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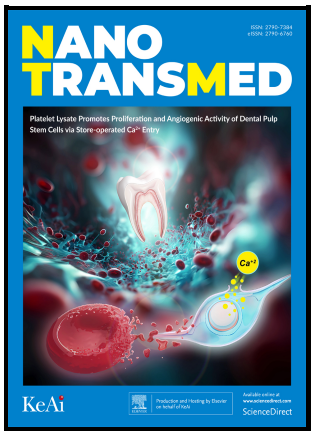

Nanoindentation: introduction and applications of a non-destructive analysis

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Nanoindentation: introduction and applications of a non-destructive analysis

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Abstract

Nanoindentation test is known as a powerful method for non-destructive characterization and analysis of mechanical properties of nanoscale materials. In this method, the indenting tip penetrates the surface of the sample by applying a force of several millinewtons to the extent of several nanometers, and the resulting force-displacement curve is used as the output of the test to calculate the mechanical characteristics of the sample, including hardness and elastic modulus, as well as to identify various mechanical phenomena such as Creep, strain hardening, surface cracking, phase transformations, creep and fracture toughness of the material are used. In this article, the Nanoindentation method is briefly introduced and its principles and basics are discussed. The application of this method is valid for analyzing the mechanical properties of a wide range of materials. The purpose of this article is to familiarize researchers and experts in engineering fields with the nanoindentation method as a non-destructive analysis and its effective use in their respective fields of application.

Keyword: Nanoindentation, hardness, Elastic modulus, Non-destructive analysis, nanoscale

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1. Introduction

Nanoindentation is essentially a testing process that involves indenting a material with unknown mechanical properties, such as elastic modulus or hardness, into another hard material with known properties[1-5]. On a macroscale, there are Brinell or Vickers tests, in which one material is indented into another. Nanoindentation is also an indentation test, but the length of penetration of the material is in the nanometer range (10−9 nm) [6-9]. Apart from the length scale, the distinguishing feature of nanoindentation testing is the indirect method of measuring the contact area between the indenter and the sample [10-13]. In nanoindentation testing, the size of the remaining indentation is very small, in the micrometer range, so it is measured indirectly by measuring the penetration depth of the indenter into the material surface [14-17]. Combining the penetration depth with the known shape of the indenter allows the contact area to be estimated [18-19]. The indentation test is a simple method for the mechanical characterization of materials with unknown properties through the contact of a material with known mechanical properties such as elastic modulus and hardness [20-24]. This test has its roots in the Mohs hardness measurement scale, in which a material that can leave a permanent scratch on another material is classified as harder. This hardness scale was first developed in 1822, where the highest hardness value for diamonds was considered to be a hardness number of 10[25-30]. Since then, various hardness testing methods have been developed, including the Brinell, Knop, Vickers and Rockwell tests, which are based on creating a depression on the surface of various materials using a special tool called an indenter [31-34]. Nanoindentation is an indentation test in which the depth of penetration or the height of the indentation created on the surface is on a nanometer scale, instead of the conventional millimeter or micrometer dimensions in conventional hardness testing methods [35-39]. The obvious characteristic of the nanoindentation test compared to conventional indentation methods is the indirect measurement of the area of the indentation created on the surface of the

sample. Unlike conventional indentation methods, the dimensions of the indentation area created during the nanoindentation test are much smaller than what can be measured directly [40-43]. Therefore, the area of this area is usually determined in the nanoindentation test by measuring the indentation depth on the surface of the sample [44-49]. with having the geometric characteristics of the indenter, it is possible to indirectly measure the dimensions of the contact area of the indenter with the sample surface. By using indentation methods, in addition to hardness, the elastic modulus, strain-hardening coefficient, fracture toughness and viscoelastic properties of materials can also be calculated [50-54]. The force-displacement curve of figure (1) is related to the case when an indenter with a spherical tip is placed on the smooth surface of the sample and sinks into the sample surface with a gradually increasing force. This curve includes a complete loading-unloading cycle called the subsidence cycle [55-59]. The force and depth of indentation are recorded for each increase in force as a measure of modulus and hardness as a function of depth of indentation. As seen in Figure (1-a), after reaching the maximum force, the force is gradually removed and the corresponding indentation depth is recorded [60-64]. The loading part of the indentation cycle consists of an initial elastic contact region and a subsequent plastic or yielding region that occurs at higher forces within the specimen [65-67]. During loading, if the phenomenon of yielding has occurred, the force-displacement curve will take a different path until it reaches zero applied load, and the indentation effect will remain on the surface of the sample [68-70]. The hardness and elastic modulus of the sample are calculated by measuring the maximum penetration depth at a certain applied load and the slope of the tangent line on the loading curve at the maximum load point (Pmax)[71-74]. In some cases, the elastic modulus of the sample can be measured using the loading area of the force-displacement curve in addition to the loading area. For a viscoelastic material, there is no linear relationship between force and penetration depth [75-

79]. This is due to the fact that for a given force, the resulting displacement depends on the speed of force application in addition to the amount of force applied. The immersion test for this category of materials is associated with creep phenomenon [80-84]. This phenomenon manifests itself in the form of a change in the penetration depth over time for a constant applied force (Figure 1-b). The analysis of the creep zone in the force-displacement curve of viscoelastic materials leads to obtaining quantitative data about the quasi-solid characteristics as well as the quasi-fluid components of the sample properties. In brittle materials, cracking on the surface of the sample is likely, especially when using pyramidal indenters (such as Berkovich's trihedral indenter and Vickers tetrahedral indenter). As seen in Figure (1-c), the crack length propagated from the corners of the indentation effect can be used to calculate the fracture toughness of the sample [85-89].

