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PARTICULAR ASPECTS IN THE CALIBRATION AND APPLICATION DYNAMIC AND STATIC HARDNESS

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Abstract – The paper summarizes some results obtained at investigation of the main quantities influencing the uncertainty of measuring dynamic hardness. Some parameters concerning the force and time behaviour for full test cycle (loading and unloading) is discussed in terms of a systematic characterization of the dynamic set up used. The technique parallels the method for static indentation hardness determination and allows direct comparisons between dynamic and static hardness measurements.

Keywords: hardness, dynamic, calibration.

1. INTRODUCTON

Recent years have seen significant improvements in dynamic hardness equipment [1]. It is now possible to monitor, with high precision and accuracy, both the depth and the force measuring system during indentation experiments [2]. However, it is mostly in static loading conditions, and questions remain, including what properties can be measured using dynamic indentation techniques. Deviations which occur when the dynamic hardness values is converted into a static hardness values amounts to between ±5% and ±15% [3]. In a static hardness test, where a predetermined load is applied for 2–15 s, the rate of deformation is typically of the order of $10^{-2}s^{-1}$. The quasi – static approximation is expected to be valid for impact velocities much than sonic velocities in either the indenter or the specimen. Dynamic hardness values, H_d about twice the static values, H_s have been measured at loading durations as short as $=10^{-3}...10^{-5}$ s and the dynamic strain rate is at least 4 to 5 orders of magnitude greater than that is achieved in a static indentation test. Deformations during impact conditions involve strain of the order of 10^3s^{-1}. However unlike in a static hardness test where the load is directly measured (or known a priori), the dynamic methods warrant measurement of incident velocity *v*, of the indenter for determining the dynamic hardness H_d. To avoid systematic errors, inertial forces and damping forces must be taken into consideration in dynamic force measurements. This requires that the masses acting between the points of force application be known, as well as the stiffness and damping of the force transducer. Conversion inaccuracies result from the comparison of test values with test undertaken by different methods. These arise because there is no clearly defined physical correlation between different test methods because of the inherent inaccuracies in the methods being compared [4]. To understand the uncertainties in the calibration of dynamic hardness, it was necessary to evaluate the main factors contributing to the uncertainty in measurements made with static and dynamic hardness testers. These factors were identified and then, where possible, verified.

2. ANALYSIS

It is specified in ISO/DIS 14577 [5] that the indenter area function should be verified with either direct or indirect measurement. Mean contact pressure resisting dynamic indentation itself is always higher than that involved at the last stages of the impact (indentation – rebound phase), because the plastic deformation has completely come to the end. This dependence becomes very marked with the soft metals, where the pressure required to produce plastic deformation dynamically are very much greater.

Presumably, if the response of the material depends only on the maximum load and not on the loading rate, each characteristic of the indentation observed at equal radius will be the same for both static and dynamic loads. The extent of the observed differences will indicate the degree to which the response differs from the assumption. Although the morphology of the deformation beneath the contact is similar in static loading and impact, some differences in the extent of deformation were identified (Fig.1).

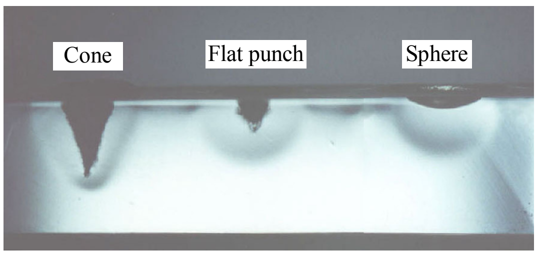

Fig.1 Plastic deformed zones for various indenters in the cross – section of polymethylmethacrylate (PMMA)

