CHAPTER 34
MEASURING HARDNESS

34.1 INTRODUCTION

Technical characteristics
• Hardness measurements • Down to submicron-size features • Adhesion evaluation • Calculation of the elastic modulus (Young's modulus)

Definition of hardness

Hardness measurement is one of the most widely applied coating testing methods. *Hardness* is defined as the material's resistance to permanent indentation by another object under the influence of a force (F). As a measure for the hardness (H), the applied load (P), and the affected surface area (A) are related through the following expression:

$$H = \frac{P}{A} \tag{34.1}$$

Over the years hardness measurements have moved from more bulky macro-level methods to micro hardness and the current state-of-the-art: nanoindentation.

34.2 HARDNESS MEASUREMENT

As part of the development of different models describing the underlying mechanical theory, different types of indenter tips have been used. These are typically made from a very hard material such as diamond. Figure 34.1 shows an overview of different tips found in the literature.

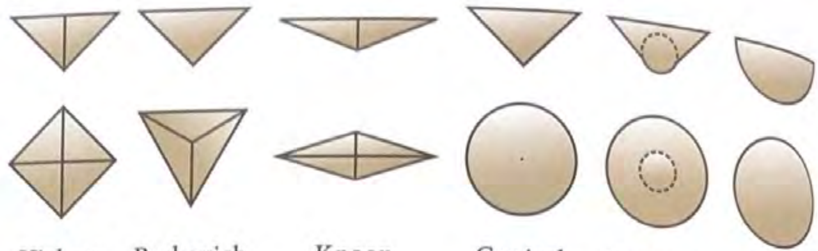


Figure 34.1: *Overview of different types of indenter tips used in connection with micro hardness measurements and nanoindentation applied over the years, in connection with the development of different theoretical models. Knoop and Vickers are used in connection with micro hardness measurements. Standard Rockwell C tests applies more spherical indenters.*

In hardness testing of surface coatings, two different types of hardness numbers are applied, which are based on measurement with two different diamond indentation geometries. The first is the Knoop hardness and the second is the Vickers hardness. The geometry of these two diamonds are calibrated in such a way that the same load will result in the same surface area, generating the same hardness value. Figure 34.2 shows the geometry of a Knoops and a Vickers diamond, respectively

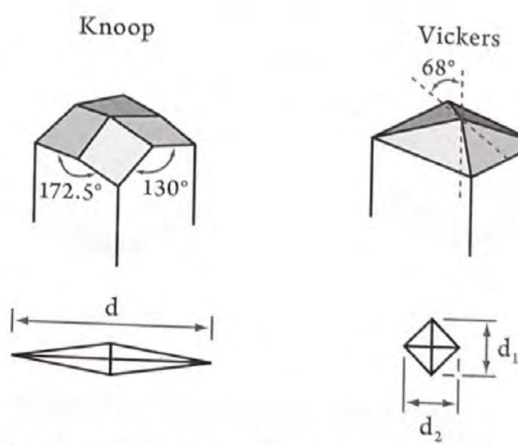


Figure 34.2: *The geometry of the Knoop and Vickers diamond, respectively.*

Due to the difference in the diamond indenter geometry of the Knoop and the Vickers hardness, the resulting formula for the hardness is also different. The expression for Vickers hardness is:

Vickers hardness

$$HV = \frac{1.8544P}{d^2} \qquad (34.2)$$

Where P is the load in kp (kilopond) and d is the length of the medium diagonal of the indentation measured in millimeters.
A simplified expression for the Knoop hardness is as follows:

Knoop hardness

$$HV = \frac{14.229P}{d^2} \tag{34.3}$$

Where P is the load in kp and d is the length of the long diagonal in millimeters.

Macro hardness

In hardness measurement it is distinguished between macro hardness and micro hardness. Macro hardness is the hardness at high loads, typically higher than 10–20 kp.

