Bulletin of the *Transilvania* University of Braşov
Series I: Engineering Sciences • Vol. 4 (53) No. 2 - 2011

TESTING THE MECHANICAL PROPERTIES OF MATERIALS AT NANOSCALE; THE NANOINDENTATION METHOD

L. COSTINESCU[1] A. MARIN[1]
C. COJOCARIU[1] D. MUNTEANU[1]

Abstract: *Within the frame of this paper a brief history of methods developed and efforts made over time, to test the mechanical properties of materials at nanoscale is presented. The development of indentation methodologies for the micro mechanical characterization of materials requires a precise understanding of the correlation between uniaxial mechanical properties and hardness. A special attention was given to the evolution of nanoindentation method.*

Key words: *nanoindentation, hardness, mechanical properties.*

1. Introduction

As defined by the Webster dictionary, nanotechnology is "*the art of manipulation of devices tiny molecular size*". Called "*the twenty-first century manufacturing technology"*, nanotechnology includes precision engineering and its development is closely related to the investigative tools at the nanoscale material [25], [30].

Recently, many efforts have been made in developing nanoindentation equipment and nanoindentation techniques for probing the mechanical properties of materials and thin film on the sub-micron and nanoscale [18], [6], [12], [32].

In this context we can say that nanoindentation method gained popularity with the development of machines that can record small load and displacement with high accuracy and precision and also because Analytical models by which the load-displacement data can be used to determine modulus, hardness and other mechanical properties [38].

2. History of Determining the Mechanical Properties

The nature of the stresses arising from the contact between two elastic bodies was first studied by Hertz in 1881. His theory is found to be very accurately describing the stress, strain, and displacement fields in the elastic specimen by comparing with the finite element simulation results. Hertz theory is to be served as verification of current finite element modeling. The problem of determining the distribution of stress within an elastic half space when it is deformed by the normal pressure against

[1] Dept. of Technological Equipment and Materials Science, *Transilvania* University of Braşov.

its boundary of a rigid punch have been considered first by Boussinesq in 1893.

Another major contribution was made by Ian N. Sneddon [24] who derived general relationships among the load, displacement, and contact area for any punch that can be described as a solid of revolution of a smooth function [34].

Although the concept of a mechanical test for metals based on forcing a strong indenter into a plane surface dates back at least 150 years [4], the modern interpretation of the hardness of a metal as the pressure resisting plastic indentation by a comparatively strong and stiff indenter of well defined geometry originated in 1900 with the work of Brinell [11].

In the Brinell test, a hard ball of diameter D, originally of hardened steel but later of cemented tungsten carbide, is pressed under a load W into the plane surface under test [1], [31].

The Meyer hardness H_M, first defined in 1908 is determined by ball indentation in exactly the same way, but it is defined as the load divided by the projected area of the indentation [11].

The Vickers test was first described in 1922, [11], [23] and was commercialized by the Firth-Vickers company. It uses a diamond indenter in the form of a square-based pyramid, with an angle of 136º between the faces.

The Rockwell test, [21] for which a patent was filed in 1914 but which was first used commercially in the early 1920s, was more suited to automated use by less-skilled labor [11].

The Rockwell hardness test method consists of indenting the test material with a diamond cone or hardened steel ball indenter. The indenter is forced into the test material under a preliminary minor load F_0 usually 10 kgf. The permanent increase in depth of penetration, resulting from the application and removal of the additional major load is used to calculate the Rockwell hardness number [34].

The development of indentation methodologies for the micro mechanical characterization of materials requires a precise understanding of the correlation between uniaxial mechanical properties and hardness. One of such fundamental correlations was found by Tabor [34], for pyramidal (Vickers) indenters.

David Tabor's contribution to the science of indentation hardness, his interest in which started with his first published paper in 1939 and continued until his last paper almost 60 years later, was immense. Tabor's book - *The Hardness of Metals*, published in 1951, has had a major influence on the subject of indentation hardness and is by far the most widely cited source in this area [11].

Perhaps the most common means of testing mechanical properties of materials at micro and nano-scale is instrumented indentation. The method was developed in 1992 by Oliver and Pharr and quickly superseded previous, more lengthy tests [19], [17], [28].

In Figure 1 we can see the original nanoindenter built in Switzerland. The basic components of the system are not so different from the systems that we see today [35].

