HNO_3 and the Au concentration in HCl and spin-speed for the coating were 500 ~ 10,000 ppm, 200 ~ 1,000 ppm and 500 rpm, respectively. The samples were annealed at 430 ~ 530 °C in a N_2 atmosphere for crystallization.

The structure of MIC poly-Si films was examined through Raman spectroscopy, x-ray diffraction (XRD), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) measurements. The depth profile of Ni in the Ni-MIC poly-Si was measured by a secondary ion mass spectroscopy (SIMS) measurement.

Ⅲ. RESULTS

A. Metal induced crystallization of a-Si using a Ni solution

Figure 1 shows the X-ray diffraction spectra of the Ni-MIC poly-Si films annealed at 500 °C for 20h with various Ni concentrations. No crystalline peak is observed for 1,000 ppm case. The crystalline Si peaks at d=3.14 Å (111), d=1.92 Å (220) and 1.64 Å (311) appear in (b) 5,000 ppm and (c) 10,000 ppm cases and the intensity of the crystalline Si peaks increases with Ni concentration.

The crystallization of a-Si was confirmed by Raman spectroscopy. Figure 2 shows Raman intensity for the Ni-MIC poly-Si films spin-coated with (a) 1,000 ppm, (b) 5,000 ppm and (c) 10,000 ppm Ni

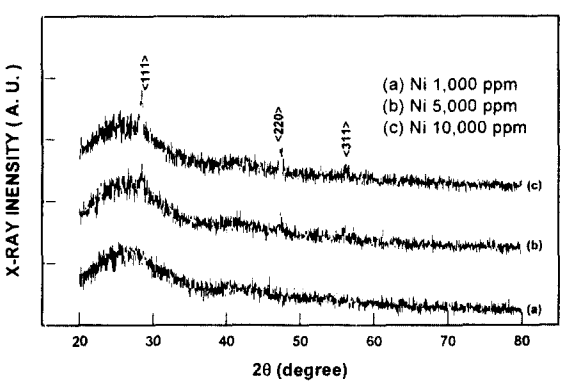


Figure 1. X-ray diffraction spectra of the Ni-MIC poly-Si films annealed at 500 °C with various Ni solutions of (a) 1,000 ppm, (b) 5,000 ppm and (c) 10,000 ppm Ni in HNO_3.

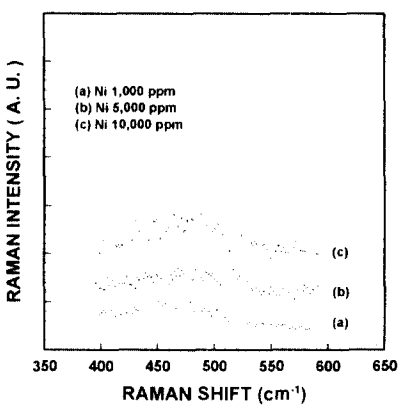
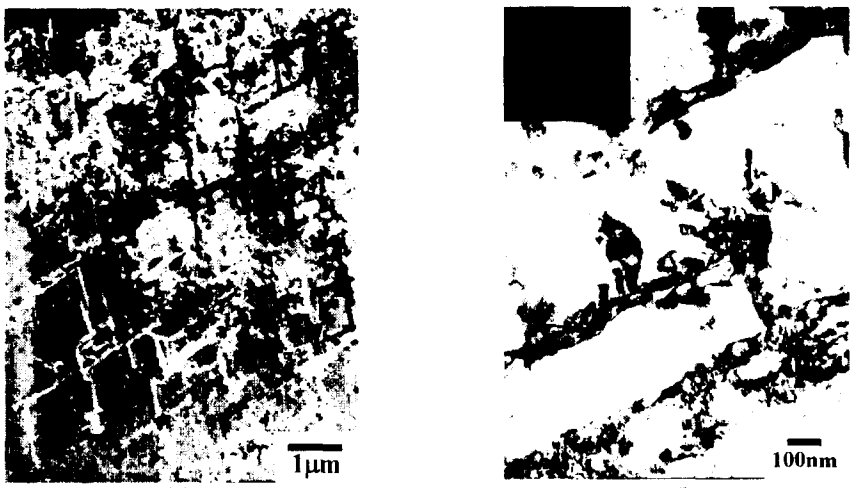


Figure 2. Raman intensity of the Ni-MIC poly-Si films annealed at 500 °C with various Ni solutions of (a) 1,000 ppm, (b) 5,000 ppm and (c) 10,000 ppm Ni in HNO_3.