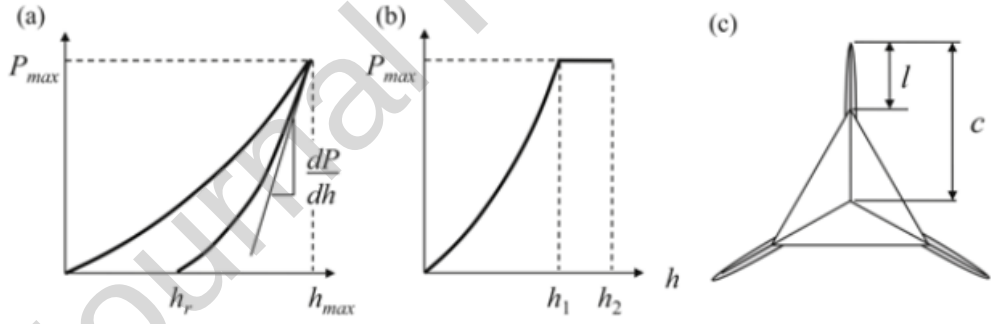

Figure 1. Force-displacement curves for (a) an elastic-plastic solid, (b) a viscoelastic solid for a spherical indenter, and (c) propagating cracks from the corners of the indenter residue on the surface of brittle material [21-22].

Nanoindentation is an effective and powerful tool to detect local elastic properties and hardness of micro- and nanostructures of materials. The basic idea of nanoindentation is simple: press a very sharp tip into the surface of a material and examine the mechanical behavior of the material based

on the tip's response[90-92]. Pioneering work dates back to Brinell's work, where he pressed a small ball into the surface of a material and measured the plastic properties of the ball bearing as the indenter. Now, further development of nanoindentation technology makes it possible to perform such mechanical tests directly on the micro- and nanostructures of materials. Since nanoindentation was introduced into the study of cement-based materials, a lot of valuable microstructural-mechanical information on cement-based materials has been published. During the last decade, nanoindentation has been used to study the microstructural mechanical properties (e.g., indentation modulus M, hardness H, creep compliance C) of cement pastes at different water-cement ratios [4,6–12,15,16,23 – 26,29–31,33,34,37,43,44,46–48,51,56,57,69,72,81,87–89,91,93-97-99,100-102-109-113]. Moreover, nanoindentation has been applied to the study of Nano silicates [20,37,38,53,111,146], silica fume [27,75], fly ash [18,27,75,94,95], slag [18,45,94,95] and metakaolin [18,28], as well as fly ash [58-60], slag [72] and metakaolin [59] and other cementitious materials [34, 76,77,79,103,108]. At the same time, the mechanical properties of the interfacial transition zone (ITZ) of cementitious composites containing aggregates and fiber-reinforced fibers have been studied by applying nanoindentation [1,41,51,52,54,55,61,80,84, 96,104–107]. The application of nanoindentation has provided insight into the microstructural mechanical properties of cement-based materials and has provided a wealth of valuable microstructural-mechanical information that is the basis for understanding and improving the macroscopic mechanical performance. Based on the microstructural information obtained by nanoindentation, many multi-scale analytical and numerical models have been proposed and successfully used to calculate the macroscopic mechanical performance of cement-based materials [2, 8, 26, 32]. Moreover, the microstructural mechanical information on cement-based materials obtained by nanoindentation provided one of the important pieces of evidence for molecular

simulation [19,63,67,73,78]. Currently, nanoindentation is becoming a common technique to characterize the mechanical properties of existing cement-based materials and may be used to quantitatively evaluate microstructural changes in the mechanical properties of newly developed cement-based materials. As the application of nanoindentation is gaining increasing interest among researchers in the field of construction and building materials, this article aims to provide an overview of this important technique applied to cement-based materials and to provide a comprehensive review of the mechanical properties of cement-based materials, measured by nanoindentation.

