



High Temperature Nanomechanical Testing **68**

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Abstract

Elevated temperature nanomechanical testing is becoming a very popular technique to unravel temperature effects on the deformation mechanisms of a number of material systems, especially in those cases where it is the only available technique for mechanical testing, like in thin-films and coatings. This chapter presents several success stories where nanoindentation and micropillar compression were applied at elevated temperature to study the temperature-dependent strength and strain rate-dependent behavior. Nevertheless, we recognize several areas which require further developments for elevated nanomechanical testing to become a widely used and robust technique: more automated approaches to

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continuously monitor and correct for thermal drift and more stable and longer-lasting new indenter materials.

1 Introduction

Several nanoindentation systems currently exist in the market capable of operating at non-ambient conditions. They not only offer new measurement capabilities that enrich our scientific understanding of basic material response, including strain rate and temperature effects on deformation mechanisms at the micro- and nanoscale, but also allow testing relevant technological materials under real operating conditions, widening the applicability of these techniques to important industrial applications, such as the hard coating industry, high temperature metallic alloys, etc.

The first attempt to develop a nanoindentation system capable of operating at elevated temperature can be traced back to 1996 when Suzuki and Ohmura [1] presented their high temperature ultramicro indentation apparatus capable of testing up to 600 °C. The measurements were affected by thermal drift and system instabilities, but since then, there have been many improvements to both the instrumentation and experimental methodology [2, 3]. Technological developments have allowed performing stable tests at different strain rates up to 900 °C with low thermal drifts in different ambient conditions. High temperature levels depend on the application of the materials being tested in order to get relevant data; for example, testing eutectic solders up to 150 °C is enough, while thermal barrier coatings require testing temperatures beyond 750 °C.

In 2004 Uchic et al. [4] presented the first experimental tests in which micropillars milled from a substrate using focused ion beam (FIB) were compressed using a flat punch indenter. The enormous benefit of this approach is that small samples can be extracted out of almost any material. A wide range of micro- and nanoscale geometries are routinely manufactured using FIB for nanomechanical testing. Moreover, the combination of nanoindentation systems with scanning electron microscopy (SEM) and transmission electron microscopy (TEM) techniques enables us to observe how the sample microstructure and morphology change as it is being deformed [5]. Another benefit is that oxidation of the sample during elevated temperature tests is drastically reduced inside the vacuum chamber. Technological developments in the high temperature capabilities by nanoindenter manufacturers provide access to study the size effects at elevated temperature, which provides invaluable information for the design of scale-dependent properties. For instance, we know that mechanical properties of materials are size-dependent and that this is influenced by temperature and ambient conditions; this knowledge is critical for the further development of high-quality micro-/nanodevices.

Many applications in the aerospace, automotive, cutting tool, and nuclear sectors require knowledge of high temperature properties; i.e., environmental and productivity drivers (i.e., removing coolant to cut dry and higher cutting speeds) require high temperature nanomechanical testing of hard coatings used in high-speed machining because of the extreme frictional heating [6].

In Sect. 2, the critical issues for performing high temperature nanomechanical testing are described. Section 3 deals with the application of high temperature nanoindentation and micropillar compression methods to study mechanical properties as a function of temperature. Rate effects, including studies of creep, strain rate sensitivity, activation volume, and activation energy, are the topics of Sect. 4. Finally, Sect. 5 makes some final remarks on the future developments that are required for more robust elevated temperature measurements in nanomechanical testing.

2 Key Issues for High Temperature Nanomechanical Testing

The most important challenges that any type of load- and depth-sensing mechanical test faces when the testing temperature is increased refer to the good functioning of the system electronics, the management of thermal drift, and the chemical stability, not only of the specimen itself but also that of the indenter tip material. Since a number of reviews have tackled some of these instrumental issues in detail [3, 7–12], this section provides only a short summary. There are other critical issues for ensuring accurate hardness and modulus results that are more application dependent and apply particularly when performing high temperature nanoindentation that will be dealt with in the next section.

2.1 Instrument Stability

In any configuration of a nanoindenter instrument, the heating stage should not affect the system electronics. For reliable high temperature tests, the transducer and the measurement electronics must be protected by some method; typical design strategies include heat shielding of components and large cooling blocks for effective heat dissipation. The transducer is normally placed behind a heat shield which has a circulating fluid around it to prevent heat reaching the transducer. Carefully placed thermocouples are sometimes used to monitor transducer temperature. The displacement calibration should not vary within the temperature range of the test.

2.2 Thermal Drift

Thermal drift is a key issue which introduces time-dependent errors in the measured displacement during nanoindentation. Thermal drift occurs from thermal expansion within the system that results in additional displacement superimposed over the measured displacement during testing. Due to the nanometer length scales involved in nanoindentation, temperature variations of less than a degree Celsius can cause significant thermal drift. It can be separated into frame and contact drift components [3]. Frame drift, which arises from temperature variations within the system components, is minimized by introducing stabilization periods prior to the

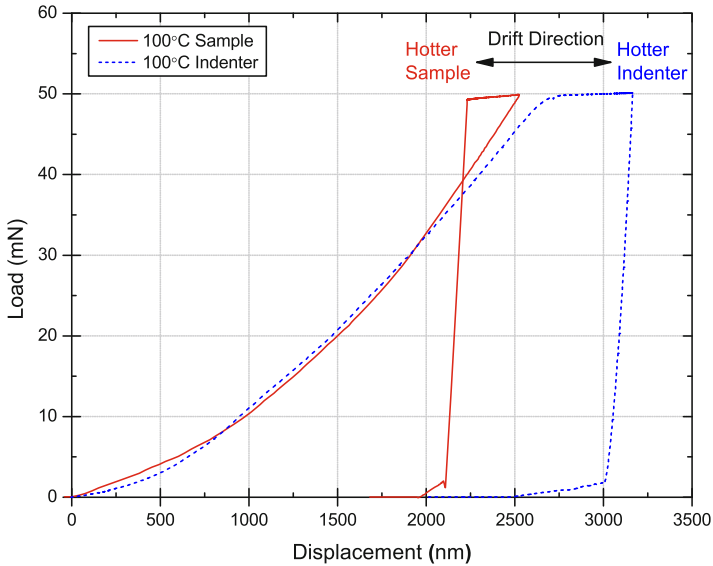


Fig. 1 Load-indentation depth curves for indentations performed under load control in Al with either the sample or the indenter tip heated to 100 °C, while the other remains at 25 °C [11]

measurements, whereas contact drift, which is due to heat transfer between the tip and the sample, is minimized by ensuring that the tip and sample are at thermal equilibrium before performing the high temperature test. There are several factors that affect the amount of heat flow at the tip-sample contact such as material properties, contact area, and ambient conditions; in vacuum, thermal equilibrium is more difficult than in air, as heat convection is suppressed. Thermal drift has even been observed to be nonlinear [9] which is particularly critical for creep experiments where load is held constant and displacement is measured for extended periods of time. In this case, a low and constant thermal drift is necessary.

Nanoindenter manufacturers offer different heating arrangements and solutions for high temperature testing in the desired conditions. Special high temperature tips with an insulating shaft are used with these systems. In one commercial system, isothermal contact is achieved by heating the probe and the sample separately using a patented control procedure [9]. Other designs are based on dual resistive heating elements that eliminate temperature gradients and employ passive tip heating for isothermal tip-sample contact. Figure 1 shows the effect of having a large temperature gradient (≈ 100 °C) between the diamond indenter and an Al sample on the load-displacement curves. The red curves correspond to a case where the indenter was at room temperature (RT) and the sample at 100 °C, resulting in heat flow toward the indenter and causing the thermal expansion of the tip. In this scenario, the system reacts by withdrawing the indenter in order to maintain the prescribed constant load, giving rise to a spurious “reverse creep” effect in the indentation depth (negative displacement drift). The opposite behavior (increasing penetration

depth with time at constant load) would be observed when the indenter was hotter than the sample, as shown for the blue curve (positive displacement drift).