Micro hardness

When you are testing the hardness of a coating you usually apply smaller loads so that the indentation depth is sufficiently small. This type of hardness measurement is normally done with a micro hardness instrument by a nanoindenter applying even smaller loads. The applied loads in connection with micro hardness measurements are usually around 5–200 p, and the diagonals of the base area is less than 100 μm.

To determine the hardness of a surface coating, it is required that the coating has a certain layer thickness to avoid measuring the hardness of the base material instead. This is especially the case when using a micro hardness instrument and less important when applying a nanoindenter. How big the least necessary layer thickness of the coating has to be is highly dependent on the hardness of the coating and the applied load. Figure 34.3 shows the least necessary layer thickness as a function of the hardness of the coating and the applied load, when using a micro hardness indenter. In the case of a nanoindenter, the layer thickness can be as low as 200 nm.

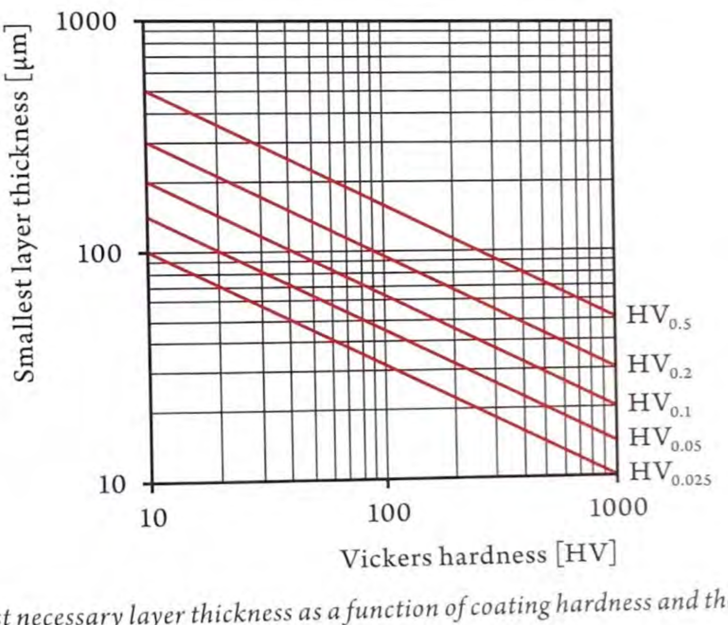


Figure 34.3: *The least necessary layer thickness as a function of coating hardness and the applied load in kp when applying a micro hardness indenter.*

Depending on the mechanical properties of the material, different generic types of loading and unloading curves can be observed (see Figure 34.4).

