1.2.2. TEM Work at Cornell University

TEM Investigations of Grain Boundaries in Silicon

My main project during my post-doc time at the Institute of Materials Science and Engineering at the Cornell University, Ithaca, New York, State, USA. The topic was actually a bit more involved since t the grain boundaries were "custom made" by sinterinng two pieces of Si together with a predetermined orientation. In the simplest case this meant two put two Si wafers with either {100} or {111} orientations on top of each other with some specific misorientation. Later this was called wafer bonding and made big waves in the Si science and engineering community. Bonding was tricky, and TEM specimen preparation was extremely tricky. But the pictures I got were rewarding and worth the effort. The Siemens Elmiskop 102 TEM was in excellent conditions, thanks to the care of Ray Coles, the technician in charge. It allowed my to employ high resolution TEM (HRTEM) and I produced spectacular images, may a kind of "first". The **picture** shows a small-angle twist boundary on a {111} plane that contains three sets of screw dislocations that are split into partial dislocations and interact to form a complex network containing intrinsic and extrinsic stacking faults at the nodes. I also "discovered" a new imaging technique (kinematic multi-beam bright field) that allowed to see all dislocations at high resolution.



The picture also shows some amorphous stuff in the boundary (probably SiO₂ lumps), so-called "double ribbons (see below).

Double Ribbons and the Stacking Fault Energies in Si

The topic is "Direct TEM Determination of Intrinsic and Extrinsic Stacking-Fault Energies"; see <u>No 18 in</u> the publications <u>list</u>. I would have counted that as a not so interesting minor technical paper but as I found out (by accident), it is one of my most cited ones with **100** citations! It might be hard to believe but that is a lot of citations for this field. Well, the article was mostly written by Barry Carter who knew a lot more about the topic than I by then (and now). It may be a bit hard to see why this article caused some minor excitement among the (probably no more than 50) cognoscenti then. But in 1979 "dissociated" dislocations were still a hot topic, in particular in semiconductors. Read the introduction of the article to find out more. So I give you some of the original pictures here.

Defects in EFG Si Ribbons



Nov. 25th 1973 is a day to remember. It was a Sunday and you were forbidden to drive your car on a German "Autobahn"! WEE had the high point of the first (world-wide) oil crisis! The crisis started the very first wave of research in what today we like to call "green" energies, in particular solar energy and solar cells. My post-doc contract with Cornell university included TEM studies in Si ribbons made by the "Edgedefined Film fed Growth" (EFG) method. I actuality did a lot of work on this topic – but never published anything! I include it here because this would started my on solar cell research, a topic that I kept up through most of my professional life. The picture gives an impression of what I was up to in 1977. Nobody has looked much at defects in silicon, and what I saw was not always clear. In fact, now, in 2022 and about 50 years later, I still don't know what these striped things are.



Weak Beam Contrast of Stacking Faults in TEM

This is a "on the side" topic that let to a highly technical paper (No. 16 on the list) for which Barry Carter did most of the work. I wouldn't have included it here except for a special reason: It contains the first HRTEM picture that was actually taken to solve a problem! The problem was that stacking faults showed unexpected contrast behavior under certain conditions but that it was usually not quit clear if what you saw really was a stacking fault or, e.g. a micro-twin. Read the paper if you want to know more. By some fancy preparations and TEM work, I was able to show by HRTEM that the defect we investigated was indeed a intrinsic / .extrinsic stacking fault combination. My former Ph. D advisor M Wilkens supplied the theory.

The picture shows it all: Two intrinsic and one extrinsic stacking fault meet at the dark area.

The paper actually caused some discussion and "comments" in the literature; see, e.g., No 39 in my publication list.



First HRTEM picture taken to solve a problem!

/ Links to:

- Chapter 2: Chapter 2: Early TEM Work in Stuttgart
- Chapter 3: TEM Work at Cornell University
- Chapter 4: Research at IBM T.J. Watson Research Center
- Chapter 5: Research at Siemens in Munich
- Chapter 6: Research at the Christian-Albrechts-Univesität zu Kiel
- Chapter 7: Hyperscripts and Teaching in Kiel