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Examination of reported size effects in ultra-micro-indentation testing

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The "indentation size effect" described in some reports of ultra-micro-indentation hardness tests has been examined to elucidate its cause. The magnitude of the effect was found to be associated with the strain-hardening propensity of the material tested, and thus similar to that observed in conventional tests with greater force, but the ranges of hardness variation were discontinuous. The relatively small size effect in ultra-micro-indentation testing seems to imply very low effective friction between indenter and specimen: a condition which could be due to 'dither' in the closed-loop servo control of ultra-micro-indentation.

1. Introduction

An "indentation size effect" – most often as increasing apparent hardness for smaller indentation – has long been known from "microindentation" and even "low-load" testing (1–50 or 20–1000 g load ranges). The modern generation of "ultra-micro-hardness" ("nano-indentation") test machines operate in a much lower load range, down to 0.1 mN ($\simeq$ 10 mg), and may, therefore, be expected to exhibit a pronounced indentation size effect extrapolated from the higher load ranges. However, the range of hardness variation reported is often similar to that observed in low-load or microindentation testing. This fact poses questions about the nature of the effect and also offers a basis for further investigation.

Ultra-micro-indentation test apparatus exerts force, with closed-loop servo control, through the medium of a calibrated spring. (Force is therefore usually defined as such, rather than in terms of the mass applied by a dead-weight loading system.) The very fine resolution of force by ultra-micro-indentation test apparatus is a major achievement which makes the testing method practicable. Displacement of the indenter is measured to ascertain depth of penetration. The provision of a precisely shaped, finely pointed, indenter is usually addressed by adoption of a triangle based pyramid (Berkovitch) indenter. Careful calibration is necessary to define the detection of first contact and to account for imperfection of tip shape.

2. Possible causes of an indentation size effect

Initial elastic-only resistance, which is characterized by a sharp fall in hardness above a very small indentation size, has been identified in ultra-micro-indentation tests of non-metals [1, 2]. Such behaviour is consistent with a requirement for a critical strain energy to trigger permanent deformation or, more probably, cracking. The behaviour is less likely in indentation of metals.

An early explanation for the size effect commonly observed in microindentation testing was based on underestimation of the indent size due to inadequate optical resolution or elastic recovery on removal of the indenter. Later opinion seems to be that this explanation has proved to be insupportable for low-load, and probably for micro-indentation, testing with a pyramidal indenter [3, 4]. The depth-sensing technique of ultra-micro indentation testing obviates these problems but introduces its own uncertainties in accounting for the compliance of the test system and in detecting initial contact.

A zero error in indenter displacement would generate an apparent indentation size effect in depth-sensing tests. This is comparable with the problem of optical resolution in microindentation testing, and may be expected to produce a similar variation of apparent hardness. A blunt or mis-shapen indenter would also introduce zero error in the effective displacement together with a variation of indentation geometry; so "the size effect" should be demonstrably different. An unequivocal indication of a blunt indenter could be sought in deviation of the force–penetration relationship near the origin.

A zero error in force measurement would also generate an apparent indentation size effect; but it is noteworthy that the effects would be expected to be opposite for dead-weight or for spring-suspension machines. (In the former cases, less weight would be applied while in the latter, less force, presumably, would be detected.)

An ever-present potential cause of an apparent size effect is inhomogeneity of the specimen, perhaps due to a sub-surface gradient of chemistry or of microstructure, or to surface hardening in polishing.

0022–2461

Friction has been shown to be responsible for a marked indentation size effect in low-load testing of some metals and the magnitude of the effect was associated with strain hardening [5, 6]. This intrinsic form of the size effect has been related to the special deformation conditions of a "plastic hinge" at the perimeter of the indentation [7]. An alternative explanation has been offered by Li *et al.* [8] in terms of their "proportional resistance" model. The important difference between these explanations is that the "plastic hinge" model assumes dominance of the boundary conditions for small indentations, whereas the other model assumes geometric similarity with varying importance of interface friction.