The main strategies used to match tip-sample temperature are based on monitoring thermal drift and adjusting the temperatures to minimize the drift. Thermocouples placed near the contact at the sample surface or even directly at the tip are also helpful to monitor the heat flow direction and act accordingly to ensure temperature matching, but this is not always feasible. The different methods for thermal drift management and correction have been thoroughly discussed by Wheeler et al. [3, 12].

2.3 Indenter Tip Material

The choice of indenter material is crucial in order to avoid tip degradation and chemical interactions at elevated temperatures. Diamond is the standard material choice for room temperature indentation because of its hardness. For high temperatures, diamond can safely be used up to 400 °C. Above 450 °C, diamond starts to oxidize and can also react with steel and other metallic species to form carbides. When in contact with transition metals like Ti, Fe, or Co, the erosion of diamond starts at even lower temperatures. High-vacuum nanomechanical testing significantly reduces oxidation and allows diamond indenters to be used at higher temperatures. As an alternative, sapphire (single crystal alumina) has no problem with oxidation, but it can react with some metals [7], and its hardness drops with temperature, which can be problematic when testing hard materials, like hard coatings. Another candidate is cubic boron nitride (cBN), which has mechanical properties closer to diamond at room temperature and superior oxidation resistance, but its hardness also decreases quickly with temperature, and it might react with some elements at high temperatures, to form more stable borides. Tungsten Carbide (WC) is inert in combination with most materials and a suitable choice for indenting softer metals and alloys. In conclusion, the indenter material should be selected according to the maximum testing temperature, chemical reactivity with the material being tested, and choice of test environment. Wheeler et al. provide an excellent review of tip material choices and interactions with samples for high temperature testing [7], and the most relevant observations are summarized in Table 1.

In addition to the premature wear caused by the interaction between indenter material and sample, adhesion effects might result in the loss of material from the tip and/or material transfer from the specimen to the indenter tip, resulting in indenter tip contamination. Both phenomena will cause reshaping of the tip and a change of the tip geometry. Increased wear of the diamond tip has been observed after a small number of indentations on a hard SiC coating at 300 °C, after which recalibration of the indenter tip area function was required [13] (see Fig. 2a, b). Degradation of a diamond tip after indentation of Al at 300 °C was also observed in that study due to transfer of material from the coating to the indenter tip, as shown in Fig. 2c, presumably coming from the oxidation of the Al surface.

Table 1 Chemical indenter material interactions during high temperature nanoindentation [7]

	Sample material classes						
	Alkali metals	Alkaline earth metals	Early transition metals	Ferrous metals	Noble metals	Coinage metals	Metalloids
Indenter material	Li, Na, K, Rb, Cs	Be, Mg, Ca, Sr	Se, Ti, V, Cr, Y, Zr, Nb, Mo, Hf, Ta, W	Mn, Fe, Co, Ni	Ru, Rh, Pd, Re, Os, Ir, Pt	Cu, Zn, Ag, Cd, In, Sn, Au, Pb, Bi	Al, Si, Ge
Diamond	Inert	Inert	Dissolves carbon/forms carbides	Dissolves carbon e.g., <i>Fe</i> ~400 °C	Dissolves carbon >1000 °C	Inert	Forms Al_4C_3 > 700 °C with Al Forms SiC > 1200 °C with Si
Carbides B_4C	Unknown	Inert	B_4C reacts with Ti and Zr	B_4C forms borides/carbides	Inert	Inert	Forms borides
SiC	Unknown	Inert	Inert	Dissolves SiC	Inert	Inert	Forms Al_4C_3 with Al
W/C	Unknown	Inert	Mostly inert – May lose carbon by diffusion	Mostly inert – May lose carbon by diffusion	Inert	Inert	Inert
Nitrides <i>cBN</i>	Unknown	Inert	Mostly inert Mo, Ti, and Cr may form nitrides/borides	Dissolves e.g., <i>Ni</i> ~300 °C	Inert	Inert	Forms borides and nitrides
Oxides Al_2O_3	Dissolves	May reduce oxide	Mostly inert Ti and Cr may reduce oxide, e.g., <i>high Cr steel</i> ~700 °C	Dissolves, e.g., <i>Ni</i> ~300 °C	Dissolves in Pd and Pt	Dissolves in Zn and Bi	Reduces oxide, e.g., Al ~400 °C

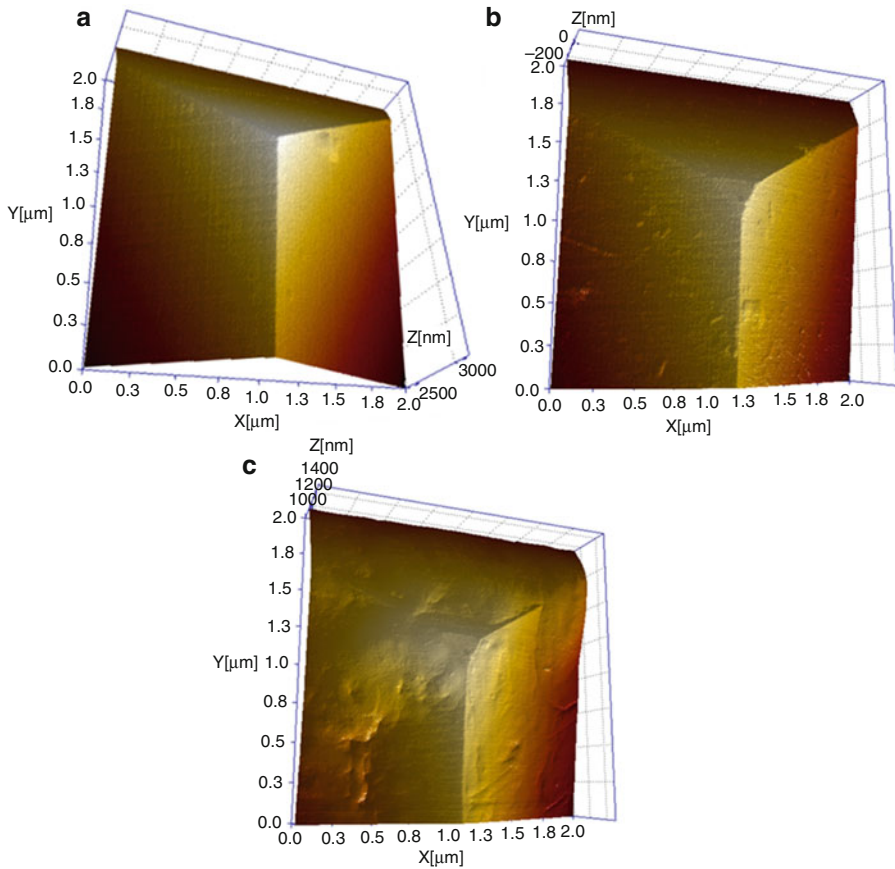


Fig. 2 AFM 3D topography images ($2\ \mu\text{m}$ scan size) of (a) a new diamond Berkovich tip; (b) after nanoindentations in SiC coating at $300\ ^\circ\text{C}$ and (c) after nanoindentation in Al coating at $300\ ^\circ\text{C}$ (From [13]). These observations emphasize the importance of carrying out indenter tip shape calibrations on a regular basis, especially when indenting and scanning hard samples and metals that readily oxidize at $T > 200\ ^\circ\text{C}$, so that the tip gets easily contaminated

2.4 Ambient Atmosphere

The ambient atmosphere during high temperature testing is an important consideration because many metals oxidized readily in air. Oxidation rates increase with increasing temperature, and the evolution of an oxide on a sample will greatly affect the measured properties. When testing metals, it is therefore necessary to reduce or eliminate oxidation in order to achieve accurate mechanical property measurements. Gas lines are usually used to purge the air around the testing area and slow down the oxidation process during testing. Argon gas is typically used to purge the chamber and establish positive pressure achieving oxygen levels of 0.01%. In other configurations, compressed dry air and inert gases (Ar or N_2 with a few percentage of H_2)

are used to purge the testing area to prevent heated gases reaching the transducer and reduce oxidation.

