Electronic excitation induced ion beam mixing

A Summary/Review

by

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1. Introduction

Ion beam mixing is a phenomenon at interface between two layers, in which the atoms of two layers mingle with each other under the influence of ion beam traversal through this. A low energy ion beam irradiation causes a ballistic mixing at the interface due to elastic collision cascades. The elastic collisions produce considerable defects in both the layers. These defects assist the diffusion across the interface, referred to as radiation enhanced diffusion. Since the energy lost by the ions in elastic collisions is called nuclear energy loss, therefore nuclear energy loss is considered to be responsible for ion beam mixing. It has been shown that the number of atoms in the mixed region is proportional to the nuclear energy loss and the ion fluence. There is large number of reports on the ion beam mixing at low energies **[1-11].** The nuclear energy loss peaks at low energies typically below 1 MeV. It was shown for the first time by Dufour et al. **[12**] that the mixing at the interface could also be induced by the large electronic excitations of high energy heavy ions with the atoms in the sample. Formation of silicides and Germanides by thermal annealing **[13-18]** has been of interest because of their applications. Ion beam mixing is an effective tool for synthesizing silicides and germanides with definite advantages over the thermal annealing process. The striking feature of ion beam mixing is that (i) it has spatial selectivity, (ii) it is rather a low temperature process and (iii) thermodynamically immiscible systems, in principle, can be mixed.

The ion beam mixing can be understood by the schematic given in **fig. 1**. The two layers after ion exposure gets mixed at the interface resulting in the mixed region of the

properties different from that of the individual two layers. If one of the two layers is very thin say about ten nm or smaller, it can result in complete mixing of the layer.

The passage of high-energy heavy ions (**swift heavy ions** or **SHI**, ions with energy typically around or more than 1 MeV/u) through insulators and certain other materials creates extensive defects along the ion path beyond certain threshold of electronic energy loss. Such **columnar defects** along the ion path are explainable by **Coulomb explosion [19]** or by **thermal spike [20,21].** When the ion passes through an insulator it creates positively charged core surrounding the ion path due to electronic excitation induced by the ion. It is followed by a strong electrostatic repulsion among these charges, taking place within 10^{-14} s, leading to atomic motion, which results finally in a cylindrical zone of modified material. The Coulomb explosion model, however, fails in the case of metals, where the high mobility of conduction electrons leads to the neutralization of charges in a fraction of a femtosecond, much before the Coulomb explosion could occur. The columnar defected zone of modified material is also called as latent ion track. It was believed that the ion track could be created only in insulators and some of the semiconductors. However in early 90's it was shown **[22]** that the ion track could be created in some metals at very large electronic energy loss S_e. The creation of ion track in metals cannot be explained by Coulomb explosion because of the fact that the positive charge is quickly smeared by abundantly available electrons. The neutralization of charges in case of insulator does not occur as the electron leaving the core do not return back whereas the in case of metals the plasmon frequency being of the order of 10^{15} Hz, the electron return to core much before the Coulomb explosion. The creation of ion track in metals is well explained by thermal spike model, where it is considered that the energy

deposited in electronic subsystem is transferred to lattice via electron phonon coupling and the temperature of lattice within a narrow nanometric size cylinder exceeds the melting point of the material transiently typically for pico second duration. Above a certain threshold, S_{eth}, of electronic energy loss S_e, the material within a few nanometers surrounding the ion path melts for duration of the order of pico second. The molten state then quenches at a very fast rate of 10^{14} K/ s, forming latent tracks. It was shown that the latent track creation in insulators could also be explained by the thermal spike model. Materials undergoing the formation of latent tracks or any effect of SHI irradiation are referred to as the materials **sensitive to S_e** *or* **sensitive to SHI**. The evolution of the lattice temperature with time is estimated by the solution of two coupled differential equations.

It appears, as it will emerge by the end of this brief review that the SHI induced mixing is a consequence of inter diffusion during transient melt phase **[23]**. The interface modification have been quantitatively explained in terms of the diffusion of species during the transient melt phase. The inputs about the diameter of columnar defect region and the duration of melt phase are taken from thermal spike model. Diffusivity so obtained in the measurements is in the range of 10^{-8} to 10^{-6} m^2 s^{-1}. Such a high diffusivity is possible only for the molten state. Thus the SHI induced phenomena at the interface is explained quantitatively, supporting the existence of transient temperature spike. This has been a significant contribution in understanding the SHI induced ion beam mixing in a joint collaboration work of IUAC Delhi, LMU Munich, Stuttgart university and CIRIL, Caen and Stuttgart university.

The ion beam mixing is quantified in terms of mixing rate and the capability of producing mixing by ion irradiation is defined by mixing efficiency. The mixing rate is determined utilizing the concentration profiles of one of the two elements of the two elements at the interface. The difference in variance due to ion irradiation is expressed as

$$\Delta\Omega^2 = \Omega^2(\Phi) - \Omega^2(0) \quad \text{.....(i)}$$

where $\Delta\Omega^2$ is the difference in variance of the irradiated system at the fluence (Φ) and that of the pristine. When the difference of the variances $\Delta\Omega^2$ of the concentrations with respect to the pristine, is plotted with fluence, it usually gives a straight line within experimental uncertainties, therefore one can write the following:

$$\Delta\Omega^2 = k\,\Delta\Phi \quad \text{.....(ii)}$$

Where k is mixing rate.

The mixing rate thus is a measure of mixing effect and can be written as

$$k = d\Omega^2/d\,\Phi \quad \text{.....(iii)}$$

The mixing rate strongly depends on the electronic energy loss in case of SHI induced mixing and on the nuclear energy loss for ion beam mixing at low energies. Generally, the mixing occurs only above a certain threshold S_{eth}. The mixing efficiency is defined as

Mixing efficiency, $\mu = k / S_e$ for SHI

$= k / S_e$ for low energy ions

According to the global thermal spike model for the nuclear stopping region, the mixing rate should linearly increase with the square of the deposited nuclear energy density **[24-**

26]. It is noteworthy to mention that such a scaling has never been observed in the nuclear stopping regime **[27,28],** but it is interesting to see that such a behavior has been observed in the electronic stopping regime, as will be discussed later. Mixing rate and the mixing efficiency have the following relationship **[29],** considering that there is always a threshold of Se, beyond which SHI induced IBM can occur,

$$\mathbf{k = \mu^2(S_e - S_{ec})^2} \text{.....} \mathbf{(3.2.1.1)}$$

Where $\mathbf{S_{ec}}$ denotes mixing threshold and μ is mixing efficiency. The mixing rate and the mixing efficiency are the two key parameters useful for discussion on the content of mixing for different systems and the ability of the energy loss processes in ion beam mixing.

The ion beam mixing experiments provide good testing grounds for theoretical model explaining the atomic motions induced by swift heavy ions, defect creation etc.

The most commonly techniques used for the studying the interface mixing has been Rutherford backscattering spectrometry (RBS) **[30],** elastic recoil detection analysis (ERDA) technique **[31],** secondary ion mass spectrometry (SIMS) for depth profile of elements at interface. X-ray diffraction for phase identification, X-ray reflectivity for quantifying mixed region, Mossbauer spectroscopy if one of the elements in the system under investigation is magnetic, cross sectional transmission electron microscopy for direct viewing the interface with sub nm resolution.

The ion beam can induce surface roughness. In such cases, extra care needs to be taken while analyzing the samples by RBS **.** The RBS spectra of a mixed layer and a roughened

layer (but unmixed layer) may appear the same. The roughness at the surface and the interface mixing produces similar effects in the RBS spectrum **[32].** Thus the roughness of the irradiated samples should be taken into account while determining the extent of the mixed layer thickness.