2. Indentation on the nanometer scale

The topic of nanofabrication started from the desire of the engineering community to measure the mechanical properties of hard thin layers and the expansion of various operations and surface processes in the 1980s [46-49]. The existing microhardness test equipment at that time was not able to apply sufficiently small forces to create a penetration depth of less than 10% of the thickness of the thin layer in order to prevent the influence of the presence of the substrate on the measured hardness. Even assuming the possibility of applying this force, it was not possible to determine the size of the resulting depression with sufficient accuracy. For example, the uncertainty in measuring the Vickers indentation effect with a diameter of 5 μm using an optical method is more than 20%. This uncertainty increases with decreasing indenter size, so that it reaches 100% for an indenter effect with a diameter of 1 μm[50-53]. Since the spatial dimensions of the indentation area cannot be measured using conventional methods, modern nanoindentation methods are usually used to measure the penetration depth of the indentation and to determine the area of the contact area, the geometric characteristics of the standard indentation are used. In this way, it is necessary for the indenter to make contact with the sample surface with a very small initial contact force, and its

initial penetration into the sample surface is considered as the penetration depth. Additional corrections are necessary considering the effect of factors such as irregularity in the shape of the indentation, deviation of the loading frame and accumulation of materials around the indentation (Figure 2). These effects lead to errors in the measurement of the penetration depth and subsequently in determining the hardness and elastic modulus of the sample. In addition, the scale of deformation in the nano-indentation test is comparable to the size of material defects such as dislocations and grain size, and therefore the use of continuous medium mechanical approximation in analyzing the results of this test will be less valid [54-58]. The results of the nanoindentation test provide the necessary data for the analysis of the elastic modulus, hardness, strain hardness, cracking, phase transformations, creep and fracture toughness of the material. The sample size and sinking effect are very small, so it is considered a non-destructive test. Also, sample preparation is an accessible process. Since the amount of deformation during this test is very small, it can be used to characterize the mechanical properties of thin layers and surface coatings. In many cases, the microstructural characteristics of coatings and thin films are significantly different from those of the bulk material. The reason for this difference is the presence of residual stresses, the preferred orientations of crystal plates and the morphology of the microstructure of the material. Therefore, this test is used in a wide range of different technologies, including chemical vapor deposition (CVD), physical vapor deposition (PVD), cathodic arc deposition, ion implantation, and functional materials. The nanoindentation analyzer is usually easy to use, controlled by a computer, and does not require a vacuum chamber or other expensive laboratory infrastructure. In the following, after examining the force-displacement curves, the applications of the nanoindentation test in different technologies will be discussed [58-62].

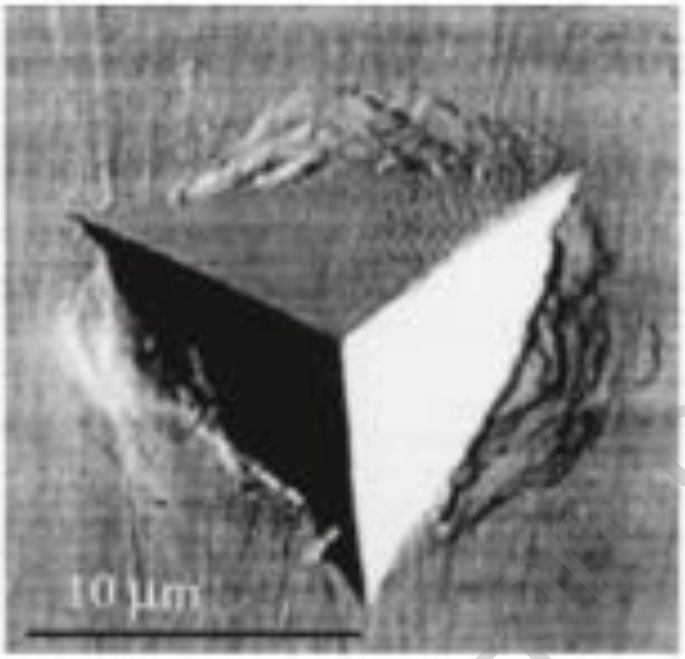

Figure 2. Atomic force image of the indentation effect created in a steel sample by the Berkovich indenter in the shape of a triangular pyramid. Accumulation of material around the sinking effect is visible [21-22].

3. Force-displacement curves

The main purpose of the nano-indentation test is to extract values related to the hardness and elastic modulus of the material by using the curve of changes in the indentation depth with increasing applied force. In this test, the penetration depth is drawn as a function of force during loading from zero to a maximum value and then loading from that maximum value until reaching zero force. In case of plastic deformation, after loading, an indentation effect will remain on the surface of the sample, whose exact dimensions cannot be measured using conventional optical methods [60-61]. Therefore, the hardness of the sample is calculated indirectly, using the geometric characteristics of the indenter and its penetration depth on the surface of the sample. Also, after passing the loading and unloading cycle, some of the deformation created on the surface of the sample is reduced due to the recovery of elastic strains. Therefore, by analyzing the initial part of the

material's elastic response during loading (the initial part of the loading curve), an estimate of the material's elastic modulus can be obtained [62].