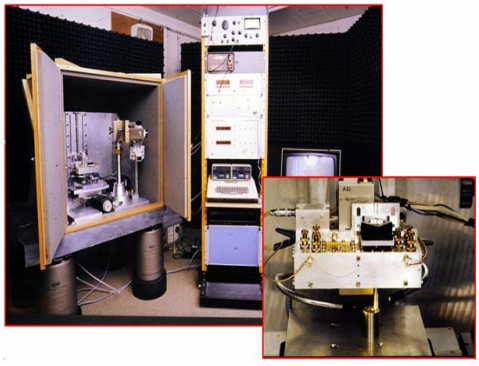

Fig. 1. *The first Nanoindenter built by Pethica, Hutchings, and Oliver, in 1983* [35]

3. Analytical Models, Comments and Discussion

By far the most successful and widespread model for nanoindentation data analysis is one in which the unloading data are assumed to arise from a purely elastic contact (Hertzian contact). This approach was developed over 40 years with contributions from a number of groups around the world.

The form most often used is that presented by Oliver and Pharr, and is known as the Oliver and Pharr method [36]. Nearly all of the elements of this analysis were first developed by workers at the Baikov Institute of Metallurgy in Moscow during the 1970's (for a review see Bulychev and Alekhin) [36], [2].

The basic assumptions of this approach are:

a) Deformation upon unloading is purely elastic;

b) The compliance of the sample and of the indenter tip can be combined as springs in series:

$$\frac{1}{E_r} = \frac{1-\nu_i^2}{E_i} + \frac{1-\nu_s^2}{E_s}, \tag{1}$$

where E_r is the "reduced modulus", E is the Young modulus, n is the Poisson ratio and i and s refer to the indenter and sample respectively.

c) The contact can be modelled using an analytical model for contact between a rigid indenter of defined shape with a homogeneous isotropic elastic half space using:

$$S = \frac{2\sqrt{A}}{\sqrt{\pi}} E_r, \tag{2}$$

where S is the contact stiffness and A the contact area. This relation was presented by Sneddon [36], [24]. Later, Pharr, Oliver and Brotzen [36], [19] where able to show that (2) is a robust equation which applies to tips with a wide range of shapes.

The first complete nanoindentation data analysis was presented by Doerner and Nix, [36], [6] who argued that, if the change in contact area is small during unloading, the indenter can be treated as a flat punch. Assuming that, during loading, all of the material in contact with the indenter is plastically deformed and that outside the contact only elastic deformation occurs at the surface, they were able to make the connection between P-h data and the contact area.

Let the distance along the indenter axis that the indenter is in contact with the sample material be h_c, the "contact depth". As, according to the afore made assumption, an extrapolation of the slope S of the unloading data at maximum load P_{max} (the maximum contact stiffness) to the displacement axis yields the contact depth of the indent, see Figure 2 [36].

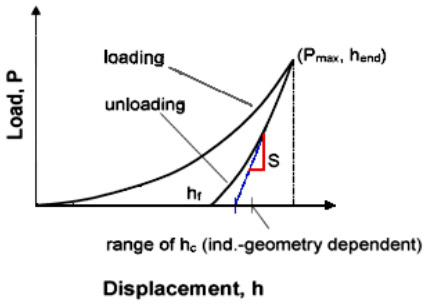

Fig. 2. *Schematic plot of the Doerner and Nix model* [36]

To find the hardness, a measure of the indentation area is needed. A convenient way to do this is to use the projected contact area at maximum load. If the tip shape is accurately known, a tip 'area function' can be generated [36]:

$$A_c = f(h_c). \tag{3}$$

Doerner and Nix measure the shape of their indenter by making a series of indents in soft brass and relating the contact depth, obtained as described above, to the contact

area measured by imaging acetate replicas of those indentations in the Transmission Electron Microscope (TEM). The hardness is obtained simply as:

$$H = \frac{P_{\max}}{A(h_c)}. \tag{4}$$

Note that this definition of the hardness is different from the conventional definition of hardness. In the nanoindentation analysis the hardness is calculated utilizing the contact area at maximum load whereas in conventional tests the area of the residual indent after unloading is used.

Since an elastic contact analysis is used, the elastic modulus of the material can be obtained. The contact area from (3) is combined with the stiffness at maximum load in (2) to find the reduced modulus in (1). If E and n for the indenter material are known, the ratio $E/(1-n^2)$ for the sample material can be obtained [36].