The interface can be formed by different combinations such as metal/silicon, metal insulator, metal-metal etc. Therefore the SHI induced mixing in different type of interfaces studied so far are discussed.

2.1 Insulator/insulator, insulator/semiconductor and metal/insulator systems

2.11 SHI induced IBM in oxide/Si and Ni_3N/SiO_2 systems

The interface mixing [**33,34]** of some metal oxide thin films (NiO, Fe_2O_3, and TiO_2) deposited on Si, by irradiation with 90 to 350 MeV Ar, Kr, Xe and Au-ions at 80 K at different fluences up to $9x10^{15}$ ions/cm^2 . The interface was analyzed by Rutherford Backscattering Spectrometry (RBS). It was shown that the irradiation of ceramic/ceramic bilayers results in very strong interface mixing, which was found to be scaling with square of S_e. The mixing was observed only if the threshold S_e for track creation was exceeded. The threshold of mixing seemed to be determined by the higher track formation threshold of two constituents forming the bilayer. Among the metal oxide/Si systems, the mixing in NiO/Si, Fe_2O_3/Si and TiO_2/Si were observed. Similarly the mixing at the interface was observed by the same group in the Ni_3N/SiO_2 systems.

It was shown [35] the linear variation of the square root of mixing rate of Ni_3N on different substrates with the S_e for these systems. For TiO_2/Si system, the SHI induced

mixing rate is found to scale nonlinearly with the ion fluence, which indicated that mixing is driven by a chemical solid-state reaction in this case.

It was argued by Schattat and Bolse **[34**] in extensive work on the interface on several metal oxide/Si system that the observed threshold of IBM is not due to the Coulomb explosion process. Several ions and energies were taken to determine the threshold for SHI induced IBM. It was shown that the observed mixing did not have any dependence on Young's modulus of the systems, suggesting no correlation with Coulomb explosion. Whereas the observed threshold of mixing had a correlation with the product of melting temperature and specific heat and therefore with the thermal spike model as proposed by Szene **[36].** Thus the ion beam mixing in theses system was explainable under thermal spike formulism. It may be noted that the thermal spike model by Szene and that of by Wang et al **[24]** have significant differences in basic assumptions of the creation of temperature spike, which are not discussed here.

SHI induced mixing in ZnO/SiO_2 **[36]** studied using different S_e and fluences indicated that the IBM is due to the molten state (occurred due to thermal spike) diffusion at interface. The SHI induced mixing in NiO/SiO_2 was also explained as interdiffusion in molten track across the interface **[37].** The SHI induced mixing is also shown to occur in the fluorides of Ba and Ca deposited on SiO_2 **[38].**

2.12 IBM of metal/polymer

Bruck et al **[39**] showed SHI induced mixing in Cu film on Teflon polymer. Despite Cu being insensitive to S_e, a very strong mixing was observed as investigated by RBS. This also had implication on adhesion of Cu film on Teflon. This was among first result on

SHI induced mixing in metal/insulator system. The cause of mixing could have been the diffusion of Cu in the transiently molten track of polymer. Metal polymer mixing by ion beam has been exploited for improving the adhesion of metal film on polymer **[40].**

It has been demonstrated recently **[41]** that the SHI induced IBM of Ni thin film on polymer results in formation of magnetic-insulator nanocomposite layer.

2.13 SHI induced IBM in CuO/glass studied by on-line ERDA

On-line studies of ion beam mixing in CuO/glass systems induced by electronic excitation were performed by Avasthi et al. **[42].** It was observed that about 28 atoms of Cu (from CuO film) and 26 atoms of Si (from glass) mix with each other for each incident I ion of 210 MeV. Apart from this diffusion of oxygen from glass to CuO was observed. The recoil spectrum of oxygen extracted from the two dimensional spectrum recorded during the irradiation is shown in **fig. 2**. The smearing of the interface as seen in figure is due to diffusion of oxygen from glass (having higher concentration) to the CuO film (having smaller concentration). This was the first experiment demonstrating the ion beam mixing by on-line ERDA. It is discussed in detail in section 2.23 and 2.24.

2.1.1 Summary on insulator /insulator, insulator/semiconductor and metal/insulator systems

The insulators are extremely sensitive in terms of defect creation by swift heavy ions and they produce ion tracks in a cylindrical regions full of defects, even at very low S_e. Therefore their exposure to SHI creates atomic motions within ion tracks and the insulator/insulator systems undergo a strong mixing at the interface. If instead of insulator/insulator system, insulator/semiconductor system is irradiated, it too undergoes

mixing but less in comparison to insulator/insulator system. Although Coulomb explosion model is well applicable for creation of ion track in insulators, still it fails to explain IBM as the experimentally determined mixing threshold had no correlation with the Young modulus of the system, which was expected if the Coulomb explosion was the cause of IBM. According to thermal spike model of Szene **[35]** the threshold of mixing should have linear variation with the product of specific heat and temperature (CT), as was found experimentally and therefore thermal spike model seems to explain the SHI induced IBM.

2.2 Metal/Si or metal/semiconductor

Metal silicides, in general are chemically more stable**.** Some of them are very good candidates for direct band material like beta $FeSi_2$, wide band gap semiconductor like SiC etc. Low electrical resistivity of metal germanides make the potential candidates for use as contact and interconnect materials than the well-known metal silicides. Recently metal germanides have opened up exciting possibilities of their use as ferromagnetic semiconductors in magnetic devices. Ferromagnetic semiconductor as the name says has semiconducting properties and long-range ferromagnetic order. Thus in brief there is wide interest in mixing of metal/Si systems.

2.21 SHI irradiation and annealing effects in the interfaces of Fe/Si multilayers

Pratima Dhuri et al **[43]** studied and compared the effects of 200 MeV Ag ion irradiation and thermal annealing of Fe/Si multilayers with overall composition Fe50Si50. Two multilayers with Fe layer thickness of 19 A (having amorphous structure) and 62 A (having crystalline structure) were taken. It was concluded by the X-ray reflectivity

studies that both the thermal annealing and heavy ion irradiation result in significant inter diffusion between the Fe and Si layers. The final product of thermal annealing depends upon the initial structure of the Fe layer whereas in the case of SHI irradiation, the formed phases are similar and is independent of initial structure. The results of SHI induced mixing were quantitatively explained in the framework of thermal spike model. The effect of S_n is taken into account in temperature spike calculations. According to an extrapolation of the low-energy ion mixing result on the Fe/Si system, the contribution to the total mixing from elastic collisions turns out to be less than 1%.

2.22 SHI induced mixing in Fe/Si

Assmann et al **[44**] performed irradiation experiments on thin ^{57}Fe layers deposited on Si with swift heavy ions. The energy loss of the ions in the Fe layer was varied between 32 and 47 keV/nm by taking different heavy ion beams and energies. The formation of iron silicide phases was studied by conversion electron Mossbauer spectroscopy (CEMS) which gave a clear evidence of very clear formation of the high temperature a-$FeSi_2$. **Fig. 3** gives the Mossbauer spectra of the pristine and the samples irradiated with different ion beams at different fluence. The occurrence of this phase which is thermodynamically stable above 960°C was an indication of high temperature conditions along the ion tracks. Thus ion beam mixing was hinted to be due to temperature spike.

Dufour et al **[12]** performed irradiation (at very high electronic energy deposition using GeV ion beam at GANIL) of Fe/Si multilayers where Fe was in crystalline form and Si was in amorphous state. It resulted in formation of totally mixed layer at a fluence of 10^{13} ions/cm^2. The temperature spike was given as possible cause of mixing.

