Materials Characterization

Modern Methods and Applications

edited by Narayanaswami Ranganathan

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Materials Characterization: Modern Methods and Applications

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Preface

Modern materials are put to use in harsh environments and high temperatures, and the determination of material properties has become a key issue. The material mechanical properties are governed by the microstructure and other physical properties. Depending on the kind of application, one is interested in local mechanical properties, typically at the surface or in the layers immediately underneath, while other applications require the knowledge of the global or bulk behavior.

This book, which is a result of a coordinated effort by researchers from five different countries, addresses the methods of determining local and global mechanical properties of a variety of materials: metals, plastics, rubber, and ceramics.

Chapter 1 is a comprehensive treatment of the nanoindentation technique, treating the basic principles, contact mechanics, and various examples of surface properties determination. While conventional properties like the hardness and the reduced modulus are discussed, more delicate tests such as nanowear, nanoimpact, and micropillar compression tests are also treated in detail, with different relevant applications.

Chapter 2 treats surface property changes in selected polymers and rubbers, measured by nanoindentation. Different aspects such as surface gradient of vulcanizate crosslinking, surface migration of low molecular weight components of rubber, and surface segregation in polymer blends are discussed.

Chapter 3 treats the static and wear resistance of dental composites with emphasis on new composites with enhanced behavior like improving the mechanical properties of fillings, resulting in longer lifetime, and by providing bactericidal function to composites.

Chapter 4 treats the global and local properties of a lead free solder. It is shown that the local properties, such as hardness, modulus, and creep are affected by the presence of a specific microstructure in the solder ball and the existence of local precipitates. The local properties can be quite different from bulk properties. The cyclic fatigue behavior of the bulk alloy is studied in detail.

Chapter 5 discusses the change in surface properties in the plastic zone accompanying a growing fatigue crack. It is shown that the plasticity index is very sensitive to local plasticity and the local changes indicate that ductility exhaustion is a precursor to fatigue crack propagation.

Chapter 6 discusses in detail the fatigue behavior of a polychloroprene rubber. Fatigue behavior is studied at different load ratios and strong indication of strain induced crystallization is observed, leading to life enhancement at high load ratios. A detailed fractographic examination is presented to illustrate the mechanisms.

Chapter 7 is a comprehensive review of the methods of determining fatigue crack growth resistance of metallic materials, including instrumented testing methods, spectrum fatigue testing, and standards for testing and analysis.

Chapter 8 treats friction and wear tests and gives a global and structured vision of the tribological tests existing from the classifications of tests according to the needs of the industry.

The final chapter presents a means of determining elastic properties by the nondestructive resonant vibrating method, developed for bulk and coated materials characterization.

It has been a pleasure realizing this collection, which will be very useful to research scholars, graduate students, and teachers.

The editor is grateful to all the 22 contributors to this book and to Pan Stanford Publishing for their help with the different processes of proofreading and editing.

N. Ranganathan
Tours, France
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Contents

Chapter 1

Advanced Nanomechanical Test Techniques

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1.1 Introduction

For many years the hardness of bulk materials and thick coatings has been determined by optical analysis of indentation marks. The development of thin coatings deposited by techniques such as physical vapor deposition (PVD) and chemical vapor deposition (CVD), to improve wear resistance, led to the requirement to measure their properties at a smaller scale. Initially such coatings were typically relatively thick (e.g., ~10 μm) and microhardness measurements could be performed to determine their hardness. However, as the thickness of the films reduced the reliable determination of their hardness by conventional optical means became impossible. Depth-sensing indentation (DSI) instruments have been developed to

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address this need and have become increasingly popular. The test technique is also called instrumented indentation testing (IIT) or nanoindentation and has progressed sufficiently for standardization to be required with the first international standard for DSI being released in 2002 and is currently in revision [1]. Provided instruments are well calibrated the data from nanoindentation tests are routinely analyzed by well-established contact mechanics treatments to provide the reduced elastic modulus and the hardness (or more strictly the mean contact pressure) of the test sample [2–3]. Conversion between nanoindentation hardness and Vickers hardness requires a little care. In addition to knowledge of the indenter geometry the actual contact areas used in the two definitions of hardness are slightly different necessitating the need for a geometric correction factor.

