JOURNAL OF MATERIALS SCIENCE 26 (1991) 6576–6580

Importance of load cell sensitivity in determination of the load dependence of hardness in recording microhardness tests

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The behaviour of microhardness under varying load was investigated with an apparatus which measured both load and diamond pyramid motion simultaneously. There have been several experiments with this type of apparatus, which are designed to measure the hardness under load of a material. This type of measurement eliminates the effect of elastic recovery after the diamond is removed from the sample. Two types of load-independent hardness have been proposed on the basis of studies performed on this type of apparatus. The first follows the theory of Tate stating that elastic recovery is responsible for the load dependence of hardness. The second, proposed by Froelich *et al.* states that the load dependence of hardness is due to surface forces. This investigation used an apparatus similar to that of Froehlich *et al.* The results indicated that the load-independent hardness of Froehlich *et al.* was an experimental artifice caused by late detection of the surface, leading to underestimation of the penetration and overestimation of the hardness. Hardness measured under load using the apparatus in the present project was found to be load dependent.

1. Introduction

Measurement of hardness using a diamond pyramid has long been a practical quality-control tool for industry. In diamond pyramid hardness, a diamond indentor is impressed on a surface at a known load for a known period of time. The area of the identation remaining, after removal of the diamond is calculated from the remaining impression width. The applied load divided by area of the remaining indentation is defined as the hardness. Accurate measurements of the hardness of brittle materials have been difficult to make, because this type of hardness is load dependent [1]. In general, measurements show that the hardness of brittle materials tends to increase with decreasing load [1]. This presents a problem to investigators, because in the load-dependent region, making comparisons between materials or treatments is difficult. There are also theoretical difficulties. Because the diamond indentor is a pyramid, the shape of an indentation should not vary with load. Because the shape of the indentation does not vary with the load, neither should the hardness.

The classical explanation of the load dependence of hardness is based on elastic recovery [2]. This is something of a misnomer because, although the explanation relies on elastic recovery, it is not the bulk elastic recovery which is important but rather the elastic recovery of the ends of the diagonals of the indentation. The argument is that the geometrical similarity of the main indentation does not apply at the ends of the diagonals. The ends of the diagonals retreat a given amount regardless of the load, and the proportion of this retreat increases as the load decreases, increasing the measured hardness.

The equation used to correct for elastic recovery is

$$H = C\frac{\text{load}}{(D + D_e)^2} \quad (1)$$

where D is the measured diagonal, D_e is the elastic recovery, and C is a constant which depends on the geometry of the diamond (1.864 for a Vickers diamond). This explanation has long been considered controversial. Mott [1] attacked it on both empirical and theoretical points of view. He argued that geometrical similarity should extend to the ends of the diagonals.

Later work by Kranich and Scholze [3], however, revived the thesis of elastic recovery. They used a microscope to determine the hardness while the diamond was still under load, eliminating the effect of elastic recovery. Their results indicated that the hardness was constant under load for a wide range of loads. The hardness of glasses during loading was not found to vary with time under load or with atmospheric conditions. They proposed that hardness under load should be considered a property of the material. Later work by Hennicke and Vaupel [4] with a Vickers indentor on a similar apparatus found that hardness was load independent for glasses above a critical load but load dependent under that load. The critical load was approximately 0.8 N for silica glass [4].

Bartinev *et al.* [5] ascribed the load dependence of hardness to kinetic effects. Long-term loading with a

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Vickers diamond led to a constant decrease in the hardness with time at all loads. For periods of time longer than 1 day, a constant hardness was obtained for loads less than 5.0 N. Gunasekera and Holloway also found a time dependence in hardness under load for Vickers diamonds [6].