For sharp wedge or conical indenters, substantial upward flow is usually observed and since elastic strains are thus negligible compared to plastic strain, the specimen can be regarded as being rigid-plastic. It was presumed that variations in indenter geometry would alter greatly the energy to dynamic indent the specimen provided the same indentation mode applied [6]. However, non-faceted indenter shapes (e.g. hemispheres) create stress fields which are more amenable to analytical solution, avoid the problems associated with characterizing the exact geometry of the tip-end shape. Thus, a range of indenter geometries $\left(60^0 < 2\theta < 180^0\right)$ may be needed to provide compete dynamic and static hardness measurements. The angles smaller then 60^0 are not considered due to two factors: the small angle cone is seldom used in practice; the small angle may result in the significant strain around the indenter that could introduce errors in calculations. While the pressure distribution under the indenter is experimentally indeterminate, the results of this constrain manifest themselves in the form of changes in the contact area and plastic zone. We think that since the deformation is volume process and it takes energy to induce it, the energy related definition of the hardness is more descriptive in particular in the case microhardness measurement where the volume of the deformed material is very small. Energy approach relates energy dissipated in the sample to the volume of the indent after withdrawal of the indenter [7]. Therefore, analyses of the main quantities influencing the uncertainty of comparison between static and dynamic hardness measurements are of particular interest:

- measuring deviation of the force and displacement measuring system;
- estimation deviation of the plastic zones size and shape beneath indenter in static and dynamic indentation.

The dependence of hardness on indentation angle is explained using the plastic zone size with respect to the contact area. When varying the cone angles the maximum hardness is reached when the plastic zone is almost as large as the contact area [8]. For small semi cone angles less than 60^0, a discrepancy is found between the static and dynamic indentations. The complex dependence of hardness on indenter angle may be explained by the strain field u under the indenter. In experiments, the values of hardness were found to be erratic when semi cone angles were less than 60^0 because the rim of impression contained deep radial fissures. The “pile-up” of surface profiles around indenters could also cause errors in estimating contact area. The effect of friction might also be a factor. It has been shown that friction can play an important role in indentation experiments, especially for small semi cone angles [6].

The variation of imprint volume with applied load (energy) and indenter angle is shown in Fig.2. This graph is interesting for a number of reasons. At first, it shows that for all angles for static loading the imprint volume increases as the apex angles decreases. For dynamic loading the opposite was found to be the case – imprint volume decreased as the apex angles decreased. In trying to explain these effects it is worth remembering that in conventional hardness testing arises geometry effect, because the contact areas are not directly proportional to the depth of penetration of the indenter when different loads are applied. To ensure that indentations are made at equal indents radii the following conditions must apply:

$$\frac{mv^2}{2} / d^3 = idem \qquad (1)$$

where d is the diameter of the indentation, $mv^2/2$ is kinetic energy of striker.

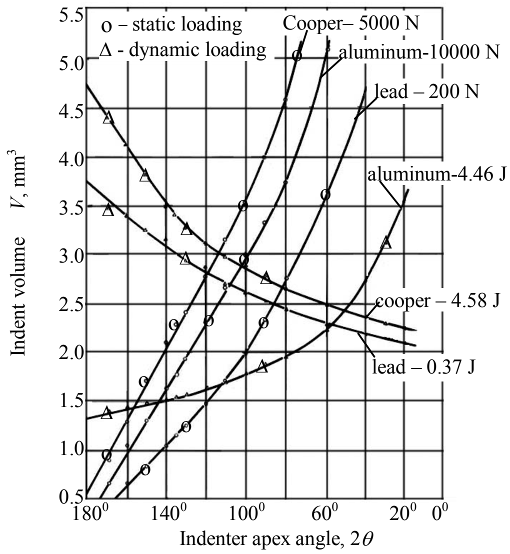

Fig. 2 Influence of apex angle on indent volume for static and dynamic indentation

A convenient experimental parameter for characterizing the indentation size is the peak load F applied to the indenter. The indentation volume (V_o) and load parameters are related by straight forward geometrical considerations, in combination with the hardness relation $H = F / A$, where A is the projected area of the indentation. This with $A = \left(3\sqrt{2}\,V_0 / cot\,\theta\right)^{2/3}$ for pyramidal or cone indenters for quasi – static loading the apparent hardness was evaluated using

$$H = 4F / \pi d^2 \qquad (2)$$

where H is the apparent hardness, F is the peak applied load, and d is the projected diameter of the indentation. An equivalent expression for the apparent hardness under conditions of impact loading is given by [3]

