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Hardness of Thin Films and the Influential Factors

İshak Afşin Kariper

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Abstract

Hardness is one of the most significant mechanical characteristics of a material. Hard materials are known for their durability. Currently, diamond is the hardest substance known in the world. Researchers have substantially worked on the production of this expensive material. Coating this material as a thin film was also the topic of a separate research. At the same time, hardness measurement, including the measurement of the hardness of a thin film was a topic of research as well. In this section, we will examine what researchers are doing for the measurement of the hardness, especially for bulk materials.

Keywords: Nanohardness, thin films, hardness, influential factors, diamond

1. Introduction

Hard materials are very useful for the industry and technology researches. SmCo5 and Nd2Fe14B can be used to produce televisions, video-recording devices, and speakers, with their magnetic-hard properties. Even consumers need and demand hard materials because they are long-lasting. Hard materials are highly effective when they are used to cover a surface. They are also used as diffusion barriers in electronic industry, as they preserve their chemical stability under high temperature and prevent the diffusion of foreign atoms. This allows electronic materials to be more durable than in the past (Mechanical corrosion is another important problem). Especially in mechanical industry, these materials (hard thin films) are very useful to prevent substances' mechanical corrosion. Based on the aforementioned reasons, nowadays, hard thin films have a significant place in coating.

In the abstract, we have underlined that hardness is one of the most important mechanical characteristics of a material. You can get insights about other characteristics of the material by

measuring its hardness. Hardness is an indicator of the material's resistance against scratching, cutting, abrasion, and puncture. Table lists the hardness of some materials in terms of absolute hardness (**Table 1**).

Mineral	Absolute hardness
Talk: $Mg_3Si_4O_{10}(OH)_2$	1
Gypsum: $CaSO_4.2H_2O$	2
Calcite: $CaCO_3$	9
Fluorite: CaF_2	21
Quartz: SiO_2	100
Topaz: $Al_2SiO_4(OH,F)_2$	200
Ruby or Sapphire: $Cr–Al_2O_3$	400
Diamond: C	1500

Table 1. Absolute hardness of some materials.

Nanoscale, that is, a few hundred nanometers in size, materials are called nanomaterials. Thanks to nanomaterials, the technology has made great progress. The properties of a material in nanoscale are very different than the ones in the bulk form, which created great advantages for the technology. Especially, coating these materials to the surface as a thin film has added new properties to the base material. However, there is very little information in the literature on the mechanical properties of thin films [1].

After coating process, the coated material can gain electrical, optical, corrosive, and cracking resistance, depending on the thickness of the film. A material with such properties will be very handy in space research, astronomy, automobile industry, and in many other areas of engineering. For example, imagine that you can cover any surface with a heat-resistant material. This can be used in a wide range of area, from space engineering to the clothing of firefighters. On the other hand, if we could cover the surface of a material with diamond, we could make many materials harder and at the same time more robust [2].

When you produce such materials, another problem that you will be faced will be how to measure the hardness of the material when it is coated with such a thin film. In the literature, the hardness of thin films is generally measured by the indentation method [3], even though it has some limitations. The hardness of the substrate also affects the measurement, for example, while measuring, the hardness of soft thin films or a soft substrate may affect the hardness of the final product even though the coating that you have produced is very hard. Thus, the type of coating and the substrate is crucial.

Researchers are currently working on the measurement of the hardness of the thin films. All of them apply 10% rule in their studies. Accordingly, when a hollow-shaped hole is made on the surface of the film, the depth of this hole should be less than 10% of the film thick-

ness. In this way, the effect of the base material to the hardness (and to the measurement) will be lower than 2% [4–8]. This is quite logical for the thin films, since their thickness is lower than 1000 nm; the depth of the hole should be around 100 nm. These units can be easily measured with the current technology [9]. In this topic, the impact of the hole's geometric shape on the mechanical properties, that is, hardness, also attracted the attention of the researchers [3]. We can see some outstanding studies in the literature about hardness of thin films in **Table 2**.

Author/s	Article	Material	Subject
M. Draissia, H. Boudemagh, and M. Y. Debili	Structure and hardness of the sputtered Al–Cu thin films system	Al–Cu thin film	Hardness of film according to % Cu [10]
• Lin-Dong Wang, • Min Li, • Tai-Hua Zhang, • Nai-Gang Liang	Hardness measurement and evaluation of thin film on material surface	None	Geometric effect [3]
• Seung Min Han, • Ranjana Saha, • William D. Nix	Determining hardness of thin films in elastically mismatched film-on-substrate systems using nanoindentation	Al/Si, Al/sapphire, W/Si, and W/glass	Hardness measurement [11]
Yu. A. Bykov, S. D. Karpukhin, Y. V. Panfilov, M. K. Boichenko, V. O. Cheptsov, and A. V. Osipov	Measurement of the hardness of thin films	TiN	Measurement of hardness and film thickness effect [12]
J. L. He, W. Z. Li and H. D. Li	Hardness measurement of thin films: separation from composite hardness	TiN, Cu	Measurement of hardness [13]
S. Chen, L. Liu, T. Wang	Investigation of the mechanical properties of thin films by nanoindentation, considering the effects of thickness and different coating–substrate combinations	Al, W	Measurement of hardness and film thickness effect [14]
Y.-G. Jung, B. R. Lawn, M. Martyniuk, H. Huang, X.Z. Hu	Evaluation of elastic modulus and hardness of thin films by nanoindentation	None	Elastic modulus and hardness of thin films [15]
S. Liu, H. Huang, Y. T. Gu	Hardness of silicon nitride thin films characterized by nanoindentation and nanoscratch deconvolution methods	SiN	Hardness of silicon nitride thin films [16]
N. Demas, C. Lorenzo-Martin, O.O. Ajayi, R.A. Erck, and I. Shareef	Measurement of thin-film coating hardness in the presence of contamination and roughness: implications for tribology	TiAlN, CrN	Hardness of thin film effect on tribology [17]

Author/s	Article	Material	Subject
P. D. Tall, S. Ndiaye, A. C. Beye, Z. Zong, W. O. Soboyejo, H.-J. Lee, A. G. Ramirez, K. Rajan	Nanoindentation of Ni–Ti thin films	Ni–Ti	Hardness of alloys thin film [18]

Table 2. Hardness of thin films in the literature.

