Experimental problem uniquely solved by TEM: Observation of dislocations and their dynamics

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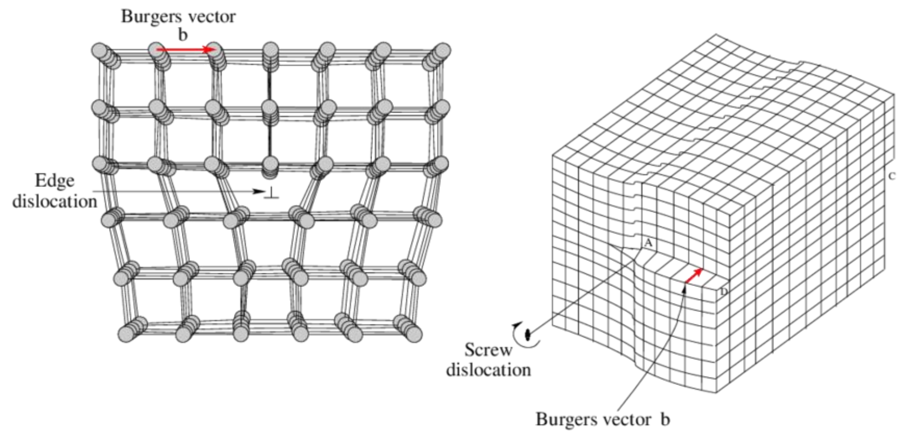
3000-year-old problem

- Why can metals be work-hardened and annealed?
- Why is steel harder than iron?

Theory of dislocations

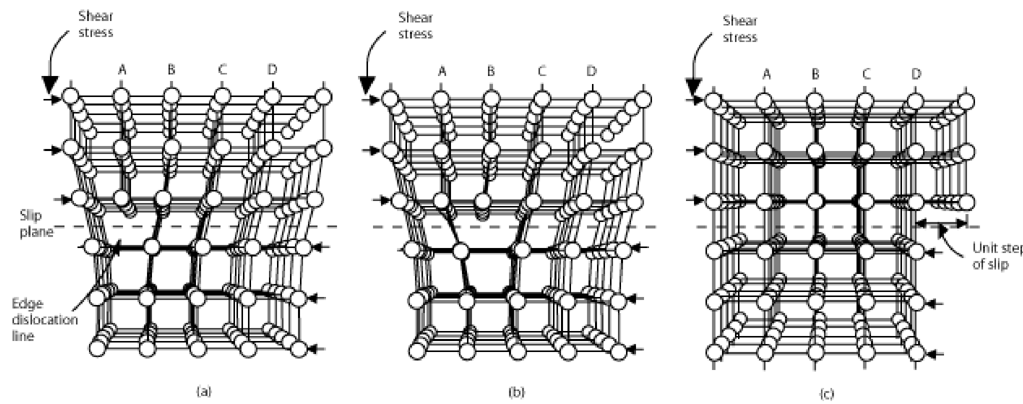
- Taylor, Orowan, and Polyani (independently) – edge dislocation, 1934.
- Burgers – screw dislocation and Burgers vector, 1939.
- Frank and Read (independently) – formation and multiplication of dislocations (Frank-Read source), 1950.



González-Viñas, W., & Mancini, H. L. (2004). *An introduction to materials science*. Princeton, NJ: Princeton University Press.

Theory of dislocations – movement and pinning

- perfect single crystals are very hard (compared to imperfect ones at least) – but also very rare
- dislocations are defects that can easily move through crystal and cause slippage
- they can pin on each-other (when there are many of them, like after work hardening) and other defects (like alloy precipitates), thus increasing the hardness again
- finally dislocations can be un-pinned and destroyed by heating (annealing) – creep and recrystallization

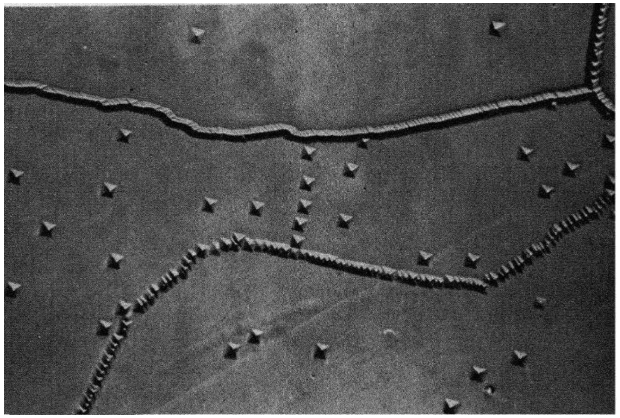


Hertzberg, R. W. (2012). *Deformation and fracture mechanics of engineering materials.* Chichester: John Wiley & Sons.