3. Case studies

In a recent discussion of displacement-sensing indentation experiments [9], Oliver and Pharr presented results for tests on a variety of materials. Of these, quartz and aluminium are selected for examination, as they represent a wide difference in expected deformation behaviour. Bearing in mind the major technical difficulties referred to above, the primary objective in this investigation was to verify the force and penetration calibrations. To this end, data were acquired by measurement of co-ordinates for several points in the traces of the "load–displacement" relationships in their Figs 4 and 5.

Assuming, as a working hypothesis, that there is no variation of hardness with indentation size, d, (or load, L), the fundamental (traditional) relationship

$$H = aL/d^2 \qquad (1)$$

leads, for force, F, and depth of indentation, P, to

$$H = bF/P^2 \qquad (2)$$

if the deformation, including elastic deformation of the indenter, is always geometrically similar. Therefore the derived linear relationship

$$P = cF^{1/2} \qquad (3)$$

is a good basis on which to test the calibrations of the instrument, because zero error and non-linearity become immediately obvious (except when the errors in force and penetration are exactly compensating).

For quartz, this last relationship, shown in Fig. 1, appeared to be exactly linear but noticeably offset from the origin. The linear correlation coefficient was determined as 1.000 and the offset error in penetration as ~ 11 nm. This disparity is admittedly very small but it proves to be significant. Calculation of hardness values with or without correcting for the apparent error produced the reported indentation size effect or virtually no size effect, as shown in Fig. 2. Even though unavoidable error in derivation of the data for smaller indentations leads to some uncertainty in the corresponding calculated hardness values, the difference between the relationships of hardness to penetration is striking.

For aluminium, the chosen test relationship was again precisely monotonic but non-linear; as shown in Fig. 3. The correlation coefficient for the second-order

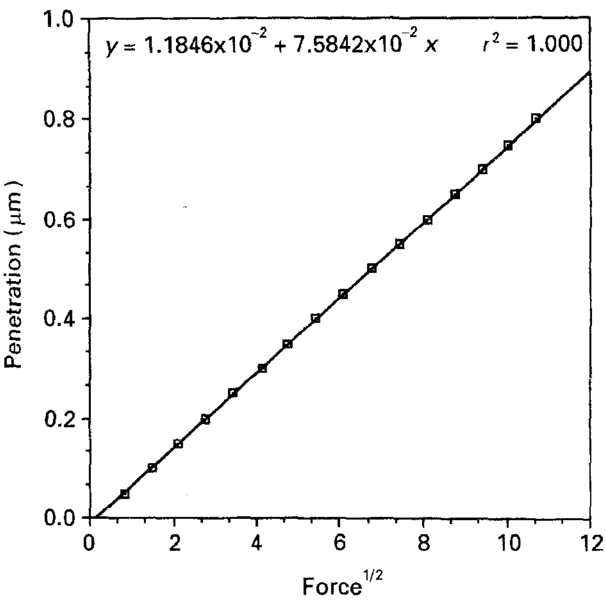

Figure 1 Force–penetration calibration for quartz; data from [9].

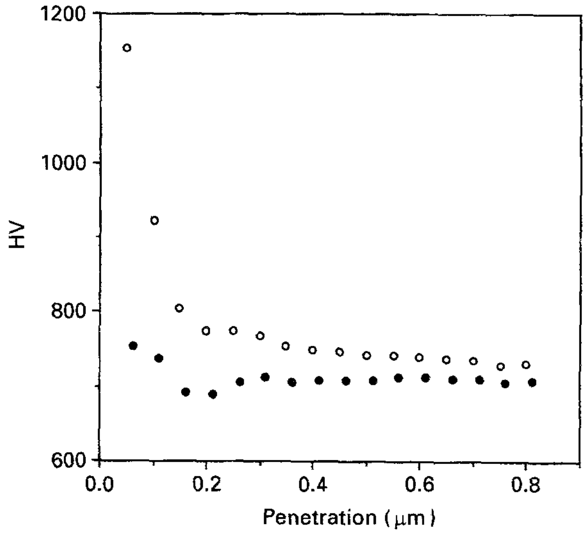

Figure 2 Hardness versus penetration for quartz; data from [9]. (○) Original, (●) corrected.

