In situ **small scale mechanical testing under extreme environments**

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The high precision offered by small scale mechanical testing has allowed the relationships between mechanical behavior and specific microstructural features to be determined to an unprecedented degree. However, the behavior of most interest to scientists and engineers is often the materials' behavior under service conditions in an extreme environment: high/low temperatures, high strain rates, hydrogen, or radiation. In this article, we detail the progress made to adapt nanomechanical testing systems and techniques to observe materials behavior *in situ* in extreme environments.

Keywords: nano-indentation; hardness; embrittlement; nuclear materials; scanning electron microscopy (SEM);

Introduction

Small scale mechanical testing (SSMT) at the micro and nano scales has become an integral part of the material science community and is widely used as a tool to evaluate either a material's mechanical property changes due to microstructural alterations or obtain new insight about its physics. It has become rather common to utilize small scale mechanical testing even for engineering applications be it evaluating coatings, irradiated samples, individual grains, welds, joints, etc.

With the wider use to engineering applications, there is a push to test the materials *in operando* conditions, such as temperature, corrosive environment, high strain rate or radiation, to assess the materials' property changes in these relevant environments.

In addition, expanding the testing conditions to these areas allows new insight into a multidimensional environmental space. This paper adumbrates current efforts in using small scale mechanical testing in extreme environments to push the envelope on what is possible using SSMT.

High and Low Temperatures

Non-ambient temperature nanoindentation is probably one of the more developed capabilities in the area of small scale mechanical testing under extreme conditions. As illustrated by Figure 1, several commercially available nanoindentation systems have been brought to the market in the last decades, which are capable of operating at non-ambient temperatures from approximately -140 ºC (*1*) up to 1000 ºC (*2–7*), including *in situ* in the scanning electron microscope (*8–10*). These developments offer new measurement capabilities that enrich our scientific understanding of fundamental material response, including strain rate and temperature effects on deformation mechanisms at the micro and nano-scale (*11*). This allows 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, and corrosion layers, etc. (*12*, *13*).

