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AN X-RAY STANDING WAVE ANALYSIS

by

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ABSTRACT

The atomic structure at the interface of single-crystal $NiSi_2$ films on Si(111) has been determined with x-ray standing waves. Two types of films have been investigated, one with the same crystal orientation as the substrate (A-type), and the other with its orientation 180^0 rotated about the surface normal (B-type). For both orientations, the Ni atoms at the interface are found to be 7-fold coordinated. The bonds across the interface are contracted by 0.04 ± 0.05 Å for the A-type film and 0.11 ± 0.03 Å for the B-type film. For the first time the strain in an overlayer has been measured by the x-ray standing wave technique, using a Moiré effect between the overlayer lattice planes and the x-ray standing wave field. The (111) interplanar distance in $NiSi_2$ is found to be contracted by 0.4%.

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1. INTRODUCTION

The hetero-epitaxial $NiSi_2$-Si(111) interface has been extensively studied in recent years. It may serve as a model system for a proper understanding of metal-silicon Schottky Barrier formation, but then the interface atomic structure has to be known accurately, including possible relaxations [1]. The $NiSi_2$ overlayer can either have the same orientation as the substrate (A-type) or can be rotated by 180^0 about the surface normal (B-type). Tung et al. [2] have developed a method to grow silicide overlayers which have either type of orientation.

The Si-dangling bonds of the Si(111) substrate can either be attached to the Si atoms or to the Ni atoms in the silicide, resulting in 7- or 5-fold coordinated Ni atoms at the interface respectively. Fig. 1 shows the difference between these two possibilities for a B-type film. The structure at the interface has been studied by various techniques [3-6]. Van Loenen et al. [4] investigated B-type overlayers with ion blocking and found that the Ni atoms at the interface are 7-fold coordinated. This result is in agreement with the TEM results by Cherns et al. [5] which favoured a 7-fold coordination for both A- and B-type films. Akimoto et al. [6], however, concluded from x-ray standing wave (XSW) analyses on A-type overlayers that the interface Ni atoms are 5-fold coordinated. Only the measurements by Van Loenen et al. [4] were sensitive enough to determine the relaxation at the interface.

With regard to the XSW method, the relevant difference between the 5- and 7-fold coordinated interface is the distance d_{IF} of the first Ni layer to the last (111) diffraction plane of the substrate (fig. 1). Using bulk bond lengths, this distance can be calculated to be 2.73 Å and 3.52 Å for the 5- and 7-fold coordinated interfaces respectively. Several recent XSW measurements with synchrotron radiation [7] have shown that with this technique it should be possible to distinguish clearly between these interface structures and to determine the relaxation as well. Accordingly, the first goal of this study is to determine with XSW the Ni coordination number for both the A- and B-type interface and to measure the relaxation at the interface. The second goal is to measure the strain in the overlayer. The lattice parameter of bulk $NiSi_2$ is 0.46% smaller than that of Si. This very small mismatch enables the growth of pseudomorphic silicide overlayers that are relatively thick. Tung et al. [2] found that $NiSi_2$ films of less than 60 Å thickness were free of dislocations. The pseudomorphic growth implies that the $NiSi_2$ lattice stretches in the directions parallel to the surface, which will be accompanied by a contraction in the normal direction. The value for the spacing of the (111)-planes is 3.135 Å for Si and 3.121 Å for bulk $NiSi_2$.

2. X-RAY STANDING WAVE ANALYSIS

The XSW formalism used here to evaluate the measured data has been described for implanted layers [8], for surface adsorbates [9] and for bulk atoms [10]. We shall here discuss its extension for the case of single-crystal epitaxial layers. If only one beam is Bragg reflected from a crystal, the photon Bloch wave eigenstate inside the 3-dimensional periodic crystal array consists of two partial waves. In the region above the surface, the incident and reflected waves interfere within the limits given by the photon coherence length. Each truly periodic overlayer will influence this interference pattern extending across the interface. For XSW this effect can be neglected as long as the layer is very thin compared to the extinction depth in the layer material. In the following we will strictly constrain the mathematics and the experimental situation to this condition.

