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Indentation size effect: reality or artefact?

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The purpose of this investigation was to study the load dependence of the microhardness, typically in the range 5–500 gf. This well known phenomena is called the indentation size effect (ISE) and was investigated for two sets of specimens: titanium and aluminium alloys. Variation of the hardness with applied load was first compared with various existing models and the surface profile, near the indent, was measured by confocal microscopy. The formation of pile-ups near the indentation print led to the correction of the indent diagonal which is found to fit well with our experimental data as well as with other results in the literature. For the materials investigated, the ISE effect is an artefact, i.e. the variation of hardness with the applied load is only a consequence of the variation of the contact surface between the specimen and the indenter.

1. Introduction

Indentation hardness testing is probably the most simple technique used to provide the mechanical characteristics of a material, and among the large range of existing methods, the Vickers test is the one in most widespread use.

The Vickers diamond pyramid hardness number (DPHN) is defined as the ratio of the applied load on the pyramidal contact area of the indentation:

$$\text{DPHN} = \alpha P/d^2 \quad (1)$$

with the geometry of the indenter (square-based diamond pyramid of face angle 136°):

$$\alpha = 2\sin 68^\circ = 1.8544$$

where P is the applied load in kgf (1 kgf = 9.806 N) and d is the indentation diagonal in mm. The units (kgf mm^{-2}) are generally omitted.

The indenter gives geometrically similar indentations, so that it follows that the hardness must be independent of the applied load and of the size of the indentation. This is true for applied loads generally greater than 5 kgf: the hardness is constant and is called the standard (macro) hardness or the bulk Vickers hardness value. In contrast, for applied loads less than 100 gf (microhardness range) it is experimentally well established that the apparent microhardness varies with applied load: in some cases it diminishes but more frequently it increases with decreasing load, and this effect is known as the "indentation size effect" (ISE) [1]. The results of load-variant hardness behaviour are represented in Fig. 1. For titanium alloy, the hardness decreases with increasing load according to the typical dependence between hardness and applied load. These observations imply that if hardness is used as a material selection criterion, it is clearly insufficient to quote a single hardness number.

Much research work has been performed to establish the source of such a variation and several possible explanations exist. The most common explanations found in the literature are experimental errors related to the smallness of the indentation (typically 1 to 10 μm).

(i) As the load is reduced, vibrations become increasingly important, the diagonal length increases and the apparent hardness decreases.

(ii) On the other hand, the surface of the specimen must be hardened by the polishing process, and increases the hardness.

(iii) The limit of resolution of the optical system is about ± 0.5 μm, thus the accuracy at the lowest loads is poor. Mott [2] suggested that the indentation appears smaller than its true size by approximately a constant amount. This effect becomes proportionately higher for small loads and raises the hardness when the load diminishes.

The second set of explanations are described by Bückle [3] as the apparent causes of error and are directly related to the intrinsic structural factors of the tested specimens.

(i) The elastic recovery of the indentation print when the load is removed is proportionately more marked for small indentations.

(ii) The volume indented is so small that it contained no dislocations so the hardness approaches the theoretical limit of the perfect crystal.

(iii) Work hardening during indentation.

(iv) Grain precipitates or impurities.

For a more complete review see Bückle [3] and Tabor [4, 5].

The ISE effect has been traditionally described through Equation 2, known as the power law or

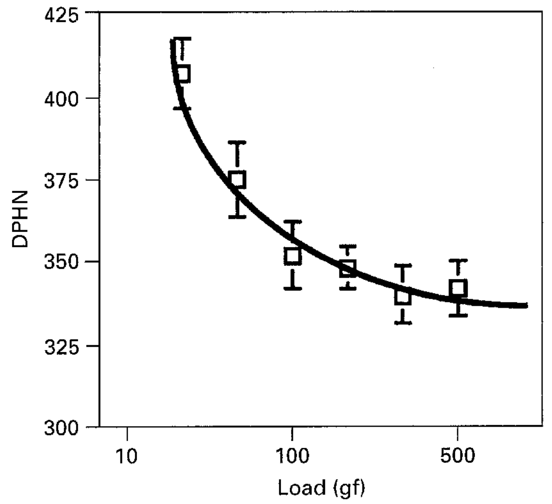

Figure 1 Plots of Vickers hardness as a function of applied load for titanium alloy TA6V.

Meyer's law (in spite of the fact that the Meyer index, n, must be restricted to the case in which a ball indenter is used):

$$P = Ad^n \quad (2)$$

where A and n are constants derived from the curve fit of the plot on a bilogarithmic scale.