Another possibility is to deposit a few nm of a passivating layer (e.g., Ti or Cr) in order to protect the surface from further oxidation. Tests should be performed at sufficient penetration depths in order to minimize the effect of the passivating layer on the measured properties.

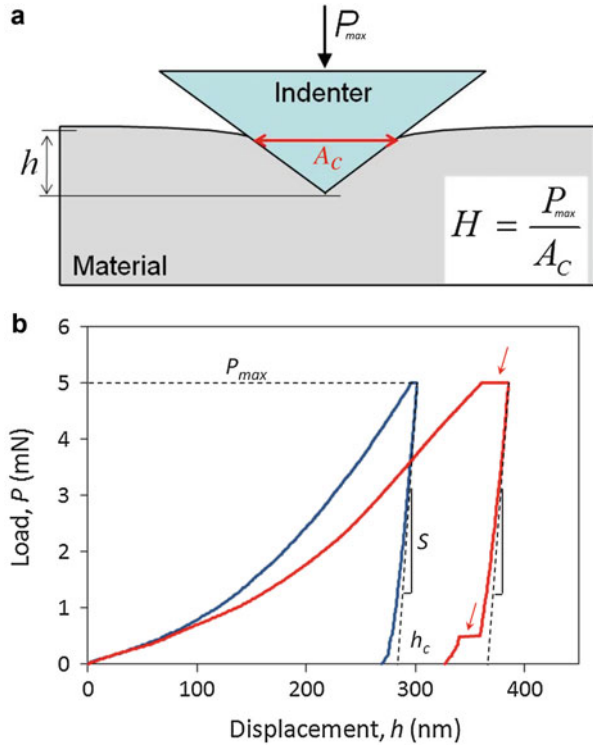
There is also the option to perform tests in an almost oxygen-free environment by housing the instrument within a high vacuum chamber which can achieve ultimate vacuum levels of 10^{-5} Pa [10]. In this case, thermal management is more difficult as heat convection is eliminated as a means of equilibrating temperatures, but at the same time, temperature stabilization is faster (particularly above 600 °C) as radiation heating assists equalization of tip and sample temperatures. Appropriate design of cooling systems and thermal drift correction methods make high temperature vacuum indentation the preferred method for testing metals that readily oxidize, where high temperature measurements up to 950 °C have been reported [10].

3 Nanomechanical Testing Studies Revealing Temperature-Dependent Strength

Up to the beginning of the twenty-first century, the study of mechanical properties at small scales was almost exclusively limited to the application of instrumented nanoindentation. However, with the availability and application of focused ion beam (FIB) milling to micromachine micrometer-scale 3D geometries with high precision, a novel nanomechanical testing discipline has emerged, by which conventional mechanical tests, like tensile, compression, or bending tests, are reproduced at the micro- and/or nanoscale. Among them, micropillar compression has become widely popular to assess mechanical properties at small scales, particularly at high temperatures. Therefore, in the following, the progress on elevated temperature nanomechanical testing has been divided into two main topics: instrumented nanoindentation and micropillar compression.

Nanoindentation involves pressing a hard tip with a well-defined geometry into the sample surface, while force and depth are measured continuously during loading and unloading. Commonly used indenter tips have self-similar geometries with depth (i.e., the ratio between contact area and contact depth is constant), like conical, pyramidal Berkovich and cube-corner indenters. For self-similar indenters, the elastoplastic deformation induced by the indenter is, in principle and if not affected by indentation size effects, independent of indentation depth and representative of an equivalent plastic strain ε_{eq} , which depends on the included angle of the indenter ($\varepsilon_{eq} \approx 0.08$ for Berkovich and ≈ 0.2 for cube-corner geometries). Despite the complex strain and stress fields underneath the indenter, hardness and modulus are commonly obtained from such nanoindentation tests, using contact mechanics and the well-known Oliver and Pharr method [14]. The reduced modulus, E_r , is calculated from the stiffness at the onset of unloading (S) and the projected area of contact between the indenter and the material (A_c), as:

Fig. 3 (a) Cross section of nanoindentation profile at peak load and (b) loading-unloading sequence for an indentation in Cu at room temperature (blue curve) and at 200 °C, indicating the parameters used to estimate hardness and modulus. Arrows point at the increased creep taking place at high temperature



$$E_r = \frac{\sqrt{\pi}}{2} \frac{S}{\sqrt{A_c}} \tag{1}$$

The unloading slope (S) is obtained from the elastic unloading, and the hardness, H , is calculated as:

$$H = \frac{P_{max}}{A_c} \tag{2}$$

where P_{max} is the maximum applied load (see Fig. 3a). By calibration into a reference material of known elastic properties (e.g., fused silica), the indenter area function can be determined relating the contact depth h_c to the projected contact area A_c .

Guidelines for accurate room temperature measurements of hardness and modulus are given in ISO 14577(1–4) [15]; however, high temperature nanomechanical testing requires adapting the measurement protocols described in the ISO approach so that the factors described in the previous section are taken into account.

Moreover, time-dependent inelastic deformation rates can increase significantly with testing temperature T , especially at high homologous temperatures $T_h (= T/T_m)$, where T is the test temperature and T_m is the melting temperature, both in Kelvin) > 0.6 , making the measurement of elastic stiffness on unloading more difficult.

Figure 3b shows the load-displacement curve for indentations performed on Cu at 200 °C, showing an increased creep deformation during the hold period, with respect to the room temperature result. If creep rates are not minimized during unloading, the contact area and elastic modulus determination can be seriously compromised; consequently, the loading histories need to be modified to include sufficiently longer hold times at peak load, to minimize the effect of creep during unloading [16]. Feng and Ngan [17] have suggested a correction term due to creep in the apparent contact compliance ($=1/S$) which has been successfully applied to correct high temperature properties data of Ni-based superalloys up to 400 °C [18].

Depending on the application, some of the critical issues described will be more relevant than others. For instance, critical requirements for testing advanced alloys are oxidation minimization and time dependency, whereas for hard coatings, indenter durability and tip wear are more of a concern. In the case of ceramic coatings, creep is usually small, and using hold times between 10 and 30 s is sufficient to ensure testing accuracy at high temperatures; in addition, oxidation is not that critical, and typically tests are performed in a purged Ar atmosphere with a diamond tip or with a cBN tip. For other materials (e.g., bcc metals, solders, and polymers), considerable time dependency occurs at lower temperatures, and care in the experimental design should be taken to ensure accurate elastic modulus measurements. In order to validate data, it is recommended to calibrate the system using a test reference sample (e.g., fused silica, tungsten) at the testing temperature and when possible compare the data with measurements performed with other techniques. The temperature dependence of the elastic modulus of the indenter used should also be taken into account as its value is used to convert from indentation reduced modulus to Young's modulus [14] (e.g., modulus of cBN decreases by $\approx 4\%$ between RT and 950 °C [7]).

Micropillar compression involves pressing a flat punch indenter onto a micropillar with a well-defined geometry. Because of the uniaxial loading imposed on the micropillar, the stress and strain are easily computed from the load-displacement data after applying the necessary corrections for substrate deflection, pillar taper, and misalignment between the punch and top surface of the pillar [19]. Material strength parameters such as yield point and flow stress can thus be computed from the resulting stress-strain curves, providing more information on the constitutive behavior of the material. Even though the same problems as those described for the case of nanoindentation might arise regarding sample-tip interactions, the local stresses are greatly reduced between the indenter flat punch and the head of the pillar, which generally alleviates the phenomena. It is therefore possible to use diamond flat punches to compress ferrous metals and/or transition metals up to relatively high temperatures, especially in vacuum environments.

3.1 High Temperature Nanoindentation Studies

It is better to discuss separately the elevated temperature nanoindentation studies performed on metals and ceramics, not only due to the very different nature of these

materials but also due to the very different scopes of the studies that have been performed in each case.

