



RESEARCH ARTICLE

Lon Beam Induced Silicide Formation at Nb/Si Interface

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ABSTRACT

*Ion-beam technique such as ion-beam mixing is very attractive as it allows one to process the materials at comparatively low temperatures and in fewer processing steps. As a result much attention has been devoted to understanding the ion-induced atomic transport processes and phase formation across metal/Si, Si/silicide, and metal/silicide interfaces. The silicide intermetallic has gained growing interest in the fields of technology as well as scientific aspect. Transition- and refractory-metal silicides are potentially attractive materials for various applications. In the present art of work the ion beam mixing of Si/Nb/Si thin film structure has been investigated by using Ag beam of energy 100 MeV at varying fluences ranging from  $1 \times 10^{13}$  to  $1 \times 10^{14}$  ions /cm<sup>2</sup>. The aforementioned specimens have been characterized by GI-XRD and XRR techniques. GI-XRD gives the clear signature of Nb-silicide Nb<sub>5</sub>Si<sub>3</sub> and NbSi<sub>2</sub> at the interface with different planes. Whereas XRR tells about the surface and interface roughness along with occurrence of ion beam mixing and interface width of the silicide phase/ phases.*

**Key words:** Ion Beam Mixing (IBM), metal silicides, energy losses, thin film.

INTRODUCTION

Morden material science (nano-science, ultra-fast spectroscopy etc.) requires new materials with exquisite properties which are often used in the novel device technologies. The fabrication of these new materials requires a quality control of properties, such as the physical parameters with the film structure and morphology. Amongst the different synthesis techniques adopted to grow metal silicides, swift heavy ion beam mixing (SHIBM) is becoming more attractive in term of its spatial selectivity, precise control, and low temperature process (Jain, and Agarwal 2011). The ion-beam mixing (IBM) with lower energy ion has been well understood with basic accepted mechanism being the nuclear energy loss (Chang, 1990; Nastasi,1994; Bolse, 1994), while the mechanism of SHIBM, which started in early nineties (Dufour, *et al.*, 1993), is being studied in detail by analysing different types of systems.

The refractory metal silicides, a class of transition metal silicides show remarkable mechanical strength, low density, creep tolerance and oxidation resistance up to high temperatures and, therefore, they are candidate materials for structural applications in oxidizing and hard-line environments (Petrovic and Vasudevan, 1999). Compared to noble-metal silicides, the application of refractory silicides, such as WSi<sub>2</sub> or TaSi<sub>2</sub>, appears more useful in high-temperature environments. Thus, Refractory metal silicides are receiving interest, due to the potential candidate materials for replacing nickel andcobalt superalloys in high temperature (1473-1873 K) applications.

EXPERIMENTAL TECHNIQUE

The Si/Nb/Si films of 40 nm (for each constituent layer) thickness were deposited in Ultra High Vacuum Technique (UHV) by e-gun evaporation at a base pressure of  $\sim 10^{-8}$  torr on Si (100) wafers. Before deposition, the Si substrate was first cleaned by using conventional method. The deposition rate was maintained at 0.1 Å /min and the substrates were kept at room temperature RT during deposition. The ion irradiations were performed using 100 MeV Ag<sup>+9</sup> beam at varying fluences ranging from  $1 \times 10^{13}$  to  $1 \times 10^{14}$  ions /cm<sup>2</sup>.

The combinations of ion energy and thickness of the layered film structure were chosen in such a way that large damage energy was deposited at the Nb/Si interface. The beam current was maintained at around 1 pA (particle nano Ampere) in order to avoid sample heating. The TRIM 2003 code was used to simulate the ion range and the deposited energy distributions by considering a detailed calculation of full damage cascades (Shown in Fig1).

The phase structure of the intermixed region was monitored via  $0.5^\circ$  grazing-angle x-ray diffraction (GI-XRD) using the Cu ( $K_\alpha$ ) line. X-ray reflectometry was also used as supporting technique for ion beam mixing investigation. The XRD and XRR techniques were employed at DAE-CSR-IUC consortium, Indor, India.

