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**DETERMINING THE ELASTIC–PLASTIC
PROPERTIES OF METALLIC MATERIALS THROUGH
INSTRUMENTED INDENTATION**

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Determining the elastic-plastic properties of metallic materials through instrumented indentation

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Resumen

En los últimos años, y asociado al desarrollo de la tecnología MEMS, la técnica de indentación instrumentada se ha convertido en un método de ensayo no destructivo ampliamente utilizado para hallar las características elástico-plásticas de recubrimientos y capas delgadas, desde la escala macroscópica a la microscópica. Sin embargo, debido al complejo mecanismo de contacto debajo de la indentación, es urgente proponer un método más simple y conveniente para obtener unos resultados comparables con otras mediciones tradicionales. En este estudio, el objetivo es mejorar el procedimiento analítico para extraer las propiedades elástico-plásticas del material mediante la técnica de indentación instrumentada.

La primera parte se centra en la metodología llevada a cabo para medir las propiedades elásticas de los materiales elásticos, presentándose una nueva metodología de indentación, basada en la evolución de la rigidez de contacto y en la curva fuerza-desplazamiento del ensayo de indentación. El método propuesto permite discriminar los valores de indentación experimental que pudieran estar afectados por el redondeo de la punta del indentador. Además, esta técnica parece ser robusta y permite obtener valores fiables del módulo elástico.

La segunda parte se centra en el proceso analítico para determinar la curva tensión-deformación a partir del ensayo de indentación, empleando un indentador esférico. Para poder asemejar la curva tensión-deformación de indentación con la que se obtendría de un ensayo de tracción, Tabor determinó empíricamente un factor de constricción de la tensión (ψ) y un factor de constricción de la deformación (β). Sin embargo, la elección del valor de ψ y β necesitan una derivación analítica. Se describió analíticamente una nueva visión de la relación entre los factores de constricción de tensión y la deformación basado en la deducción de la

ecuación de Tabor. Un modelo de elementos finitos y un diseño experimental se realizan para evaluar estos factores de constricción. A partir de los resultados obtenidos, las curvas tensión-deformación extraídas de los ensayos de indentación esférica, afectadas por los correspondientes factores de constricción de tensión y deformación, se ajustaron a la curva nominal tensión-deformación obtenida de ensayos de tracción convencionales.

En la última parte, se estudian las propiedades del revestimiento de cermet Inconel 625-Cr₃C₂ que es depositado en el medio de una aleación de acero mediante un láser. Las propiedades mecánicas de la matriz de cermet son estudiadas mediante la técnica de indentación instrumentada, haciendo uso de las metodologías propuestas en el presente trabajo.

Abstract

In recent years, along with the development of MEMS technology, instrumented indentation, as one type of a non-destructive measurement technique, is widely used to characterize the elastic and plastic properties of metallic materials from the macro to the micro scale. However, due to the complex contact mechanisms under the indentation tip, it is necessary to propose a more convenient and simple method of instrumented indentation to obtain comparable results from other conventional measurements. In this study, the aim is to improve the analytical procedure for extracting the elastic plastic properties of metallic materials by instrumented indentation.

The first part focuses on the methodology for measuring the elastic properties of metallic materials. An alternative instrumented indentation methodology is presented. Based on the evolution of the contact stiffness and indentation load versus the depth of penetration, the possibility of obtaining the actual elastic modulus of an elastic-plastic bulk material through instrumented sharp indentation tests has been explored. The proposed methodology allows correcting the effect of the rounding of the indenter tip on the experimental indentation data. Additionally, this technique does not seem too sensitive to the pile-up phenomenon and allows obtaining convincing values of the elastic modulus.

In the second part, an analytical procedure is proposed to determine the representative stress-strain curve from the spherical indentation. Tabor has determined the stress constraint factor (stress CF), ψ and strain constraint factor (strain CF), β empirically but the choice of a value for ψ and β is debatable and lacks analytical derivation. A new insight into the relationship between stress and strain constraint factors is analytically described based on the

formulation of Tabor's equation. Finite element model and experimental tests have been carried out to evaluate these constraint factors. From the results, representative stress-strain curves using the proposed strain constraint factor fit better with the nominal stress-strain curve than those using Tabor's constraint factors.

In the last part, the mechanical properties of an Inconel 625- Cr_3C_2 cermet coating which is deposited onto a medium alloy steel by laser cladding has been studied. The elastic and plastic mechanical properties of the cermet matrix are studied using depth-sensing indentation (DSI) on the micro scale.

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Chapter 1 : Introduction

1.1. Research background

The instrumented indentation technique is currently used to determine the local mechanical properties of metallic bulk materials and mechanically characterize coatings in the modern engineering industry. The instrumented indentation technique continuously records the depth of the indentation. Compared with the conventional mechanical measurement techniques, e.g. tensile test, compression test, it provides abundant information on the materials' properties (Hardness, Young's modulus, plastic property, residual stress, etc). Moreover, this technique is a non-destructive one and the specimens are easy to prepare. Consequently, it can be applied to an in-working structure.

In recent decades, there have appeared a large number of analytical or numerical methodologies to extract the elastic-plastic properties of metallic materials. However, due to the complexity of the mechanical contact behavior in the indentation measurement, these methodologies are not a straightforward procedure. They need numerical assistant or experimental empirical parameters. Moreover, these methodologies are usually not convenient and limited to a specific range of material properties. From the experimenter point of view, it is necessary to have a more direct and simple analytical methodology to determine the properties of the materials, which could be consistent with measurements from the conventional test.

1.2. Reason for the study and outline of the thesis

In this study, the main aim is to improve the methodologies of the instrumented indentation technique to determine the elastic-plastic properties of metallic bulk materials. As regards the mechanical properties of metallic materials and the characteristics of the different indenter tips, the analytical methodologies in this study can be divided into two parts: the first part is related to a novel analytical methodology for extracting the elastic properties using a Berkovich indenter. The other part is a new analytical process for extracting the plastic properties using a spherical indenter. The outline of the thesis as follows.

Chapter 2 focuses on the theoretical issues involved in the measurement with the indentation technique. It begins with a review of the literature from the classic mechanical theory and its relationship with indentation on elastic or elastic-plastic bulk materials. Then it summarizes the current methodologies for measuring the elastic-plastic properties and discussing controversial views on them. Finally, from experimental experience, the influences of contamination on the indentation process will be discussed.

Chapter 3 focuses on the analytical methodology for extracting the elastic properties using a sharp indenter. Based on the previous analytical study for the loading curve of a sharp indenter, a novel instrument indentation methodology is proposed theoretically for bulk metallic materials, which is experimentally validated using some common metallic materials. From the result, the value of the elastic properties obtained from this methodology can be comparable with those measured from conventional tests.

Chapter 4 is dedicated to the analytical procedure for determining the plastic properties for metallic bulk materials using spherical indentation. Carrying on from the formulation of the Tabor equation, the analytical relationship between the stress constraint factor and strain constraint factor are firstly demonstrated within the framework of Hollomon's power law. An alternative procedure for extracting the plastic properties of metallic materials is proposed, which is validated from the numerical and experimental analysis.

Chapter 5 presents research into the mechanical characterization of Inconel cermet coatings using the instrumented indentation test at the micro-scale. Compared with the

Inconel bulk materials, the reinforcing Cr_3C_2 particles greatly improve the mechanical properties of the coating.

The last chapter summarizes the conclusions and highlights the contributions in the work. Some ideas and future work are also discussed.

Chapter 2 : Literature review

2.1. Theoretical background

2.1.1. Hertz contact

Based on the classic theory of elasticity and continuum mechanics, Hertz [1] was the first to obtain an analytical solution to two elastic axi-symmetric objects that are pressed together under external loading. Fig.2.1. shows two contacting elastic spheres with diameters d_1 and d_2 pressed together with force F . E_1, ν_1 and E_2, ν_2 as the respective elastic Young's modulus of the two spheres, the radius of the circular contact area is given as [2]:

$$a = \sqrt[3]{\frac{3F}{8} \frac{(1-\nu_1^2)/E_1 + (1-\nu_2^2)/E_2}{1/d_1 + 1/d_2}} \quad (2.1)$$

The pressure distribution on the contact boundary is hemispherical, as shown in Fig.2.1. At the contact boundary, the stress has a hemispherical distribution with the contact zone of $2a$ in diameter.

The normal pressure distribution under a spherical indenter was given as:

$$\frac{\sigma_z}{p_m} = -\frac{3}{2} \left(1 - \frac{r^2}{a^2} \right)^{1/2} \quad r \leq a \quad (2.2)$$

Equations (2.1) and (2.2) could also be used for the contact between a sphere and a flat surface or a sphere and an internal spherical surface.

Fig. 2.1 (a).Two elastic spheres pressed in contact with an external force F ; (b).Schematic of stress distribution on the contact boundary.

The maximum contact pressure p_m , will occur at the center point of the contact area.

$$p_{\max} = \frac{3F}{2\pi a^2} \quad (2.3)$$

In cylindrical coordinates, the analytical solutions for stresses beneath the surface of a semi-infinite, elastic plate, indented by elastic sphere are given by [1, 3]:

$$\begin{aligned} \frac{\sigma_r}{p_0} &= \frac{3}{2} \left\{ \frac{1-2\nu_2}{3} \frac{a^2}{r^2} \left[1 - \left(\frac{z}{u^{1/2}} \right)^3 \right] + \left(\frac{z}{u^{1/2}} \right)^3 \frac{a^2 u}{u^2 + a^2 z^2} + \left(\frac{z}{u^{1/2}} \right) \left[u \frac{1-\nu_2}{a^2 + u} + (1+\nu_2) \frac{u^{1/2}}{a} \tan^{-1} \left(\frac{a}{u^{1/2}} \right) - 2 \right] \right\} \\ \frac{\sigma_\theta}{p_0} &= -\frac{3}{2} \left\{ \frac{1-2\nu_2}{3} \frac{a^2}{r^2} \left[1 - \left(\frac{z}{u^{1/2}} \right)^3 \right] + \left(\frac{z}{u^{1/2}} \right) \left[u \frac{1-\nu_2}{a^2 + u} - (1+\nu_2) \frac{u^{1/2}}{a} \tan^{-1} \left(\frac{a}{u^{1/2}} \right) + 2\nu_2 \right] \right\} \\ \frac{\sigma_z}{p_0} &= -\frac{3}{2} \left(\frac{z}{u^{1/2}} \right)^3 \frac{a^2 u}{u^2 + a^2 z^2} \\ \frac{\tau_{zr}}{p_0} &= -\frac{3}{2} \left(\frac{rz^2}{u^2 + a^2 z^2} \right) \left(\frac{a^2 u^{1/2}}{a^2 + u} \right) \end{aligned} \quad (2.4)$$

Where: p_0 is the mean pressure beneath the indenter;

$$u = \frac{1}{2} \left[\left(r^2 + z^2 - a^2 \right) + \left[\left(r^2 + z^2 - a^2 \right)^2 + 4a^2 z^2 \right]^{1/2} \right]$$

The principal stresses in the rz plane can be expressed as:

$$\sigma_{1,3} = \frac{\sigma_r + \sigma_z}{2} \pm \left[\left(\frac{\sigma_r - \sigma_z}{2} \right)^2 + \tau_{zr}^2 \right]^{1/2}$$

$$\sigma_2 = \sigma_\theta \quad (2.5)$$

The maximum stress occurs on the z axis and the corresponding principal stresses can be expressed as:

$$\sigma_1 = \sigma_2 = \sigma_x = \sigma_y = -p_{\max} \left[(1 - |\xi_a|) \tan^{-1} \left(\frac{1}{\xi_a} \right) \right] (1 + \nu) - \frac{1}{2(1 + \xi_a^2)} \quad (2.6)$$

$$\sigma_3 = \sigma_z = \frac{-p_{\max}}{1 + \xi_a^2} \quad (2.7)$$

Where: $\xi_a = z/a$: a parameter of non-dimensional depth below the surface.

Fig. 2.2: Magnitude of the stress components below the surface as a function of the maximum pressure of the contacting spheres [2].

Fig.2.2. shows the distribution of the ratio of principal stresses to $p_{\max}$ along the z axis. It should be noted that in Fig.2.2, the Poisson' ratio was taken as 0.3. Note that the shear stress reaches a maximum value of $0.3 p_m$ slightly below the surface shown and plastic flow will occur first at the point beneath the surface corresponding to $z=0.48a$ [4]. From the Tresca failure criteria, the principal shear stress on the deformation plane is given as:

$$\tau_{\max} = \frac{\sigma_1 - \sigma_3}{2} \quad (2.8)$$

The plastic flow will occur when the shear stress at the point is equal to half the yield stress, σ_y . From Eq.2.3, the relationship between the mean pressure under indenter and yield stress can be expressed as:

$$p_m \approx 1.1\sigma_y \quad (2.9)$$

When the mean pressure approximately reaches the value of $1.1\sigma_y$, along the z axis, the point beneath the surface corresponding to $z=0.48a$ initially develops plastic strain. As the load increases, the plastic zone (which is surrounded by elastic material) simultaneously expands, causing the indentation process to commence the elastic-plastic regime.

2.1.2. Indentation on elastic-plastic materials

Hill [5] proposed the solution for the quasi-static expansion of an internally pressurized spherical shell of perfect elastic-plastic material. Researchers have extended the expanding cavity models (ECM) based on Hill's work for indentation with different indenter tips or indented materials [6-8]. For conical indentation, based on von Mises yield criterion and the assumption of material incompressibility, ECMs can be expressed as:

$$\frac{H}{\sigma_y} = \frac{2}{3} \left[1 + \ln \left(\frac{1}{3} \frac{E}{\sigma_y} \tan \alpha \right) \right] \quad (2.10)$$

Where α is the cone angle of conical indenter.

Subsequently, R Narasimhan [9] proposed modified ECMs based on the Drucker-Prager yield criterion for the perfect elastic-plastic materials without considering the strain-hardening effect. After that, Gao et al. [10] extended ECMs for power law hardening and linear hardening materials.

Fig.2.3. shows the simplified stress field in the spherical indentation in expanding cavity models. The contact radius, a of the spherical indenter is surrounded by a hemispherical hydrostatic core. This core is surrounded by an incompressible hemispherical plastic zone with radius r_c . The plastic zone is also surrounded by an elastic zone. As the penetration depth increases, the volume of core will be occupied by the spherical indenter with radial expansion da , while, the volume of plastic zone expands in the elastic region at radial movement dr .

Fig. 2.3: Schematic of the simplified stress distribution in ECMs in spherical polar coordinates.

Using Henckey' deformation theory, the stress, strain and displacement in an internally pressurized spherical shell can be derived for Hollomon's power law hardening materials, in which both the von Mises yield criterion and the material incompressibility are assumed [11, 12]. In Hollomon's equations, the true stress strain curve from a uniaxial tensile or compress test can be expressed as:

$$\sigma = E\varepsilon \quad \text{for } \sigma \leq \sigma_y \quad (2.11a)$$

$$\sigma = K\varepsilon^n \quad \text{for } \sigma \geq \sigma_y \quad (2.11b)$$

Where E is the elastic modulus, σ_y is the yield stress, K is the strength coefficient and n is the strain hardening exponent.