The force-displacement curve for common types of indenters is similar to Figure 3. The elastic modulus (*E) of the sample is calculated using the area of the indented contact area with the sample surface (A) and the slope of the loading curve (dP/dh) at the maximum force point (P_{max}) as follows:

$$E^*=1/2\ \sqrt{\pi}\ /\sqrt{A}\ dP/dh$$

The hardness of the sample can also be calculated using the force applied per unit area of the residual indentation effect as follows:

$$H = \frac{P}{A}$$

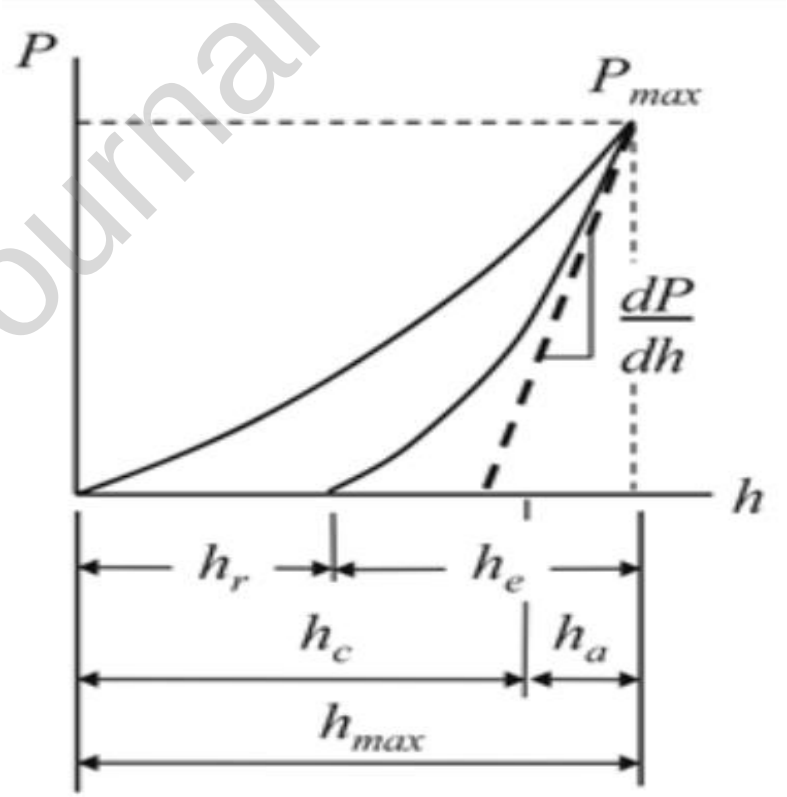

Figure 3. Loading-unloading curve for nano-indentation test with maximum applied force Pmax and penetration depth h_{max}. The depth of the indented contact area with the sample surface hc and the slope of the elastic area of the loading curve dP/dh are used to calculate the hardness and modulus of the sample. hr and he are respectively equal to the depth of the residual effect on the surface and the amount of displacement related to the elastic deformation recovered during loading [21-22].

In practice, the nanoindentation test is used to characterize a wide range of materials from soft polymer materials to hard pseudo-diamond thin films. The shape of the force-displacement curve obtained from this test is a rich source of information not only for calculating the hardness and elastic modulus, but also for identifying non-linear phenomena such as phase transformations, surface cracking and delamination or separation of the surface thin film. An overview of the observed force-displacement curves for the mentioned phenomena is given in Figure 4. Therefore, it should be noted that in some cases, the permanent deformation or the residual sinking effect on the surface of the sample is not only caused by the plastic silane of the material, and it may be related to the phenomenon of surface crack propagation or phase transformations on the surface of the sample [63].