Fig. 2 shows schematically the interface region of a thin film on top of a substrate. In the substrate, the distance between diffraction planes (which are here parallel to the surface) is d_H (= $1/|\vec{H}|$). It is further assumed that the overlayer contains atoms of a different element, which are arranged in N lattice planes, having a spacing d_{OL} and starting at a distance d_{IF} from the last substrate plane. In the case of $NiSi_2$ these planes run through the Ni atoms as indicated in fig.1.

The intensity of the standing wave pattern at position $\vec{r}$ close to the surface is given in normalized form by [8]

$$\frac{I(\theta)}{I_o} = 1 + R(\theta) + 2\sqrt{R(\theta)}\ \cos(\nu(\theta) - 2\pi\vec{H}\cdot\vec{r}), \tag{1}$$

where the reflectivity R and the phase factor ν can be computed from dynamical theory as a function of the reflection angle θ [10]. In the present case, $\vec{H}\cdot\vec{r} = z/d_H$ where z is the direction perpendicular to the diffraction plane. When rocking through the Bragg reflection range the phase factor ν will change by π, causing the antinodes which at the low angle side are lying halfway between the substrate diffraction planes, to shift half a lattice spacing inwards. This is also shown in fig. 2.

We are interested in the fluorescent radiation that is emitted by the atoms in the overlayer planes. These atoms are exposed to the standing wave pattern generated in the substrate. In general the distances d_H and d_{OL} will not be equal and the positions of the overlayer atoms with respect to the standing wave nodes will shift from layer to layer. This will cause a kind of Moiré effect as described below. The fluorescence yield is given by

$$Y(\theta) = C\int_o^\infty I(\theta,z)\,\rho(z)\,dz\,, \tag{2}$$

where ρ(z) is the z-projected density distribution of the fluorescence selected overlayer atoms [8] and C includes all constant parameters.
Assuming that all atoms are at their exact lattice positions and stacked in N planes, we may take

$$\rho(z) = \sum_{k=o}^{N-1} \delta(z - d_{IF} - kd_{OL})\,. \tag{3}$$

Substituting (1) and (3) into (2) gives after some straightforward calculations:

$$\frac{Y(\theta)}{CI_oN} = 1 + R(\theta) + 2\sqrt{R(\theta)}\ f_c\,\frac{\sin(\pi N\gamma)}{N\sin(\pi\gamma)}\ \cos\left[\nu(\theta) - 2\pi\delta - \pi(N-1)\gamma\right], \tag{4}$$

where we introduced the distance parameter $\delta = d_{IF}/d_H$ and the parameter $\gamma = (d_{OL}/d_H) - 1$ which is related to the strain and the mismatch of the overlayer. We also introduced the coherent fraction f_c, which is in the present model the fraction of overlayer atoms which are positioned exactly in the N planes. The terms in eq.(4) containing γ describe the Moiré effect. For $\gamma = 0$ there is no change in lattice spacing across the interface and the Moiré effect disappears. Eq.(4) can be written in the general form:

$$\frac{Y(\theta)}{CI_oN} = 1 + R(\theta) + 2\sqrt{R(\theta)}\,F\ \cos(\nu(\theta) - 2\pi D)\,, \tag{5a}$$

where

$$F = f_c\,\frac{\sin(\pi N\gamma)}{N\sin(\pi\gamma)}\,, \tag{5b}$$

and

$$D = \delta + \frac{1}{2}(N-1)\gamma . \tag{5c}$$

The parameters F and D describe all possible yield curves and these are the parameters that can be determined experimentally. A complete structure determination requires the determination of both δ and γ. In principle δ can be determined by measuring an overlayer with a thickness of one lattice plane, because for $N = 1$ the parameter γ drops out of F and D. Once δ is known, γ can be determined by measuring a thicker layer. In general, it is necessary to measure at least two samples with different thicknesses. The thinner sample will be relatively sensitive to δ and the thicker one will be relatively sensitive to γ. Knowledge of the thickness of the overlayer is important, because it cannot be directly determined from the shape of the fluorescence yield.

In the model, the spacing d_{OL} is assumed to be uniform. This may not be true; the spacing will vary slightly if the strain is inhomogeneously distributed over the film. The measurements reported in this paper, however, can well be described by eq.(5) and the results are not very sensitive to such variations in d_{OL}.

