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**"In-situ diffraction and imaging studies of hetero-epitaxial growth of semiconductors"**

by

**Peter A. Bennett and John A. Venables**

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## ABSTRACT

We are building a high resolution, energy filtered reflection high energy electron diffraction (RHEED) instrument for use in studying epitaxial growth, primarily in semiconductor strained layer systems, such as germanium-silicon. This instrument is now at a minimal operational stage. Secondly, we are continuing in-situ studies of reactions in ultrathin (<100 Å) films during annealing, using primarily an ultrahigh vacuum scanning electron microprobe instrument. To this end, we have developed special methods for analyzing RHEED patterns, and for numerical fitting of Auger lineshapes to determine stoichiometry.

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## SCIENTIFIC OBJECTIVES

We are developing ultrahigh vacuum in-situ diagnostic techniques for monitoring epitaxial growth, and are using these to study two classes of systems: $Ge_xSi_{1-x}$ strained layers on Si substrates, and silicide formation.

In the case of strained layers, the primary technique is high resolution reflection high energy electron diffraction (RHEED), supplemented with (dark field) imaging in reflection mode (REM), using a 15 keV scanning electron microscope (SEM). One objective is to study explicit kinetic effects in the growth of strained layers, for which the use of in-situ diagnostics is essential. We will characterize the accomodation of overlayer lattice parameter as a function of $Ge_xSi_{1-x}$ alloy composition (hence, lattice misfit), overlayer thickness, substrate temperature and deposition rate on a nominally flat substrate. Secondary kinetic and structural effects will also be studied, such as the influence of surface orientation (100) vs (111), and the influence of atomic height steps. Some fraction of this work will involve the development of quantitative methods of measuring and analyzing diffraction lineshapes ("RHEED streaks").

In the case of silicide formation, a combination of RHEED and Auger techniques will be used. Of interest is the progress of solid phase epitaxy by contact reaction for ultrathin (<100 Å) metal films on silicon substrates. We have recently observed that epitaxial constraints, which lower interface free energy, play a dominant role in the reaction kinetics. Thus, in comparison with "conventional thin film" (~1000 Å) systems, we find that reaction temperatures are dramatically lowered, phase formation sequences altered, and phases that are metastable or unstable in the bulk may be formed in thin film systems. We are making systematic measurements of phase formation as a function of film thickness and annealing temperature, to empirically characterize these reactions. It is apparent that from such phase formation diagrams, information about the fundamental reaction processes may be deduced, such as the increasing/decreasing importance of epitaxial misfit/bulk diffusion as overlayer thickness is reduced below 100 Å. These measurements have required the development of advanced techniques for analyzing RHEED patterns and Auger lineshapes.

## STATUS

A. Equipment development

The RHEED apparatus is shown in Fig. 1. At present it is operational at a minimal level using a conventional phosphor display + vidicon system. Diffraction patterns may be digitized using a micro-computer system. The vidicon system was used to measure the rocking curves in pub. #3. The deflection coils and their power supplies have been constructed, but not yet tested and characterized. The reverse viewing optical system, which was quite an elaborate UHV project, is constructed, but not yet installed. The energy filter system is designed, but not yet constructed. We will likely use the scanning system in stages as it is developed, first with vidicon only, then Grigson scanning, then energy filtered scanning. No electron spectrometer (shown on the top flange in Fig. 1) has been funded in this grant.

We recently completed design of a dual e-gun deposition system for simultaneous deposition of Si and Ge. It has not yet been built. In order to begin the semiconductor epitaxy studies, we have built simple tungsten boat evaporation/sublimation units that operate at close working distance. These will suffice for initial studies at low coverages and low rates.

We are working to enhance the SEM system, primarily to achieve single atomic step imaging capabilities. This involves improving the mechanical stability, magnetic shielding and field cancellation, and adding a diffraction aperture system. Work on each of these items has been initiated.

B. Results

On the semiconductor epitaxy project, we have developed simple evaporators that can deliver several Å/minute of either Si or Ge with a carbon contamination level of ~ 0.2% as measured by Auger. We have grown Ge islands on a Si(100) substate, and are doing exploratory measurements of island size (determined from diffraction broadening) as a function of preparation conditions, such as substrate temperature and deposition rate.