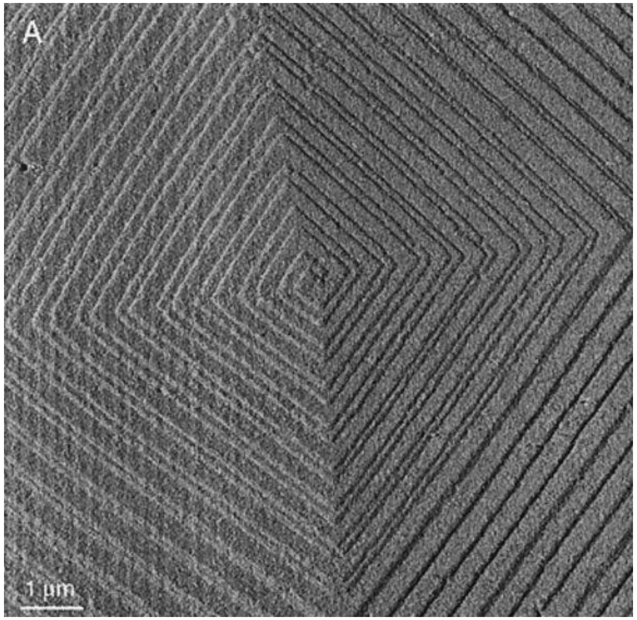
Theory was essentially complete in late 40's with little experimental evidence for existence of dislocations!

Observation of dislocations – first attempts

- etching – observation of strain patterns at the intersection of dislocation with the surface
- observation of crystal growth around screw dislocation
- many X-ray diffraction attempts (smeared spots…), but nothing besides twinning and grain sizes



Dislocation pits on the etched surface of LiF.[1]



Crystal growth around a screw dislocation in calcite (AFM image).[2]

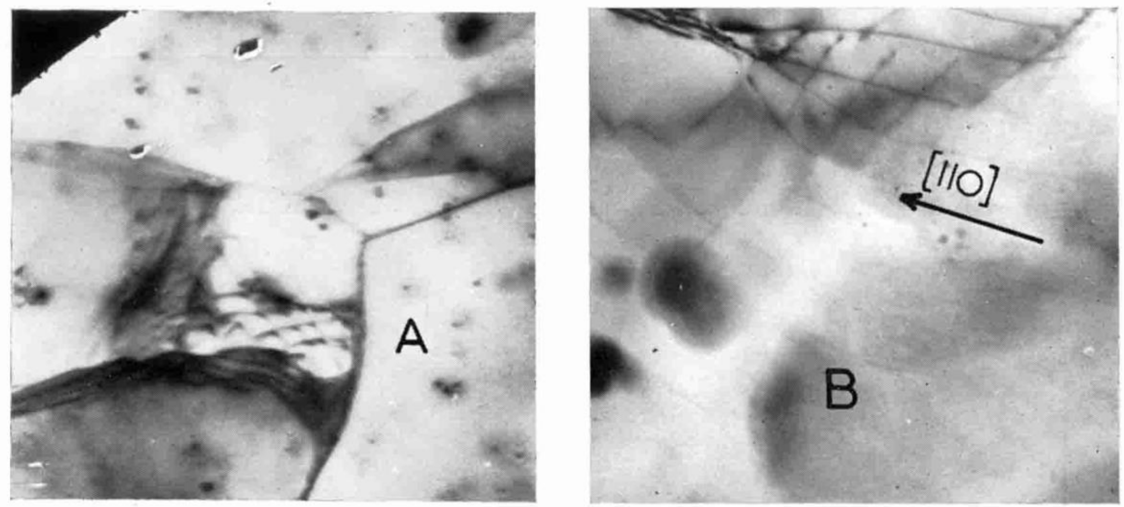
[1] J. J. Gilman and W. G. Johnston. *Dislocations and Mechanical Properties of Crystals (a report of the 1956 Lake Placid Conference).* p. 116. John Wiley and Sons.
[2] Teng, H., Dove, P. M., Yoreo, J. J. (2000). *Geochimica Et Cosmochimica Acta, 64*(13), 2255-2266.

