MATERIALS CHARACTERISATION PART II: TIP GEOMETRY OF THE VICKERS INDENTER FOR MICROINDENTATION TESTS

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Abstract

This is the second of two papers by the authors associated with materials characterisation methods based on hardness testing. It is important to have knowledge of the tip geometry of the indenter employed in the hardness test as this affects the correctness of the value of contact area parameter used to determine the mechanical properties. In this paper outcomes of a study concerned with the tip geometry of the Vickers micro-indenter are presented. Results from experiment are compared with results from published works and the most current accepted analytical models. A new non-contact methodology based on a residual imprint imaging process is developed and further compared with other methods using experimental and numerical analysis over a wide range of material properties. For confirmation, an assessment was undertaken using numerical dimensional analysis which permitted a large range of materials to be explored. It is shown that the proposed method is more accurate compared with other methods regardless of the mechanical properties of the material. The outcomes demonstrate that measuring contact area with the new method enhanced the overall relative error in the resulting mechanical properties including Hardness and Young's modulus of elasticity. It is also shown that the value of contact area using actual indenter geometry obtained from experimental load displacement analysis or FEM numerical analysis is more accurate than the value obtained from the assumption of perfect indenter geometry and hence can be used for materials with low strain hardening property.

Keywords: Vickers indenter; micro-indentation; finite element modelling (FEM); 3-D optical profilometry, brittle material

1 INTRODUCTION

The elastic properties of a target material can be calculated by applying a solution proposed by [1], on the assumption that indentation depth exceeds the radius of curvature of the indentation tip. However, the method has limited use for materials exhibiting linear elastic behaviour under indentation load. The method, based on the elastic plastic constitutive material law, can be applied when the following two assumptions are satisfied. Firstly, the unloading phase of the load displacement curve is assumed elastic, having an elastic recovery depth h_e. Secondly, the reloading path is expected to follow the unloading phase of the first indentation until maximum indentation depth of the previous indentation is achieved.

The expression derived by [1] to describe the elastic unloading curve obtained from the elastic load-displacement relationship for the case of an indentation by a rigid conical indenter on a linear elastic half-plane, is given as

$$P = \frac{2E\tan\alpha'}{\pi(1-v^2)}{h_e}^2 \tag{1}$$

$$h_e = \frac{\pi}{2}a\cot\alpha' \tag{2}$$

Differentiating Eq. (1)

$$\frac{dP}{dh_e} = \frac{4E\tan\alpha'}{\pi(1-v^2)}h_e = S_s \tag{3}$$

Substituting Eq. (2) into Eq. (3),

$$\frac{dP}{dh_e} = \frac{2\delta E}{\sqrt{\pi}(1-v^2)}\sqrt{A_c} \tag{4}$$

-where P is the maximum load of the indenter, E and v are the Young's modulus and Poisson's ratio of the specimen respectively, α' is the effective half-angle of the indenter which takes into account the geometric half-angle of the indenter and the residual imprint from the previous indentation.

δ is the indenter correction factor, which approaches unity in the case of a small deformation of an elastic plastic material caused by an indenter of a rigid asymmetric smooth profile. Eq. (4) represents the conventional relationship to determine the elastic properties in depth sensing indentation. Bulychev [2] found this relationship suitable for cylindrical punch and spherical indenters, while [3] showed that the relationship is also true for all types of pyramidal indenters.

A validation of the elastic unloading phase and equivalent effective angle leads to the possibility of direct use of Sneddon's elastic relation Eq. (1) and [3] have shown that for a range of indenter shapes, the relation given by Eq. (4) is applicable.