3. EXPERIMENT

The samples (6 x 10 x 0.38 mm^3 in dimension) were cut from a Si(111) wafer. An important problem was to clean these thin samples and at the same time preserve the crystal quality. Stresses related to an inhomogeneous temperature distribution over the sample during annealing can easily cause imperfections and complicate XSW experiments. The samples were cleaned using the "Shiraki method" [11]. As the last step of this method, the samples were annealed at 800°C in a ultrahigh vacuum system with a base pressure of 5×10^{-11} Torr. Auger measurements after cleaning showed that the carbon contamination was less than 5% of a monolayer. In order to keep the temperature over the sample as homogeneous as possible, radiative heating was used. This was done by mounting a somewhat larger piece of Si-wafer just below the sample and heating it to approximately 1100°C. In this configuration no current is passing through the sample and it needs to be clamped only very slightly, again minimizing the stress. In one case, Si clamps were used, in the other two cases Ta clamps. Both configurations worked well and the temperature variation over the sample was within ± 10°C.

The Ni was deposited by direct sublimation from resistively heated Ni wires at deposition rates in the range from 0.5 to 2.0×10^{14} atoms/cm^2/min. The method of Tung et al. [2] was used to grow overlayers with either A- or B-type orientation. Van Loenen et al. [3] found that a deposition of at least 5×10^{15} Ni atoms/cm^2 is needed to grow a uniform B-type NiSi$_2$ overlayer. Accordingly we deposited 6×10^{15} Ni atoms/cm^2 at room temperature to make our thinnest B-type sample. In order to form the epitaxial NiSi$_2$ overlayer, the sample was subsequently annealed at 550°C for 5 min. This treatment resulted in a film containing $N = 8$ Ni planes. In order to grow a thick B-type sample, first a thin NiSi$_2$ layer was grown and then a new layer of approximately 4×10^{15} Ni atoms/cm^2 was deposited and annealed. This was repeated until a silicide layer with a total thickness of approximately 35 Ni planes was grown. The A-type sample was grown by depositing 16×10^{15} Ni atoms/cm^2 and annealing. The thickness of the deposited layers was checked with medium-energy ion scattering (100 keV H$^+$) during growth. After the XSW measurements, the deposited quantities were determined more accurately using high-energy scattering (2 MeV He$^+$). The N values are given in table 1.

The samples were grown at the FOM-Institute, and the XSW analyses were performed at HASYLAB. In order to protect the samples from contamination during transport and analysis (all in air), the samples were capped by an amorphous layer of Si with a thickness of 50 Å.

The XSW measurements were done at the Wiggler beamline in HASYLAB. The experimental set-up is schematically shown in fig. 3 and is described in detail elsewhere [12]. The synchrotron light from the 32 pole Wiggler is monochromatized by a double-crystal monochromator (with an asymmetrically cut second crystal) and directed onto the sample. Scans through the rocking curve are made by varying the energy of the monochromatized beam, while the sample remains fixed in position [12]. The (111) Bragg reflected x-ray intensity is detected by a NaI(Tl) detector. The fluorescent radiation is detected by a Si(Li) solid state detector.

The energy of the incoming x-ray beam was 12.4 keV in the case of the thin B-type sample and 14.0 keV for the two other samples. For each sample a rocking curve was measured versus $\theta - \theta_B$, where θ_B is the Bragg angle. A typical curve is shown in fig. 4a. The measured Ni K_α fluorescence yields for the three samples are shown in figs. 4b-d. For each sample a high-resolution double-crystal topograph [13] was taken to survey the sample perfection and the sample mounting which can easily cause lattice strain. Perfect sample regions (4-10 mm^2) were then selected for the XSW measurements.

4. RESULTS AND DISCUSSION

The structural parameters D and F (eqs.(5b) and (5c)) were determined for each of the samples using the analysis described in [8]. The results of the χ^2- fits are shown as solid curves in figs. 4a-d, the optimal values for the parameters F and D are given in table 1.