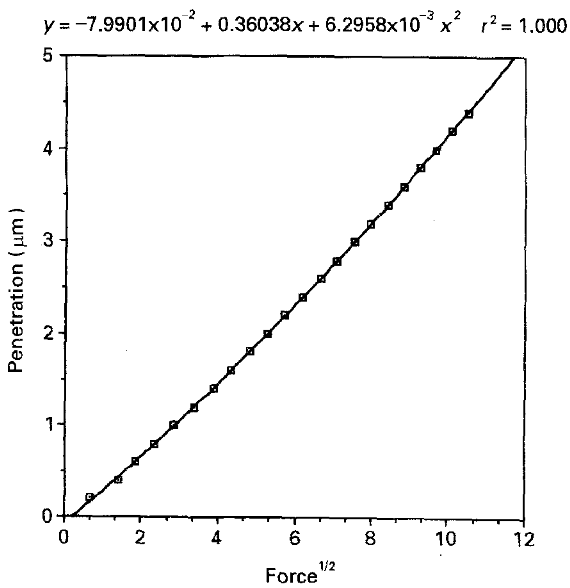

Figure 3 Force–penetration calibration for aluminium; data from [9].

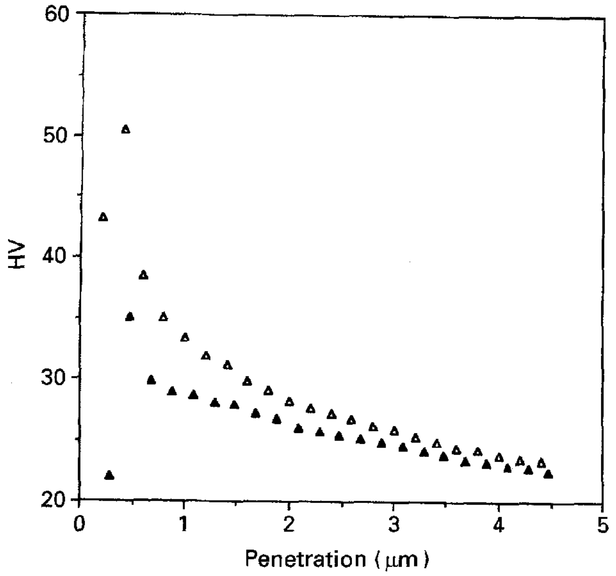

Figure 4 Hardness versus penetration for aluminium; data from [9]. (△) Original, (▲) corrected.

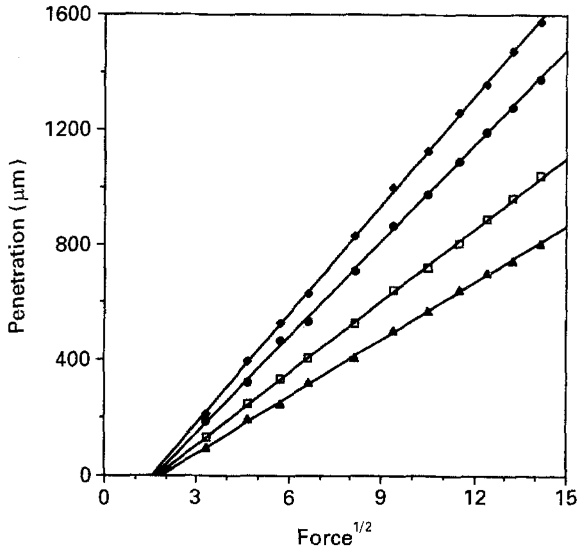

Figure 5 Force–penetration calibration for four steels; data from [10]. Fig. 4. Steel (▲) 1, (□) 2, (●) 3, (◆) 4.

polynomial regression equation is 1.000. Again there is an offset in the penetration calibration, amounting in this case to ~ 80 nm. The calculated hardness values show a significant diminution of the size effect when this offset error is taken into account, but not constant hardness, (see Fig. 4). The minimum hardness, being very close to the minimum to be expected from *macro*-indentation testing, is particularly interesting.