The Vickers indenter is usually assumed to be a rigid body possessing a geometry that accords with a perfect pyramidal shape having a face angle θ, of 68°. The projected contact area A_p to depth relationship proposed by [4] is given by:

$$A_P = 4\,{h_c}^2 tan^2\theta \tag{5}$$

The actual projected area of a Vickers indenter (blunting indenter) is similar to that given for a blunting Berkovich indenter [5] as described by [3] and [6], can be determined from

$$A_P = C\,(h_c + h_d)^2 \tag{6}$$

-where, h_d is the distance between the blunted edge and the cone-end. Neglecting this value will result in an underestimation of the projected area. This relation also shows that the projected area is a function of contact depth h_c. Thus, the accurate determination of contact depth and indenter tip radius is crucial for the determination of elastic properties when using Eq. (4). The contact depth is obtained from the expression

$$h_c = h_{max} - \varepsilon \frac{P_{max}}{dP/dh_e|h_{max}} \tag{7}$$

-where ε is a constant depending on Vickers indenter geometry. The value of ε may change in magnitude between ε = 0.75 as proposed by [3] and ε = 0.785 as proposed by [6, 7] over a wide range of exponents of the unloading elastic-plastic indentation curve relationship.

It is found from numerical simulation and experimental works proposed by many researchers that the indentation material will exhibit a pile–up around the plastic imprint should the ratio of E/σ_y be large enough, typically greater than 30. This type of deformation will result in the deviation of the actual contact depth, h_c, from the value derived by Eq. (7). Cheng and Cheng [8] found this deviation to be more than 30% in some cases. Oliver and Pharr [9] reported that the values due to pile-up are only significant when $h_r / h_{max} > 0.7$, this principle value again being relevant for the case of $E/\sigma_y > 30$. This criterion strictly limits the possibility of use of the Oliver & Pharr approach, Eq. (7) to accurately determine h_c for typical materials. Therefore, the accurate measurement of the projected contact area, A_p is considered as a primary challenge to reduce the errors in the determination of mechanical properties such as hardness, and reduced modulus E_r.

It is recognized that h_c is affected by the material behaviour during the indentation process, which is shown, either pile-up or sink-in. The available methods to approximate h_c based on the material behaviour may result in over-estimation or under-estimation of the material property values.

A knowledge of indenter geometry is key to improving and validating the accuracy of the microindentation test. Moreover, the accurate representation of the indenter's geometry has a great effect on the results of area function. The area function in [5] was determined based on the Oliver & Pharr method of continuous stiffness measurements of the Berkovich nanoindentation experimental test, which requires a continuous depth sensing technique. This requirement has limited the application of that approach in engineering conditions. However, instrumented indentation machines require the complete set of loading-unloading indentation depth curves to compute the material properties and this is not practically possible for all material systems such as those that are brittle. These materials exhibit crack initiation and propagation within the indentation process. A simpler and more effective methodology needs to be developed to characterise the material properties using a standard microindentation hardness test machine. This work seeks to provide that solution.

Although fused silica has been used widely as a reference standard for nanoindentation testing, the indentation test mechanism of most brittle material systems remains difficult to interpret using general load displacement curves. There are many reasons behind these difficulties. Firstly, the lack of constitutive material laws to describe the mechanical behaviour of particular brittle material families which exhibit permanent changes in density under very high pressures (densification behaviour) resulting in difficulties to describe the load indentation results in terms of mechanical properties. Keryvin [10] proposed more reasonable constitutive models implemented in FEM software by using a material sub-routine. The results show that the pressure-induced densification (PID) constitutive law can be used to interpret the mechanical response of the load displacement curve and to describe the densified zones underneath the indenter.

Secondly, a wide range of brittle material such as glasses and metallic glasses can exhibit high levels of sink-in and pile-up, covering a broad range of contact behaviour compared with metallic materials, and results in a demanding challenge to determination of mechanical properties using the load indentation results. Based on results of material behaviour during indentation tests, the values of projected area calculated via direct analysis of the load displacement curve, possess high levels of error and are generally unreliable for accurate prediction of material properties due to the occurrence of pile-up and sink-in.