Oliver and Pharr (O&P) [36], [17] made a critical improvement to the method proposed by Doerner and Nix (D&N). Sneddon's contact solution [36], [24] predicts that the unloading data for an elastic contact for many simple indenter geometries (sphere, cone, flat punch and paraboloids of revolution) follows a power law that can be written as follows:

$$P = \alpha \cdot h^{\mathrm{m}}. \tag{5}$$

In this equation P is the indenter load, h is the elastic displacement of the indenter and α and m are constants. Oliver and Pharr apply this formulation to determine the contact area at maximum load as it is valid even if the contact area changes during unloading. To do this, they derive the following relationship for the contact depth from Sneddon's solutions [36], [24]:

$$h_c = h_{end} - \theta \frac{P_{\max}}{S}, \tag{6}$$

where θ = 0.72, 0.75 and 1, for cone, sphere and flat-punch-geometry respectively.

The equation (6) can be found also as follows:

$$h_c = h_{\max} - \varepsilon \frac{P_{\max}}{S}. \tag{6'}$$

The procedure for O&P analysis is to then fit a power law function to the unloading segment. This yields the contact stiffness as slope of this function at maximum load. This slope in addition to the appropriate value of q is used in order to determine the actual contact depth so that it is finally possible to derive the indentation modulus (2) and the indentation hardness (3) from the measurement. Figure 3 shows a schematic sketch of such an analysis [36], [17].

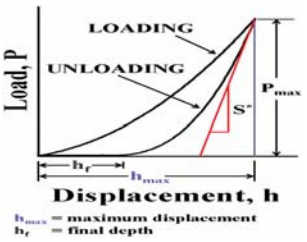

Fig. 3. *An indentation force-displacement curve* [29]

Both values, indentation modulus as well as indentation hardness, depend strongly on the area function $A(h_c)$ and the accuracy with which it is determined [36].

Since 1992, the analysis method proposed by Oliver and Pharr [17] has been established as the standard procedure for determining the hardness and elastic modulus from the indentation load-displacement curves for bulk materials. In the Oliver-Pharr method, the projected contact area between indenter tip and material is estimated using the equations for the elastic contact of an indenter of arbitrary shape on a uniform and isotropic half space [24]. The indentation

modulus and hardness of the material can thus be calculated without the necessity of imaging the indentation after the experiment. The Oliver-Pharr method was initially developed for analyzing indentations in bulk materials, not for films on substrates, and no information about a possible substrate is included in the analysis. The Oliver-Pharr method is, however, frequently used by researchers to interpret indentations performed on thin films in an attempt to obtain approximate film properties regardless of the effect of substrate properties on the measurement. The accuracy of such a measurement depends on the film and substrate properties and on the indentation depth as a fraction of the total film thickness [3], [7], [20], [16], [22]. To minimize the effect of the substrate on the measurement, the indentation depth is often limited to less than 10% of the film thickness [20]. This empirical rule is not always reliable, especially if the elastic mismatch between film and substrate is large. The 10% rule is also not useful for very thin films when experimental issues make it difficult to obtain accurate results for very shallow indentations. Evidently there exists a need for a method that can be used to analyze thin-film indentation data for indentation depths where the substrate effect cannot be ignored [10].

Indentation methods are finding increasing use in the study of mechanical properties of bulk and thin-film materials over a wide range of size scales. There are basically two theoretical branches applied in indentation research, which are classified by the indentation size scale.

One is traditional continuum mechanics while the other is newly developed strain gradient plasticity theory [8], [9], [27], [14].

Continuum mechanics has enjoyed a tremendous success in many disciplines of engineering, in which typical length scales of components and deformation are larger than millimeters. In recent years, continuum mechanics has been applied to microelectronic industry, where the characteristic length scales are very small, typically from 0.1 to 10 microns. Accordingly, several experiments have been developed to measure mesoscale mechanical properties [34].

Recent experiments have shown that materials display strong size effects when the characteristic length scale associated with non-uniform plastic deformation is on the order of microns. For example, Fleck et al. [8], [9] observed in the twisting of thin copper wires that the scaled strength increases by a factor of three as the wire diameter decreases from 170 to 12 microns, while the increase of work hardening in simple tension is negligible. In bending, of ultra thin beams, Stolken and Evans [27] observed a significant increase in the normalized bending hardening as the beam thickness decreases from 100 to 12.5 microns, while data from simple tension displays no size dependence. For an aluminum-silicon matrix reinforced by silicon carbide particles, Lloyd [13] observed a substantial strength increase when the particle diameter was reduced from 16 to 7.5 microns with the particles volume fraction fixed at 15%. More convincing experimental evidence of the size dependence of material behavior at the micron level comes from the micro-indentation or nano-indentation hardness test. The measured indentation decreases from 10 microns to 1 micron [34], [26], [14], [15].

The classical plasticity theories cannot predict this size dependence of material behavior at the micron or nano scale because their constitutive models possess no internal length scale.