2.23 On-line study of SHI induced mixing at interface

The studies of SHI induced mixing by on-line ERDA were initiated by Avasthi et al [23,42], an aspect reported for the first time, utilizing the capabilities of the ion beam in terms of modification as well as characterization. On-line studies was performed on mixing in Fe/Si system induced by electronic excitation. This work reported on the dependence of mixing on the electronic excitation in quantitative numbers. It was reported that about 56 atoms of Fe and 85 atoms of Si mix with each other for each incident Au ion of 230 MeV and 33 atoms of Fe and 33 atoms of Si mix with each other for each incident I ion of 210 MeV. It was also shown that mixing in Fe/Si does not take place, in the case when oxygen concentration at the interface is high. Both the spectra are recorded for the same number of incident ions. This was later studied in detail by a number of experiments as given below to test the hypothesis of thermal spike model for SHI induced mixing.

2.24 Test of the hypothesis of transient molten state diffusion for swift-heavy-ion induced mixing

Srivastava et al **[23]** tested the proposed hypothesis that swift-heavy-ion induced mixing is a consequence of a transient molten state diffusion by detailed study of 230 MeV Au ion induced mixing at an Fe/Si interface monitored online by elastic recoil detection analysis. An extensive data analysis was made to determine the experimental diffusivities of Fe in Si, and vice versa, believed to be occurring during the temperature spike. The required ion track size and duration of the transient melt phase was calculated theoretically from the thermal spike model by the CIRIL group. The close resemblance of the experimental diffusivities to the liquid state diffusivities reported in the literature was the first comprehensive verification of the hypothesis that SHI induced mixing is a

consequence of inter diffusion during transient melt phase following the passage of swift heavy ion.

Under the framework of thermal spike model, it is assumed that inter-diffusion across the interface takes place in the transient melt phase. The diffusion coefficient or diffusivity of the elements at the interface during transient melt phase was determined. The distribution of Fe and Si before and after irradiation at various fluences is shown in **figure 4**. The diffusion coefficient (D) is estimated as follows:

$$\mathbf{D = \Delta\Omega^2 / 2tn} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (v)$$

where tn = effective diffusion time and $\Delta\Omega^2$ is the difference of variance of the pristine and that at fluence (Φ). The diffusion time during the transient melt phase is obtained by the following relation:

$$\mathbf{tn = (\Phi/\Phi c) \times ts} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (vi)$$

Where ts = duration of melt phase, Φ = maximum fluence and critical fluence $\Phi c = 1/(2r)^2$ is the fluence for complete overlap of the ion tracks, and r is the track radius.

The diffusivity at the interface was estimated to be far higher than expected in solid state diffusion. It was found to be of the order of liquid state diffusivity. Thus it supported the hypothesis based on thermal spike model to explain the SHI induced mixing.

It was argued that the occurrence of SHI induced mixing between Fe and Si is surprising since crystalline Si is known to be insensitive to SHI. It was, however, shown that Si also

melts near the Fe/Si interface. Such explanation were also given for Ni/Ti interface **[46].** The schematic in **figure 5** explain the melting of Si at the interface adjacent to Fe.

2.26 SHI induced mixing in Ti/Si and Ni/Si

Sisodia et al **[47]** used high electronic deposition by 350 MeV Au+ ions at different fluences for SHI induced mixing in Ti/Si. Rutherford backscattering spectra (RBS) and the X ray reflectivity measurements indicated mixing at the interface. SHI induced mixing lead to the formation of titanium di-silicide ($TiSi_2$) phase, which was evident by grazing incidence X-ray diffraction data.

The irradiation of Ni/Si and Ti/Si systems by 95 MeV Au ions at a fluence of 10^{13} ionscm^2 and subsequent annealing lead to the formation of silicides [**48**]. The layers of Ni (15 nm) and Ti (18 nm) were deposited by e-gun evaporation on Si substrate. Rutherford backscattering spectroscopy and X-Ray refectivity were used to characterize the pristine and irradiated samples. The large electronic excitation due to SHI irradiation produces defects in the system. Thermal annealing of the SHI irradiated Ni/Si system provided the required energy to the atoms for diffusion across the interface. The defects produced by SHI irradiation enhance the interface diffusion during annealing.

2.27 SHI induced interface modification in Ni/Ge

The formation of Ni_2Ge alloy by 100 MeV ion irradiation of Ni/Ge bilayer structure at different temperatures and fluences was studied by Som et al **[49**]. The content of mixing was determined by Rutherford backscattering spectrometry. High resolution cross-sectional transmission electron microscopy was performed for thickness determination and structure determination and this made a very clean a clear experiment with strong

evidence. Alloy formation was discussed on the basis of swift heavy ion irradiation induced effects. **Figure 6** shows cross section TEM images at the interface along with high resolution image to confirm the Ni_2Ge phase.

2.28 SHI induced interface modification in Co/Ge bilayer

Som et al **[50]** reported on interface modification in Co/Ge bilayer system using 100 MeV Au ion irradiation at different temperatures and fluences. Interfacial changes induced by irradiation were studied by Rutherford backscattering spectrometry and cross-sectional transmission electron microscopy. The irradiation at low temperature did not show any appreciable interface mixing whereas the room temperature irradiation resulted in formation of a thin intermixed layer. It was on the basis of synergetic effects of nuclear and electronic energy loss processes. It was proposed that in the presence of large number of S_n induced point defects; S_e induced thermal spike leads to atomic mobility across the Co/Ge interface. Although it is possible to explain the result without considering the synergy effect. It is possible that in the case of the liquid nitrogen temperature the transient temperature spike does not reach the molten state and this could be the cause of no mixing, whereas at room temperature, both the Co and Ge (being S_e sensitive) undergo transient melt state, resulting in mixing at the interface.

2.29 Mixing in Cu/Ge system by swift heavy ions

Sarvesh Kumar et al. [**51,52]** investigated the ion beam mixing in Cu/Ge bilayer, which were irradiated by 120 and 140 MeV Au ions and Cu/Ge multilayer by 120 MeV Au ions. The concentration versus depth profiles at the interface were determined by Rutherford backscattering of the pristine and irradiated samples. The pristine as well as irradiated Cu/Ge multilayers samples were annealed at 200 °C temperature. The

irradiated annealed samples showed considerable mixing in comparison to pristine annealed due to radiation effected diffusion. On the basis of several experiments with different ions and energies it was concluded that the IBM increases with S_e and fluence. Irradiation at RT gave higher mixing than that at liquid nitrogen temperature.

2.30 SHI induced mixing in a-Si/V/a-Si

Diva et al **[53]** carried out for investigation of ion beam mixing in Si/V/Si system induced by electronic excitation. Au ions of 120 MeV were taken at different fluences upto 10^{14} ions/cm^2 for this purpose. The mixing at the interface was investigated by secondary ion mass spectroscopy and Rutherford backscattering spectroscopy. A linear dependence of the atomic mixing with the fluence was observed. Strong mixing was observed at the fluence of 10^{14} ions/cm^2. Swift heavy ion induced mixing was explained by the interdiffusion at interface during transient melt phase. The contribution of nuclear energy loss to the observed mixing was sown to be insignificant on the basis of the reported results of mixing at low energy ions. On the basis of investigations in several metal/a:Si systems it was concluded that the content of mixed layer [53] is dependent on the electron phonon coupling of the metal layer. A factor η was defined [21] to represent the sensitivity of material to Se. The content of mixing [53] also varies well with η, which depends on the electron phonon coupling. This again is a strong support to thermal spike model in explanation of SHI induced IBM.