$$H_d = \frac{1}{2} m \left(v_i^2 - v_r^2\right) / V_r \qquad (3)$$

where H_d is the dynamic hardness, m is the mass of the indenter, v_i is the impact velocity, V_r is the volume of the permanent indentation. In the present study, an approximation of (2) was used which does not require the direct measurement of the actual indentation volume [6]:

$$H_d = \frac{1}{2} m \left(v_i^2 - \frac{3}{8} v_r^2\right) / V_a \qquad (4)$$

where the apparent indentation volume $\left(V_a\right)$ is determined by assuming that the radius of curvature of the indentation is equal to the radius of the indenter. Another means of evaluating the plastic response is through the application of Mayer's low:

$$F = ad^n \qquad (5)$$

where a and n are the constants, d is the indentation diameter. Tabor [3] was able to relate the experimentally derived quantity, n, with the work – hardening index, m, of the test materials and correlating this with the results from uniaxial tensile tests. This relationship of $m = n - 2$ can be applied only in the case of indentation by spherical indenters. For pyramidal or conical indenters the related indentation size effect (ISE) index is found by fitting experimental data to the (4). When $n = 2$, the hardness is independent of indentation size, when $n > 2$ there is "reverse" ISE. The peak impact force can be considered as an important measure of the indentation kinetic energy, if the usual errors that appear when the measured forces ore impulsive type ones, are restricted (Fig.3).

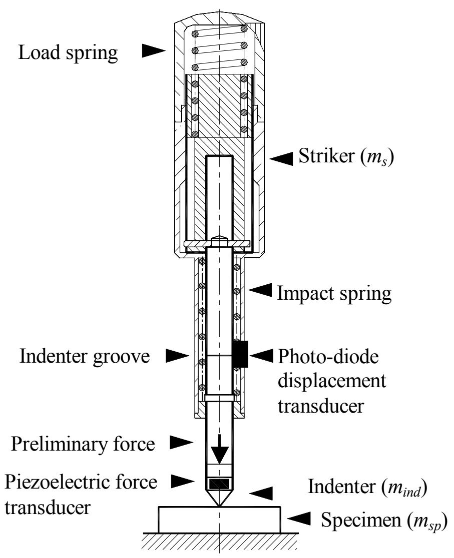

Fig. 3 Dynamic indentation tester: a schematic diagram of the main test frame and instrumentation, showing the placements of the sample, load and displacement transducers.

At dynamic rates, a piezoelectric load cell is used [6]. A pair of photo - diode transducers is used to measure the displacement. The resolution of the pair is ± 0.1 μm. Both the load and displacement transducers are directly coupled to the sample. This feature eliminates the need to correct for the compliance of the load frame. The signal from the piezoelectric transducer is processed by the charge amplifier and displayed on the memory oscilloscope. In the same time the driving signal of the photo-diode is also displayed on the same oscilloscope. The period between the rising front of the driving voltage and the rising front of the transducer signal represents the delay time for dynamic indentation force. To allow comparison between the kinetic energies at impact in the two types of the concept of the "equivalent impact energy" can be introduced. For a three mass system "striker – indenter – specimen" (Fig.3) impact problem this is defined as: equivalent impact energy E = energy impact to the striker up to the time when the masses m_s and m_{ind} have a common velocity, where m_s is the mass of the striker and m_{ind} is the mass of the indenter that is free to move.

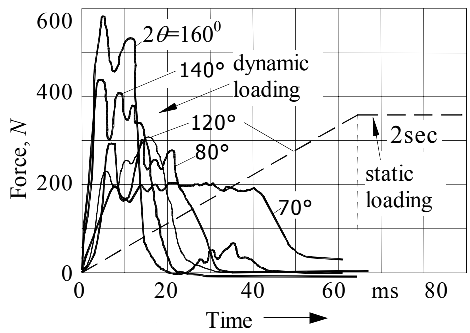

Fig. 4 Data recorded during the test force of dynamic and static indentation for various cone indenter apex angles, 2θ

According to the contact mechanics equations this requires that the masses acting between the increasing spring and the points of force application be known, as well as the stiffness and damping of the force transducer. This behaviour can be explained by the experiments described in Fig.5.