3.1.1 Metals and Semimetals

Several elevated temperature nanoindentation studies have focused on studying the effect of temperature on the hardness of metals. Due to the thermally activated nature of plastic deformation, temperature is expected to increase the dislocation nucleation rate (due to thermal fluctuations), the dislocation mobility, and the diffusion-mediated flow rate [20]. There are two main advantages associated with the use of nanoindentation to study thermally activated plastic flow. Firstly, only a small volume of material is needed for the test, so that either nanoindentation is the only available technique capable of measuring the mechanical properties of some materials (as it is the case for thin-films and some nanostructured metals), or it can be used in a high-throughput manner in a combinatorial material's discovery approach. And secondly, but not less important, depending on the scale of the nanoindentation test with respect to the characteristic size of the microstructure, it can be used to decouple different deformation processes: from the onset of dislocation nucleation for very small indentations to collective dislocation dynamics when the indentation is large but smaller than the grain size, including the effect of grain boundaries when the indentation size encompasses several grains. Several nanoindentation works on the effect of temperature in each case can be found in literature.

For instance, the effect of temperature (up to $\approx 0.2 T_m$) on the very early stages of plasticity during low-load nanoindentation of fcc and bcc oxide-free metal surfaces has revealed interesting phenomena on the dynamics of dislocation nucleation. For example, indentations on pure (100) platinum (bcc) revealed that temperature promotes a monotonic decrease in the critical load for dislocation nucleation and that the serrated nature of plastic flow was more severe as temperature was raised from 20 to 200 °C, indicating that these nucleation events were stress biased and thermally activated [21]. Nanoindentation of (001) tantalum (fcc) also exposed a change in the shape of the loading curves from one large pop-in marking the onset of plasticity at 25 °C to multiple pop-ins at 200 °C [22], due to the thermal activation of screw dislocations at temperatures above $0.15 T_m$. Other nanoindentation studies have also shown that the variability of the critical load for dislocation nucleation is due to thermal fluctuations, while in the case of metallic glasses, for which the onset of plastic deformation occurs by the nucleation and propagation of a shear band, the variability in the critical load is dominated by structural fluctuations [23].

Higher up in the length scale where the indentation is governed by the collective behavior of dislocations, several studies have also focused on the temperature-dependent single crystal behavior, either by indenting single crystals or individual grains far from grain boundaries in polycrystalline metals. For instance, some high temperature nanoindentation studies have focused on studying the competition between plasticity and pressure-induced phase transformations in single crystal silicon. At room temperature, it is well known that upon loading Si suffers a phase transformation from the diamond structure to a metallic tetragonal Si phase, which back-transforms to amorphous Si upon unloading, giving rise to a well-known

pop-out event [24]. However, several regimes have been observed at elevated temperature. While the room temperature behavior remained fairly constant up to 200 °C, at higher temperatures the pop-out disappeared, and the elastic modulus decreased substantially, due to the formation of amorphous silicon during loading [25]. However, the hardness of Si dropped substantially for temperatures > 350 °C, which was attributed to the transformation to another high-pressure metallic Si phase [26], while for even higher temperatures (between 500 and 650 °C), thermally activated dislocation flow controlled the indentation process, without evidence of any pressure-induced phase transformations [10].

Another interesting application of nanoindentation at the single crystal level is to use the nanoindentation response of grains with varying crystallographic orientation, in combination with single crystal plasticity (SCP) simulations, to determine the critical resolved shear stress (CRSS) of the different slip and twinning systems in magnesium [27]. In another study, the temperature evolution of the CRSS of the different deformation modes was determined using a sphero-conical indenter in the temperature range of 25–300 °C, demonstrating a transition in plasticity from basal slip and extension twinning-dominated flow at room temperature to basal and prismatic slip dominance at 300 °C [28].

The case of single crystal indentation cannot be completely discussed without making reference to the indentation size effect (ISE). ISE manifests itself in many single crystals as an increase in hardness with decreasing depth of penetration and becomes important at depths less than approximately 1 μm, as a result of the larger contribution of strain gradients caused by the density of geometrically necessary dislocations [29]. Nanoindentation in Cu at 200 °C has shown a significantly reduced ISE contribution, suggesting that thermal activation plays a major role in determining the length scale for plasticity [30].

Several elevated temperature studies can also be found in the regime where the nanoindentation imprint encompasses several grains, and therefore, plasticity is controlled by the polycrystalline behavior. Hot indentation hardness studies of polycrystalline aluminum (up to 100 °C) [13], gold (up to 200 °C) [25], and copper (up to 400 °C) [31] have shown a decrease of hardness with temperature, consistent with their polycrystalline bulk behavior. The high temperature indentation of polycrystalline metal alloys (Ni-W [32]) has shown that the extent of the hardness drop depends on grain size, being less pronounced for finer grain sizes, although this observation cannot be extended to other material systems, where a higher hardness drop with temperature has been found for smaller grain sizes. Another interesting example can be found in the case of polycrystalline tungsten, an important metal for the nuclear industry, which has been tested in vacuum up to 950 °C [16], the highest temperature reported in literature for a nanoindentation test. Hardness dropped from 6 GPa at 25 °C to 2 GPa at 950 °C, while the elastic modulus remained nearly constant up to 800 °C and increased for higher temperatures. The latter was, however, attributed to artifacts on the measured stiffness caused by the high creep rates encountered at $T > 750$ °C, as explained above.

Finally, the case of high-strength nanostructured metals deserves further attention. Numerous studies have reported on the development of nanostructured

materials, including nanograined, nanolaminated, and/or nanotwinned metals, as a strategy to produce high-strength materials exploiting the well-known “smaller is stronger” effect caused by the interaction of dislocations with the high density of boundaries and interfaces introduced in these materials. However, whether the high strength of such nanostructured metals will remain at elevated temperature or whether other deformation mechanisms will soften the response, even more than in the corresponding bulk coarse-grained counterparts, is a question that is still under debate. The development of elevated temperature nanoindentation is contributing to clarify this, as in many cases, these materials are produced in low dimensions (thin films and/or small volumes) and, therefore, cannot be tested by other means. For instance, the elevated temperature hardness of Cu/Nb nanolaminates was studied by nanoindentation up to 400 °C [33]. While the nanolaminates retained a substantially high hardness at elevated temperature, it was demonstrated that the hardness drop with temperature was layer thickness dependent and that below a certain critical layer thickness, the hardness drop increased significantly, as a result of thermally activated dislocation transmission at the Cu/Nb interfaces. Other studies in Cu/W nanolaminates, for which the W layer is significantly stronger than the Cu layer, demonstrated a dramatic reduction in properties at 200 °C, as a result of diffusion-mediated flow triggered by the high applied stresses and the small thickness of the layers [34]. These examples illustrate that the current knowledge on the mechanical behavior of nanostructured metals is still in its infancy and it is expected that the use of high temperature nanomechanical testing techniques will contribute to new developments in this area.

3.1.2 Ceramics

Even though the case of ceramic materials is mentioned after metals, it should be stated that the development of high temperature nanoindentation was initially driven by the hard coating industry, for which the elevated temperature performance of ceramic wear-resistant coatings is critical, due to the high temperatures that are reached at the contact between the cutting tool and the workpiece during machining. As a matter of fact, several examples of hot hardness measurements using cBN tips in hard ceramic coatings, like nitrides, carbides, and oxides, at temperatures up to 750 °C, can be found in literature [6, 35–37]. The hot hardness of nitride-based hard coatings, such as (Ti, Al)N and TiCN, was reviewed by Beake et al. [6], who highlighted the importance of measurements at elevated temperature, as the room temperature hardness is not always a good indicator of optimum cutting tool life. For instance, TiAlN coatings have a higher hardness (30 GPa) than AlCrN (25 GPa) at room temperature, but the hardness decreases more rapidly at higher temperatures for TiAlN, dropping to 15.4 GPa at 500 °C, while AlCrN retained a hardness of 20.5 GPa [37].

Worth mentioning is the case of fused silica, whose high temperature hardness and modulus were studied up to 400 °C [38], indicating an anomalous behavior with a slight softening and an increase in modulus with temperature, as opposed to other glasses such as soda-lime glass, where hardness and modulus both drop with temperature [25]. This is important as fused silica is the indentation reference material used for tip area calibration, which suggests that future development should consider other reference materials for elevated temperature nanoindentation.