2.31 SHI induced mixing in Co/Si

Chakraborty et al **[54]** performed ion beam mixing studies on a multilayer heterostructure of a-Si(50 nm)/Co(50 nm)/a-Si(50 nm) on Si(100), prepared by an e-beam evaporation technique under UHV conditions. The multilayer was irradiated by 120MeV Au ions with fluence of 10^{13} to 10^{14} ions cm^{-2}. The interface was examined by Secondary ion

mass spectrometry, which indicated increase in the mixing with fluence. **Fig. 7** gives the SIMS spectra of the pristine and irradiated samples at different fluences, indicating significant mixing at the interface. The investigations by x-ray diffraction confirmed the formation of different phases of cobalt silicides, Co_2Si, CoSi and $CoSi_2$. **Fig. 8** gives the X ray diffraction confirming the silicide phase, without annealing. The above work suggests that swift heavy ion irradiation can be used for making buried layer silicide at room temperature avoiding thermal annealing. The cross sectional TEM showed that deposited Si was in a-Si form at the interface, which could play an important role in maximizing the effect of SHI irradiation. It is to be noted that the irradiation of Co on Si did not yield significant mixing as reported in the next section. The metal layer on a-Si results in greater mixing than those on the Si substrate simply because the a-Si layer is sensitive to SHI irradiation whereas Si substrate which is crystalline is not sensitive to SHI irradiation.

In another independent study, Bhattacharya et al **[55]** irradiated thin films of Co deposited on Si, by 120 MeV Au beam irradiation at a fluence of 10^{13} ions/cm^2 and subsequently subjected to thermal annealing to induce interdiffusion at the interface. The irradiated and subsequently annealed samples were analyzed by Rutherford backscattering spectroscopy. **Figure 9** shows the RBS spectra of the pristine, irradiated and irradiated annealed samples. Respective crystalline cobalt silicide phases were identified by grazing incidence X-ray diffraction. Complete intermixing of Co and Si in the interfacial region to form CoSi was achieved following thermal annealing (at 400 ^{o}C) of the SHI irradiated CoSi system. The radiation enhanced diffusion mechanism was invoked to explain the mixing in SHI irradiated and subsequently annealed samples. Thermally assisted (ion generated) defect migration across the CoSi interface enhanced defect mediated atomic mobility, which led to the formation of cobalt silicides, in

accordance with the thermodynamically favored route. The post SHI irradiation annealing temperature required for the formation of crystalline phases (400 ^{o}C), is lower than that reported for low energy ion beam mixing cases where post irradiation annealing temperature in excess of 700 ^{o}C is required for the formation of phase. Due to lower processing temperatures, SHI irradiation and subsequent annealing may be considered as a promising silicidation technique in solid state technology.

2.32 SHI induced modifications in In/Se layer

It was observed that irradiation of In/Se bilayer by 40 MeV Si ions and subsequent annealing leads to the formation of the InSe phase **[56].** The temperature required for the formation of InSe phase was much lower than that required in the annealing of pristine In/Se pristine layer. It is similar to the observation of Co/Si case **[55].**

2.2.1 Summary on metal/semiconductor systems:

Different groups studied a large number of systems. Taking a view of all the results reported so far, the mixing in metal Si system is well explained by thermal spike considerations where it is assumed that during the transient melt phase the metal and Si species mix together due to enhanced diffusivity. Detailed joint experiments by IUAC Delhi, LMU Munich, MPI Stuttgart groups in conjunction with thermal spike calculations by CIRIL group comprehensively established this hypothesis of SHI induced mixing. Formation of high temperature phase a- $FeSi_2$ by the mixing of Fe and Si is additional proof of the existence of temperature spike.

2.3 SHI induced mixing in metal/ metal interface

The diffusion of oxygen across CuO/float glass interface under the influence of electronic excitation induced by SHI irradiation was observed and explained as a diffusion process

in transient melt phase. SHI induced IBM in oxide/oxide and metal/a-Si has also been explained on similar basis. The Coulomb explosion cannot be ruled out as a possibility for these cases of IBM. Therefore the SHI induced mixing in metal/metal system becomes important to investigate as the possibility of Coulomb explosion is completely ruled out.

2.3.1 IBM induced by swift heavy ion irradiation in Fe/Zr multilayers

Jaouen et al **[57]** studied SHI induced ion beam mixing and phase change for Fe/Zr multilayers. The multilayers had a modulation of 7.6 nm and an overall composition $Fe_{69}Zr_{31}$. The Zr layers were amorphous whereas the Fe layers were crystalline (bcc), having a strong (110) texture. The phase transformation and the composition changes were analyzed by the X-ray diffraction, RBS and Mossbauer spectroscopy. A complete mixing was observed at a fluence of 10^{13} U/cm^2. The electronic energy depositions in Zr and Fe by ion beam were 4.5 and 6.9 keV/nm respectively. Based on the experimental observation, It was suggested that mixing occurs in a two-stages. At first stage, the diffusion of iron atoms in the amorphous zirconium layers takes place across the interface. Subsequently in the second stage, ion bombardment leads to a generalized transformation in the entire film, similar to the plastic deformation phenomena as reported in irradiation in amorphous alloys.

Since both Fe and Zr are S_e sensitive and the U ions deposited very high electronic energy loss in the bilayer, which is more than the S_e threshold for both the layers. Therefore a complete mixing takes place.

2.3.2 SHI induced mixing induced in metallic bilayers

Leguay et al **[58]** established from SHI induced IBM experiments that high density of electronic energy deposition can induce damage creation in some metallic targets beyond certain threshold as high as, of the order of a few 10 keV/nm. The electronic excitations

induced ion beam mixing in metallic bilayers. Ni/Ti bilayers, irradiated at 80 K with GeV Ta ions was investigated by the X-ray and neutron reflectometry, X-ray diffraction, electron microscopy. A very strong interdiffusion across the interface indicating the ion beam mixing was reported in Ni/Ti system as a result of dense electronic excitations. The electronic energy depositions in Ni and Ti by the ion beam were 5.9 and 3.2 keV/nm respectively. Both Ni and Ti being S_e sensitive and the Ta ions depositing very high electronic energy loss in the bilayer, a very strong mixing takes place.

2.3.3 SHI induced mixing in Fe/Ni multilayer

Srivastava et al **[59]** carried out investigations on the effect of 120 MeV Au ion irradiation on a Fe/Ni multilayer. The characterization of pristine and irradiated films were made through X-ray diffraction, X-ray reflectivity, magneto-optic Kerr effect and four probe resistivity measurements. The formation of $FeNi_3$ and FeNi alloy phases, evidenced by XRD, were observed due to SHI induced IBM. The saturation magnetization of the film increased after irradiation because of the high magnetic moment of the mixed phase. The increased saturation magnetization after irradiation was an evidence of $FeNi_3$ phase formation. The measured resistivity of the pristine and irradiated multilayers are in agreement with the theoretically predicted values, considering the contributions from interfaces and grain boundaries. It is suggested that the mixing takes place due to an interdiffusion during the transient melt phase predicted by the thermal spike model for the consequences swift heavy ion beam irradiation. The diffusivity across the interface was determined using the experimentally obtained mixed layer thickness and the values of track radius as well as duration of melt state from thermal spike model. It was again found to be of the order of the diffusivity seen in the

liquid state. Thus here too the SHI induced mixing is confirmed to be due to the inter diffusion during transient melt phase.