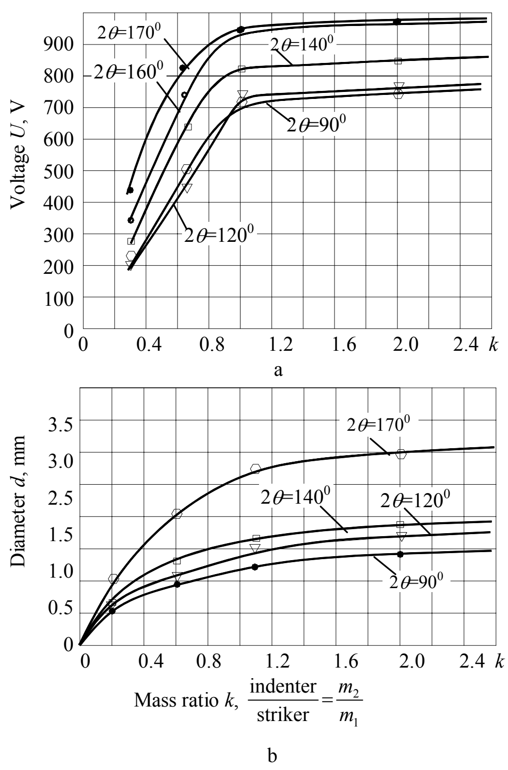

Fig. 5 Relation between voltages U (V) (a), indent diameter (b) and mass ratio indenter/striker (standard hardness block, HRC 22.1)

Although it is difficult to obtain an accuracy estimate of m_L, it is possible to use the "effective mass" concept. The equivalent impact energy is equal to the striker kinetic energy in a fixed load spring, and energy impact to the specimen is this value when the striker velocity is zero. If the mass of the specimen is ignored, then the equivalent impact energy is $E^* = \frac{m_s v_s^2}{2}\left(\frac{m_{ind}}{m_s + m_{ind}}\right)$. When we apply this concept to impact ion the specimens with various masses m_{ind} is replaced by a single lumped mass m_L (when $m_L > m_{ind}$).

Therefore for the giving constant values of the dynamic hardness for the testing specimen changes in kinetic energy of the impact could by not in changing of impact velocity of indenters and fall, but in changing mass of the impacting striker. This condition verifies correlation between dynamic and static hardness values. Different impulse load amplitudes and pulse shapes could be obtained by changing the mass of striker – indenter – specimen system, by varying apex angle of the indenter or by impact velocity. In the case of a dynamic excitation, the applied input force and the measured effective force are not equal.

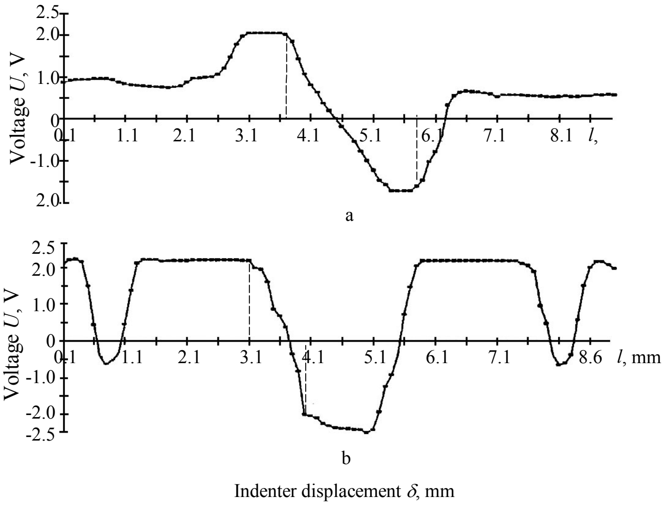

Fig. 6 Photo-diode calibration characteristics in relation of the form of groove: (a) stick out (1x2 mm); (b) rectangular (1x2 mm).

