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Project Title: EXPLORATORY STUDY ON MICROANALYSIS OF THIN FILMS BY BACKSCATTERING TECHNIQUES

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OVERALL OBJECTIVES

1. To use backscattering spectrometry in the 1-2 MeV range for the determination of the composition and the detection of interdiffusion in thin film structures of interest to photovoltaic conversion.

2. To investigate the solid-phase epitaxial growth (SPEG) of Si layers at low temperatures obtained by interdiffusion processes.

3. To investigate backscattering spectrometry in the sub-MeV range and to assess its possibilities and problems.

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PLANNED ACTIVITY FOR LAST SIX MONTHS

1. Investigate electrical properties of the SPEG layer.

2. Investigate the possibility of SPEG with metal films other than Pd as transport medium.

3. Investigate BS below 1 MeV

EXPLORATORY STUDY ON MICROANALYSIS OF THIN FILMS BY BACKSCATTERING TECHNIQUES

by

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ABSTRACT

I. SPEG

To be potentially useful, SPEG layers must have controllable electrical characteristics, particularly with regards to conductivity type and resistivity value. Efforts were therefore undertaken to establish that such a control is possible.

We added one processing step to the standard sample fabrication procedure by vacuum-depositing a thin layer (nominally 5Å) of Sb before the deposition of the amorphous Si layer, as shown in diagram labelled Caltech 4 (C4). Anneal steps remained unchanged. The resulting SPEG layer was analyzed in four different ways: (a) Auger Electron Spectroscopy (see C5), performed after the uppermost Pd_xSi_y layer was removed, confirmed that Sb was present within the SPEG layer of Si. (b) The I(V) characteristics of the SPEG layer against the Si substrate (<100>, 1-10 Ωcm p-type) was rectifying, and the layer itself afforded good electrical conductivity and easy ohmic contacting with W wires via the Pd_xSi_y layer (see C6). (c) A scanning microprobe picture of a cleaved sampled taken in the electron-beam-induced current mode established the presence of a depletion region more than 1 μ below the surface (see C7 and C8). (d) Hall effect data indicated that the SPEG layer was n-type, had an average free carrier concentration of about 10^{19} cm^{-3} and an average electron mobility $\bar{\mu}_n$ of about 40 cm^2/Vs (see C9). These results apply to a typical sample obtained with initial layers of about 1000Å Pd, 5 Å Sb and 1 μ of amorphous Si annealed in vacuum, first at 280° for 30 min and then under slowly increasing temperatures (≳ 0.2 °C/min) up to 525 °C. The evidence that the layer is n type and well doped is convincing. We plan to determine the properties of the doped SPEG

layers more accurately, and to try other methods of introducing the dopant species of various kinds. The main problems are inadequate process control and marginal sensitivity of analytical tools.

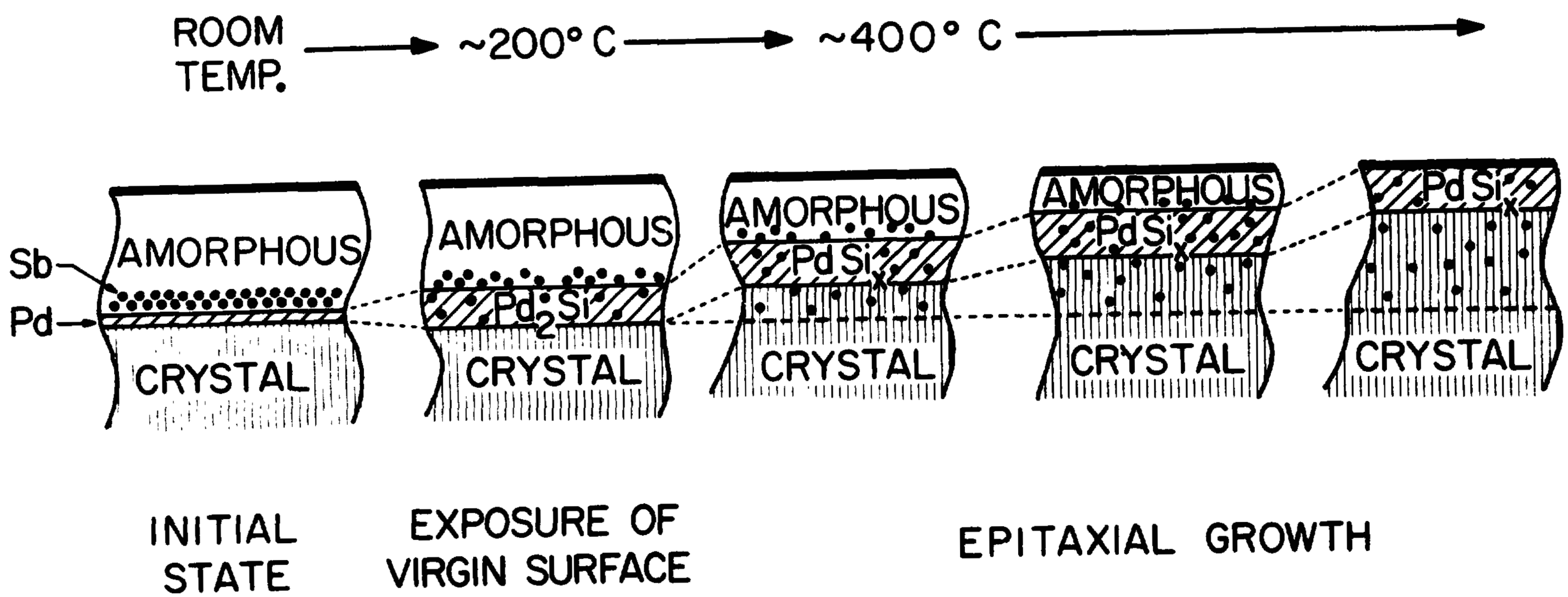
SPEG with Pd_2Si as transport layer has two stages (see C10): (a) Initial fast (~10 Å/s at 500°C) transient regime. The growth starts with islands which ultimately join to form a layer (C11). The final thickness is equal to that of the initial Pd_2Si layer (C12). (b) Steady state regime. The growth is much slower (~ 1 Å/s), linear, and independent of the thickness of the transport medium (C13). The rate is thus not diffusion-limited.