Together with Equation 1 it is obvious that if n is lower than 2, the hardness rises when the load diminishes, when $n = 2$ (Kick's law) the hardness does not depend on the load, and for n greater than 2 the hardness diminishes with the applied load. The literature gives values ranging from 1.5 (work-hardened copper) to 4 for lead telluride [1].

2. Materials and experimental procedure

2.1. Materials

The samples studied were standard TA6V titanium alloy which is used in surgical applications and 2024-T3 aluminium alloy which is used in aeronautical applications.

2.2. Hardness measurements

Microhardness measurements were made with a Leitz Durimet microhardness tester at load levels ranging from 5 to 2000 gf and at a constant indentor dwell time of 15 s. The diagonals of the indentation print were measured by optical microscopy (magnification 400 ×) or by "scanning confocal microscopy" (SCM): $G = 6000$. In order to minimize error, the hardness measurements were systematically repeated up to ten times.

2.3. Profile measurements

Each experiment was conducted as follows: after indentation, the image of the indentation mark was obtained and the surface profile measured by SCM with a height precision less than 0.02 μm for all experimental loads. Computer programmes were written to calculate the height of the pile-ups near the indent, and 10 lines were observed for each indent.

3. Results and discussion

Equation 2 fails to explain the observed variations of microhardness with load, and several authors proposed different corrections to provide a satisfactory explanation of the ISE effect. If we consider that the ISE effect is an artefact, it is possible to obtain the true hardness, H_0, by correcting the applied load or by correcting the indentation length.

Hays and Kendall [6] assumed that as load P is applied to a sample, P would be partially affected by a smaller resistive pressure, and introduced the effective indentation load: $P - W$ where W is the material resistance to the initiation of plastic flow, e.g. the minimum applied load required to cause an indentation. With this correction, it follows that:

$$P - W = Kd^2 \quad (3)$$

where K is constant for a given material. Li and Bradt [7] discussed this method and showed that the load correction is too large to have a physical meaning. They proposed a second correction, called the "Proportional Specimen Resistance" (PSR) model. As for [6], the load is corrected but the correction is proportional to the indentation size: $P_r = a_1 d$. The effective indentation load and the indentation size are then related as:

$$P = a_1 d + a_2 d^2 \quad (4)$$

where a_1 is a coefficient related to the proportional resistance of the test specimen, and a_2 is constant. Li and Bradt noted that this relation is of the same general form that has been applied by Bernhardt [8] and by Fröhlich *et al.* [9] when the representation of the applied load to the indentation size effect is represented by a polynomial series in place of a Meyer relationship. The PSR model was developed by Li and Bradt on Knoop microhardness of single crystals of rutile and cassiterite and the coefficient, a_1, was attributed to the elastic resistance of the test specimen and the friction at the indenter facet–specimen interface. When the applied load is greater than a critical value, P_c, the hardness becomes load independent (see Fig. 1).

We now propose to combine Equation 4 with the standard Vickers hardness formulae (Equation 1) to obtain:

$$\text{DPHN} = 1854.4\,(a_2 + a_1/d) = H_0 + B/d \quad (5)$$

The equation we obtain is the same as the empirical equation established by [10–12]. We illustrate this diagrammatically in Fig. 2 where the representation of hardness against the reciprocal length of diagonal ($1/d$) will show a linear dependence. The relationship is linear, the intercept with the hardness ordinate represents the bulk hardness of the material and the slope, B, is the dependence of hardness with the applied load.

If the ISE effect is an artefact, the hardness (HV_c) must be constant, if the print diagonal is corrected (d_c). From Fig. 2, the mathematical correction becomes obvious:

$$HV/HV_c = d_c^2/d^2 = 1 + B/(dH_0)$$

or

$$d_c^2 = d^2 + (B/H_0)d \qquad (6)$$

where the subscript, c, refers to the mathematical correction. The dotted line in Fig. 2 represents the corrected hardness HV_c. The values of B, H_0 and the mathematical correction B/H_0 found for TA6V(2024-T3) (Fig. 2) are, respectively, as follows:

Absolute hardness, $H_0 = 322(78)$ kgf mm^{-2}
Slope, $B = 731(147)$ kgf mm^{-2}µm
Correction length, $B/H_0 = 2.27(1.88)$ µm

