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Film Hardness Evaluation in Hard Film/Substrate Composites by Conical Indentation

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The hardness of the bulky or covered materials is typically obtained through indentation techniques. Due to the complexity of the influence of the micro- or nanohardness of the coating and the substrate on the measuring of the composite hardness, various mathematical and geometrical models based on the area law-of-mixtures' advance are founded. The present study offers mathematical and geometrical modelling of the coefficient of the area law-of-mixtures' for conical indentation. The project imprints are considered as disks and the coefficients α, β of the area law of mixtures become ratios of circle surfaces. The hardness of the composite and the substrate is expressed as a function of the imprint projected dimensions and the applied load. Finally, the film contribution is determined from the proposed modelling of the area law-of-mixtures' model.

Key words: hardness, non-destructive testing, hardness test, microindentation, modelling.

Твердість об'ємних матеріялів або їхніх покриттів зазвичай визначається за допомогою техніки індентування. Через складний вплив мікро- або нанотвердости покриття та підкладинки на міряння твердости композиту використовуються різні математичні та геометричні моделі, засновані на

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удосконаленому правилі сумішей. У даній роботі запропоновано математичне та геометричне моделювання коефіцієнта правила сумішей для конічного індентування. Проєкції відбитків вважаються дисками, а коефіцієнти α, β правила сумішей дорівнюють відношенню площ кіл. Твердість композиту та підкладинки виражається як функція розмірів проєкцій відбитків і прикладеного навантаження. Внесок від плівки визначається моделюванням в рамках правила сумішей.

Ключові слова: твердість, неруйнівний контроль, випробування на твердість, мікроіндентування, моделювання.

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1. INTRODUCTION

The indentation test is a great way to characterize a number of mechanical properties of materials at the micro/nanoscale. It has been identified as one of the key features of the estimation of mechanical properties, and it has been used successfully to determine the thin film hardness coated material.

In materials science, hardness is a measure of the resistance to localized plastic deformation induced by the mechanical indentation; there are three categories of mechanical hardness measurements: scratch, indentation, and rebound. The indentation hardness value was determined by dividing the value of the applied force of indentation with the contact imprint surface [1–19]. In general, the microhardness or nanohardness and elastic modulus of an elastic–plastic material can be evaluated from the force–displacement curve [1–3].

Owing to equipment limitations such as the tip of the penetrator shape, it is difficult to achieve significant experimental results in the depth value [4]. Thus, to get the real film hardness by the technique of micro- or nanoindentation, it is necessary to establishing geometrical and mathematical modelling.

Since the efficient data of the geometric form of indenter is the key of indentation, we have chosen to use an indenter of a conical geometric form to penetrate coated materials, where we used the area law-of-mixtures models to determine the coating hardness as function of the imprint depth, the composite hardness H_c and of the substrate hardness H_s.

We have considered the projected imprints of the substrate and the composite as disks of the radius r^*, r respectively, and the coefficient α of the area law-of-mixtures as ratio of circle surface.

Finally, geometrical and mathematical modellings have been used to establish the expression of the coating micro- or nanohardness of the coated material, wherever discussions and comparisons of figures and formulas were presented.

2. COMPOSITE HARDNESS OF MONOLAYER COATED MATERIAL

To avoid the complexity of the influence of the micro- or nanohardness of the coating and substrate on the measured composite hardness, numerous mathematical models based on the area law-of-mixtures advance [14–24] have been developed and established to separate the microhardness or nanohardness of film from the substrate [5, 6, 23, 24].

The area law-of-mixtures models presuppose the validity of a linear law of mixtures to express the measured composite hardness H_c, as a function of the film hardness H_f, and of the substrate hardness H_s.