It has been demonstrated by previous researchers of microindentation that most materials exhibit pile-up or sink-in, around the indentation imprint, and it is increasingly recognized that the estimation of the residual indentation projected area based on the analysis of the unloading part of the load displacement curve can produce errors of up to 50% or more. Some authors [11-13] suggested that the most appropriate and accurate method is to directly image the indentation using a well calibrated high-resolution atomic force microscope (AFM) and obtain the projected contact area of the indentation from the AFM image processing software. Recent developments in indenter tip scanning probe microscopy (ITSPM) based on contact mode imaging have been concerned with residual imprint image processing without any major influence on the sample or any great damage to the imprint image. However, the ITSPM technique has disadvantages when compared with AFM. The slower image scan operation, and the probe used in the scanning process usually has a higher blunting tip radius due to the use of a much wider pyramidal geometry which requires a larger force to scan the whole indentation surface and results in greater surface damage compared with AFM. Therefore, this technique is more widely used in the height image application.

Charleux [14] proposed a new post-mortem ITSPM method for residual imprint observation using a height based imaging technique produced by built-in scanning probe microscopy (SPM). The SPM method was compared with three different direct methods by analysis of the load displacement curves obtained from numerical analysis and experiment, covering a wide range of material properties. It was shown that the new method systematically leads to lower error levels regardless of the type of material.

Gerberich [15] proposed that the residual depth of penetration indicated by the line profile obtained from the AFM probe image after the nanoindentation test is about 65% of the residual depth of penetration obtained by the same nanoindentation SPM. This large discrepancy was attributed to the fact that the geometry of the AFM probe tip is much sharper than the tip used for indentation and imaging ITSPM. However, in such applications the indentation residual relaxation should be considered by assuming a delay time after the indentation process in order to achieve the final residual indentation depth. Lilleodden [16] proposed that the relaxation of the residual indentation was caused by dislocations on the indentation surface, which may affect the accuracy of results for the following reasons: Firstly, the indentation depth was less than or the same as the surface roughness of the specimen. Secondly, the AFM probe tip was the same as the one with which the indentation had been made, in which case results would not be useful.

Therefore, the scanning probe geometry should be carefully considered in the case of both AFM and ITSPM, based on the indentation size and material type. However, with the development of microscopic imaging processes, the systematic residual imprint imaging makes the indentation results more reliable and faster to obtain.

The aim of this Paper is to introduce a new non-contact methodology, based on residual imprint imaging, to reduce the discrepancy in the results determined from other methods. It will provide increased precision in the measurement of the projected contact area and overcome issues of damage associated with the probe contact. In this study a quantitative and qualitative 3-D topography analysis of residual imprint from microindentation tests were developed using a three-dimensional optical profiler instrument Wyko NT1100 coupled with Wyko vision 64 analysis software supported by FEA to determine the indentation geometry and indenter area function. This new approach has been tested and validated on amorphous materials.

2 MICROINDENTATION TESTS: EXPERIMENTAL PROCEDURE

Fused silica samples with known mechanical properties were chosen as a test material for this work. The material was prepared to standard metallographic and microindentation test specification as described in [5].The three-dimensional optical profiler instrument (Wyko) was used to investigate the morphology of the specimen surface after indentation. The Wyko system is a first-rate tool for characterizing surface height variations and quantifying the degree of sink-in or pile-up due to plasticity, having excellent spatial resolution.

The scanned indentation imprint images from the Wyko system were examined with a projected top view and in radial line mode. The new technique was employed to calculate the projected contact area of residual imprints using Wyko Vision 64 software analysis. The contact edges between the indenter and the specimen were obtained in radial line mode with reference to projected top view images, edges can be identified by a discontinuity in the slope of the cross sectional shape of the line profile. In addition, the radial line can clearly show any pile-up or sink-in around the indentation. During the maximum loading stage, the contact edge produced from a general pyramidal indenter such as that used with Vickers indentation equipment can exhibit either sink-in or pile-up and in some cases both sink-in and pile-up may occur simultaneously.