We have determined D for two B-type samples with different number of lattice planes N. Eq.(5c) then provides two equations with δ_B and γ_B as unknowns. Solving this, we obtain:

$$\delta_B = 1.089 \pm 0.009 ; \qquad \gamma_B = -0.0088 \pm 0.0013 .$$

Multiplying δ_B by d_{111} gives the distance of the first Ni layer relative to the last (111) diffraction plane in the crystal:

$$d_{IF}^{B}(\mathrm{exp}) = 3.41 \pm 0.03 \text{ Å} .$$

Comparing this result with the corresponding distances for the 5- and 7-fold coordinated interfaces (2.73 Å and 3.5 Å), we conclude that the B-type film is 7-fold coordinated and that the distance d_{IF}^{B} is 0.11 ± 0.03 Å smaller than the value calculated from bulk bond lengths. This change in distance is the result of a contraction of the Si-Si bond across the interface and/or an angular distortion of the Si-Si and Si-Ni bonds on either side of it (see fig. 1b). The precise location of the distortion could not be deduced from the present measurements. The physical origin of the interface contraction is not yet understood.

The measured distance d_{IF}^{B} agrees well with the ion channeling analysis by Van Loenen et al. [4]. They found a value of 3.45 ± 0.08 Å for a thin B-type overlayer, which corresponds to a contraction by 0.07 ± 0.08 Å. To illustrate the sensitivity of XSW in determining δ, fig. 4b also shows the normalized yield curves expected for 5- and 7-fold coordinated interfaces, using bond lengths and γ as determined above.

The sensitivity for the strain parameter γ is illustrated in fig. 4d for the thickest B-type film. The solid line is the best fit from which the strained value for γ is determined. The dashed line is the normalized yield for a NiSi$_2$ film which is unstrained in the perpendicular direction ($\gamma = -0.0046$). From $\gamma_B = -0.0088$, the spacing between the (111)-planes in the B-type NiSi$_2$ film is computed:

$$d_{111}(\mathrm{NiSi_2\ film}) = 3.108 \pm 0.004 \text{ Å} .$$

This is 0.4% smaller than the bulk value. Thus the stretching parallel to the surface as is implied by the pseudomorphic growth, is accompanied by a contraction in the perpendicular direction. From elasticity theory the perpendicular component $\varepsilon_\perp$ of the strain in the layer can be related to the parallel component $\varepsilon_{||}$ [14]. Here these components are defined with respect to bulk NiSi$_2$. One can write:

$$\varepsilon_\perp = -\alpha\varepsilon_{||} ,$$

where α is positive and can be written in terms of the elasticity constants. In this case the ratio α (not be be confused with the Poisson ratio) cannot be calculated, because the elasticity constants in NiSi$_2$ are unknown. For a few other crystals with the fluorite structure (e.g. CaF$_2$, SrF$_2$ and ThO$_2$) the elasticity constants are known [15] and for these crystals α falls in the range from 0.85 to 0.95. From the experimental results for $\varepsilon_{||}$ and $\varepsilon_\perp$ we can determine an experimental value for α of NiSi$_2$:

$$\alpha = 0.9 \pm 0.3 .$$

This value is nicely within the range of the theoretical α values for the other fluorite type crystals.

Using ion channeling and blocking, Van Loenen et al. [3] measured for thin B-type NiSi$_2$ films on Si(111) a d_{111} of 3.094 ± 0.007 Å. This is somewhat smaller than the value determined here. The difference may be due to the fact that ion scattering is more sensitive to the outer layers, which could be relaxed and cause steering of the ion beam, whereas with x-ray standing waves an average of the spacing over the whole overlayer is obtained.

The difference between A- and B-type oriented silicide is only a rotation of 180° about the surface normal and therefore α should be the same for both orientations (pseudomorphic growth) and therefore the perpendicular strain components should be equal as well. Thus we may take

$\gamma_A = \gamma_B$. This enables the determination of the distance parameter for the A-type sample, even though only one thickness was investigated. Using eq.(5c), we obtain:

$$\delta_A = 1.109 \pm 0.016,$$

which gives

$$d^A_{IF}(\exp) = 3.48 \pm 0.05 \text{ Å} .$$

Thus also the A-type $NiSi_2$ interface is 7-fold coordinated, but with a contraction of 0.04 ± 0.05 Å. The contraction is smaller than that found at the interface of the B-type silicide, though the error margins on the determined values do not allow us to conclude to a significant difference with a high degree of confidence. At any rate, a substantial contraction is not to be expected for the A-type overlayer system, as it represents the natural stacking sequence of (111) planes on crossing the interface.

