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AFM AND *SEM* INVESTIGATIONS OF ION BEAM SYNTHESIZED Mg_2Si PRECIPITATES IN Si SUBSTRATES

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The Mg_2Si phase was fabricated using ion beam synthesis with high doses of $^{24}Mg^+$ implantation in Si substrates, followed by rapid thermal annealing. The chemical composition of the as-implanted and of samples annealed under different conditions was studied by X-ray energy dispersive spectroscopy. The effect of the different doses and annealing time on the surface morphology was investigated by scanning electron microscopy and atomic force microscopy.

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1. Introduction

Formation of Mg_2Si precipitates in Si is of interest for the creation of silicon-based materials with new photoelectric and thermoelectric properties. Magnesium silicide (Mg_2Si) is a narrow band-gap semiconductor with an indirect band gap of 0.6-0.8 eV [1,2]. This material is expected to be applied in highly efficient solar cells, and has potential detector applications in the 1.2-1.8 μm infrared range relevant for optical fibres [3,4]. Ion beam synthesis (IBS) is one of the most suitable techniques employed to form silicides [5].

In the present work, the results of composition and surface morphology investigations of thin layers of Mg_2Si precipitates in Si substrates, prepared by IBS, are reported.

2. Experimental details

Four types of samples were prepared by single (samples 31-44) and double (samples 11-24) implantations of $^{24}Mg^+$, followed by annealing in a vacuum of 6.65×10^{-3} Pa at 500 ºC for three different times – 30, 60 and 300 s. The implantation conditions and annealing regimes are given in Table 1. The implantation was performed into (111)-oriented, n-type, 800 Ω cm, Czochralski (Cz) silicon. During it, the substrate temperature rose to about 230 ºC due to the incident ion beam with a power density of 0.48 Wcm^{-2} (beam current density of 12 μA cm^{-2}). The experimental IR spectra indicate unambiguously the presence of the Mg_2Si phase in all of our samples [6].

The surface morphology of the samples was examined using a Jeol JSM-840A scanning electron microscope (SEM). The chemical compositions were studied by X-ray microanalysis, using X-ray energy dispersive spectroscopy (EDX) on a Link analytical AN 10/95S spectrometer. The qualitative and quantitative analyses were carried out at an accelerating voltage of 4 keV, due to

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sample thicknesses of about 150 nm. More detailed pictures on the surface for some of the investigated samples were obtained using a Supra 35 electron microscope. The surface roughness was estimated using a Digital Instruments Nanoscope IIIa atomic force microscope, in air.

3. Results and discussion

Fig. 1 shows AFM images of silicon samples, as-implanted with Mg^+. The calculated difference in the sputtering yields for Si (1.22 at/ion) and Mg. (2.17 at/ion) [7] led to an increasing surface roughness of the samples with increasing implanted dose. The values of surface roughness (R_{max}) and root mean square (rms) are given in Table 1. The mean Mg concentrations calculated by SRIM [7] and obtained experimentally by EDX are in good agreement (Table 1).

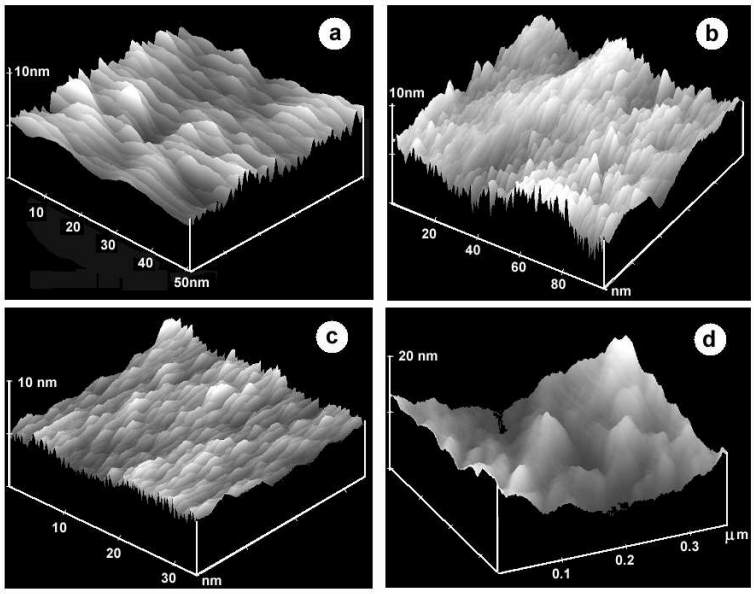

Fig. 1. AFM images of as- implanted samples: (A) - 11; (B) – 21; (C) - 31; (D) – 41.