SPEG with Ni silicide in lieu of Pd silicide as transport medium has been demonstrated by backscattering spectrometry (C14). As a whole, Ni SPEG is quite similar to that of Pd SPEG (C15). The transient is less distinct, because slower, and in the steady state regime, the boundaries of the transport medium lose their definition as the growth proceeds, which indicates poorer quality of the grown layer. Reactions require temperatures which are typically 90°C above the corresponding temperatures of Pd SPEG.

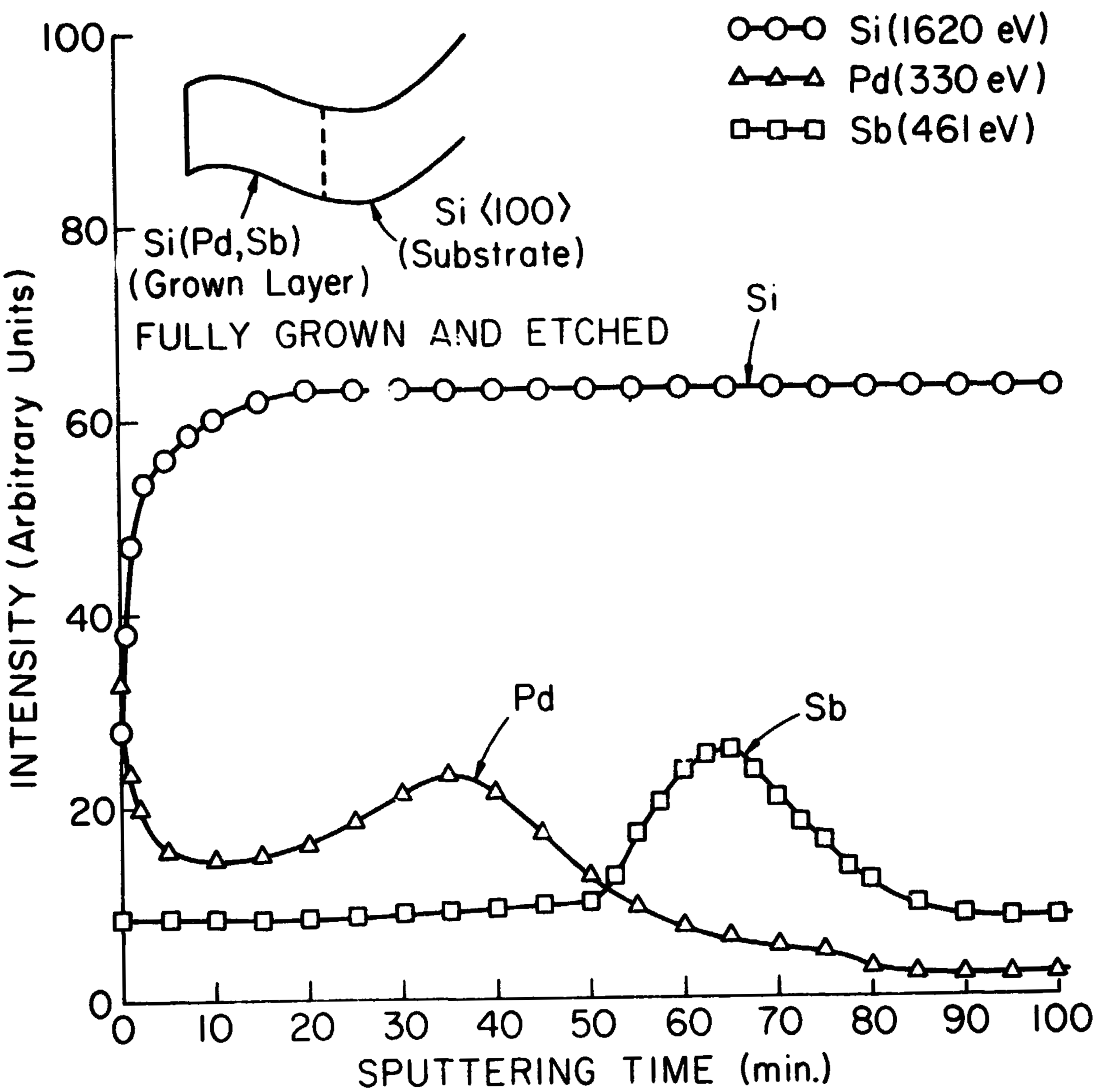
II. BACKSCATTERING SPECTROMETRY (BS)

To develope BS at energies below 1 MeV, depth calibration must be provided first by absolute measurements of stopping cross sections. Typical past errors of ± 10% are undesirably large. We have compared various methods of data evaluation and their errors with computer programs tested against real ^4He spectra. All methods can attain a few %, but values derived from films of known mass per unit area are generally most reliable. Main sources of errors are caused by detector noise and background signals. Absolute measurements are planned next.

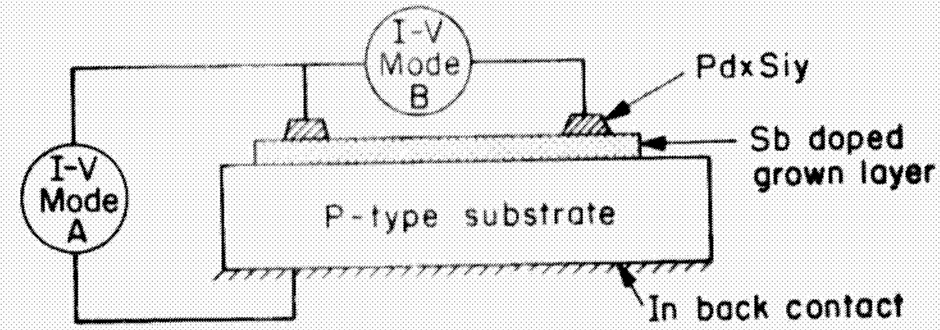
Summaries of main results, major problems, planned activity and renewal proposal are given in C16 to C19.