2.3.4 Summary on metal/metal system: A close perusal of the above investigations in metal/metal systems reveals that almost complete mixing takes place when the layers are thin bilayers or multilayers, when the two layers are miscible and the electronic energy deposition is more than threshold for creating defects in these metals. When the two layers are immiscible, the IBM does not take place even if the S_e is beyond S_e threshold. However the mixing in immiscible system occurred at very high fluence.

The metal/metal layers become important for investigation of IBM because the SHI induced mixing due to the Coulomb explosion is ruled out. The energy deposited in electronic sub-system is quickly smeared out by conduction electrons and the Coulomb explosion does not occur. Other way to explain the same is that the plasmon frequency in metallic systems is of the order of 10^{15} Hz and therefore electron returns time to core is about 10^{-15} sec and the Coulomb explosion occurs in 10^{-14} sec. The electron return time being an order of magnitude smaller than explosion time, the positive core in the wake of ion passage is neutralized and the possibility of Coulomb explosion is ruled out.

2.3.5 Other on-line experiments to support diffusion during transient melt phase:

2.3.5.1 Transient enhanced diffusion of oxygen in Fe mediated by large electronic excitation

The transport of oxygen atoms in Fe film was monitored by on-line ERDA measurement **[60].** Three separate experiments were performed to ensure the reproducibility and to have authenticated data for the observed phenomenon. The experiments were performed using Au ions of 210, 243 MeV and I ions of 210 MeV ~from the Munich MP Tandem accelerator. The electronic energy loss (S_e) values for these ions in Fe are around 46, 47,

and 36 keV/ nm, respectively. The corresponding values for nuclear energy loss (S_n) are two orders of magnitude smaller. The recoils were detected by a position sensitive gaseous ionization detector ~having a solid angle of 5.7 msr at a scattering angle of 37°. Large solid angle allows the possibility of on-line monitoring and the position sensitive feature is used for the correction of kinematic broadening. It was evident from the recoil spectra shown in **figure 10** that the content of oxygen inside iron, and the full width at half maximum ~FWHM of the oxygen peak, increased with the ion fluence. This observed increase in FWHM indicated that the oxygen kept on diffusing in the Fe film with fluence. Although this observation is different from the ion beam mixing, yet there is a similarity in terms of atomic mass transport across the interface. The lattice temperature rise and decay is governed by two coupled differential equations. The lattice temperature variation with time is shown in **figure 11** as estimated using the coupled differential equations of thermal spike model. In this O absorption experiment the transport of oxygen inside the Fe film can be considered as mixing of oxygen with Fe at the interface of the Fe film and vacuum. The S_e values in the present experiment were larger than the threshold for creation of latent tracks in Fe film. The observed result is explained considering that the diffusion of oxygen in Fe takes place during the transient melt phase. From the fluence dependence of FWHM of the oxygen peaks, the diffusivity of oxygen in Fe was determined where the track radius and the duration of temperature spike were taken from thermal spike calculations.

The experimental evidences of the existence of transient temperature spike induced by SHI were comprehensively given by Avasthi et al. **[60].**

2.4 SHI irradiation studies of thermodynamically immiscible systems

2.4.1Study on the SHI induced interface modification of Pt/C and Ni/C

Te two systems are chosen in such a way that one S_e sensitive layer is common and one system is miscible and the other is not. It was reported by Gupta et al **[61]** that the mixing in the Ni/C multilayer and demixing in the Pt/C multilayer under 100 MeV ion irradiation, takes place. It is to be noted that Ni/C is miscible whereas Pt/C is immiscible. In both the multilayers, C is S_e sensitive. The electronic energy depositions by ion beam in Ni, Pt and C are 3.0, 3.7 and 1.4 keV/nm respectively.

In another experiment [62] as a collaboration between, Stuttgart university, IUAC Delhi and RBS College Agra, it was shown that there is smoothening at interface of Fe/Au as a result of SHI irradiation. The system Fe/Au being immiscible, again supports earlier observation [61] that SHI irradiation causes demixing at the interface of immiscible system.

2.4.2 SHI induced IBM study on the Fe/Bi interface

Gupta et al recently **[63]** reported that the Fe/Bi interface, which has positive heat of mixing, remains immiscible despite strong electronic energy deposition. It has been shown earlier that very strong mixing at interface is expected if both the layers at the interface are S_e sensitive. In the present case, both the Fe and Bi are Se sensitive, still no mixing is observed due to the fact that the heat of mixing is positive and thermodynamical concepts prevail, resulting in no interdiffusion at interface. This observation is a bit in contrast, with what is observed in low energy ion beam mixing. It has been reported that the mixing, the ion beam mixing can occurs in thermodynamically immiscible systems at low energies and high fluences, due to force recoils, cascade

collisions at the interface region. *SHI irradiation at very high fluence should be investigated for te possibility of SHI induced IBM in immiscible systems.*

2.4.3 Modifications in Tb/Fe amorphous multilayers under SHI irradiation

Richomme et al **[64]** studied the SHI induced interface modifications in amorphous Tb/Fe (an immiscible system) multilayers by irradiation with Kr, Xe, Pb and U ions at various fluences and were investigated by Mossbauer spectrometer. The demixing of Fe and Tb atoms at the interfaces was observed, which indicated that immiscible systems do not undergo SHI induced mixing. At very high fluence, the samples exhibit magnetic properties of amorphous alloys giving indication of mixing. The electronic stopping power threshold of mixing was suggested to be between 15 and 32 keV/nm. On the basis of experiments, one can say that the SHI induced mixing can occur in immiscible systems only at high fluences.

3. Further tests of thermal spike model in explanation of SHI induced mixing

By the conclusions drawn in section 2.1, 2.2, 2.3 and 2.4, it is quite convincing that the SHI induced mixing can be explained well in the framework of thermal spike model. There have been further theoretical calculations and experiments to test the explanations more rigorously as discussed below.

It is well known that mono-atomic ion beam of any ion at their Bragg peak energy do not create tracks in Si **[65**]. It was proposed **[23]** and theoretically predicted by CIRIL group in CAEN, France that although Si does not undergo a molten phase, still a very shallow region at the interface can be transiently molten because the temperature of molten metal layer is higher than that of the melting temperature of Si. The Si at the interface in small region gets melt due to heat energy transfer from the adjacent metal melt at higher temperature. It works well for the cases where the melting point of is more than that of

Si like in case of Fe,Co, V, etc The experiments were carried out at IUAC **[66,67**] taking such combinations where the S_e sensitive layer has the melting point more than that of other layer which is not sensitive to S_e, such as Fe and Au. Such a system showed ion beam mixing as expected. In another case where the S_e sensitive layer has the melting point lower than that of other layer which is not sensitive to S_e, such as Fe and . It did not show mixing as expected.

4. Conclusion on SHI induced IBM and future prospects

The cause of mixing is essentially a transfer of the energy deposited by the ions in the electronic subsystem to the lattice to cause atomic displacements. Out of the two possible models of generating atomic motions by SHI, namely Coulomb spike and thermal spike, the thermal spike definitely has an edge in explaining the results of SHI induced IBM as has been discussed in previous sections.