In one form, all models express the composite hardness of monolayer-coated material according to the following area law-of-mixtures:

$$H_c = \alpha H_f + \beta H_s, \ \alpha + \beta = 1 . \tag{1}$$

Jönsson and Hogmark models (Fig. 1) is one of the most model; it gives the composite microhardness H_c, in function of the substrate microhardness H_s, the coating microhardness H_f, the surface of the thin film S_f, the substrate surface S_s and the total the imprint surface S_c as follows [4–6, 7–19]:

$$H_c = \frac{S_f}{S_c} H_f + \frac{S_s}{S_c} H_s . \tag{2}$$

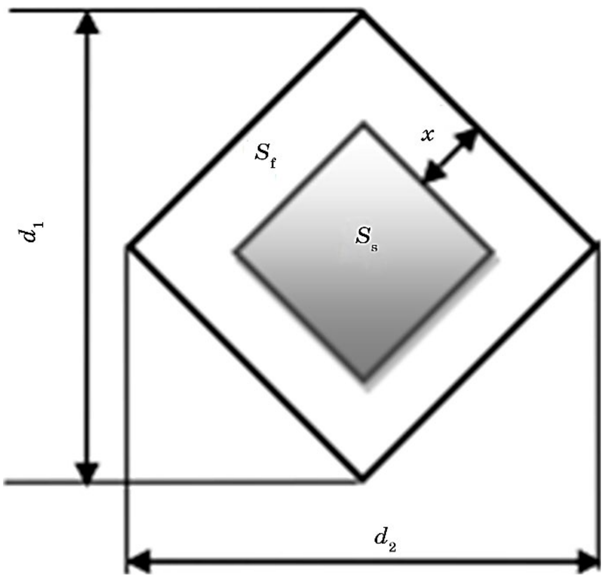

Fig. 1. Semi-load-supporting areas of monolayer composite in Jönsson and Hogmark model [7].

From Figure 1, the composite surface gets the following expression:

$$S_c = d^2 / 2,\ d = (d_1 + d_2) / 2. \quad (3)$$

Then, the substrate surface becomes as

$$S_s = (d / \sqrt{2} - 2X)^2. \quad (4)$$

Subsequently, the surface of the coating becomes

$$S_f = S_c - S_s = d^2 / 2 - (d / \sqrt{2} - 2X)^2. \quad (5)$$

3. MATHEMATICAL CONCEPTS OF THE RIGHT CIRCULAR CONE

The right circular cone is a three-dimensional geometric form; it has a flat round face on one side and a pointy end on the other side. It has a line that touches its apex point in a perpendicular of the centre of its round base. Moreover, its apex lies just over its base centre. Its conical surface is a ruled surface formed by fixing one end of a line segment at the cone vertex and sweeping the other around its circular base perimeter.

In a Cartesian co-ordinate system, the right cone of a round base of the height H and the radius R, oriented along the Z-axis, with a vertex pointing down and a base positioned at $Z = H$ (Fig. 2), can be described as by the following equation:

$$x^2 + y^2 - (R / H)^2 Z^2 = 0, \quad (6)$$

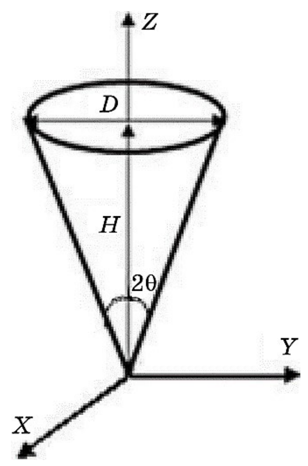

Fig. 2. Right cone in Cartesian co-ordinates.

where $x^2 + y^2 = r^2$; then, the characteristic equation of the right con becomes

$$r^2 - (R / H)^2 Z^2 = 0 . \quad (7)$$

4. HARDNESS OF CONICAL INDENTATION

4.1. Massive Material Hardness

The hardness values were defined as the ratio of the applied force to the resulting indentation area. According to the conical form of the indenter, the hardness can be given by the following expression [1–19]:

$$H_c = F / S . \quad (8)$$

The indentation of a right cone of a circular base of diameter D and height H into the tested surface makes an imprint of conical forms. Its projection is a disk area (Fig. 3) of a radius r and a surface S [20]:

$$S = \pi r^2 . \quad (9)$$

The hardness H_{Con} as function of the applied force F and the surface

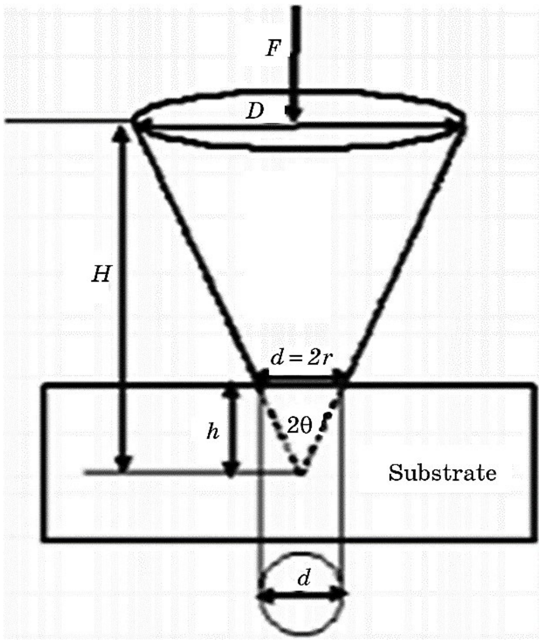

Fig. 3. Principle of penetration of the right cone.

of the resulting imprint gets the following equation:

$$H_{\mathrm{Con}} = F / \pi r^2 . \tag{10}$$

Since $\mathrm{tg}\theta = r/h = R/H \to r = R/H$, the hardness becomes, according of the load F, the cone dimension (H, R), and the imprint depth h:

$$H_{\mathrm{Con}} = \frac{H^2 F}{\pi (hR)^2} . \tag{11}$$

4.2. Composite Hardness Modelling

Whereas, there are always complexities of the measurement of the coating micro- or nanohardness of covered surfaces, and it is indispensable to generate a model for the coupled thin coating–substrate behaviour under penetration.

According to the area law-of-mixtures approach, the composite hardness of a monolayer coating (substrate + film) takes the following additive law [17, 29, 30]:

$$H_{\mathrm{c}} = \alpha H_{\mathrm{f}} + (1 - \alpha) H_{\mathrm{s}} . \tag{12}$$

The coefficient α can be written as ratio of the projected surfaces of the resulting imprints as the following [21]:

$$\alpha = S_{\mathrm{f}} / S_{\mathrm{c}} . \tag{13}$$

The indentation of a sufficiently hard indenter of a right cone shape into a composite material of a coating thickness e results a cone imprint shape of a circular base of radius r and height h. Its projection presents the composite imprint (Fig. 4). Then, the projection of the composite imprint gets a disk form of a surface:

$$S_{\mathrm{c}} = \pi r^2 . \tag{14}$$

So, the composite hardness can be expressed by the following equation:

$$H_{\mathrm{c}} = F / \pi r^2 . \tag{15}$$

From Figures 4 and 5, the projected surface of the substrate imprint gets the following expression:

$$S_{\mathrm{s}} = \pi (r^*)^2 = \pi (r - \Delta r)^2 . \tag{16}$$

The projected surface of the film imprint takes the following formula:

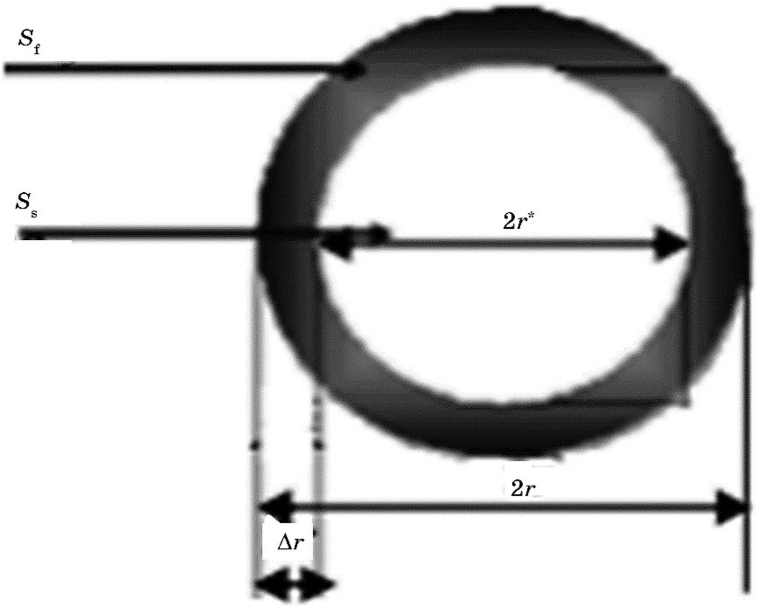

Fig. 4. Cross-section of load-supporting areas of film and substrate.