With fix positioning of the optical transducer at a distance of 2,0 mm from indenter groove and by using the subtraction circuit, the calibration characteristics have been recorded by controlled change of reflected curve (Fig.6). Calibration was carried out so that the determining of the most suitable value of output voltage U for a given suitable form of indenter groove will be achieved. For the purpose of studying the linearity of conversion of the indenter displacement into an electrical signal, a gauge such as presented in [8] has been designed. It may be noted (Fig.6) that curves have its minimum at x=4…6 and that the first steeper and falling part is less non-linear than the second rising parts of the curves. The advantage of the falling part is, apart from less non-linearity, also greater sensitivity of displacement conversion into an electrical signal.

Because the plastic deformation reduces the maximum load one would expect the load in the elastic-plastic case to be less than that in the elastic case, as is indicated above. A larger extent of elastic recovery in the depth of the residual contact impression and a smaller plastic zone size relative to the contact dimension are both associated with the higher ratio of hardness–to–modulus (H/E) in impact. Traditionally, nominally sharp pyramidal or conical indenters have been used for indentation experiments, primarily because they will create the large shear stresses necessary to cause plastic flow. The measurement accuracy of the instrument can be examined in two ways:

- mean measurement inaccuracy refers to deviations which occur when individual measurements are repeated using the same method. When measurements are taken with various impact devices of deviation of the mean value will not exceed approximately $\pm 3\%$.
- conversion inaccuracies result from the comparison of test values with tests undertaken by different methods. These arise because there is no clearly defined physical correlation between different test methods because of the inherent inaccuracies in the methods being compared

3. RESULTS AND DISCUSSION

Dynamic indentation experiments were carried out with different specimen materials in the velocity range 5-30 m/s. In Fig.7 it is shown experimental contact force-time $F = f(t)$ and displacement – time $\delta = f(t)$ curves of dynamic indentation for various indenters' apex angles have been obtained from oscilloscope records of the kind.

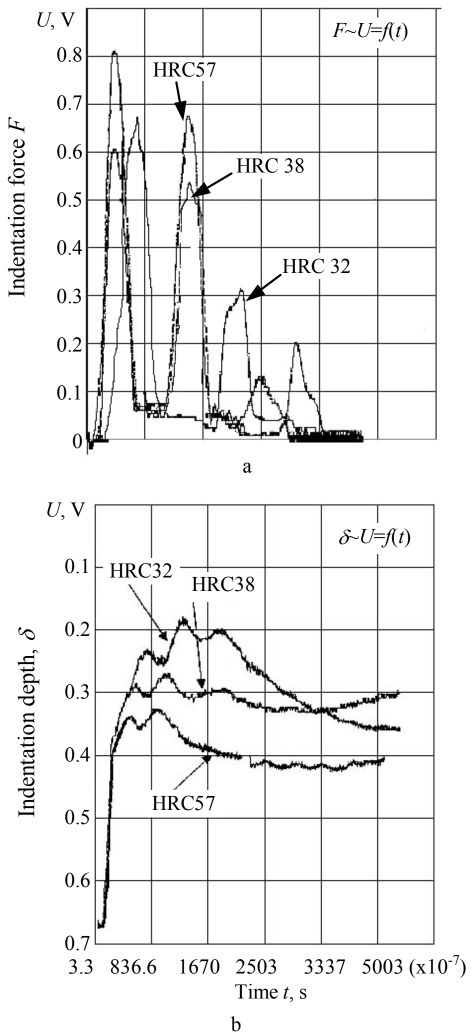

Fig. 7 Diagrams of the force $F \sim U = f(t)$ (a) and displacement $\delta \sim U = f(t)$ (b) for various hardness standard blocks

In addition to the indirect verification using reference blocks it is possible to realize the calibration by direct verification of the test force in the dynamic and static loading. Finding difference between mean hardness values obtained by the standardized tester and that obtained by a testing machine carried out the verification [4]. Yet another condition presumed to be valid method of comparison dynamic and static hardness is that the inertial resistance provided by the indented material (i.e. resistance to displacement of the specimen from the path of the penetrating indenter) should be negligible when compared to its resistance to plastic deformation (which equals H_d). The inertial resistance offered by the specimen material equals $0.5\rho v_{av}^2$ per unit volume of the specimen material displaced, where ρ is the density of the specimen material and v_{av} is the average velocity of the indenting cone. Thus the critical velocity is obtained as 120 m/s [4]. As the maintenance of constant impact energy can only result from pre-determined values for spring constant and spring path, it follows that the effective kinetic energy acting at the point of impact is itself not constant. If the impact velocity v_i is measured to determine the kinetic energy, than it will still contain both error and disturbance effects unlike the rebound velocity v_r .