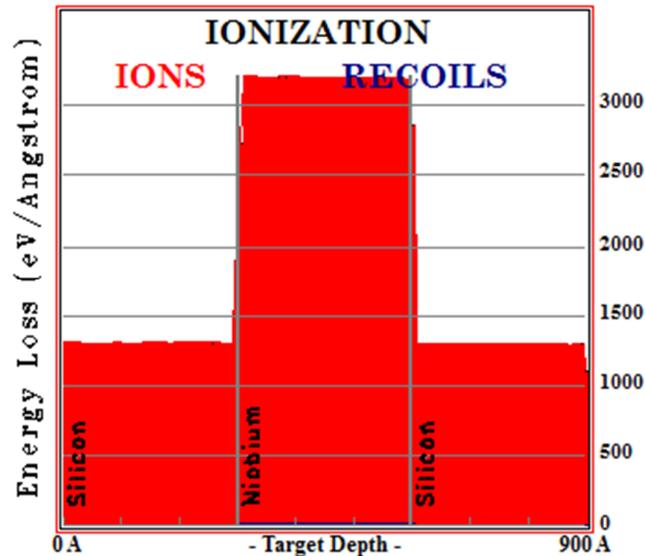


Fig. 1: Graphical representation of the Energy Loss as obtained by TRIM code

## RESULTS AND DISCUSSION

### GI-XRD Study:

Grazing incidence X-Ray diffraction (GIXRD) was carried out using D8 diffractometer. The GIXRD patterns were measured in the  $2\theta$  range of  $25-75^\circ$  with a glancing angle of  $0.5^\circ$  using Cu- $K_\alpha$  radiation in order to prevent the substrate peak. XRD patterns were analyzed by using PCPDFWIN program.

The XRD pattern of as deposited thin film (Fig 5.4a) giving the separate crystalline Bragg peaks of  $Nb_5Si_3$  phase corresponding to planes [213 and 101] and  $NbSi_2$  phase with [103] crystal plane. Besides that the system also contains oxygen in the form of Niobium oxide. It has been verified by PCPDFWIN files, that the peaks at  $2\theta = 53.75^\circ$  and  $2\theta = 40.57^\circ$ ,  $41.48^\circ$  corresponds to  $NbO_2$  and  $NbO_{1.929}$  respectively. This oxygen presence is well supported by RBS simulation also. This system is irradiated at different fluences as  $1 \times 10^{13}$ ,  $5 \times 10^{13}$  and  $1 \times 10^{14}$  ions/cm<sup>2</sup>.

The sample irradiated at lowest dose  $1 \times 10^{13}$  ions/cm<sup>2</sup> (Fig 5.4b) shows a small diminution of the peak corresponding to  $2\theta = 46.72^\circ$ , and a new low intensity peak raising (shown by red colored upward arrow in Fig 5.4b) at  $2\theta = 30.55^\circ$ . This new peak can be attributed to  $Nb_5Si_3$  phase having [004] crystal orientation. Except this there is no significant change in other silicide or oxide peaks.

At an irradiation dose of  $5 \times 10^{13}$  ions/cm<sup>2</sup> (Fig 5.4c) many of the peaks disappear, suggesting the amorphization of the layered structure as a result of irradiation. Though peak at  $2\theta = 38.27^\circ$  remains at the same position with decreased intensity and peaks at  $2\theta = 41.48^\circ$  and  $2\theta = 53.75^\circ$  suffer no change. The irradiation at highest fluence of  $1 \times 10^{14}$  ions/cm<sup>2</sup> causes re-crystallization of the pre-amorphized layered structure, which in turn appears in the form of bragg peaks. As stable silicide,  $Nb_5Si_3$  and  $NbSi_2$  phases have been formed with different crystal orientations [213, 101 and 004] at this highest fluence.

Thus, GI-XRD measurements revealed that irradiation promotes the transformation of atoms at the interface and leads to the formation of new mixed compound in the form of metal silicide.

