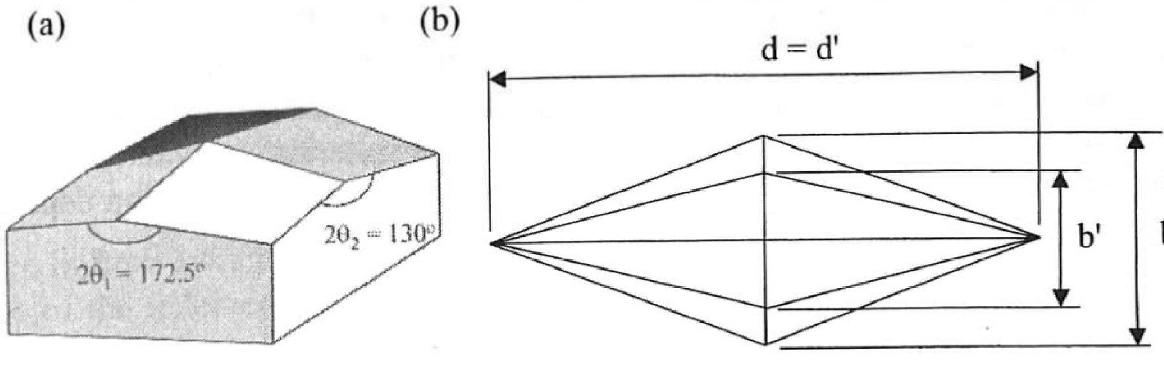

Fig. 2.4 (a) Geometry of a Knoop indenter. (b) The length of the long diagonal of the residual impression remains approximately the same from full load to full unload. The size of the short diagonal reduces from b to b' due to elastic recovery during unloading.

In Eq. 2.3.5b, α is a geometry factor found from experiments on a wide range of materials to be equal to 0.45. The ratio of the dimension of the short diagonal b to the long diagonal a at full load is given by the indenter geometry and for a Knoop indenter, $b/a = 1/7.11$. The primed values of a and b are the lengths of the long and short diagonals after removal of load. Since there is observed to be negligible recovery along the long diagonal, then $a' \approx a$. When H is small and E is large (e.g. metals), then $b' \approx b$ indicating negligible elastic recovery along the short diagonal. When H is large and E is small (e.g. glasses and ceramics), then $b' \ll b$. Using measurements of the axes of the recovered indentations, it is possible to estimate the ratio E/H for a specimen material using Eq. 2.3.5b.

2.4 Load-Displacement Curves

The principal goal of nanoindentation testing is to extract elastic modulus and hardness of the specimen material from experimental readings of indenter load and depth of penetration. In a typical test, load and depth of penetration are recorded as load is applied from zero to some maximum and then from maximum load back to zero. If plastic deformation occurs, then there is a residual impression left in the surface of the specimen. Unlike conventional indentation hardness tests, the size (and hence the projected contact area) of the residual impression for nanoindentation testing is too small to measure accurately with optical techniques. The depth of penetration together with the known geometry of the indenter provides an indirect measure of the area of contact at full load, from which the mean contact pressure, and thus hardness, may be estimated. When load is removed from the indenter, the material attempts to regain its original shape, but it prevented from doing so because of plastic deformation. However, there is some degree of recovery due to the relaxation of elastic strains within the material. An analysis of the initial portion of this elastic unloading response gives an estimate of the elastic modulus of the indented material.

The form of the compliance curves for the most common types of indenter are very similar and is shown in Fig. 2.5. For a spherical indenter, it will be shown in Chapter 5 that the relationship between load and penetration depth for the loading portion for an elastic–plastic contact is given by:

$$h = \frac{1}{2}\left(\frac{P}{\pi R_i H} + \frac{3}{4}\frac{\sqrt{P\pi H}}{\beta E^*}\right) \qquad (2.4a)$$

For the elastic unloading, we have from Eq. 1.2g:

$$h = \left[\frac{3}{4E^* R'^{1/2}}\right]^{2/3} P^{\frac{2}{3}} \qquad (2.4b)$$

For a Berkovich indenter, it will be shown in Chapter 5 that the expected relationship between load and depth for an elastic–plastic contact is given by:

$$h = \sqrt{P}\left[\left(3\sqrt{3}H\tan^2\theta\right)^{-\frac{1}{2}} + \left[\frac{2(\pi-2)}{\pi}\right]\frac{\sqrt{H\pi}}{2\beta E^*}\right] \qquad (2.4c)$$