On the silicides project, we have developed several innovative techniques and used them to study in detail the Ni/Si(111) thin film system. In order to minimize systematic experimental uncertainties (espec. film thickness and annealing temperature), we have developed a masked deposition technique whereby a well defined overlayer thickness profile is generated, and annealed in a single experimental run. This requires the microanalysis/imaging capability of the SEM/SAM instrument, and is illustrated in Fig. 1 (pub. 1). Early results have been presented as a "kinetic phase formation diagram" (Fig. 3, pub. 1) showing the ordered phases that appear as a function of deposited thickness and annealing temperature in the range of 0-25 Å and 50-700 C, respectively. Structure identification using the RHEED patterns required development and/or refinement of methods using Kikuchi patterns and "rocking curves", as described in pubs. 2 and 3. In particular, we have shown that depth information may be derived from a surface diffraction method (RHEED) without using complicated and "unproven" dynamical calculations. Thus, we have shown that Kikuchi patterns provide bulk (vs. surface) symmetry information from the diffraction geometry (vs. intensities), and that crude bulk crystallographic information may be derived from rocking curves using a modified kinematic treatment (pub. 3). More recently we have developed numerical Auger lineshape analysis techniques that complement diffraction methods by providing information about disordered phases. An example of the spectral decomposition of the Si LVV line is shown in Fig. 2 (attached). Fig. 3 (attached) shows the evolution of the silicide compounds during thermal annealing. This work demonstrates that accurate quantitative Auger analysis for a sample with multiple stoichiometries is possible provided that a stable numerical fitting is used, and reliable "fingerprint" spectra for the single phase compounds are available. This work is described in presentations 3, 4, 6 and 7.

## IMMEDIATE PLANS

An immediate goal is to image single atomic steps on Si(111) using the SEM in dark field image mode. We will then look at the facetting transition, whereby the vicinal surface (with regular spaced single atom steps) separates into wide terraces separated by steep facets in a reversible phase transition [R. J. Phaneuf and E. D. Williams, PRL58 (1987) p. 2563]. Secondly, we will study Si homoepitaxy with imaging, to better understand the transition from terrace nucleated growth (leading to RHEED oscillations) to step flow growth (steady RHEED intensity). Thirdly, we are working to prepare UHV cleaned "curved" (lenticular) Si(100) surfaces. For this surface, single atom steps with regular terrace spacing are thermodynamically stable, and we hope to use this substrate to make systematic studies of the effect of surface steps on epitaxial growth, both for Si overlayers (homoepitaxy) and eventually, for strained overlayers of $Ge_xSi_{1-x}$ alloys.

In the silicide system, we will study the effect of surface reconstruction on the phase formation diagram by growing on a sputtered Si(111) surface, and comparing to results for growth on the flat, annealed surface.

Lastly, we are continuing collaborative efforts to prepare UHV cleaned silicon surfaces in the TEM and STEM atomic resolution electron microscopes, operated in the high resolution electron microscope facility at Arizona State University. These microscopes have been operating here since about June 1988. Present difficulties (for silicon studies) involve specimen fragility and vacuum conditioning (outgassing), which will soon be under control.

## PERSONNEL

1. A. P. Johnson, graduate research assistant; MS thesis "A Surface Phase Diagram for Thin Nickel Films on Silicon(111)", awarded Aug. 1987.
2. J. R. Butler, graduate research assistant; PhD thesis area: thin film silicides.
3. X. Tong, graduate research assistant; PhD thesis area: semic. epitaxy.
4. M. R. Wood, undergraduate hourly; technical help (mechanical).
5. R. Tatro, undergraduate hourly; technical help (computers).

