Nanoindentation: High Temperature

1. Introduction

Instrumented nanoindentation has become a ubiquitous method to characterize the mechanical properties of materials on the small scales necessary for such applications as thin film coatings and micromechanical devices, for which bulk tests are difficult or impossible (for more specific information, see Nanoindentation Techniques). The extension of nanoindentation techniques to elevated temperatures is a relatively recent development, and a routine testing technique with established best practices has not yet emerged. However, the potential impact of high-temperature nanoindentation is extremely high for both engineering applications where components or devices are expected to see elevated temperatures in manufacture or in service, and scientific investigations where thermally activated phenomena are of interest.

The introduction of high temperatures to a sensitive technique challenges our ability to measure properties with resolution on the nanoscale, and thus a significant focus of the field has been on refinement of the technique. Parallel with its development, however, high-temperature nanoindentation has also been applied to study a wide range of temperature-sensitive materials phenomena, including temperature-dependent mechanical properties, deformation mechanisms, rheology, and phase changes.

2. Technique Evolution

Since the inception of conventional hardness testing and through the subsequent development of more sophisticated instrumented indentation equipment, researchers have always adapted the available testing techniques and equipment to incorporate high-temperature capabilities. For example, ‘hot hardness’ tests on the macro- and microscale have been performed since the 1960s. Later, in the 1990s, when instrumented indentation on the micro- and nanoscale became widely used and accepted, high-temperature micro- and nanoindentation experiments became more regular, driven by both the scientific and industrial communities eager to mechanically test at the decreasing scales of new technologies, at relevant processing and service temperatures.

A broad survey of the early high-temperature micro- and nanoindentation studies (after Schuh et al. 2006) reveals a trade-off between resolution and temperature (Fig. 1). Tests above 500 °C have typically been limited to larger scales, while nanoscale tests have been restricted to relatively low temperatures—in many cases, the maximum testing temperature has not exceeded 200 °C. In the following sections, current limitations for achieving high resolution at the finest scales, including thermal drift and sample oxidation, are discussed. These topics frame the major concerns of the field today, which continues to expand the working range to include high-resolution testing at temperatures up to and above 500 °C.

3. Instrumentation

As the field of high-temperature nanoindentation is relatively new, custom instruments abound, though several commercial instruments are also available from Hysitron (Minneapolis, MN, USA), MTS Systems (Eden Prairie, MN, USA), Micro Materials (Wrexham, UK), and Fischer-Cripps Laboratories (Sydney, NSW, Australia). Whether custom or commercial, most instruments are essentially standard room-temperature instrumented nanoindentation systems, augmented with appropriate heating elements as well as shielding and cooling systems for temperature-sensitive components such as force and displacement transducers and sensors.

Specific heating methods across the various available instruments range from localized sample stage heating to heating of the entire instrument chamber. Regardless of the specific heating arrangements, temperature is generally maintained to within 0.1 °C. Thermal isolation of the sensitive measurement

![Figure 1](image-url)

The interplay between temperature and indentation scale established through several micro- and nanoindentation studies (reviewed in Schuh et al. 2006). The points define a region of temperature and scale (shaded), which are accessible using current techniques and instrumentation.