In this chapter, we tried to formulize and explain the methods that can be used to measure the hardness of a thin film, the parameters affecting the hardness of a thin film, and the impacts of other parameters such as temperature, elasticity module, which also affect hardness, on the thin films.

2. Methods

The logic of hardness measurement is to indent the material to be tested with another material, whose hardness is known, and measure the resistance that the tested material displayed against it. The material that is used to measure the hardness (indenter) should be much harder than the tested material. Thus, mechanical defects that may occur during the measurement would be equilibrated. The geometric shape of the indenter is also important. It is usually chosen among the geometric shapes whose area can easily be calculated, such as ball (sphere), pyramid, or cone.

The usual method to achieve the hardness is to form a scratch, having an area (A), using an indenter by applying a specific force (F). Therefore, hardness H is

$$H = \frac{F}{A} \tag{1}$$

The area (A) formed on the surface of the material is negatively proportional with the hardness of the material. The following precautions should be taken while making the measurements:

1. The measurement should be made on a flat surface.

2. The thickness of the test material should be at least 10 times bigger than the depth of the indent (Therefore, microhardness measurements are not very suitable for thin films).

3. Hardness measurement should be made at the center of the sample.

    Currently, four methods can be mentioned for hardness measurement, as described in the following sections.

2.1. Brinell's hardness test

Brinell's hardness test consists of indenting a sphere with a 10 mm diameter into the material (**Figure 1**).
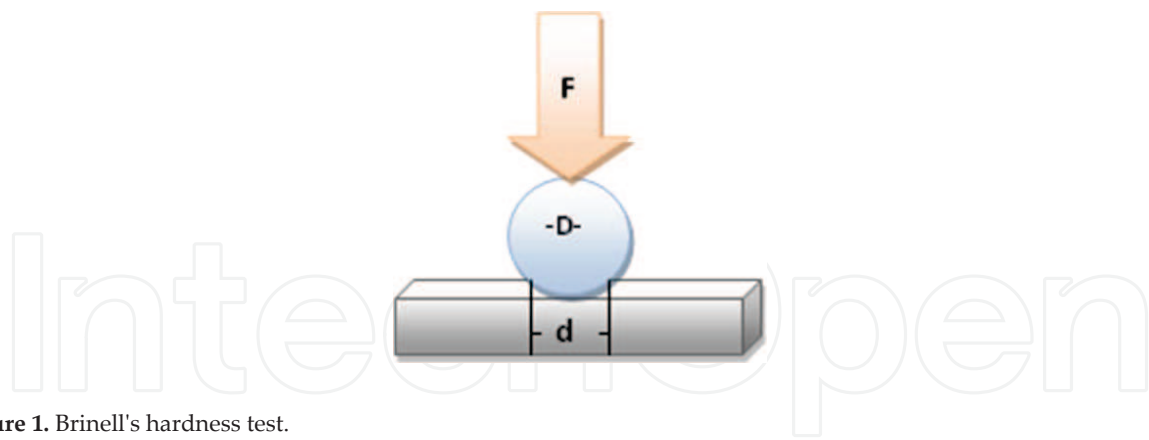
Figure 1. Brinell's hardness test.

Here, F is the applied force; D is the diameter of the indenter; and d is the diameter of the indentation.

$$H = \frac{2F}{\mu D(D - \sqrt{\square D^2 - d^2})} \tag{2}$$

The force to be applied is calculated according to $F = CD^2$, where C is the loading degree, varying for each material.

2.2. Rockwell's hardness test

It is similar to the Brinell hardness test; however, in this test, the holes between the surface of the sample and the indenter is equilibrated by applying a force of 10 kg. Then, an additional major load is applied and removed, while the preliminary load is still maintained (**Figure 2**).
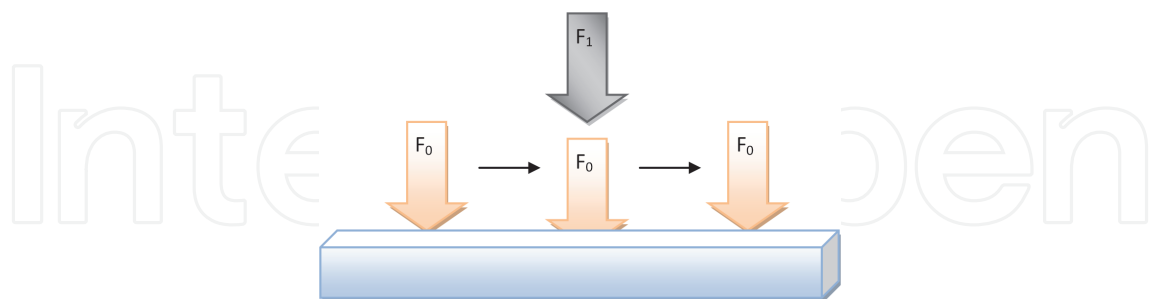

Figure 2. Rockwell's hardness test.