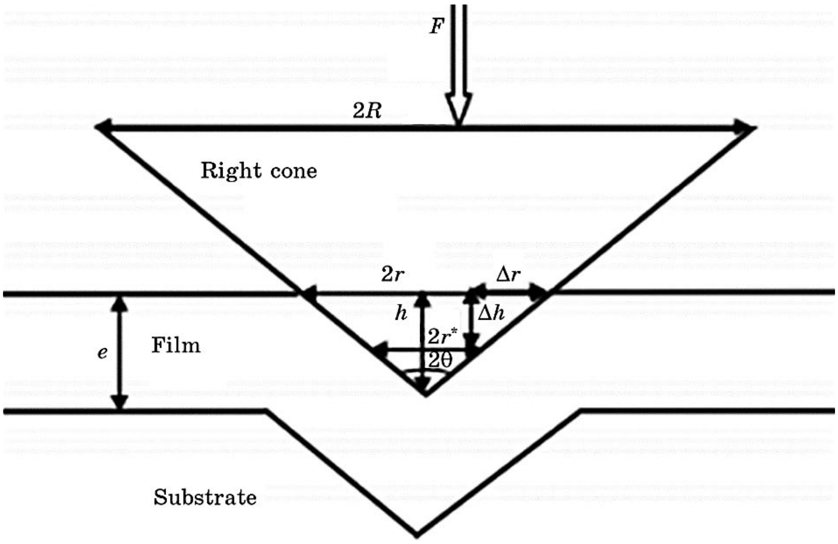

Fig. 5. Section scheme of the cone indentation on a coated solid.

$$S_f = S_c - S_s = \pi(r^2 - (r - \Delta r)^2 . \tag{17}$$

So, substituting Eqs. (14) and (17) into Eq. (13), one can obtain the coefficient α of the law of the surfaces' mixture:

$$\alpha = \frac{S_f}{S_c} = \frac{2\Delta r}{r} - \left(\frac{\Delta r}{r}\right)^2 . \quad (18)$$

From Figure 5, we can write:

$$\frac{\Delta r}{\Delta h} = \frac{R}{H} = \frac{r}{h} \to \Delta r = \frac{R}{H}\Delta h = \frac{r}{h}\Delta h . \quad (19)$$

By replacing Eq. (18) into Eq. (19), the area coefficient α becomes

$$\alpha = \frac{2\Delta h}{h} - \left(\frac{\Delta h}{h}\right)^2 , \quad (20)$$

where the film deformation is considered as $\Delta h = Ce$, and the area fraction α gets

$$\alpha = 2\frac{Ce}{h} - \left(\frac{Ce}{h}\right)^2 . \quad (21)$$

The constant C can be expressed in relation to the apical semi-angle θ of the indenter. It is equivalent to $\cos^2\theta$ for a plastic film and to $(1 - \sin\theta)$ for cracked film [28].

In case of a coated material of a plastic film, the coefficient α gets the following formula:

$$\alpha = \frac{2e}{h}\cos^2\theta - \left(\frac{e}{h}\cos^2\theta\right)^2 = \cos^2\theta\left(\frac{2e}{h} - \frac{e}{h}\cos^2\theta\right). \quad (22)$$

But, if cracks develop in the film, the coefficient α is defined as follows:

$$\alpha = \frac{2e(1-\sin\theta)}{h} - \left(\frac{e(1-\sin\theta)}{h}\right)^2 . \quad (23)$$

From Eq. (12), the film hardness H_f becomes the function of the composite hardness H_c, the substrate hardness H_s, and the coefficient α as follows:

$$H_f = \{H_c - (1-\alpha)H_s\} / \alpha . \quad (24)$$

5. DISCUSSION AND COMPARISON TO KNOWN FIGURES AND FORMULAS

In accordance with the above geometrical and mathematical modelling of the coefficient α, the following cases can be noted.

1. In the first case, the indenter does not penetrate the film of the coated material; in this course, the area fraction α was discussed in the states of plastically deformed film, when cracks develop into the film.

