A NEW METHOD TO EVALUATE BOTH HARDNESS AND ELASTIC MODULAUS OF COATED THIN FILMS.

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Abstract: There are several methods to study the mechanical properties of coated materials. In this paper we report a novel method to determine the mechanical properties of a coated thin film by nano indentation.ich is a very popular and advanced method to evaluate the mechanical properties of a film under low load conditions which is a maximum limit of 500mN. In this paper the films are applied with different thickness on the silicon substrates, the thickness values are taken like 400nm, 700nm and 1000nm.

Key Words: Nano indentation, thin films, coatings, hardness, elastic modulus, mechanical properties.

Introduction: The nature of the stresses arising from the contact between two elastic bodies was first studied by Hertz in 1881[1]. 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.

Another major contribution was made by Ian N. Sneddon 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.

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, 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. 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.

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.

The Vickers test was first described in 1922, 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, 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. 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 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. 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, 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,

Theory: 2.1 Mathematical Formulae: The most common means of testing mechanical properties of materials at micro and nano-scale is an instrumented indentation. The method was developed in 1992 by Oliver and Pharr [1] and quickly superseded previous, lengthy tests.

A popular application of contact mechanics is found in depth-sensing or instrumented indentation. This type of indentation testing is usually applied to depths of penetration in the sub-micron range and is termed "nanoindentation". In this type of test, the applied load and the depth of penetration of an indenter into the specimen are recorded and used to indirectly determine the area of contact and hence the hardness of the test specimen. It is the most popular application of nanoindentation testing is that of thin films, the method is equally useful for any specimen whose mechanical properties over small size scales need to be measured. Surface modified layers, individual phases or grains in a ceramic are typical examples. The testing finds common application in the biological field (teeth and bone), the semiconductor industry, ceramics, thin films, polymers, and most recently, MEMs testing, etc.

The three-sided Berkovich indenter [] is the most popular geometry for nanoindentation testing since the tip of the indenter can be made very sharp and avoids the line of conjunction usually found in a Vickers indenter. The face half-angle of a Berkovich indenter is $65.27°≈65.3°$. This gives the same projected area to depth ratio as a four-sided Vickers

indenter (face angle 68).

The essential element of a depth-sensing or instrumented indentation test is the load-displacement curve. Usually, such a curve consists of a loading part (containing both elastic and plastic deformations) followed by an unloading part.

In the initial part of the loading response, it is important to understand that there is a transition from purely elastic contact to a plastic contact even for a Berkovich indenter. For an initial elastic contact, the mean contact pressure increases with increasing load as predicted by the Hertz contact equations. At the condition of a fully-developed plastic zone, the mean contact pressure levels off to a constant value with increasing load and this value of mean contact pressure is called the hardness. Once the penetration depth becomes larger than the tip radius, the pyramidal shape of the indenter becomes the dominant geometrical feature of the indentation

It is the data taken on the unloading that is used to determine the area of contact at the maximum load. This is done by using equations of contact for a conical indenter where we mathematically transform the actual pyramidal geometry into an equivalent cone. That is, we find a cone angle that gives the same area-to depth ratio as the actual pyramidal indenter.

The contact area can be calculated from geometry for a Berkovich indenter of face angle "θ", and then the area is give by

$$A = 3\sqrt{3}h_c^2 \tan^2\theta \qquad 1$$

Where **hc** is the distance measured vertically from the indenter tip.

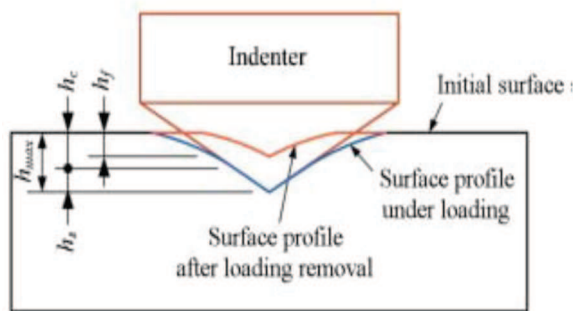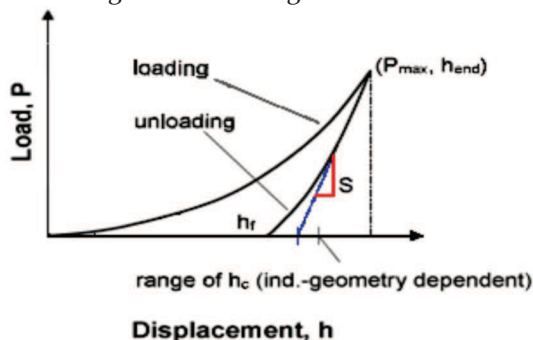Fig1: Sketch of indentation profile