Table 1. Summary of the implantation conditions, annealing temperature and time, surface roughness, calculated and experimental values of Mg concentration.

Sample	Ion implantation $^{24}Mg^+$ Energy, Doses	RTA	rms (nm)	R_{max} (nm)	SRIM Mg (at %)	EDX Mg (at %)
11	40 keV, 5×10^{16} cm^{-2}	as implanted	0.187	2.189	12.3	5.3
12	plus	500 °C, 30 sec				7.5
13	15 keV, 5×10^{16} cm^{-2}	500 °C, 60 sec				7.0
14		500 °C, 300 sec				8.0
21	40 keV, 1×10^{17} cm^{-2}	as implanted	0.811	5.417	21	13.9
22	plus	500 °C, 30 sec				
23	15 keV, 1×10^{17} cm^{-2}	500 °C, 60 sec				16
24		500 °C, 300 sec				14
31	40 keV, 2×10^{17} cm^{-2}	as implanted	0.393	0.959	21	16
32		500 °C, 30 sec				20
33		500 °C, 60 sec				25
34		500 °C, 300 sec				18
41	40 keV, 4×10^{17} cm^{-2}	as implanted	0.787	6.458	35	40.8
42		500 °C, 30 sec				46.6
43		500 °C, 60 sec				45.3
44		500 °C, 300 sec				36