This was still inconclusive, at best gave only dislocations crossing the surface and the most important theoretical predictions were untestable (like moving dislocations)!

First direct observation of dislocations - TEM

- 1955-56, Peter Hirsch, X-ray crystallographer (PhD under Bragg, the younger).
- TEM reached nanometer resolutions at the time, so considered a better prospect for finding dislocations.
- Problems – thin enough foils to get transmission (hammered gold, etched aluminium), interpretation (possibility of Moire patterns).
- First dislocations were observed in gold, but aluminium was annealed and produced nicer pictures, so it was published first!

Fig. 7



1000 Å

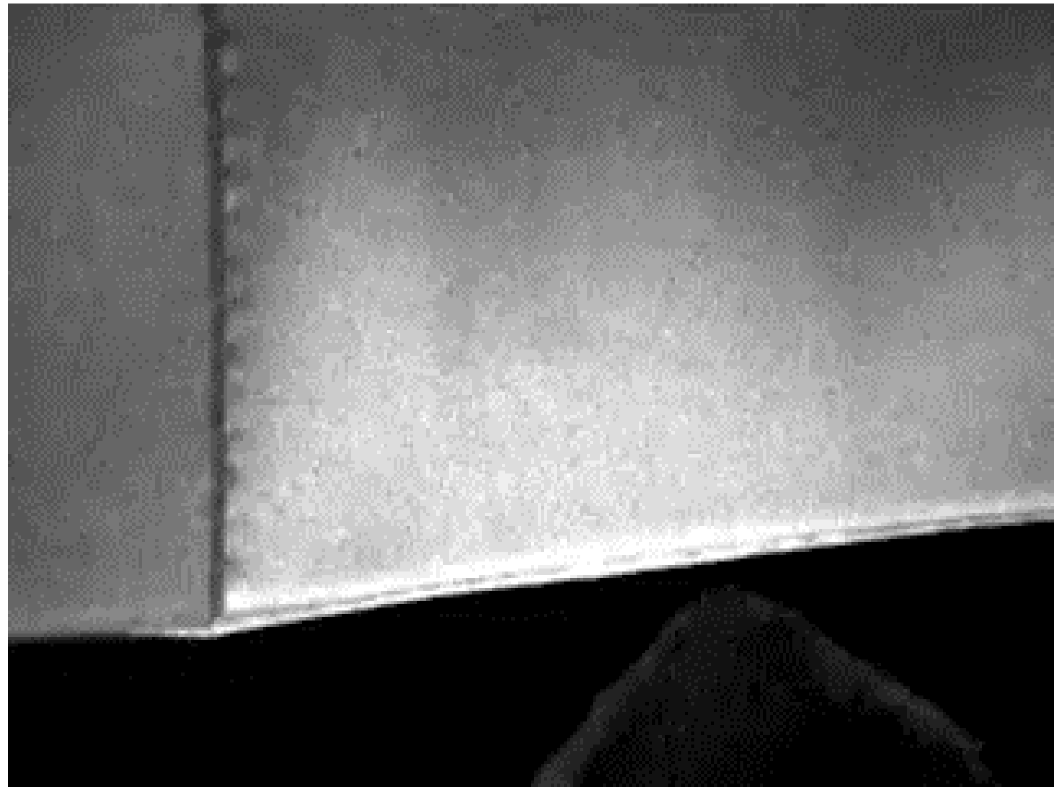
Mag. ×100 000

A, B :—Square cross-grids of dislocations. The dislocations in B are approximately parallel to [110] directions.

Hirsch, P. B., Horne, R. W., Whelan, M. J. (1956). LXVIII. Direct observations of the arrangement and motion of dislocations in aluminium. *Philosophical Magazine, 1*(7), 677-684.