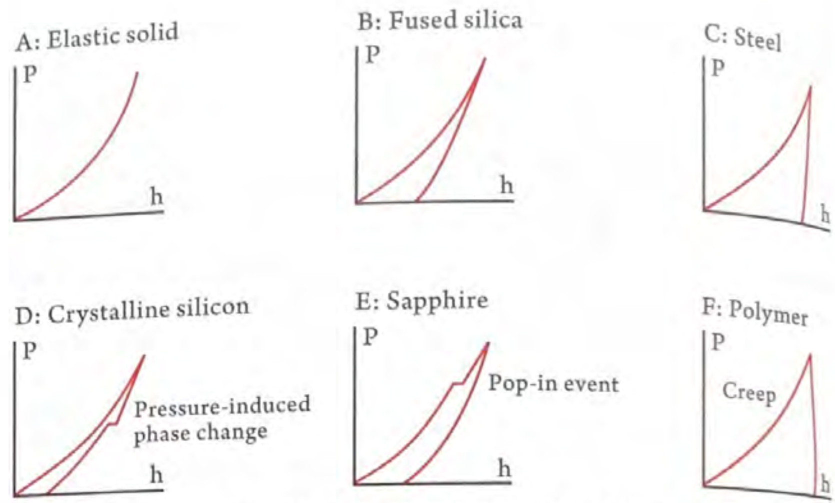


Figure 34.4: *Different scenarios of load/unloading curves. A: Perfect elastic solid. B and C: Material revealing typical plastic and elastic properties with some irreversible deformations. D: Example of a crystalline material (e.g. silicon) that reveals a pressure induced phase change, where the cubic diamond structure of silicon (Si-I) transforms into a metallic phase (Si-II) under increasing indentation load. Such phase changes can be reversible and may therefore be observed both in the loading and unloading curves. E: A material revealing a 'pop-in' event caused by sub-surface cracking or the movement of dislocations displacements. F: A polymer material that reveals creep (permanent material deformation caused by the applied load).*

The hardness of a film can be determined from the loading/unloading curve as the maximum applied load (P_{max}) divided by the residual indentation area (A_r). The projected residual area can be measured directly by using the indentation tip as a *scanning probe microscope* (SPM)—see Section 36.13, or *ex situ* by using a light microscope or an electron microscope.

$$H = \frac{P_{max}}{A_r} \tag{34.4}$$

The hardness can be determined in two ways: (i) as a function of the indentation depth or (ii) as a single hardness value as in more conventional macro indentations. From, e.g. Equation (34.4), it is evident that the harder the material, the smaller the indentation area will be when applying the same load.

Clearly, it will be based on some approximation to come from the SEM/SPM image of the projected indentation area to the actual contact area during the indentation. A number of different theories and models have been developed to correlate the indenter area and the applied force to calculate the hardness. One of the more famous models (and cited papers in the field) has been developed by Oliver and Pharr (see below). Besides this, there are other models, which will also be addressed below.

34.2.1 MODELS FOR CORRELATING INDENTER AREA AND SURFACE HARDNESS

The Martens hardness

The Martens hardness, H_m, is given by the maximum displacement in the point of maximum indentation depth and maximum load (h_{max}, P_{max}):

Martens hardness, H_m

$$H_m = \frac{P_{max}}{A_s} \tag{34.5}$$

The maximal indentation depth (h_{max}) is used to calculate the contact surface area, A_s, based on the indenter geometry.
For a perfect Berkovich indenter, the relationship is: $A_s = 24.5\ h_{max}^2$

The indentation hardness

The indentation hardness, H_I is defined slightly different:

Indentation hardness, H_I

$$H_I = \frac{P_{max}}{A_p} \tag{34.6}$$

Where the hardness is the projected contact area A_p.

Doerner and Nix

Doerner and Nix have suggested that the indenter can be treated as a flat punch, introducing a contact area function: $A_c = f(h_c)$, whereby the hardness is given by:

Doerner and Nix hardness, H_{DN}

$$H_{DN} = \frac{P_{max}}{A(h_h)} \tag{34.7}$$

Doerner and Nix calibrated their tip area by applying a series of indentations in soft brass as a function of the indentation depth combined with electron microscope images.

Oliver and Pharr

Oliver-Pharr hardness

Oliver and Pharr improved the time-consuming tip characterization method proposed by Doerner and Nix by suggesting that the unloading curve for simple indenter geometries (sphere, cone, flat punch, etc.) follows a simple power law:

$$P = \alpha h^m \tag{34.8}$$

In this equation, P is the indenter load, h is the elastic displacement of the indenter, and α and m are constants. Oliver and Pharr applied this formulation to determine the contact area at maximum load as it is valid, even if the contact area changes during unloading. To do this, they derive the following relationship for the contact depth:

$$h_c = h_{end} - \theta \frac{P_{max}}{S} \tag{34.9}$$

Where θ = 0.72, 0.75 and 1, for cone, sphere and flat-punch-geometry, respectively, and S is the slope of the unloading curve, as seen in Figure 34.5 below. An Oliver-Pharr analysis will fit the power law function to the unloading curve, yielding the contact stiffness (S) as the slope. Combining this with the tip dependent value of θ the contact depth can be calculated. Finally, it is possible to calculate the hardness from the measurement. Figure 34.5 shows a schematic plot of such an analysis.