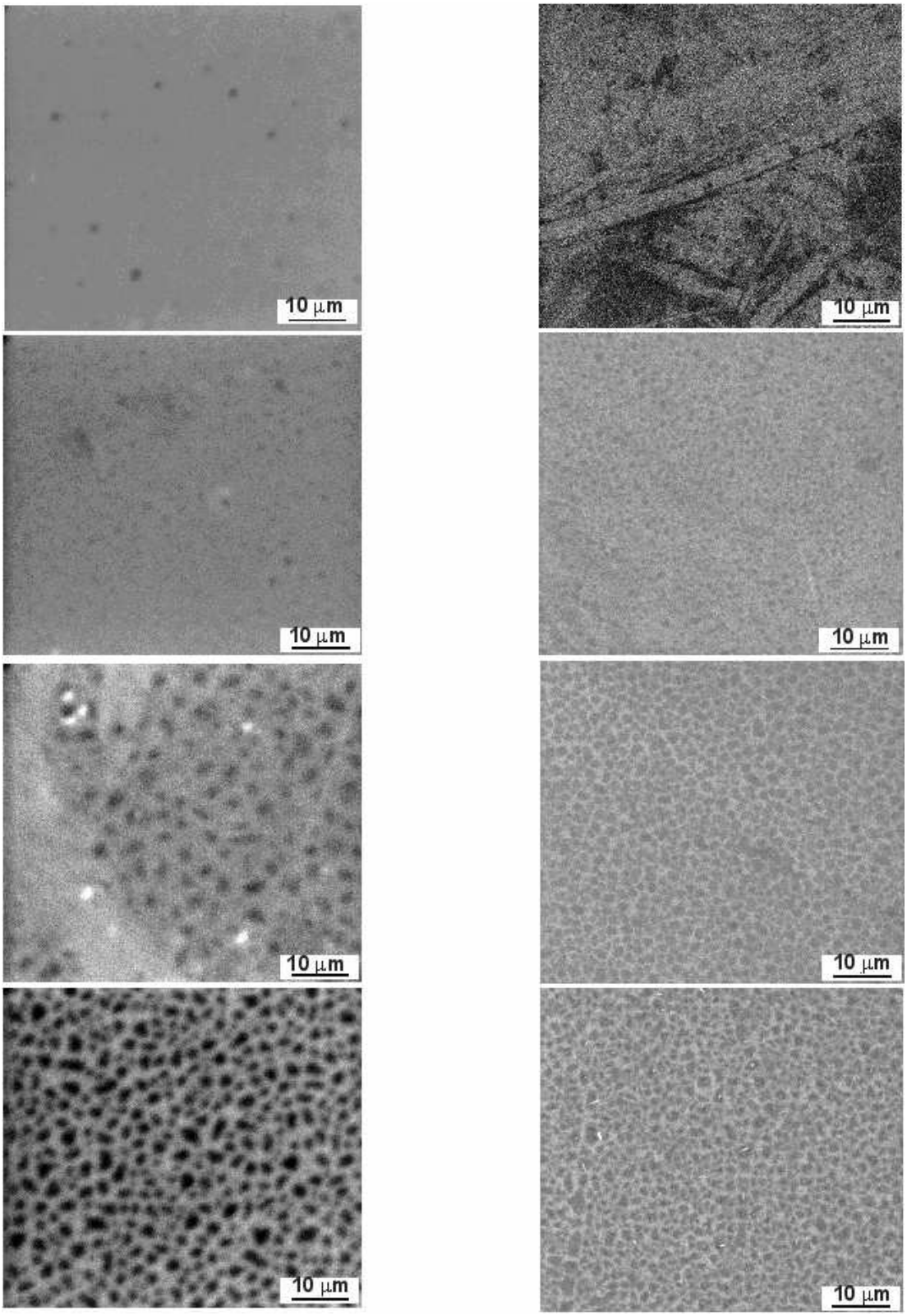

Fig. 2. SEM microphotographs of as-implanted and annealed samples at 500 ^{o}C for 30 s, 60 s and 300 s: left – samples 11, 12, 13, 14; right – samples 41, 42, 43, 44.

Fig. 2 shows SEM microphotographs of various samples. Since Mg is a fast diffusing dopant in Si (3.5×10^{-10} cm^{-2} s^{-1} at 370 ˚C), its redistribution towards the Si surface occurred with the increase of the annealing time. As a result of this process, the density of the Mg_2Si precipitates on the Si surface increased. The mean size of the Mg_2Si for the highest annealing time was about 3 μm for s.14 and about 8 μm for s.24. It can be seen from Fig. 2 (right side) that with increasing annealing time, a mosaic structure develops.

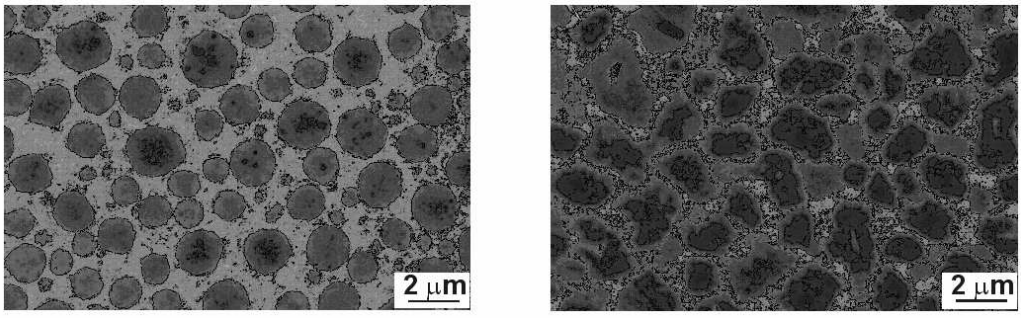

Fig. 3. SEM microphotographs obtained by Supra 35 electron microscope for the samples 34 and 44.

Fig. 3 (samples 34 and 44) clearly shows that the density of Mg_2Si precipitates increased with increasing implanted dose. The mean size of the formed clusters on the Si surface increased from 1 μm to about 2 μm. At the same time, the shape of precipitates changed from a spherical to an irregular form.

4. Conclusions

The effect of rapid thermal annealing on the surface morphology of Mg_2Si precipitates in silicon, obtained by ion beam synthesis, was investigated. The results revealed surface microtopography evolution, depending on the implantation conditions and annealing time. The surface roughness of the samples, and the size and density of the Mg_2Si precipitates were strongly influenced by the implantation dose and energy, and by the the annealing time.

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