Froehlich *et al.* [7] devised a hardness machine which allowed for continuous determination of hardness during loading. Their instrument measured the penetration and the load electronically, and they found that if the hardness was calculated conventionally (load/penetration^2), it decreased sharply with increasing load. Their data showed evidence of a different kind of load-independent hardness which, according to them, was applicable to all materials. Fig. 1 is typical of their results although the data are from the present study. Fig. 1 is a plot of load divided by penetration versus penetration. The data were obtained using a glass slide. The features are typical of Froelich *et al.*'s data for all materials. Froehlich *et al.* analysed the data using a truncated power series. They expanded load in terms of penetration depth, as follows:

$$L/P \;=\; C_0 + C_1 P \qquad (2)$$

where P is the penetration, L is the load, C_0 and C_1 are constants.

The data were then plotted as load/penetration versus load. The slope of the resulting line was proportional to load/penetration^2, which is proportional to hardness. Froelich *et al.* claimed that the squared term represents the volume deformation of a material, while the linear term represents the surface energy of the deformation. Froelich *et al.* found this slope was constant, and all deviations from a constant slope could be explained by features such as work-hardened layers or soft hydrated surface layers. All of the materials they tested: soda glass, PVC plastic, brass and two ceramics, had a non-zero Y intercept, the value of which was considered to be a material property related to surface energy. The decrease in the hardness with decreasing load was then proposed to be due to an increase in the importance of the volume deformation term over the increase in surface energy. Later their work was repeated by Frischat [8] with similar results. The purpose of the present investigation was to repeat the work of Froelich *et al.* using a similar apparatus.

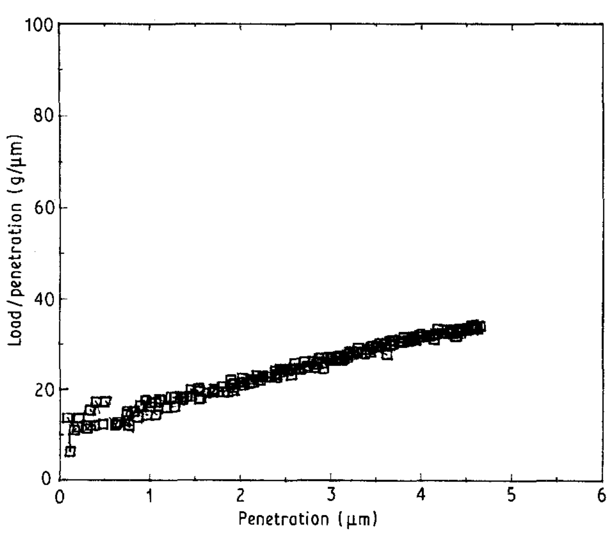

Figure 1 A load-independent hardness graph typical of Froelich *et al.* The slope of the line is proportional to hardness. These data were collected by measuring the penetration from the point at which the load was 0.2 g, thus simulating their apparatus. (——) Theoretical elastic deformation of a plane surface by a punch calculated from the equation of Sneddon [9].

2. Procedure

The instrument used for this investigation is drawn in Fig. 2. An inductive displacement transducer (LVDT) is used to measure displacement, and a 20 N strain-gauge load cell is used to measure the load. Both Knoop and Vickers diamonds may be used, but the Vickers diamond is preferred, because it is less sensitive to alignment errors. The load is supplied by the loading arm. The capstand nut lowers the arm into the sample. Loading rates are variable and less than 1 μm s^{-1}. The load cell also deforms, so the deformation rate varies during loading. The microscope slides (ISI-1704) used in this investigation were tested in the as-received state. The material were tested in air. The samples were clamped to the load table by spring-steel clamps which supplied a force of more than 100 N.

The instrument was interfaced to a computer for data collection. Locating the surface of the sample is the most difficult part of the measurement. The initial penetration of the sample was detected by a statistical programme which worked as follows. The voltage output of the strain-gauge load cell was measured by a 16-bit A-to-D converter. The sensitivity of the load cell was then calculated using the random spread of measured loads when the part was unloaded. During measurement, 50 data points were collected at 0.02 s intervals. The last five points of each of these data sets were tested to determine if the diamond had contacted the sample. If the diamond had not touched the sample the load cell zero was reset. By resetting the load cell zero every second, medium-term drifts in the load-cell outputs could be accounted for. Once contact with the sample was detected, the last 100 points before contact were saved. The initial position