2.3. Vickers hardness test

In this test, force F is applied by a diamond indenter, in the form of a right pyramid with an angle of 136° between opposite faces (**Figure 3**). The calculations are made by measuring the diagonals of the indentation left on the surface of the material.
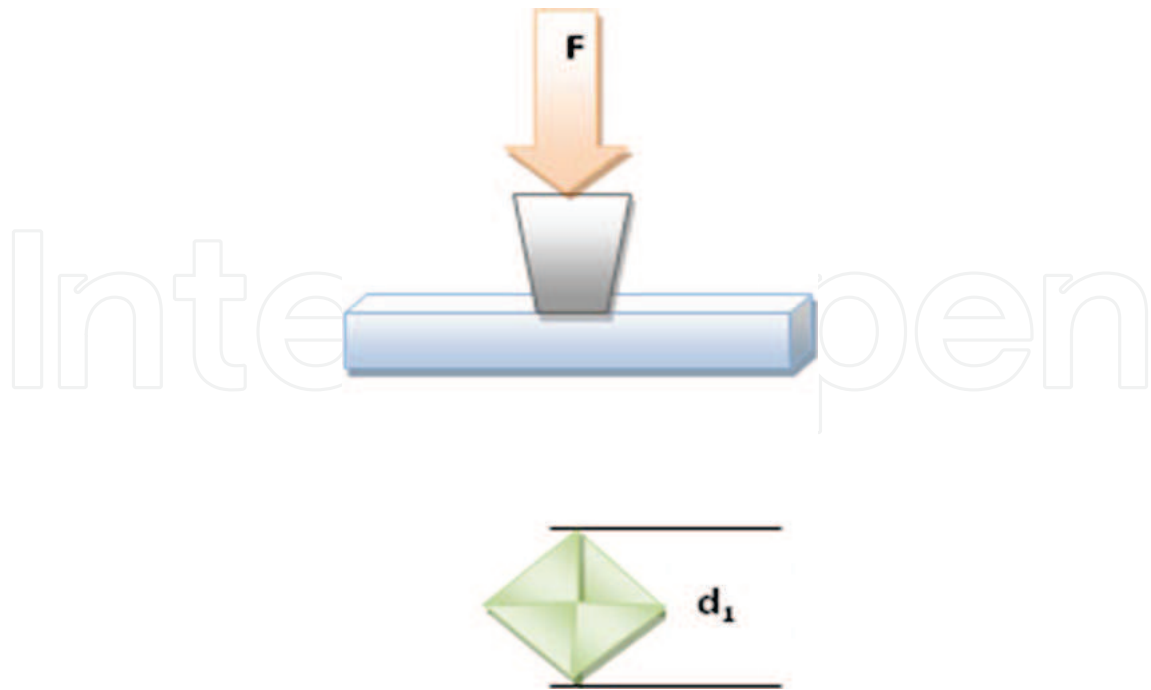
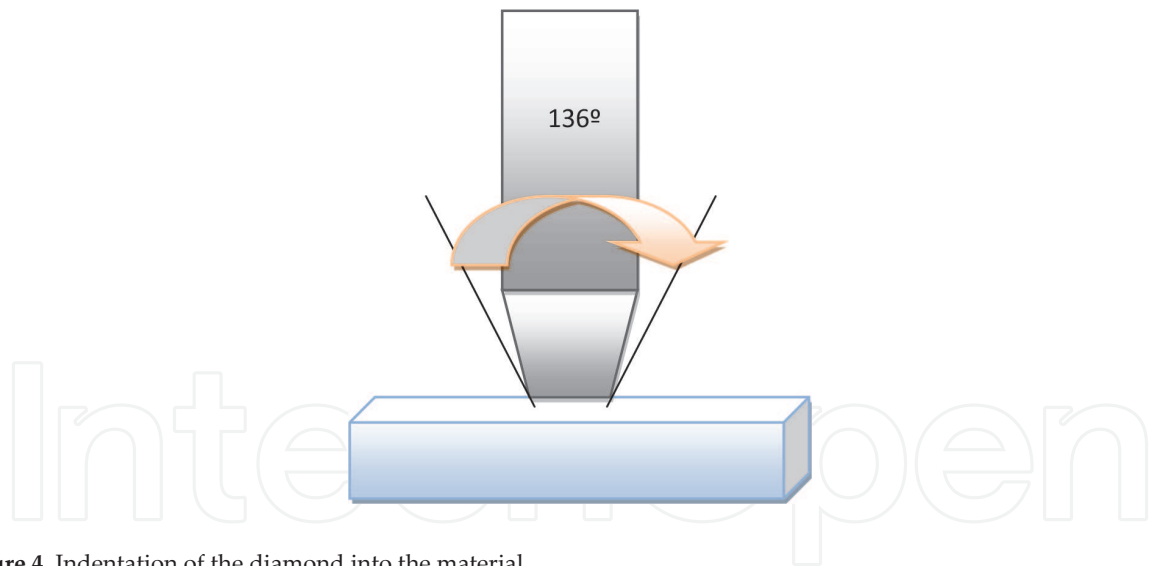**Figure 3.** Vickers test and the indentation left in the surface of the material.

Figure 4. Indentation of the diamond into the material.

Using d_1 displayed above, d can be calculated as $d = (d_1 + d_2)/2$. And the hardness can be calculated as

$$H = 1.8544 \frac{F}{d^2} \qquad (3)$$

Hardness of thin films can be measured with this method by using AFM devices.

2.4. Microhardness test

This method is suitable to measure the hardness of micron-sized samples. Similar to the Vickers test, a diamond indenter, in the form of a right pyramid, with an angle of 136° or 172°, is used (**Figure 4**). The hardness is

$$H = 14.2 \frac{F}{d^2} \qquad (4)$$

where d is the length of the indented edge [19].

3. Hardness of thin films

Researchers started to conduct researches on hard thin films and to produce them, because it became more and more important for the industry and the technology. Since the hardness measurement is as important as the thin films themselves, many researchers have focused on this issue as well. In various studies conducted until today, it has been indicated that the hardness of thin films can be calculated by indent images taken through nano-indentation device or AFM device (**Figure 5**).