In the case of pile-up, the material appears at above the reference surface due to plastic flow around the indenter during indentation, and the phenomenon is commonly observed with low strain hardening metallic materials. In the case of sink-in, material flows to a depth below the reference surface, a phenomenon observed in high yield strength materials such as fused quartz. During the unloading stage, and in the case of the contact edge exhibiting residual pile-up behaviour, the cross-section of the contact area measurement should commence from the lowest point of the imprint to the highest point on the pile-up material. Continuous measurement of the highest points on each of the sections around the imprint form the contact zone boundary regarded as belonging to the imprint area. The inherent surface roughness of the material may make the high point positions of pile-up on the contact edge profile unclear. Therefore, in order to overcome this effect, the imprint is considered to lie inside the specified circular area and the radial line mode rotated by a small angle β_i along an axis parallel to the projected top plane and running through the lowest point of the imprint. Repeating this process from 0 to 360^o leads to determination of the whole contact edge.

Figure 1 shows key steps of the proposed technique to calculate the contact area: (a) Represents the projected top view of the three-dimensional optical image of the Vickers indentation on fused silica with maximum load of 2.94N, (b) projected top view with different orientation of rotated angle, β_i , (c) full cross-section graph showing the radial line mode position at particular (r, β_i) , which indicates that the position of the contact edge of microindentation is above the original surface level, representing pile-up behaviour around the indentation, and (d) the process is repeated for all required values of β resulting in a full description of the contact edge boundary, specified by the black line. This describes the novelty and key differences of this new approach.

3 MICROINDENTATION TESTS: RESULTS AND DISCUSSION

3.1 Projected area determined by 3-D optical profilometry

Commercial micro-indentation testing and 3-D optical profilometry (non-contact Wyko instrument) methods have been coupled to measure the mechanical properties of fused silica. A series of micro-indentation tests at different indentation loads were applied to the fused silica. The method to determine the area function is based on the analysis of the two-dimensional image sets obtained from micro-indentation results. The contact area, A_c, and indentation depth profiles corresponding to projected top view and radial line mode were extracted directly from the imprint images data analysis software as is shown in Figure 2. In image (a) a radial line position at particular (r, β_i) across the 3-D optical profile-meter image and two arrows, 1 and 2, are located on it. These arrows identify the positions of the contact edge between the specimen and the indenter along the line. Image (b) gives the line profile across the radial line mode on the indentation image along the direction indicated by arrows 1 and 2 shown in image (a). Moreover, the positions of arrows 1 and 2 in images (a) have identical scale and hence a one-to-one correspondence with arrows 1 and 2, respectively, in image (b). The distance between the two arrows in frames (b) is measured directly from the Wyko image. Repeating this process from around the indentation image will lead to identification of the whole contact counter (black line), and then the contact area directly determined across the counter edge, black line in image (a), that is related to the residual imprint behaviour of either the sink-in or pile-up.

However, very small discrepancies in the positioning of the arrows are unavoidable. Vickers hardness values were determined from the measurements of residual indentation size using the Wyko images Further information from the residual Vickers indentation can also be measured such as the angle at the apex and the residual depth which help to identify the elastic recovery depth and densification values.

To calculate the hardness corresponding to a given indentation, the projected contact area of the indentation was measured using the top projected mode with reference to the radial line mode of the Wyko image, and a subsequent image analysis program.

3.2 Projected area determined by load displacement analysis

A series of Vickers indentation tests were carried out at six pre-determined maximum load values in the range of 10-1000gf (approximately 0.1-10N). Three indentations tests were performed at each peak load, and for each indentation the impression diagonals were measured three times with a resolution of 0.1µm. The average of the group results are presented in this study with thermal drift neglected. Figure 3 shows the typical load indentation curves obtained from this experimental work.

The contact area Ac of the Vickers indentation was determined using the same methodology as that used for the Berkovich indenter as described in [5]. However, the main disadvantage of the method is the inability to include effects of pile-up. To accommodate this constraint we have chosen fused silica as the reference material as this exhibits very little pile-up during deformation. The regression analysis was obtained according to a non-perfect indenter approach describe by [3, 5], and given by Eq. (6) using Sigma plot software. This analysis gives the value of C =33.1,and $h_d = 60$ nm. The experimental results shown in Figure 4 indicate that the deviations of the determined area function from the perfect Vickers geometry are attributed to the indenter tip blunting which affects the best-fit value of $h_d$and the effective half-included angle change resulting in deviation in the best-fit value of parameter C that is equal to 24.56 for the perfect indenter.

