IBM Progress Report 1979

- I was a busy beaver, it seems. The report was delivered orally, that's why the pictures have not captions. Real high quality pictures were passed around, the written version was just a kind of memory prop.
  - I had to scan this report in a picture format (jpeg) so I could process the result and make the partially badly faded pages readable. I added a few comments where sensible.

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Program Review

H. Foell

Research Activities in 1979 were focused on the following items:

i) Weak-beam contrast of stacking faults in TEM (together with C.B. Carter (Cornell) and M. Wilkens (Max-Planck-Institut, Stuttgart). Earlier work was concluded and published.

ii) Design and assembly of a large-area thinning machine for Si. Large areas (~1cm^2) of very thin Si sheets (5μm-20μm) are required for SAM studies of grain boundary diffusion in Si. First runs with single crystalline Si have been successful, thinning of poly-Si is in progress.

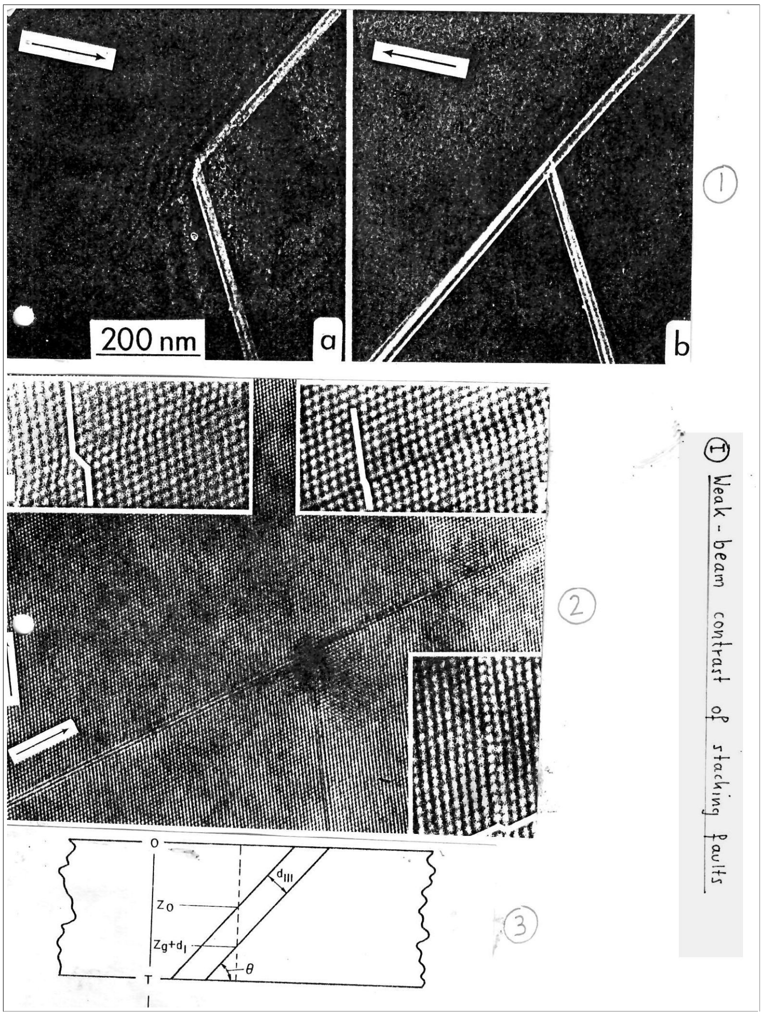
iii) Anodic etching of defects in p-type Si. It was found that p-type Si can be etched preferentially at defects in an electro-chemical cell with diluted HF as electrolyte. Depending on the applied voltage only electrically active defects or all defects can be etched. This etching method can partially replace EBIC and chemical etching, and provides a powerful technique for pinpointing electrically active defects in the TEM.