3.2 High Temperature Micropillar Compression

As discussed above, with the increasing availability of FIB systems, micropillar compression has become a very popular technique for testing materials at small length scales, as it allows the determination of complete stress-strain curves, rather than just hardness values. As such, the number of studies that make use of this technique at elevated temperature is increasing exponentially.

However, there are two questions that one should address when trying to relate micropillar results with bulk mechanical behavior, even before considering elevated temperature testing. One is whether Ga⁺ ion implantation caused by FIB milling [39] influences the results, and the other is whether the results are affected by the size of the pillars. Neither of these questions has an unambiguous answer, as they are very material dependent and, sometimes, even interrelated. Regarding the effect of Ga⁺ implantation during FIB milling, it is estimated to extend only a few tens of nanometers below the surface, resulting also in an increase of dislocation density and point defects, which can undoubtedly affect the results, especially if the initial material contains a very low defect density [39]. However, in materials with conventional initial dislocation densities, the effect of FIB milling should be less important, but this has to be confirmed in each case to avoid undesirable effects. For instance, the strength of polycrystalline Al micropillars milled by FIB was shown to decrease due to Ga segregation at the grain boundaries [40]. Regarding the effect of pillar size, this is also very dependent on the material system. It is well known that as the pillar size decreases, plasticity might be limited by the availability and operation of dislocation sources [41], but the extent of the size effect will depend on the bulk strength of the material. It has been shown that for soft metals, such as pure fcc Al, Cu, Ni, or Au, the results are usually affected by large size effects [40, 42] but that those are strongly reduced or even diminished for strong metals and ceramics, yielding results that are representative of the intrinsic yield strength of the material [43–45]. In the latter case, an increase in temperature might reveal a pillar size effect, as the intrinsic yield strength of the material drops substantially, as has been demonstrated for bcc metals [46] or for ionic solids such as MgO [47] and LiF [45].

Therefore, and for the reasons stated above, care should be taken to extrapolate micropillar compression results to macroscopic behavior in pure metals and ceramics. There are cases, however, which are free of Ga + implantation-induced effects and pillar size effects, from which very relevant temperature effects have been obtained. For instance, it has become a very useful technique to study brittle-to-ductile transitions in brittle ceramics such as spinel (MgAl₂O₄) [43] or silicon [48]. The technique is also finding a clear application on the study of high temperature strong metallic alloys, as is the case for Ni-based superalloys, whose strength is controlled by the spacing of the γ' precipitates, at a scale much lower than the pillar size and which are stable at temperatures up to 650 °C. Micropillar compression tests on Ni-based superalloys up to 630 °C in vacuum resulted in modulus, yield, and flow stress values comparable to those from macroscopic tests [8]. It has also revealed itself as a very useful technique to study the mechanical behavior of thermal barrier coatings, like aluminide bond coats on Ni-based superalloys [49]. This study showed

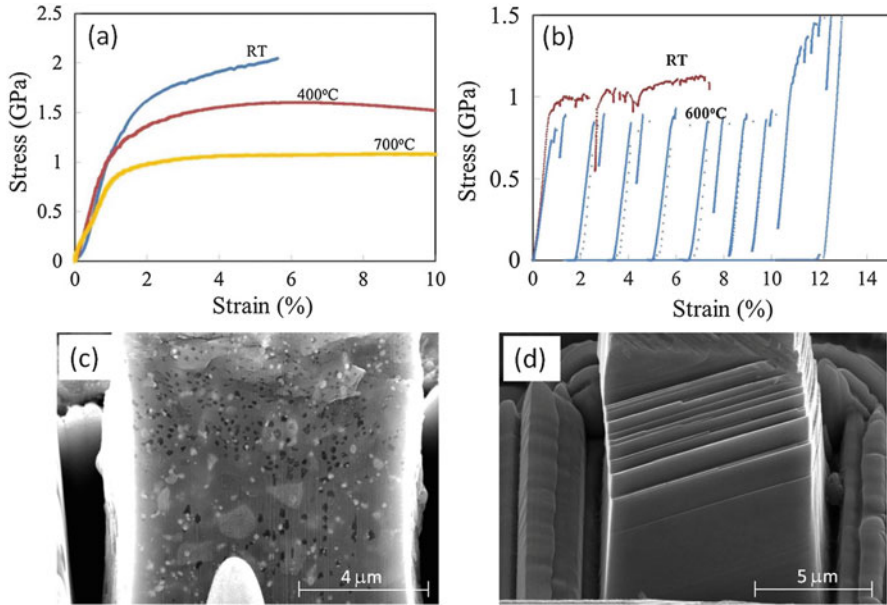


Fig. 4 Stress-strain curves of (a) bond coat – showing limited strain hardening and reduced yield stress at high temperatures – and (b) Ni-based superalloy, showing large load drops at high temperatures. Morphology of the pillars after compression of (c) bond coat at 800 °C – showing uniform deformation without slip band formation – and (d) superalloy at 600 °C, showing a large number of slip bands [48]. The results are useful on elucidating the change of deformation mechanisms with temperature

that the elastic modulus of the bond coat remained nearly constant from RT to 800 °C, whereas the yield stress decreased by $\approx 50\%$ (Fig. 4a). At 600 °C the yield strength of the superalloy sample decreased by $\approx 20\%$, with large load drops observed in the stress-strain curves associated with the formation of slip bands, which increases with temperature [49] (Fig. 4b). Uniform plastic deformation (grain boundary sliding) without slip bands was observed in the bond coat at high temperature (Fig. 4c), whereas multiple slip bands were observed in the Ni-based superalloy compressed micropillar (Fig. 4d), where the density and severity of the slip banding increased with temperature.

Another application where micropillar compression has turned out very useful to reveal high temperature deformation mechanisms is that of nanolaminates, mentioned in the previous section. Although some pillar size effects are possible [50], these are usually negligible provided the pillar size is much larger than the layer thickness, which controls the nanolaminate strength [50–52]. Several examples can be found in literature for metal/metal (i.e., Cu/Nb [51], Cu/Zr [53], and Cu/Cr [54]) as well as metal/ceramic (i.e., Al/TiN [55], Al/SiC [56], and Cu/TiN [57]) nanolaminates. Many of those studies have shown that increasing temperature might trigger stress-assisted diffusion, limiting the high temperature strength of these materials, especially for the thinnest layer thicknesses. For instance, Fig. 5 shows

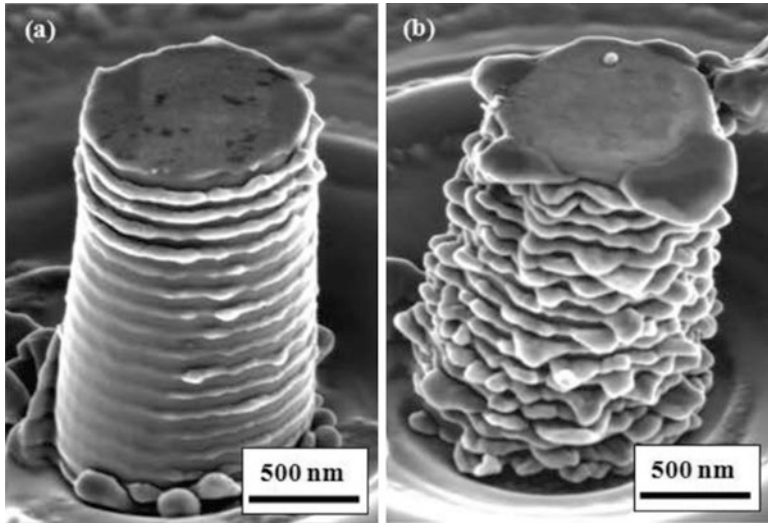


Fig. 5 SEM micrograph of Al/SiC nanolaminate micropillars deformed up to a plastic strain of 8% at (a) 23 °C and (b) 100 °C [56]. Plastic deformation is limited and localized in the Al layers at room temperature, whereas at 100 °C, extrusion of the Al layers is much more dominant along the micropillar, revealing the role played by stress-assisted diffusion on deformation

two Al/SiC nanolaminate micropillars compressed at room temperature and 100 °C. Large extrusions of the Al layers can be observed at the surface of the pillar tested at 100 °C, as a result of the activation of the stress-assisted diffusion, limiting the elevated temperature strength of these materials.