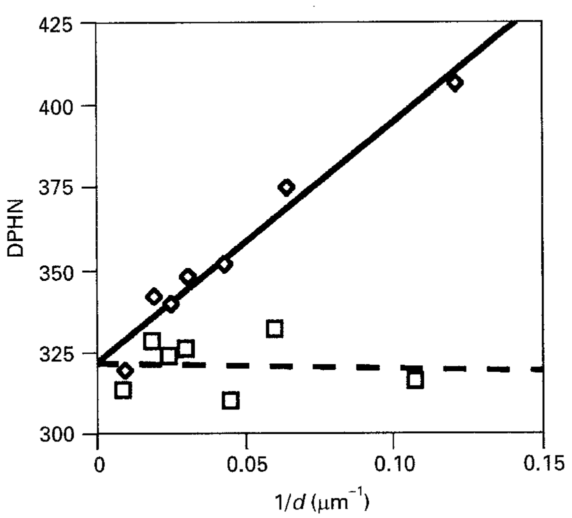

Figure 2 Variation of microhardness with the reciprocal length of the print diagonal for titanium alloy TA6V. The corrected hardness HV_c (Equation 6) is represented by the dotted line. Key: □ HV_c; ◇ HV. HV = 731.2 d^{-1} + 322.3.

For pyramidal indenters, and prior to any elastic recovery on unloading, all indentations should have the same shape. If elastic recovery changes the shape of the indentation, it is not believed to alter the length of the recorded diagonals. To understand how elastic recovery influences the microhardness, Farges and Degout [13] proposed in a recent paper a simple geometrical model where the deformation of the material around the indentation print is connected with the diagonal correction. Fig. 3 shows the geometrical model obtained by Farges and Degout where the dashed zone corresponds to the bulge formation. On the assumption that a significant amount of the load is supported by the bulge area, the indentation area must be corrected; then the corrected diagonal length, d', is related to the measured diagonal d:

$$d'^2 = d^2 + 4fd(2)^{1/2} \qquad (7)$$

It is to be noted that Equation 7 is of the same form as Equation 6 with $B/H_0 = 4f(2)^{(1/2)}$.

To test whether the idea of these authors is a reasonable approximation, we examined the indentations print by SCM (Lasertec) and scanning electron microscopy. Fig. 4a shows the impression of indentations made on 2024-T3 with a load of 200 gf. The ridge is clearly marked (see the enlargement in Fig. 4b), and the lip height, defined as the height of materials displaced along the edges of the indentation, was measured