So the lines started running around, and from there on everything became clear.[1]

- Solutions for problems
- Aluminium was beaten, annealed, then etched with HF to 0.5 µm thickness. (Gold was just beaten.)
- Tilting the sample changed the contrast, but lines were still there.
- Switching to higher brightness setting (not suitable for diffraction) revealed moving lines!
- Moving dislocations showed many of the characteristics predicted by theory (movement along glide planes, Frank-Read sources).


[2]

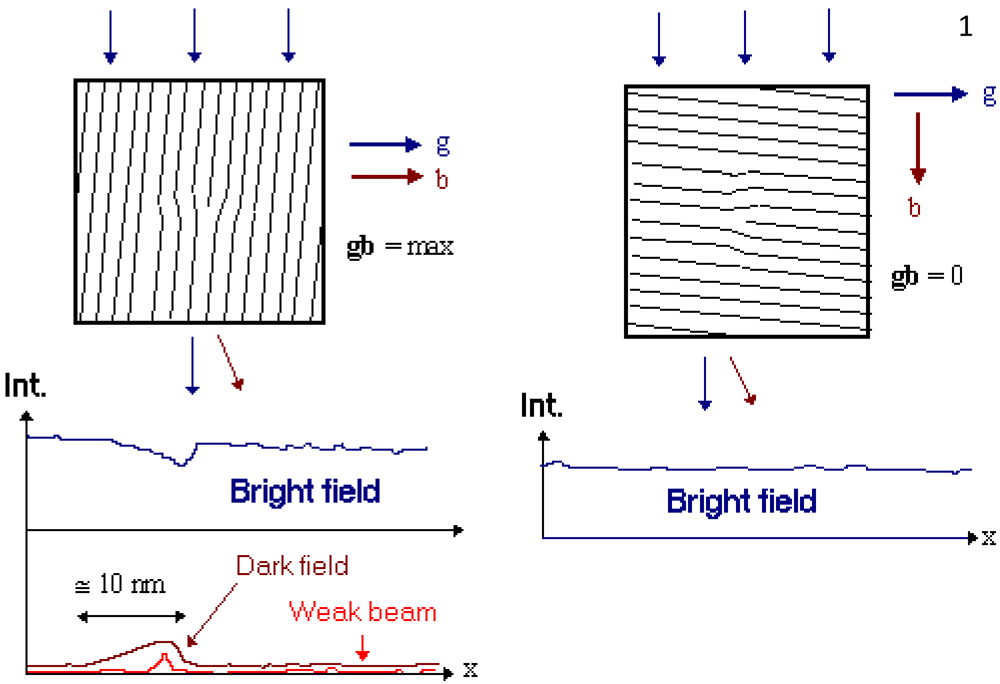
Generation, movement and pinning of dislocations near grain boundary in $SrTiO_3$.

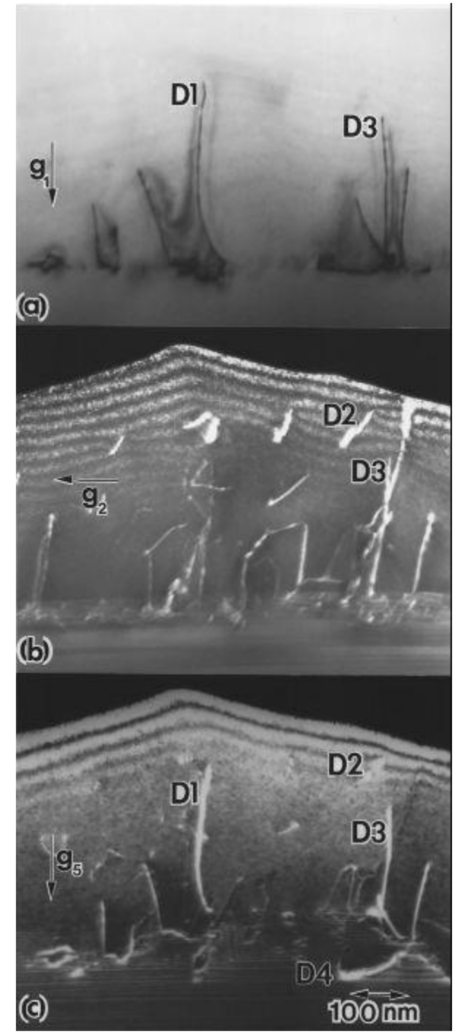
[1] AIP Oral History Interviews: Interview with Peter Hirsch.