iv) Corrosion of Pd_2Si "Bubbling" of Pd_2Si contacts was found to occur in the Si-line environment about 2 weeks after the contacts have been made. The reasons for this lift-off of the silicide were unclear and it was conceived as a serious problem with respect to future device application. It could be shown that the bubbling was due to an electrochemical reaction between the Si and the silicide in exposed areas (edge of the contact-pinholes) with HF (present as contaminant in the atmosphere) acting as electrolyte. "Bubbling" thus is a corrosion phenomena, possibly assisted by specific interface properties.

v) High resolution TEM of defects in ion-implanted Si (together with T.Y. Tan) Ion implantation usually produces an amorphous layer and crystal lattice defects below the amorphous-crystalline interface. Knowledge about these defects is very limited, but very unusual configurations had been identified, e.g. planar defects on {113} planes. Lattice imaging helped to further the understanding of implantation-produced defects and lead to considerable progress in modelling defect properties.

vi) TEM of Si-silicide interfaces in cross-section (together with P. Ho and K.N. Tu). Several Si-silicide systems have been studied in cross-section (some of them at high resolution) after a method for preparing cross-sectional specimens had been developed. Impo .ervations were: a) The Si-Pd_2Si interface is rather smooth if .. silicide grew epitaxially and slightly rougher if it grew non-epitaxially; the relative smoothness S (= average thickness of silicide divided by roughness amplitude) was found to be ~20 or ~10, resp. The Pd_2Si surface can be very rough (S ~ 3) Lattice imaging shows the absence of a glassy interface layer, the presence of misfit-like dislocations and no facetting of the interface. b) The Si-$NiSi_2$ interface is very rough and heavily facetted for {100} Si (S ~ 2) and has a relatively smooth surface which is also facetted. The silicide is perfectly epitaxial on {100} Si whereas it growth in a twin relationship to the matrix on {111} Si. It is still facetted in this case, but much smoother (S ~ 20) than on {100} Si. Within a facet the interfaces are atomically flat for both {100} and {111} substrate orientations as shown by direct lattice imaging. Misfit dislocation networks of edge dislocations with b = a/2<110> or b = a/6<112> were found in the direct epitaxial case or in the twin case, respectively. c) The Si-PtSi interface is extremely rough (S ~ 3) whereas the surface is very smooth (S ~ 20); in marked contrast to Pd_2Si. Silicides currently investigated are Ni_2Si, NiSi, Pd_2Si formed at different temperatures and the Al-Ti-Pd_2Si system.

They allowed 2 pages.
I presented 6 topics. No 4 was actually confidential. The big topic was the last one - silicides.

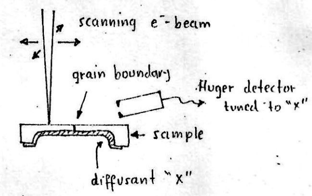


We know this topic.
It was presented under . 3.1 TEM Work at Cornell University /
[3.4 Weak Beam Contrast of Stacking Faults in TEM]
While the TEM work was done at Cornell, writing it all up (and come up with the theory) took time and the paper was send out in Jna 1980, one year after me joining IBM

(II) Large area thinning of Si

Goal: Homogenous thinning of Si wafers (ϕ = 2¼" ···· ¼") both single - and poly - crystalline to final thicknesses of 40 µm ···· 2 µm

Application: a) intended: Internal friction measurements (B. Berry). Grain - boundary diffusion in Si (P. Ho)



A thin layer of the diffusant "x" is evaporated on the sample back-side. The appearance of "x", upon heating the sample, on the frontside is watched by SAM.

b) possible: Preparation of samples with a large area transparent to the electron beam in TEM for studying special defects.

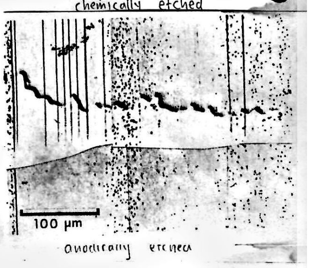
Achievement A large area thinning apparatus has been designed and built. First runs were successful.

I actually did built the damned thing, and it worked!.
It just was never used by the people it was intended for. They were afraid of the rather dangerous chemistry involved.