(a) (b)

Figure 3. (a) TEM bright field images of the Ni-MIC poly-Si film spin-coated with 10,000 ppm Ni solution and then annealed at 500 °C for 20 hours, (b) the central region of the needlelike c-Si network i (a) and its electron diffraction.

standard absorption solution and then annealed at 500 °C for 20h. The optical phonon peak of crystalline Si is clearly observed at 516.64 cm^{-1} together with a small broad peak at 480 cm^{-1} caused by remaining amorphous silicon. Note that there is no optical phonon peak of crystalline Si when 1,000 ppm Ni solution is used. The peak at ~520 cm^{-1} increases with Ni concentration.

Figure 3 shows TEM bright field image of the Ni-MIC poly-Si film spin-coated with 10,000 ppm Ni solution and annealed at 500 °C for 20h. In Fig. 3 (a), a needlelike morphology of the Ni-MIC poly-Si is seen, which is due to the migration of $NiSi_2$ precipitates and is resulted from the growth of crystalline silicon in the amorphous matrix. Extensive crystallization of two or more overlapping single-crystal networks, differentiated by the difference in diffraction contrast, is seen. The width of needlelike single crystal Si needle is about ~ 1,700 Å and is very similar to the low-implantation-dose samples.[12] Earlier observation[13] of Ni induced crystallization of a-Si revealed that the onset temperature for crystallization of a-Si was significantly reduced in the presence of $NiSi_2$ precipitates and occurred at 500 °C. Crystallization was mediated by the migration of the $NiSi_2$ precipitates. In the initial stage, nucleation occurs randomly at the {111} faces of the individual precipitate. The orientation of the $NiSi_2$ precipitates within the a-Si determines both the orientation of initial crystalline structure and the subsequent growth direction of needlelike crystallites. All kinds of crystallites grow in <111> directions[12], normal to a $NiSi_2$ {111}. Following nucleation of crystallites, needlelike c-Si grows at the $NiSi_2$/c-Si interface as the precipitates migrate through the a-Si. Figure 3 (b) shows the central region of the needlelike c-Si network in Fig. 3 (a) and its electron diffraction pattern. The edges of the growing needlelike crystallites are observed in Fig. 3 (b). The longest needle, in right-hand side, appears to be broken into two growth fronts, each with a $NiSi_2$ precipitate at the leading edge. In the electron diffraction pattern, the halo at Si (111) spacing indicating an amorphous Si is observed. Electron diffraction indicates that the direction of growing needlelike c-Si is <111> and some a-Si phases exist in between c-Si phases.

B. Metal induced crystallization of a-Si using a (Ni+Au) solution

Figure 4 shows XRD diffraction spectra of the (Ni+Au) MIC poly-Si films with various annealing temperatures. The Ni and Au concentrations were 2,500 ppm and 500 ppm, respectively. No crystalline peak was observed at 450 °C. The crystalline Si peaks at d=3.14 Å (111), d=1.92 Å (220) and 1.64 Å (311) are observed for poly-Si films annealed at above 470 °C. The intensity of crystalline Si peaks increases with annealing temperature as expected.

Figure 5 shows XRD diffraction spectra of the (Ni+Au) MIC poly-Si films annealed at 500 °C for 20h with various Ni/Au concentration ratios. The total concentration of (Ni+Au) is fixed 1,000 ppm. The clear <111>, <220> and <311> peaks of crystalline Si appear in (a), (b) and (c) and the intensity increases with Au concentration in the (Ni+Au) mixing solution. The only Ni solution of 10,000 ppm case is added in which there is a smaller peak than that of (Ni+Au) mixing solution. From the experimental results shown in Fig. 1 through 5, it is clear that the metal induced crystallization accelerates by adding Au in Ni solution.

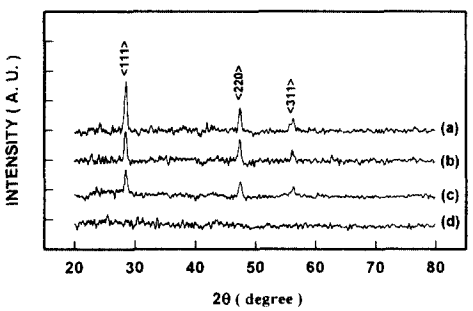


Figure 4. XRD diffraction spectra of the (Ni+Au) MIC poly-Si films with various annealing temperatures of (a) 530 °C, (b) 500 °C (c) 470 °C and (d) 450 °C.