Over the last 25 years commercial nanoindentation test instruments (also called nanoindenters) have improved their resolution, their calibrations, and their ability to very precisely position where the indentations are made to obtain highly localized and accurate mechanical property information. Additionally, they have expanded the range of test techniques beyond simple nanoindentation, with several including some capability for nanotribological measurements (e.g., nanoscratch and nanowear testing), which has consequently greatly expanded their range of applications. There is a range of commercial nanoindenter designs, including electrostatic or capacitive actuation, and vertical or horizontal loading configurations. The design of one popular commercial test instrument, the NanoTest system from Micro Materials, combines electrostatic actuation with horizontal loading and an open test platform that has enabled its further development into a true multifunctional nanomechanical/nanotribological test instrument where tests can be performed with a range of contact geometries (Fig. 1.1) and environmental conditions (Fig. 1.2 illustrates the temperature and strain rate test envelope) [4]. The various tests provide complementary information and the data obtained can often more usefully map onto the actual conditions that the materials experience in use. It is becoming possible to move beyond basic characterization to the development of increasingly accurate prediction of the surface behavior.

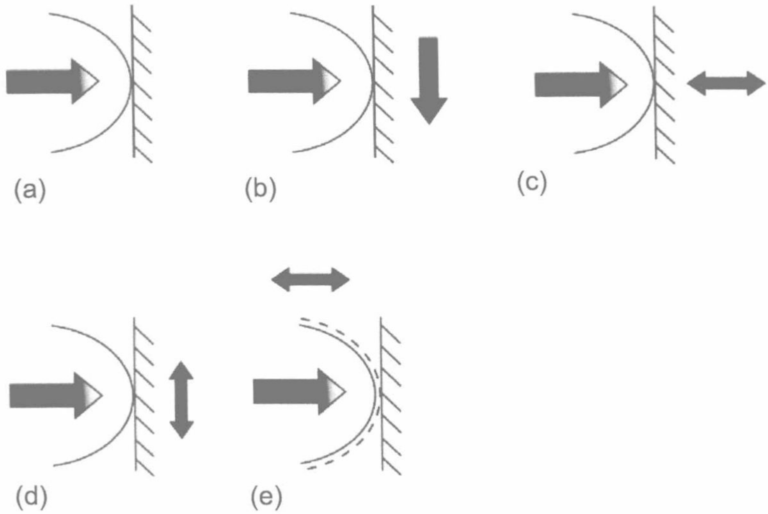

Figure 1.1 Range of nanomechanical testing capability in a commercial instrument (NanoTest system) illustrated for a spherical test probe. (a) Indentation contact, (b) scratch, (c) contact fatigue or impact by sample oscillation, (d) nanofretting or reciprocating wear, and (e) nanoimpact by probe impulse.

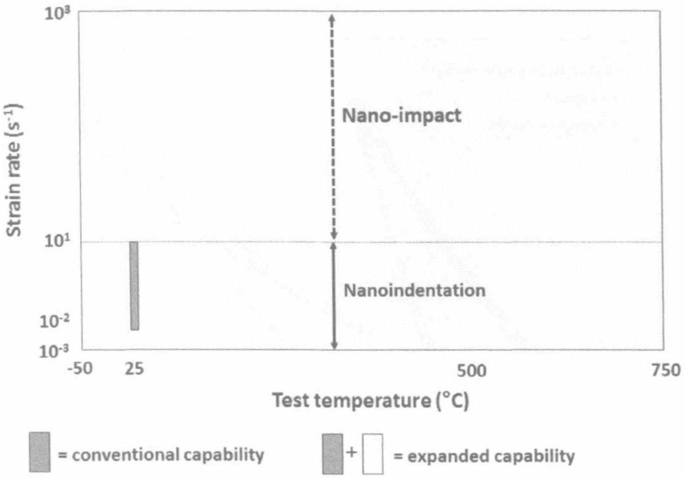

Figure 1.2 Temperature and strain rate test capability in a commercial instrument (NanoTest system).