3.3 Indentation contact area determined from numerical modelling

Numerical simulations were created using ABAQUS 6.14.1 software to determine the area function of a pyramidal Vickers indenter. An equivalent axisymmetric conical indenter approximated the pyramidal indenter represented in Figure 5 with a 'spherical cap' tip radius and specific inclination angle, each of the Vickers pyramid indenter and its equivalent conical indenter, having the same projected area versus the contact depth. Lichinchi [17] have shown that there are no significant differences in the load–indentation curves when comparing the 3-D pyramidal indenter model with the axisymmetric conical indenter model. The indenter is considered as a rigid cone with a spherical end exhibiting an effective half-included angle and tip radius of 1.25 µm to match the results obtained from the Scanning Electron Microscopy (SEM) images.

A series of FEM simulations for a Vickers indenter were performed using a wide range of tip geometry (tip radius and inclination angle). Load displacement curves of numerical modelling were compared with experimental results at different indentation loads. The best match results were then obtained using the two stage objective function. The objective function used in this study is the root mean square error (RMSE).

In this work a parametric study was developed using simulation space to determine the tip geometry for a given set of indentation data employing a script programming language (python) interfaced with ABAQUS FEM. The proposed programme will automatically search for a range of values of tip geometries until the optimization is achieved. For the axisymmetric conical indenter under investigation, two parameters were varied within the two stages. In the first stage, tip radius was varied from 0.1 to 2.5µm using a small indentation depth of h < 1 µm with an increment of 0.01µm. The optimal tip radius from the first optimization stage is used as an input data for the second parametric study for which inclination angle was varied from 68° to 75° using an indentation depth of h > 1.25 and increment of 0.1°. The numerical results were then stored into a database to form a simulation space. The results were structured in an excel file program for interpretation.

3.4 FEM supported determination of the area function

Figure 6 shows the comparison between the load displacement curves from theory and experiment for three different maximum loads using optimal tip radius of 1.25 µm and inclination angle of 74.1°. The two curves exhibit good correlation and only a minor deviation occurs in small loads due to the elastic-perfectly plastic material constitutive material law used in the FEM, which is not fully adequate in explaining the behaviour of fused silica material.

The projected area and contact depth were calculated using the same procedure as that used with the FEM Berkovich indentation [5]. The projected area is calculated for the best match between the loading unloading curves from theory and from experiment using the two stage optimization approach. A series of FEM simulations were carried out at various indentation loads enabling the indenter projected area A_P to be plotted against contact depth h_c (area function of Vickers indenter) for the examined fused silica reference material, Figure 7.

4 DISCUSSION OF FEM AND EXPERIMENTAL RESULTS

Contact area was investigated at different indentation loads using experimental and numerical results. Three different experimental methods were employed to determine the contact area. The first method was carried out using the Martin & Oliver method to analyse the experimental load displacement curve for determination of the actual indenter geometry area function A_{MO}, as proposed in section (3.2). The second, newly proposed method, employed a 3-D optical Profilometer image processing technique to determine the contact area A_{PM} , as described in section (3.1). The third method was that proposed by the Oliver & Pharr method in which the contact area for a perfect indenter geometry is obtained using, A_{OP}, Eq. (5). The numerical approaches were developed to determine the contact area using the actual indenter geometry derived from FEM analysis, A_{FEM} as proposed in section (3.4).