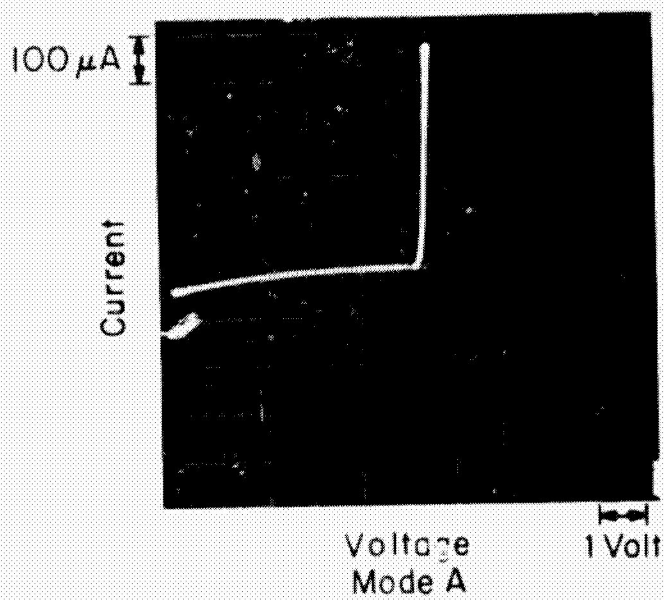

ROOM TEMP.
~200° C
~400° C
Sb
Pd
AMORPHOUS
CRYSTAL
Pd2Si
PdSix
INITIAL STATE
EXPOSURE OF VIRGIN SURFACE
EPITAXIAL GROWTH

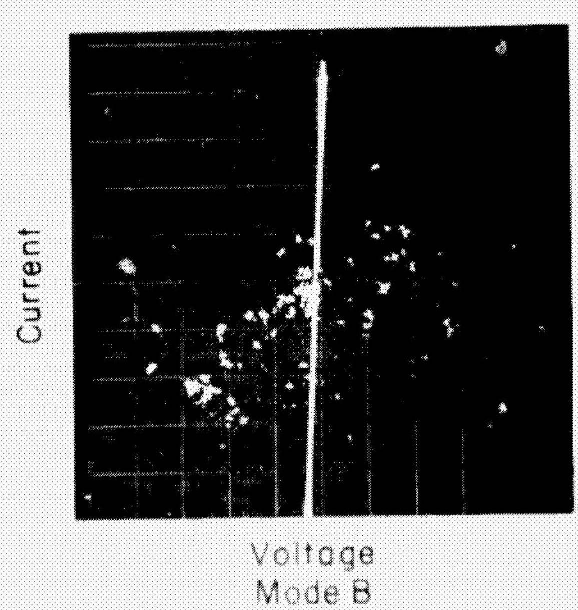
SPEG

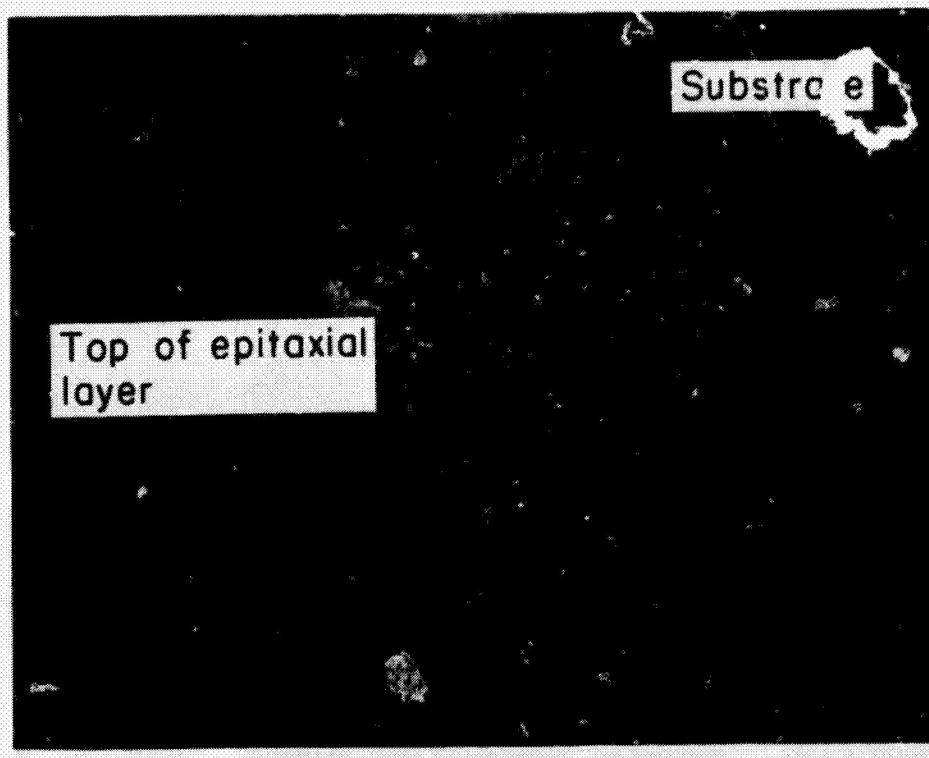


Si(1620 eV)
Pd(330 eV)
Sb(461 eV)
Si(Pd,Sb)
(Grown Layer)
Si ⟨100⟩
(Substrate)
FULLY GROWN AND ETCHED
Si
Pd
Sb
INTENSITY (Arbitrary Units)
SPUTTERING TIME (min.)
0
10
20
30
40
50
60
70
80
90
100


I-V
Mode
B
PdxSiy
Sb doped
grown layer
I-V
Mode
A
P-type substrate
In back contact