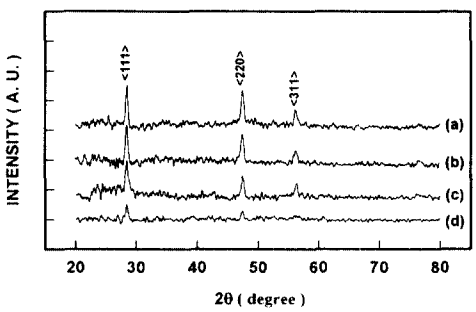


Figure 5. XRD diffraction spectra of the (Ni+Au) MIC poly-Si films annealed at 500 °C for 20h with various Ni/Au concentration ratios of (a) 1/3, (b) 1 and (c) 3, respectively. The total concentration of (Ni+Au) is fixed 1,000 ppm.

Figure 6 shows TEM bright field images of the (Ni+Au) MIC poly-Si films spin-coated with a (Ni+Au) solution of a mixture of (2,500ppm Ni and 500ppm Au) and annealed at (a) 470 °C and (b) 450°C for 20h. In Fig. 6 (b), the morphology of needlelike c-Si are observed and these are very similar to the needlelike crystallites, which are formed due to the migration of $NiSi_2$ precipitates. The width of needlelike single crystal Si is about ~ 1,500 Å and is very similar to that of Ni-MIC poly-Si by Ni solution.[11] It is noted that there is no XRD crystalline peak at the annealing temperature of 450 °C as shown in Fig. 6, but needlelike crystallites are observed in the TEM image of the (Ni+Au) MIC poly-Si film. In the case of Au induced crystallization, Au segregation to the phase boundary between poly-Si and a-Si is observed during crystallization because of a phase decomposition of Si and Au[14]. But there is no Au segregation in the phase boundary of the poly-Si and a-Si in Fig 6(b). And the a-Si was fully

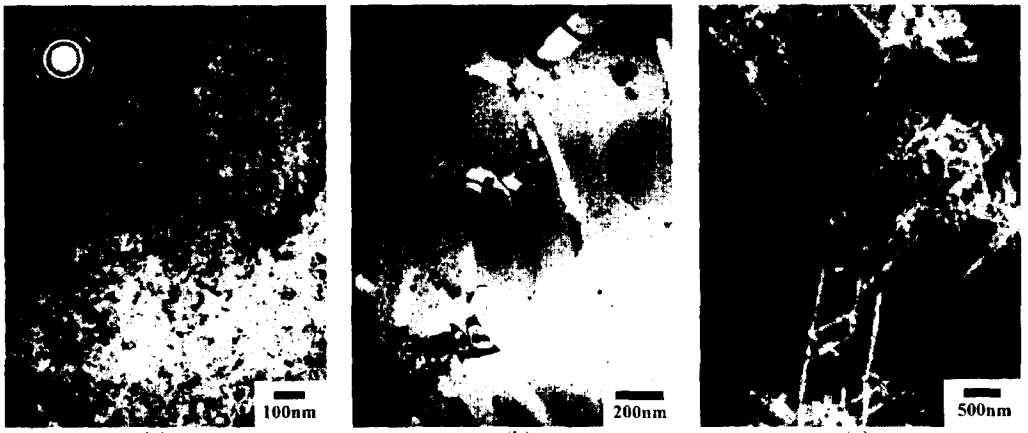


Figure 6. TEM bright field images of the thin silicon films spin-coated with Ni and Au mixing solution (2,500 ppm Ni and 500 ppm Au) and then annealed at (a) 470 °C and (b) 450 °C for 20h. The image for the thin silicon film using 10,000 ppm, which is annealed at 470 °C, is inserted in (c).

crystallized at 470 °C. There is no amorphous phase in the electron diffraction pattern of Fig. 6(a). For the comparison, a TEM bright field image (c) of the thin silicon film, coated with 10,000 ppm Ni solution and then annealed at 470 °C for 20h, is added. In the case of a 10,000 ppm Ni only solution, needlelike morphology of the crystallites is seen, which is due to the migration of $NiSi_2$ precipitates. In the case of metal silicide mediated crystallization of a-Si, metal silicide acts as a nucleus for crystallization, so the crystallites increase with metal concentration. In Fig. 6(a), a-Si film is fully crystallized by (Ni+Au) metal induced crystallization in spite of low metal concentration. It is clear contrast to Fig. 6(c) for the Ni solution without Au, in which some needlelike crystallites exist within a-Si matrix.