One can summarize the following points on the basis of a large experiments performed so far.

i. The two layers should be thermodynamically miscible, i.e. the heat of mixing should be negative) to get SHI induced mixing. In the immiscible systems, demixing occurs.
ii. One of the two layers must be S_e sensitive in order to have SHI induced mixing and the the mixing rate is quite large when both the layers are S_e sensitive. Even If one of the layers is S_e sensitive, the SHI induced mixing takes place if the melting point of the S_e insensitive layer is smaller than that of S_e sensitive layer.
ii. SHI induced mixing has a threshold of S_e, and the IBM increases with ion fluence.

iii. The SHI induced mixing is shown to be resulting from interdiffusion at the interface during transient molten state.

iv. The experiments of ion beam mixing in several metal/a:Si system shows that the content of mixed layer depends on the electron phonon coupling of the metal layer.

v. SHI induced mixing has not been reported in thermodynamically immiscible system which is in contrast with the low energy induced ion beam mixing of similar system. *This is an area, which requires further experiments and investigation.*

vi. Irradiation followed by annealing results in the formation of mixed phase at lower temperature than that obtained in annealing of the pristine samples, which is because of the enhanced diffusivity due to defects in irradiated sample.

vii. SHI irradiation of Metal/insulator system with immiscible combination results in formation of metal-insulator nanocomposite (metal particles embedded in insulator). *This is an important area of investigation where the kinetics for the growth of nanostructures needs to be investigated with low energy as well as high energy.*

viii. For the mixing in the metal/a:Si, dependence [53] of mixing rate with electron phonon coupling can be a guideline for predicting the amount of mixing in metal/a:Si system.

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Figure captions

Fig. 1: Schematic of ion beam mixing
Fig. 2 Recoil spectra of oxygen for Cuo film on glass.
Fig. 3 Mossbauer spectra of pristine and irradiated Fe/Si by different swift heavy ions.
Fig. 4 Distribution of Fe and Si at different fluences as recorded by high resolution RBS.
Fig. 5 Schematic of the ion beam mixing showing molten Si, only at the interface, whereas Fe melts in the whole thickness of the film within ion track.
Fig. 6: TEM images of the interface showing the Ni_2Ge phase.
Fig. 7 SIMS spectra of the pristine and SHI irradiated Co/Si.
Fig. 8: XRD of the SHI irradiated Co/Si showing the formation of Cobalt silicide
Fig. 9 RBS of the pristine and irradiated and annealed samples of Co/Si.
Fig. 10 Recoil spectra of oxygen at different fluences for the Fe/Si showing the increase of oxygen and subsequent diffusion in the Fe film.
Fig. 11 The temperature of Fe lattice as a result of passage of 210 MeV Au in Fe according to the thermal spike model.

Figure 1

From Ph.D. thesis, 2002, S.K. Srivastava, JNU Delhi

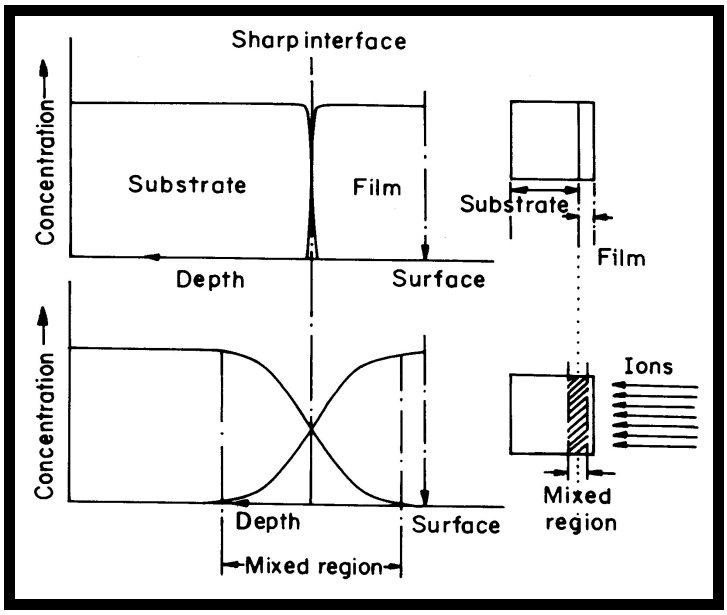

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Figure 2

from Ref. 42

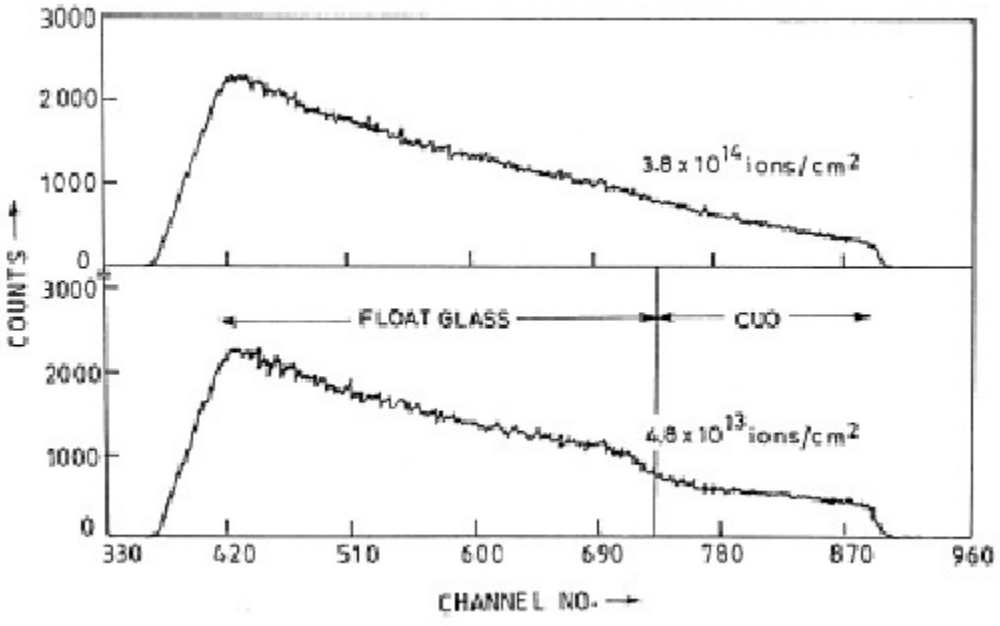

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Figure 3

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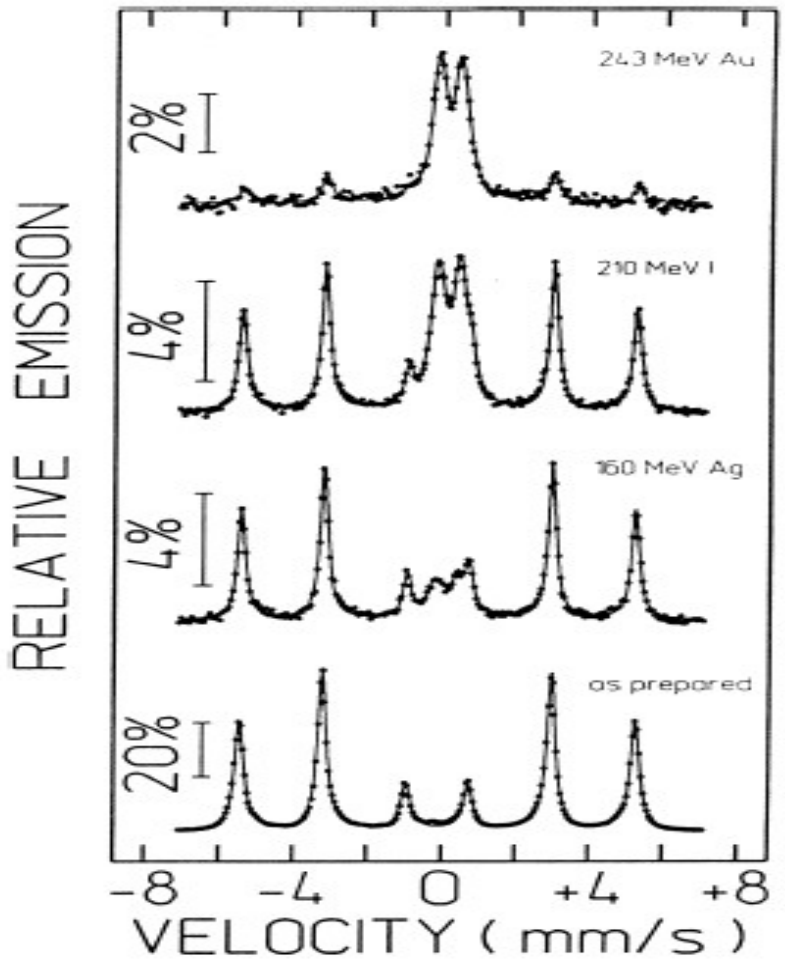