(III) Anodic etching of defects in p-type Si

Basics: Anodical dissolution of Si requires holes. Therefore preferential etching of defects in n-type Si is possible if the defects can act as generation centers for holes. Preferential etching in p-type Si was thought to be impossible

Experimental: Preferential etching of defects in p-type Si is possible at low voltages (≤ 5 V). The etching behaviour depends on the applied voltage: At very low voltages (~ 0.3 - 0.5 V) only electrically active defects (as defined by EBIC) are etched; at higher voltages all defects are etched.



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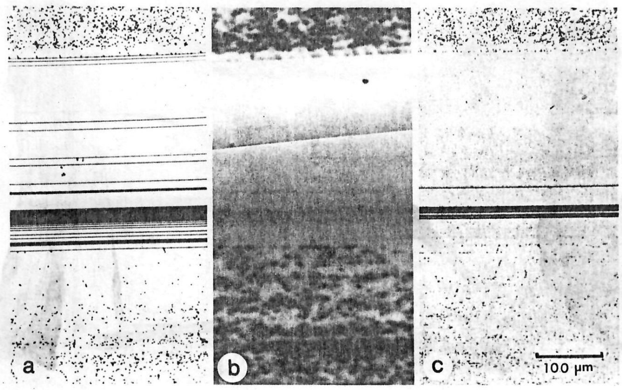


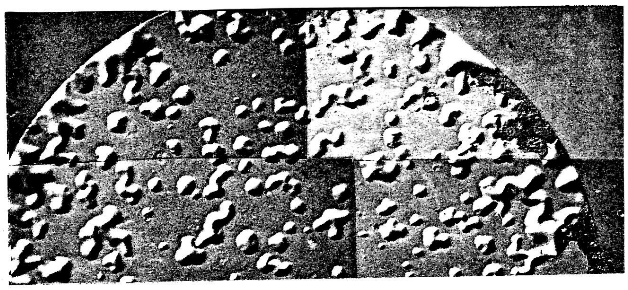
anodically etched at 1V — EBIC — anodically etched at 0.4V

That project was a beauty.
It came into being . because I was stupid but quite successful. It should change my (professiona) life forever.
I shall have much to say about this.

Advantages: Very quick and cheap method for revealing defects in p-type Si (solar cell material). Superior resolution compared to EBIC. No ambiguities as in chemical etching. Transfers defect etching from black art (chemistry) to exact science (physics). Potentially applicable for all semiconductors.

(IV) Corrosion of Pd_2Si

Background: Pd_2Si contacts on devices made in the pilot line showed "bubbling" after 2 - 4 weeks. The reasons for that were unclear

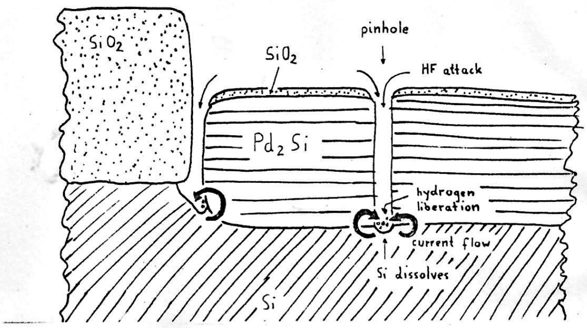


(6) bubbles in Pd_2Si contact

Experimental observations: Bubbling occurs within a few hours if the specimen is immersed in very diluted HF. The Si below the bubble is partially dissolved. Bubbling was explained to be a corrosion phenomena with HF as the active agent and the potential difference between Pd_2Si and Si as the driving force.