Fig 2: Loading and Unloading curve

As mentioned previously, the main outcome of a depth-sensing indentation test is the load-displacement curve. For hardness measurements, the maximum load is selected so as to be certain that a fully developed plastic zone in the specimen material has been achieved. This is sometimes difficult to achieve in the case of nanoindentation testing of thin films whereby it is required to limit the total penetration depth to usually less than 10% of the film thickness [1],[4],[5],[6] (to avoid or reduce influence from the substrate). For this reason, it is important to have a very sharp tip for thin film testing. At full load, a hold period is often applied to account for creep effects before the indenter is unloaded. Creep and thermal drift in nanoindentation testing are very important sources of error and are difficult to distinguish. In both cases, for a constant load hold period, the depth reading may change. In the case of thermal drift, the depth reading may increase (deeper into the specimen) with time or decrease. When creep occurs, the depth reading usually increases. Thermal drift errors are usually brought about by changes in the dimension of the contact (indenter, indenter shaft, and specimen) from thermal expansion or contraction due to temperature changes during testing.

After maximum load, or the optional hold period, the applied load is reduced and the resulting penetration depth recorded. The unloading process is usually assumed to be an entirely elastic affair. The elastic strains relax during unloading and the surface of the material attempts to recover its original shape. Full elastic recovery is usually prevented from happening by the presence of the plastic zone. The incomplete elastic recovery can easily be identified on the load displacement curve. For a fully elastic contact, such as what can happen with a spherical indenter, the unloading curve lies on top of the loading curve. The area enclosed between the loading and unloading curves represents the energy lost as heat during plastic deformation. The slope of the unloading curve at any point is called the contact stiffness.The elastic unloading curve can be used with elastic equations of contact to determine the area of the contact under load and hence the hardness.

The included angle of the Berkovich indenter (65.3°) gives the same projected area for the same depth of penetration as the Vickers indenter (angle 68°).

It is the data taken on the unloading that is used to determine the area of contact at the maximum load. This is done by using equations of contact for a conical indenter where we mathematically transform the actual pyramidal geometry into an equivalent cone. That is, we find a cone angle that gives the same area-to depth ratio as the actual pyramidal indenter. This can be calculated from geometry.

If d is the length of one side of the triangular impression, then the projected area of contact is given by:

$$A_P = (\sqrt{3}/4)\, d^2 \qquad 3$$

Treating that the contact is an axis-symmetric cone, the contact between a rigid conical indenter and an elastic half space is found from

$$P = \frac{\pi a}{2}\, E^* a \cot\alpha \qquad 4$$

Where α is the effective cone semi-angle (70.3° for a Berkovich indenter). The quantity $a\cot\alpha$ is the depth of penetration h_c measured at the circle of contact.

The depth beneath the specimen free surface within the circle of contact is given by

$$h = \left(\frac{\pi}{2} - \frac{r}{a}\right) a \cot\alpha \quad r \le a \qquad 5$$

Setting r=0 and substituting into equation 5, then we obtain:

$$P = \frac{2E}{\pi} h^2 \tan\alpha \qquad 6$$

Historically, it was noted that the initial unloading response for many materials was linear. This implied that the contact conditions were those of a cylindrical punch instead of a cone where:

$$P = 2aE^* h \qquad 7$$

Where a is the radius of the indenter equal to the radius of the circle of contact and where it should be noted that the depth of penetration is linearly dependent on the load. Then the derivative of the load P with respect to the displacement h (the contact stiffness) is given by:

$$\frac{dP}{dh} = 2\,\frac{2E\tan\alpha}{\pi}\, h \qquad 8$$

Substituting into equation 6, then we get

$$P = \frac{1}{2}\frac{dP}{dh}\, h \qquad 9$$

If the total depth of penetration is h_{max}, at load Pmax, then as the load is removed, the indenter moves through a distance h_e as shown in Fig.1.

At $P = P_{max}$ the displacements $h_r = 0 = h_e$ and $h_r = a = h_a$. From Eq.5 at r = a, the plastic (or contact) depth h_c is found from:

$$h_c = h_t - \left[\frac{2(\pi - 2)}{\pi}\right]\frac{P_{max}}{dp/dh} \qquad 10$$

Where P_{max} and dP/dh are measured during an experiment. The square-bracketed term in Eq.10 is often given the symbol ϵ and evaluates to 0.72 but it is common practice to use a value of 0.75 since this has been shown to account for non-uniformities in the material response as the load is withdrawn.