To analyse the variations in the accumulated plastic strains as a function of depth beneath the indentation in a specimen material and to gain an estimate of the differences in plastic zone size and shape with respect to the loading rate, microstructural analyses of the indentation zones were performed on the materials for which stress-strain curves were provided. Similar to static testing, the indenter is placed in contact with the specimen before the striker is impacted on to the incident bar – indenter. By choosing a long incident bar, normal impact is ensured in the experiment. The indentations imprint size on the specimen measured after and depth of the indentation – before and after the cycles of indentation to calculate the dynamic hardness. There is no way of describing how to determine all these parameters in advance, thus the system can only be calibrated by comparing the resulting (F,δ,t) diagrams with operator observations.

The quasi-static nature of the deformation during the dynamic indentation can also be demonstrated by comparing in detail the indentations formed under dynamic and static conditions at the same strain level (Fig.8). The size of the plastic zone that surround an indentation site is dependent on the ratio (H/E) and d relation of the form

$$b=\left(\frac{E}{H}\right)^{1/2}\frac{d}{2\left(\sqrt{2}\pi\tan\theta\right)^{1/3}} \qquad (6)$$

where b is the radius of a hemispherical plastic zone centred at the point of initial contact, d - the diameter of the imprint and 2θ the apex angle of cone indenter. Thus, the smaller plastic zone sizes (at given d) for impact loading suggest a dynamic hardness higher than the static value. For a rate sensitive material, the stress-strain curves reveal that the yield stress increases with strain rate. Accordingly, the plastic zone characteristics should vary under static and dynamic indentations also.

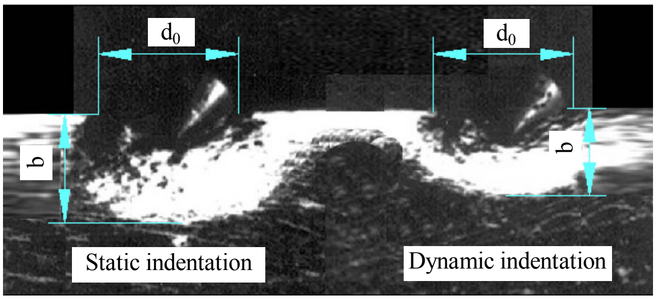

Fig.8 Micrographs of deformed plastic zones in cross-section for static and dynamic indentation and equal radii of indents

To analyse the variations in the accumulated plastic strain as a function of depth beneath the indentation in a specimen material and to gain an estimate of the differences in plastic zone size and shape with respect to the loading rate, microstructural analyses of the indentation zones were performed. Unlike the previous rebound methods, which have used the principles of rigid body (ball indenter) dynamics for determining the dynamic hardness, the current experimental scheme utilizes triplex dynamic scheme “striker – indenter – specimen”.

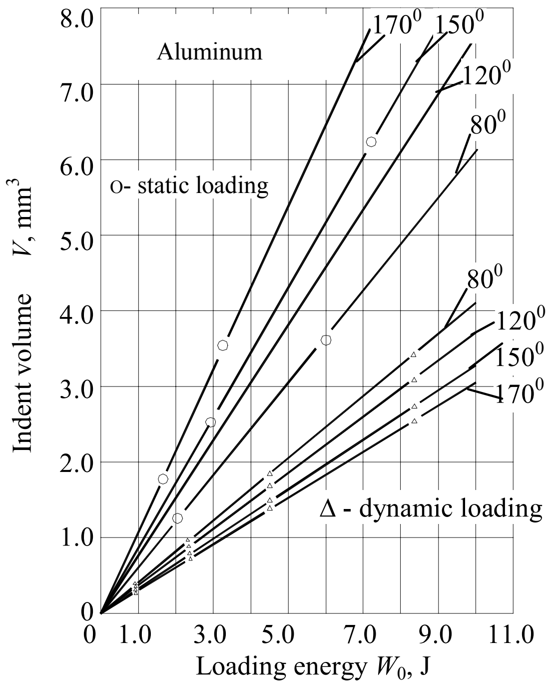

Fig. 9 The variation of indent volumes with loading energy and contact apex angles for aluminium (static and dynamic values)