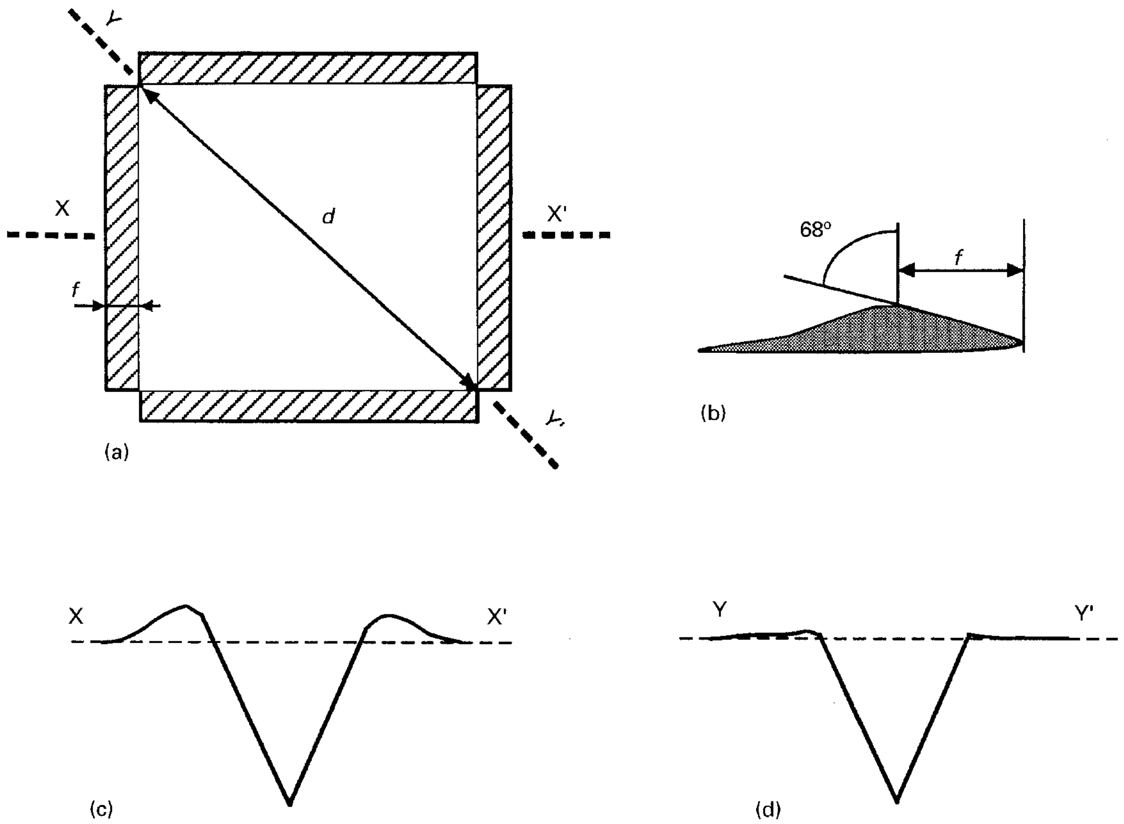

Figure 3 (a) Farges and Degout [13] geometrical model for the bulge formation. (b) Representation of the pile-up. (c) and (d) Profile in the X–X′ and the Y–Y′ directions.

