MATERIAL CHARACTERIZATION OF FORAMINIFER A VIA NANOINDENTATION

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Abstract: The paper presents results of the materials characterization of planktonic foraminifera via nanoindentation. Four types of planktonic foraminifera are investigated. The obtained results are discussed.

Keyword: planktonic foraminifera, nanoindentation,

1. Introduction

Instrumented indentation test (IIT) is used in last years to characterize the mechanical properties of different type of materials, thin films and multilayer materials. The method is attractive because testing is done on small samples in a local area. There are many cases where the conventional experiments cannot be carried out due to e.g. the geometry, the size and the build-up of the specimen. Such limitations occur for example for small volumes or if a thin layer on a substrate is to be investigated. Nanoindentation (NI), where an indenter is pressed normal onto the surface of the specimen. Such limitations occur for example for small volumes or if a thin layer on a substrate is to be investigated. Nanoindentation (NI), where an indenter is pressed normal onto the surface of the specimen, had been proven within the last decade to be a promising technique to investigate the mechanical properties of small volumes.

The relatively non-destructive character of these tests makes it possible to investigate the material properties in a small volume instead of a separately manufactured specimen. The testing process itself is non-destructive, leaving tested samples available for other analyses. The global variables load and displacement are monitored during the experiment by instrumented measuring devices, leading us to the notion of IIT. A disadvantage of indentation tests however is the complicated inhomogeneous deformation showing up in the specimen. Therefore it is not possible to conclude the local variables stresses and strains form the global measured variables load and displacement. As a result of instrumented indentation test only indentation modulus and indentation hardness can be calculated.

In present paper IIT is used for material characterization of Foraminifera.

2. Theoretical background of Instrumented indentation test.

The theoretical background of the indentation testing is given in [5]. Here we include the explanation in [5] for completeness. Instrumented indentation testing is similar to a hardness test in that a rigid probe is pushed into the surface of a material. Traditionally hardness tests consist of the application of a single static force with a specific tip shape. The output of these hardness testers is a single indentation value that is a measure of relative penetration depth of the indentation tip into the sample. The calculation of this single valued measurement requires the area of the residual hardness impression to be measured either optically or by microscopy.

IIT is an improvement to traditional methods because there is no need to measure the area of the residual impression. With instrumented indentation testing the area of contact is calculated from the load-displacement history which is measured simultaneously and continuously over a complete loading cycle. One of the main benefits of IIT is the calculation of material properties without the need to measure the contact area directly once the indenter is withdrawn from the material.

In tensile testing Young’s modulus E is calculated as the slope of the stress-strain curve when the deformation is elastic. Young’s modulus is an intrinsic property of a material; the only way to change E is to change the atomic structure of the material. Hardness is directly proportional to the yield stress and is generally smaller of it by a factor of about 3. Hardness is not an intrinsic property due to the fact that hardness can be altered by cold-working, heat treating and other means. Elastic modulus and hardness are important to design engineers because they deliver information on how a material will behave under various stresses and strains. IIT has also been used to calculate complex modulus of polymers, and fracture toughness in ceramics and glasses [1].

The typical IIT test has following steps in force-time history (Figure 1):

0. Indenter approaches the test surface until the contact is realized.
1. (Loading step) the indenter is driven into the surface until the maximum force or penetration depth is reached.
2. (Hold time step) the force applied to the sample is held constant for a period of time determined by the user.
3. (Unloading step) the indenter is withdrawn from the sample (up to 90% of the peak force).
4. The force applied to the material is held constant for a user specified period.
5. The indenter is withdrawn from the sample.
Hardness has conventionally been defined as the resistance of a material to permanent penetration by another harder material with measurement being made after the test force has been removed, such that elastic deformation is ignored. Instrumented indentation hardness provides the ability to measure the indenter penetration $h$ under the applied force $F$ throughout the testing cycle and is therefore capable of measuring both the plastic and elastic deformation of the material under test. Figure 2 is a typical load displacement hysteresis curve obtained from an elastic/plastic material and Figure 3 shows the schematic representation of the indent under load and in the unloaded condition.

