CHARACTERIZATION OF SEMICONDUCTORS BY MEV HE$^+$ BACKSCATTERING SPECTROMETRY, CHANNELING AND DOUBLE CRYSTAL DIFFRACTION

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Abstract

Double Crystal Diffractometry (DCD) is used to characterize the strain produced by 2 MeV He$^+$ at room temperature to simulate a Rutherford Backscattering Spectrometry(RBS) analysis of bulk Si, Ge and GaAs. The irradiation-induced strain is very small in both Si and Ge and persists after 15 min annealing at 400°C. The strain is significant in GaAs and largely reversible upon annealing. The capabilities of DCD are further demonstrated on an example involving characterization of a CoSi$_2$ epilayer grown on a Si(111) substrate whose surface is slightly offset from the (111) plane by an angle ϕ_s in the [1$\bar{1}$0] direction. There is a very small misorientation between the Si(111) and CoSi$_2$(111) planes. The misorientation angle, α, between these two planes is a fixed fraction 0.017 of the offset angle, ϕ_s, up to $\phi_s = 4°$. A simple geometrical model is proposed to explain the observation. The model agrees quantitatively with the experiment.

I. Introduction

To develop and make semiconductor devices, it is important to have nondestructive techniques. RBS is a common analytical tool for the analysis of composition and thickness of thin film[1]. In this paper, the strain induced by an RBS analysis in (100) oriented Si, Ge and GaAs crystals and the recovery by subsequent furnace annealing are presented. Due to the high sensitivity of the x-ray diffraction to the strain in a single crystal and the nondestructive nature of x-rays for semiconductors, DCD is an outstanding technique to detect very small radiation damage levels[2,3].

DCD is also an important tool for characterizing heteroepitaxial structures[4]. This is illustrated by the measurement of a very small misorientation angle($< 0.07°$) between the epitaxial CoSi$_2$(111) layer and the offset Si(111) substrate[5].

II. Strain Induced by RBS Analysis

A 2 MeV He$^+$ beam was incident on (100) oriented Si, Ge and GaAs(100) crystals at room temperature, with a typical RBS dose of $\sim 10^{16}$/cm^2. X-ray rocking curves were taken before and after RBS analysis. After room temperature measurements, the samples were annealed for 15 min at temperatures increasing in steps of 100°C up to 400°C in flowing Ar gas.

RBS is routinely used to characterize near surface layers and thin films. There is not much information about radiation damage induced by He$^+$ in Si in the MeV range. A recent paper[6] states that at room temperature 1 MeV He$^+$ has a threshold dose of 2×10^{17}/cm^2 for flaking, which is an order of magnitude above the typical dose of RBS analysis. DCD study considers 250 KeV Si$^+$ implantation in Ge, Si and GaAs at room temperature. In both Si and Ge, the strain induced by irradiation rises linearly at low doses. The strain in Ge rises 10 times faster than the strain in Si[7]. In our experiments, the induced strain in both Si and Ge is very small. The rocking curves only show a small broadening of substrate peak after RBS [Fig.1(a),(b)]. We do not observe a great difference of induced strain between Si and Ge. The reason for this contrast with Si$^+$ irradiation is related to the difference in the experimental conditions, in particular the mass and energy of the incident ions. Figure 2 demonstrates the strain recovery for Si after annealing at 400°C for 15 min. Although detectable, the observed strain relaxation is small. The results for Ge are similar.

In contrast to Si and Ge crystals which show only a small broadening of the rocking curves upon irradiation, the GaAs rocking curves develop a definite structure [Fig.1(c)]. It indicates that a layer with a strain on the order of 0.1% is created. Paine et al.[8] measured the strain induced by 100 KeV He$^+$ irradiation in GaAs at room temperature as a function of dose and established that the strain rises linearly with the dose for strain below 0.4%. We thus conjecture that the strain in our experiment falls in that region, meaning that the strain rises linearly with