Figure 4

from Ref. 23

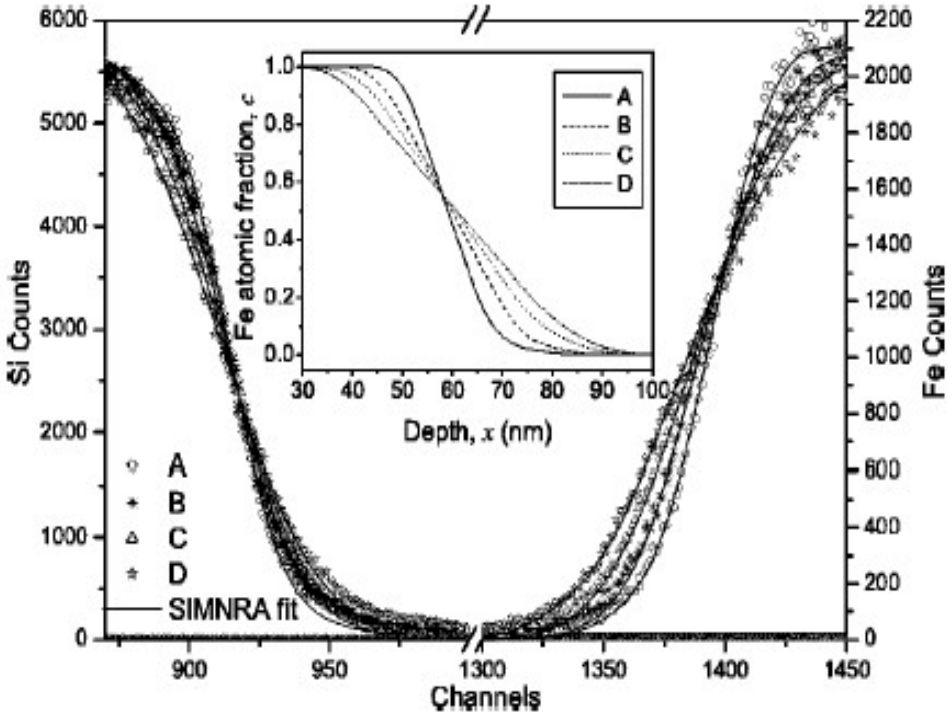

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Figure 5

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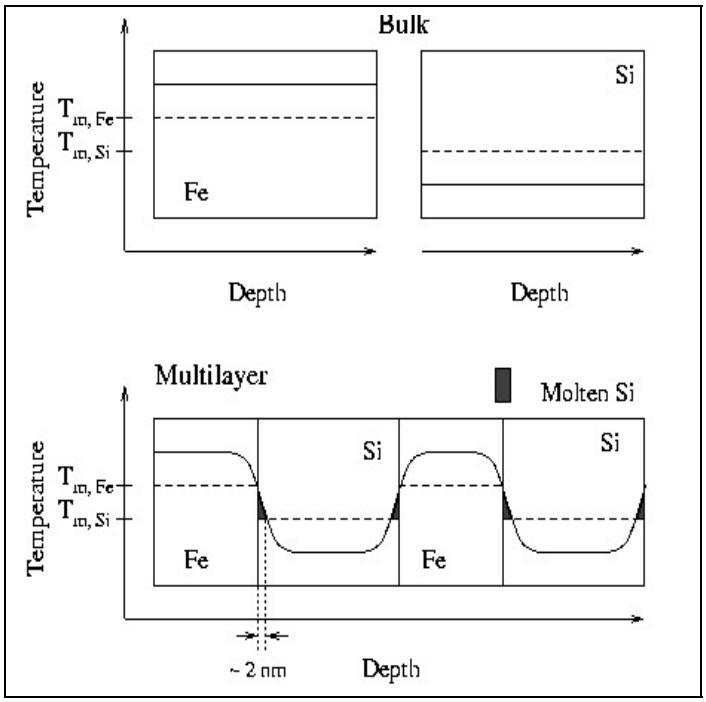

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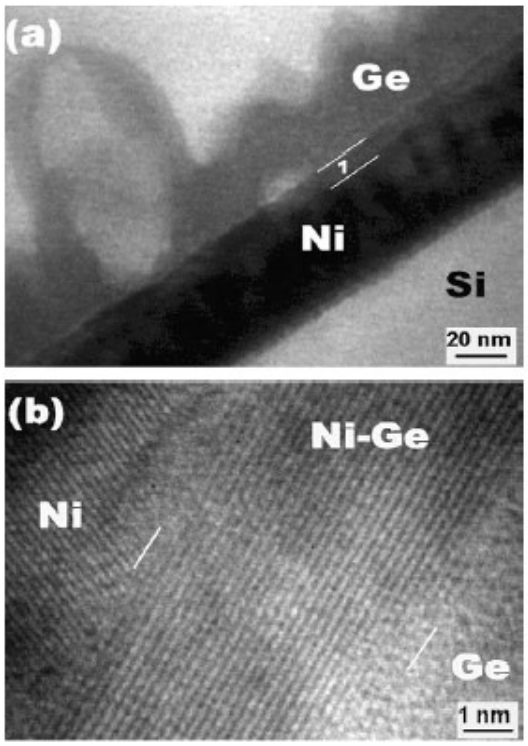

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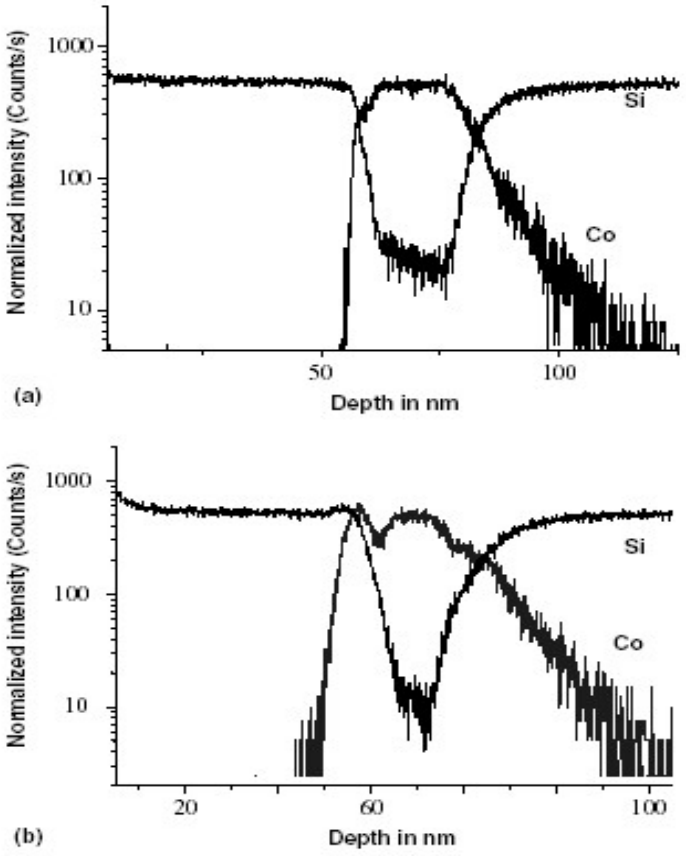