In the plastic region, the stress, strain and displacement components can be expressed in the spherical polar coordinates (r, θ, ϕ) as:

$$\sigma_{rr} = \frac{2}{3}\sigma_y \left[\left(\frac{1}{n} - 1 \right) - \frac{1}{n} \left(\frac{r_c}{r} \right)^{3n} + \frac{r_c^3}{r_o^3} \right] \quad (2.12a)$$

$$\sigma_{\theta\theta} = \sigma_{\phi\phi} = \frac{2}{3}\sigma_y \left[\left(\frac{1}{n} - 1 \right) + \left(\frac{3}{2} - \frac{1}{n} \right) \left(\frac{r_c}{r} \right)^{3n} + \frac{r_c^3}{r_o^3} \right] \quad (2.12b)$$

$$\varepsilon_{rr} = -\frac{\sigma_y}{E} \frac{r_c^3}{r^3} \quad (2.12c)$$

$$\varepsilon_{\theta\theta} = \varepsilon_{\phi\phi} = \frac{\sigma_y}{2E} \frac{r_c^3}{r^3} \quad (2.12d)$$

$$u = \frac{\sigma_y}{2E} \frac{r_c^3}{r^2} \quad (2.12e)$$

Where r_c is the radius of the elastic plastic boundary, r_o is the out radius of the spherical shell.

The internal pressure in the inner part of the spherical shell can be expressed as:

$$\frac{p_i}{\sigma_y} = \frac{2}{3} \left[\left(1 - \frac{r_c^3}{r_o^3} \right) + \frac{1}{n} \left(\frac{r_c^{3n}}{r_i^{3n}} - 1 \right) \right] \quad (2.13)$$

When $r_o \rightarrow \infty$, the solution in Eq.(2.13) can be changed to:

$$\frac{p_i}{\sigma_y} = \frac{2}{3} \left[1 + \frac{1}{n} \left(\frac{r_c^{3n}}{r_i^{3n}} - 1 \right) \right] \quad (2.14)$$

In addition, due to the occupation of the indenter in the hydrostatic core as the penetration depth increases, the radical expansion of the hemispherical core for the spherical indentation can be expressed as:

$$\pi a^2 dh = 2\pi a^2 du \Big|_{r=a} \quad (2.15)$$

Where h is the indentation depth, we can obtain $h = R - \sqrt{R^2 - a^2}$,

By differentiating Eq. (2.12e), the rate of change of the radial displacement at any r in the plastic zone is:

$$du \Big|_{r=a} = \frac{3}{2} \frac{\sigma_y}{E} \frac{r_c^2}{a^2} dr_c \quad (2.16)$$

Incorporating Eq.(2.16) into Eq.(2.15), we can obtain:

$$3r_c^2 dr_c = \frac{E}{\sigma_y} \frac{1}{R} a^3 da \quad (2.17)$$

As the dh can be expanded through Taylor's expansion, integrating Eq. (2.15) can get:

$$\left(\frac{r_c}{a} \right)^3 = \frac{1}{4} \frac{E}{\sigma_y} \frac{a}{R} \quad (2.18)$$

By substituting Eq.(2.18) into Eq.(2.14), the expanding cavity models for the spherical indentation of power law hardening can be expressed as:

$$p_i = \frac{2}{3} \sigma_y \left\{ 1 + \frac{1}{n} \left[\left(\frac{1}{4} \frac{E}{\sigma_y} \frac{a}{R} \right)^n - 1 \right] \right\} \quad (2.19)$$

Similarly, the expanding cavity models for the conical indentation of power law hardening can be expressed as:

$$p_i = \frac{2}{3} \sigma_y \left\{ 1 + \frac{1}{n} \left[\left(\frac{1}{3} \frac{E}{\sigma_y} \cot \alpha \right)^n - 1 \right] \right\} \quad (2.20)$$

From Eq.(2.19) and (2.20), as the geometry of indenter is confirmed, the mean stress on the hemispherical core for Hollomon's power law materials is dependent on Young's modulus, E , yield stress, σ_y and hardening exponent, n . However, it should be noted that the mean stress, p_m compressed on the indenter is not totally converted into the hydrostatic pressure, p_i at the hemispherical core. Consequently, the actual mean pressure, p_m is not equal to the radial directional pressure at the core p_i . In order to estimate the indentation mean pressure, p_m , Johnson[6], Studman et al[13] suggested that extra stress (proportional to the yield stress) should be added to the radial directional pressure at the core p_i . More recently, Kang et al.[14, 15] studied again the expanding cavity model in a spherical indentation to evaluate the flow stress, and suggested that the difference between the mean pressure and the hydrostatic pressure is proportional to the flow stress measured from the tensile test as shown in Eq. (2.21).

$$p_m = p_i + k \sigma_t \quad (2.21)$$

The value of k in Eq. (2.21) ranged from 0.43 to 0.83 dependent on the material's properties, which was determined from the 13 metallic materials in the experiment.

Furthermore, the expanding cavity model simplifies the indentation process as the movement of a hemispherical cavity, which is different from the realistic indentation process. Plastic deformation should diversify the stress distribution at the boundary of the core. Most of all, the pile up effect is not considered in this model, the ECMs can be used only for shallow penetration. The materials, especially high E/Y or low hardening exponent easily show pile up at deep penetration depth. This effect could widen the contact area and disperse the stress distribution beneath the indenter. Kang et al.[14, 15] tried to reduce the pile up effect by

measuring the residual indentation. Jiang et al. [16] also introduced a correction factor related to the indentation value to take this pile up into account. However, it should be noted that these corrections are limited to a certain range of selected material properties. Due to the complexity of the physical process in pile up, the possibility of extracting the elastic properties of materials from the expanding cavity models seems difficult.

2.1.3. Sneddon's solution

In the elastic indentation with a punch indenter, the contact area is constant, which is equal to πa^2 in the indentation process. Sneddon found a simple equation between the load, F and the penetration depth, h [17]:

$$F = \frac{4\mu a}{1-\nu} h \quad (2.22)$$

Where a is the radius of punch indenter, μ is the shear modulus.

Then by differentiating Eq.(2.22), we can obtain:

$$S_u = \frac{2}{\sqrt{\pi}} \sqrt{AE_r} \quad (2.23)$$

Where $\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i}$. $S_u = dF/dh$ is the initial unloading stiffness.

From Eq.(2.23), Young's modulus could be estimated from the initial unloading slope in the Sneddon analysis. G.M.Pharr et al.[18] extended the Sneddon analysis for the indentation of an elastic half space by any punch described as a solid of revolution of a smooth function. Consequently, the measurement of Young's modulus using contact stiffness, S_u , and the contact area can be done with an arbitrary indenter. Conversely, the contact area can be estimated indirectly when the elastic properties and the contact stiffness are known.

As regards the correction of the Sneddon equation, two correction factors should be considered based on numerical analysis[19]. One factor is related to the bodies of revolution. King [20] proposed that a correction should be added to the right-hand part in Eq.(2.23), for a triangle, it is 1.034, and for a square 1.012. The other factor γ is related to inward radial displacements of materials under the indenter. Li et al.[21] used Sneddon analysis to make

comparison between numerical and experimental results on five materials and found that this equation can only predict the trend of contact radius but could not determine very accurate values. J.M. Collin et al. [22] explained that this deviation might be attributed to the use of elastic- perfectly plastic model in simulation without considering the strain hardening and argued that this equation does not take into account the inward radial displacements of materials under the indenter. They used the formulation proposed by Hay and Woff [23] by introducing a fact γ to determine the contact radius during spherical indentation as:

$$a = \frac{3\pi R(\sqrt{\Delta h} - 1)}{4B} \quad (2.24)$$

Where $\Delta h = 1 + \frac{4BS_u}{3\pi RE_r}$, $B = \left[\frac{N(1-2\nu)(1+\nu)}{N(1-\nu^2) + (1-\nu_i^2)} \right]$, $N = \frac{E_i}{E}$,

For the spherical indenter: $\gamma = 1 + \frac{2a}{3\pi R} \left[\frac{N(1-2\nu_s)(1+\nu_s)}{N(1-\nu_s^2) + (1-\nu_i^2)} \right]$

For the conical indenter: $\gamma = 1 + \frac{2(1-2\nu)}{4(1-\nu)\tan\alpha}$

It should be noted that when using Eq.(2.24) to estimate the contact radius, the value of a/R is limited to 0.25 according to their experimental observations.

2.2. Measurement of the elastic properties for metallic materials by indentation

2.2.1 Estimation of contact area from unload curve

The most famous method for extracting the elastic properties is the Oliver-Pharr method (Multiple-point) [24]. Fig.2.4. shows the schematic cross section of indentation and corresponding indentation loading-unloading versus penetration depth.

In Fig.2.4.(a), the penetration depth, h , consists of the contact depth, h_c , and the displacement of surface at the perimeter of the contact.

$$h = h_c + h_s \quad (2.25)$$

Where $h_s = \varepsilon \frac{P_{\max}}{S_u}$, $\varepsilon = 1$ for the flat punch in the Sneddon equation, for the conical and paraboloid indenters, $\varepsilon = 0.72$ and 0.75 , respectively.

Fig. 2.4:(a) Schematic cross section of indentation (b) full indentation load-unload versus penetration depth [24]

The contact stiffness can be measured from the slope of the tangent line to the unloading curve at the maximum loading point. The relationship between penetration depth, h , and indentation force, P , during unloading can be expressed as:

$$P = B(h - h_f)^m \quad (2.26)$$

Where B , m and h_f can be determined by the least square fitting procedure.

The initial slope at the maximum load, P_m can be expressed as:

$$S_u = Bm(h_m - h_f)^{m-1} \quad (2.27)$$

The compliance of the load could be estimated prior to the measurement. The total measure compliance C , involves the compliance of the load frame, C_f and the specimen compliance, S_u .

$$C = C_f + \frac{\sqrt{\pi}}{2E_r} \frac{1}{\sqrt{A}} \quad (2.28)$$

As the contact depth and the geometry of indenter are confirmed, the contact area can be estimated through an iteration process when the indent on the material is made with the known Young's modulus. Normally, fused silica is used as the reference material. The relationship between the contact area and the contact depth can be approximated as Eq. (2.29):

$$A = c_0 h_c^2 + c_1 h_c^1 + c_2 h_c^{1/2} + c_3 h_c^{1/4} + c_4 h_c^{1/8} + c_5 h_c^{1/16} \quad (2.29)$$

Where the first term $c_0 = 24.5$ for an ideal Berkovich tip, $c_0 = 2.598$ for ideal cube corner tip and $c_0 = -\pi$ for the ideal conical-spherical tip.

As regards the Oliver-Pharr methodology, the main problem is the influence of the pile up effect, which results in the underestimation of the contact depth in Eq.2.25 and the overestimation of Young's modulus. The pile up effect will be discussed in Section 2.4.1. As a result, researchers are trying to find other methods to measure the elastic properties without knowing the contact area.

2.2.2 Loading curve

Several researchers have attempted to investigate the shape of the loading curve. Their studies concluded that the loading curve for an ideal sharp indenter should follow Kick's equation [25-27]:

$$F = Ch^2 \quad (2.30)$$

The indentation load, F shows a linear relationship with the square of the indentation depth h , and C is the curvature of the loading curve. This value can be obtained from the experimental force-penetration curves. Several efforts have been made to obtain an analytical expression for the C parameter. Hainsworth and Page [28], Hainsworth and Chandler [29] determined an expression for C by combining Young's modulus and the hardness :

$$C = E \left(\Phi_m \sqrt{\frac{E}{H}} + \Psi_m \sqrt{\frac{H}{E}} \right)^{-2} \quad (2.31)$$

In equation (2.31), E and H are Young's modulus and the hardness, respectively, and Φ_m and Ψ_m are empirical parameters obtained from experiments using known materials property. These empirical parameters are only dependent on the type of indenter used; for a Berkovich indenter, $\Phi_m = 0.194$ and $\Psi_m = 0.930$. The effect of the blunt tip and indentation size can be ignored at deeper penetration depths. K. K. Jha et al. [30] proposed an analytical method for determining the empirical parameter for the indenter in the Hainsworth model. Then,

J. Malzbender et al. [31] derived an analytical expression for the $P - h^2$ relationship, which was verified experimentally [32, 33]:

$$F = E_r \left(\frac{1}{\sqrt{c_0}} \frac{\sqrt{E_r}}{\sqrt{H}} + \frac{\varepsilon}{\beta} \sqrt{\frac{\pi}{4}} \frac{\sqrt{H}}{\sqrt{E_r}} \right)^{-2} h^2 \quad (2.32)$$

M. Troyon and M. Martin improved the expression formulated by Malzbender by taking two correction factors into account [34]. One factor had been pointed out by Hay et al [23, 35] in previous discussion on Sneddon's solution in 2.1.3. The other factor is ε , related to indenter geometry, and can be expressed as [36]:

$$\varepsilon = m \left\{ 1 - \frac{m-1}{m \left[\chi \left(\frac{m}{m-1} \right) \right]} \right\} \quad (2.33)$$

Where: $\chi(t) = \int_0^1 \frac{x' dx}{\sqrt{1-x^2}}$, m is the index from the curve fitting of the unloading curve in

Eq.(2.26).

However, their analytical expressions involve two parameters, i.e., hardness and Young's modulus. Consequently, the model could only be used to estimate either E or H when one of these parameters is known. In practice, neither hardness nor Young's modulus is known; thus, it is not convenient to use this model.

2.2.3 Energy method

The energy method is an alternative way of extracting the material's properties from the loading and unloading curves in indentation process. Fig.2.5. shows indentation curves of loading-unloading for elastic plastic materials, in which W_{tot} is the total work, W_p is the irreversible work, W_e is the reversible work.

Using dimensional analysis and the finite element method, Cheng, Y.-T., et al. [37, 38] proposed linear fitting related to the work of indentation, and material's properties. For the conical indenter, the ratio of reversible, W_e , to the total work, W_t , has a linear relationship with the ratio of hardness to the reduced Young's modulus, E_r .

$$\frac{H}{E_r} = \kappa \frac{W_e}{W_{tot}} \quad (2.34)$$

Where $\lambda = 1.50 \tan(\theta) + 0.327$ $\kappa = 1/[\lambda(1 + \gamma)]$ for $60^\circ \leq \theta \leq 80^\circ$.

Fig. 2.5: Schematic diagram of the loading-unloading curve of the indenter, W_{tot} is the total work, W_p is the irreversible work, W_e is the reversible work.

By combining Eq. (2.23) and (2.34) to eliminate contact area, A , reduced Young's modulus, E_r can be estimated. Soon afterwards, D Ma et al.[39] investigated the relationship between Young's modulus and non-ideally sharp indentation parameters. Based on an analytical analysis and the finite element analysis, they concluded that there is an approximated one to one correspondence between the ratio of nominal hardness/reduced Young's modulus and the ratio of elastic work/total work for any definite bluntness ratio of a non-ideally sharp indenter. For the spherical indenter, Ni.W et al. [40] used a similar method and deduced:

$$\frac{H}{E_r} = 0.5928 \left(\frac{h_{max}}{R} \right)^{0.62} \left(\frac{W_u}{W_t} \right) \quad (2.35)$$

Where values of h_{max}/R range from 0.05 to 0.5.