Finally, the high temperature strength of a wide range of chromium nitride-based hard coatings has also been evaluated using in situ micropillar compression up to 500 °C [58]. This allowed the first direct measurement of the high temperature compressive strength, rather than the hardness, of such coatings. The addition of Si and/or Ti to the coatings was found to be beneficial for decreasing the effect of temperature on coating strength.

4 Nanomechanical Testing Studies Revealing Time-Dependent Behavior

Another area of active research in nanomechanical testing is the investigation of time-dependent plasticity, such as strain rate effects and creep behavior, especially with the emergence of elevated temperature testing. The possibility of determining strain rate sensitivity, activation volumes, and activation energies at the nano- and microscale is very appealing, as these parameters can give essential information to unravel the rate-controlling deformation mechanisms as a function of applied stress, strain rate, and temperature.

The strain rate sensitivity of plastic flow of a material is based on the response of the underlying deformation mechanisms to a change in stress or strain rate. An important relationship for understanding the interplay between strain, stress, temperature, material properties, and microstructural properties is the Mukherjee, Bird, and Dorn (MBD) semiempirical relationship [59]:

$$\dot{\epsilon} = A \frac{DGb}{kT} \left(\frac{b}{d}\right)^p \left(\frac{\sigma}{G}\right)^n A' \sigma^n \exp\left(\frac{-Q}{RT}\right) \quad (3)$$

where $\dot{\epsilon}$ is the steady-state strain rate, D is the appropriate (lattice or grain boundary) diffusivity, G is the shear modulus, b is the Burgers vector, k is the Boltzmann constant, T is the test temperature in Kelvin, d is the grain size, p is the grain size exponent, σ is the applied stress, and n is the creep exponent. The diffusivity, the strain rate sensitivity, and the factor $A(b/d)^p$ are creep mechanism dependent. Under constant temperature and microstructure, the relation between strain rate and stress level reduces to:

$$\dot{\epsilon} = A'' \sigma^n \quad (4)$$

and the strain rate sensitivity m , defined as $m = \partial \ln \sigma / \partial \ln \dot{\epsilon}$, is given by $m = 1/n$, with a value between 0 (rate-independent limit) and 1 (linear Newtonian flow). Increased contribution of grain boundary sliding and diffusion processes is usually observed at higher m values, while lower m values usually indicate dislocation glide-controlled deformation processes and a larger tendency for strain localization.

Additionally, the activation volume represents a physical volume proportional to the number of atoms involved in the thermally activated rate-controlling deformation mechanism [20]. The applied stress works on this volume to assist deformation by reducing the energy barrier. The apparent activation volume V for plastic deformation is defined as [20]:

$$V = \sqrt{3} kT \frac{\partial \ln \dot{\epsilon}}{\partial \sigma} \quad (5)$$

Combining Eqs. (4) and (5) with the definition of strain rate sensitivity, it follows that:

$$V = \sqrt{3} \frac{kT}{\sigma m} \quad (6)$$

Different deformation mechanisms usually involve specific activation volumes, as summarized in Table 2, where V^* represents the number of unit volumes in terms of the magnitude of the Burgers vector b , as $V^* = V/b^3$.

It is possible to use nanomechanical testing, especially when subjected to varying temperatures, to unravel strain rate effects. Several strategies have been put forward, of which the most important are summarized below.

Table 2 Typical activation volumes measured for some relevant characteristic deformation mechanisms

Mechanism	Activation volume (V^*)
Cobble creep/GB-mediated deformation	$<0.1b^3$
Dislocation nucleation	$1b^3-10b^3$
Cross-slip	$10b^3-100b^3$
Dislocation-dislocation interactions	$100b^3-1000b^3$
Dislocation forest cutting	$>1000b^3$

4.1 Constant Strain Rate Tests

One strategy to unravel strain rate effects is to compare nanoindentation and/or micropillar compression experiments carried out at varying strain rates. In the case of nanoindentation, this requires making some assumptions on the definition of a so-called indentation strain rate. The definition of an indentation strain rate makes use of the self-similarity of conical and pyramidal indenters and the fact that the representative plastic strain imposed by the indenter and the indentation pressure (hardness) are constant with indentation depth, as underlined above. Under these circumstances, it is possible to define an indentation strain rate $\dot{\epsilon}_{\text{ind}}$ as \dot{h}/h (displacement rate divided by actual displacement). Since for pyramidal indentation, the loading curve follows the relation, $P = Ch^2$ [60], where P is the load, h is the displacement, and C is the indentation curvature (i.e., resistance of the material to indentation), it follows that:

$$\dot{\epsilon}_{\text{ind}} = \frac{1}{2} \frac{\dot{P}}{P} \quad (7)$$

and therefore, a constant indentation strain rate test can be easily programmed in any indentation instrument by imposing a proportional loading rate over the entire indentation test. Several studies [61] have demonstrated that an analogy can be drawn between rate effects in uniaxial testing and in indentation as hardness (H) is related to the flow stress (σ_y) by a constraint factor (C_f) which depends on the indented material and has a value of $\approx 2-3$ for many metallic materials [61], such that $H = C_f \sigma_y$. Eq. (3) can then be rewritten as:

$$\dot{\epsilon}_{\text{ind}} = B' H^n \exp\left(\frac{-Q}{RT}\right) \quad (8)$$

so that the strain rate sensitivity can be obtained from the slope of a log-log plot of hardness versus indentation strain rate:

$$m = \frac{d(\ln H)}{d(\ln \dot{\epsilon}_{\text{ind}})} \quad (9)$$

while the activation volume can be computed from:

$$V = 3\sqrt{3} \frac{kT}{Hm} \quad (10)$$

With the currently available indentation instruments, indentation strain rates in the range 10^{-1} s^{-1} to 10^{-4} s^{-1} are easily accessible, although the lowest indentation strain rates are only accessible when thermal drift is extremely low and constant, since a typical indentation test at these conditions at a maximum depth of 1–2 μm might last between 30 and 60 minutes.

Additionally, when tests are performed at varying temperatures, it is also possible to determine the activation energy. Taking the logarithmic on both sides of Eq. (8) yields:

$$\log(\dot{\epsilon}_{\text{ind}}) = \log B' + n \log(H) - \frac{Q}{RT} \quad (11)$$

so that the activation energy Q can be computed from the slope of $\log(\dot{\epsilon})$ versus $(1/T)$. Several studies that use this approach can be found in the literature giving reasonable results. For instance, nanoindentation tests on high-purity indium [62], from room temperature to 75 °C, yielded a stress exponent for indentation creep of 7.3 and an activation energy of 78 kJ/mol, in agreement with the activation energy for self-diffusion in the material. The activation energy ($\approx 0.3 \text{ eV}$) and volume ($\approx 1\text{b}^3$) of single crystal platinum have also been determined from the onset of plasticity during nanoindentation up to 200 °C by Mason et al. [63], but the method required a large number of experiments and a complex mathematical approach. An example of determination of activation parameters from nanoindentation data performed at three different strain rates and temperatures on nanotwinned (NT) and coarse-grained (CG) Cu is shown in Fig. 6. The strain rate sensitivity for NT-Cu was found to be always higher than that for CG-Cu at both room and elevated temperature. An activation energy of 65 kJ/mol was found for CG-Cu, which is reasonable for a homologous temperature < 0.2 , while a higher activation energy of the order of 104 kJ/mol was obtained for NT-Cu.

In the case of micropillar compression, driving a constant strain rate (CSR) compression test is straightforward provided the indentation instrument is capable of imposing a constant displacement rate. For instance, the mechanical behavior of Cu/Cr multilayer films and $\text{Cu}_{20}\text{Cr}_{80}$ at.% alloys was studied by CSR micropillar compression up to 300 °C [64]. A transition from shearing and crack formation across the Cu/Cr interfaces leading to anomalous grain growth or beading of the nanocrystalline Cu layers was observed at elevated temperatures. On the other hand, the $\text{Cu}_{20}\text{Cr}_{80}$ alloy film exhibited the highest strengths with negligible temperature dependence.