1.2 Nanoindentation

1.2.1 *Contact Mechanics Theory*

Illustrative nanoindentation curves on fused silica, single-crystal tungsten and sapphire (0001) with a sharp pyramidal Berkovich diamond indenter are shown in Fig. 1.3. For a given indenter geometry the load–displacement curve recorded in a nanoindentation test can be thought of a "fingerprint" for a material as it contains information about the elastic and plastic properties of the sample under test.

The slope of the unloading curve at any point is called the contact stiffness. In this analysis, the reduced modulus, E_r, is calculated from the stiffness at the onset of the unloading, S, and the projected area of contact between the probe and the material, A_c, as

$$E_r = \frac{\sqrt{\pi}}{2\beta} \cdot \frac{S}{\sqrt{A_c}} \tag{1.1}$$

where β is the correction factor for the shape of the indenter. There is some ongoing debate over the exact value, though β is commonly taken as 1.034 for the Berkovich indenter geometry. The reduced indentation modulus, E_r, is directly determined in a nanoindentation test (also referred to as E'). However, the plane

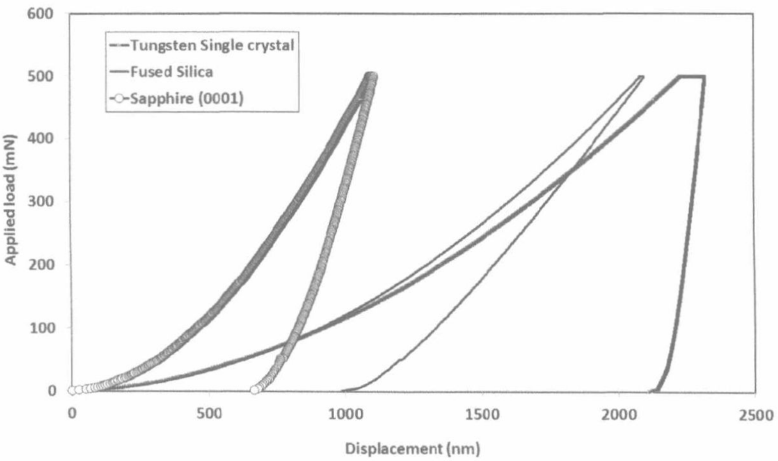

Figure 1.3 Nanoindentation curves on fused silica, single-crystal tungsten and sapphire (0001).

strain modulus, $E^* = E/(1 - \nu^2)$, can also be quoted (e.g., in ISO 14577 [1]) and when reporting nanoindentation data it is therefore necessary to specify clearly which modulus is being reported (E_r, E^*, or E). Conversion of the directly measured reduced modulus to the elastic (Young's) modulus of the sample requires that its Poisson's ratio be known or be reliably estimated. As elastic displacements occur both in the specimen and in the indenter (the indenter is not completely rigid), the elastic modulus of the sample is calculated from E_r using

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i} \quad (1.2)$$

where E and E_i, and ν and ν_i are the elastic modulus and the Poisson ratio of the tested material and the indenter, respectively. For diamond indenters, E_i and ν_i are 1141 GPa and 0.07, respectively. When a diamond indenter is used (i) on fused silica with $\nu = 0.17$, a reduced modulus of 69.6 GPa gives $E = 72$ GPa and (ii) for ceramics and hard coatings ν is typically 0.2–0.25. For sapphire with $\nu = 0.235$, an E_r of 314 GPa is equivalent to $E = 410$ GPa. (iii) For tungsten with a Poisson ratio $= 0.28$, $E_r = 320$ GPa converts to $E = 409$ GPa. The mean pressure or hardness (H) can be calculated as

$$H = P/A_c \quad (1.3)$$

where P is the applied load. Nanoindentation hardness is defined as the load divided by the projected contact area. However, in the definition of Vickers hardness the actual rather than projected area is used, resulting in a geometric scaling factor of 0.927 [5] and after correction for units a final relation of

$$H_V = 0.094545 H_{IT} \quad (1.4)$$

where H_V = Vickers hardness and H_{IT} is the DSI hardness.

1.2.2 *Practical Considerations*

1.2.2.1 Reference materials for calibration

Fused silica has proved the most popular material for calibrating nanoindentation instruments and the test probes (indenters) they use. It is inexpensive, highly polished, and mechanically homogeneous and has relatively little time-dependent behavior due to its