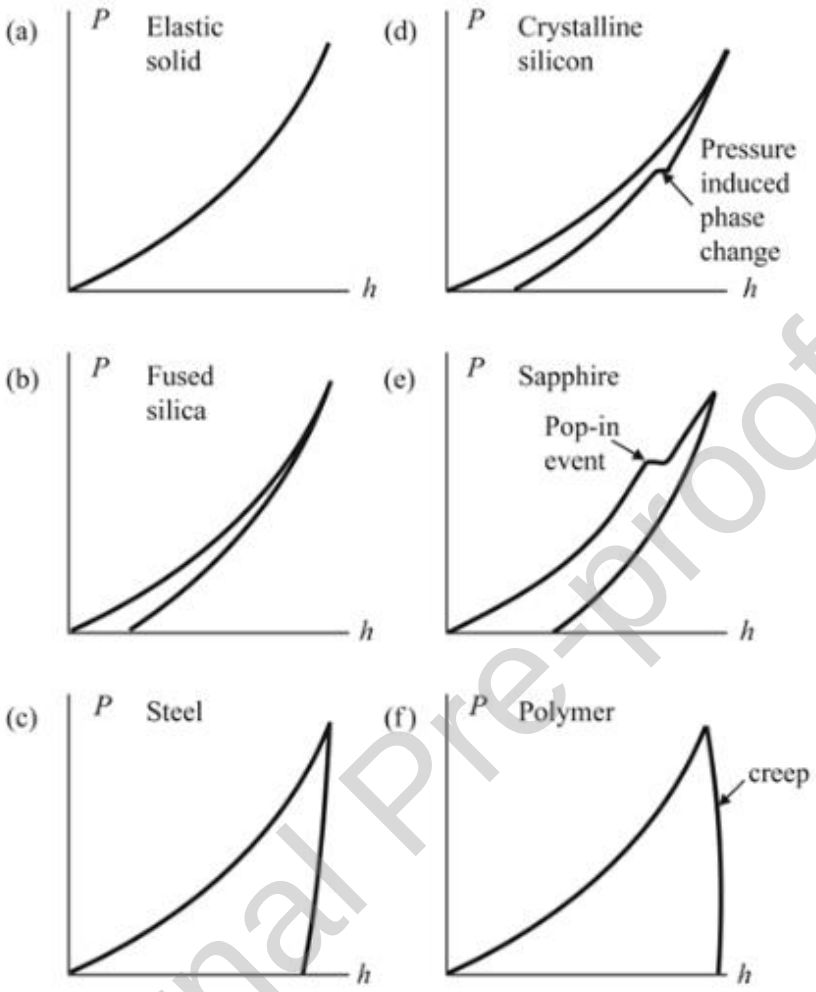

Figure 4. A schematic of the observed force-displacement curves for (a) elastic solid, (b) brittle solid, (c) soft solid, (d) crystalline solid, (e) brittle solid containing cracking during loading and (f) polymer under creep (The amount of applied force is of the order of a few million newtons and the amount of displacement is of the order of a few nanometers [21-22].

4. Applications of Nanoindentation

4.1. Fused silica

The force-displacement curve obtained from the nanoindentation test for the fused silica sample using the Berkovich indentation is shown in Figure 5. By applying a force of 50 mN, the

penetration depth of 624.9 nm was obtained. By using the numerical parameters obtained from the curve, especially the slope of the tangent line to the loading curve at the point of maximum load, the elastic modulus (*E) and the hardness of the sample are equal to 72.45 GPar and 9.54 GPa, these are in full agreement with the tolerances determined using other measurement methods. Some researchers use fused silica as a reference material to calibrate the nanoimmersion device and validate the data obtained from it. However, this is subject to error due to the occurrence of time-dependent creep behavior during its loading by the indenter. For example, in the nanoindentation test using a cubic pyramid indenter under a constant force of 10 millinewtons, after 10 seconds, the penetration depth increases by 5 nm. In order to minimize the effect of creep on the slope of the loading curve in the nanoindentation test of fused silica as a reference, it is necessary to keep the maximum load on the sample for at least 10-15 seconds before the loading stage[64-66].

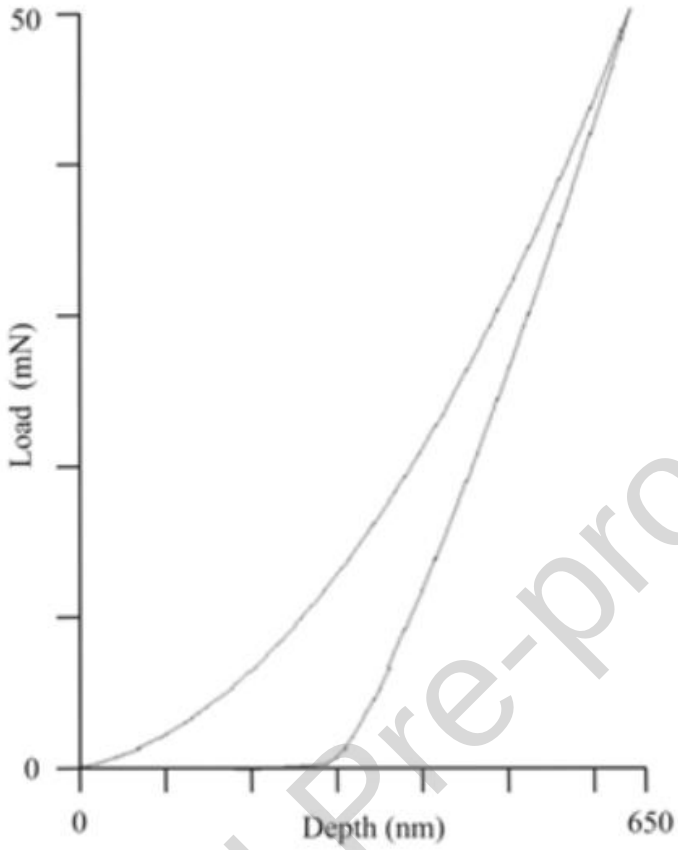

Figure 5. The force-displacement curve resulting from the nano-indentation test for the fused silica sample using a Berkovich indenter at a maximum force of 50 micronewtons and a maximum depth of 625 nm [21-22].

4.2. Super hard thin layers

Using the nano-indentation test for the characterization of super-hard materials is challenging. The penetration depth of the Berkovich sinker on the surface of this class of materials is of the order of its tip radius and is comparable to the roughness of the sample surface. On the other hand, the production of ultra-hard thin films is associated with a significant residual stress, which affects the hardness value measured by the nanoindentation method. To overcome these challenges, correction methods have been developed by considering all factors affecting the results of the

nanoindentation test. Another interesting challenge is how to measure the hardness of materials that have a higher hardness than diamond. The force-displacement curves for two thin film samples, the first TiN film and the second TiN/α-Si_3N_4 composite film, are given in Figure 6. In the nanocomposite sample, the amorphous silicon phase limits the growth of the TiN structure and consequently increases the hardness of the nanocomposite thin layer from 26 GPa to 60 GPa. As it can be seen, the depth of penetration in the surface of this class of ultra-hard materials is about 80 nm with the application of a maximum load of 4 million newtons [67-68].