Plastically deformed film. In this situation, we consider two types of indenters' tip. The first is very sharp cone, where the apical semi-angle θ is small ($\cos^2\theta \approx 1$), and consequently, the area fraction α becomes as

$$\alpha = \frac{2e}{h} - \left(\frac{e}{h}\right)^2. \tag{25}$$

Then, the replacing of Eq. (25) into Eq. (24) gives the film hardness H_f as the function of the composite hardness H_c, the substrate hardness H_s, the film thickness e, and the depth h as the following:

$$H_f = \frac{H_c - \left(1 - \frac{2e}{h} + \left(\frac{e}{h}\right)^2\right) H_s}{\frac{2e}{h} - \left(\frac{e}{h}\right)^2}. \tag{26}$$

The second is cone of blunt tip; the apical semi-angle θ is approximately right ($\cos^2\theta \approx 0$), and the area fraction α becomes as

$$\alpha = 2e\cos^2\theta / h. \tag{27}$$

Then, the film hardness H_f becomes the function of the composite hardness H_c, the substrate hardness H_s, the film thickness e, the apical semi-angle of the indenter θ, and the depth h:

$$H_f = \frac{H_c - \left(1 - \frac{2e\cos^2\theta}{h}\right) H_s}{2e\cos^2\theta / h}. \tag{28}$$

Cracks develop in the film. For a very sharp cone, the apical semi-angle θ of the indenter is approximately null ($\sin\theta \approx 0$), and

$$\alpha = \frac{2e}{h} - \left(\frac{e}{h}\right)^2. \tag{29}$$

Then, the film hardness H_f gets the following formula:

$$H_f = \frac{H_c - \left(1 - \frac{2e}{h} + \left(\frac{e}{h}\right)^2\right) H_s}{\frac{2e}{h} - \left(\frac{e}{h}\right)^2}. \tag{30}$$

But, for the case of a blunt cone $(1-\sin\theta)^2 \approx 0$, the coefficient α becomes

$$\alpha = 2e(1-\sin\theta)/h\,. \tag{31}$$

Then, the film hardness can be written as follows:

$$H_\mathrm{f} = \frac{H_\mathrm{c} - \left(1 - \dfrac{2e(1-\sin\theta)}{h}\right)H_\mathrm{s}}{2e(1-\sin\theta)/h}\,. \tag{32}$$

2. In the second case, the film of the coated material penetrated by the indenter (Fig. 6), the film deformation is known; it gets $\Delta h = e$. In the course of this assumption, the area fraction becomes as the following:

$$\alpha = 2\frac{e}{h} - \left(\frac{e}{h}\right)^2. \tag{33}$$

Then, the film hardness H_f becomes the function of the composite hardness H_c, the substrate hardness H_s, the film thickness e, and the depth h as the following:

$$H_\mathrm{f} = \frac{H_\mathrm{c} - \left(1 - \dfrac{2e}{h} + \left(\dfrac{e}{h}\right)^2\right)H_\mathrm{s}}{\dfrac{2e}{h} - \left(\dfrac{e}{h}\right)^2}\,. \tag{34}$$

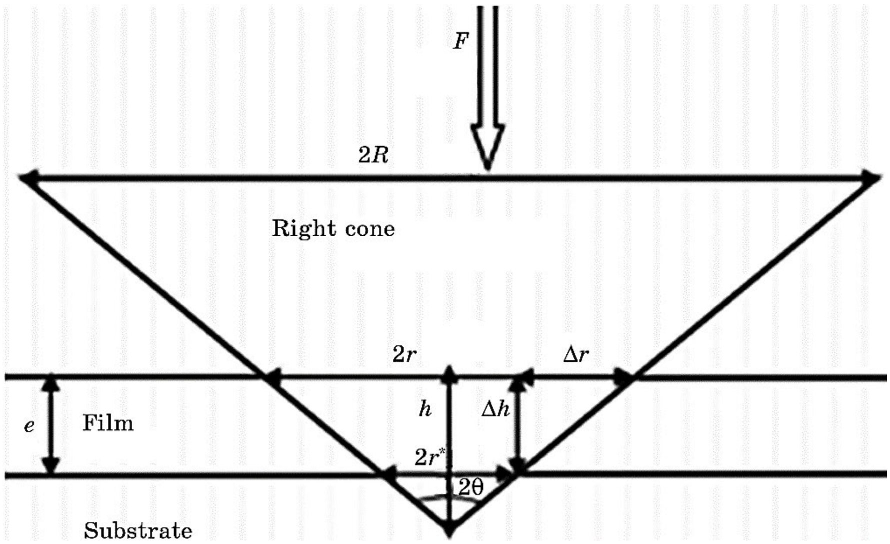

Fig. 6. The scheme of the right cone penetration.