the dose of irradiation. This conjecture is supported by other data: irradiation of Ne, Si and Te all yield linear dose dependences below strains of $\sim 0.3\%$[9]; a 15 MeV Cl irradiation established that at 0.4%, the strain ceases to rise[10]. Figure 1(c) shows the strain relaxation upon post furnace annealing, which is similar to that described for the Cl irradiation[10]. The thermal annealing behavior of the strain recovery in GaAs is clearly different from that in Si and Ge.
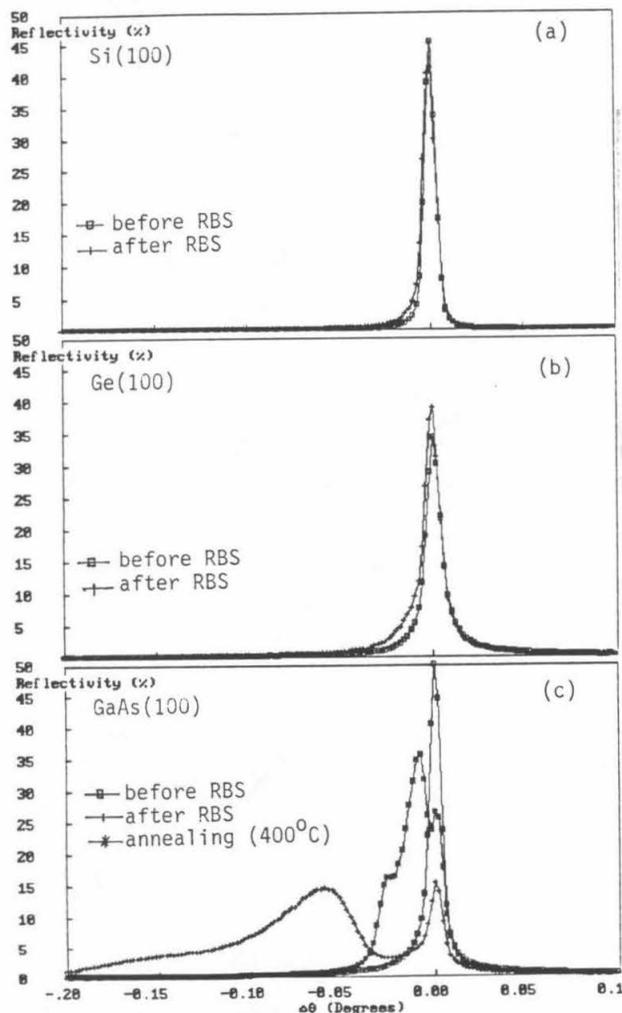

Figure 1. X-ray rocking curves from (400) diffraction plane of (100) oriented Si(a), Ge(b), and GaAs(c) before and after RBS analysis.

These results indicate that RBS is practically a nondestructive analytical method for Si and Ge crystals in terms of lattice strain. This is not the case for GaAs. Channeling investigations can require much larger exposure than conventional RBS studies; damage-induced strain may then become significant even for Si and Ge.
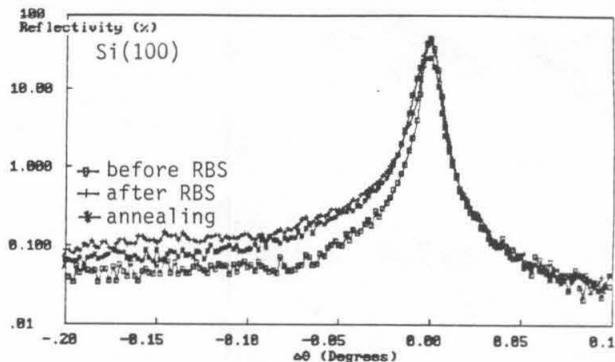

Figure 2. Log plot of x-ray rocking curves from (400) diffraction plane of Si(100) before and after RBS analysis, and after furnace annealing.

III. Misorientation between CoSi$_2$(111) and Si(111)

A set of mechanically polished (111)Si wafers of three typical offset angles, $\phi_s \approx 0.1°, 2°, 4°$, were used as the substrates for studying the misorientation effect. Wafers were cut towards the $[1\bar{1}0]_s$ direction with offset angles ϕ_s. The Si surface was cleaned by the usual RCA procedure. The residual oxide layer was stripped off by either flash heating to 900°C or Si beam self cleaning in the UHV chamber. The base pressure was 10^{-10}Torr. A stochiometric film of CoSi$_2$ was deposited on the Si substrate heated to about 550°C in a vacuum of 10^{-9}Torr, by keeping the flux ratio of the Si and Ge deposition beams close to 2:1.

Samples of (111)CoSi$_2$ epilayers on (111)Si substrates ranging in thickness from 100Å to 2000Å, were prepared under similar MBE conditions. RBS and channeling were employed to characterize the stochiometry, thickness, and crystal quality of the epitaxial layer. Some samples were analyzed by both plane-view and cross-sectional TEM to reveal pinholes, dislocation networks and interface structures. Back diffraction Laue x-ray was used to measure the offset angle, ϕ_s. DCD from symmetrical (111) and (333) diffraction planes were used to obtain the average perpendicular strain, $\epsilon^\perp$, and the misorientation angle, α. The sample was mounted such that the $[111]_s$ direction of the Si substrate was in the plane of the incident beam $\hat{k}_i$ and of the surface normal $\hat{n}$ (i.e., x-ray beam $\hat{k}_i$ was in the $(11\bar{2})$ plane)[Fig.3]. From the two rocking curves, the average perpendicular strain, $\epsilon^\perp$, and the misorientation angle, α, can be obtained [Fig.4],

$$\epsilon^\perp = -\cot\theta_B \cdot \frac{\triangle\theta_I + \triangle\theta_{II}}{2},$$

$$\alpha = \frac{\triangle\theta_I - \triangle\theta_{II}}{2},$$

where θ_B is the Bragg angle, and $\triangle\theta_{I,II}$ is the rocking curve peak separation for the two diffraction configurations I,II of Figure 4. Results from back reflection Laue and DCD analyses are summarized:

(1) The $[111]_f$ direction of the CoSi$_2$ film lies between the surface normal $\hat{n}$ and the $[111]_s$ direction of the Si substrate [Fig.3].