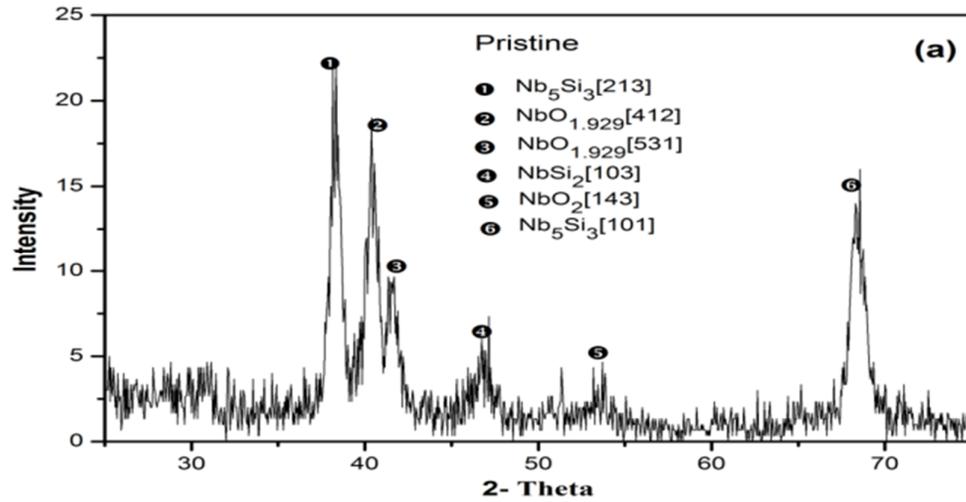


Fig. 2(a): GI-XRD curve of unirradiated Si/Nb/Si thin film system

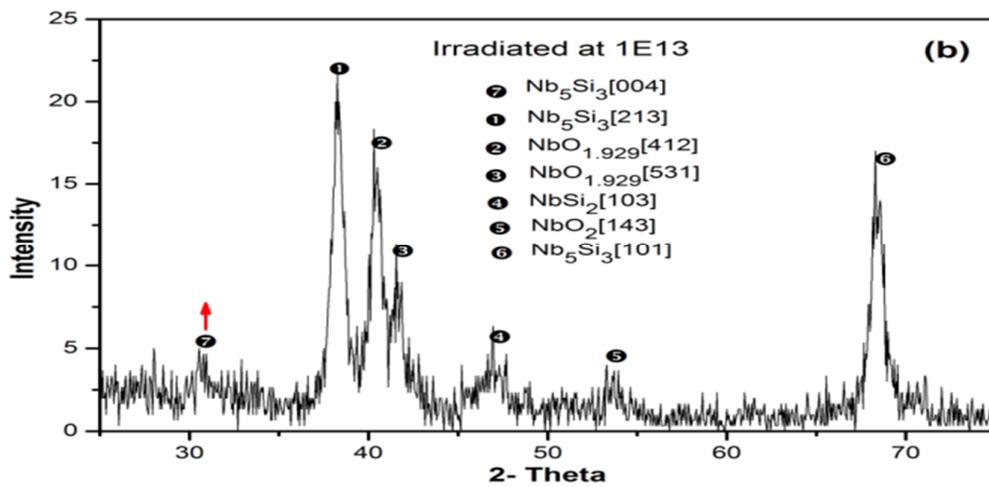


Fig. 2 (b): GI-XRD curve of irradiated Si/Nb/Si thin film system at  $1 \times 10^{13}$  ions/cm<sup>2</sup>