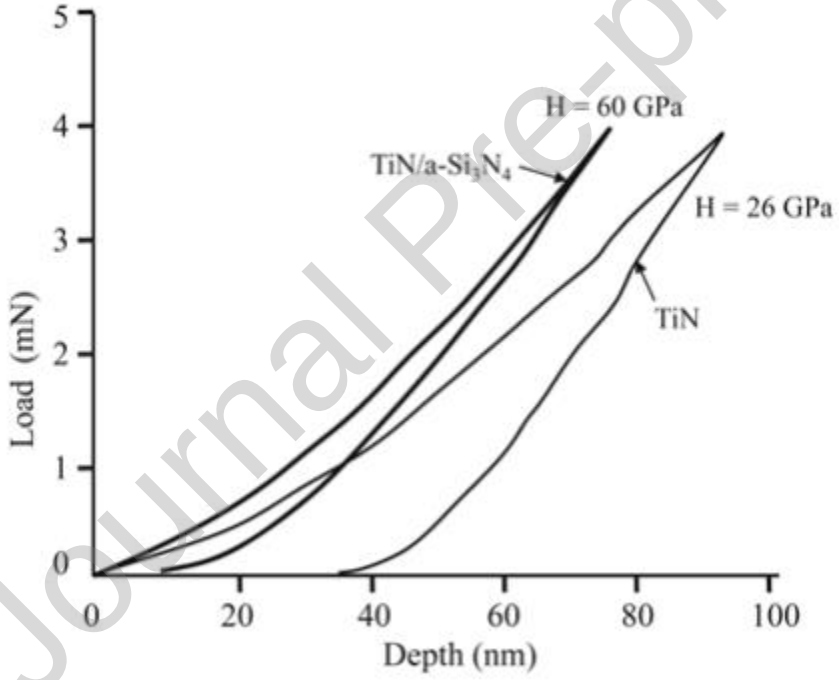

Figure 6. The force-displacement curve obtained from the nanoindentation test for two thin film samples, the first is a pure TiN film and the second is a TiN film doped with amorphous silicon phase, using a Berkovich indenter at a maximum force of 4 millinewtons and a maximum depth of 80 nm [21-22].

4.3. Creep phenomenon in polymer film

One of the applications of nanoindentation test is to estimate the viscoelastic properties of polymer materials and biological materials. The force-displacement curve obtained from the nanoindentation test for the polymer film sample using a spherical indenter is shown in Figure 7. In these materials, the occurrence of creep phenomenon in the sample causes a negative slope in the initial part of the loading curve, which is the main characteristic of polymer materials[69-70].

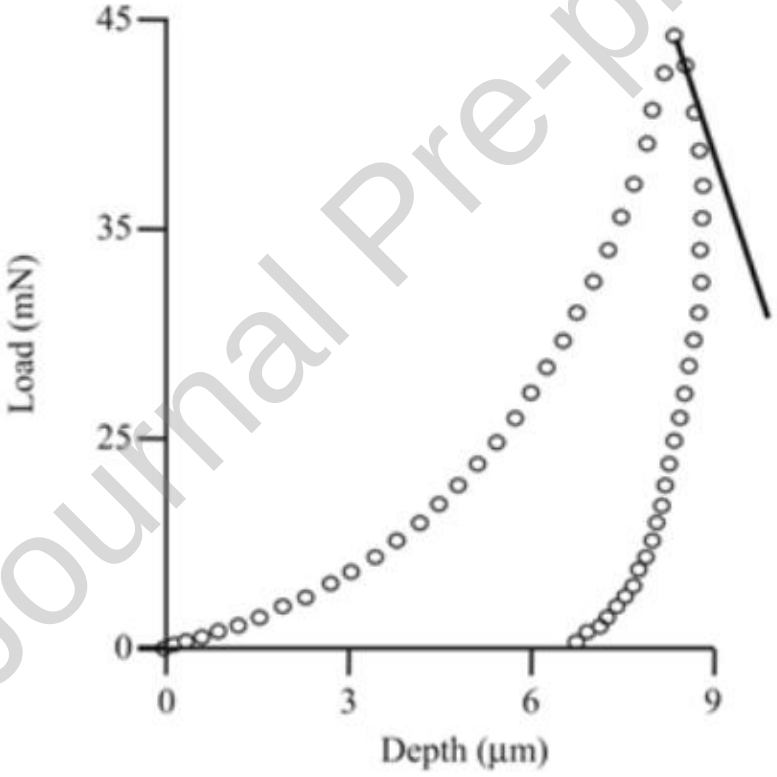

Figure 7. Common force-displacement curve obtained from nanoindentation test on polymer film using a spherical indenter. The occurrence of creep phenomenon during the test has caused a negative slope at the beginning of the loading curve [21-22].