In this study, the analyses concentrated on the determination of the relative error, R_e, between the true contact area A_c estimated through Sneddon's Eq. (4) and the contact area predicted by each method. Based on the Sneddon's method the relative deviation in the contact area will give roughly the same deviation in the hardness value and half of the Elastic modulus value (see Eq. (4))

The, R_e is given by the expression Eq. (8)

$$R_e = \left(\frac{A - A_C}{A_C}\right) 100\% \tag{8}$$

Figure 8 shows a comparison of the relative error in values obtained from theory and experiment *(A_{MO}, A_{PM}, A_{OP}, and A_{FEM})*, at different indentation loads. The relative error Eq. (8) between the true projected contact area estimated from experiment data by Sneddon's Eq. (4) and each of the other methods is plotted. Each bar refers to one of the four methods obtained at different indentation loads. The use of such a procedure will allow for comparison between the numerical and experimental tests.

The success rate of the method chosen was based on their capability to match the true value of A_c, within ∓ 10 % error, which is still realistic from a numerical and experimental point of view. As displayed, the R_e values estimated from the four methods systematically underestimates the contact area.

The A_{MO}, and A_{FEM}, methods perform well only for loads less than 3N, though both methods give reasonable results for the fused silica sample. Increases in the indentation load greater than 3N leads to increase yield strain rate and results in an unexpectedly high error level. This suggests that both methods were optimized using this material as a reference for microindentation load of up to 3N. Based on observation of the results, the A_{MO}, method performs better than the A_{FEM}, method although each underestimates the contact area.

The determination of the contact area using perfect indenter geometry A_{OP}, clearly leads to a severe underestimate of the contact area due to inaccurate estimation of the contact geometry by assuming finite sharp tip radius. Poon [18, 19] observed that the values of the indentation load increase for a larger tip radius at the same indentation depth compared with the sharp indenter.

The overall performance of the proposed new method, using 3-D optical profilometry and image processing, for A_{PM} , is more reliable compared with the other direct methods .

The main advantages of the proposed method are firstly, that it is a non-contact measurement technique, which eliminates all effects of indenter shape and force values in comparison with other imaging techniques such as AFM, and ITSPM. Secondly, it does not necessarily require use of a highly expensive instrumented indentation machine to extract the mechanical properties from load displacement curves. Thirdly, the contact area measurement obtained from this method is not related to the load indentation, such as is the case with the Oliver & Pharr method [20], therefore the contact area is insensitive to the frame and specimen compliances, i.e. stiffness issues.

Fourthly, this method is compatible with any tip blunting values, and does not require a tip calibration process. Finally, it is a realistic method for any indentation load and fully accounts for material sinking or pile-up behaviour.

5 DIMENSIONAL ANALYSIS OF CONTACT AREA FROM FEM

Dimensional analyses were employed to investigate the effect of elastic and plastic parameters on the contact area. Two sets of material constitutive law were examined to cover a wide range of contact geometry and material properties. The first constitutive law, used for an isentropic elastic plastic material with power law strain hardening (EPH) driven by the tensile behaviour (stress, σ, and strain, ϵ), is given by Eq. (9). This is commonly used in the numerical simulation of metallic alloys [21].

$$\sigma = \begin{cases} E\varepsilon & , for : \sigma < \sigma_y \\ \sigma_y \left(E/\sigma_y\right)^n , for: \sigma > \sigma_y \end{cases} \tag{9}$$

The second constitutive law, is the Linear Drucker-Prager law for plasticity (LDP) of soil and granular material [22], which is widely used to explain the influence of the hydrostatic stress component on yielding stress [23]. Such a model can be used to describe the deformation behaviour of metallic glass and polymer materials [24]. The linear plastic Drucker-Prager model is given by Eq. (10):

$$F = q + \beta\, \sigma_m - \sigma_{yc} = 0 \tag{10}$$

Where: $q = \sqrt{\frac{3}{2} S_{ij} S_{ij}}$ is the Von Mises equivalent stress, and S_{ij} is the stress deviator, $\sigma_m = \sigma_{kk}/3 = -P$, where P is the hydrostatic pressure stress, σ_{yc} is the compressive stress, and β is the friction coefficient

$$S_{ij} = \sigma_{ij} - \sigma_m \varepsilon_{ij} \tag{11}$$

The friction coefficient, β, is considered based on the assumption that in the case of pure shear loading $\sigma_m = 0, \beta$ may be neglected resulting in, $q = \sigma_{yc}$. Secondly, in the case of pure hydrostatic loading the stress $q = 0$, $\beta = -\sigma_{yc}/P$. Therefore, β may be considered as the ratio between the compressive yield stress of the material and the hydrostatic pressure stress in pure hydrostatic loading.