In fact, these hardness measurements are also performed in a way similar to Vickers testing that we have explained above. The copper triple needle probe, which is used for tapping mode in AFM device, is indented to the surface by applying a particular force.
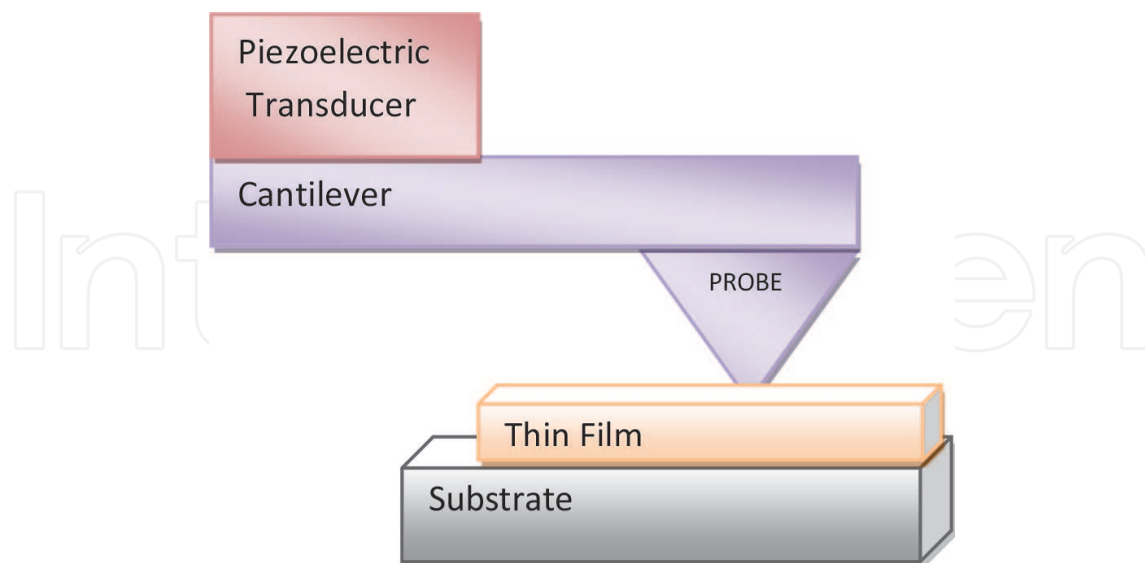

Figure 5. Hardness measurement by AFM.

The hardness of thin films can be determined using Vickers formulas (Eq. (3)), through the pyramid-shaped indentation seen on the surface image of the film. Here, measuring the

hardness of the substrate along with the thin film is very important. Otherwise, you may end up measuring the hardness of the substrate, instead of the hardness of the nanometer-thick film. Therefore, it may be useful to compare the hardness of the substrate with the hardness of the film. To prevent this, researchers often use the following formulas:

$$E_c = \frac{\pi S}{2\beta\sqrt{A}} \tag{5}$$

where E_c: composite modulus,

S: measured stiffness,

B: Berkovich-type constant: 1.034.

Mechanical problems that may occur due to the substrate can be eliminated by shifting to the Young module E_m value:

$$E_m = \frac{\left(1 - v_m^2\right)}{\dfrac{1}{E^*} - \dfrac{1 - v_i^2}{E_i}} \tag{6}$$

where v is the Poisson ratio and E is the Young module, representing the characteristics of the indenter and tested material with subindices "i" and "m". In this process, Young's module can be computed by replacing the indices with the tested film and the substrate, and the hardness of the film can be calculated.

Many researchers have worked on the hardness of thin films and described the parameters affecting the hardness of thin films. Some structural characteristics of thin films affect their optical, electrical, and mechanical properties. For example, it has been proven that electrical resistance of a thin film can be increased by decreasing its thickness; or film thickness and optic band gap are negatively proportional. However, researchers are a little late to investigate the impact of these parameters on the mechanical properties of thin films. On the other hand, there are researchers, such as Kariper, who have demonstrated that surface tension and surface energy of thin films are correlated with film thickness, average grain size, and number of crystalline per unit area.

According to literature, López et al. showed that film thickness and film hardness are negatively correlated [20]. Cavaleiro and Louro also observed that hardness is negatively correlated with grain size [21].

Due to the deformation of thin films, Ferro et al. could only show that there is a negative relation between thickness and hardness of the film: (i) the boundary between the film surface and the base prevents disordered structures and cracks, (ii) at the same time, nanocrystal nature of the films increases disordered structures. Therefore, it has been concluded that the decrease in the film thickness increases film tension of the area during the creation of the indent and causes

the indent to be deeper; and this process prevents plastic deformation. These explanations have convergent parts with our interpretation [22].