4. Soft Biomaterials

For most people, a soft material is one that can be touched by hand or deformed by the naked eye without applying excessive force [25,115-118]. This simply reflects the property of being much more flexible than many technical materials. In fact, the terms "soft" and "rigid" do not say anything specific about hardness or plastic deformation, but only mean that soft biomaterials are not mineralized in their healthy state [26,119-121]. Soft biomaterials, such as globular proteins [27,122-124], cancer cells [28,125-127], arteries [29,128-130], cartilage [30,131-132], and the brain [31,133-135], change over multiple length scales, from the molecular to the cellular level, and from the tissue to the organ level. The complex hierarchical structure described above allows characterization within a wide range of physiologically relevant time scales and elastic modulus ranges (Figure 8). Many constitutive models, such as linear elastic, hyperelastic, viscoelastic, and pyroclastic models, are widely used for the mechanical characterization of soft biomaterials. In fact, these models can be categorized into different aspects of the constitutive response, especially those that distinguish them from typical engineering materials.

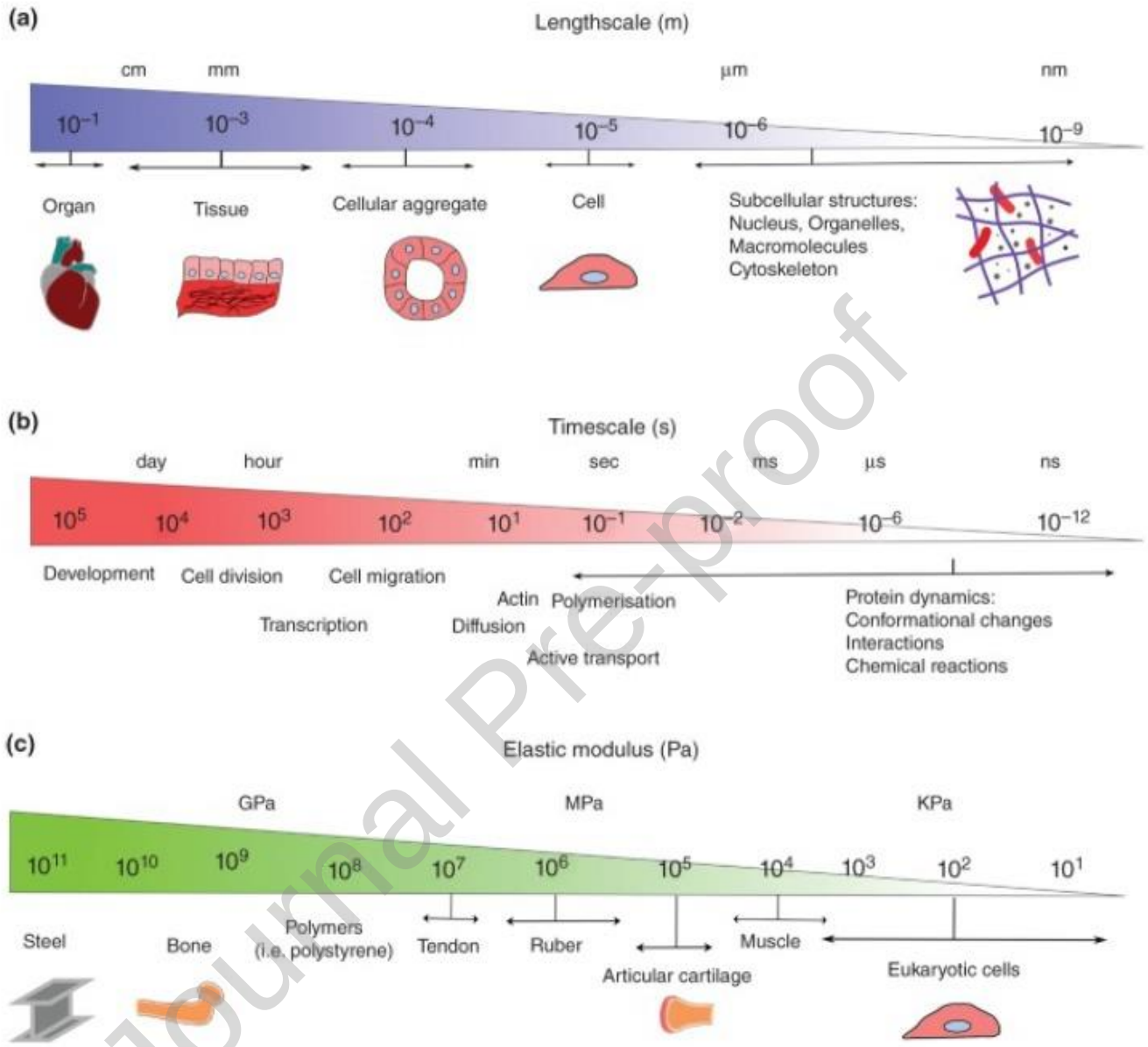

Figure8. Soft biomaterials at multiple scales. (a) Length scales from molecular to organ levels. (b) Time scales of different physiological processes. (c) Comparison of elastic moduli of different typical materials [145].