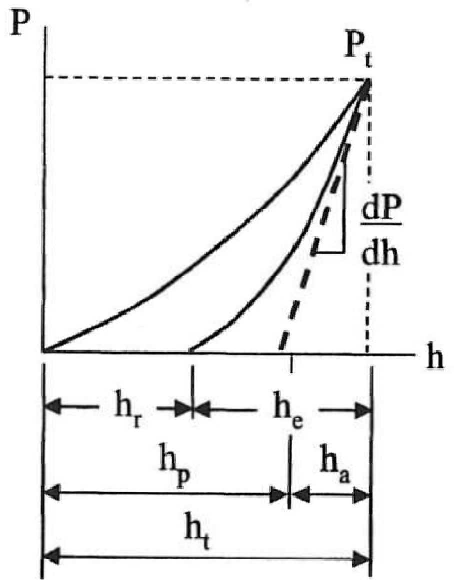

Fig. 2.5 Compliance curves, loading and unloading, from a nanoindentation experiment with maximum load P_t and depth beneath the specimen free surface h_t. The depth of the contact circle h_p and slope of the elastic unloading dP/dh allow specimen modulus and hardness to be calculated. h_r is the depth of the residual impression, and h_e is the displacement associated with the elastic recovery during unloading.

Upon elastic unloading we have from Eq. 1.2m:

$$h = \sqrt{P}\left(\frac{\pi}{2E^*}\right)^{\frac{1}{2}}\left(\frac{\pi}{3\sqrt{3}}\right)^{\frac{1}{4}}\frac{1}{\tan\theta'} \tag{2.4d}$$

where in Eqs. 2.4b and 2.4d the quantities R' and θ' are the combined radii and angle of the indenter and the shape of the residual impression in the specimen surface. The dependence of depth on the square root of the applied load in Eqs. 2.4a to 2.4d is of particular relevance. This relationship is often used in various methods of analysis to be described in Chapter 3.

In subsequent chapters, the methods by which elastic modulus and hardness values are obtained from experimental values of load and depth are described along with methods of applying necessary corrections to the data. In most cases, methods of analysis rely on the assumption of an elastic–plastic loading followed by an elastic unloading — with no plastic deformation (or "reverse" plasticity) occurring during the unloading sequence.

The indentation modulus is usually determined from the slope of the unloading curve at maximum load. Eq. 2.4e shows that the indentation modulus (here expressed as E^*) as a function of dP/dh and the area of contact.

$$E^* = \frac{1}{2}\frac{\sqrt{\pi}}{\sqrt{A}}\frac{dP}{dh} \tag{2.4e}$$

The indentation hardness is calculated from the indentation load divided by the contact area. The contact area in turn is determined from the value of h_p (see Fig. 1.3) and the known geometry of the indenter (Table 1.1). The value for h_p is found by an analysis of the load-displacement data (see Fig. 2.5).

Variations on the basic load - unload cycle include partial unloading during each loading increment, superimposing an oscillatory motion on the loading, and holding the load steady at a maximum load and recording changes in depth. These types of tests allow the measurement of viscoelastic properties of the specimen material.

In practice, nanoindentation testing is performed on a wide variety of substances, from soft polymers to diamond-like carbon thin films. The shape of the load-displacement curve is often found to be a rich source of information, not only for providing a means to calculate modulus and hardness of the specimen material, but also for the identification of non-linear events such as phase transformations, cracking, and delamination of films. Fig. 2.6 shows a schematic of some of the more commonly observed phenomena. It should be noted that in many cases the permanent deformation or residual impression is not the result of plastic flow but may involve cracking or phase changes within the specimen.