Therefore the equations to estimate the reduced Young's modulus and hardness explicitly can be expressed as:

$$E_r = \frac{1}{F_{max}} \left[0.4657 \left(\frac{dF}{dh} \right)^2 \left(\frac{h_{max}}{R} \right)^{0.62} \frac{W_e}{W_t} \right] \quad (2.36)$$

$$H = 0.276 \left(\frac{h_{\max}}{R} \right)^{1.24} \left(\frac{W_e}{W_t} \right)^2 \left(\frac{dF}{dh} \right)^2 \frac{1}{F_{\max}} \quad (2.37)$$

However, J. Alkorta et al. [41] argued that there is a significant deviation in Eq.(2.34) linearity. The parameter κ is dependent on the pile up and hardening exponent. It also showed that the deviation related to the energy ratio method could be even higher than that related to the Oliver Pharr method for soft materials.

2.3. Mechanical characterization of metallic materials through spherical indentation.

2.3.1. Spherical indentation regime.

Many pioneers have studied the spherical indentation regimes [6, 42-50]. Generally, the spherical indentation process can be divided into three distinct regimes: elastic, elastic-plastic and fully plastic (illustrated in Fig. 2.6).

Fig. 2.6: Schematic representation of the plastic zone expansion during the spherical indentation process: (a) elastic, (b) elastic-plastic, (c) fully plastic[51]

2.3.1.1. Elastic regime

In the elastic regime, the indented materials can be recovered after unloading. From the curve of mean pressure versus load, Tabor [42] found that the initial yield point corresponding to the onset of plastic deformation which occurred when $P_m = 1.1\sigma_y$. Johnson [6] proposed a dimensional parameter $A = E_r/a(\sigma_y/R)$ and proved theoretically that the elastic regime was valid

up to $A=2.53$. This regime is consistent with the Hertz elastic theory. The researchers verified these conclusions with experimental data or FEM and there is no disagreement in this regime.

2.3.1.2. Elastic-plastic regime

In this regime, the mechanical material response consists of the elastic and plastic regimes. The mechanical state in the indentation is different from the uni-axial tensile or compression state. There have been many studies related to the boundary of the elastic plastic regime. The controversial discussion is mainly concentrated on the subdivision of elastic-plastic transition and the boundary between the elastic-plastic and full plastic regime. As the increase in indentation load exceeds the elastic limit and the onset of plastic deformation occurs, the plastic zone expands upward and outward, which is constrained by surrounding elastic materials. Park et al.[48] observed that the elastic-plastic transition can be divided into two parts: the elastically dominated part ($1.07 \leq P_m/\sigma_r \leq 2.1$), and the plastically dominated part ($2.1 \leq P_m/\sigma_r \leq 3$) which is dependent on the work hardening property. They also found a ‘pseudo-Hertzian’ ($1.07 \leq P_m/\sigma_r = 1.5$) behavior in the elastically dominated transition, which had also been observed by Mesarovic and Fleck[47]. The existence of the ‘pseudo-Hertzian’ explained why the plastic zone did not break through the indented surface. Fig.2.7 shows the schematic of regime division based on the finite element observations [48].

Fig. 2.7: Schematic representation of the relationship between p_m/σ_r and $E^*a/\sigma_y R$ based on the finite element observations[48].

However, O.Bartier and X.Hernot [49] studied the parabolic and spherical indentation phenomenologically and pointed out that there is no ‘pseudo-Hertzian’ for both indenters from the analysis in FEM, they also pointed out that ‘pseudo-Hertzian’ was not caused by the break in the plastic zone through the indented surface according to the FEM observation. Additionally, they proposed two novel non-dimensional expressions: $(dF/da)*(a/F)$ and $(da/dh)*(h*a)$, to study the indentation regime. Z. Song and K.Komvopoulos [50] had also theoretically studied the post-yield deformation in FEM which can comprise a four deformation regime: linear elastic-plastic, nonlinear elastic-plastic, transient fully plastic, and the steady-state fully plastic. Due to the complex indentation process in the elastic plastic regime, there is no agreement on the boundary in this regime. The material with different properties should manifest a different boundary of elastic plastic regime.

2.3.1.3. Indentation in fully plastic regime

As regards the boundary of fully plastic regime, there are no unified values to define the boundary between the elastic-plastic and full plastic regime. The majority of empirical values are based on simulation or limited experimental data.

Johnson [44] showed the initial fully plastic at $\Lambda = E_r/a(\sigma_y/R) = 40$; Mesarovic and Fleck [47] showed the initial fully plastic at $\Lambda = E_r/a(\sigma_y/R) = 40-50$ for elastic perfect plastic materials. Park and Pharr showed the fully plastic regime began at $\Lambda = E_r/a(\sigma_y/R) = 50-200$; Plane and blank [52] found the boundary between the elastic-plastic regime and fully plastic regime was at $\Lambda = E_r/a(\sigma_y/R) = 80$ for different kinematic and isotropic materials. O.Bartier and X.Hernot studied the materials with high E/σ_y ratio, when $\Lambda = E_r/a(\sigma_y/R) = 220$, the indentation behavior reached full plastic regime.

2.3.2. Determination of plastic property through spherical indentation based on representative strain

In 1908 Meyer had found that for many materials, the mean pressure increased with a/R according to the simple power law [53]:

$$p_m = k \left(\frac{a}{R} \right)^m \quad (2.38)$$

Where, p_m is the mean pressure, a/R is ratio of indentation radius to the ball radius, m is the Meyer' index.

In Eq.(2.38), k and m are constants. Meyer's equation was verified by further experimental studies[42, 54] and they suggested that the Meyer index, m , was related to the hardening exponent, n . The value of the Meyer index, m is approximately equal to $n+2$.

Following Meyer's work, Tabor suggested that the ratio a/R represented the indentation strain. He assumed that the mean pressure, p_m in the fully plastic regime was proportional to the representative stress, σ_r , and the impression radius was proportional to the corresponding representative strain, ε_r . Consequently, the representative stress and strain can be expressed as[42]:

$$\varepsilon_r = \beta \frac{a}{R} \quad (2.39)$$

$$\sigma_r = \frac{F}{\psi \pi a^2} \quad (2.40)$$

Where β is indentation strain constraint fact, a is contact radius, R is the radius of indenter, ψ is indentation stress constraint fact, F is the indentation load.

Tabor determined the parameter β and ψ from empirically experimental data with a spherical indentation. Generally, the indentation strain constraint β is equal to 0.2 and the indentation stress constraint ψ is equal to 2.8-3.2. It should be emphasized that the representative strain defined in the Tabor equation is an average value. The strain constraint fact, β , should vary with the depth of the indentation for which the tangent of corresponding contact point could be equal to the same angle of sharp indenter [6, 55]. Most of researchers agreed with the formulation of Tabor's equation, but argued the choice of the value of ψ and β [56-62].

O.Richmond et al.[57] used the finite difference method for solving the indentation problem in rigid-perfectly plastic materials to evaluate Tabor's equation. They predicted that throughout the penetration the mean pressure was equal to 3 times the yield stress and the stress constraint factor, β was about 0.32. They concluded that the discrepancy between their result

and Tabor's equation was due to not taking the work hardening in the theory model for rigid-perfectly plastic materials into account.

Lee,CH et al [63] used the finite element method to compute the elastic-plastic solution of ball indentation with steel SAE4340, and gave the value of 2.77 as the stress constraint factor in the full plastic regime. They also compared the average strain defined by O.Richmond with the plastic zone volume under the indenter. They found that the proportionality factor between the mean effective strain and the constraint radius is not constant and varies from 0.2 to 0.12 over the range of a/R up to 0.3, which indicated that the strain constraint factor defined by Tabor is an average value in the indentation process. Their analysis supported Tabor's equation. A similar evaluation using FEM had been made by G.B.Sinclair et al [64].

Y.Tirupataiah et al [45, 65] evaluated the empirical equation of Tabor using experiment data and concluded that the stress constraint factor CF determined in the case of iron, steel, copper and its alloys, aluminum alloy in solution-treated, aged and averaged conditions, lay within the range of 2.4 to 3.0. They also extended Matthews' analysis of indentation for work hardening fully plastic material by a sphere [66]. The stress constraint factor can be expressed related the hardening exponent:

$$\psi = \frac{6}{2+n} \left(\frac{40}{9\pi} \right)^n \quad (2.41)$$

There was a discrepancy between the theoretical stress CF and the experimental result. In the case of steel, on an aged aluminum alloy, and Cu-Al, the theoretical stress CF can give a good prediction. However, in the case of materials exhibiting a low strength value, like iron, copper, Cu-Zn, the model in Eq.(2.41) gave a poor prediction. Moreover, through examining the experimental data of data from steel and copper, he pointed out that there was an obstacle when using Tabor's equation, the stress CF was taken within the range of 2.6-3, which resulted in a relatively large variation for the value of the strain CF. On the other hand, if the strain CF was taken as 0.2, it also made the large variation in the value of the stress CF [54].

Taljat et al.[46] analyzed the spherical indentation process using the finite element method for steel with a hardening exponent in the range from 0 to 0.5. They evaluated Tabor's empirical

equation and calculated that the stress constraint factor was equal to 3 for the hardening exponent 0, it decreased to below 3 for materials with a high hardening exponent, which was consistent with the report in Tirupataiah [45]. Recently, Yetna N’jock et al. [67] simplified this numerical result and proposed a methodology to extract the stress strain curve from the macro spherical indentation for the metal of steel and brass, in which two kinds of Hollomon and Ramberg-Osgood power law constitutive equations were considered, respectively.

Nevertheless, some researchers argued the formulation of representative strain in Tabor’s equation. Milman et al. [68] defined the representative strain by analyzing the variation in area during indentation. In the fully plastic regime, the average strain in the direction of depth can be expressed as:

$$\varepsilon_r = -\int_{s_1}^{s_2} \frac{dS}{S} = -\ln \frac{a}{2Rh_c} \quad (2.42)$$

It is not necessary to define the extra empirical parameters in this definition. Recently, K Fu et al.[69] used the definition of representation strain by Milman and proposed an iterative method to extract the flow stress curve using spherical indentation. In their study, the stress constraint factor was taken as 3. It seems very robust, however, they did not extract the specific value of the material properties, only compared the shape of the representative stress strain curve obtained from spherical indentation with that of the nominal stress strain curve.

Ahn et al. [51] developed a shear strain definition by differentiating the displacement in the direction of depth. The equation is:

$$\varepsilon_r = \frac{\beta}{\sqrt{1 - (a/R)^2}} \frac{a}{R} \quad (2.43)$$

Where β is the material independent constant, the value is 0.1.

Xu et al.[60] used the formulation of indentation strain in Ahn et al. [51] and proposed a methodology to extract the engineering stress strain curve from the spherical indentation. With the help of finite element simulation in a defined range of metallic materials, they found the indentation stress constraint factor is only dependent on the hardening exponent, n , which is consistent with the previous experimental and numerical studies [45, 46, 66]. However, the

indentation strain constraint factor depends on both n and the ratio of E to σ_y . As regards the experimental measurement in practice, this method needs abundant computation with FEM, which is not convenient and has the limitation of the dimensionless method. Additionally, the ambient factors, such as the imperfect indenter tip, residual stress, roughness of specimen, have not been considered.

Recently, Klidindini et al. [70, 71] defined a new formation of indentation strain which is consistent with Hertz theory. The new definition of indentation strain has also been evaluated using finite element simulation. From the experimental data in CSM, the new method can not only estimate Young's modulus of indented materials in the shallow penetration depth and capture the harden property, but the influence from the effect radius of imperfect indenter is also avoided. The new indentation strain is:

$$\varepsilon_r = \frac{h}{2.4a} \quad (2.44)$$

Although this method manifests more physical meaning, they have not given the indentation stress factor. The method can only estimate the tendency of representative stress strain curve approximately.

From the literature, there is no uniform agreement among researchers as regards the relationship between the mean pressure and the extent of the indentation. Additionally, most of the strain constraint factor or stress factor in these definitions is obtained from empirical experimental data or FEM simulation data. More research into the methodology for extracting flow stress is needed in future.

2.3.3. Another methods to extract flow stress by spherical indentation

2.3.3.1. Dimensional method

Dimensional analysis is widely used to explore the terms of indentation with the help of finite element analysis[72]. This method needs a large number of FEM computations within a certain range of the material properties defined as the power law equation. The dimensional function has established a relationship between the indentation characteristics and material properties [16, 73-80]. Π theorem is the key theorem in dimensional analysis, which could reduce

complex physical problems to their simplest form [81, 82]. If the physically meaningful equation involving Γ independent physical variables, and they can be expressed in terms of r independent physical units, the original equation can be rewritten in terms of $n-k$ dimensional parameters $\Pi_1, \Pi_2, \Pi_3, \dots, \Pi_{\Gamma-r}$. In the analysis of the dimensional method for spherical indentation, the indentation load, F should be a function of the independent parameters of the material properties [83]:

$$P = f(E_r, \sigma_r, n, h, R) \quad (2.45)$$

Where σ_r is the representative stress.

By apply the Π theorem, Eq. (2.45) can be changed to:

$$P = \sigma_r h^2 \Pi_1\left(\frac{E_r}{\sigma_r}, n, \frac{h}{R}\right) \quad (2.46)$$

Other indentation responses can also be used to develop the dimensional function, such as, load curvature, elastic energy, exponent of loading curve, contact stiffness, etc. After that, an artificial neural network (ANN) was combined with the dimensional function to predict the material properties [84, 85]. However, there are shortcomings in this method [86, 87]. The dimensional function is established with the assistance of FEM, in which the indentation process is simulated in an ideal state. The real experimental environment is not considered totally in the simulation: the roughness of the surface, the imperfect indenter, the noise of the machine, the influence of friction etc [59]. Consequently, using dimensional functions deduced from FEM in a real experimental test is suspicious. Moreover, the uniqueness of the dimensional function is a controversial topic. It is very complex and the time consumed to perform the FEM computations to develop the dimensional functions, which are mainly dependent on the range of materials in the simulation. It should be noted that the majority of studies related to the dimensional method use the power law hardening model in their in FEM model, although in real materials the constitutive equation might be different. Nevertheless, the dimensional analysis can give useful functions when combining the material parameters and indentation response and give a theoretical guideline for the experimental study.

2.3.3.2. Obtention of material properties from the load-displacement curve

Unlike the unloading-displacement curve, there is more information related to material properties from the loading curve. Moreover, the shape of the loading curve is not greatly influenced by the friction coefficient [88]. The physical parameters extracted from the unloading curve in the experimental data, e.g., contact stiffness, are not as stable as those obtained from the loading curve, e.g., load curvature. Many researchers have tried to extract the material properties from the load-displacement curve [89-93].

The most direct inverse method is to compare the loading curve from the experiment to that obtained from the finite element simulation. Based on the optimization algorithm and a given constitutive equation, the unknown material properties can be extracted using the optimization process. Another method is to define the analytical equation to describe the loading curve, in which the unknown material parameters are involved. Derivated from the Oliver-Pharr method, Malzbender et al.[92] proposed that the loading curve be expressed as:

$$h = \sqrt{\frac{F}{E_r}} \left[\frac{1}{2\pi R f(n)} \frac{\sqrt{FE_r}}{\sigma_r} + 0.75 \frac{\sqrt{\sigma_r}}{\sqrt{4/\pi E_r}} \right] \quad (2.47)$$

Where $f(n)$ is function of the hardening exponent that takes into account pile up or sink in[94].