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Figure 8
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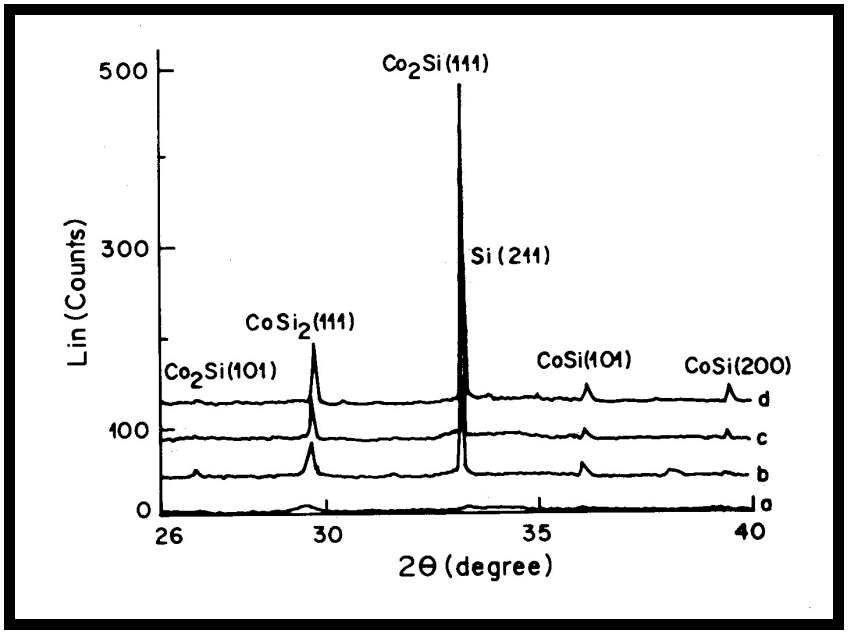

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Figure 9
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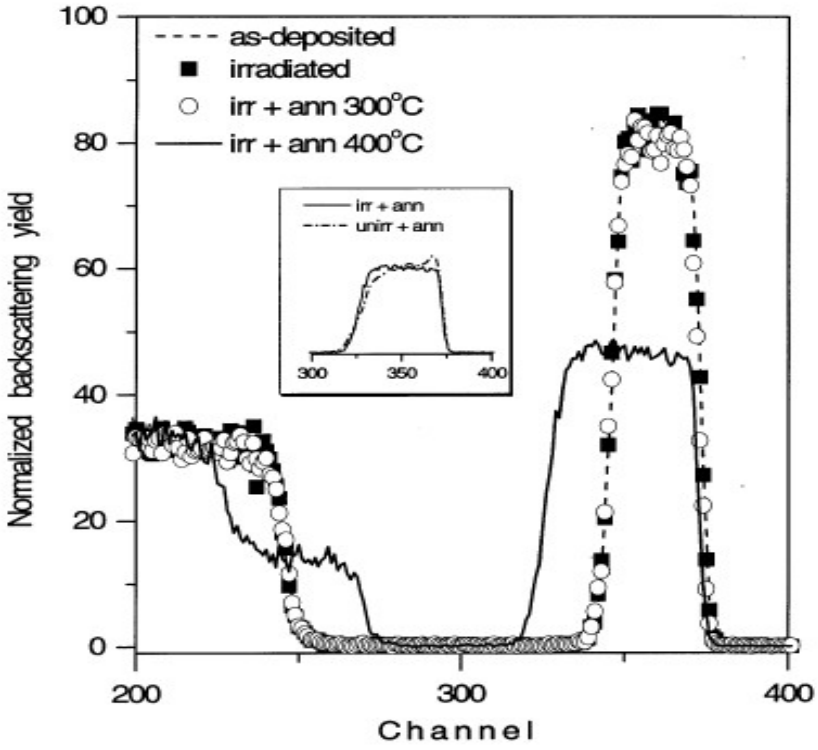

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Figure 10

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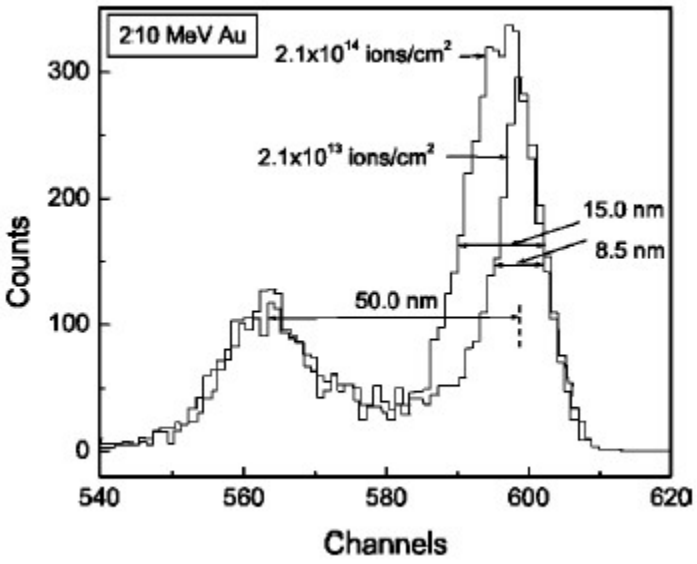

Figure 11

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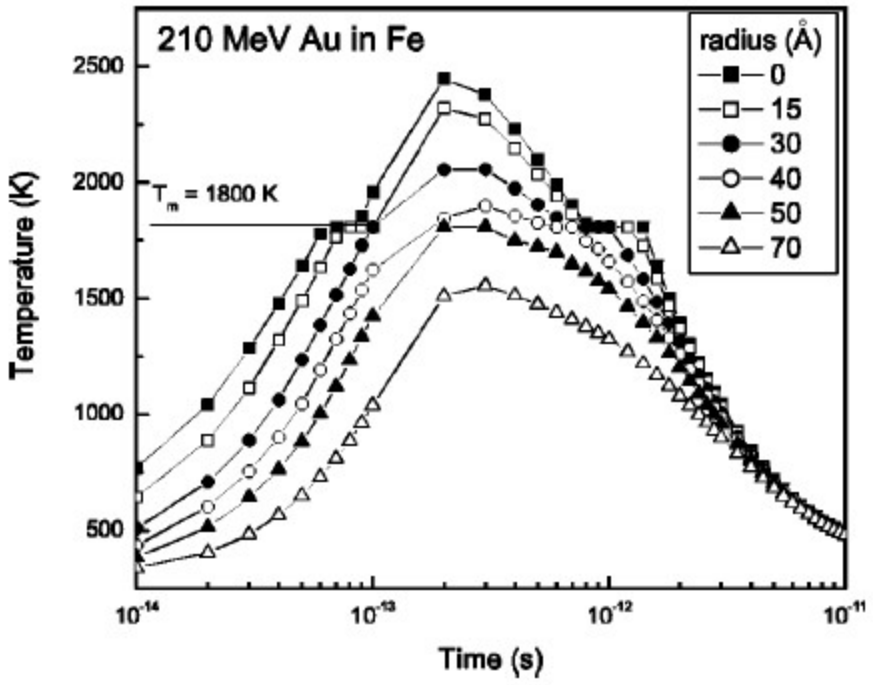

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