That "bubbling" was kind of embarrasing.
That's why it was kept confidential

Model for bubbling:



(7)

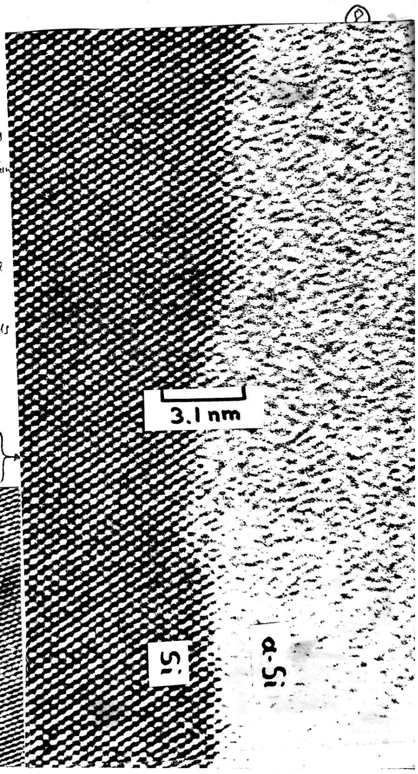
A local element between Si and Pd_2Si is formed in exposed areas (edges of contact; pinholes); the electrochemical reaction proceeds under dissolution of Si and liberation of H. HF, present in traces in the atmosphere, acts as electrolyte. The liberated hydrogen is mobile in the Pd_2Si - Si interface and gets trapped at interface imperfections. Eventually H_2 will form by reaction with other hydrogene and high-pressure cells form in the neighbourhood of the electrochemical reaction. In the final stage, the pressure is so high that the Pd_2Si is lifted off. This model is supported by the observation (Si-line) that bubbling in devices is sensitive to the humidity of the atmosphere. A possible factor which would support bubbling in Pd_2Si is the possible presence of voids in the Pd_2Si - Si interface.

(V) High resolution TEM of defects in ion-implanted Si

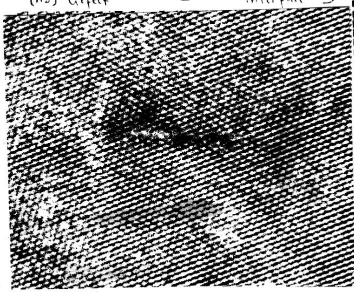
Basic goal: Identification of radiation-induced defects in Si; in particular nature of the {113} defect (planar defect on {113} planes observed after all kinds of irradiation at $T \geq 400°C$). This goal has not yet been achieved.

Highlights: Although the detailed structure of the {113} defect is still mysterious, existing pictures inspired extensive modelling (T.Y. Tan). Observations of interest were: crystalline-amorphous interface in ion-implanted samples; nature of damage below amorphous interface; formation of microcracks after hydrogene implantation (This supports bubble model)

(8)



{113} defect (9) crystalline-amorphous interface



Quite an interesting little project.
I do believe that I propduced another "firsts" here.

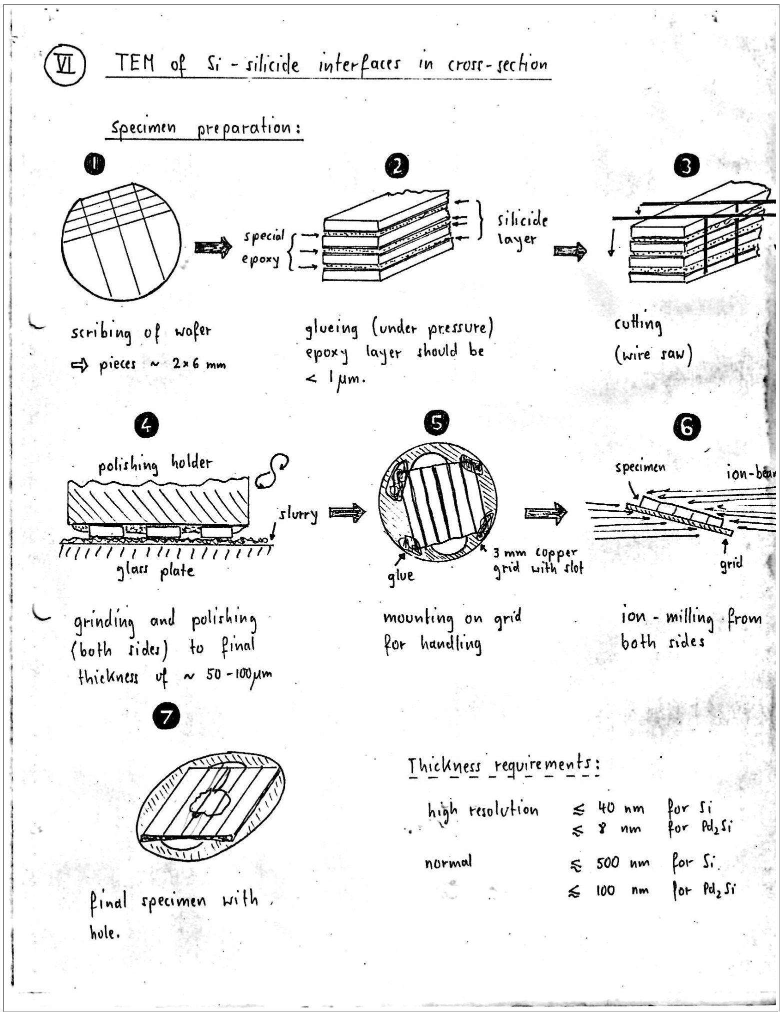
stacking fault with dislocations and small microcracks after H_2^+ implantation