The MIC temperature of a-Si decreases to 450 °C by adding Au into a Ni solution. The a-Si, spin-coated with a solution of 2,500 ppm Ni and 500ppm Au can be fully crystallized by 470 °C annealing for 20h. The crystallization accelerates with increasing (Ni+Au) concentration and with increase in annealing temperature. Even in the presence of Au, the crystallization of a-Si appears to proceed through the migration of $NiSi_2$ as in the case of Ni MIC.

It is shown that the formation of $NiSi_2$ is a "nucleation controlled reaction" which has been explained in detail by d'Heurle et al.[15] They observed that a balance exists between the opposing forces due to the surface creation energy of the new surface (Δs) and the bulk free energy of the reaction (ΔG per unit volume). Practically, Si-rich silicides ($NiSi_2$, $CoSi_2$ etc) are formed at the silicide/a-Si interlayer by the reaction. Therefore, the surface creation energy of the new surface (Δs) is reduced

Earlier observations[14,16] of Au silicide formation revealed that the Au_4Si phase was formed at the very low temperature of 180 °C. The Au_4Si has the same structure of cubic as c-Si and a higher lattice constant than that of c-Si. Table II summarizes the lattice constants of $NiSi_2$, Si and Au_4Si.

Since Au addition decreases the lattice mismatch between $NiSi_2$ and c-Si, the nucleation temperature of $NiSi_2$ is lowered due to the reduction of the interfacial energy ($\Delta\sigma$). The addition of Au

may also change the free energy of the reaction : $Ni(Au)Si + Si \rightarrow Ni(Au)Si_2$, because of the higher solubility of Au in $NiSi_2$ than in NiSi increasing the entropy of mixing. In a previous report[14], the solubility of Au in Ni silicides was estimated 0.1 at. % in Ni_2Si, 1 at. % in NiSi and 4 at. % in $NiSi_2$. The combined effect (decrease of $\Delta\sigma$ and increase of ΔG) appears to enhance the $NiSi_2$ induced crystallization by Au addition and thus leads to a lower metal induced crystallization temperature.

Table II. Lattice constants of $NiSi_2$ and Au_4Si [14,17]

	Phase	Structure	Lattice constants (Å)	Silicide formation temperature (°C)
Ni	$NiSi_2$	Cubic	5.403	$\geq$ 250
Au	Au_4Si	Cubic	5.505	130
Si	c-Si	Cubic	5.430	

Figure 7 shows the temperature dependence of conductivity for the Ni-MIC poly-Si films annealed at 500 °C for 20h with various Ni/Au concentration ratios. The total concentration of (Ni+Au) is fixed at 1,000 ppm. The conductivity activation energy and the room temperature conductivity are 0.48 ~ 0.52 eV and ~ 10^{-5} S/cm, respectively. The Ni solution of 10,000 ppm case is added. There is no hopping conduction, which is reportedly seen in unhydrogenated amorphous silicon. It is noted that laser crystallized poly-Si films show an activated form of conductivity.[18]

Figure 8 shows AFM image of the Ni-MIC poly-Si film annealed at 500 °C for 20 h using 10,000 Ni solution. The surface morphology of poly-Si film plays a very important role for the poly-Si TFTs[19].

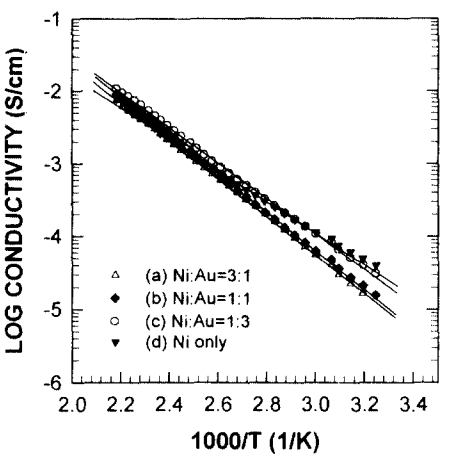


Figure 7. The temperature dependence of the conductivity for the Ni-MIC poly-Si films crystallized at 500 °C for 20h with various Ni/Au concentration ratios. The total concentration of (Ni+Au) is fixed 1,000 ppm.

