Single crystal thin film mechanical behavior measured with nanoindentation

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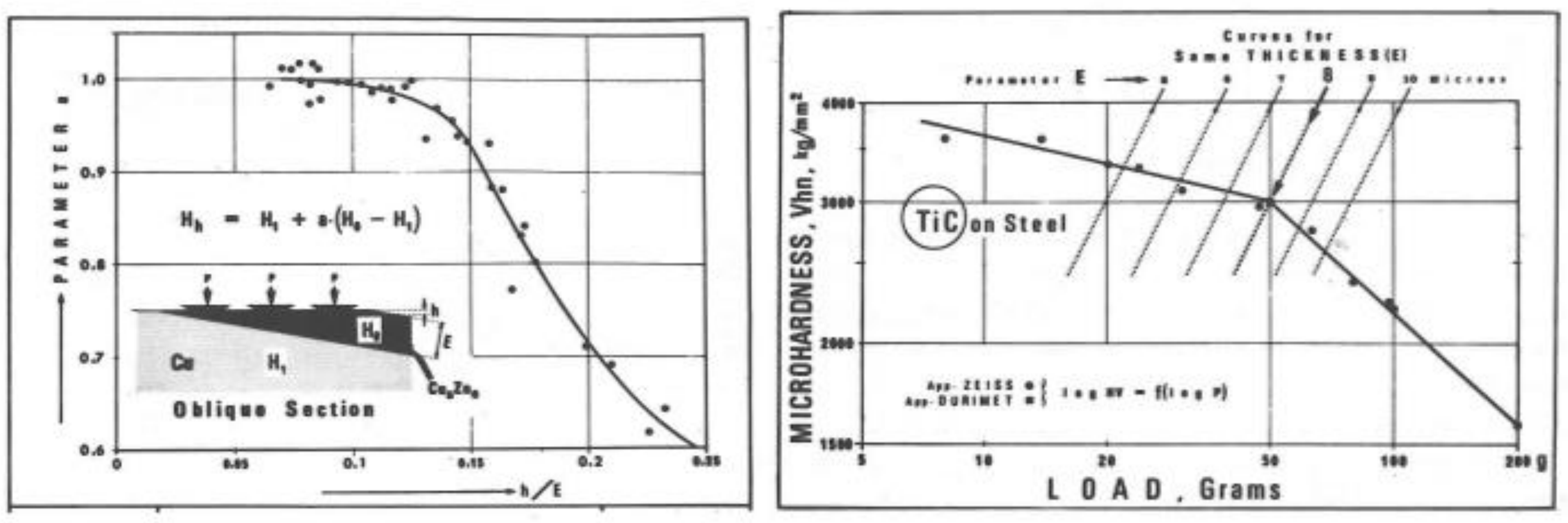
Motivation

One of the most common methods to measure the elastic modulus and hardness of thin films is to use nanoindentation and the well-known "10% rule of thumb". The 10% rule of thumb has evolved to the understanding that elastic modulus and hardness can be taken at 10% of the film thickness with no or little influence from the substrate, even though only hardness was stated in the original Bückle paper. While this guideline may hold true for some film-substrate systems and film thicknesses (greater than 1000 nm), it cannot and should not be applied universally. It will be shown on single crystalline copper films on sapphire, grown by thermal evaporation (50, 100, and 300 nm thick) that the hardness can be evaluated but the elastic modulus cannot be properly measured when compared to bulk single crystal copper. It will be demonstrated that the elastic modulus is a long range property that is substantially influenced by the substrate even at indentations of 10% of the thickness. Using the initial Hertzian elastic portion of the load-displacement curve before a pop-in occurs does not allow for the elastic modulus of copper to be measured. The findings reveal that the 10% rule should not be applied to evaluating the elastic modulus of thin films and hardness can be measured for some film system up to 60% of the film thickness.

Origins of the 10% rule of thumb

- First mentioned by H. Bückle in 1973
- THICK (8 µm) hard coating on a soft substrate
- Only hardness – Vickers indenter

„It is found that for hard coatings on less hard substrates the intrinsic hardness of the coating is measured with indentations the depths of which are less than a tenth of the coat thickness. The hardness curve resulting from measurements made on the surface under increasing load is therefore affected by a displacement corresponding to the value of h = E/10 [h is thickness, E is the coating displacement]."