Knowing γ and the thickness of the overlayers, and using eq.(5b), the coherent fraction f_c can be computed from the parameter F. The coherent fraction is a measure of the crystalline quality of the silicide film. For the thin B-type sample we find $f_c = 0.66$, for the A-type sample $f_c = 0.92$ and for the thick B-type sample $f_c = 0.85$. The A-type and the thick B-type overlayers are highly perfect. The lower quality of the thin B-type film probably relates to random surface displacements induced by the presence of the a-Si cap or by impurities diffusing through the cap.

The reason that Akimoto et al. [6] found a 5-fold coordination for the interface Ni atoms, instead of the 7-fold coordination found here and by others [4,5], may be related to layer inhomogenities and/or substrate strain in the samples that Akimoto et al. have used. These effects will always reduce the coherent fraction, which is close to ideal in our study. Furthermore, Akimoto et al. used relatively thick overlayers (≈ 35-115 Ni planes) for which the thickness was less accurately determined (using a quartz-crystal thickness monitor). For thick overlayers the measurement becomes very sensitive to the perpendicular strain, making it difficult to deduce the distance parameter δ, see eq. (5c). Moreover, in such thick layers dislocations could be formed, leading to a partial (and possibly inhomogeneous) relief of the strain.

5. CONCLUSION

X-ray standing wave measurements allow an accurate determination of relaxed distances at the interface of an epitaxial thin-film system. The lattice strain in the film can be measured as well.

For both A- and B-type oriented $NiSi_2$ films on a Si(111) substrate the Ni atoms at the interface are found to be 7-fold coordinated. The bonds across the interface show a contraction of 0.04 ± 0.05 Å for the A-type film and 0.11 ± 0.03 Å for the B-type film.

In order to be pseudomorphic with the substrate, the $NiSi_2$ lattice must be slightly stretched in the lateral direction. This stretch is accompanied by a perpendicular strain of opposite sign. The latter strain component has been measured with x-ray standing waves. We find a contraction of the (111) interplanar distance by 0.4%.

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Table 1

The experimentally determined parameters for the three investigated samples. The thicknesses have been measured by RBS. The parameters *F* and *D* describe the shape of the fluorescence yield curves and have been determined by fitting the data in figs. 4b-d.

$NiSi_2$ overlayer	thickness N (no. of Ni planes)	*F*	*D*
B-type	8.2 ± 0.8	0.65 ± 0.02	1.057 ± 0.006
A-type	21.1 ± 2.1	0.87 ± 0.02	1.021 ± 0.003
B-type	36.5 ± 3.7	0.72 ± 0.01	0.933 ± 0.003

FIGURE CAPTIONS

Fig. 1

The atomic structure of B-type NiSi$_2$ films on Si(111), showing the $(1\bar{1}0)$ planes with (a) 5-fold and (b) 7-fold coordinated Ni atoms at the interface. Open circles denote Si atoms, filled circles denote Ni atoms.

Fig. 2

A schematic of an interface of a thin film, showing the principles of the x-ray standing wave method. Note that the positions of the overlayer atoms with respect to the standing wave antinodes are shifting from layer to layer. (For details see text).

Fig. 3

A schematic of the experimental set-sup, showing the monochromator and the detectors.

Fig. 4

The reflectivity and the Ni K$_\alpha$ fluorescence yield for the NiSi$_2$-Si(111) samples. The circles are the data points, the solid lines are the best fits. The data were obtained using the (111) Bragg reflection at an x-ray energy of 12.4 keV for the case of thin B-type sample (curve (b)) and 14.0 keV for the two other samples (curves (a), (c) and (d)). Curves (b), (c) and (d) are given vertical offsets of 1, 3 and 5 units respectively.

(a) Typical rocking curve of the Si(111) substrate, taken on the sample covered with A-type NiSi$_2$.

(b) B-type sample with N = 8.2. The dashed and dash-dotted lines represent the yield curves of the 5-fold and 7-fold coordinated interfaces respectively, using the distance parameter δ calculated from bulk bond lengths and $\gamma = -0.0088$.

(c) A-type sample with N = 21.1 .

(d) B-type sample with N = 36.5 . The dashed line is the yield curve when the distance parameter has its optimal value ($\delta = 1.089$), but the strain parameter is changed into the value for bulk NiSi$_2$ ($\gamma = -0.0046$).