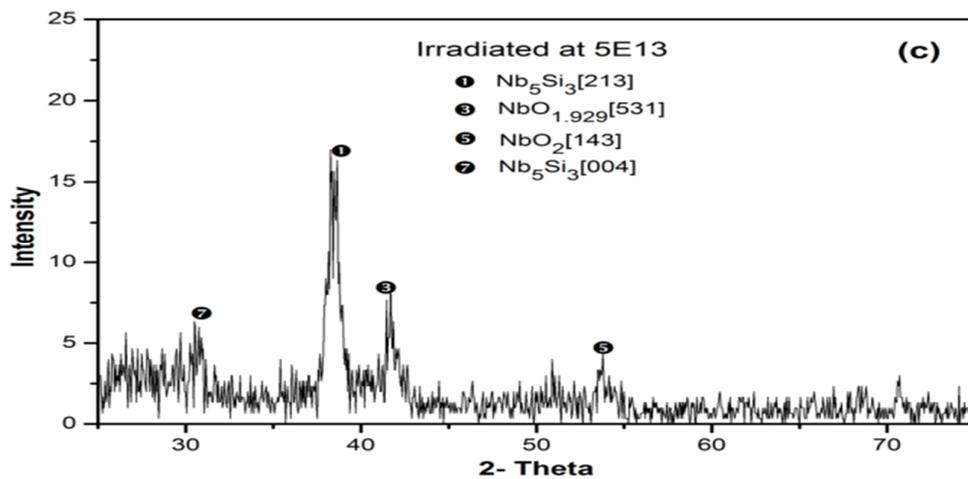


Fig. 2(c): GI-XRD curve of irradiated Si/Nb/Si thin film system at  $5 \times 10^{13}$  ions/cm<sup>2</sup>

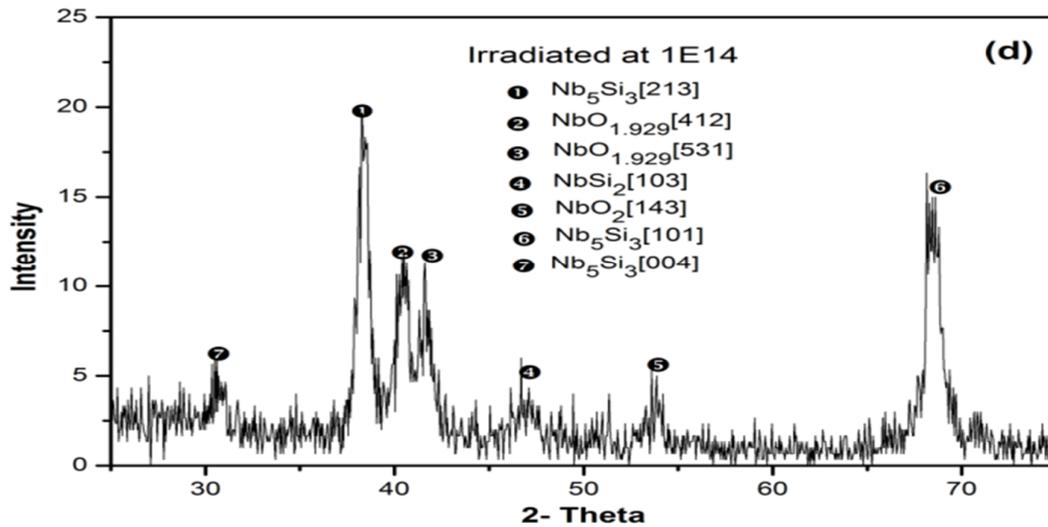


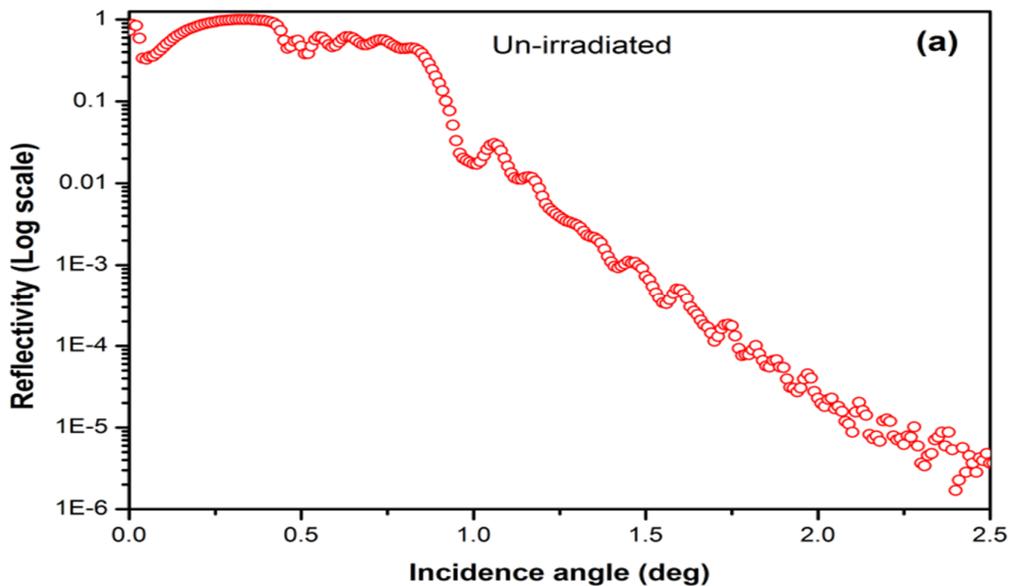
Fig. 2(d): GI-XRD curve of irradiated Si/Nb/Si thin film system at  $1 \times 10^{14}$  ions/cm<sup>2</sup>