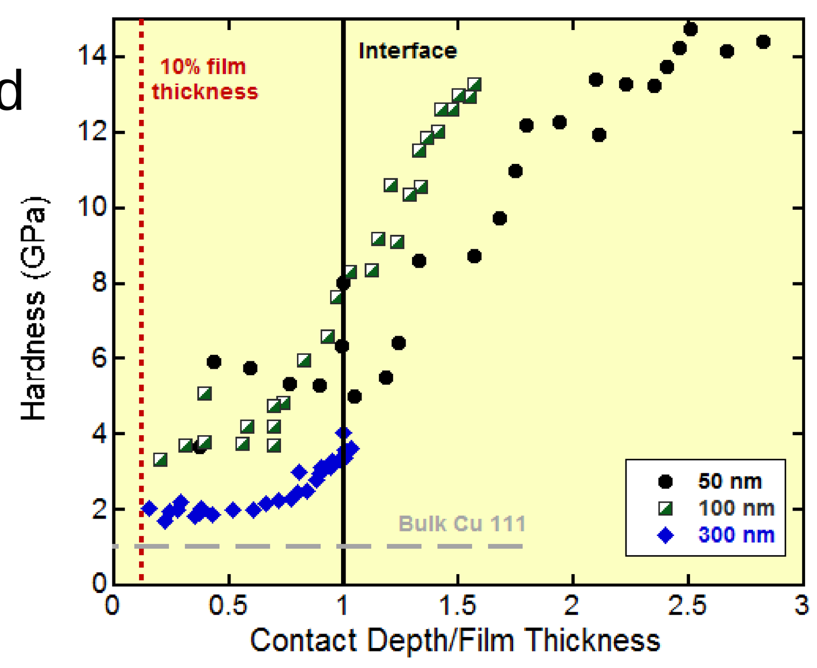


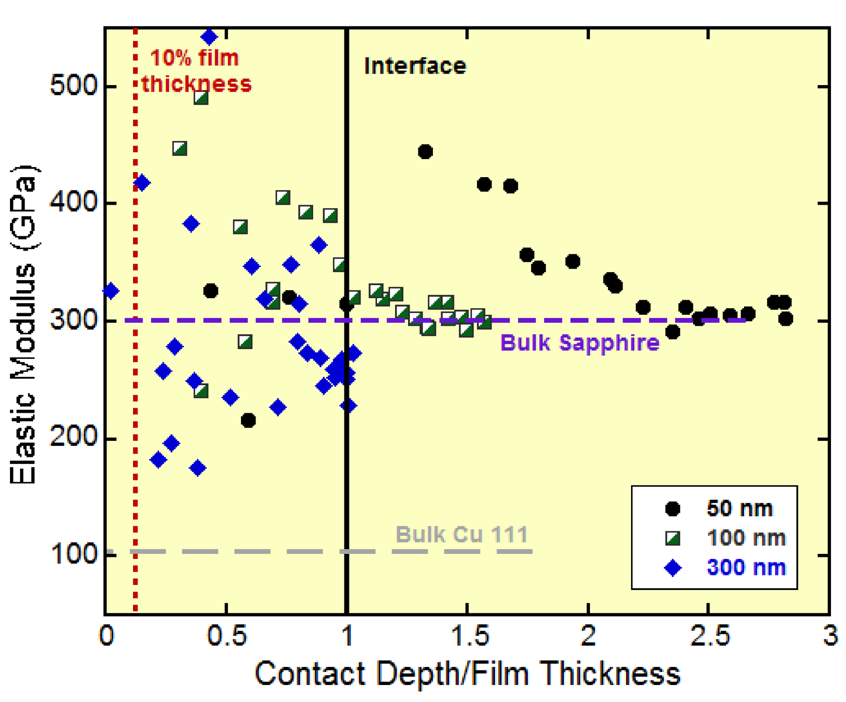
- No mention of elastic modulus
- Thin films < 1 µm?
- Soft on hard systems?

H. Bückle, ch. 33, p. 453-494, in The Science of Hardness and Its Research Applications (1973) eds. J.H. Westbrook & H. Conrad, published by ASM

Hardness and Elastic Modulus

- Below 10% film thickness, hardness could not be evaluated with the loads used.
- Generally, hardness increased with decreasing film thickness.
- Sapphire substrate is probed well before the interface is reached
- For 300 nm film, hardness plateau was observed for about 60% of the film thickness.