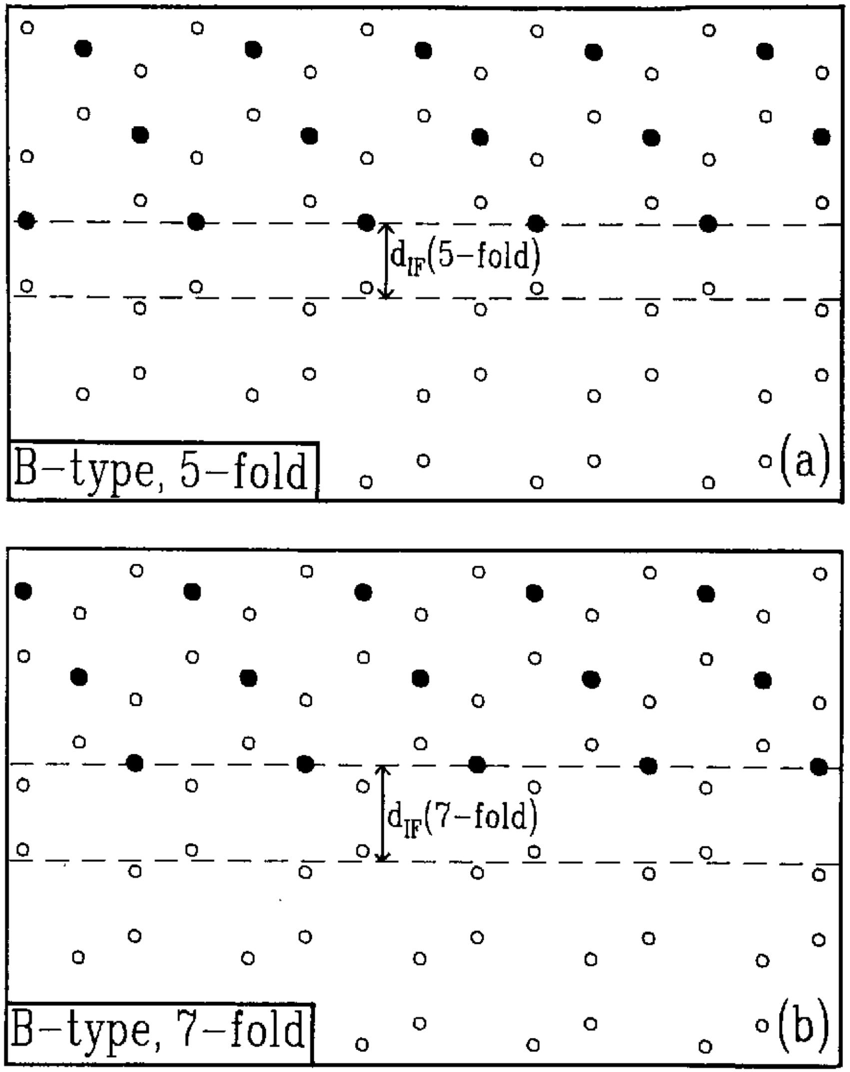
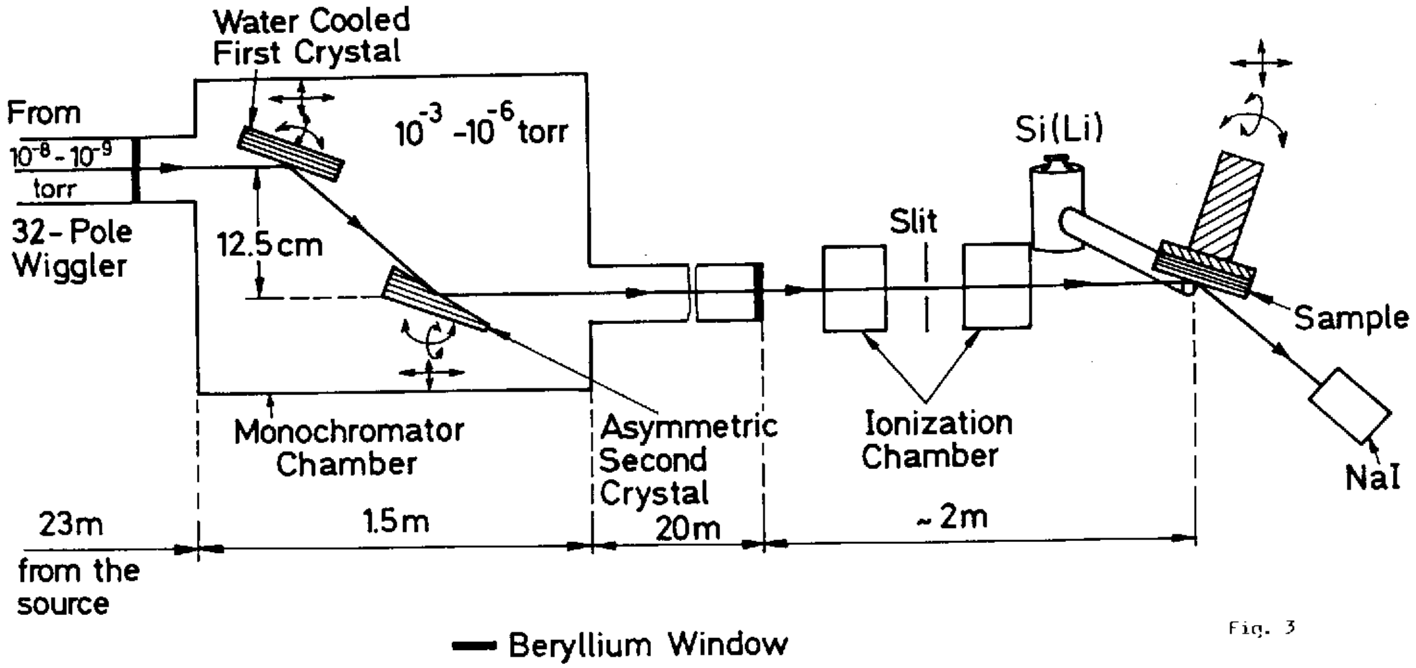