Once a value for h_c has been determined, the area of contact is found from Eq.1 where, for a Berkovich indenter (α = 70.3°),

$$A = 24.5\, h_c^2 \qquad 11$$

From the above equations the reduced elastic modulus is found that

$$E^* = \frac{dP}{dh}\frac{1}{2}\frac{\sqrt{\pi}}{\sqrt{A}} \qquad 12$$

Experiments and finite element analysis shows that a correction factor β is needed for Eq. 12. The correction factor is applied as the factor $1/\beta$ to the measured value of dP/dh. Accordingly we have:

$$E^* = \frac{1}{\beta}\frac{dP}{dh}\frac{1}{2}\frac{\sqrt{\pi}}{\sqrt{A}} \qquad 13$$

and so Eq. 10 becomes

$$h_c = h_t - \epsilon\beta\frac{P_{max}}{dp/dh} \qquad 14$$

where dP/dh is the actual experimental quantity. There are various estimates and explanations of the value of β in the literature, the most popular being that of King at 1.034[3].

From this test the hardness of a thin film is obtained by the well known relation:

$$H = \frac{P_{max}}{A} \qquad 15$$

Experimental Details:

The Nanoindentation tests are planned to carrying on a Nano-indenter XP (MTS) tester. All the experiments are conducting by the Berkovich indenter, which was made of diamond.
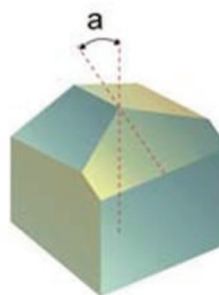
The system is equipped with a continuous stiffness measurement (CSM) module and thus is capable of giving the mechanical properties as a function of depth.

- Maximum Depth - 500 µm
- Depth Resolution - <0.01 nm
- Maximum Load - 500 mN (10N high load)
- Load Resolution - 50 nN

Fig3: Nano- Indenter[xp] (Mts) Tester

Fig 4: Berkovich indenter

3.1 Description:

The NanoIndenter performs indentation tests by driving a diamond indenter into the specimen surface and dynamically collecting the applied force and displacement data. Material properties are derived from the load and depth data. Specimens are typically relatively smooth and flat. Test positions are targeted using an optical

imaging system with a 250X display. To perform the indentation test on CSM type system, the typical indenter used as Berkovich indenter.

Micro hardness testing on a very small scale is conveniently carried out using a Berkovich three-sided pyramidal indenter, since the facets of the pyramid may be constructed to meet at a single point rather than a line, which usually results at the apex of a four-sided pyramidal indenter. The Berkovich indenter is thus very useful for the investigation of the mechanical properties of thin films such as optical coatings, paint, and hard coatings on machine tools. For this experimental work the coating materials are prepared and synthesized by processes like Physical vapour deposition (PVD), chemical vapour deposition (CVD) and sol-gel method.

Results And Disscusion:

Nano-indentation experiments have been conducted on polycrystalline thin films of Copper, 400, 700 or 1000 nm in thickness, on silicon substrates. The load-displacement curves exhibit periodic bursts in indentor penetration depth, which is interpreted chiefly to be a consequence of the nucleation of dislocations. The first departure from elastic deformation appears to occur when the maximum shear stress at the tip of the indentor reaches the theoretical shear strength of copper, and is, as anticipated, essentially independent of film thickness. The bursts of dislocation nucleation, occurring at approximately constant indentation loads, are separated by an elastic deformation response which conforms to Kick's law for the sharp Berkovich indentor and which can be estimated solely from the elastic properties of the film. The overall elastoplastic response of the film to nano-indentation, which comprises elastic deformation of

the film and microplasticity in the film arising from dislocation nucleation, appears to be sensitive to the film thickness in that the resistance to nano-indentation systematically decreases with increasing film thickness. This trend is fully consistent with curvature measurements on film–substrate systems which reveal that the 'average' yield strength of the thin film increases with decreasing film thickness.
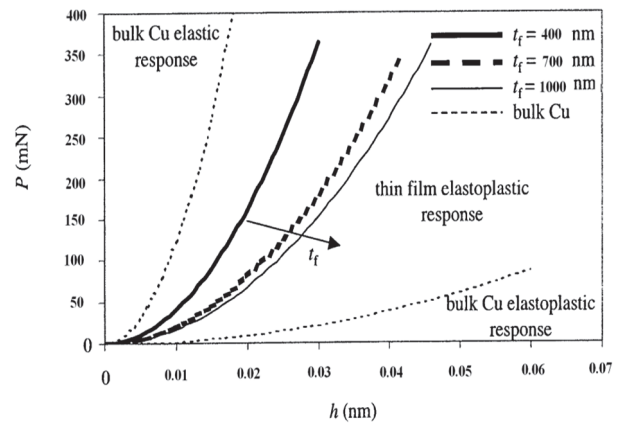

Figure 5: Variations shown in the resistance to indentation.

5. **Conclusion:**

Current developments and trends in microelectronics are focused on thin film. The application of nano indentation technique is used in microelectronics in order to extract the mechanical properties of the thin layers, which is a important factor in the area of reliability analysis. In this work copper was chosen as the material for the thin film in view of its recent emergence as the interconnect material of choice in microelectronics devices.

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