Nayebi et al. [90] assumed that the load curve could be expressed as:

$$h = A(\sigma_y, n) F^{B(\sigma_y, n)} \quad (2.48)$$

Where A, B are dependent on the yield stress and strain hardening exponent.

The functions of A and B were confirmed by the finite element simulation, in which Young's modulus was fixed, the yield stress and the hardening exponent were selected from within a defined range. A similar analytical equation was developed by Beghini et al. [91]. This method with analytical formation has more physical meaning than the dimensional method. However, indented material with different properties might have the same loading-displacement response [95], the problem of uniqueness of solution still exists in this method. Otherwise, the functions are dependent on the selected material properties, which could restrict the universal utility.

2.4. Contamination in the experimental data process

In the instrumented depth sensing technique, the contact area can be estimated from the load-unloading curve. Many factors could influence the extraction of material properties, for instance, the roughness of the specimen, the friction ratio between the indenter and the indented material, tip rounding, the pile up and sink in [96-99]. In this section, the contamination which could be encountered in the experimental data process will be discussed and summarized.

2.4.1. Pile-up or sink-in phenomenon

Pile up or sink in is a very complex physics phenomenon in the indentation process. The extent can be evaluated by $c^2 = a_c^2 / 2Rh$, which is mainly dependent on the value of the hardening exponent, n [46, 94, 100]. $c^2 < 1$ implies that the material shows sinks-in, while $c^2 > 1$ means that material shows piles up. In Eq. (2.25) and Fig.2.4 (a), the contact depth, h_c is always lower than the total penetration depth, h . However, the pile up phenomenon will take place when the indenter is applied on soft materials. Fig.2.8 shows obvious pile-up in three-dimensional AFM topography images [101]. Obviously, Eq.25 cannot correctly describe the real relationship between the contact depth, h_c , and the penetration depth, h .

Fig. 2.8: AFM image for typical indentations on the Au thin film, three dimensional views[101]

With the help of finite element simulation, A.Bolshakov and G.M.Pharr [102] found that when the pile up was obvious, the contact area estimated from analyses of the load-displacement curves underestimated the true contact area by as much as 60%, which consequently leads to the hardness and elastic modulus being overestimated. In their conclusion, they advised that when the ratio of residual depth to maximum depth is $h_f/h_{\max} < 0.7$, the pile up

is not significant, and the Oliver-Pharr can method can give a reasonable result. Recently, M. Yetna N’jock et al. [103] proposed a similar criteria from experimental data, defined as the ratio of the residual indentation depth to the indentation depth at the maximum load. For materials in which the ratio is equal to 0.83, the Oliver-Pharr method can lead to an accurate contact area. When the ratios are higher or lower than 0.83, pile up or sink in dominates, respectively. M. G. Maneiro [104, 105] proposed a new procedure for determining Young’s modulus of elastic plastic materials using a spherical indenter, which is not affected by the pile up or sink in. When they assumed that the radius is constant during the unloading process, they derived that the residual impression radius can be expressed as:

$$R_r = R_i \left(\frac{h_t + h_r}{2h_r} \right) - \frac{1}{8} \frac{(h_t + h_r)^2}{h_r} + \frac{h_r}{2} \quad (2.49)$$

As the residual radius confirmed, the combined radius can be calculated by:

$$\frac{1}{R} = \frac{1}{R_i} - \frac{1}{R_r} \quad (2.50)$$

Young’s modulus of material can be obtained by the slope of the linear fitting between $P/h_e^{3/2}$ versus the combined radius, R in Hertz’s equation. Although this method is robust, its validity is limited when the pile up or sink in phenomenon is significant.

Care should be taken on the measurement on soft materials with a high E/σ_y value or low hardening exponent, n by the nanoindentation technique. Although the contact area can be measured using AFM, it would increase the cost of the measurement.

2.4.2. Measurement of contact stiffness

2.4.1.1. The slope of the initial unload curve

Contact stiffness is a very important parameter when using depth sensing indentation. The contact stiffness can be estimated from the slope of the initial unload curve through the power law fitting process in Eq.2.27. There is some discussion on the extraction of contact stiffness from the slope of the initial unload curve [106].

Oliver Pharr concluded that the paraboloid was the preferred geometry for the Berkovich indenter and the power law index for the unloading curve ranges from 1.25 to 1.51 [24]. G.M.Pharr and A.Bolshakov [106] proposed a simple model using the concept of the effective shape based on a finite element and found that the effective indenter was dependent on the material properties, E/H , which contributed to the variety of m in the power law fitting. However, from the FEM analysis, the Berkovich indenter is equal to the conical indenter with a cone angle of 70.3° , in which the power law index for the unloading curve is a constant value ($m=2$). Gong et al. [107] performed the indentation test on three typical brittle materials with a Berkovich indenter and argued that the unloading data of Berkovich indenter should be based on the conical indenter ($m=2$) by introducing an extra term to describe the effect of the residual contact stress.

Moreover, there were studies related with the proper fitting method to obtain the slope of the initial unload curve. From the finite element analysis, V.Marx and H.Balke[108] advised that the slope be obtained from the tangent through the upper 5% of the curve but not to power fitting the whole unloading curve. The linear fitting on the upper 33% of the unload curve can also obtain a reasonable value. Jianhong Gong et al.[109] also proposed that the unloading curve be expressed as:

$$\frac{P}{h} = a_1 + a_2 h \quad (2.51)$$

Then the initial unloading curve was determined as:

$$S_u = a_1 + 2a_2 h_{\max} \quad (2.52)$$

In addition, the incomplete or irregular unloading curve is very commonly observed. Hu Huang and Hongwei Zhao [110] presented a partial fitting method by fitting a portion of the unloading data corresponding to about 10-40% of the maximum indentation load in the unloading curve to determine the residual depth h_f . Alternatively, based on the energy method, K.K.Jha et al.[111] proposed a new expression of contact stiffness which was a function of the elastic energy constant and was independent of the peak indentation load.

2.4.1.2. Continuous Stiffness Measurement (CSM)

An alternative method for measuring the contact stiffness is to use the continuous stiffness measurement. The continuous stiffness is determined by superimposing a small oscillating excitation force amplitude F_o at a frequency ω on top of the quasi-static applied load and can be expressed as[97]:

$$S_u = \left[\frac{1}{\frac{F_o}{z_o} \cos \phi - (K_s - M\omega^2)} - \frac{1}{K_f} \right]^{-1} \quad (2.53)$$

Where F_o is the oscillating excitation force amplitude; z_o , the oscillating excitation displacement amplitude; K_s , the support spring stiffness; M , the mass of the indenter column; and K_f , the frame stiffness.

In the Continuous stiffness measurement (CSM), the amplitude and frequency of the superimposed oscillation mostly affect the data collection system [112]. The CSM with 45 Hz oscillation frequency and 2 nm amplitude would normally produce reasonable values of contact stiffness [97]. The inherent noise should also be considered, which would scatter the post data analysis. The effective contact point should be identified at the initial indentation. Fig.2.9 (a) shows that the noise of the stiffness at the initial portion is worse than that in the middle part of the harmonic stiffness-depth curve. Meanwhile, the worn tip at the shallow penetration depth could pollute the raw experimental data in the continuous stiffness measurement. To avoid the noise in CSM, the raw data needs to be smoothed in the post data process. Fig.2.9 (b) shows that the relationship between the harmonic stiffness, S_u , versus the indentation load, F , in a natural logarithm is almost linear. Consequently, the raw experimental data can be smoothed by the fitted linear regression for the harmonic stiffness, S_u , versus the indentation load, F (in a natural logarithm $\ln$ - $\ln$ plot) . Fig.2.9 (a) also shows the plot of the harmonic stiffness, S_u , versus the indentation load, F , (in $\ln$ - $\ln$ plot) fitted from the penetration depth 10nm. By back-extrapolating the fitted curve, the smooth harmonic stiffness-depth curve can be obtained.

Fig. 2.9: (a) harmonic stiffness-depth curve for measurement on steel vs the harmonic stiffness fitting curve (b) the linear fitting of harmonic stiffness, S_u , versus the indentation load, F , in a natural logarithm.

2.4.3. Deficient indenter

Due to the manufacture or the period of use, the indenter tip may be imperfect. Figs. 2.10 and 2.11 show the imperfect indenters of the Berkovich and conical-spherical indenter under AFM and SEM, respectively.

Fig. 2.10: Three-dimensional AFM rendering image of the lightly (a) and heavily (b) used indenters, and contour plot of the surface of the lightly (c) and heavily (d) used indenter shape in the near-apex region with 2 nm contour spacing after interpolation and filtering [113]

Fig. 2.11: Scanning electron microscope image of a worn diamond spherical-conical indenter (dotted line – ideal radius) [61].

Both the shape and radius of indenter tip can be changed with time. S.J. Bull reported that the radius of the Berkovich indenter (which was approximately 50 nm when new) increased by about 1nm per sample tested over a period of six months [114]. As a result, a periodical calibration is recommended. Prior to the measurement, the calibration of the tip is inevitable, which could be done using the iterative method in the reference materials with known elastic properties in the Oliver-Pharr method. However, this method has some limitations. This calibration procedure in Eq. 2.29 has large errors associated with its application to data obtained from materials which show significant pile up or sink in at the perimeter of the indentation. Furthermore, the computation of the true value of the contact area A_c is a controversial issue [115].

As regards the measurement on metallic materials using spherical indentation, the effect indenter radius should be considered [116-118]. As the geometry of the spherical indenter is imperfect, the effective indenter radius at a given depth is different from the nominal radius. Fig.2.12 shows how an imperfect sphere affects the radius of the effective sphere.

According to the geometric relationship, the effective radius indenter, R_{eff} can be expressed as [116-118]:

$$R_{eff} = \frac{a^2 + h_c^2}{2h_c} \quad (2.54)$$

In which, the contact depth, h_c and contact radius, a can be measured using a profiler instrument or the Oliver-Pharr method on the reference fused silica material.

Fig. 2.12 : Schematic of the relationship between the imperfect sphere and the effective sphere[118]

Conclusion

In this chapter, the basic theories related to the indentation in different regimes and the latest methodologies to determine the elastic-plastic properties of metallic materials, together with the contamination in the experimental data are summarized. From the literature, the contact mechanism under the indenter is still unknown to us, especially when the indentation state is beyond the elastic regime. As regards the methodologies, there are still deficiencies in them, although they were widely validated using experimental data or FEM simulation. However, some empirical parameters in the methods are based on certain experimental data or FEM simulation, but a lot of contamination has not been considered, which would lead to large variations in the results. In all, it is necessary to develop a more stable and convenient analytical procedure for the instrumented indentation technique.

Chapter 3 : A novel methodology for determining Young's modulus of elastic-plastic materials using instrumented sharp indentation

3.1. Introduction

In cases in which it is necessary to determine the local properties of engineering materials, or in those cases in which it is necessary to characterize coatings, interlayers or films mechanically, the instrumented sharp indentation technique is presented as the best alternative, and in some instances, the only one for estimating their mechanical properties [119]. The main mechanical properties measured by this technique are the elastic modulus, E , and hardness, H [24, 120]. The peak load, P , the maximum penetration depth, h , the residual depth after unloading, h_r , and the slope of the upper portion of the unloading curve, known as the contact stiffness, S_u , are the parameters that can be measured directly from the experimental indentation curves. The fundamental equations used to obtain H and E by indentation tests, are:

$$H = \frac{P}{A_c} \quad (3.1)$$

Where P is the load and A_c is the projected contact area at that load; and

$$E_r = \frac{\sqrt{\pi}}{2\beta} \frac{S_u}{\sqrt{A_c}} \quad (3.2)$$

Where E_r is the reduced modulus and β is a constant that depends on the geometry of the indenter. Additionally, E_r can be related to the elastic modulus of the indented material through:

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \quad (3.3)$$

Where E and ν are the Young's modulus and Poisson's ratio, respectively, for the bulk material and E_i and ν_i are the Young's modulus and Poisson's ratio, respectively, for the indenter.

However, the success of equations (3.1) and (3.2) to provide the actual values of hardness and elastic modulus depend on the precision with which the projected contact area is estimated. This area can be estimated by the indirect measurement of the last point of contact between the indented material surface and the indenter. This point is determined by the geometry of the indenter and the penetration depth, h , through the contact depth, h_c

$$h_c = h - h_s \quad (3.4)$$

Additionally, h_s can be derived from equation (3.5):

$$h_s = \varepsilon \frac{P}{S_u} \quad (3.5)$$

Where ε is constant that depends on the indenter geometry. Elastic contact analysis shows that $\varepsilon=0.75$ for sharp indenters [121]. From the calibration procedure, A_c versus h_c is can be expressed according to Eq. (3.6).

$$A_c = c_0 h_c^2 + c_1 h_c + c_2 h_c^{\frac{1}{2}} + c_3 h_c^{\frac{1}{4}} + \dots \quad (3.6)$$

Where c_i are constants determined by curve fitting procedure[121]. For an ideal sharp indenter, the contact area can be reduced to:

$$A_c = c_0 h_c^2 \quad (3.7)$$

Where the constant $c_0=24.5$. The remaining constants of Eq.(3.6), describe deviations from the ideally indenter geometry. Any change in the edge radius of the tip necessarily implies a new calibration process

However, there are some problems with this methodology. Firstly, the Oliver and Pharr analysis methodology does not account for the pile up effect around the indenter which means that the true contact depth should be measured from a position above the original surface. Another shortcoming of this methodology is the time consumption of calibrating the tip. Careful tip calibration is essential for the methodology of the Oliver and Pharr method. Thus a periodical calibration is recommended for Eq. (3.6). Moreover, the computation of the true value of the contact area, A_c , is a controversial issue [115]. Fig.3.1 shows two typical phenomena that may affect the elastic modulus value obtained by the indentation through the Oliver and Pharr methodology. The first is associated with the different plastic behaviour of the tested material as compared to the reference material (e.g. fused silica) used in the calibration of the function area. As a consequence of the indentation process, the surface of the material in contact with the indenter may show a pile-up or sink-in effect, resulting in a contact depth different from those predicted by Eq. (3.4). The second is the roundness tip effect, characterized by the radius, r , and the effective truncation length, h_0 (Fig. 3.1). Successive indentation tests may produce a progressive wear of the tip, which results in a gradual increase in the radius of the tip edge.

Fig. 3.1: Pile-up and sink-in phenomena developed during an indentation test on elasto-plastic material, using an actual indenter with blunt depth.