Based on each of the above constitutive material law models, the dimensional analysis of material properties effect on the contact area is given by Eq. (12):

$$A_c = \begin{cases} h_m^2 \prod_{EPH}(v, \sigma_y/E\,, n) \\ h_m^2 \prod_{DPP}(v, \sigma_{yc}/E\,, \beta) \end{cases} \tag{12}$$

A series of FEM simulations were performed using an equivalent two-dimensional axisymmetric model developed in a previous section (3.4) to investigate the effect of (EPH, and LDP) constitutive models on the contact area. At each simulation, load displacement data and height indentation image were extracted and analysed. In the both models the Poisson's ratio and the Elastic modulus were fixed at values of 0.3, and 100 GPa respectively. However, it was found from the dimensional analysis of material properties that only the parameters (σ_y, n) in the case of the EPH constitutive model, and (σ_{yc}, β) in the case of the LDP constitutive model have a major influence on the contact area. Accordingly, the values of (σ_y,n), and (σ_{yc},β) are modified in the numerical simulations of EPH, and LDP respectively.

Table (1) shows the range of values for the dimensionless parameters, for both the EPH, and LDP constitutive models, used in the Numerical simulations.

Three numerical methods was developed to predict the contact area: the first method based on Oliver & Pharr determined the contact area using Eq. (5) for a perfect indenter geometry $A_{perfect.}$ The second method based on Martin and Oliver, used the optimized value of the indenter geometry derived from FEM analysis A_{actual} section (3.4). The third method used the newly proposed approach in which the contact area was determined by analysis of the residual imprint based on indentation height image processing and indentation curves after each simulation.

5.1 Discussion and analysis of FEM results

The relative error, R_e, were obtained from each of the three methods as plotted in Figures 9 (a1-a2), 10 (a1-a2) and 11 (a), and the absolute relative error, $|R_e|$, also plotted in Figures 9 (b1-b2), 10 (b1-b2), and 11 (b) for EPH, and LDP material constitutive laws respectively. The magnitude of error at particular tensile and compressive yield strain values, σ_y/E, and σ_{yc}/E were highlighted .

A parametric study was carried out on both material law equations to determine the contact area for a given set of material properties based on indentation data using a script programming language (python) interfaced with ABAQUS FEM [25]. In this arrangement the true contact area, A_{true} is directly measured from the indentation results using ($\pi\, r_c^2$) and the two methods of contact area $A_{perfect}$, and A_{actual} were estimated from load displacement curves based on the indenter geometry. The A_{pm} was estimated from the indentation height image processing and indentation curves using Python script subroutine interfaced with ABAQUS. The output data base file from ABAQUS of the residual imprint images will then be imported into Gwyddion software as a (GSF) format in order to use this algorithm for image processing analysis.

Based on the FEM observation of the material laws (EPH, and LDP), the magnitude of, R_e, given by Eq. 13 was found to strongly depend on the specimen mechanical properties and the indenter geometry which produced different contact behaviour, either pile-up or sinking in.

$$R_e = \left(\frac{A - A_{true}}{A_{true}}\right) 100\% \tag{13}$$

In case of the EPH type material, the relative error measurements for perfect indenter geometry, $A_{perfect}$ were calculated.in Figure 9 (a1, a2) shows an underestimation of the contact area for materials exhibiting strain hardening of $0.1 \leq n \leq 0.4$ over a wide range of tensile strain values of $0.001 \leq \sigma_y/\mathrm{E} \leq 0.01$. This suggests an underestimation of contact area when pile-up occurs and agreement with results predicted by [26].