However, one of the drawbacks of the constant strain rate approach to determine strain rate effects is that a large number of tests are needed at varying strain rates, in order to overcome the experimental scatter inherent in any nanomechanical test. An emerging method that is gaining popularity and that overcomes the above described limitations is the use of strain rate jump tests. Strain rate jump testing is well known in conventional macromechanical testing, and has been extended to nanomechanical testing, as described in the next section.

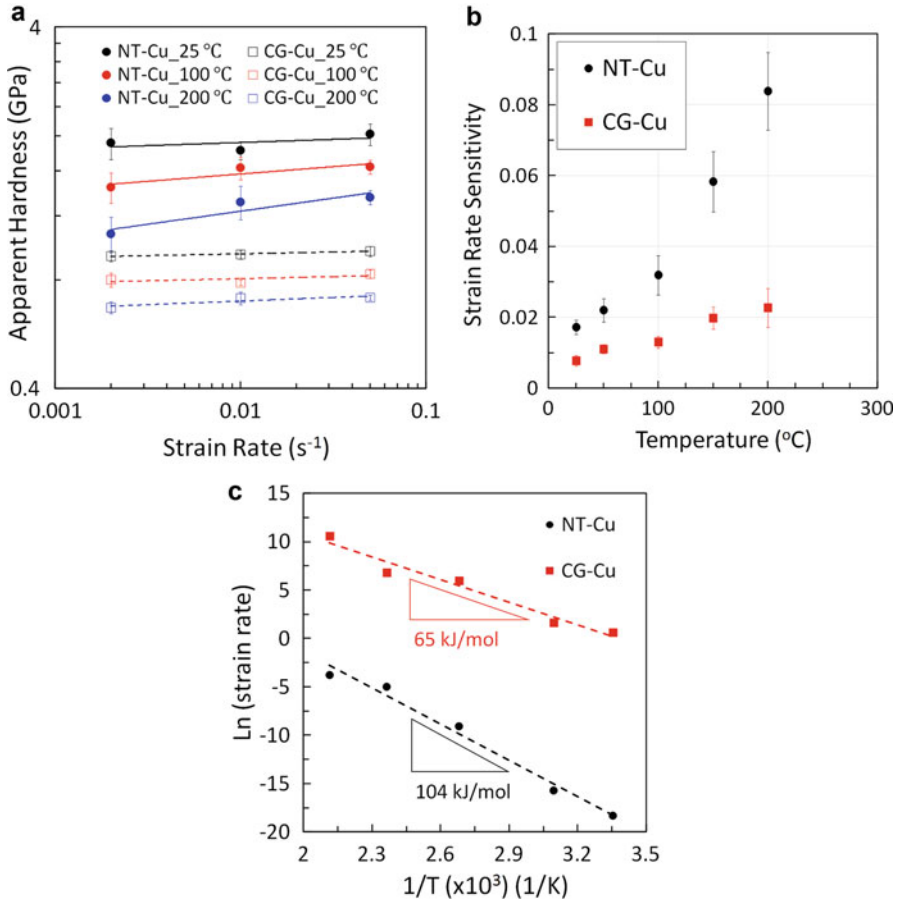


Fig. 6 (a) Hardness as a function of strain rate, (b) strain rate sensitivity as a function of temperature, and (c) Arrhenius plots of the temperature dependence of the strain rate to determine the apparent activation energy of nanotwin Cu (NT-Cu) and coarse-grained Cu (CG-Cu)

4.2 Strain Rate Jump Tests

In a strain rate jump (SRJ), the change in stress resulting from a sudden jump in the imposed strain rate is used to determine the strain rate sensitivity (m) and activation volumes (V^*). The method can either be applied to a nanoindentation or a micropillar compression test and was first proposed in the framework of nanoindentation by Alkorta et al. [65] and Maier et al. [66]. The main advantages over the constant strain rate method are that the strain rate sensitivity can be determined from a single indentation experiment by introducing several strain jumps, as shown in Fig. 7 [83], minimizing the inherent scatter associated with different nanomechanical tests, and the reduction in indentation time, which eliminates problems associated with thermal drift effects.

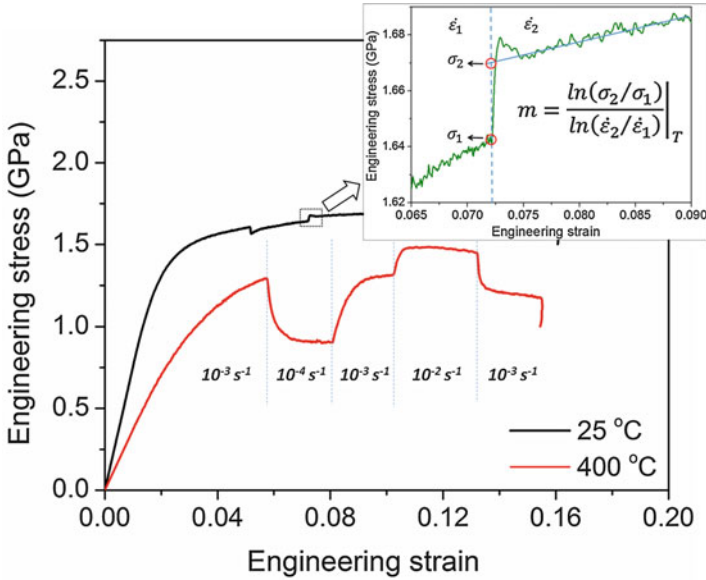


Fig. 7 Example of micropillar compression stress-strain curves for a Cu/Nb nanolaminate [83], showing the jumps in flow stress due to the change in imposed strain rates (as indicated in the figure) at 25 and 400 °C. The size of the strain rate jumps suggests a larger increase in the strain rate sensitivity (SRS) with temperature. The inset shows a higher magnification image of one of the jumps: a linear curve is fitted to the constant strain rate segment after the jump and extrapolated to the jump strain. Stress levels σ_1 and σ_2 are measured, and the strain rate sensitivity is calculated from Eq. (12)

The strain rate sensitivity is calculated by using Eq. (12) from hardness/flow stress and strain rate before and after each jump. Ideally, the hardness should be measured continuously using continuous stiffness measurement during testing; otherwise, an apparent hardness value should be used, although the latter introduces some errors, as the elastic contribution to the indentation depth is not accounted for.

$$m = \frac{\ln(\sigma_2/\sigma_1)}{\ln(\dot{\epsilon}_2/\dot{\epsilon}_1)} \Big|_T = \frac{\ln(H_2/H_1)}{\ln(\dot{\epsilon}_2/\dot{\epsilon}_1)} \Big|_T \quad (12)$$

Examples of the temperature-dependent activation parameters obtained from SRJ and CSR tests are given in Table 3. Wheeler et al. [67] used SRJ nanoindentation tests at different temperatures to measure activation parameters for UFG and CG aluminum. In this case, the activation energy was obtained using the modulus-compensated hot hardness analysis of Sherby and Armstrong [68] from the slope of a logarithmic plot of H/E against reciprocal absolute temperature. Mohanty et al. [69] used SRJ micropillar compression tests on nanocrystalline nickel films from 25 to 100 °C, from which strain rate sensitivity and activation volume values were determined as a function of temperature. Elevated temperature SRJ micropillar compression tests

Table 3 Some examples of application of high temperature nanomechanical testing for the determination of strain rate effects.