(10)

large microcrack

(11)

Rather tricky defects here.

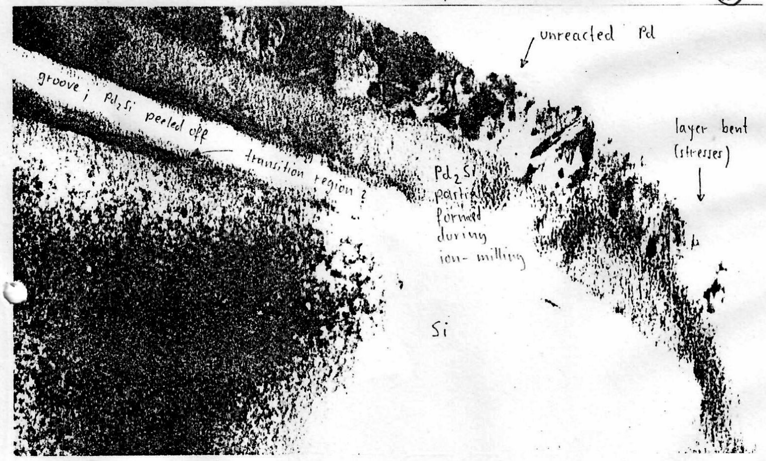


That page goives an idea of what it menat ot prepare a TEM sample.

Microscopy: Siemens Elmiskop 102 (Cornell) for high resolution
JEOL 200 B for normal mode.

Major problem: Different transparency of specimen left and right of interface(s). Distinguishing artifacts from true interface features. Artifacts might be due to specimen heating during ion-milling; stress-relief when specimen is thin (bending and peeling off), preferential milling of one component, sputtering of atoms on Si surface, ununderstood contrast effects.

(12)

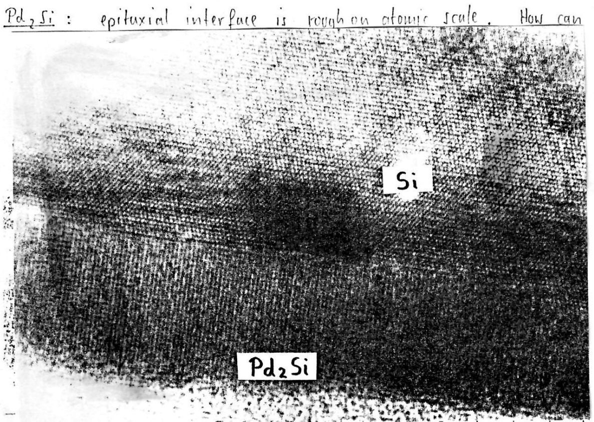


Peeling often observed for Pd_2Si formed at low temperatures, possibly because of presence of voids in interface.
Is the "transition region" (see also next picture) real or caused by Pd, which was sputtered on to this area and reacted?