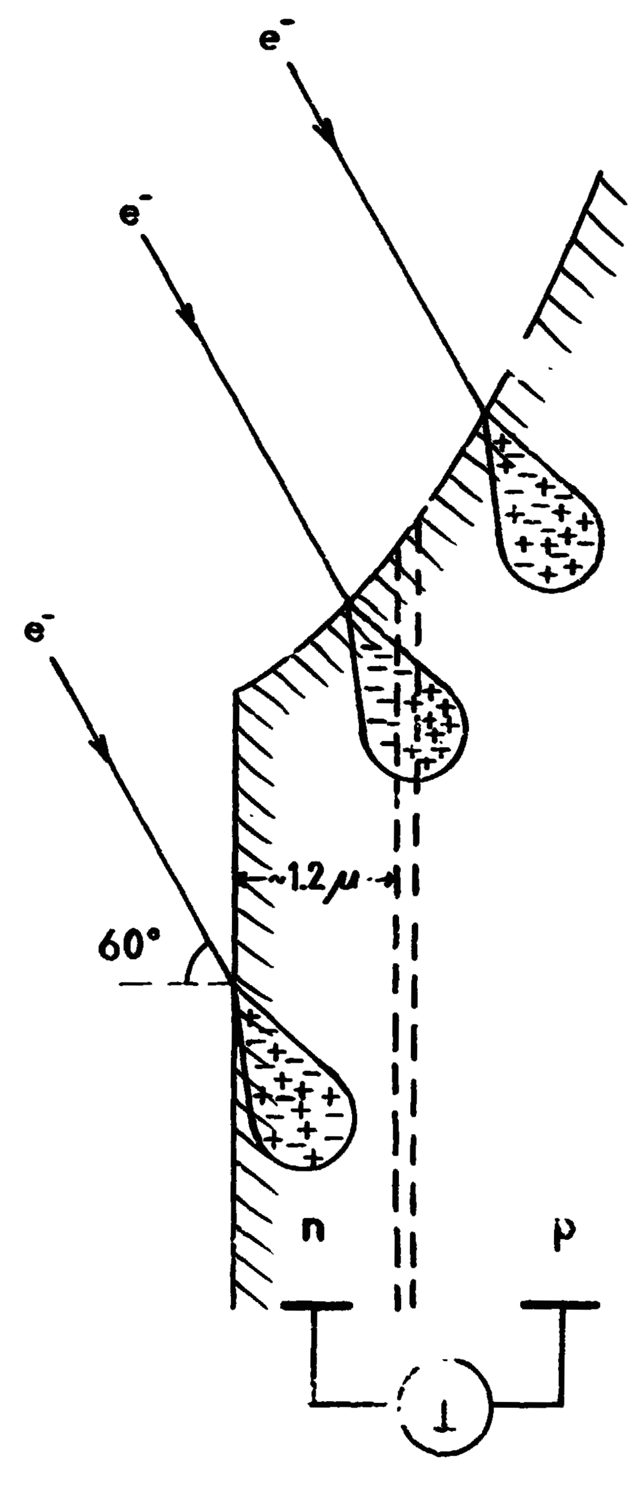

100 μA
Current
Voltage
Mode A
1 Volt


Current
Voltage
Mode B



2nd electron Mode   20μ

EBIC Mode



EBIC

HALL EFFECT

Data obtained at room temperature with van der Pauw patterns. Values are averages over the whole thickness (~ 1 μm) of the SPEG layer.

	SPEG Layer	Comparison with bulk single crystal	
Conductivity type	n	n	
Mobility μ_n	~ 40-50	140	cm^2/Vs
Carrier Concentration	10^{19}	10^{19}	cm^{-3}
Resistivity	~ .02	. J04	Ωcm