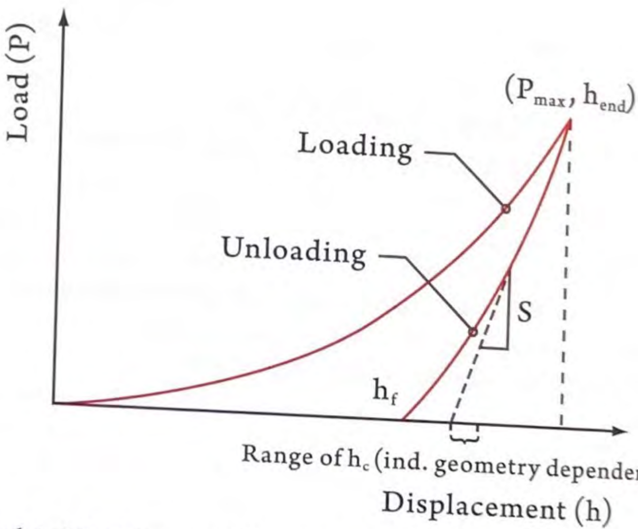
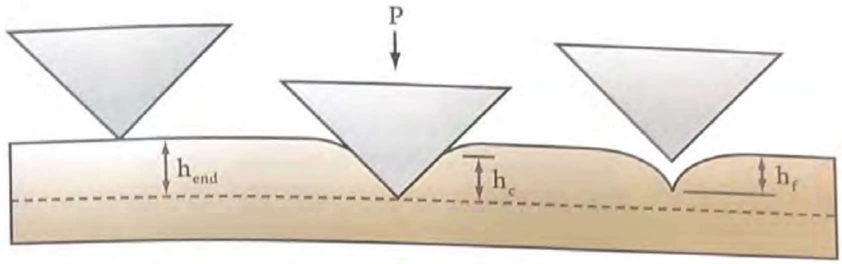


Figure 34.5: *Schematic plot of the Oliver and Pharr model and the different indenter depths (h_{end}, h_c and h_f).*

The depth h_{end} is the maximum indenter depth before unloading. After unloading, the material elasticity reduces the depth to h_f. h_c is the actual depth relevant for the indenter area due to surface deformations as shown in Figure 34.6.



re 34.6: *The relation of the maximal indenter depth h_{end}, the actual relevant indenter depth h_c and the left over indenter footprint, corresponding to an indenter depth h_f.*

From the hardness (H) and the slope of the unloading curve, S = dP/dh, it is possible to calculate the reduced Young's Modulus (E_r):

$$E_r = \frac{1}{\beta}\frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_p(h_c)}} \qquad (34.10)$$

Where $A_p(h_c)$ is the projected area of the indentation at the contact depth h_c, and β is a geometrical constant close to one. $A_p(h_c)$ is often approximated by fitting polynomial, as shown below for a Berkovich tip:

$$A_p(h_c) = C_0 h_c^2 + C_1 h_c^1 + C_2 h_c^{1/2} + C_3 h_c^{1/4} + \dots + C_8 h_c^{1/128} \qquad (34.11)$$

Where C_0 for a Berkovich tip is 24.5, while for a cube corner (90°) tip it is 2.6. The reduced modulus E_r is related to Young's modulus, E, of the test sample through the following relationship:

$$\frac{1}{E_r} = \frac{(1-\nu_i^2)}{E_i} + \frac{(1-\nu^2)}{E} \qquad (34.12)$$

The subscript i indicates a property of the indenter material and ν is Poisson's ratio. For a diamond indenter tip, E_i is 1140 GPa and ν_i is 0.07. Poisson's ratio of the sample, ν, generally varies between 0 and 0.5 for most materials and is typically around 0.3.