The fact that sizes of the plastic zone are almost identical in shape under static and dynamic indentation conditions (Fig.9) provides a strong proof for the applicability of quasi-static conditions during the dynamic indentation. If the specimen mass is small, the duration, *t*, is also small and the force, *F*, must be larger than that generated by a more massive specimen. The measuring deviation of the zero point of the depth measurement can be obtained from this standard deviation of the regression curves $F = f(t)$ and $\delta = f(t)$ for static and dynamic loading. In an indentation experiment with a sharp pyramidal or conical indenter, the highest applied load, *F*, is almost invariably found to relate to the maximum penetration depth, *h* (measured after the removal of the load) and thus after elastic recovery of any surface flexure, but ignoring any small elastic recovery of the depth. It can be concluded that quasi-static conditions should be maintained during the impact process. This requires that the impact duration must be long enough for the passage of a number of elastic waves back and forth in the elastic indenter and also within the plastically deforming region in the specimen.

Consideration of the geometry shows that similarity can only be achieved if the plastic zone is adjusted to suit the applied load (Fig.8). Experiments have consistently shown that during the rebound phase of the impact process, the imprint shape relaxes elastically (because of the release of the stored elastic energy) mainly in the depth direction. Therefore, it is quite difficult to complete the imprint volume on the basis of the value of imprint diameter and simple analytical expressions. The above problem can be overcome in terms of the unrelaxed imprint volume, i.e. the volume of the imprint at the end of the penetrating phase of the indentation process. Also, the deformation within the impact indentations has become much more pronounced, causing the contours of these indentations appear somewhat irregular, compared with the static indentations. The distinguishing features are:

- the impact indentation is much shallower than the static indentation;
- the central area of the impact indentation has been pushed down, apparently as a result of shear failure;
- the outer portion of the impact profile form a rim with a much lower shape that of a comparable static indentation.

The results of the initial calibration were used to make corrections to the force value of the dynamic hardness tester. The series of test were they repeated using the corrected values. The dynamic hardness tester used the results of these tests to assess the uncertainty in the measurement of the force. Due to the practical reliability of comparative accuracy hardness test blocks are used to control the accuracy of hardness testers on a daily basis. Hardness measurements obtained with normally operating hardness testers should almost agree with the hardness values of applicable test blocks. The dynamic hardness value, defined, as the velocity quotient is dependent on the parameters of the instruments chosen i.e. the impact energy and the apex angle of the conical indenter tip. Therefore the curves are valid only for the specific impact device.

The verifications covered the force required to produce hardness indents, deformed plastic zone size beneath the indenter, and the characterization the depth sensor on the dynamic hardness tester. Dissipated energy is derived out of whole load – displacement data obtained during the experiment by discrete integration of force being function of displacement.

5. CONCLUSIONS

The most important error sources are identified and a procedure for system calibration is suggested. Deviations, which occur when the dynamic hardness value is converted into a static value amounts to between $\pm 3\%$ and $\pm 12\%$. As the impact device is calibrated for vertical impact, it is necessary to make subtractions on measurements values from other directions. Comparisons of applied loads for imprint and extent of plastic zones beneath indenters for static and dynamic indentation at equal indentation radius show that predictions of dynamic hardness using data from static loading are inadequate.

A reduction of the dynamic hardness measuring systems uncertainties requires that the internal structure of the transducers has an important influence on the calibration method and has be taken into consideration. The conditions under which the errors are significant are identified and must be include the measurement uncertainty into test result and into the hardness conversion tables.

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