For comparison with the aluminium, data for some steels tested with a similar instrument was extracted from a recent exposition of ultra-micro-indentation tests by Bell *et al.* [10]. These four steels had been heat treated to provide different levels of hardness. Once again, verification of the calibrations produced precise linear relationships, converging fairly closely at a common initial force (see Fig. 5). The worst correlation coefficient was 0.999 and the offset errors in penetration calibration ranged from 121–200 nm. Taking these offsets into account almost eliminated the

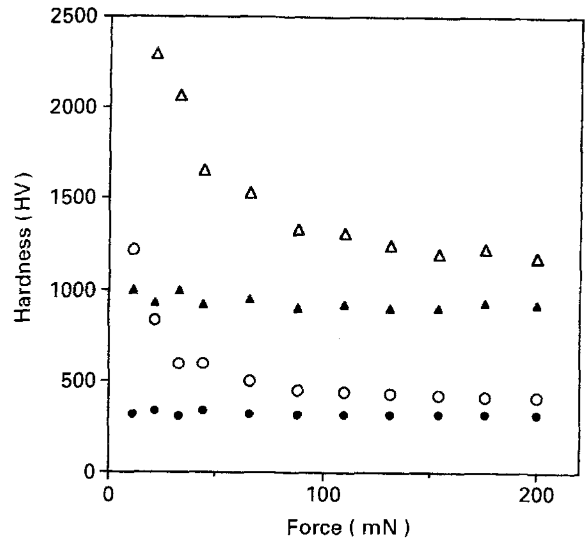

Figure 6 Hardness variation for steels 1 and 3 in Fig. 5. (△) 1, old; (▲) 1, new; (○) 3, old; (●) 3, new.

original marked indentation size effect. Data for two of these steels, selected for clarity of illustration, are shown in Fig. 6. Against expectation (from a presumption of a probable strain-hardening effect), a slight residual indentation size effect is more apparent for the hardest material; but this could arise from slight error in the data.

4. Discussion

It seems clear from the smooth monotonic force–penetration relationships, the derived forms of which are quite linear for quartz and the steels, and from the consistent near-nullification of the indentation size effect, that the data obtained by scrutiny of small-scale graphs is accurate enough to support the subsequent analysis. Even less variation in the relationships could have been attained by statistical smoothing but, in the interests of objectivity, this was not done.

Because the ultra-micro-indentation data originated from measurements made under load, there is no question that the indicated indent size is affected by elastic recovery; and, indeed, elastic compliance of the indenter and support system might be expected to produce the opposite effect in depth-sensing tests: larger apparent indentation. Furthermore, the supposed measurement 'error' in conventional indentation testing with greater load, which has been attributed to elastic recovery [11], is identified by the same analysis, based on Equation 3, used above to recalibrate penetration under load. The similar observations from tests attended by opposite elastic effects are persuasive confirmation that the size effect does not arise simply from unaccounted elastic behaviour.

The fact that the size effect can be reduced to negligible proportions by recalibration of the test data indicates the importance of a small offset but not its cause. Interpretation of the initial behaviour as a calibration error or a genuine size effect requires other knowledge, or an act of faith. For example, Frischat [1], following Froelich *et al.* [12], focused on a constant hardness regime; whereas Mason *et al.* [2]

emphasized the variation of apparent hardness in comparable behaviour, in preference to Frischat's description.

The procedure for determining first contact in rapidly changing initial indentation behaviour affects, and should be affected by, the perceived behaviour. The quest for very fine resolution in the force signal is not enough, because the penetration at low force is very small too – so a zero error is important. Detailed knowledge of the procedure would be necessary to deduce the initial complex behaviour accurately. However, apart from the initial behaviour, a stable constant hardness may be inferred. This constancy is a special finding and akin to a material property: a concept that has found broad utility. Arguably this constant hardness should be identified as a matter of course. Comparison with this stable reference condition offers a basis for elucidating the nature of the indentation size effect.

Of the materials now considered, quartz – for which a marked change of behaviour (incidence of cracking) might be expected – shows the smallest offset (11 nm) from the linear calibrating relationship. Recalibration of the data for the metals reveals considerably larger offsets. The linear relationships for the steels intersect at a common force of ~ 2 mN, which is about ten times the threshold level for detecting contact and must therefore be judged to be coincidental. More significant is the observation that the magnitude of the offset decreases with greater hardness of the steels.