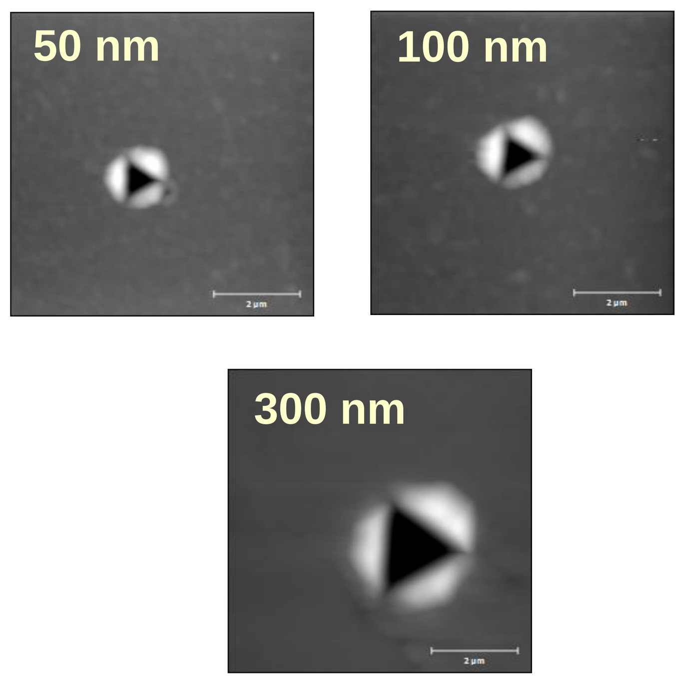


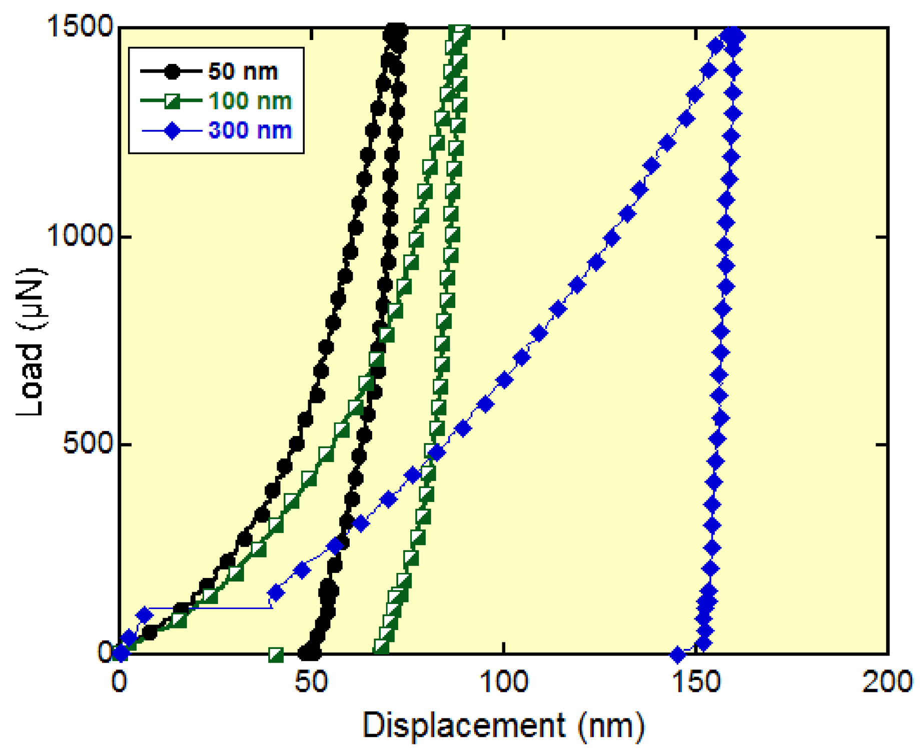


- At 10% the film thickness the (average) elastic modulus was found to be that near Sapphire for all film thicknesses.
- All films illustrated a lot of scatter of elastic modulus values between 200 and 400 GPa
- Bulk Cu 111 values were never reached (approx. 110 GPa).