5. Views and opportunities

Despite significant progress based on the current research-oriented user base, it is generally agreed that a more detailed understanding of the mechanisms required for accurate constitutive data and

anatomical distribution is necessary, opening further opportunities in medical fields such as pathology and biochemistry [136-138]. For many soft biomaterials, mechanical characterization in an in vivo environment may be of greater significance in understanding the physical biology. Compared to ex vivo testing, perfusion pressure, tissue degradation, and boundary conditions may cause some changes in mechanical properties. Recent studies have shown that mechanical properties can change dramatically within just a few minutes after death. Therefore, in vivo testing may be a better (or only) strategy to gain insight into the mechanical behavior of some soft biomaterials. Validating tests under real operating conditions and even extreme environmental conditions is an important step for future research. In fact, most soft biomaterials operate under multiple complex loading conditions, so many potential details may be missed by some measurements under ideal experimental conditions [139-141]. In particular, some results obtained in one loading limit mode do not necessarily match the material response in another mode. In addition, biomaterials may exhibit novel behaviors under some extreme environmental conditions, such as high electric fields, high magnetic fields, strong radiation, and extreme low/high temperatures, which will improve our understanding of their applications in some fields, such as medical research and space exploration. Compared with most studies focused on mechanical characterization, nanoindentation can also be developed as a powerful tool in the field of mechanobiology. Recent discoveries have directly linked development, cell differentiation, physiology, and disease to mechanical responses at the cellular and tissue levels [142-145]. Insights into mechanical signaling from the macro- to nanoscales can provide a better understanding of the relationship between composition, structure, and properties of biomaterials. Elastography techniques (e.g., magnetic resonance and ultrasound elastography) can also serve as non-invasive tools to quantify the mechanical behavior of soft biomaterials in vivo. Although these

techniques are still in their infancy and have not been fully validated or validated, they hold promise for a wide range of potential applications in various areas of pathology when combined with nanoindentation techniques.

6. Conclusion

- The nano-indentation test is used for non-destructive characterization and analysis of the mechanical properties of materials at the nano scale. In this method, an indenter with very small dimensions is used under a force of several millinewtons or less to prevent the influence of the substrate on the hardness value measured for the nanometer coating. The resulting force-displacement curve is used to calculate the hardness and elastic modulus as well as to identify the phenomena of creep, strain hardening, surface cracking, phase transformations, creep and fracture toughness of the material. In addition, this method can be used to measure the hardness of nanometer and submicron phases in composite and nanocomposite materials. In this article, a brief introduction of the nanoindentation method was given first, and then its application to analyze the mechanical properties of a wide range of materials was discussed.
- Nanoindentation is a powerful quantitative method for determining mechanical properties from very small volumes. Carefully conducted tests that meet the assumptions of O&P elastic contact analysis can easily achieve an accuracy of a few percent for indentations as small as 10 nm. Practitioners must always be aware of the effects of deviations from these assumptions on nanoindentation results. Accurate calibration of load, displacement, and machine compliance is important, as is practical accounting for tip geometry and configuration that minimizes the effects of thermal drift and plasticity. If one wishes to measure surface layers and other small volumes while avoiding the effects of surface

conditions and nearby free surfaces and interfaces, the range of effective indentation sizes must be determined empirically. Pile-up, size effects and anisotropy can introduce systematic variations that need to be taken into account. Nanoindentation is an area of intensive research and development, and data analysis methods are constantly being improved. Future developments are expected to include further improvements in DCA techniques, including the routine incorporation of computer simulations (e.g. FEM) and quantitative imaging methods to aid in the interpretation of DSI data, as well as the incorporation of acoustic methods.

- Nanoindentation is often used to characterize the mechanical properties of materials. In this technique, the applied load and penetration depth are measured on a very small scale as an indenter is forced into the test material. Penetrant testing requires minimal material preparation and can be performed multiple times on a single sample. It is particularly well suited to materials in bulk form as well as thin films, coatings, and modified surfaces. Based on classical contact mechanics, the theory and practice of nanoindentation testing has been developed to measure a wide range of material properties. This chapter provides a comprehensive overview of the fundamentals of nanoindentation. It begins with background information on penetration theory, with emphasis on the relevant Hertzian contact analysis and Sneddon solutions. It then presents common indenter types, followed by a discussion of the two most commonly measured properties: hardness and elastic modulus. It discusses guidelines and best practices for determining contact stiffness and contact area, as well as corrections for thermal drift and machine compliance. The presentation also includes representative penetration methods for characterizing residual stresses, time-dependent deformation of metals and polymers, fracture toughness of brittle

materials, and adhesion of coatings to substrates. Computer models have proven to provide valuable information on the internal deformation fields that can be correlated with the indentation response. Unique impression features and uncertainties due to material heterogeneity, as well as remaining challenges and future directions are also discussed.

Data availability

All data generated or analyzed during this study are included in this published article

Author statement

We know of no conflict of interest associated with this publication, and there has been no financial support for this work that could have influenced its outcome as corresponding author, I confirm that the manuscript has been read and approved for submission by all the named authors.

Declaration of Competing Interest

We wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome.

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Interest.

Authors Contributions

Zaid H. Mahmoud, H. N. K. AL-Salman: Writing – review & editing.

Ehsan kianfar: Writing – review & editing, Writing – original draft, Supervision, Investigation

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