[2] Kondo, S., Mitsuma, T., Shibata, N., & Ikuhara, Y. (2016). Direct observation of individual dislocation interaction processes with grain boundaries. *Science Advances, 2*(11).

TEM – still by far the leading technique for dislocation imaging

- Quantitative results are possible as well – like Burgers vector analysis.
- Intensity of beam diffracted from dislocation is proportional to $\vec{g} \cdot \vec{b}$.

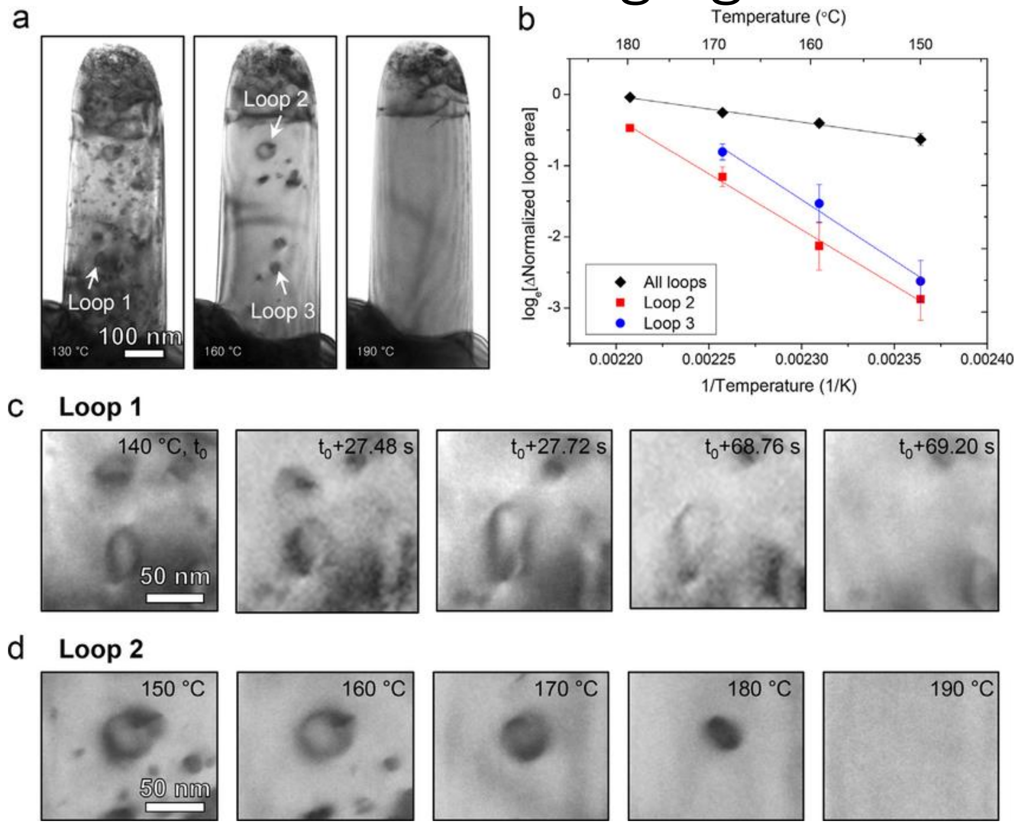




Burgers vector analysis of dislocations in GaN film. (a) is taken in bright field mode, while (b) and (c) are taken in dark field mode with different diffraction vectors.[2]

[1] https://www.tf.uni-kiel.de/matwis/amat/def_en/index.html
[2] Cherns, D., Young, W., & Ponce, F. (1997). *Materials Science and Engineering: B, 50*(1-3), 76-81.

TEM – still by far the leading technique for dislocation imaging



Annihilation of dislocations by annealing in a pillar of Al. Case (c) shows a dislocation than vanished by "escaping through the surface". Case (d) shows a dislocation loop that shrank by diffusion of vacancies. Rate of heating is 5K/min.

Lee, S., Jeong, J., Kim, Y., Han, S. M., Kiener, D., & Oh, S. H. (2016). *Acta Materialia, 110*, 283-294.

Thank you!