## PUBLICATIONS

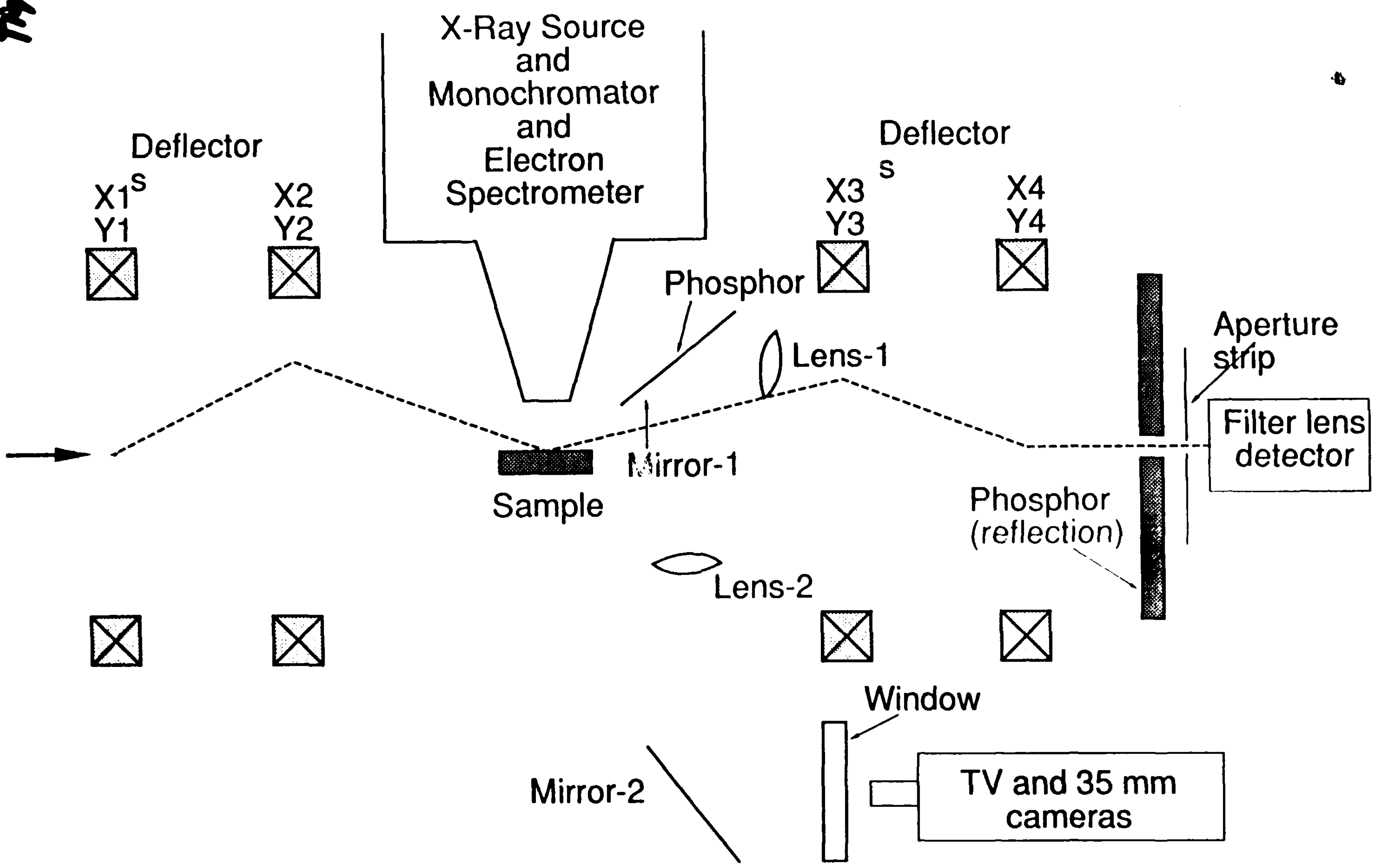
1. "A Phase Formation Diagram for Precursors to Epitaxial Growth of $NiSi_2$ on Si(111)," P.A. Bennett, A.P. Johnson and B.N. Halawith, Phys. Rev. B37 (1988), p. 4268.
2. "Thin Film Crystallography Using RHEED 'rod intensity profiles': Ni/Si(111)," P.A. Bennett, X. Tong and J.R. Butler, Jour. Vac. Sci. Tech. B6 (1988) p. 1336.
3. "Ultrathin film growth of silicides studied using microprobe RHEED and Auger" P. A. Bennett, J. R. Butler and X. Tong, Jour. Vac. Sci. Tech. (submitted).

## PRESENTATIONS

1. "RHEED studies of thin film formation with a UHV-SEM" P. A. Bennett and A. P. Johnson, Wickenburg workshop, Wickenburg, AZ Jan 11, 1988.
2. "Precursor Structures in the Formation of Epitaxial $NiSi_2$/Si(111)" P. A. Bennett, J. R. Butler and X. Tong , PCSI-15, Asilomar, CA Feb. 1, 1988.
3. "Thin film growth studies using microprobe RHEED and Auger" P. A. Bennett, seminar presented at IBM, Yorktown heights, June 1988.
4. "Thin film growth studies using microprobe RHEED and Auger" P. A. Bennett, J. R. Butler and X. Tong, 35th Amer. Vac. Soc. mtg. , Atlanta, GA, Oct. 3, 1988 (invited).
5. "Structure identification in thin films using RHEED" P. A. Bennett, J. R. Butler and X. Tong, Wickenburg workshop, Wickenburg, AZ Jan 4, 1989.