4. Conclusions

Current developments and trends in microelectronics are focused on thin layers and novel materials. This leads to application

of different test and measurement methods, which are capable to measure basic mechanical properties of such materials on micro-scale and nano-scale. Currently nanoindentation technique is used in microelectronics in order to extract material properties of thin layers, which is important factor in the area of reliability analysis [33].Based on the first model of Indentor, many instruments cover a mechanical properties characterization of thin films, coatings and substrates, as can be seen in the Figure 4 [37].

Fig. 4. *Modern Nanoindentation Platform, CSM Instruments type* [37]

In order to finde the starting moment of radial bearings, a mechanical system was designed and produced, (Figure 5) [5].

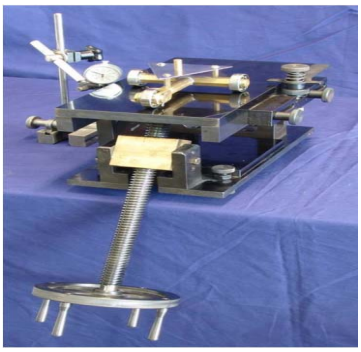

Fig. 5. *Mechanical system consisting of a device of three radial bearings and a highprecision tribometer* [5]

Acknowledgements

The author A. Marin thanks to the Sectorial Operational Programme Human Resources Development (SOP HRD), ID76945 financed from the European Social Fund and by Romanian Government.

References

1. Brinell, J.A.: *Way of Determining the Hardness of Bodies and Some Applications of the Same.* In: Teknisk Tidskrift **5** (1900), p. 69-87.
2. Bulychev, S.I., Alekhin, V.P., Shorshorov, M.K., Ternovskii, A.P., Shnyrev, G.D.: *Determining Young's Modulus from the Indenter Penetration Diagram.* In: Industrial Laboratory (USSR) (English translation of Zavodskaya Laboratoriya) **41** (1975), p. 1409-1412.
3. Burnett, P.J., Rickerby, D.S.: *The Mechanical Properties of Wear-resistent Coatings. 1. Modeling of Hardness Behavior.* In: Thin Solid Films **148** (1987) No. 1, p. 41-50.
4. Calvert, F.C., Johnson, R.: *On the Hardness of Metals and Alloys.* In: Philosophical Magazine Series 4, **17** (1859), p. 114-121.
5. Cozma, R.: *A Study of Global Friction Coefficient and the Starting Moment Variation for Radial Bearings.* In: Bulletin of the *Transilvania* University of Braşov (2009) Vol. 2 (51), Series I, p. 41-46.
6. Doerner, M.F., Nix, W.D.: A *Method for Interpreting the Data from Depth-sensing Indentation Instruments.* In: Journal of Materials Research **1** (1986), p. 601-616.
7. Fabes, B.D., Oliver, W.C., McKee, R.A., Walker, F.J.: *The Determination of Film Hardness from the Composite Response of Film and Substrate to*