#### XRR Analysis:

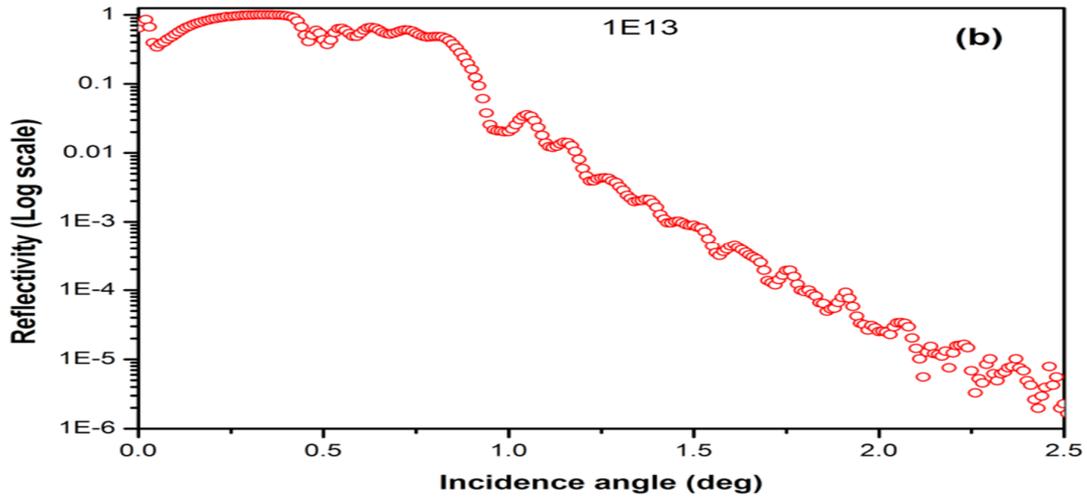
Silicide formation due to interdiffusion across the metal/silicide or metal/Si interfaces can be probed by the non-destructive and highly sensitive techniques of x-ray reflectivity (XRR). In XRR the intensity of x-rays specularly reflected (i.e. angle of incidence = angle of reflection) from the sample surface at grazing incidence  $\theta$  is measured as a function of wave vector transfer  $Q_z$  ( $\text{\AA}^{-1}$ ), perpendicular to the reflecting surface.

$Q_z$  is calculated as  $(4\pi/\lambda) \sin \theta$ , where  $\lambda$  is the wavelength of incident beam and  $\theta$  is the angle of incidence with respect to the sample surface. Using the reflectivity profiles, the composition and density gradient in a layered structure can be extracted.