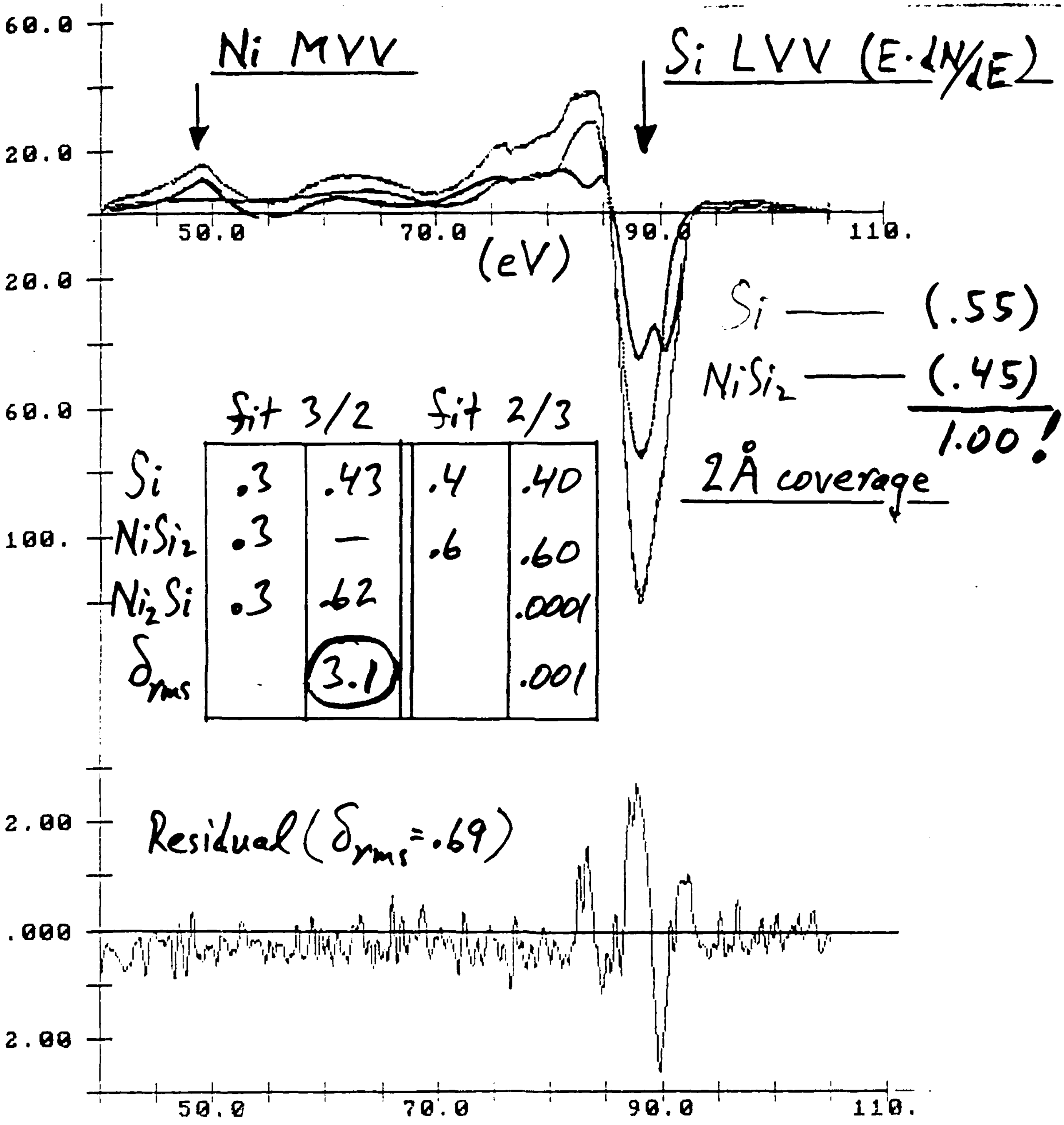
6. "Compound formation in the Ni/Si(111) ultrathin film system studied with numerical Auger lineshape analysis" J. R. Butler, X. Tong and P. A. Bennett, March APS mtg. 1989.
7. "A phase formation diagram for ultrathin film growth of nickel silicides" P. A. Bennett, J. R. Butler and X. Tong, Spring Mat. Res. Soc. mtg. April 24, 1989 (**invited**).