Venkatraman and Bravman suggested that—based on the Hall–Petch effect—as the grain size decreases, structural dislocation decreases, and this process increases the flow stress [23].
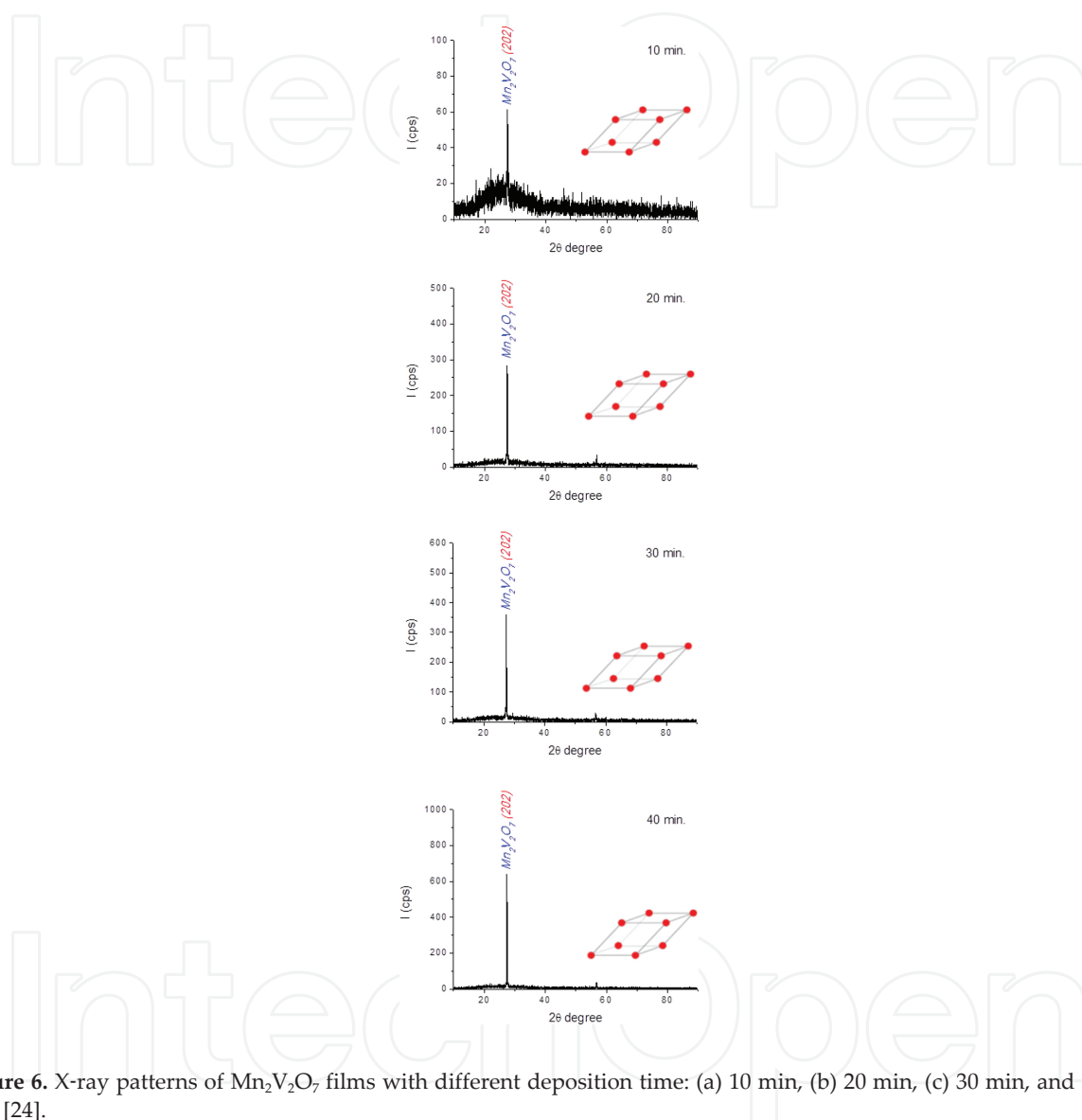

Figure 6. X-ray patterns of $Mn_2V_2O_7$ films with different deposition time: (a) 10 min, (b) 20 min, (c) 30 min, and (d) 40 min [24].

Kariper has published enormous and very clear works about the hardness of thin films and the influential factors [24]. He has demonstrated that parameters of thin films such as film thickness, grain size, surface roughness, etc. have affected the hardness of thin films. He had coated amorphous glass substrates with $Mn_2V_2O_7$ thin films. He showed that thickness and average grain size of the films varied with deposition time (The graphs and figures are taken from (and reorganized) the paper of Kariper published in the *International Journal of Minerals, Metallurgy and Materials*, in 2015). The lattice parameters of $Mn_2V_2O_7$ thin films were: $a = 0.6879$ nm, $b = 0.7973$ nm, $c = 1.0948$ nm, and the angles are $\alpha = 87.92°$, $\beta = 72°$, $\gamma = 83.29°$. The location

of atoms in the plane is displayed in **Figure 6** and **Tables 3** and **Tables 4**. The peaks of the films were indexed with ASTM values (47-0342).

The researcher had measured the film thickness through AFM tapping mode; he calculated average grain size and number of crystalline per unit area through Scherrer's formulas; the obtained results are presented in **Figures 7** and **Figures 8**.
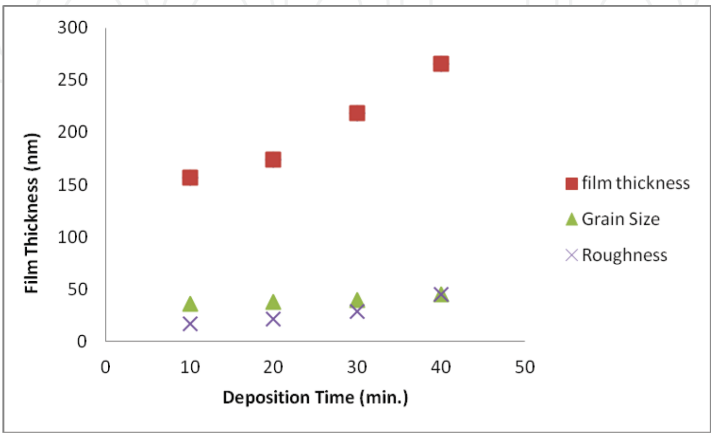

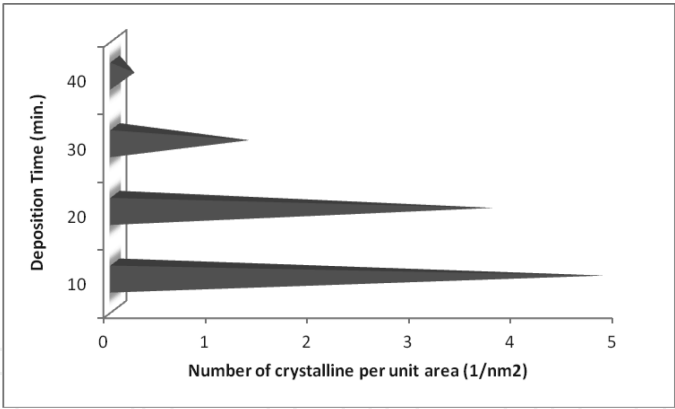**Figure 7.** Film thickness, average grain size and roughness versus deposition time [24].