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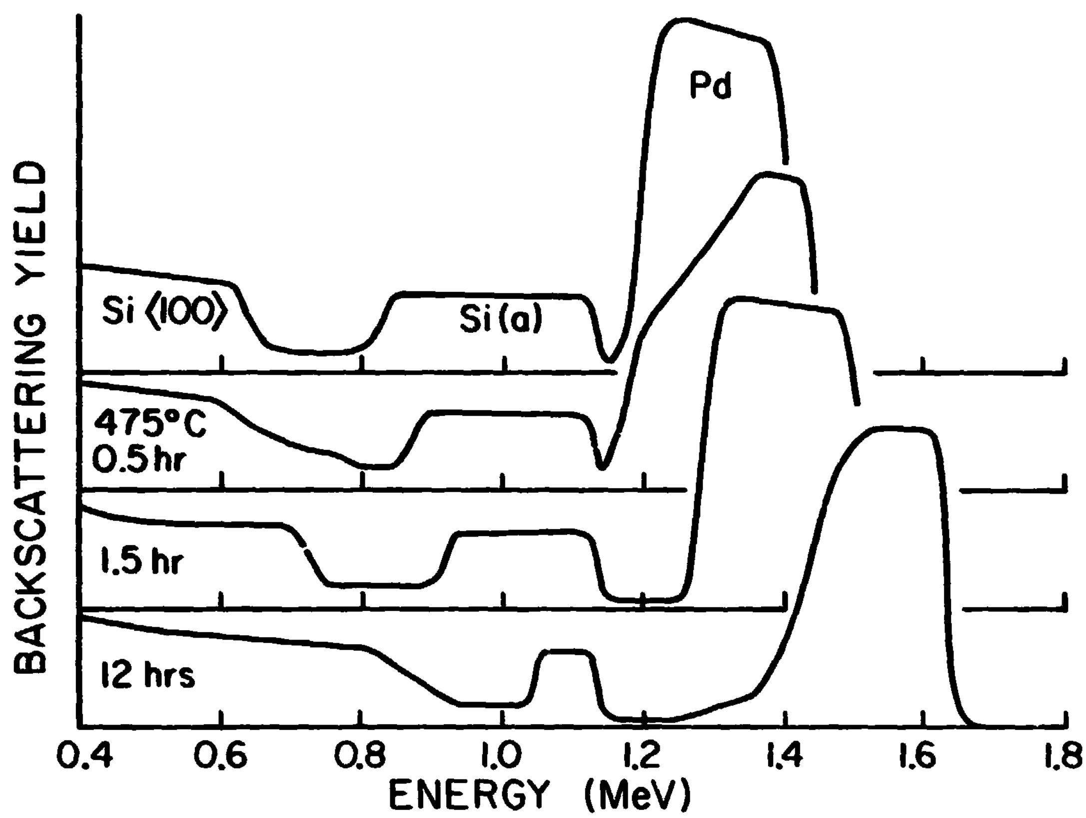
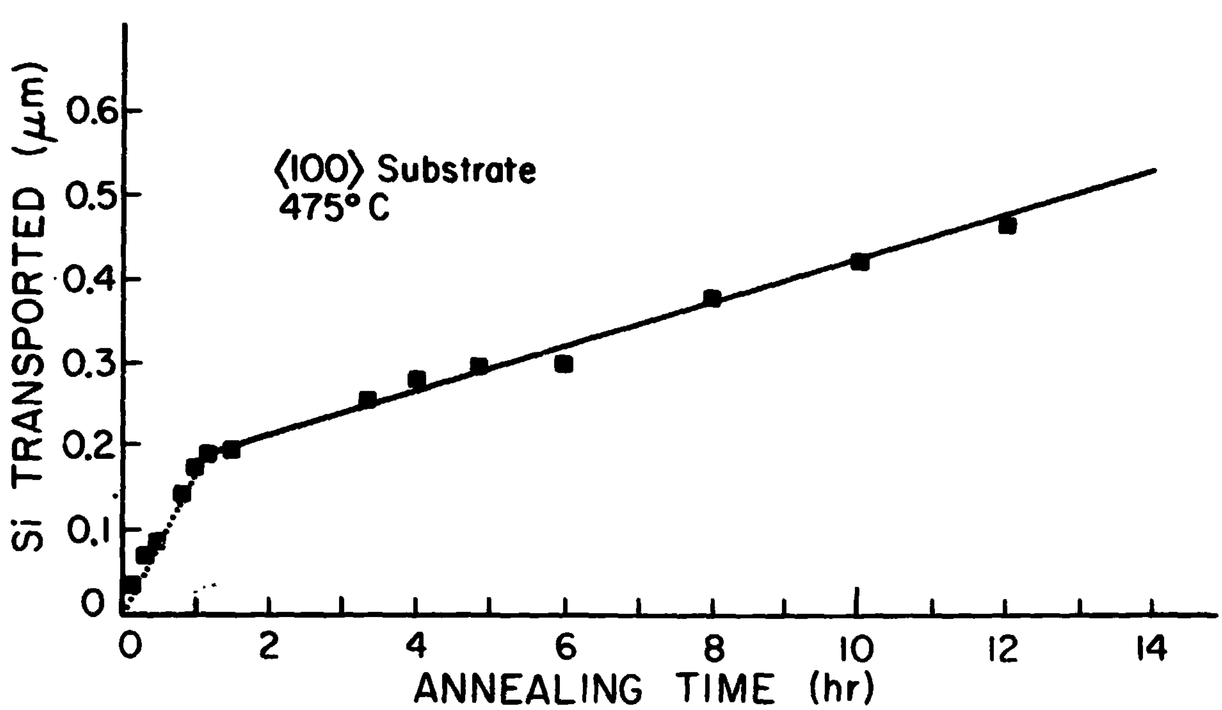
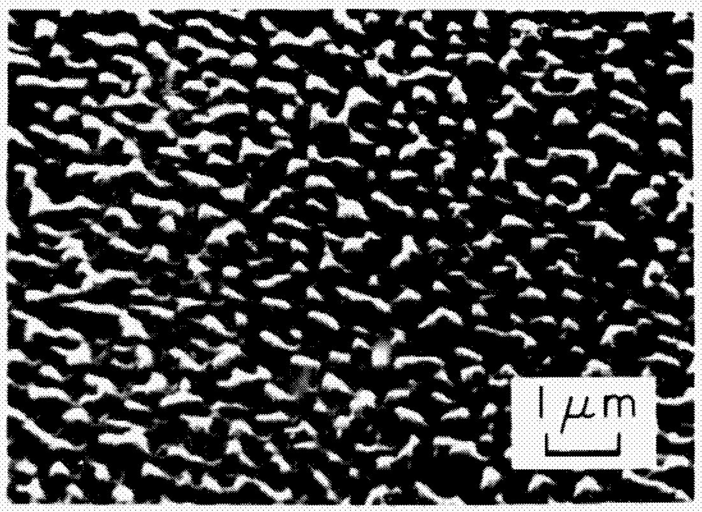
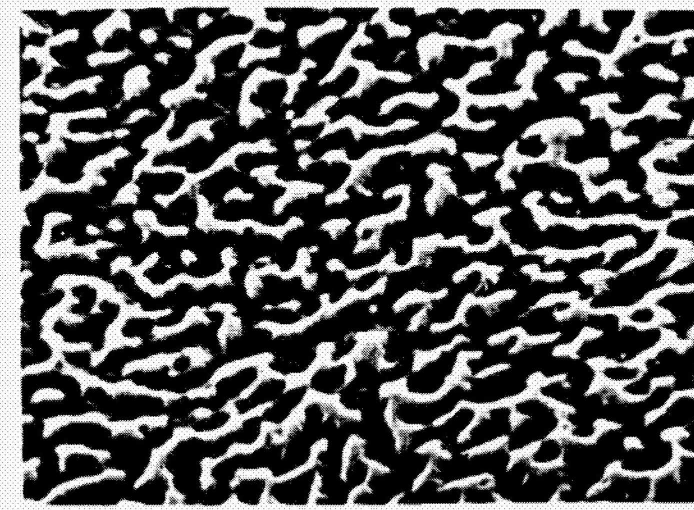

Pd
Si ⟨100⟩
Si (a)
475°C
0.5 hr
1.5 hr
12 hrs
BACKSCATTERING YIELD
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
ENERGY (MeV)