To overcome these difficulties, alternative methodologies have been developed in order to provide a reliable value of the elastic modulus and hardness through instrumented indentation tests. Dimensionless functions could be used to take into account the effect of sink-in or pile-up, to characterize the elastic-plastic properties of the materials [122, 123] [72] [124] [125]. Another possibility is to try to extract information on the loading branch of the indentation process. Loading curve for an ideal sharp indenter should follow the Kick law equation [25-27]:

$$P = Ch^2 \quad (3.8)$$

The indentation load, P , shows a linear relation with the square of the indentation depth h , and C is the curvature of the loading curve. This value can be obtained from the experimental force-penetration curves. Several efforts have been made to obtain an analytical expression for the C parameter. Hainsworth and Page [28], Hainsworth and Chandler [29] determined an expression for C by combining Young's modulus and the hardness :

$$C = E \left(\Phi_m \sqrt{\frac{E}{H}} + \Psi_m \sqrt{\frac{H}{E}} \right)^{-2} \quad (3.9)$$

In Eq.(3.9), E and H are the Young's modulus and hardness, respectively, and Φ_m and Ψ_m are empirical parameters obtained from experiments using known material properties. The effect of the blunt tip and indentation size can be ignored at deeper penetration depths. Thereafter, J.Malzbender et al.[31] derived an analytical expression for the $P - h^2$ relationship, which was verified experimentally [32, 33]:

$$P = E_r \left(\frac{1}{\sqrt{c_0}} \frac{\sqrt{E_r}}{\sqrt{H}} + \frac{\varepsilon}{\beta} \sqrt{\frac{\pi}{4}} \frac{\sqrt{H}}{\sqrt{E_r}} \right)^{-2} h^2 \quad (3.10)$$

M. Troyon and M. Martin improved the equation formulated by Malzbender by taking some correction factors into account [34]. However, their analytical equations involve two parameters, i.e., hardness and Young's modulus. Consequently, the model could only be used to estimate either E or H when one of these parameters is known. In practice, neither hardness nor Young's modulus is known; thus, it is not convenient to use this model.

In this study, based on the analytical loading expression proposed by Malzbender et al.[31], a simple methodology for instrumented sharp indentation is developed from a continuous record of the indentation load and the contact stiffness with the penetration depth. An explicit expression, depending on two experimental parameters directly extracted from the instrumented sharp indentation tests, has been proposed in order to obtain the elastic modulus of elastic-plastic materials. The Young's modulus of five commercial elastic-plastic materials has been calculated through the proposed methodology using a Berkovich diamond tip. These values were compared with those determined by the Oliver-Pharr methodology and those measured through tensile tests and impulse excitation of vibration tests. The comparative study shows considerable agreement between the values obtained from the proposed indentation model and the other traditional tests.

3.2. Analytical method

Considering an ideal geometry of the Berkovich tip [19], the projected area described by Eq. (3.6) can be reduced to Eq. (3.7). If Eq. (3.7) is substituted into Eqs. (3.1) and (3.2), the hardness, H , and the reduced Young's modulus, E_r , can be expressed as:

$$H = \frac{P}{c_0 h_c^2} \quad (3.11)$$

$$E_r = \frac{\sqrt{\pi}}{2\beta} \frac{S_u}{\sqrt{c_0 h_c^2}} \quad (3.12)$$

By combining Eqs. (3.11) and (3.12) a relationship between the indentation load, P , and the unloading contact stiffness, S_u , can be obtained:

$$\frac{P}{S_u^2} = \frac{\pi}{4\beta^2} \frac{H}{E_r^2} \quad (3.13)$$

According to Eq. (3.13), the quotient between the indentation load and the square of the contact stiffness, P/S_u^2 , is proportional to hardness, H , and inversely proportional to the square of reduced modulus, E_r . For homogeneous materials, H and E_r are usually constant with the penetration depth in a fully plastic regime [126], which occurs at very small penetration depths

during the sharp indentation tests. Eq.(3.13) can be rearranged to provide the following relationship:

$$\frac{H}{E_r} = \frac{4\beta^2}{\pi} \frac{E_r P}{S_u^2} \quad (3.14)$$

By substituting Eq. (3.14) into Eq. (3.10), a relationship between the indentation load, P , and the penetration depth, h , is obtained:

$$h = \sqrt{P} \left[\frac{1}{\sqrt{c_0}} \sqrt{\frac{\pi}{4\beta^2}} \frac{S_u}{E_r \sqrt{P}} + \varepsilon \frac{\sqrt{P}}{S_u} \right] \quad (3.15)$$

By substituting Eq. (3.8) into Eq. (3.13), a linear relationship between the contact stiffness S_u and the penetration depth h is found:

$$S_u = 2\beta E_r \sqrt{\frac{C}{\pi H}} h = C_s h \quad \left(\frac{Ch^2}{S_u^2} = \frac{\pi}{4\beta^2} \frac{H}{E_r^2} \right) \quad (3.16)$$

Where $C_s = 2\beta E_r \sqrt{\frac{C}{\pi H}}$, it can be experimentally obtained from the slope of the S_u - h curve. Consequently, a continuous measurement of the indentation load and contact stiffness as a function of the penetration depth is needed in order to apply this methodology.

The relationship between the indentation load, P , and the penetration depth, h , can be rewritten as a function of two parameters: the P - h curvature, C , obtained by linear fitting of the P - h^2 experimental data; and, C_s , experimentally obtained from the slope of the S_u - h curve. By substituting Eq. (3.8) and (3.16) into Eq. (3.15):

$$P = \left[\frac{\sqrt{\pi}}{2} \frac{1}{\sqrt{c_0}} \frac{1}{\beta} \frac{1}{E_r} \frac{C_s}{\sqrt{C}} + \varepsilon \frac{\sqrt{C}}{C_s} \right]^{-2} h^2 \quad (3.17)$$

By comparing Eqs. (3.17) and (3.8), an expression for Young's modulus, depending on experimental parameters directly obtained from the sharp indentations tests can be written as:

$$E_r = \frac{\sqrt{\pi}}{2} \frac{1}{\beta} \frac{1}{\sqrt{c_0}} \frac{C_s^2}{C_s - \varepsilon C} \quad (3.18)$$

It should be noted that Eq. (3.18) has been obtained under the assumption of an ideally sharp geometry. However, actual Berkovich indenters always have a small edge radius ranging

from 20 nm to 200 nm (Fig. 3.1) and an edge tip rounding associated to wear will occur as explained above [127]. Therefore, in order to use Eqs.(3.8) and (3.16), the data affected by the edge rounding should be discarded. The reliability of Eq. (3.18) will depend on the accurate determination of the C_s and C parameters. Consequently, the contribution of the edge tip rounding has to be eliminated. This was achieved by selecting the portion of the curve, where the load versus the square of the penetration depth ($P-h^2$) and the contact stiffness versus the penetration depth (S_u-h) showed a linear behaviour. In fact, from the experimental results, it was found that removing the initial part of curves (i.e. for very shallow penetration depths) resulted in the linear behaviour predicted by Eqs (3.8) and (3.16) [128-130].

Similarly, by substituting Eq. (3.18) into Eq. (3.13), an expression for the hardness, depending on the two parameters: C and C_s , is obtained:

$$H = \frac{C_s^4}{c_0 (C_s - \varepsilon C)^2} \frac{P}{S_u^2} \quad (3.19)$$

3.3 Experimental procedure

The depth sensing indentations tests (DSI) were performed on Nanoindenter XP (NanoInstruments Innovation Center, MTS systems, TN, USA) using the continuous stiffness measurement methodology (CSM). A Berkovich diamond indenter was selected [131]. In the CSM module, continuous loading and unloading cycles were conducted during the loading branch by imposing a small dynamic oscillation of 2 nm and 45 Hz on the displacement signal and measuring the amplitude and phase of the corresponding force [97]. Consequently, the contact stiffness can be measured continuously during the penetration depth. A total of 60 indentations were made in the displacement control, using the CSM module on five commercial metallic alloys: carbon steel (F-114); aluminium alloy (Al1050); brass; polycrystalline tungsten; and fused silica. 2 μm of maximum penetration depth was selected to make the CSM indentation tests on carbon steel and aluminium; 3 μm for the brass alloy; and 1 μm for tungsten. Prior to the indentation tests, the surfaces of all materials were ground and then polished in two

steps, using a mechanical polisher (Labopol-5, Struers, Copenhagen, Denmark), finishing with a colloid suspension of 0.05 μm .

In order to check the Young's modulus values obtained by equation (3.18), the Oliver-Pharr methodology (O-P) to calculate the elastic modulus was also applied to the indentation tests. A previous calibration procedure of the function area, A_c , was carried out on fused silica. The area function coefficients of equation (3.6) were calculated through an iterative use of equations (3.2), (3.3) and (3.6). This function area was used to calculate the elastic modulus of all materials according to the O-P methodology.

Tensile tests were also carried out on carbon steel (F-114), aluminium (Al1050), and brass. The tensile tests were carried out on a universal INSTRON 8501 testing machine with a 10kN load cell at room temperature using a displacement rate of 0.5 mm/min. Cylindrical or bar specimens were loaded with force control until fracture. The specimens were manufactured according to the ASTM.E8/E8M standard [132]. The gage length was 12.5mm for bar and cylindrical samples.

Additionally, impulse excitation tests were carried out on carbon steel (F-114), aluminium (Al-1050), and brass alloy samples. The impulse excitation tests were carried out on a Grindosonic MK5TM instrument, and proper location of the impulse point and transducer were based on ASTM E8 [133]. The test was repeated until five consecutive resonant frequencies that lie within 1% of each other, were obtained. The average value was used to determine the Young's modulus of the specimen. The dimensions and weights of the sample and the frequencies from the impulse excitation test are listed in Tables 3.1. The elastic modulus values obtained from tensile tests and the impulse excitation tests were compared with those obtained from indentation tests through the O-P methodology and the proposed methodology.

In the case of silica, the elastic modulus value provided by the manufacturer was used for calibration and as the nominal reference value. In the case of tungsten, a Ti-Y₂O₃ tungsten alloy was selected. The elastic modulus value reported for this alloy was 382 $\pm$ 23 GPa [134]. This value was considered as the nominal reference value in this work.

In addition, microindentation tests were carried out on all metallic alloys. 10 hardness tests on each material were performed using a Vickers indenter. The indentation load ranged from 0.5 N to 3 N with a dwell time of 20 s. The microhardness was obtained dividing the indentation load by the projected area of the residual imprint.

Table 3-1: Parameters of the samples for the impulse excitation test.

Material	Diameter (mm)	Width (mm)	Thickness (mm)	Length (mm)	Weight (g)	Frequency (Hz)
Steel	5.9	-	-	349.3	76	7360±30
Brass	16.0	-	-	654.3	1103	2638±20
Al 1050	-	29.9	2.9	433.3	103	841±1

3.4 Results and Discussion

Fig. 3.2 shows the $P-h^2$ and S_u-h curves obtained from instrumented indentation tests carried out on fused silica using the actual Berkovich tip. The initial part of both curves was not linear at shallow penetration depths: less than approximately 400 nm for $P-h^2$ curve (Fig. 3.2(a)), and 50 nm for S_u-h (Fig. 3.2(b)); i.e. the initial part was characterized by nonlinear tendency, most probably due to the roundness effect of the tip edge. When the penetration depth increased above those critical depths, both curves, $P-h^2$ and S_u-h , were characterized by a linear tendency, which corresponding to that predicted by Eqs. (3.8) and (3.16), respectively. These equations were formulated for an ideal sharp indenter. Consequently, only the values corresponding to penetration depths greater than the critical ones fulfilled the test conditions under which the assumption of ideal geometry of the Berkovich indenter was acceptable and, therefore, Eq. (3.18) could be used to calculate a correct value of the Young's modulus. Fig. 3.2 also shows a representative linear fitting process for penetration depths higher than the critical depths on experimental curves, $P-h^2$ and S_u-h , obtained from indentation tests on fused silica. The critical depths, h_{critical} could be identified by the intersection point between the original experimental curve and linear proportional slope curve by removing the initial curved regime.

A similar procedure has been described by others authors to obtain a similar critical depth [130]. Similar behavior was observed for all studied materials and similar procedure was followed to determine the corresponding critical depth.

Fig. 3.2: Linear fitting procedure of the $P-h^2$ curve (a) and the S_u-h curve (b) to calculate the C and C_s parameters, obtained from indentation tests on fused silica. The roundness effect of the tip is shown via a zoom of the initial part of both curves. The critical point determination is also included.

Fig.3.3 shows the $P-h^2$ (Fig.3.3a) and S_u-h (Fig.3.3b) curves obtained from instrumented indentation tests carried out on steel, brass, aluminium and tungsten. All curves were characterized by an initial non-linear portion and followed by a linear portion for depths

greater than a given value of the penetration. The critical penetration depths were different and depended on the curve and the material analysed.

Fig. 3.3: P - h^2 curves (a) and S_u - h curves (b) obtained from instrumented indentation tests carried out on steel, brass, aluminum and tungsten.

Table 3.2 shows the $h_{critical}$ values obtained from the analysis of the P - h^2 and S_u - h curves for all elastic-plastic materials. The values of the critical penetration depth for P - h^2 curve affected by the tip roundness were ranged from 500 nm to 600 nm. Meanwhile, the values of the critical penetration depth extracted from S_u - h curves, were less than 150 nm. The initial part of both curves, and consequently the value of this critical depth, may be strongly affected by the tip edge radius. Similar phenomenon was reported by other researchers [27, 130, 135].

Table 3-2: The h_{critical} values, slopes C and C_s obtained from the analysis of the $P-h^2$ and S_u-h curves for all elastic-plastic materials, respectively; and corresponding linear regression coefficient, R^2 .

Material	Curves $P-h^2$			Curves S_u-h		
	h_{critical}	C (Pa)	R^2	h_{critical}	C_s (Pa)	R^2
Steel	500	9.08×10^{10}	0.99987	140	1.04×10^{12}	0.99863
Brass	600	4.00×10^{10}	0.99973	150	5.55×10^{11}	0.99921
Al1050	450	2.63×10^{10}	0.9999	120	4.48×10^{11}	0.99921
Tungsten	350	1.57×10^{11}	0.99942	70	1.65×10^{12}	0.99945
Fused silica	400	1.26×10^{11}	0.9998	50	2.77×10^{11}	0.99955

Fig. 3.4 : $P-h^2$ curves (a) and S_u-h curves (b) obtained from instrumented indentation tests carried out on steel with blunt tip and new tip, respectively.

The Berkovich tip was blunt and an old one in previous study. Figure 3.4 shows the $P-h^2$ curves (a) and the S_u-h curves (b) obtained from instrumented indentation tests carried out on steel with a blunt tip and a new tip, respectively. The critical depths estimated with the new tip are lower than with the old one. However, the parameters C and C_s (from linear curve fitting for the different tip radii) were similar, which could provide consistent Young's modulus with the proposed methodology.

The proposed procedure, shown in Fig. 3.2, has been carried out on both curves and for all materials. Table 3.2 shows the values of C and C_s obtained for the different materials. Substituting the values of these parameters into Eq. (3.18), the corresponding values of the elastic modulus could be obtained for each material. Table 3.3 shows the Young's modulus values determined by Eq. (3.18) (values designated as *proposed methodology*), through the Oliver-Pharr methodology (values designated as *O-P*), tensile and impulse excitation tests. The Young's modulus measured from tensile test and impulse excitation test were closed. These values of elastic modulus were considered as the nominal one. The Young's moduli measured by the Oliver-Pharr methodology were larger than those obtained from tensile and impulse excitation tests. This phenomenon may be a consequence of the pile-up effect developed by the plastic strain induced on the metallic materials during the indentation process [102].