Illustrating problems

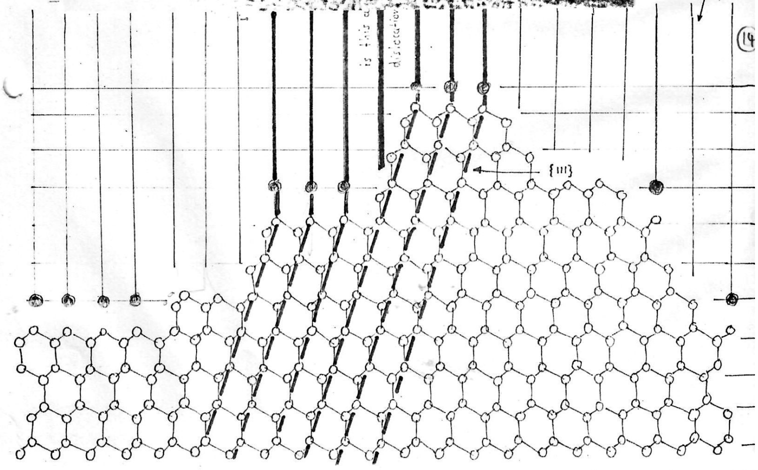
Results: Discussed by pictures

Pd_2Si: epitaxial interface is rough on atomic scale. How can that be?



(13)

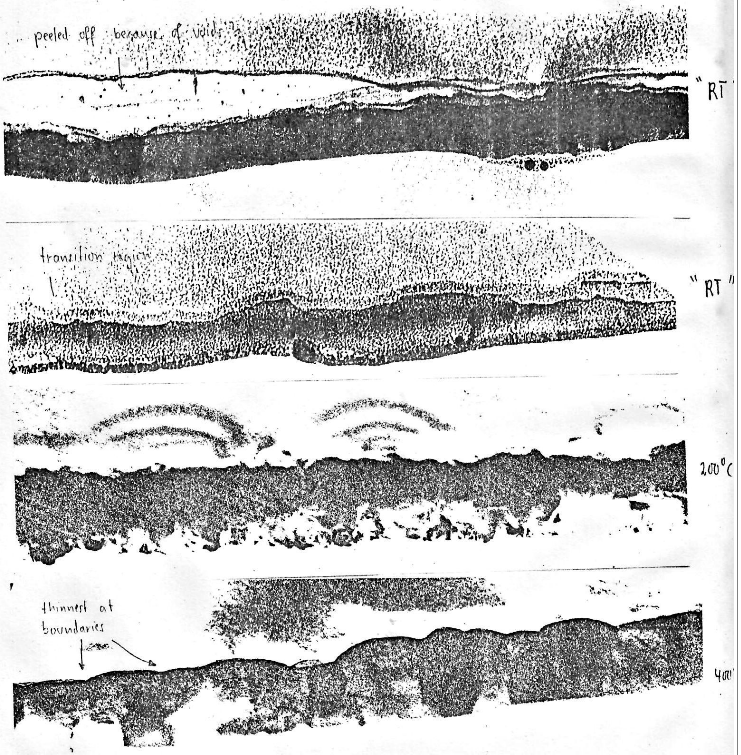
(14)



Definitely the first HRTEM picrture of a heterogeneous interface.

A Burgers-like circuit show ending Pd_2Si {2240} fringes. Are these interface dislocations? What is the meaning (and definition) of dislocations in the interfaces of crystals with different structure?

Pd_2Si interface structure changes with annealing temperature (15)



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Nickel - silicides

Epitaxial $NiSi_2$: (formed by anneal at 800°C) Heavily facetted; very rough on {100} Si; rather smooth on {111} Si and twinned with respect to matrix. Interfaces atomically smooth within a facet. Misfit dislocations are present

pictures: facetted $NiSi_2$ on {100} Si; direct lattice image; misfit disl.
facetted $NiSi_2$ on {111} Si; direct lattice image; misfit disl.

Pt Si

rough interface, smooth surface (contrast to Pd_2Si)

Al - Ti - Pd_2Si - Si system

Pd-rich region grows; interface gets rough with annealing