Figure 8. Number of crystalline per unit area and dislocation density of the films versus deposition time [24].

Deposition time (min.)	Film thickness (nm)	Grain size (nm)	Roughness (nm)
10	157	36	17
20	174	38	21
30	218	40	29
40	266	45	45

Table 3. Some parameters of the thin film according to deposition time [24].

Deposition time (min)	Number of crystalline per unit area (1/nm²)
10	4,81
20	3,72
30	1,32
40	0,192

Table 4. Number of crystalline per unit area of the thin film according to deposition time [24].
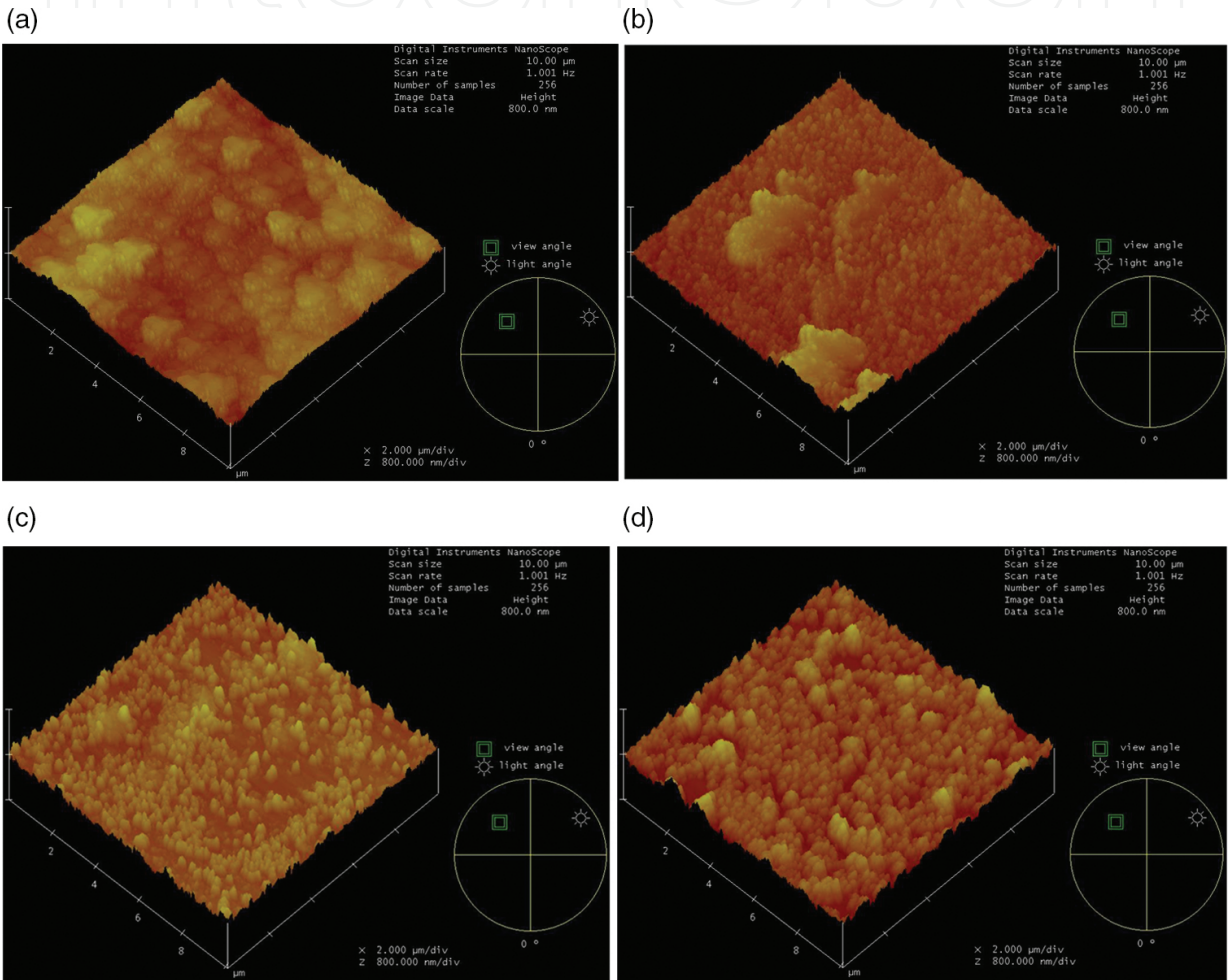

Figure 9. AFM images of the films with different deposition times (a) 10 min, (b) 20 min, (c) 30 min, and (d) 40 min [24].

He also stated that surface roughness affects hardness and published AFM surface images (**Figure 9**).

Afterward, he plotted the important parameters of thin films, such as film thickness, average grain size, surface roughness, and number of crystalline per unit area, in the form of a graph to find out if they are correlated with hardness (**Figure 10** and **Figure 11**, and **Table 5 (a)** and **(b)**).
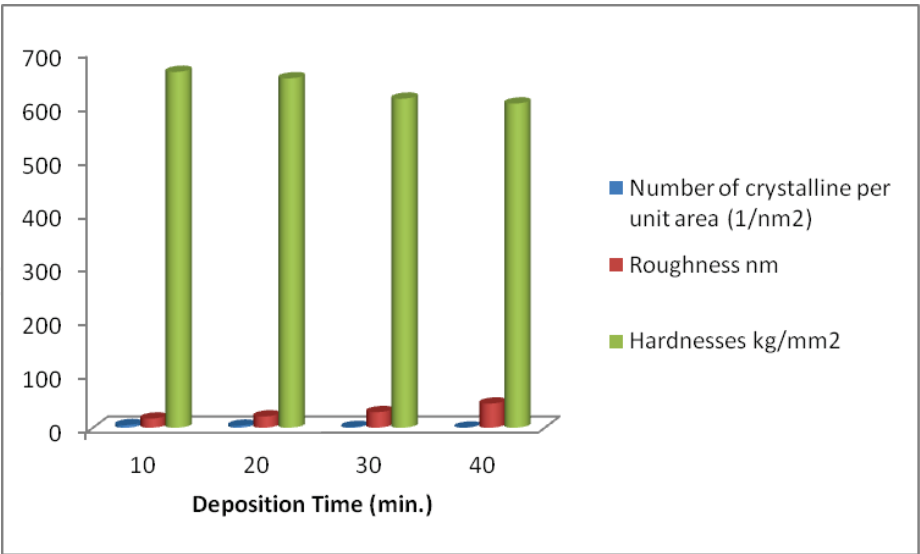
Figure 10. Relation between film thickness, average grain size, and hardness [24].