As a consequence, it seems that the Oliver and Pharr method is not using a correct value of contact area, A_c , to calculate the Young's modulus. This circumstance demonstrates the inaccuracy of using a fused silica samples to calibrate the function area when materials with different elastic-plastic behaviour are tested by nanoindentation. In view of this, it is suggested that the calibration of the contact area would be done with a material as close as possible to the one being test. The ideal situation would be to calibrate the contact area on the same material to be characterized. This is in fact what happens when the O-P method was applied on fused silica, which was the material used as a reference material to calibrate the contact area function (Table 3.3). In this particular case, the Young's modulus obtained by the O-P method and by the one proposed in this paper are almost the same. The Young' modulus

values determined through the proposed methodology have errors ranging from 0.15% to 3.9 % when compared to those obtained by tensile and impulse excitation tests (Table 3.3). Unlike what happened to the O-P method, these results indicate that the proposed methodology seems to be not so sensitive to the plasticity phenomena that occur under the indenter tip, providing values that are closer to the reference ones. Moreover, in the proposed method the calibration procedure of the contact area is not needed. Instead, two parameters C and C_s have to be calculated by linear fitting, which makes the proposed process simpler and faster.

Table 3.3: Young's modulus obtained from different techniques: nanoindentation using the Oliver-Pharr methodology; nanoindentation using the methodology proposed in this paper; tensile tests; and impulse excitation tests.

Material	O-P ⁽³⁾ (GPa)	Tensile Test (GPa)	Impulse Excitation Test(GPa)	Proposed Methodology (GPa)	error%
Steel (F114)	246.0±5.5	209.0±10	210.5±1	211.2±3.0	0.57
Brass	116.7±3.7	95.0±10	99.7±1	102.2±3.1	0.37
Al-1050	90.6±1.8	76.3±5	75.8±1	79.3±3.5	3.9
Tungsten ⁽¹⁾	401.8±7.2	-	-	388.6±3.1	1.73
Fused silica ⁽²⁾	73.0±0.5	-	-	75.0±0.1	0.40

¹ Young's modulus reference value[136]: 382±23 GPa

² Young's modulus provided from the manufacturer: 74.7±0.2 GPa

³ Oliver-Pharr methodology: Coefficients of the area function for the tip calibrated on fused silica:

$$c_0=24.5, c_1=-19,859, c_2=768,388, c_3=-3,181,026, c_4=2,569,101$$

Fig.3.5 shows the hardness values determined by microindentation tests versus indentation load. In order to evaluate the capabilities of the proposed methodology to estimate the hardness of each material from nanoindentation tests, the values obtained from Eq. (3.19) were plotted as horizontal lines.

Fig. 3.5 : Hardness values determined by microindentation tests at different indentation loads for each material analyzed. The hardness values obtained from Eq. (3.19) were included by a horizontal line for each material. The hardness data for fused silica provided by the manufacturer and obtained from Eq. (3.19) were also included in the attached table.

The microhardness for all materials analysed depended on the indentation load, the values decreasing as the load increased. This behaviour has been widely reported and it is known as the indentation size effect. It is related to the ability of the indented material to activate mechanisms in order to accommodate the deformation induced by the indentation process [137]. The hardness values obtained by the proposed methodology, Eq. (3.19), were comparable although slightly higher than those obtained by microhardness tests, especially at the higher loads used. The same behaviour was observed for the fused silica sample when compared to the nominal value of hardness provided by the manufacturer. Additionally, a remarkable difference was observed between the values obtained on tungsten at different loads. The proposed

methodology gives hardness values up to 21% higher than those corresponding to microhardness tests at maximum load. The discrepancy observed between the hardness values obtained by microhardness tests and the proposed methodology could be related to the difference in behaviour assumed by Eq. (3.1) or (3.19) and the real behavior, as these equations consider the indented material as a continuum media. However, the actual behaviour of the material might not follow this assumption, because phenomena like the generation of dislocations within the material to accommodate the strain induced by the indentation process, residual stresses generated on the sample surface as a consequence of the polish process, or different surface treatments, are not taken into account in Eq. (3.1) or (3.19).

Conclusions

In summary, a simple method for calculating the Young's modulus of bulk elasto-plastic materials has been proposed and experimentally validated. The proposed method requires the fulfilment of the linear variation condition for the $P-h^2$ and S_u-h curves. Under this requirement, a procedure is followed to select $P-h^2$ and S_u-h data not affected by the tip roundness. The proposed method has proven to be able to provide Young's modulus values very similar to those measured by other traditional testing techniques. This work has shown that the proposed method can successfully reduce the detrimental effects of the tip roundness and the plasticity phenomena on the Young's modulus estimation through the sharp instrumented indentation on elastic-plastic materials.

The extracting procedure is as follows:

- (a). Make indentation tests with a sharp indenter on a suitably prepared surface, at different penetration depths.
- (b). Acquire a continuous record of indentation load and contact stiffness with the penetration depth.
- (c). Represent the indentation load, P , versus squared penetration depth, h^2 , and the contact stiffness, S_u , versus penetration depth, h , curves.

- (d). Determine the critical depth. Select the data corresponding to the penetration depths greater than the critical one.
- (e). Determine the slopes C and C_s from $P-h^2$ and S_u-h curves, respectively, through the linear fitting of the data corresponding to penetration depths greater than the critical one.
- (d). Substitute the values of C and C_s into equation (3.18) and (3.19) and calculate the Young's modulus and nanoindentation hardness.

Chapter 4 : A new analytical procedure to extract representative stress strain curve by spherical indentation

4.1. Introduction

The main mechanical properties measured by this technique are the Young's modulus, E , and hardness, H by sharp indenter [24, 120]. However, awareness of basic Young's modulus or hardness is not sufficient, more *in-situ* material properties are necessary. The methodology for measuring materials properties such as flow stress, creep property and fracture is necessary through an instrumented indentation technique [61, 138-140]. This work focuses on the methodology for determining the *in-situ* flow stress of metal using a spherical indenter with an instrumented depth sensor. The spherical indenter has become more popular recently. Unlike the sharp indenter such as the Berkovich or Vicker indenter, the experimental curve from the spherical indenter provides more information, as this type of indenter has a smooth transition from elastic to elastic-plastic contact[141].

There were many methodologies for the extraction of flow stress using the spherical indentation technique. One category belongs to mathematical methods (e.g. the dimensionless method, and Artificial Neural Networks (*ANN*)) [16, 73-76, 78, 142-144]. However, these methods need a large amount of computing time. Additionally, the unique solution and sensitivity to defects in the tip in these methods is still under discussion [87, 125]. Another category to obtain the material parameters from spherical indentation is by defining the representative strain and stress. As regards practicality in the experimental measurement, these

methods are more convenient and can be used directly. Following the work of Meyer [53], Tabor defined the representative strain at the contact edge of the spherical indentation and empirically determined the values of ψ and β as 2.8 and 0.2 based on a lot of tensile test data on common metals [42]. Most of researchers agreed with the formation of Tabor's equation, but disputed the choice of the value of ψ and β [56-62]. Nevertheless, some researchers argued the formulation of representative strain in Tabor's equation [51, 60, 68, 69].

From the literature, there is not uniform agreement among researchers as regards the relationship between the mean pressure and the extent of indentation. Additionally, most of the strain constraint factor or stress factor in these definitions is obtained from empirical experimental data or FEM simulation data. There are few analytical studies to explain these constraint factors. The main purpose of this research is to study the analytical procedure for extracting the stress strain curve using spherical indentation. A new consideration of the relationship between the strain and stress constraint factors is studied theoretically, which could lead to a better understanding of the empirical values of the constraint factors in Tabor's equation. This chapter is organized as follows. Section 4.2 starts from theory background of spherical indentation to extract the stress strain curve, and a new analytical expression will be proposed to determine the values of the constraint factor in Tabor's equation. In section 4.3, the FEM simulation and experimental test will be described. In section 4.4, results and discussion will be shown. Finally, some significant conclusions will be summarized.

4.2. Theoretical background

4.2.1. The analytical relationship between the stress constraint factor and the strain coefficient in Tabor's equation

In 1908 Meyer found that for many materials, the mean pressure increased with a/R according to the simple power law [53]:

$$p_m = k \left(\frac{a}{R} \right)^m \quad (4.1)$$

In which, p_m is the mean pressure, a/R is ratio of indentation radius to the ball radius, m is the Meyer' index.

In Eq.(4.1), k and m are constants. Meyer's equation was verified by further experimental studies [42, 54] and they suggested that the Meyer index, m was related to the hardening exponent, n . Following Meyer's work, Tabor proposed the concept of representative strain or stress, through which the mean pressure in Meyer's equation can be converted into the true stress strain curve. He assumed that the mean pressure, p_m at fully plastic regime was proportional to the representative stress, σ_r , and the impression radius was proportional to the corresponding representative strain, ε_r . Consequently, the representative stress and strain can be expressed as[42]:

$$\varepsilon_r = \beta \frac{a}{R} \quad (4.2)$$

$$\sigma_r = \frac{P}{\psi \pi a^2} \quad (4.3)$$

Where β is indentation strain constraint factor, a is the contact radius, R is the radius of indenter, ψ is the indentation stress constraint factor, P is the indentation load.

Tabor determined the parameter β and ψ from empirically experimental data. Generally, the indentation strain constraint β is equal to 0.2 and the indentation stress constraint ψ is equal to 2.8-3.2 [145]. It should be emphasized that the representative strain defined in the Tabor equation is an average value. The strain constraint factor, β , should vary with indentation depth through which the tangent of the corresponding contact point can be equal to the same angle of a sharp indenter [6, 55]. The fact is that the equivalent stress and strain at an arbitrary point beneath the indenter should be equal to the uniaxial stress strain curve from the result of FEM, although the equivalent stress and strain of the material vary from point to point [75, 146] .

The representative stress strain curves in the formation of Tabor' equation could be comparable to those measured from the tensile test. The true stress strain curve from the uniaxial tensile or compress test can be expressed as Hollomon equations:

$$\sigma = E\varepsilon \quad \text{for } \sigma \leq \sigma_y \quad (4.4)$$

$$\sigma = K\varepsilon^n \text{ for } \sigma \geq \sigma_y \quad (4.5)$$

Where E is the elastic modulus, σ_y is the yield stress, K is the strength coefficient and n is the strain hardening exponent.

Substituting Eq. (4.2) and (4.3) into the Hollomon equation, we can obtain:

$$\frac{P}{\psi\pi a^2} = K \left[\beta \left(\frac{a}{R} \right) \right]^n \quad (4.6)$$

Eq. (4.6) represents the relationship between the load, P and the ratio of contact radius to indenter radius according to the power law in fully plastic regime. Transforming Eq. (4.6) into the natural logarithm gives:

$$\ln P = \ln \left[\frac{K\pi\psi\beta^n}{R^n} \right] + (n+2)\ln a \quad (4.7)$$

In the same way, according to Hertz's equation in the elastic regime, the loading and contact radius can be expressed as:

$$P = \frac{4E_r}{3R} a^3 \quad (4.8)$$

Where $\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i}$, E , ν , E_i and ν_i are the Young's modulus and Poisson's ratio

for the bulk material to be measured and for the indenter, respectively.

Transforming Eq.(4.8) into formation of the natural logarithm gives:

$$\ln P = \ln \left[\frac{4E_r}{3R} \right] + 3\ln a \quad (4.9)$$

Fig. 4.1: Eq. (4.7) and (4.9) in the same $\ln a - \ln P$ coordinates, 'e' is the intersection point.

Plotting Eq. (4.7) and (4.9) in $\ln a$ vs $\ln P$ coordinates is shown in Fig. 4.1. By extending the two lines, there should be an intersection point 'e' for different linear slopes. The 'e' point is the transitional point of the elastic and the fully plastic regimes.

In a previous work, Johnson [44] suggested that the spherical indentation process can be divided into three distinct regimes: elastic, elastic-plastic and fully plastic period. Y.J.Park and G.M.Pharr used FEM and showed that the elastic-plastic regime can be subdivided into two parts: (1). an elastically-dominated regime, in which the indentation behaviour was dependent on an elastic property; (2). a plastically-dominated regime in which the indentation behavior depended on the work hardening characteristics of the material [48]. Fig.4.2 shows a schematic representation of the relationship between p_m/σ_r and $(E/\sigma_y)(a/R)$ based on the finite element analysis.

Fig. 4.2: Schematic representation of the relationship between p_m/σ_r and $(E/\sigma_y)(a/R)$ based on the finite element analysis[48], in which the representative strain was defined as $\varepsilon_r=0.2a/R$.

The intersection point 'e' can be considered as a simplistic interpretation of the transition regime. Additionally, this intersectional point 'e' may be approximated to the yield point of the uniaxial stress strain curve, in which the stress-strain is fitted by a Hollomon equation. Therefore, the representative strain and stress at 'e' point can be express as:

$$\varepsilon_r = \beta \left(\frac{a_e}{R} \right) \quad (4.10)$$

$$\sigma_r = \frac{P_e}{\psi \pi a_e^2} \quad (4.11)$$

By combining Eq. (4.10) and (4.11), we can obtain:

$$\psi^* \beta = \frac{R}{\pi E} \frac{P_e}{a_e^3} \quad (4.12)$$

By combining Eq. (4.12) with Hertz's equation in Eq.(4.8), we can obtain:

$$\phi = \psi^* \beta = \frac{4}{3\pi} \frac{E_r}{E} \quad (4.13)$$

In equation (4.13), the product, ϕ of stress constraint fact ψ and strain constraint fact β is constant, which is dependent on the ratio of reduced Young's modulus, E_r to Young's modulus E . According to equation (4.13), the constraint factors can be determined if one of them is known.

Based on empirical data in Tabor's equation, the value of stress constraint factor is selected as 3 for common engineering metals in literatures [61, 69, 147]. However, there are few studies on the analytical solution for the stress constraint factor. Shield [148] studied the axially symmetric plastic flow of rigid-plastic non hardening materials using the Tresca yield criterion. He found that the average pressure beneath the flat punch indenter was equal to $2.8\sigma_y$. If the von Mises criterion is used, the average pressure beneath the flat punch indenter was $3.2\sigma_y$. Unlike the punch indenter, where the contact area is constant, the spherical one offers a gradual transition of the contact area. It could be assumed that the spherical indenter is corresponding to a punch indenter at a given penetration depth. As a result, the value of stress constraint factor is taken as 3.2, which is consistent with analytical solution by Shield [148]. As the product of stress and strain constraint factors is constant, according to equation (4.13), the strain constraint factor can be determined.

2.2. Estimation of the contact radius

Another important parameter is the contact radius, a , which can be used to predict the representative strain. In the case of a flat punch indenter, Sneddon's analysis proposed an estimation expression of contact radius, a :

$$a = \frac{S_u}{2E_r} \quad (4.14)$$

Where S_u is the contact stiffness, it can be obtained from the initial of unloading curve or the harmonic stiffness in CSM.

G.M.Pharr et al.[18] extended Sneddon' analysis for the indentation of an elastic half space by any punch described as a solid of revolution of a smooth function, from which the contact radius could be determined when the reduced Young' modulus and initial unloading slope of the unloading are known.

4.3. FEM and Experiment procedure

4.2.1. FEM model

Commercial finite element analysis Software ABAQUS v 6.13 was used to simulate the Spherical indentation Process[149]. A 2D axisymmetric model with $100 \mu\text{m} \times 100 \mu\text{m}$ dimension was used. The meshing type was CAX4R, a 4-node bilinear axisymmetric quadrilateral, reduced integration and hour glass control. The refine meshing size was 50 nm in the vicinity of indenter tip for accurate data acquisition. The spherical indenter with a radius of $10 \mu\text{m}$ was modelled as axisymmetric analytical rigid. The FEM meshing is illustrated in Fig.4.3. The movements of the nodes along the axis symmetry were limited along the y-axis, and the nodes at the bottom were fixed along the y-axis. The contact friction was chosen as 0.1 [62, 74]. The simulations were carried out on common metals from the literature which were considered isotropic, and Hollomon's power law strain hardening. The yield criterion was von Mises including large deformations.