Fig. 1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From
10^{-8} - 10^{-9} torr
32-Pole Wiggler
10^{-3} - 10^{-6} torr
12.5 cm
Si(Li)
Slit
Sample
Monochromator Chamber
Asymmetric Second Crystal
Ionization Chamber
NaI
23m from the source
1.5m
20m
~2m
Beryllium Window

Fig. 3

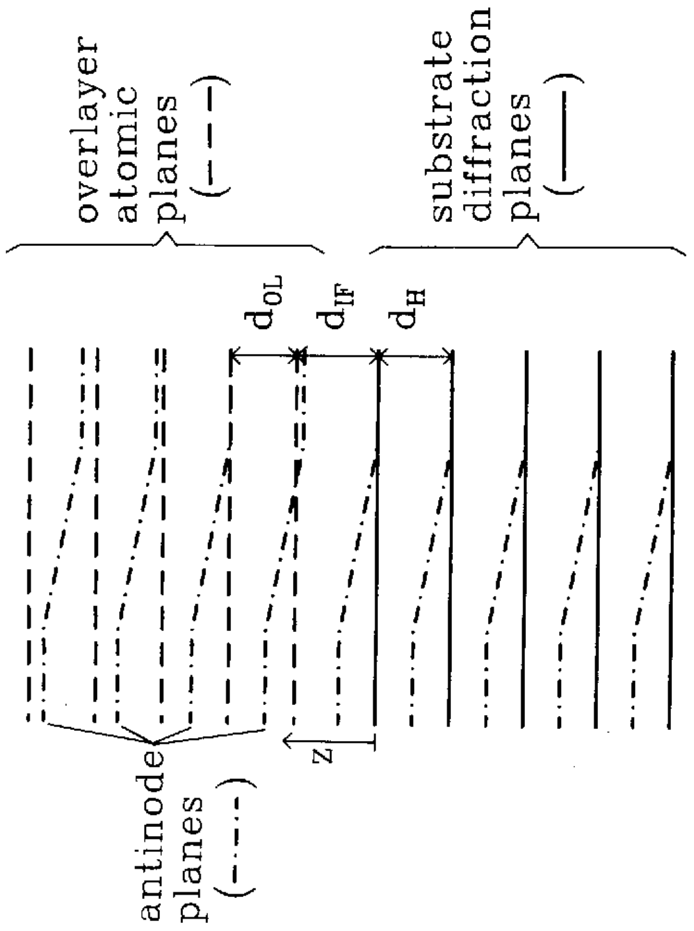
overlayer atomic planes (– – –)
substrate diffraction planes (——)
d_{OL}
d_{IF}
d_H
Z
antinode planes (–·–·–)