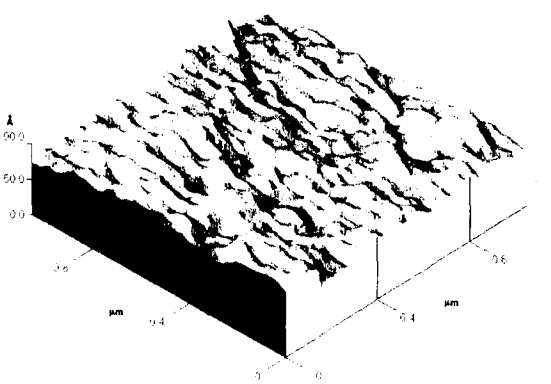


Figure 8. AFM image of the Ni-MIC poly-Si film annealed at 500 °C for 20h.

The RMS (root mean square) surface roughness of the Ni-MIC poly-Si film is about 8 Å.

IV. DISCUSSION

Figure 9 shows a schematic diagram of the growth of needlelike crystallites within a-Si. When the a-Si, spin-coated with Ni solution is annealed at above 400 °C, the $NiSi_2$ precipitates are formed in the a-Si matrix and then they act as nucleation sites. Although all precipitates can act as nucleation sites, substantial growth of Si occurs for <110> oriented precipitates with reported to film surface normal. It shows that only <110> oriented nucleus, having four <111> faces perpendicular to a-Si surface, leads to the growth of needlelike crystallites as shown in Fig. 9. But the nucleus, with inclined <110> direction, does not lead to the growth of crystallites because of the intersection of the {111} faces with top or bottom surface. The growth of (111) faces can be explained by the fact that the surface energy of the (111) plane in Si is the lowest compared to any other orientation[20].

In the case of $NiSi_2$ precipitate induced crystallization, the crystalline Si nucleates on one or more of the eight {111} faces of the octahedral $NiSi_2$. However only <110> oriented precipitates grow extensively. The orientation of the $NiSi_2$ precipitates within the a-Si determines both the orientation of initial crystalline structure and the subsequent growth direction of needlelike crystallites (in Fig. 3 and 6). The angle between the needlelike crystallites is close to 70.5° (corresponding to the angle between the (111) and (111) faces which are perpendicular to the (110) face). Such needlelike crystal growth is similar to the MIC phenomena published in the previous reports[6].

Figure 10 shows Ni depth profiles in the Ni-MIC poly-Si films using 10,000 ppm Ni solution (a) and using a sputter–deposited 4 Å Ni layer (b). The samples were annealed at 500 °C for 20h. The Ni concentration in the MIC poly-Si using Ni solution is about 1.2×10^{18} cm^{-3} which is much lower than that

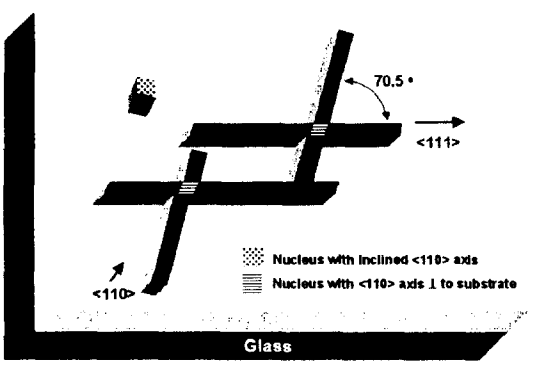


Figure 9. Schematic diagram of the growth of needlelike crystallites in the a-Si network in two orientations of the <110> axis of the nuclei. Just only <110> oriented nuclei perpendicular to the film surface lead to growth of needlelike crystalline Si.