Fig. 4.3 : FEM meshing in simulation

4.2.2 Experimental procedure

Depth sensing indentations tests (DSI) were carried out on a Nanoindenter XP (NanoInstruments Innovation Center, MTS systems, TN, USA) using the continuous stiffness measurement methodology (CSM). A conical (angel 60°) spherical diamond indenter with a tip radius of $10\mu\text{m}$ was selected. A scanning electron microscope was used to measure the radius of tip before the test to verify the radius of the indenter. Continuous loading and unloading cycles were conducted during the loading branch by imposing a small dynamic oscillation of 2 nm and 45 Hz on the displacement signal and measuring the amplitude and phase of the corresponding force [97, 150]. Consequently, the contact stiffness was continuously measured as a function of the penetration depth during the experiment. Prior to the indentation tests, the samples were ground and polished in two steps, using a mechanical polisher (Labopol-5, Struers, Copenhagen, Denmark) and finished with colloidal suspension of silica of $0.05\mu\text{m}$. A total of 100 indentations were made in a displacement control on five samples of commercial metallic alloys: a carbon steel (F-114), a brass alloy, and an aluminum alloy (Al-1050). A maximum penetration depth of 2,000 nm was selected to make the indentation tests on all the materials, which satisfies the limitation of correction factor in the contact radius, to make sure that the last contact position could be in the profile of the spherical part of indenter [151]. The average of these curves is selected to converge into the representative stress strain curve of the spherical indentation which can be comparable with the true stress strain curve from the tensile test [138]. Tensile tests were also carried out on the carbon steel (F-114), aluminum (Al-1050), and brass alloy samples. They were carried out on a universal INSTRON 8501 testing machine with a 10 kN load cell at room temperature under displacement control using a displacement rate of 0.5 mm/min. The cylindrical specimens were machined in accordance with the ASTM E8 standard [132]. The gauge length was 12.5 mm for the samples.

4.4. Results and discussion

4.4.1. Analysis of the FEM result.

Fig.4.4 show the comparison between the FEM and Hertz loads on the elastic materials with Young's modulus $E=100\text{GPa}$. The FEM load is consistent with the one from Hertz equation, which could validate the correction of FEM modeling.

Fig. 4.4.Comparison between the FEM and Hertz loads on the elastic materials with Young's modulus $E=100\text{GPa}$

The material properties of metals are given in Table 4.1. The actual contact area can be taken into account by identifying the last node which maintains contact with the indenter [151].

Table 4-1: Common engineering metal materials with work hardening were selected in the evaluated analysis. (Poisson ratio =0.3)

Materials	E (GPa)	σ_y/E	σ_y (MPa)	n	predict m in fem
Steel alloy	210	0.0069	1460	0.12	2.11
Aluminum	70	0.0057	400	0.1	2.12
Copper	128	0.000078	10	0.5	2.50
Brass	96	0.00061	59	0.36	2.4
Iron	211	0.0033	71	0.26	2.27
Inconel 600	170	0.0015	263	0.293	2.32

Fig. 4.5 (a) shows the indentation curve from FEM. The steel alloy shows the highest resistance against the penetration, and the indentation curves of soft metal like Iron, Copper and

Brass show a low load at the same penetration depth. The indentation behaviors for the three soft metals are indistinguishable from the load curves. Therefore, it can be seen that with the only load curve from the spherical indentation, the plastic property could not be determined uniquely. The contact areas are obtained from the last contact node in the FEM of each step, and the penetration depth versus contact area shown in Fig. 4.5 (b). The values of the contact area are different from each other at a given depth of penetration. Meanwhile, the contact area of material is increased by decreasing the value of the hardening exponent. However, in the Oliver Pharr method, the cross sectional area of tip and the contact area of the indenter and the specimen, at a given depth, are treated implicitly as if they were the same [152]. For this reason, it is recommended to calibrate the area function of the indenter with a reference material that has similar materials properties to the measured one.

Fig. 4.5: (a) Load versus penetration depth curve (b) Contact area A versus penetration depth .

Tabor had concluded the slope of the linear regression is Meyer' index m which is equal to $n+2$, where n is the hardening exponent in the Hollomon equation. Table 4.1 also shows Meyer' index, m obtained from the slope of the regression of $\ln(P)$ on $\ln(a)$, from which the value of hardening exponent, n is approximately equal to the input one.

In order to check the reasonable determination of constraint factors in Tabor's equation. The tensile stress strain curve is compared with representative stress strain curves from different constraint factor (CF): stress CF=3.2 in this study; stress CF=3 according to Tabor's

equation. Recently, Kalidindi et al.[71] used a new definition of the representative indentation strain from the transformation of Hertz's equation:

$$\sigma_r = \frac{F}{\pi a^2}, \quad \varepsilon_r = \frac{4}{3\pi} \frac{h}{a} \approx \frac{h}{2.4a} \quad (4.15)$$

Though the new definition of representative strain proposed by Kalidindi et al.[71] is consistent with Hertz's equation, the representative stress strain haven't been compared with the nominal tensile stress strain curve. In this FEM study, the representative strain proposed by Kalidindi et al.[71], in which the stress CF=3, is also included to compare with tensile stress strain curve. Fig.4.6 shows the comparison of tensile stress curve and representative curves from FEM results. The representative stress strain curve using the CF=3.2 in the cases of Iron, Inconel alloy, copper and Brass best agrees with the nominal true stress strain curve than those curves obtained from Tabor or Kalidindi' equation. However, the initial part of the true stress strain curve and the representative stress strain curves in steel and aluminum shows large derivation between them. From Fig.4.2, when the values of ratio $(E/\sigma_y)(a/R)$ range from 50-200, the fully plastic deformation is reached. For the metals of Iron, copper, and brass, the fully plastic regime is developed for a penetration depth less than 5nm and which correspond to a representative strain less than 0.003. However, for the metals of aluminum and steel, the commence of fully plastic regime need more deeper penetration depth at about 100nm and which correspond to representative strain more than 0.05. In this study, the product, ϕ is attained from the intersection between the elastic regime and the fully plastic regime, in which the effect of the transition from elastic to plastic regime is ignored. Consequently, assumption is only suitable for the metal which the full plastic regime is developed at shallow penetration depth. For metals with longer transition of elastic plastic regime, significant derivation of the initial part of the representative stress-strain curve is attained regarding the true stress-strain curve. The representative stress strain from Kalidindi and Tabor equation lead to large deviation for some materials with short and large transition of elastic plastic regime.

Fig. 4.6: Comparison of the tensile stress curve and representative curves from FEM results

4.4.2. Analysis of the experimental results

In the tensile test, according to Hollomon's equation, the mechanical properties can be obtained from the stress strain curve. The Young's modulus, yield stress which is determined by means of the 0.2% strain rule, the hardening exponent and grain size are shown in Table.4.2.

Table 4-2: Mechanical properties of steel, brass and Al-alloy				
Materials	E(GPa)	Yield stress(MPa)	Hardening exponent n	Grain size (μm)
steel	210	780	0.26	15
brass	100	320	0.15	20
Al-alloy	70	220	0.1	10

In order to check the radius of spherical indenter, as showed in Fig.4.7, the best fitting spherical radius is found to be about $9.2 \mu\text{m}$ from SEM measurement which is smaller than the nominal value. From the zoomed figure, the spherical indenter is worn at shallow penetration depth. In addition, Inherent noise of continuous stiffness measurement and the roughness of specimen could scatter the representative stress strain curve. Consequently, penetration depths less than 10 nm will not be utilized for extraction of representative stress strain curve

Fig. 4.7. Scanning electron microscope image of a worn diamond conical-spherical indenter

Fig. 4.8 shows the average load vs. penetration depth curves for the three indented materials done with a Spherical tip. At the defined penetration depth, a higher indentation load is necessary for the steel. The indentation load of the Aluminum is the lowest.

Fig. 4.8: Load versus average penetration depth curves of the indented materials

When the Young's modulus was confirmed by tensile test and the contact stiffness can be measured by CSM, the contact radius can be estimated by Sneddon's equation [17]. It should be noted that, when using Eq. (4.14) to estimate the contact radius, the Young's modulus is assumed as constant during the indentation process [71, 153]. Fig.4.9. shows the linear regression of $\ln(P)$ versus $\ln(a)$ for the experimental materials. The values of hardening exponent from the slope are in good agreement with those obtained from the tensile experiments.

Fig. 4.9: Linear regression of $\ln(P)$ versus $\ln(a)$ for the experimental materials.

In the experimental analysis, the value of stress constraint factor is taken as 3.2, and the strain constraint factor is confirmed from Eq. (4.13). In order to verify the reasonability of the confirmation of constraint factors from experimental data, the representative stress strain curve extracted from this study will be compared with the curve extracted from Tabor's method. Fig.4.10. shows the plots for the indentation stress strain curve using the constraint factors in the study, the indentation stress strain curve using the constraint factors in Tabor's equation and the true stress strain curve extracted from the tensile test.

Fig. 4.10: Curve plots for the representative stress strain curve, tabor stress strain curve and the true stress strain curve extracted from the tensile test. (a) steel, (b) brass, (c) aluminum.

The representative stress strain curve using the constraint factors in the study shows good agreement with the true stress strain curve from tensile test. The deviation in the initial part of the representative stress strain curve could be due to the transition from the elastic regime to the plastic regime, which has been explained in the previous part. The products of the strain constraint factor and the stress constraint factor for the materials studied are also shown in the figure.

Conclusions

The main conclusions of this chapter can be summarized as follows:

- (1) In this study, a new insight into the constraint factors in the formation of Tabor's equation is described analytically. The product, ϕ of the strain constraint factor, β and stress constraint factor, ψ is constant at the transition position between the elastic regime and the fully plastic regime, which is related to the Young's modulus of the materials in contact.
- (2) An experimental and FEM procedure are carried out to evaluate the analytical procedure for extracting the representative stress strain curve from the spherical indentation. The representative stress strain curve using the proposed constraint factors in the study shows a good agreement with the nominal true stress strain curve.
- (3) Due to the drawback of the formulation of Tabor's equation, the representative stress strain

curve derived from Tabor's could have a large deviation at the initial strain period for the materials with a longer elastic-plastic transition defined by $(E/\sigma_y)(a/R)$. In future, the new formulation of representative strain for spherical indentation needs more study.

Chapter 5 : Micro-scale mechanical characterization of Inconel cermet coatings deposited by laser cladding

5.1. Introduction

Nickel-based superalloys are characterized by a high-temperature oxidation and corrosion resistance. They are commonly used to produce components exposed to extreme environmental conditions during their service life, such as aircraft and power-generation turbines, rocket engines, etc. [154, 155]. However, the high prices of these alloys, compared with stainless steel, constitute a restriction in their industrial application. Consequently, an economical alternative is to use Ni-based superalloys as coatings to protect a cheaper substrate material, such as a steel alloy [156-158]. Laser cladding is a coating technique in which the filler material, in the form of powder or wire, is melted using a laser beam as a heat source and deposited onto a substrate. It is a welding coating technique and represents an alternative to the traditional thermal spray methods [159]. The main advantages coming from the use of this technique are due to the versatility of the laser beam that allows the production of different types of coatings at greater thicknesses, with strong metallurgical bond with the substrate, minimal dilution with the base material, and a good surface finish [155, 160].

The commercial name Inconel is used to identify a group of Ni-based superalloys mainly made up of Ni and Cr. These alloys have been demonstrated as suitable to be deposited as a coating by laser cladding. The microstructure, the wear behaviour, and the mechanical properties of these coatings have been studied [157, 159, 161-169]. In addition, recent research

into the fracture and failure mechanism of Ni-base laser cladding coatings was carried out using *in-situ* tensile tests [170]. To improve the tribological properties of the metallic coatings applied by laser cladding, a solution is to introduce ceramic particles into the filler material. Different combinations of ceramics and metal alloys have been verified as suitable to be deposited by laser cladding to obtain cermet coatings [171]. It has been shown that Inconel 625 and Cr_3C_2 particles could be used for this purpose [169]. However, few researches about the local mechanical behaviour in a micro-scale of Inconel cermet coatings have been carried out.

The aim of this work is to study the mechanical properties in the micro-scale of Inconel 625- Cr_3C_2 cermet coatings deposited by laser cladding. The properties of the cermet matrix were compared with those of an Inconel 600 bulk specimen, also deposited by Laser Cladding. In addition, for a more complex analysis of the coatings, the properties of unmelted Cr_3C_2 particles, embedded in the in the cermet matrix, was also evaluated. Depth sensing indentation tests (DSI) were also carried out.

5.2. Materials and experimental procedures

5.2.1. Materials

An Inconel 625- Cr_3C_2 cermet coating was deposited by laser cladding onto Gr₂₂ ferritic steel (ASTM A387). Inconel 625 and Cr_3C_2 powders were supplied by Sulzer-Metco (MetcoClad 625 and Metco 70C-NS, respectively). The Inconel 625 powders were mechanically mixed with the 20wt% of Cr_3C_2 before processing the cermet coatings. The composition of Inconel 625 powder and the Inconel 600 bulk are presented in Table.5.1.

Table 5-1: Chemical composition of Inconel 625 powders and the Inconel 600 bulk specimen

Product	Weight percentage (nominal)					
	Ni	Cr	Mo	Nb	Fe	Others
Inconel 625 powder	58-63	20-23	8-10	3-5	≤ 5	< 2
Inconel 600 bulk	72-78	14-17	-	-	6-10	< 2

5.2.2. Experimental procedures

A Rofin-Dilas High-Power Diode Laser (HPDL) with a wavelength of 940 nm and a maximum output power of 1300 W was used. Argon was applied as the protective and powder carrier gas. To deposit the cermet coatings, the laser beam power was fixed at 900 W, the scanning speed at 15 mm/s, the powder feeding rate at 16.5 g/min, and the flux of Ar between 14 and 15 L/min. The substrates were coated by 10 single clad tracks with a 40% overlap between two adjacent tracks [169].

Metallographic samples were prepared in plain-view section. The coated specimens were ground with SiC paper up to 1200 grit to remove the superficial roughness of the coatings. Successively, they were polished in a diamond slurry of up to 1 μm nominal size. Finally, the polished surfaces were cleaned in deionised water and then by ultrasound in acetone and propanol. The same procedure was followed to obtain a polished surface of the Inconel 600 bulk sample.