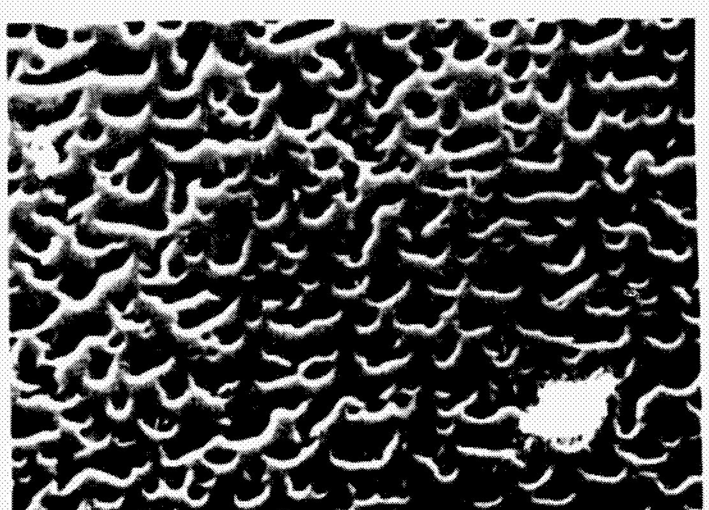

⟨100⟩ Substrate
475° C
Si TRANSPORTED (μm)
0
0.1
0.2
0.3
0.4
0.5
0.6
0
2
4
6
8
10
12
14
ANNEALING TIME (hr)