Oliver and Pharr proposed calculation technique for determination of indentation hardness and indentation modulus of a sample based on the measured force-displacement curve. The first parameter in the calculation stream is contact stiffness $S$. This contact stiffness is determined as the slope to the relationship between force and displacement when the indenter just begins to withdraw from the material. At this point the material response is considered entirely elastic. For the unloading segment, the dependence of $P$ on $h$ is given by following approximation

$$P(h) = \alpha (h - h_f)^m.$$  \hfill (1)

where $h_f$ is the depth of the residual impression after the probe has fully withdrawn from the sample Figure 3. The parameters $m$, $h_f$ and $\alpha$ are best fit constants. Once $m$, $h_f$ and $\alpha$ have been calculated and the equation for the approximation force is differentiated with respect to displacement $h$ and evaluated at the maximum displacement $h_{max}$ yielding

$$S = \frac{dP}{dh} \Big|_{h_{max}} = \alpha m (h_{max} - h_f)^{-1}.$$  \hfill (2)

The contact depth $h_c$ can be calculated as $h_c = h - h_s$. The value of $h_c$ is dependent on the exact shape of the indenter and material response to the indentation; ‘sink-in’ and ‘pile-up’ around the indenter also affect the value. The deflection of the sample surface $h_s$ is calculates as $h_s = \lambda \frac{P}{S}$. For pyramidal, conical and spherical indenters $\lambda = 0.75$. Than the contact depth is calculated from $h_c = h - 0.75 \frac{P}{S}$. This expression for the contact depth allows determining of the contact area from the load-displacement history and there is no need from optically measure or image the residual impression. This differentiates nanoindentation from traditional hardness tests.

The reduced modulus is determined by $E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_c}}$.

The indentation modulus $E_{IT}$, is calculated as

$$E_{IT} = \left(1 - \nu_{\text{sample}}^2\right) \left[\frac{2}{S} \sqrt{\frac{A}{\pi}} - \frac{1 - \nu_{\text{indenter}}^2}{E_{\text{indenter}}} \right]^{-1}.$$  \hfill (3)

where $E_{\text{indenter}}$ and $\nu_{\text{indenter}}$ material properties of the indenter. The calculation of the indentation modulus requires knowledge of the sample Poisson's ratio. Indentation hardness $H_{IT}$ is defined as the ratio of the maximum load to the contact area at that load.
Many applications and research papers in last years shows that the nanoindentation is a valuable tool for evaluating the mechanical properties of a wide range of materials - metals, polymers, biomaterials, nanostructured materials etc. [3].

3. Material characterization of planktonic foraminifera via IIT

Shells of planktonic foraminifera date back hundreds of millions of years.

Planktonic foraminifera are aquatic creatures which are often less than a millimeter in size and resemble grains of sand to the naked eye. In generally between 0.10 mm and 1.00 mm in diameter and averages about 0.33 mm, equal to the fine sand grade of sediments.

Four types of foraminifera are investigated via IIT. The samples are delivered from prof. T. Schanz, Bochum University.

Globigerina bulloides (GB)
Uvigerina peregrina (UP)
Globorotalia tuncatulinoides (GT)
Orbulina universa (OU)

3.1. Sample preparation

Using two component epoxy clay foraminifera are fixed on punch and prepared for IIT. A single sample of foraminifera from each type is used for the indentation tests.

Nanoindentation test involve indenting a specimen by different indendentation depth for each sample. IIT. Nanoindentation experiment [4] was realized using Nano Indenter G200 (Agilent Technologies). This indenter is equipped with a Berkovich three-sided diamond pyramid with centerline-to-face angle 65.3º and 20nm radius at the tip of the indenter. The minimum load possible to be applied is 10 mN, and the maximum load is 500 mN. Displacement recording resolution is 0.01 nm and the load recording resolution is 50 nN. The device is equipped with an optical microscope with 2 objectives of magnification respectively 250x and 1000x.

A. Determination of $E_{IT}$ and $H_{IT}$ for GB

\[ E[GPa] = 6.787/6.759 \]
\[ H = 0.27/0.079 \]
\[ h[nm] = 3000/3500 \]

B. Determination of $E_{IT}$ and $H_{IT}$ for UP

\[ E[GPa] = 19.726/11.608 \]
\[ H = 0.344/1.194 \]
\[ h[nm] = 3000/3500 \]

443
C. Determination of $E_{IT}$ and $H_{IT}$ for GT

\[ E_{[GPa]} = 32.887/33.831/28.931 \]

\[ H = 0.18/1.412/0.9 \]

\[ h_{[nm]} = 2000/1500/1500 \]

\[ E_{[GPa]} = 65.4 \]

\[ H = 5.4 \]

\[ h_{[nm]} = 200 \]

D. Determination of $E_{IT}$ and $H_{IT}$ for OU

\[ E_{[GPa]} = 19.726/11.608 \]

\[ H = 0.344/1.194 \]

\[ h_{[nm]} = 3000/3500 \]

\[ E_{[GPa]} = 99.772 \]

\[ H = 5.621 \]

\[ h_{[nm]} = 200 \]

4. Results and discussion

Indentation experiments can provide information in a local area about the mechanical properties of foraminifera, namely indentation hardness and indentation modulus. However, it can be concluded that the number of nanoindentation tests must be large due to the specific object and the measurement scatter.

Acknowledgments

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5. References


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