Single Crystal 111 Cu films on Sapphire

- 50, 100, 300 nm Cu 111 films on Sapphire & bulk 111 Cu
- Indent with 300 nm radius Berkovich tip on a TriboIndenter
- Loads 100 µN and 10,000 µN, indents imaged after
- Pop-ins observed in 100, 300 nm
- Oliver-Pharr used to evaluate:
  - Hardness
  - Elastic Modulus
- Pile-up was ignored for now



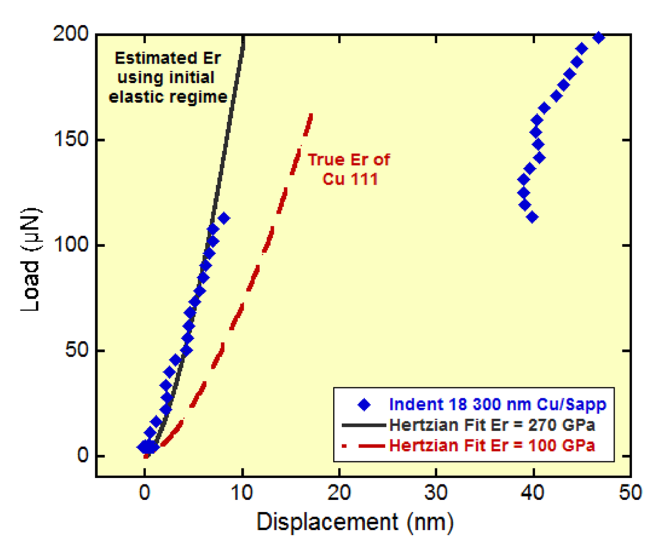


Above: SPM images of indents. Left: Representative Load-Displacement curves of three different films

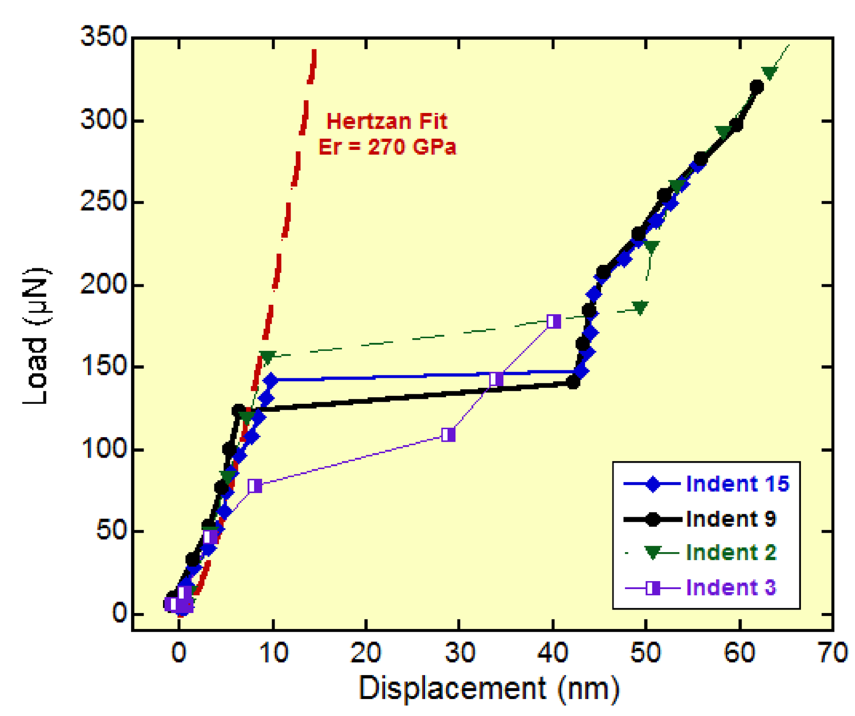
Hertzian Theory and Elastic Modulus

- First pop-ins are elastic and less than 10% film thickness. Can they be used to measure thin film elastic modulus?
- With Hertzian Theory, keep tip radius constant, but change Er (reduced elastic modulus) to fit initially elastic pop-in

$$P = \frac{4}{3} E^* R^{1/2} \delta^{3/2}$$



Example: Er = 270 GPa (close to Sapphire) fits the initial elastic loading of the 300 nm Cu film. The true Er for Cu 111 is close to 100 GPa.



Er = 270 GPa fits the initial elastic loading of the 300 nm Cu film of several indents.

Hertzian theory cannot be used to measure thin film elastic modulus with elastic pop-ins

Summary

- Hardness, short range property, can be measured up to 60% of film thickness
- Elastic Modulus, long range property, can not be measured