Fig. 1



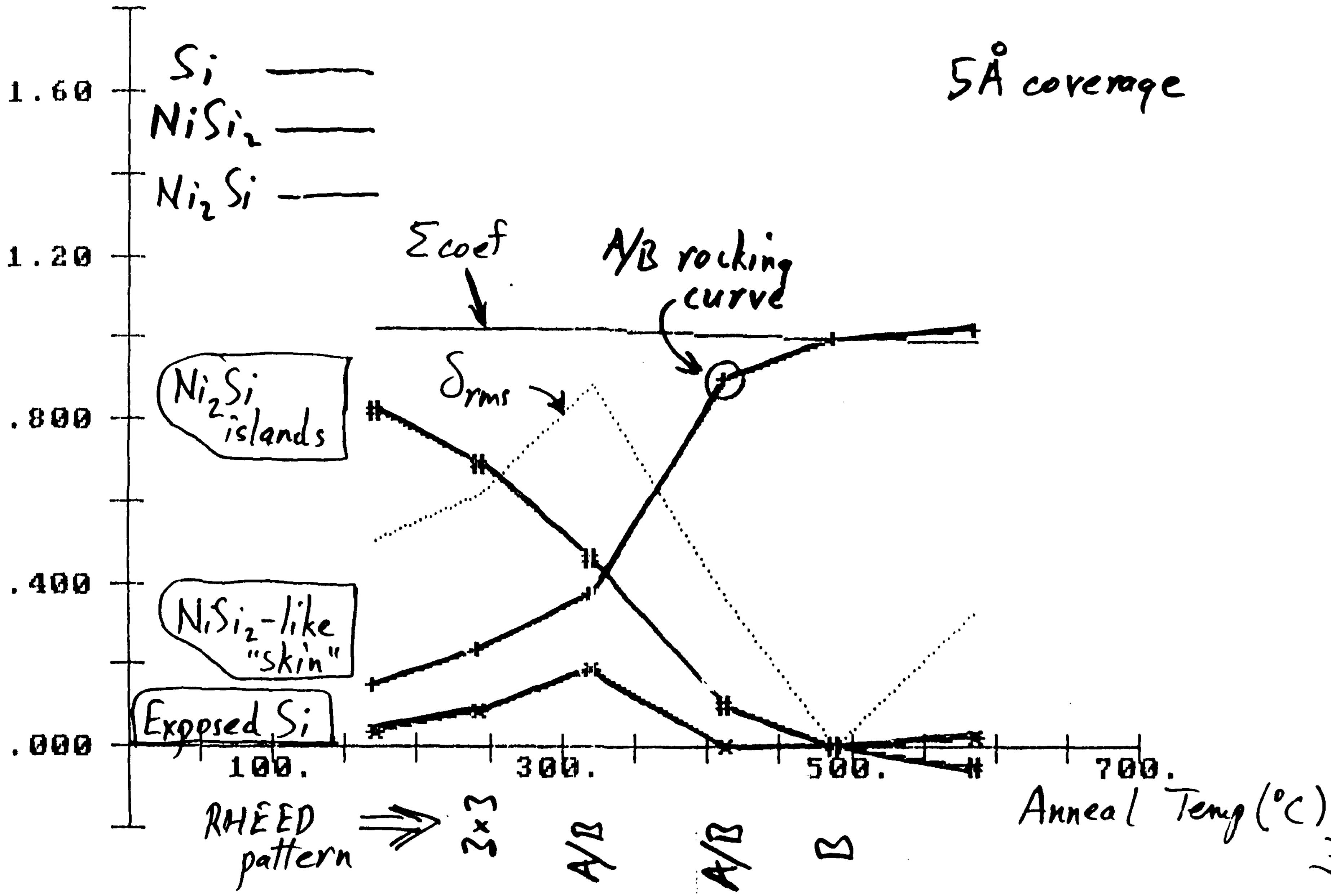
X-Ray Source and Monochromator and Electron Spectrometer
Deflectors
X1 Y1
X2 Y2
Deflectors
X3 Y3
X4 Y4
Phosphor
Lens-1
Aperture strip
Filter lens detector
Mirror-1
Sample
Phosphor (reflection)
Lens-2
Window
Mirror-2
TV and 35 mm cameras

Fig.




| | fit 3/2 | | fit 2/3 | |
|---|---|---|---|---|
| Si | .3 | .43 | .4 | .40 |
| $NiSi_2$ | .3 | — | .6 | .60 |
| $Ni_2Si$ | .3 | .62 | | .0001 |
| $\delta_{rms}$ | | (3.1) | | .001 |

# Fig. 3



5Å coverage
Si
NiSi2
Ni2Si
1.60
1.20
.800
.400
.000
100.
300.
500.
700.
Anneal Temp (°C)
Σcoef
A/B rocking curve
δrms
Ni2Si islands
NiSi2-like "skin"
Exposed Si
RHEED pattern
3×3
A/B
A/B
B


Fig 7