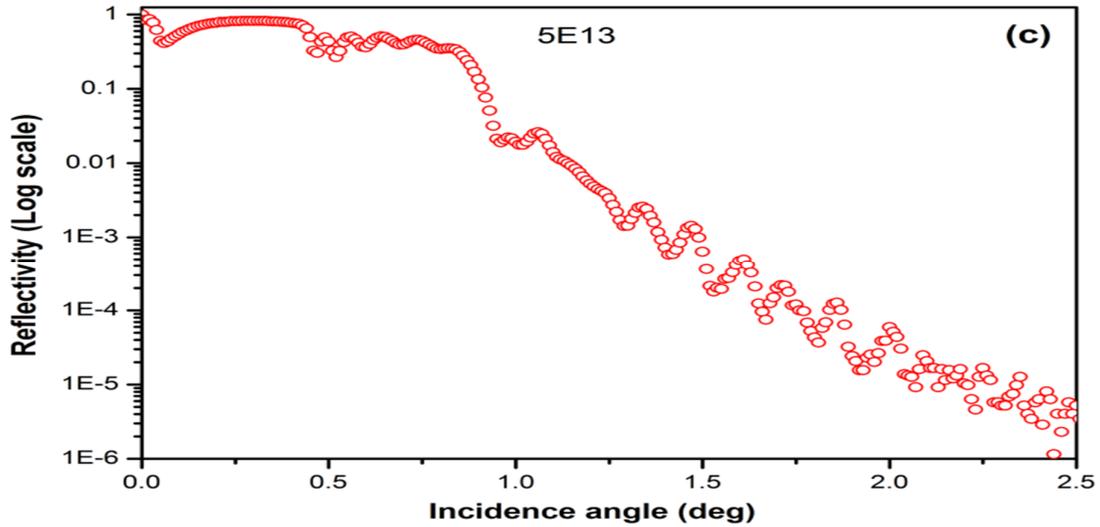
It is calculated from the profile that the thickness of Nb and Si is  $\sim 40$  nm. Hence the deposition was in the right order of accuracy. On comparing the reflectivity profiles of Fig 3 (a)-(d), it can be seen that there is an appreciable change in the Bragg's reflection peaks which signifies considerable change in the structure and composition of the film as a result of irradiation. This change can be attributed in the form of the silicide formation at the interfaces of layered structure.



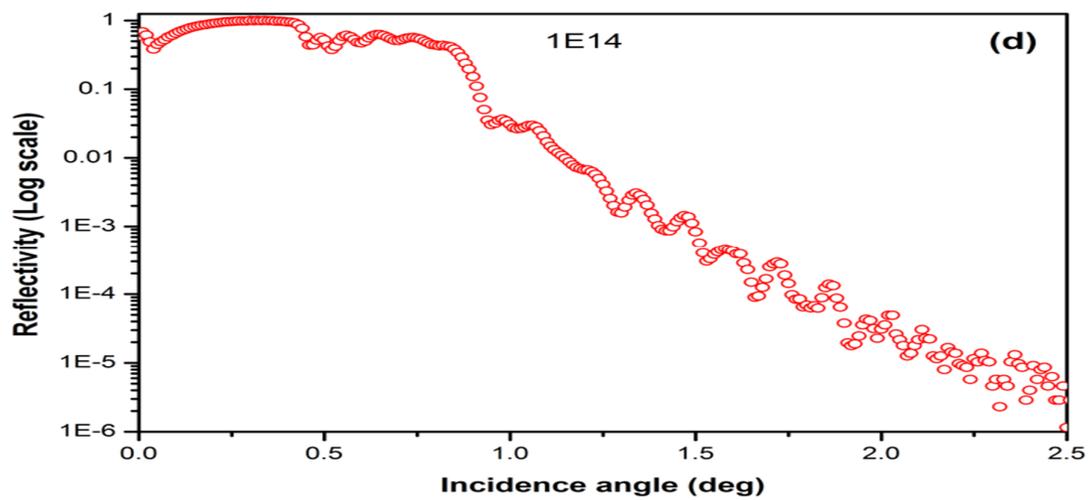
**Fig. 3(a):** Reflectivity curve of unirradiated Si/Nb/Si thin film system



**Fig. 3(b):** Reflectivity curve of irradiated Si/Nb/Si thin film system at  $1 \times 10^{13}$  ions/cm<sup>2</sup>



**Fig. 3(c):** Reflectivity curve of irradiated Si/Nb/Si thin film system at  $5 \times 10^{13}$  ions/cm<sup>2</sup>



**Fig. 3(d):** Reflectivity curve of irradiated Si/Nb/Si thin film system at  $1 \times 10^{14}$  ions/cm<sup>2</sup>

**CONCLUSIONS**

In the present work Swift heavy ion induced modifications at Si/Nb/Si thin film system has been investigated. From the GIXRD and XRR measurements there is clear signature of Nb-silicide  $Nb_5Si_3$  and  $NbSi_2$  at the interface with different planes. In this way the work is concluded on silicide formation due to the effect of ion beam induced atomic migration.

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