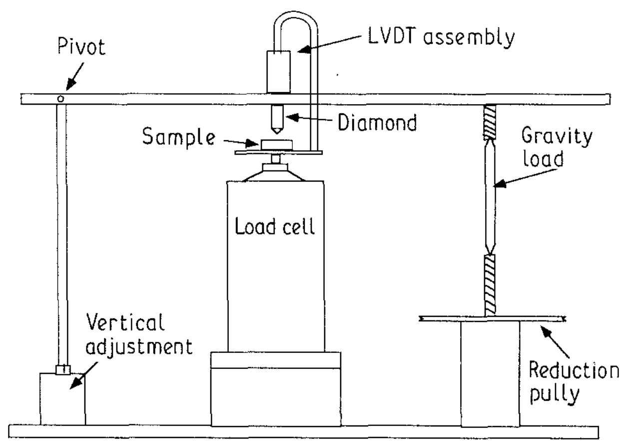

Figure 2 Drawing of the testing instrument. The diamond is loaded by gravity to a total weight of 600 g, at a rate of less than 1 μm s^{-1}.

of penetration was determined by averaging five points. When the measured load of five points was more than ten standard deviations of the short-term random load cell noise, from the pre-set zero load, the diamond was considered to have contacted the sample. (Ten standard deviations was used because there was a problem with low-frequency drifts, which were not counted in the random load cell noise.) The load cell was stable enough so that a load of 0.0002 N could be detected using this method. This is ten times more sensitive than was the case of the instrument designed by Frischat [8], who stated that his instrument had a sensitivity of 0.002 N or 0.2 g. Because both investigations used basically the same 20 N strain-gauge load cell, the statistical programme extended the sensitivity of the load cell by ten times. We also note that Frischat reported that he did not computerize his apparatus but measured his loads with an X–Y recorder. A load of 0.2 g on a full-scale graph of 10 cm for a load of 200 g will displace the pen 0.1 mm. This is barely detectable. The LVDT used in this study to measure penetration depth was accurate to 0.08 μm.

3. Results

Following the construction of the equipment described above, tests were undertaken to qualify the instrument. The results obtained by Froehlich and Frischat could not be reproduced on this instrument. The Y intercept was not zero and the hardness was not load independent.

Results for a soda–lime–silica glass are shown in Fig. 3, which follows the practice of Froehlich *et al.* of plotting the load/penetration versus penetration. The slope of the line decreases monotonically, and the Y intercept of the line is equal to zero within experimental error. This graph is typical of the brittle materials, and shows the hardness falling with increasing load. The hardnesses were of the same general value as that given by Froehlich *et al.* [7] and Frischat [8]. The hardness of the glass slide shown is 3.7 GPa at a 10 N load.

The data in Fig. 1 were also evaluated, using the methods of Frischat and Froehlich *et al.* to calculate the L2VHD, or load-independent hardness of Froelich *et al.*, of the glass slide. The L2VHD of the slide would be 2.1 GPa. Froehlich *et al.* reported hardnesses for the surface and the bulk separately, as they interpreted the data as having two slopes. L2VHDs of 1.6 and 2.5 GPa were reported for the surface and bulk, respectively. The intercepts were reported as 7.6 and 1.5 mN M^{-1} for the surface and bulk, respectively. Frischat did not report any Y-intercept for his glass.

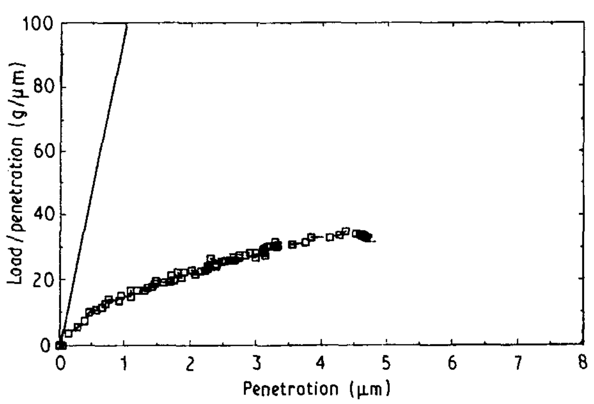

Figure 3 A Froelich-type plot of a soda–lime–silica glass tested in this investigation. The curve of the data passes through zero, and its slope decreases monotonically. The trigger load for this set of data was 0.02 g which is the limit of our apparatus.