Depth sensing indentations tests (DSI) were carried out with a Nanoindenter XP (MTS systems Co.), on the polished surfaces of the samples, by using the continuous stiffness measurement methodology (CSM) [97]. Continuous loading and unloading cycles were conducted during the loading branch by imposing a small dynamic oscillation of 2 nm and 45 Hz on the displacement signal and measuring the amplitude and phase of the corresponding force. Consequently, the contact stiffness was continuously measured as a function of the penetration depth during the experiment. Two different batches of indentation tests were carried out on the Inconel bulk and cermet samples. For each batch, an indentation matrix of 10x10 indentations, spaced 50 microns between them, was made in displacement control. The first batch of indentation tests was carried out using a Berkovich diamond indenter with a tip radius of 50 nm. A maximum penetration depth of 1000 nm was selected to carry out the DSI tests with the Berkovich tip. The aim of these tests was to obtain values of the Young's modulus (E) and hardness (H) of the studied materials. Both properties were obtained following the Oliver-Pharr methodology [20]. The other batch of indentation tests was carried out using a spherical diamond indenter with a tip radius of 9.3 μm . The aim of these tests was to study the local plastic

properties and obtain the indentation stress-strain curve of both samples. A maximum penetration depth of 1,500nm was selected to carry out the DSI tests with the spherical tip. Prior to making the indentations, a tip calibration procedure was carried out using the bulk Inconel 600 alloy as the reference material, in accordance with the CSM methodology [121, 156, 172]. The nominal elastic modulus was set to 214 GPa and the real contact area of the indenter was iteratively obtained through the following equation.

$$A_c = c_0 h_c^2 + c_1 h_c + c_2 h_c^{1/2} + c_3 h_c^{1/4} + \dots \quad (5.1)$$

Where c_i are constants determined by the curve fitting procedure. The first term was set as 24.5 for an ideal Berkovich indenter, meanwhile for the spherical indenter, the first and second terms were set as $-\pi$ and $2\pi R$, respectively, where R is the spherical indenter radius.

In addition, Vicker microhardness indentation tests were carried out on the polished surfaces of the materials studied, with a maximum load of 300 gf and a dwell time of 12 s.

5.3. Result and discussion

5.3.1. Study of the cermet coating microstructure

In a previous work [169], the microstructure of the cermet matrix was analysed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). It was observed the presence of unmelted Cr_3C_2 particles randomly distributed in the Inconel matrix. Additionally, Cr-rich carbides of stoichiometry $M_7\text{C}_3$, radially distributed around the unmelted Cr_3C_2 particles, were also observed. They were formed *in-situ* during the laser cladding.

5.3.2. DSI tests with Berkovich indenter tip

Fig.5.1 shows a representative indentation load vs. penetration depth curves obtained from the DSI tests with the Berkovich tip. The indentation loads reached at the maximum penetration of 1000 nm for the tests carried out in the cermet matrix were higher than those obtained from the Inconel bulk. Additionally, the Cr_3C_2 carbides showed the highest indentation loads.

Fig. 5.1 :Representative penetration depth vs. load curves obtained from DSI tests performed with the Berkovich tip on the cermet matrix, the Inconel 600 bulk and the unmelted Cr_3C_2 particles.

Fig.5.2 shows representative examples of Young's modulus vs. penetration depth and Hardness vs. penetration depth curves obtained from the DSI tests. The Young's modulus of the materials studied were slightly constant with the penetration depth. Examples of the results of the variations of H with the penetration depth are shown in Fig.5.2 (b), (d), and (f). The unmelt Cr_3C_2 particles showed constant values of H with the penetration depth. However, the cermet matrix and the Inconel 600 bulk showed a continuous decreasing tendency of the hardness values with the penetration depth. As the values of E remain constant, it is possible to discard the dependence of the H values to errors of the area function calibration of the indenter tip. Consequently, the hardness evolution observed with the penetration depth may be a material response to the indentation process. This phenomenon is known as indentation size effect (ISE) [173] and the hardness values obtained on the cermet coating could be affected by this effect. Different models were suggested to analyze the ISE and calculate the asymptotic hardness (H_0) at full plasticity condition. This hardness value is comparable to the Vickers one at the macroscale. Nix and Gao [173] proposed a methodology to determine the asymptotic hardness. These authors suggested the presence of geometrically necessary dislocations (GNDs) in the material structure, in addition to statistically stored dislocations (SSDs) produced during uniform straining, that leads to an extra hardening component that becomes bigger as the contact impression decreases in size [99]:

$$\frac{H}{H_0} = \sqrt{1 + \frac{h^*}{h}} \quad (5.2)$$

Where H represents the apparent hardness; H_0 , the asymptotic hardness; h , the penetration depth, and h^* , the characteristic h from which H become independent of the penetration depth. Eq.(5.2) implies that a plot H^2 vs. $1/h$ results in a straight line that intercepts the y-axis at H_0^2 .

Fig. 5.2 : Representative result of Young's modulus and hardness variation with penetration depth obtained from the DSI tests with Berkovich tip in (a) (b) Inconel 600 bulk, (c) (d) cermet matrix, and (e) (f) unmelt Cr_3C_2 particles.

Fig. 5.3 shows a representative plot of H^2 vs. $1/h$ for the Inconel 600 bulk (a) and cermet matrix (b), respectively. A good linear fitting of the data is obtained in accordance with the Nix and Gao model.

Fig. 5.3 : Example of H^2 vs. $1/h$ curves for the Inconel 600 bulk (a) and cermet matrix (b) obtained from a Berkovich indenter.

Table.5.2 summarizes the average hardness measured from the DSI tests carried out with the Berkovich indenter, the values of H_0 calculated with the Nix and Gao model, and finally the values of Vicker microhardness (HV) that could be used to verify whether the values of H_0 were correctly calculated.

Table 5-2: Average hardness measured in DSI tests with Berkovich indenter (H), asymptotic hardness (H_0), and Vickers microhardness (HV).

Material	E (GPa)	H (GPa)	H_0 (GPa)	HV (GPa)
Inconel 600 bulk	211 ± 7	3.7 ± 0.2	2.4 ± 0.3	2.7 ± 0.1
Cermet matrix	243 ± 12	7.2 ± 0.6	3.85 ± 0.3	4.5 ± 0.07

The asymptotic hardness values obtained on the Inconel bulk and cermet coating were similar to those obtained by Vickers microhardness tests. In addition, the average hardness value of the cermet matrix was 60% higher than that of the Inconel 600 bulk. The greater hardness of the cermet matrix is connected with the formation *in-situ* of the previously described M_7C_3 carbides. The Young's modulus of the cermet matrix was 15% higher than that obtained on the Inconel 600 bulk. It is possible to suppose that M_7C_3 carbides, embedded into

the Inconel matrix, promote load distribution phenomena that leads to higher values of E measured. The values of E obtained for the Cr_3C_2 particles are similar to those reported by others authors ($E=373\text{GPa}$) [174].

5.3.3. DSI tests with Spherical indenter tip

Fig.5.4 shows representative load vs. penetration depth curves obtained in DSI tests carried out using the spherical indenter tip. A higher indentation load is necessary for the cermet matrix with respect to the Inconel 600 bulk to achieve the same penetration depth, in agreement with the results obtained using the Berkovich tip. It should be noted that there are multiple discontinuities in the curve of the unmelted Cr_3C_2 particle. This phenomenon is known as “pop-in” and it is associated with the formation of cracks in the material during the tests. Materials with hexagonal lattice structure, such as the Cr_3C_2 powders particles used in this work, could present this behaviour. Other examples of materials demonstrating this phenomenon are the Sapphire, the GaN, and the ZnO [175]. In the case proposed in Fig.5.4, the first pop-in happens at an indentation depth of around 400 nm, the second one at 550 nm, and the third one at around 700 nm. There is no pop-in phenomenon during the unloading process. As the pop-in affects the results, only the data prior to the first pop-in event were used in the data analysis.

Fig. 5.4: Penetration depth versus load from Spherical indentation on cermet matrix, Inconel bulk and unmelted Cr_3C_2 .

Indentation tests with a spherical tip represent the best choice for characterising the strain hardening of the cermet matrix which offer a gradual transition from the elastic to the elastic-plastic regime. The indentation stress vs. strain curve can be obtained using the model proposed by S.R. Kalidindi et al.[71]. As the value of the Young's modulus of the materials studied was measured using the Oliver-Pharr method on the Berkovich indentation data, the contact radius (a) could be estimated according to Sneddon's equation [17]:

$$a = \frac{S_u}{2E_r} \quad (5.3)$$

where S_u represents the harmonic stiffness obtained by applying the CSM method and E_r is the reduced Young's modulus, that could be expressed as a function of the Young's modulus and Poisson's ratios of the studied material (E , ν) and the indenter tip (E_i , ν_i):

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \quad (5.4)$$

The indentation stress (σ_{ind}) and the indentation strain (ε_{ind}) can be expressed respectively as [25]:

$$\sigma_{ind} = \frac{P}{\pi a^2} \quad (5.5)$$

$$\varepsilon_{ind} = \frac{h}{2.4a} \quad (5.6)$$

The slope of the initial linear part of the indentation stress-strain curves, obtained from the cermet matrix and the Inconel 600 bulk, is approximately equal to the reduced Young's modulus obtained from the Oliver-Pharr analysis of the Berkovich indentation tests (in the zoomed parts in Fig.5.5). The plastic regime of the indentation stress strain curves were fitted to Hollomon's equation[176]. The indentation stress-strain curve for the cermet matrix shows a strain hardening value of more than twice that obtained for the Inconel 600 bulk. Probably, the M_7C_3 carbides, distributed around the unmelted Cr_3C_2 particles, could produce a distortional effect of the cermet matrix and consequently, they may produce a hardening effect. This result is in agreement with that obtained from the Berkovich tests (Table 5.2).

Fig. 5.5 : Indentation stress-strain curve for the cermet matrix and the Inconel 600 bulk.

Fig. 5.6: Indentation stress strain curve for unmelted particle Cr_3C_2 .

Fig.5.6 shows the indentation stress-strain curve for an unmelted Cr_3C_2 particle. The fitting of the experimental data was limited to those under the first pop-in event. The curve

obtained is linear with a slope that corresponds to the Young's modulus (364GPa) of an unmelted Cr_3C_2 particle. The critical point marked in red in the curve is the first pop-in point, at which the initial cracking process is produced. The mean pressure at this pop-in point is about 18 GPa which is consistent with the values of hardness obtained from the Vicker indentation in literature [174, 177].

Conclusions

In this chapter, DSI tests were carried out with Berkovich and spherical tips on a Inconel 625- Cr_3C_2 cermet coating to evaluate its elastic-plastic properties at the micro-scale. An Inconel 600 bulk specimen was used as the reference sample. Shown below is a summary of the main results obtained:

1. With the test carried out with the Berkovich tip, the hardness value of the cermet matrix was 60% higher than that of the Inconel 600 bulk, while the Young's modulus of the cermet matrix was 15% higher as regards that obtained on the Inconel 600 bulk. Additionally, the hardness of cermet matrix and the Inconel 600 bulk showed an indentation size effect which obeyed the Nix-Gao model.
2. In the spherical nanoindentation test, the indentation stress-strain curve in the plastic regime for the cermet matrix showed a strain hardening value of more than twice that obtained for the Inconel 600 bulk.
3. The unmelted Cr_3C_2 reinforced particle was also characterized. The hardness values did not show any indentation size effect. Meanwhile, there were multiple discontinuities in loading-displacement curve of unmelted Cr_3C_2 obtained from spherical indentations. The first pop-in event was observed at a penetration depth of about 400 nm and the corresponding average pressure was similar to the hardness measured from the Vicker indentations.

Chapter 6 : Conclusions and future work

6.1. Conclusions

In this work, the analytical methodologies for extracting the elastic-plastic properties of metallic materials using the instrumented indentation technique have been proposed. The main contributions of this thesis are the following:

- A simple analytical method for calculating the Young's modulus and hardness of bulk elastic-plastic materials with the Berkovich indentation has been proposed and experimentally validated. The proposed method requires the $P-h^2$ and S_u-h curves to show a linear behaviour. If this requirement is fulfilled, a procedure for selecting the $P-h^2$ and S_u-h data that are not affected by the tip bluntness is followed. The proposed method gives values of Young's modulus that are very similar to those measured using traditional testing techniques, i.e. the tensile and impulse excitation test. Meanwhile, it also provides hardness values that are comparable to those obtained by microindentation. This work has shown that the proposed method can successfully reduce the detrimental effect of tip bluntness and the plasticity phenomena under the indenter tip on the estimation of Young's modulus by sharp instrumented indentation on elastic-plastic materials. In addition, the calibration of the contact area is not necessary in the proposed method, which makes it simpler and faster.
- A new analytical procedure is proposed to extract the representative stress strain curve of metallic material through spherical indentation. Based on the formation of Tabor's equation, a new insight into the stress and strain constraint factors is studied analytically.

From the intersection point between the Hertz equation and fully plastic period which obeys Hollomon's equation, the product, ϕ of the strain constraint factor, β and stress constraint factor, ψ is constant at the transition point between the elastic regime and the fully plastic regime, which is related to the Young's modulus of contact materials. Both experimental and FEM procedures are performed to evaluate the analytical analysis for extracting the representative stress strain curve from the spherical indentation. The stress constraint factor is taken as 3.2. Consequently, the strain constraint factor could be obtained from the constant product, ϕ . From the FEM and experimental results, the representative stress strain curve, using the proposed constraint factors in the study, shows a good agreement with the nominal true stress strain curve. It should be noted that due to the limitation of Tabor's equation in the fully plastic regime, the representative stress strain curve derived from the new proposed procedure could be more suited to materials with a shorter elastic-plastic transition defined by $(E/\sigma_y)(a/R)$.

- Depth sensing indentation tests were carried out with Berkovich and spherical tips on an Inconel 625-Cr₃C₂ cermet coating to evaluate its elastic-plastic properties at the micro scale. With the test carried out using the Berkovich tip, the hardness value of the cermet matrix was 60% higher than that of the Inconel 600 bulk, while the Young's modulus of the cermet matrix was 15% higher than that obtained from the Inconel 600 bulk. Additionally, the hardness of the cermet matrix and the Inconel 600 bulk showed an indentation size effect which obeyed the Nix-Gao model. The spherical indentation stress-strain curve in the plastic regime for the cermet matrix showed a strain hardening value of more than twice that obtained for the Inconel 600 bulk. Moreover, the unmelted Cr₃C₂ reinforced particle is also characterized. The first pop-in event was observed at a penetration depth of about 400 nm and the corresponding average pressure was similar to the hardness measured from the Vicker indentations.

6.2. Limitation and Future work

Although the proposed methodologies could accurately determine the elastic-plastic properties of metallic material, further studies are needed to improve their efficiency and accuracy. In this work, the elastic-plastic properties of metallic material are obtained from different indenters. Consequently, changing the tip could make the measuring procedure more complex. It is necessary to develop a methodology for extracting the elastic-plastic properties with an identical indenter.

The method for measuring Young's modulus and hardness with the Berkovich indenter requires the critical depth from which the $P-h^2$ and S_u-h curves show a linear behaviour to be identified. However, in the case of measuring the coating system, the penetration depth of indenter should less than 10% percent of the matrix height. As a result, the proposed method will be not valid when the critical length is longer than 10% percent of the matrix height in the coating system. An indenter with smaller face angle is necessary to use the proposed method. In future, the proposed method for measuring the elastic property will be validated by a proper coating system.

In the method of extracting the representative stress-strain curve using a spherical indenter, the contact radius is estimated from Sneddon's equation. However, corrections of Sneddon's equation related to bodies of revolution and inward radial displacements of materials under the indenter are not considered. In future, these corrections will be incorporated to improve the proposed method. Moreover, the analytical procedure for extracting the representative stress strain curve with a spherical indenter is based on Tabor's equation, however, due to the the limitation of Tabor's equation, the representative stress-strain curve could give rise to a large derivation in the materials with a longer elastic-plastic transition defined by $(E/\sigma_y)(a/R)$. It is important to study this problem and develop a new definition of representative strain.

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