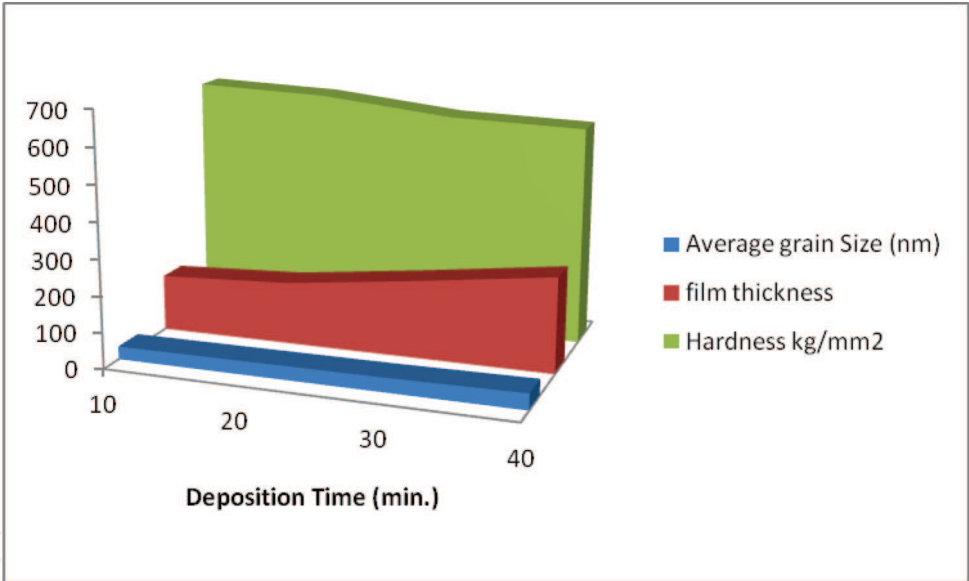**Figure 11.** Relation between number of crystalline per unit area, roughness, and hardness [24].

Deposition time (min.)	Number of crystalline per unit area (1/nm²)	Roughness (nm)	Hardness (kg/mm²)
10	4,81	17	663
20	3,72	21	651
30	1,32	29	613
40	0,192	45	604

Table 5. (a) Some parameters of the thin film according to hardness [24].

Deposition time (min.)	Film thickness (nm)	Average grain size (nm)	Hardness (kg/mm²)
10	157	36	663
20	174	38	651
30	218	40	613
40	266	45	604

Table 5. (b) Some parameters of the thin film according to hardness [24].

He achieved important findings with the outcomes of these graphs:

$H = -1.6343t + 1237.9$ (*H*: hardness, *t*: film thickness)

$H = -0.1222D + 117.09$ (*H*: hardness, *D*: average grain size)

$H = 0.0738N + 44.162$ (*H*: hardness, *N*: number of crystalline per unit area)

$H = -0.3931R + 276.76$ (*H*: hardness, *R*: roughness)

In spite of some small experimental errors, he suggested that these formulas can be combined, and can be expressed as follows:

$H = 0.01845N - 0.4086t - 0.03055D - 0.0983R + 418.978$.

4. Elasticity module of thin films

Hooke is a British scholar who lived in London between the years 1635 and 1703, and he provided great convenience to the concept of strength with the law he has discovered. Hooke has formed his law for isotropic material as follows:

For instance, let us take two different steel bars and apply two different forces (*F*1 and *F*2) to these bars in the dynamometer. Steel bars will be affected by two different tensions, $\sigma 1$ and $\sigma 2$, where $\sigma_1 = F_1/A_1$ and $\sigma_2 = F_2/A_2$. We can find out the elongation of the bars, $\Delta L1$ and $\Delta L2$. The ratio of these elongations to $L01$ and $L02$ gives us the stretching. It can be find "elasticity module," *E*, which is one of the main characteristics of a material, by writing the formulas of these elongations and divide them to the tensions that we have calculated above. Thomas Young first found this value; thus, it is called as "Young's modulus" in the literature.

Here, we cannot measure the elongation of flexible thin films, because we have coated them on a substrate. However, we can get insights about their elasticity module and elasticity indirectly and make comparisons ($\sigma = E\varepsilon$, where *E* is elasticity module and ε is unit elongation).

$1/E\alpha - 1.6343t + 1237.9$ (*t*: film thickness)

$1/E\alpha - 0.1222D + 117.09$ (*D*: average grain size)

$1/E\alpha \ 0.0738N + 44.162$ (*N*: number of crystalline per unit area)

$1/E\alpha -0.3931R + 276.76$ (*R*: roughness)

$1/E\alpha\ 0.01845N - 0.4086t - 0.03055D - 0.0983R + 418.978.$

Therefore, the elasticity of a flexible thin film is negatively correlated with average grain size, film thickness, and surface roughness, whereas it is positively correlated with the number of crystalline per unit area. On the other hand, since most of the flexible thin films are polymer or organic composites, it is quite difficult to obtain crystalline structure of these films. Therefore, it is more appropriate to make a relationship between film thickness and elasticity module.