Materials	Method	Properties investigated	Maximum test temperature [ref]
UFG and CG Al	SRJ nanoindentation	m , V^* , and Q	250 °C [67]
NC-Ni	SRJ micropillar compression	m , V^* , and Q	100 °C [69]
GaN	SRJ micropillar compression	m , V^* , and Q	480 °C [71]
GaAs	SRJ micropillar compression	Q + effect of lithography vs. FIB machining	400 °C [72]
InSb	SRJ micropillar compression	m , V^* , and Q	300 °C [70]
Cu/Cr	CSR micropillar compression	Q	300 °C [64]
Decagonal Al-Ni-Co quasicrystals	SRJ/CSR micropillar compression	Size-temperature-strength deformation map	500 °C [73]

have also been performed in InSb at temperatures up to 300 °C to investigate the changes in strain rate sensitivity above and below the ductile-to-brittle transition temperature [70]. Observations of the deformation modes, activation energies, and activation volumes confirm that a transition from partial to perfect dislocation gliding occurred at the ductile-to-brittle transition temperature, i.e., around 150 °C for InSb.

4.3 Creep Testing

Finally, a special mention of creep testing is necessary in the framework of elevated temperature nanomechanical testing, as several studies [74, 75] can be found in the literature making use of this approach for nanoindentation experiments. A nanoindentation creep experiment typically consists of a fast loading segment to a maximum indentation load, followed by a constant load segment over which the evolution of the indentation depth is followed for a long period of time [62, 76]. Typically, a nanoindentation creep curve has two regimes – an initial transient phase characterized by a sudden increase in depth, followed by a “steady-state-like” regime in which the rate of penetration stabilizes to a roughly constant value. This is similar to the primary and steady-state regimes of creep observed in bulk tensile testing. However, there are certain fundamental differences in these two types of testing that make results unlikely to agree. In uniaxial creep testing, the specimen is subjected to a uniform stress field, while in nanoindentation, the material under the tip experiences a complex triaxial state of stress, with the material undergoing plastic deformation under large strain gradients in the deformed volume. Moreover, in bulk testing, after certain time, the entire specimen deforms uniformly in the steady-state creep regime, and no contribution from the primary transient stage is present

toward the overall strain rate, as opposed to the deformation field found under the indenter tip, which is often represented as an expanding hemispherical volume; as deformation proceeds, the elastic-plastic boundary expands, and the fresh material that enters the plastic zone will exhibit primary creep deformation, while the already deformed volume within the plastic zone will show steady-state creep. Hence, there will be contributions from both primary and steady-state creep stages in indentation creep measurements. By increasing the dwell time, the contribution from primary creep can be minimized and is the prime motivation for selection of a large time scale for creep measurement (> 1 hour). A major issue while conducting creep experiments is therefore thermal drift, which can introduce profound artifacts for such long-time creep tests. The different existing approaches for dealing with thermal drift and suggestions for proper corrections are given in [3]. A fairly extended method for dealing with drift issues is the reference creep method that relies on the continuous measurement of the contact stiffness using dynamic nanoindentation during local long-term creep tests, first introduced by Weihs and Pethica [76]. In this method, a small sinusoidal oscillation is superimposed onto the force, and the contact stiffness S is measured continuously during the test. The contact stiffness S is then used to calculate the true contact area A_c using Eq. (1) assuming that the reduced modulus of the material (E_r) remains constant and is measured early in the test, when the error from thermal drift is negligible. The contact depth h_c is then determined from the contact area through the known area function of the indenter $A_c = f(h_c)$. Finally, the corrected indentation depth (h) is obtained from the contact stiffness (S) and contact depth (h_c) accounting for the elastic sink-in around the indenter using:

$$h = h_c + \varepsilon \frac{P}{S} \quad (13)$$

where P is the applied load and ε is a geometrical constant which depends on the indenter geometry and is equal to 0.75 for a Berkovich indenter [14]. The application of the method used for correcting long-term nanoindentation creep data of UFG-Au obtained at 100 °C [79] is shown in Fig. 8. For instance, following this method, the strain rate sensitivity of UFG-Al [77] and NC-Ni [78] up to 200 °C and UFG-Au [79] up to 400 °C was successfully measured during long-term (up to 2 h) experiments.

An interesting alternative to indentation creep testing is the use of stress relaxation tests, where the indentation depth is kept constant and the hardness allowed to relax with time. The main advantage of this approach is that a more constant volume is interrogated, avoiding the effects of primary creep from fresh material that enters the plastic zone in creep experiments. However, most indentation instruments are inherently load controlled, and performing a displacement-controlled experiment requires a very stable system. For instance, indentation stress relaxation experiments using a flat punch have been used to extract the activation parameters of polycrystalline copper [80].

Finally, some recent creep studies can also be found in the case of micropillar compression, which is free from the problems inherent in indentation creep testing, as the applied stress is constant and uniform. For instance, micropillar compression creep tests were conducted on nanocrystalline nickel at 125 °C, and the results were directly

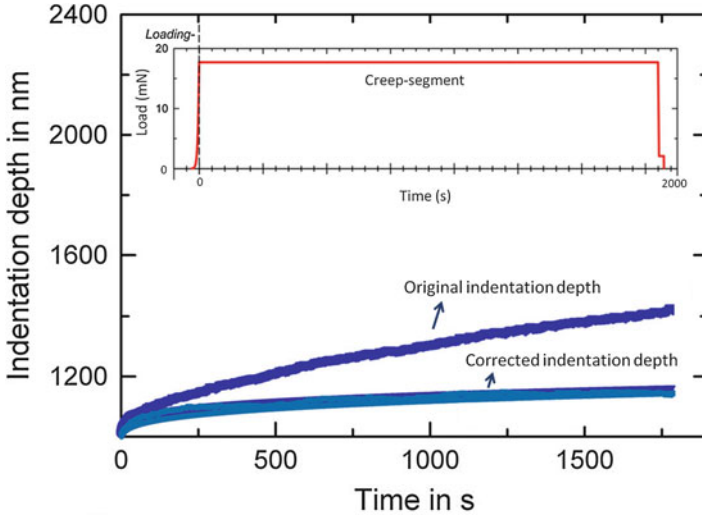


Fig. 8 Long-term nanoindentation creep test on UFG-Au starting at 1000 nm indentation depth obtained at 100 °C, showing original and corrected indentation depth data [79]. The upper curve in the inset represents the applied load which is kept constant during the creep segment. The large effect of thermal drift on the measured displacement of the indenter was corrected using the described method [77]

compared with nanoindentation creep tests [81]. Interestingly, the stress exponent under the two test conditions was found to be almost the same, indicating a similar creep mechanism. However, the strain rate measured by nanoindentation creep was about 100 times faster than that by uniaxial creep, which was attributed to the complex stress state in the plastic zone and to appropriate selection of the indentation constraint factor.

In any case, micropillar compression creep tests are not free from drift effects either, due to the large duration of the tests. Long-time (> 3 h) micropillar compression experiments at constant loads and temperatures ranging from 60 to 160 °C have been reported in pure Sn dendrites and the Sn-Ag₃Sn eutectic constituent in a lead-free Sn-3.5Ag solder alloy [82]. Drift correction for these very long tests at high temperature was particularly challenging, and drift was measured every 30 minutes during the test to account for drift rate changes during the tests. Different stress exponents and activation energies were found for low ($n = 1$, $Q = 36$ kJ/mol) and high stresses ($n = 4$, $Q = 97$ kJ/mol) indicating a transition from a diffusional creep mechanism to a dislocation-climb-based mechanism.

5 Future Direction and Open Problems

Several nanoindentation systems currently exist in the market capable of operating at non-ambient conditions. This chapter presents several success stories studying the effect of temperature on the strength and strain rate effects of a number of relevant

material systems, based on elevated temperature nanoindentation and micropillar compression experiments.

Nevertheless, the reader should be aware that the experimental difficulties mentioned in the introduction for carrying nanomechanical testing at elevated temperatures, such as thermal drift effects and sample-tip interactions, are still far from being completely overcome. More automated approaches to continuously monitor and correct for thermal drift and more stable and longer-lasting new indenter materials are still needed for elevated nanoindentation testing to become a widely used technique. It is also necessary to develop and refine the test methodology, particularly with the advent of high temperature testing in vacuum. So it is required to adapt the ISO 14577 to high temperatures with testing protocols that minimize thermal drift, oxidation and wear of the tip and sample, and time-dependent nonelastic deformation so that contact mechanics can be applied. Moreover, the accuracy of instrument calibration should be validated on reference materials with known properties at the tested temperatures.

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