4. Discussion

The disagreement between the results Froehlich *et al.* [7] and the results obtained using the new instrument requires an explanation. It is thought likely that the difference between the data produced in this investigation and that of Froehlich *et al.* was due to a more accurate measurement of the penetration by the instrument described above. Because the load on the diamond is very low during the initial penetration, accurate detection of the surface is difficult. If the surface is detected late, the penetration will be underestimated. This error is very difficult to eliminate, because the fraction of penetration before detection is proportional to the square root of the sensitivity of the load cell. A load cell which has a sensitivity of one-hundredth of the full-scale load will not detect the first tenth of penetration by a diamond pyramid. Froehlich *et al.* used a 100 N load cell for their work, which requires a sensitivity much less than 0.001 N to give a meaningful result. We do not know the actual sensitivity of their load cell, but it is unlikely a 100 N would be as sensitive as a 20 N cell which we used.

In the case of Frischat [8], it was assumed that the surface would be detected when the load was equal to the sensitivity of his load cell. Fig. 1, which reproduced Frischat's results, was obtained by collecting data using a trigger load of 0.02 N, which according to Frischat, is equal to the load-cell sensitivity used in his investigation. (The zero point of penetration, from which all depths are calculated is set equal to the position of the diamond at the trigger load.)

Although at first glance Froelich *et al.*'s explanation seems plausible, there are several problems with the data, even as originally presented. The first is that the hardness of a material measured under load in both Frischat's and Froelich *et al.*'s papers varies more than the hardness measured statically. Indeed, in Froelich *et al.*'s paper, a brass sample, which had a reasonably load-independent static hardness, had a hardness which was very load dependent when measured under load. It seems unlikely that surface forces would cease to exist upon elastic recovery. Also, in Froelich's *et al.*'s original paper, all of the hardnesses measured under load at loads less than 0.25 N were higher than the loads measured statically. This is most difficult to explain on the basis of elastic recovery. Another problem with the idea of a non-zero y-intercept due to surface forces is that an apparatus which measures hardness under load will measure both plastic deformation and elastic deformation.

Theoretically, it is clear that, even if the plastic hardness approaches infinity at low loads (a condition predicted by the theory of Froelich *et al.*), elastic deformation will become important. The equation given by Snedden [9] for the penetration of a circular cone into an elastic half space is

$$\text{load} = \frac{4v\cot(\alpha)}{\pi(1 - \nu)} P^2 \tag{3}$$

where v is larmor rigidity modulus $E/1 - \nu$, and α the half horizontal angle of the cone, 22.5° for a Vickers diamond, ν is Poisson's ratio. The deformation at 1.00 N load on a typical glass slide of 70 GPa elastic modulus [10] can be calculated as 0.9 μm. The elastic hardness is load independent and will plot on a Froelich plot as a straight line through zero. We have added that line to Fig. 1. If the elastic deformation is taken into account, there is no way that a graph of load/penetration versus load can have a non-zero Y intercept.

Using data taken from Frischat [8], the magnitude of the effect of the missed estimation of the surface can be shown. Fig. 4 is calculated from data for a chalcogenide glass $Se_{40}Ge_{40}As_{20}$. The uncorrected line is calculated from the intercept and slope data given in the article by Frischat. The corrected line is calculated by using the hardness of the sample at 2 N. This hardness was used to estimate the undetected penetration at a load of 0.002 N, which is the value given by Frishat for the sensitivity of his load cell. This undetected indentation is then added to the reported penetration, and replotted, giving the corrected line. It can be seen from the graph that this error is large enough to eliminate the Y intercept. A visual extrapolation of the corrected curve intercepts the X axis at a positive value. This is because the hardness measured under load, by Frischat, remains load dependent, even with the correction, due to a missed estimation of the surface position.