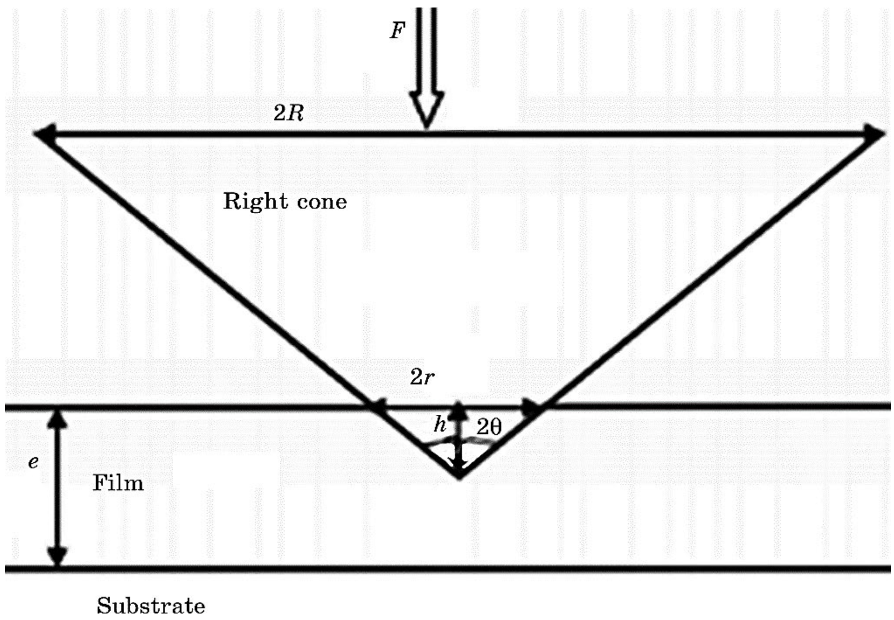

Fig. 7. The scheme of the film indentation.

3. In the third case, when there is not an effect of the substrate on the coated material and the cone penetrate only the thin film (Fig. 7), the film deformation Δh is equivalent to the depth h, then, the area fraction gets the following formula:

$$\alpha = \frac{2\Delta h}{h} - \left(\frac{\Delta h}{h}\right)^2 = 1. \tag{35}$$

Then, the area law-of-mixtures approach can be simplified as follow:

$$H_c = H_f. \tag{36}$$

6. CONCLUSION

This work presents geometrical and mathematical modelling aimed at separating the contribution of the substrate to the hardness of solid surface monolayer when the indenter is a cone; it is established the area law-of-mixtures expressions of monolayer covered materials.

The indentation of a solid monolayer coating by a non-deformable conical tip grants the simple expression of the area law-of-mixtures model and permits to find a relationship between the nano- or micro-hardness film H_f, the composite hardness H_c, the substrate hardness H_s, the film thickness e, and sizes of the resulting conical imprint r, h.

The monolayer indentation by a sharp conical penetrator ($R << H$) products a coefficient expression α for breaking film equal to that of the plastic film, which makes to conclude the decreasing of the area imprint and the phenomena occurring during and after the tests (cracking, deformation, *etc.*).

Similar to the Jönsson and Hogmark model, this study shows that the microhardness of the thin film of solid monolayer can be determined through the measurements of the composite hardness H_c, the substrate hardness H_s, the imprint sizes r, h, the film thickness e.

Finally, we think that the characterization of the bulky and coated material hardness by the indentation of a conical tip shape has the theoretical and experimental importance in field of the contact mechanic and the mechanical characterization of the solid materials, which will carry to extend the areas of the application of the indentation and scratch tests by an indenter of the con tip form.

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