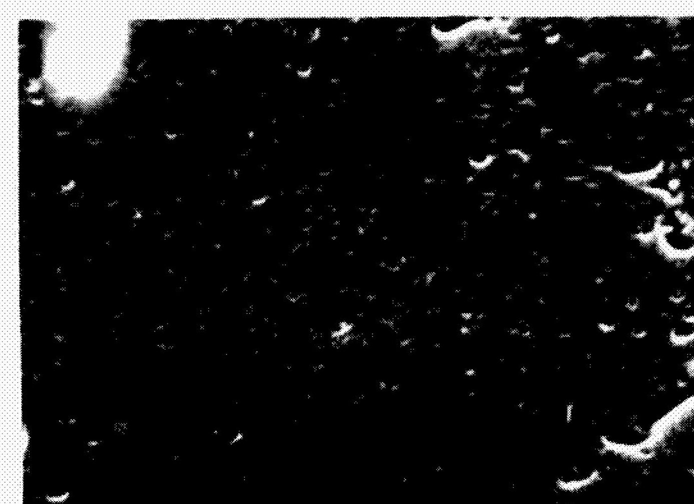
475°C 30 min



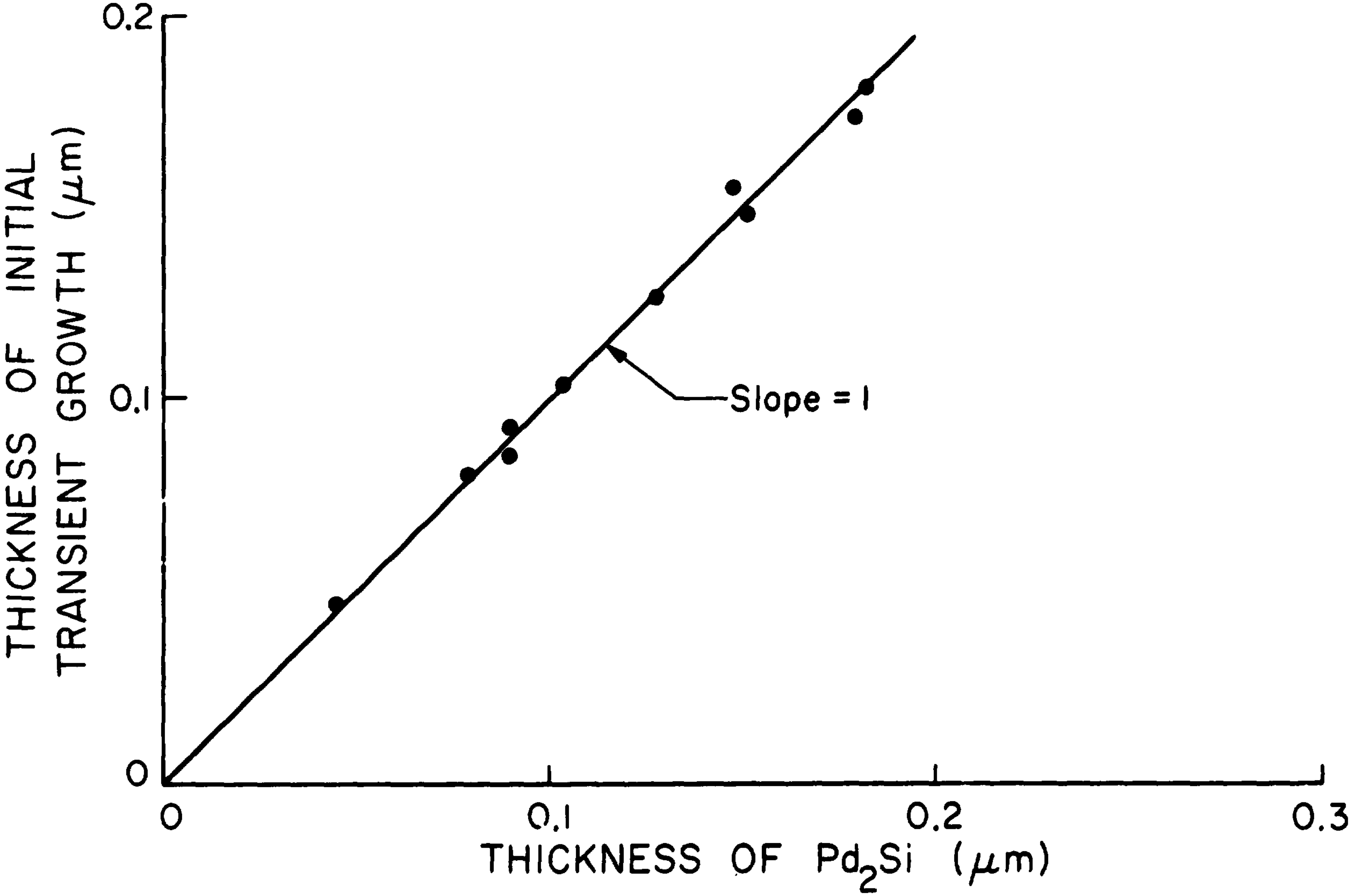
50 min

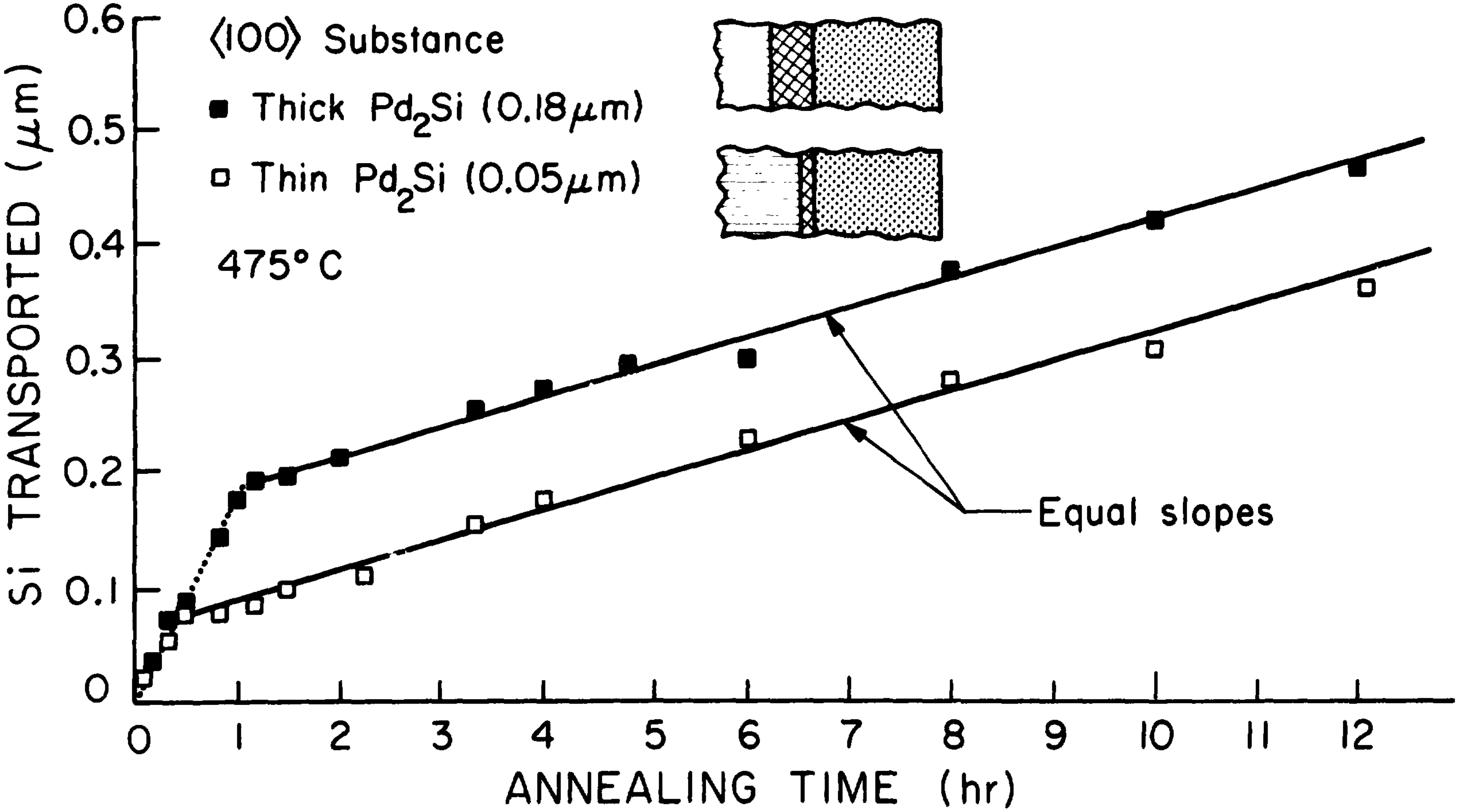


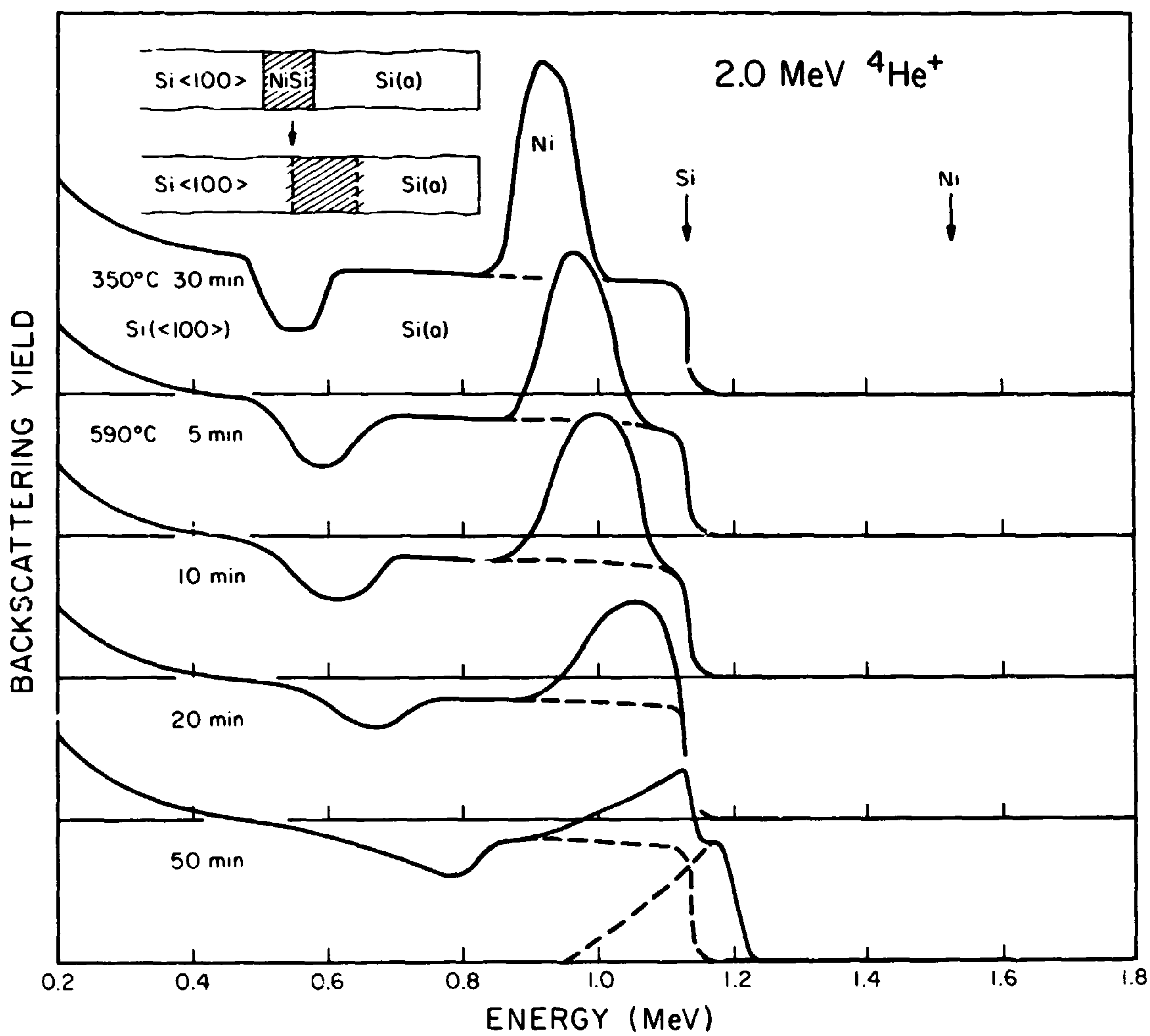
70 min



90 min


THICKNESS OF INITIAL TRANSIENT GROWTH (μm)
0.2
0.1
0
0
0.1
0.2
0.3
Slope = 1
THICKNESS OF Pd2Si (μm)


⟨100⟩ Substance
Thick Pd_2Si (0.18μm)
Thin Pd_2Si (0.05μm)
475° C
Equal slopes
Si TRANSPORTED (μm)
0
0.1
0.2
0.3
0.4
0.5
0.6
ANNEALING TIME (hr)
0
1
2
3
4
5
6
7
8
9
10
11
12



2.0 MeV $^4He^+$
Si<100>
NiSi
Si(a)
Si<100>
Si(a)
Ni
Si
Ni
350°C 30 min
Si(<100>)
Si(a)
590°C 5 min
10 min
20 min
50 min
BACKSCATTERING YIELD
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
ENERGY (MeV)

COMPARISON OF SPEG WITH Pd AND Ni

	Pd	Ni	
SAMPLE			
Substrate	<100>	<100>	
Metal Film	~1000	~1000	Å
Si(a) Film	~1	~1	μm
FORMATION OF TRANSPORT MEDIUM			
Typical Temperature	280	350	°C
Typical Time	30	30	min
Composition by BS	$\frac{Pd}{Si} = \frac{2}{1}$	$\frac{Ni}{Si} = \frac{1}{1}$	
by X-rays	Pd_2Si	NiSi	
TRANSPORT OF Si			
Typical Temperature	~500	~590	°C
Transient Rate	~10	~2	Å/s
Form	island	no island	[illegible])
Steady State Rate	~1	~1	Å/s
Structure	epitaxial	epitaxial	(poor)
Apparent Activation Energy	~4	~4	eV

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SUMMARY OF KEY RESULTS

1. Doping of SPEG layers is possible;proof obtained with Sb and established via:
   - I(V) characteristics
   - Hall effect measurements
   - EBIC μ-probe pictures
   - AES
2. SPEG with Pd_2Si has two stages:

   Initial transient regime
   - 10 A°/s rate