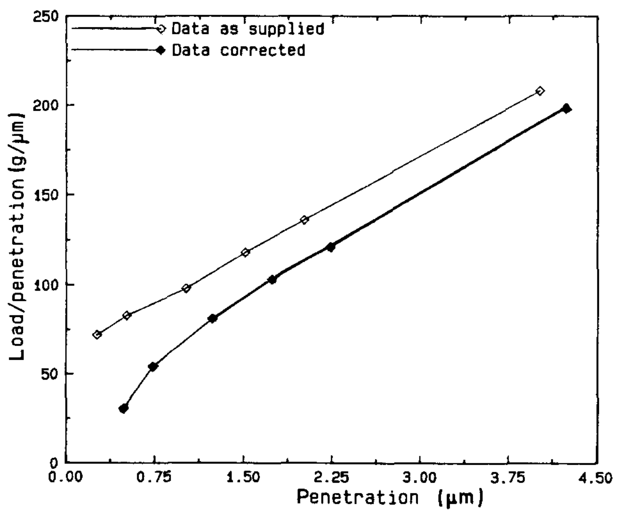

Figure 4 Frischat's data can be corrected for the late-penetration error. Data supplied in his paper [8] for $Se_4OGe_4OAs_2O$ were used to calculate the data as-supplied. The penetration at 0.2 g was calculated from the hardness at 200 g and added to correct the data. Even this sensitivity, of one-thousanth of the full-scale load, was large enough to remove the Y-intercept and remove the linearity of the graph.

TABLE I Penetration of brass by an indentor estimated by data from Froehlich *et al.* [7]

Load (N)	Penetration from remaining indentation (μm)	Penetration measured while indenting (μm)	Difference (μm)
0.10	1.76	0.97	0.79
0.50	3.93	2.75	1.18
1.00	5.56	4.39	1.17
1.50	6.82	5.36	1.19
2.00	7.97	6.71	1.16
			av. 1.10

Using data from Froehlich *et al.* [7], the difference between the penetration measured under load and the penetration measured after removing the diamond pyramid can be calculated. Brass was used for this calculation, because indentation diagonals in brass do not change length on the removal of the indentor [2], and the hardness of the brass used by Froehlich *et al.* in their paper was nearly constant. The hardness of brass measured conventionally was given as 122 kg mm^{-2} by Froehlich *et al.* The constants used for calculating the measured depth of penetration under load were also given in the paper. The penetration at any load could be calculated using these data. The penetrations we calculated during loading and after unloading using these data are given in Table I. It is seen that the penetration of the diamond measured by these two methods has a relatively constant difference of 1 μm. If material were extruded from beneath the indentation, this difference would increase proportionally with the penetration. The low reading at 10 g is due to a different function being used to calculate the hardness at that load. Froelich *et al.* reported different data for surface hardness. This relatively constant difference suggests that the difference occurred at the beginning of the penetration before the load cell detected the surface of the part being indented.

The discrepancy with the data of Kranich and Scholze is more difficult to explain. The hardness of all brittle materials measured by the machine used in the present study decreased with increasing load throughout the load range tested. Also, if the error due to late surface detection is subtracted from data supplied by Frischat, the hardness under load remains load dependent. We suggest that the difference is due to the dynamic nature of the test. During loading, the load is constantly increasing as the diamond penetrates the sample. Because of this constant increase, the hardness never reaches its equilibrium value.

5. Conclusions

The load-independent hardness of Froehlich *et al.* [7] is an experimental artifact caused by their use of a load cell which was not sensitive enough to detect the early penetration of the surface by an indentor. The hardness measured under load by our instrument was load dependent, and if we correct Frischat's hardness data for late penetration, it also is load dependent. Of course, only Frischat can make any final determination relating his data to load dependence.

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Received 27 September 1989
and accepted 29 October 1990