(2) The magnitude of the misorientation angle α is proportional to the offset angle ϕ_s [Fig.5]. The ratio is 0.017 and does not depend on the layer thickness t_f.
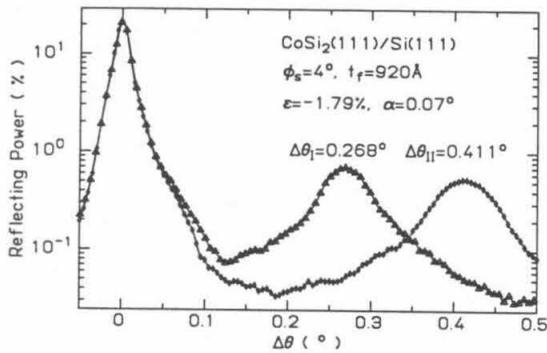
Figure 3.   X-ray rocking curves from the (111) symmetrical diffraction plane of the same CoSi$_2$/Si sample, for two different configurations.
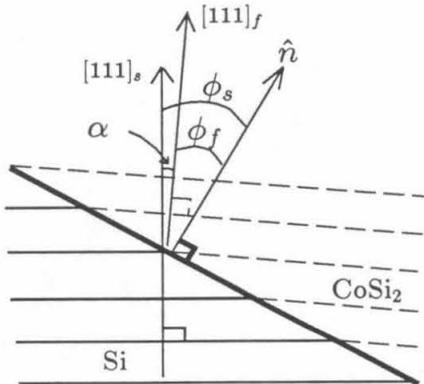

Figure 4.   Misorientation between the (111) planes of the substrate Si and the epilayer CoSi$_2$.

Based on the experimental findings and current knowledge about the interfacial structure of the epitaxial silicide and Si (in particular, CoSi$_2$ on Si(111))[11,12], we propose a simple geometrical model to correlate the geometrical quantities of the epilayer and substrate at the interface. Assuming "length matching" across the interface[Fig.6],

we obtain the following relation

$$l_e = \frac{d_f^\perp}{\sin\phi_f} = \frac{d_s^\perp}{\sin\phi_s} = l_s.$$

Simple algebra gives the misorientation angle $\alpha(\equiv \phi_f - \phi_s)$

$$\alpha = \epsilon^\perp \times \tan\phi_s,$$

to the first order in α for small α, where

$$\epsilon^{\perp,\parallel} = (d_f^{\perp,\parallel} - d_s^{\perp,\parallel})/d_s^{\perp,\parallel}$$

is the x-ray strain. This geometrical model predicts that α is proportional to ϕ_s for small offset angles ϕ_s. The least squares fitted linear function from the experimental data are compared with model calculation. Excellent agreement is obtained [Fig.5]. Misorientation between epilayer and offset substrate has been observed for other heteroepitaxial structures[13].
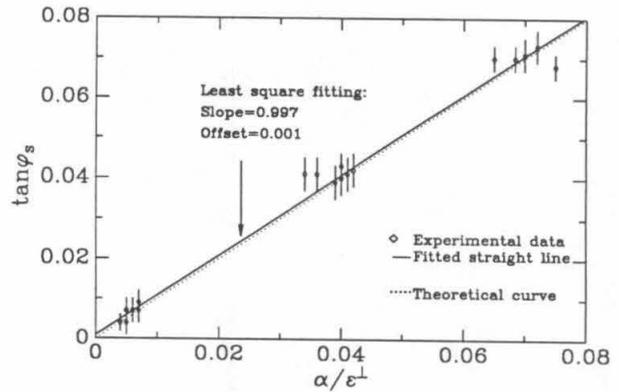

Figure 5.   Relationship between the misorientation angle α and the offset angle ϕ_s of CoSi$_2$/Si(111) bicrystal.
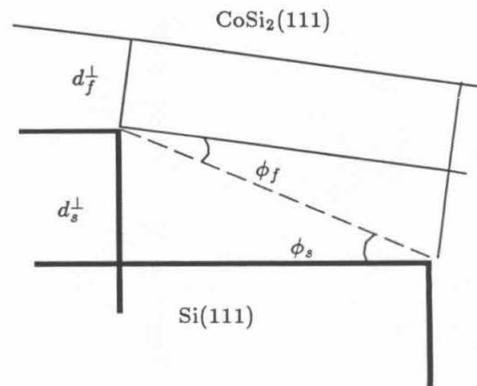

Figure 6.   Geometrical model of the misorientation between the CoSi$_2$(111) epilayer and the Si(111) substrate.

Acknowledgement

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