   - island growth
   - thickness = that of Pd_2Si layer

   Steady State regime
   - 1 A°/s rate, independent of Pd_xSi_y layer thickness
   - continuous film growth

3. SPEG with NiSi also works; generally similar to SPEG with Pd_2Si:
   - Corresponding process temperatures are typically ~ 90°C higher for Ni
   - Layer quality presently inferior to obtained with Pd_2Si

4. Identified best methods for stopping power measurements below 1 MeV from backscattering spectra.

MAJOR PROBLEMS

SPEG

Control of fabrication processes

- cleanliness of interfaces
- purity of deposited layers
- amount of doping
- annealing ambient

Characterization of final product

- limitations of analytical tools

BS

Limitations in system

- detector noise
- preamplifier noise

Limitations in target

- purity
- thickness } density
- mass/cm^2 } density

PLANNED ACTIVITY FOR NEXT SIX MONTHS

1. Characterization of doped SPEG layers.

2. Doping of SPEG layers by other dopants or other incorporation methods.

3. Compare SPEG with regrowth of amorphous Si layers on single crystal Si substrates.

4. Obtain stopping cross sections in 0.3 - 1.0 MeV range.

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PLANNED RENEWAL REQUEST

Major Goals

Investigate SPEG

Assess practical potential of SPEG to solar cell, and to semiconductor devices generally

Liberate SPEG from single crystal substrate

Develop sub-MeV backscattering spectrometry

Estimated Dates and Costs

August, 1976 to August, 1978 (24 Months) $150,000.00

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