Comparing the data for the steels and for the aluminium, it is evident that the relatively small offset error for aluminium was determined by the choice of a polynomial description of the P versus $F^{1/2}$ relationship. It must be admitted that extrapolation of a linear relationship for the upper range of larger indent size would have indicated a much larger offset: at least 500 nm. Alternatively, a non-linear tail to the calibration below 10 mN for each steel might indicate smaller offsets similar to, or less than, that calculated for aluminium.

These observations may be rationalized on the basis that divergence from a linear P versus $F^{1/2}$ relationship increases according to the sequence of materials quartz, hard steel, less hard steel and soft aluminium. This sequence may be characterized by increasing propensity for plastic deformation and strain hardening. It seems reasonable to infer that the strength of the indentation size effect is governed by strain hardening. The inference is supported by a similar finding for other materials indented with a Vickers indenter and greater force [3, 7].

Association of the size effect with plastic behaviour establishes that accommodation of the initial indentation according to expectation of varying elastic response would be inappropriate: a polynomial description of the P versus $F^{1/2}$ relationship is misleading. The marked variation of hardness originally reported for the indentation tests examined masks the fact that a characteristic constant hardness and superimposed size effect can be identified. The revealed size effect is consistent with the "plastic hinge" model of a boundary effect proposed earlier [7].

Identification of a credible cause for the indentation size effect associated with these ultra-micro-indentation tests does not, in itself, solve all the patent problems. Major questions remaining are "why is the identifiable constant hardness (for aluminium at least) no greater than for low-load hardness testing?", and "why does the size effect for ultra-micro-indentation not match the extrapolation expected from larger indentations?"

Because a principal factor in the size effect in low-load testing of iron and of aluminium (*inter alia*) has been identified as friction [5, 6], it is reasonable to suppose that the minimal size effects in these ultra-micro-indentation tests could be a consequence of particularly low-friction conditions. The cause may then be sought in the conduct of ultra-micro-indentation testing; and the closed-loop servo control of indentation is an obvious candidate. 'Dither' in the operation, inadvertent or deliberately introduced to improve resolution, would undoubtedly reduce the effective friction – and reduce the apparent hardness, as vibration from an external source is known to do [13].

Diminution of the size effect at this very small scale seems to be contrary to the findings of Gane and Cox [14] who observed continuously increasing hardness with smaller indent size. However, their results relate to spherical indentation, unknown friction conditions and measurement in the relaxed state; so direct comparison would be difficult. System-induced low friction in ultra-micro-indentation testing might be difficult to prove; but it does offer a feasible explanation for the disparities with other tests.

5. Conclusion

Data extracted from published force–penetration graphs for ultra-micro-indentation hardness tests yielded smooth monotonic P versus $F^{1/2}$ relationships with small zero offsets. It seems likely that these offsets reflect the absence of values for initial indentation and, therefore, inability to recognize the original non-linear calibrations.

The linear P versus $F^{1/2}$ relationships identified for quartz and some heat-treated steels correspond to constant hardness values for these materials. An obviously non-linear P versus $F^{1/2}$ relationship for soft aluminium indicated diminished hardness variation compared with that originally reported. A consistent view of the P versus $F^{1/2}$ relationships would be that initial divergence from a linear relationship was larger for greater propensity for plastic deformation with strain hardening.

The indentation size effect, shown to be associated with strain hardening, could be due to surface hardening during polishing; but it is also consistent with the previously proposed "plastic hinge" model of the size effect observed in low-load testing [7].

The size effect reported for the aluminium appears similar in magnitude to that observed for comparable tests with larger load. However, the minimum hardness calculated corresponds to the plateau level found from *macro*-indentation testing. Therefore, there is actually a marked disparity between the size effects observed in low-load or in ultra-micro-indentation hardness tests: one does not follow from the other.

Diminished size effect in ultra-micro-indentation testing indicates good maintenance of geometric similarity to very small scale. This implies very accurate indenter shape, accurate measurements and also very low effective friction. It is suggested that 'dither' in the closed-loop servo control of indentation, might be responsible for the low friction.

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