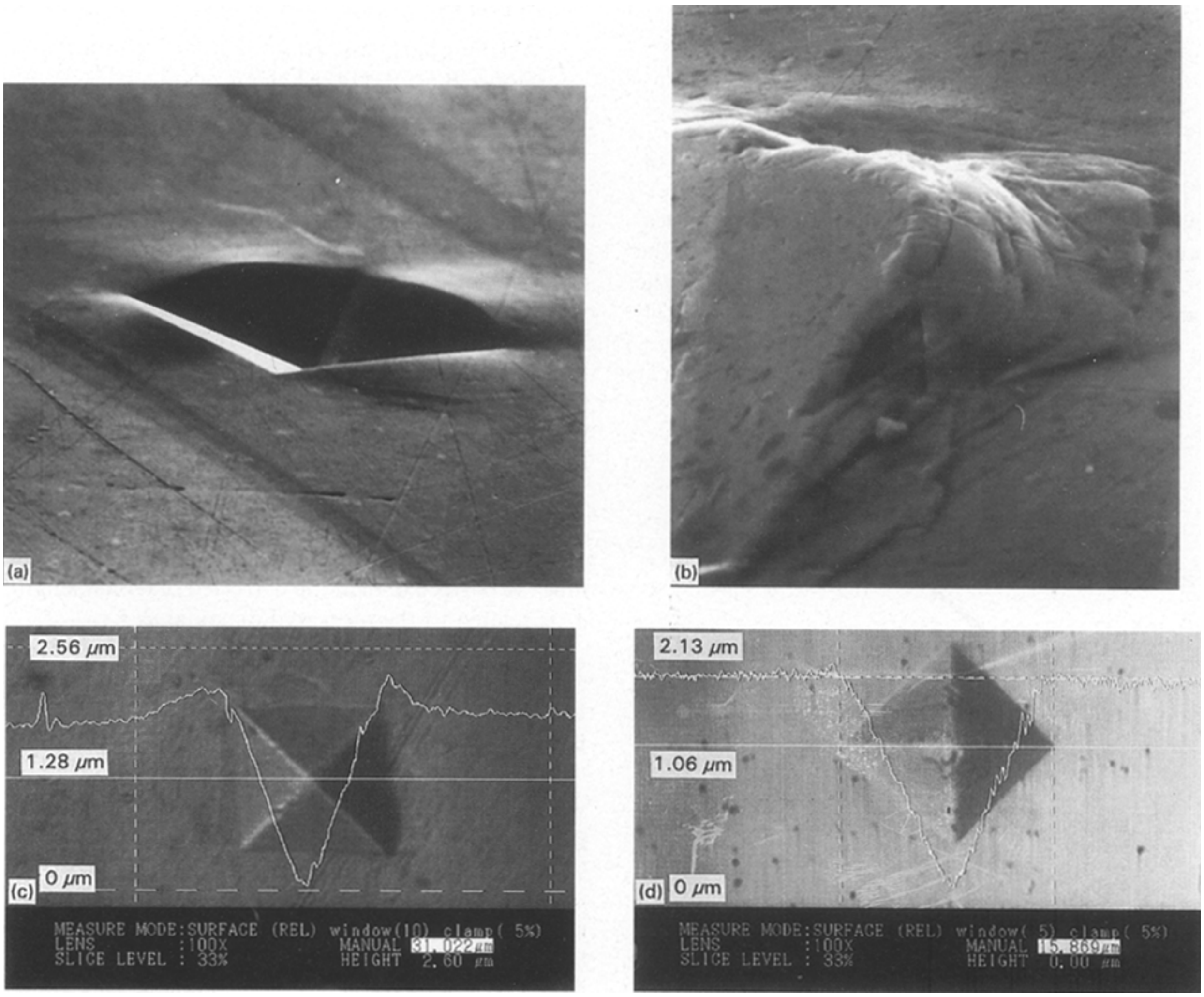

Figure 4 Surface morphology of the indentation print: (a) scanning electron microscopy ($P = 200$ gf, aluminium alloy); (b) enlargement of the bulge ($P = 1000$ gf, aluminium alloy); (c) scanning confocal microscopy showing the surface profile in the X–X′ direction ($P = 50$ gf, titanium alloy); (d) scanning confocal microscopy showing the surface profile in the Y–Y′ direction ($P = 50$ gf, titanium alloy).

by SCM. Fig. 4c and d shows typical images of the scanning line after indentation, respectively, in the X–X′ and the Y–Y′ directions.

Fig. 5 shows the correlation between the mathematical corrected diameter d_c and the geometrical corrected one, d', by SCM for aluminium and titanium alloys. The correlation is quite perfect in spite of the constant difference between ± 0.5 µm corresponding to the limit of resolution of the optical device. It can be noted that the ridge was observed at very low indentation loads [14].

In a recent work, (1 0 0), (1 1 0) and (1 1 1) surfaces of Mo and W single crystals were investigated by Stelmashenko *et al.* [15]. For loads below 10 gf each plane was found to exhibit a significantly different hardness value: the (1 0 0) is the hardest and the (1 1 1) the softest. When the applied load rises to 1 kgf, a continuous decrease in hardness was found for all planes, and the difference between them disappears. On the other hand, considerable pile-up formation was found for all indents. The maximum height of the pile-up reaches 17–20% of the depth of the indent for the (1 0 0) plane, 6–8% for (1 1 0) and 5–7% for the (1 1 1) surface. These results agree well with our observations: the absolute hardness of the different surfaces (1 0 0), (1 1 0) and (1 1 1) seems to be the same, but the variation of hardness with load depends on the amount of material in the pile-ups, and therefore on the crystallographic orientation.

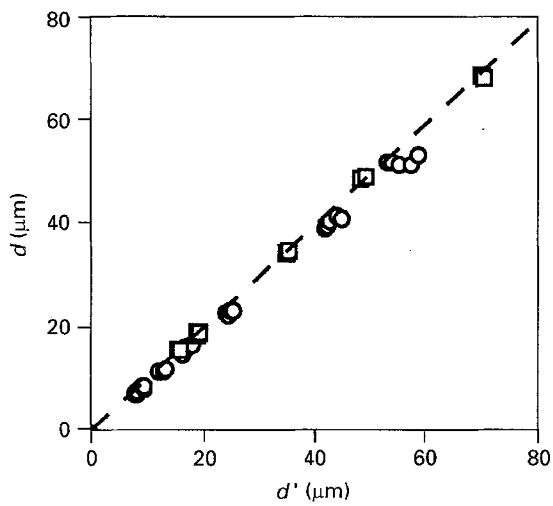

Figure 5 Correlation between the mathematical corrected diameter ($d_c^2 = d^2 + (B/H_0)d$) and the geometrical corrected one ($d'^2 = d^2 + 4fd(2)^{1/2}$). Key: □ 2024-T3, $d_c = 0.94 - 0.40d'$; ○ TA6V, $d_c = 0.98 + 0.13d'$.

4. Conclusion

To conclude, the ISE effect is an artefact for the material investigated (TA6V and 2024-T3). The true hardness is constant (independent of the applied load) but the measured hardness rises with decreasing load because the contact surface between the indenter and the specimen is greater than the surface measured by the diagonal length. If the diagonal is corrected for the bulge, we find DPHN $= H_0 =$ constant. It seems that the harder the material is, the greater is the ISE effect. For harder materials, the volume of material in the pile-up corresponds to the volume indented and, for softer ones, the pile-up decreases as the recovery and elastic compressive stress increases [15]. Two values are needed to define the hardness of a material: the true hardness, H_0, and the dependency of hardness with load which is characterized by a_1 (Equation 4) or the slope B (Equation 5) and related to the lip height. More work is needed to relate the coefficient B (or the pile-up height) to the physical properties of the materials investigated.

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