Fig. 2

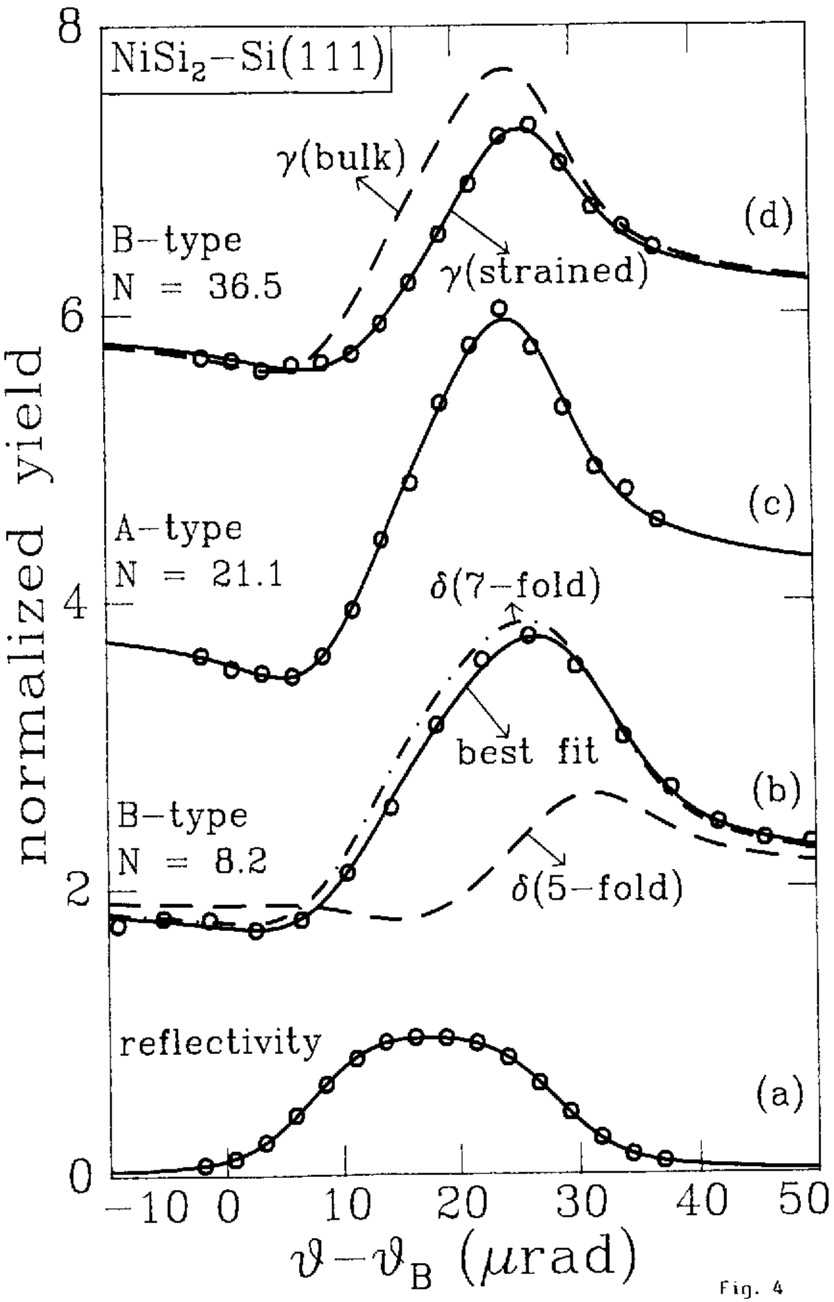
NiSi$_2$–Si(111)
normalized yield
$\vartheta-\vartheta_B$ (μrad)
8
6
4
2
0
−10
0
10
20
30
40
50
γ(bulk)
γ(strained)
B–type
N = 36.5
(d)
A–type
N = 21.1
(c)
δ(7–fold)
best fit
B–type
N = 8.2
(b)
δ(5–fold)
reflectivity
(a)

Fig. 4