Figure 9 (b1, b2) shows the absolute relative error measurements $|R_e|$ for perfect indenter geometry, $A_{perfect}$, . As displayed, the $|R_e| > 10\%$ for all material properties. This suggests that the perfect indenter geometry with sharp indentation edge does not consider the piling up behaviour of the examined materials for a given yield strain values, as well as providing a lower load displacement curves compared with the actual indenter geometry.

Figure 9 (a1, a2) presents the relative error measurements for actual indenter geometry A_{actual} . The results demonstrated underestimation $R_e \leq 10\%$ of the contact area for all range of tensile strain values with strain hardening of, $n \leq 0.1$. It is also clearly noticed that R_e of A_c for strain hardening $n \geq 0.1$ is overestimated. The strain hardening increases with the increase in the tensile strain σ_y/E of the strain hardening range $0.1 \leq n \leq 0.3$. The overestimation of contact area at this stage is due to the sinking in behaviour. The over estimation behaviour exhibit decreasing trends with increasing σ_y/E for $n = 0.4$.

Figure 9 (b1, b2) shows the absolute relative error measurements $|R_e|$ for the actual indenter geometry, A_{actual}. This method performed well for all material properties with $n \leq 0.2$, which produces a value of $|R_e| \leq 10\%$ where the materials show no or little pile-up behaviour. The results also suggest that this method is not suitable for material with high strain hardening values.

Figure 10 (a1,a2) shows that the $A_{perfect}$ method systematically tends to underestimate the A_c for materials with friction angle range $0^o \leq \beta \leq 30^o$, these measurements has been recorded irrespective of the magnitude of compressive yield strain, σ_{yc}/E (i:e regardless of the pile-up or sinking in material behaviour).

Figure 10 (b1, b2) shows the magnitude of the absolute relative error, $|R_e|$ is higher than that for the other methods. This may be attributed to the fact that the perfect indenter (sharp tip geometry) modelling will lower the resulting of force and consequently A_c. The absolute relative error of the contact area decreases with the increasing compressive yield strain for the friction angle range $0^o \leq \beta \leq 30^o$

Figure 10 (a1, a2) represents the relative error measurements of actual indenter geometry A_{actual} for LDP type materials, this method systematically tends to underestimate the A_c when the compressive yield strain is lower than 0.02 and friction angle range $0^o \leq \beta \leq 30^o$, and to underestimate the results within the whole range of compressive yield strain properties and $\beta = 30^o$. This suggests an inability to cope with pile-up material behaviour. Figure 10 (b1, b2) demonstrated that although the value of $|R_e| > 10\%$, this method performs well with materials exhibiting high compressive yield strains (> 0.02%)

Figure 11 (a, and b) shows the numerical results of the newly proposed method to determine the contact area in the case of EPH and LDP for different combinations of (n, σ_y/E) and (β ,σ_{yc}/E) respectively. The numerical simulations demonstrate that the relative error of this method performs very well with both material constitutive laws giving a 100% success rate within a specified relative error target of less than 10%.

6 CONCLUSIONS

The effect of variation in the tip geometry of the Vickers hardness stylus on the determination of contact area in instrumented microindentation tests was analysed and compared with different theories. A new method is proposed for the determination of contact area based on residual imprint measurements using 3-D optical profilometry supported by Vision 64 and Gwyddion software for image processing and analysis. For evaluation, results for contact area computed using the new method have been compared with results from three other numerical and experimental methods based on data for fused silica reference material. An assessment using dimensional analysis has permitted a wide range of contact geometry and material properties to be explored. It has been shown that the proposed method is more accurate compared with other methods regardless of the mechanical properties of the material under test. The outcomes show that measuring contact area with the new method improves the overall relative error in the obtained mechanical properties such as Hardness and Young's modulus of elasticity. We have also to emphasize the fact that the measurement of contact area using actual indenter geometry obtained from experimental load displacement analysis or FEM numerical analysis is more accurate than the contact area measurement from the assumption of perfect indenter geometry and can be used for materials with low strain hardening property.

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