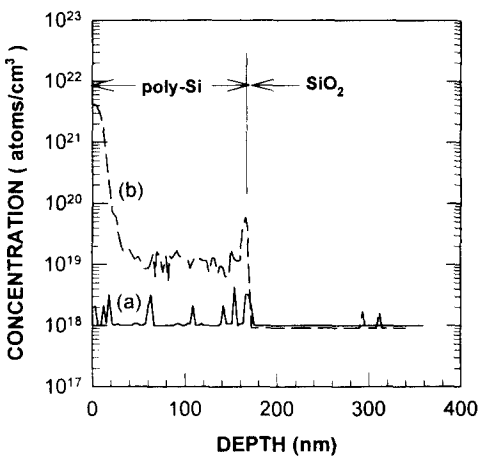
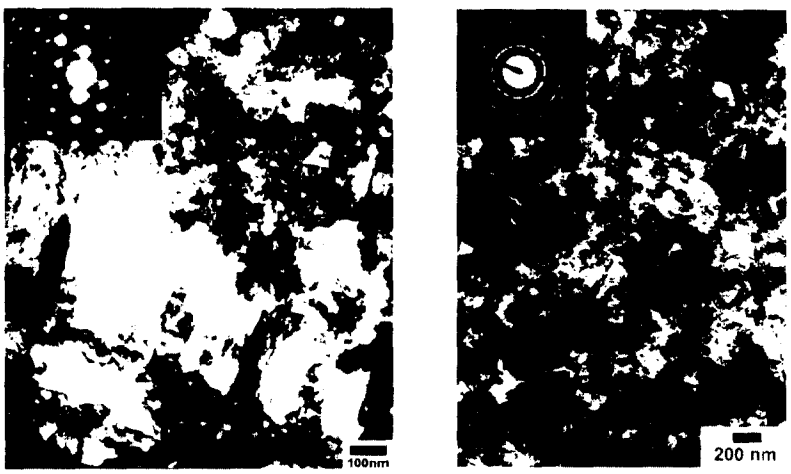


Figure 10. Depth profiles of Ni in the Ni-MIC poly-Si films spin-coated with 10,000 ppm Ni solution (a) and 4 Å Ni sputter-deposited (b). The samples were annealed at 500 °C for 20 h.



(a) (b)

Figure 11. TEM bright field images and TED patterns of the Ni-MIC poly-Si film annealed at 500 °C for 20hrs with 10,000 ppm Ni solution(a) and 4 Å Ni sputtered-deposited (b).

($>10^{19}$ cm^{-3}) in the Ni-MIC poly-Si using thin (~4 Å) Ni layer on a-Si:H. Note that the Ni concentration in the MIC poly-Si using 4 Å Ni layer decreases rapidly with Si depth. The Ni pile at the interface between the poly-Si and SiO_2 is resulted from the migration of $NiSi_2$ precipitates to the interface. In the metal silicide mediated crystallization, the precipitates act as nucleation sites and crystallization of the entire film proceeds via the migration of the $NiSi_2$ precipitates, which trailed epitaxial c-Si. With increasing Ni concentration in the a-Si films, the density of $NiSi_2$ precipitates increases and thus the crystallization accelerates.

Figure 11 shows that the TEM bright field images for the Ni-MIC poly-Si films with 10,000 ppm Ni solution (a) and 4 Å Ni sputtered-deposited (b). The films were fully crystallized at 500 °C for 20h. In Fig. 8(a), needlelike crystallites of the Ni-MIC poly-Si are seen, which are resulted from the growth of crystalline Si by the migration of $NiSi_2$. The TED pattern shows that the needlelike crystallites are strongly <111> oriented and no a-Si phase exists. In the case of 4 Å Ni, there are no needlelike crystalline phases due to the higher density of $NiSi_2$ precipitates, which resulted in the crystallization through the a-Si depth. The TED image of this poly-Si has spotted ring due to small grain poly-Si.

V. CONCLUSION

We have succeeded in Ni induced crystallization of a-Si using Ni standard absorption solution and (Ni+Au) mixing solution. The a-Si, spin coated with 5,000 ppm Ni solution and (Ni+Au) mixing solution (2,500 ppm Ni and 500 ppm Au), can be fully crystallized by annealing at 500 °C and 470 °C, respectively. The crystallization accelerates with increasing metal concentration in a-Si films. Migration of the $NiSi_2$ precipitates results in the growth of the needlelike crystallites in <111> directions. Even in the presence of Au, the crystallization of a-Si is appears to proceed through the migration of $NiSi_2$ precipitates as in the above case of Ni MIC. The Ni concentration in the poly-Si using 10,000 ppm Ni

solution was found to be a 1.2×10^{18} cm^{-3}.

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