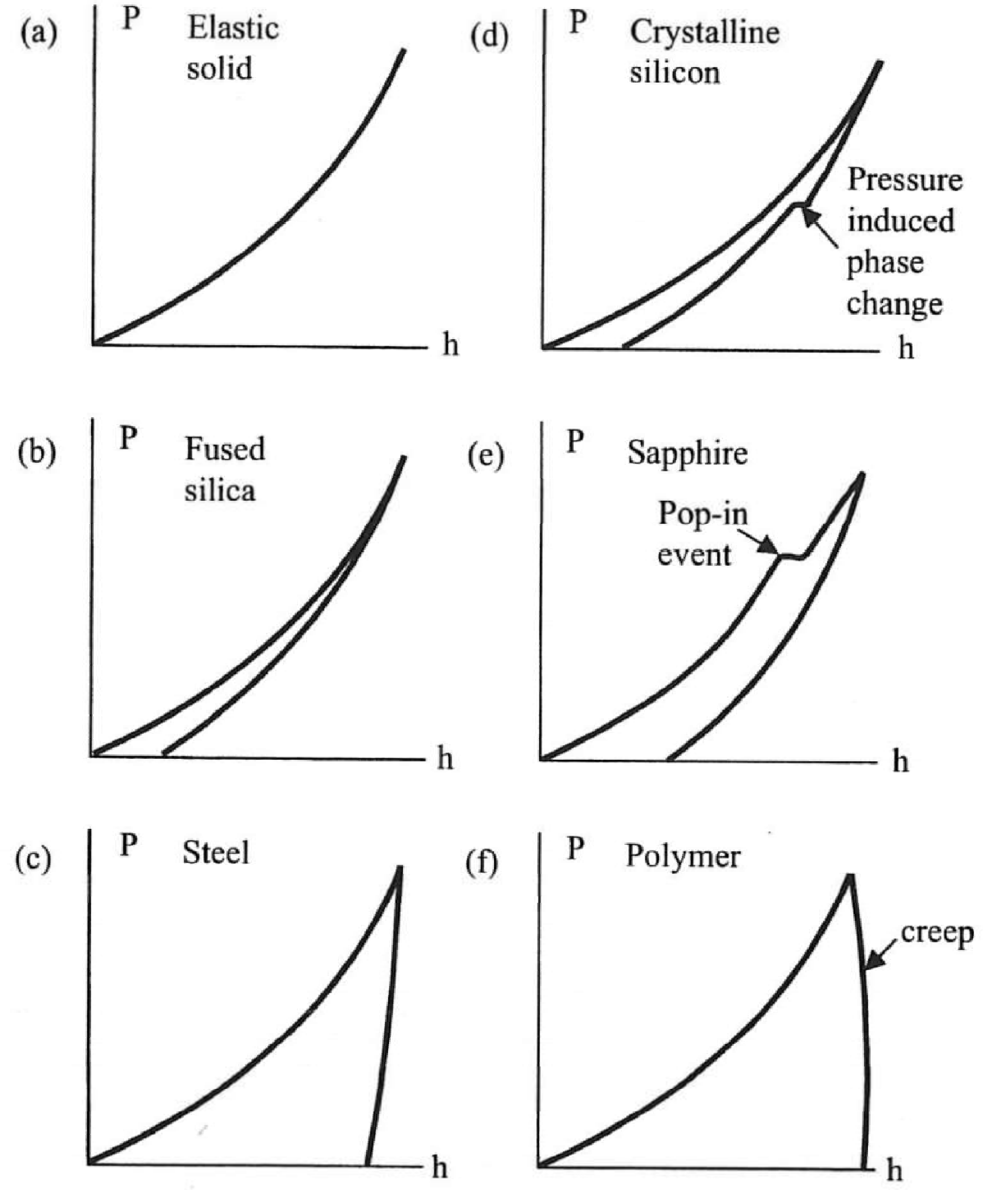

Fig. 2.6 Schematic examples of load-displacement curves for different material responses and properties. (a) Elastic solid, (b) brittle solid, (c) ductile solid, (d) crystalline solid, (e) brittle solid with cracking during loading, and (f) polymer exhibiting creep.

2.5 Experimental Techniques

Despite the mature evolution of nanoindentation test instruments, the process of undertaking such a test requires considerable experimental skill and resources. Such tests are extremely sensitive to thermal expansion from temperature changes and mechanical vibration during testing. It is necessary to ensure that the specimen and the instrument are in thermal equilibrium. For example, han-