5. Tensile and yield strength

Tensile and yield strength of thin films are among the features that cannot be measured directly. For the films whose hardness is measured, these features can be computed indirectly as below:

$$TS = \left(\frac{H}{2.9}\right)\left(\frac{n}{0.217}\right)^{n} \tag{7}$$

$$YS = \left(\frac{H}{3}\right)(0.1)^{n} \tag{8}$$

where TS: tensile strength;

YS: yield strength;

n: strain hardening exponent. Cahoon et al. [25] stated that they were able to calculate tensile and yield strengths with less than 2% error using these formulas. But, the computation of "n" value was posing problems. Thus, Pavlina and Van Tyne [26] have made some experiments and arranged these formulas as below:

$$YS = -90.7 + 2.876H \tag{9}$$

$$TS = -99.8 + 3.734H \tag{10}$$

Therefore, tensile and yield strengths can be directly computed from hardness measurements. Combining these with Kariper's formulas,

$YS = 3469.5 - 4.700t$ (t: film thickness)

$YS = 246.05 - 0.35144D$ (D: average grain size)

$YS = 36.309 + 0.21224N$ (N: number of crystalline per unit area)

YS = 705.26 – 1.130R (R: surface roughness)

Total YS = 0.21224N – 4.700t – 0.35144D – 1.130R + 1114.279.

Similarly, for TS,

TS = 4522.51 – 6.1024t (t: film thickness)

TS = 337.41 – 0.456D (D: average grain size)

TS = 65.100 + 0.2755N (N: number of crystalline per unit area)

TS = 933.62 – 1.467R (R: roughness)

Total TS = 0.2755N – 6.1024t – 0.456D – 1.467R + 1464.66.

As can be seen from these formulas, tensile and yield strengths are negatively correlated with film thickness, average grain size, and surface roughness (in other words, tensile and yield strengths of a thin film decrease as these parameters increase), whereas they are positively correlated with the number of crystalline per unit area (in other words tensile and yield strengths increase as the number of particles per unit area increases).

6. The relationship between temperature and hardness

The variation of the hardness of a material with temperature is another important issue. The most significant study of this area has been conducted by Volinsky et al. [27], on gold and copper films, in which they have attempted to explain the correlation of yield strength with temperature. Volinsky et al. have formulized their findings as below:

$$YS(T) = YS_0 + AT^2 + BT \tag{11}$$

where A and B are constants, whereas YS_0 is the yield strength of the material at 0°C. Combining this with Kariper's formulas,

$YS_0 + AT^2 + BT$ = 3469.5 – 4.700t (t: film thickness)

$YS_0 + AT^2 + BT$ = 246.05 – 0.35144D (D: average grain size)

$YS_0 + AT^2 + BT$ = 36.309 + 0.21224N (N: number of crystalline per unit area)

$YS_0 + AT^2 + BT$ = 705.26 – 1.130R (R: surface roughness)

$YS_0 + AT^2 + BT$ = 0.21224N – 4.700t – 0.35144D – 1.130R + 1114.279.

We can transform them into Kariper's formulas as described above. With the help of these formulas, yield strength of a film can be computed by measuring its hardness and the parameters such as film thickness, average grain size, number of particles per unit area, and surface

roughness. This may allow us to get rid of very expensive XRD operations. The only reason for not using these formulas is that we have not measured the hardness of Manganese–Vanadate films at 0°C yet.

7. Sensitivity coefficient

While measuring the hardness of a material, many parameters, such as force, diameter, depth, time, etc. affect the outcome of the measurement. These parameters also cause an uncertainty on measurement. The relationship of this uncertainty with the hardness is given as below:

$$c = \frac{\Delta H}{\Delta x} \qquad (12)$$

where Δx is the input parameter that has been changed. With sensitivity coefficient, we can get insight about the accuracy of hardness measurement. The opposite is also true. Sensitivity coefficients can be computed from hardness measurements. Most of the sensitivity coefficients can be obtained from the literature.

Table 6 shows the list prepared by Ludvik [28], showing the hardness the thin films produced until now.

Material	Microhardness (GPa)	Classification
c-BN	50–70	Superhard
CN	50–60	Superhard
nc-TiCN/SiCN	30–60	Hard–Superhard
nc-TiN/SiN	30–50	Hard–Superhard
TiN	20–30	Hard
SiCN	25–35	Hard
SiC	20–30	Hard
SiN	20	Soft–Hard
SiON	10–20	Soft
TiO_2	About 10	Soft
Ta_2O_5	About 10	Soft
Nb_3O_5	About 10	Soft
SiO_2	About 10	Soft

Table 6. Microhardness of PVD and CVD films with commonly used substrates [19].

8. Conclusion

Nowadays, the mechanical properties of thin films, which have a very important role in the development of nanotechnology, attract the attention of the researchers. The hardness of the material changes when its surface is covered with a hard or soft material, which especially makes the material more useful in many areas of the industry and technology. Examining the factors that affect the hardness of the material will make a very big contribution to the literature, as well as to the industry and technology.

In this section, we briefly discussed what hardness is, what the logic of hardness measurements is, and how they are performed. We mostly focused on the hardness of thin films. We extended the formulas of Kariper, who conducted the most comprehensive studies in this area, and we revealed that we can calculate elasticity module, Young's module, yield strength, and tensile strength of a thin film indirectly, or at least that we can make comparisons using these formulas. In addition, we combined the temperature change formulas of Volinsky et al. with Kariper's formula and saw that we can get insight about the characteristics of a thin film, such as film thickness, average grain size, and number of crystalline per unit area from different hardness measurements of the material performed at different temperatures.

Therefore, in this chapter, we presented the methods required to measure the hardness of a thin film, which are frequently emphasized by the researchers. We examined the factors that affect the hardness of thin films, which is one of the most important properties, and we showed how Kariper formulized them. Moreover, we explained the impact of these parameters on various properties of a thin film, such as elasticity module, Young's module, yield strength, and tensile strength, and we introduced significant novelties and hypothesis to the literature.

We hope that this study will be a milestone for other researchers who work in this area.

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