Nanometer Scale Indentations. In: Journal of Materials Research **7** (1992) No. 11, p. 3056-3064.
8. Fleck, N.A., Hutchinson, J.W.: *A Phenomenological Theory for Strain Gradient Plasticity.* In: Journal of the Mechanics and Physics of Solids **41** (1993) No. 12, p. 1825-1857.
9. Fleck, N.A., Muller, G.M., Ashby, M.F., Hutchinson, J.W.: *Strain Gradient Plasticity: Theory and Experiment.* In: Acta Metallurgica et Materialia **42** (1994), p. 475-487.
10. Han, L., Vlassak, J.J.: *Determining the Elastic Modulus and Hardness of an Ultrathin Film on a Substrate Using Nanoindentation.* In: Journal of Materials Research **24** (2009) No. 3, p. 1114-1126.
11. Hutchings, I.M.: *The Contributions of David Tabor to the Science of Indentation Hardness.* In: Journal of Materials Research **24** (2009) No. 3, p. 581-589.
12. Joslin, D.L., Oliver, W.C.: *A New Method for Analyzing Data from Continuous Depth-Sensing Microindentation Tests*. In: Journal of Materials Research **5** (1990), p. 123-126.
13. Lloyd, D.J.: *Particle Reinforced Aluminum and Magnesium Matrix Composites*. In: International Materials Reviews **39** (1994), p. 1-23.
14. Ma, Q., Clarke, D.R.: *Size Dependent Hardness of Silver Single Crystals.* In: Journal of Materials Research **10** (1996), p. 853-863.
15. McElhaney, K.W., Vlassak, J.J., Nix, W.D.: *Determination of Indenter Tip Geometry and Indentation Contact Area for Depth-Sensing Indentation Experiments.* In: Journal of Materials Research **13** (1998), p. 1300-1306.
16. Mencik, J., Munz, D., Quandt E., Weppelmann, E.R., Swain M.V.: *Determination of Elastic Modulus of Thin Layers Using Nanoindentation.* In: Journal of Materials Research **12** (1997) No. 9, p. 2475-2484.
17. Oliver, W.C., Pharr, G.M.: *An Improved Technique for Determining Hardness and Elastic-Modulus Using Load and Displacement Sensing Indentation Experiments.* In: Journal of Materials Research **7** (1992) No. 6, p. 1564-1583.
18. Panich, N., Yong, S.: *Improved Method to Determine the Hardness and Elastic Moduli Using Nano-Indentation*. In: KMITL Science Journal **5** (2005) No. 2, p. 483-492.
19. Pharr, G.M., Oliver, W.C., Brotzen, F.R.: *On the Generality of the Relationship between Contact Stiffness, Contact Area, and Elastic Moduli during Indentation*. In: Journal of Materials Research **7** (1992) No. 3, p. 613-617.
20. Pharr, G.M., Oliver W.C.: *Measurement of Thin-film Mechanical Properties Using Nanoindentation.* In: MRS Bulletin **17** (1992) No. 7, p. 28-33.
21. Rockwell, S.P.: *The Testing of Metals for Hardness*. In: Transactions of the American Society for Steel Treating **2** (1922), p. 1013-1033.
22. Saha, R., Nix W.D.: *Effects of the Substrate on the Determination of Thin Film Mechanical Properties by Nanoindentation.* In: Acta Materialia **50** (2002) No. 1, p. 23-38.
23. Smith, R., Sandland, G.: *An Accurate Method of Determining the Hardness of Metals, with Particular Reference to Those of a High Degree of Hardness*. In: Proceedings of the Institution of Mechanical Engineers **1** (1922), p. 623-641.
24. Sneddon, I.N.: *The Relation between Load and Penetration in the Axisymmetric Boussinesq Problem for a Punch of Arbitrary Profile.* In:

International Journal of Engineering Science **3** (1965), p. 47-57.
25. Stamatin, I.: *Nanomateriale, Aplicaţii în biosenzori, surse de energie, medicină, biologie* (*Nanomaterials Applications In Biosensors, Renewable Energy, Medicine, Biology*). In: Elemente de Nanotehnologie, Universitatea din Bucureşti, 2008.
26. Stelmashenko, N.A., Walls, A.G., Brown, L.M., Milman, Y.V.: *Microindentations on W and Mo Oriented Single Crystals: an STM Study*. In: Acta Metallurgica et Materialia **41** (1993), p. 2855-2865.
27. Stolken, J.S., Evans, A.G.*: A Microbend Test Method for Measuring the Plasticity Length Scale.* In: Acta Materialia **46** (1998), p. 5109-5115.
28. Stratil, P.: *Analysis of Instrumented Indentation.* In: Ph.D. Thesis, Masaryk University, Brno, 2009.
29. VanLandingham, M.R.: *Review of Instrumented Indentation.* In: Journal of Research of the National Institute of Standards and Technology **108** (2003) No. 4, p. 249-265.
30. Vedinas, I., Cretu, E.: *Elemente de nanotehnologie* (*Elements of Nanotechnology*). Bucureşti. Universitatea Titu Maiorescu, 2007.
31. Wahlberg, A.: *Brinell's Method of Determining Hardness and Other Properties of Iron and Steel*. In: Journal of Iron and Steel Institute **59** (1901), p. 243-298.
32. Woirgard, J., Dargenton, J-C.: *An Alternative Method for Penetration Depth Determination in Nanoindentation Measurements*. In: Journal of Materials Research **12** (1997*)*, p. 2455-2458.
33. Wymysłowski, A., Dowhań, Ł., Wittler, O., Mrossko, R., Dudek, R.: *Application of Nanoindentation Technique to Extraction of Thin Films Properties through Experimental and Numerical Analysis.* In: Materials Science-Poland **28** (2010) No. 3, p. 655-662.
34. Yao, Z.: *Development of an Indentation Method for Material Surface Mechanical Properties Measurement*. In: Ph.D. Thesis, West Virginia University, 2005.
35. https://agilenteseminar.webex.com. Accessed: 14.06.2011.
36. http://www.nanoindentation.cornell.edu/Model/elastic_model.htm. Accessed: 06.07.2011.
37. www.csm-Instruments.com. Accessed: 24.09.2011.
38. www.slideshare.net/viet4777/nano-indentation-lecture2. Accessed:13.06.2011.