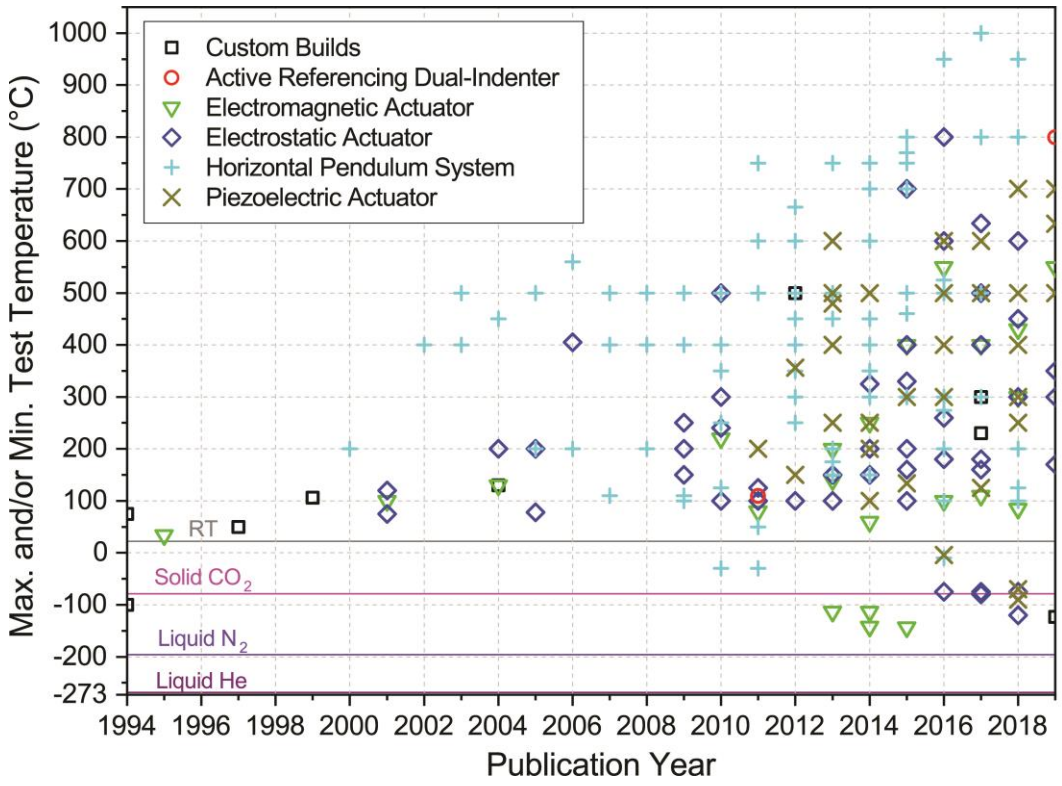

Figure 1 - Maximum and/or minimum published small scale mechanical testing temperatures for various system configurations in recent years (*14*) **- updated from** (*15*)**.**

The major difficulties faced when testing under non-ambient temperature conditions are instrument stability, accounting for thermal gradients, management of thermal drift, and chemical stability, not only of the specimen itself, but also of the indenter tip material. Several recent reviews discuss these issues in detail (*12*, *15*). The key developments that have allowed pushing the temperature limits are the use of individual heating/cooling of the indenter tip and the specimen to ensure thermal equilibrium at the contact (*16*) and the development of more advanced environmental control, either by enclosing the instrument into a purging chamber filled with Ar or by using a high vacuum chamber, as it is naturally found *in situ* within the electron microscope. Measurement temperatures as high as 1000 ºC have been actually reported in the case of stand-alone high temperature, vacuum indentation instruments (*6*, *7*).

However, non-ambient temperature nanoindentation is far from being a well-established technique and further developments are required to increase reliability and reproducibility and to facilitate the widespread use of the technique. More automated approaches to continuously monitor and correct for thermal drift, and more appropriate indenter tip materials are still needed. Diamond is the standard material choice and can be safely used up to around 550 ºC in vacuum. At higher temperatures, diamond starts to oxidize and can also react with carbide-forming metals, like steels, Ti or Co (*17*). As an alternative, cubic boron nitride (cBN) can be used, but its hardness decreases quickly with temperature, it might react with some elements at high temperatures to form more stable borides (*15*), and it is non-conductive, which limits its use in the electron microscope. Nevertheless, even if more stable indenter tip materials are developed, the harsh conditions encountered at the indentation contact will inevitably lead to quick blunting of the tip due to tip-sample reactions and/or deposition of sample material (*18*), limiting the lifetime of the expensive indenter tips to performing just a few indents. Therefore, future developments call for dramatically new approaches, where the high temperature nanoindentation indenters can be produced in mass, such as lithography techniques, and treated as affordable consumables that can be thrown away and exchanged automatically after performing just a few indents.

Alternative geometries may be necessary for extreme temperatures: spherical or flat punch/microcompression geometries to reduce the influence of blunting or using mutual indentation approaches to prevent reactions between dissimilar tip/sample materials.

While high and low temperature capabilities for SSMT are now established and growing, the larger materials science community is still limited to commercial systems restricting testing to ambient conditions. The remaining instrumental difficulties will still need to be addressed in order to make non-ambient temperature testing gain widespread usage. But it is without a doubt that this new tool in the material science toolbox will provide many valuable insights into materials physics and engineering applications in years to come.

High Strain Rates

High strain rates are commonplace in everyday life. High rates can occur simply from the impact of dropping an object onto a hard surface, a ballistic impact from a projectile, or during an automobile crash. In order to allow design structures resistant to these occurrences, it is important to understand the deformation behavior of materials at these high strain rates. Conventionally, this is accomplished at large scales using high speed universal testing frames for dynamic tension testing (*19*) or a Kolsky (Split Hopkinson) bar apparatus (*20*), which allows strain rates from 10^2 to 10^3 s^{-1} to be investigated - Figure 2. Higher rates can be accessed by using a miniaturized Kolsky bar, dynamic/ballistic indentation (*21*) or a pressure-shear plate impact test (*22*). However, in order to study the high strain rate behavior of individual microstructural features, there is considerable interest in developing micromechanical means of high strain rate testing.

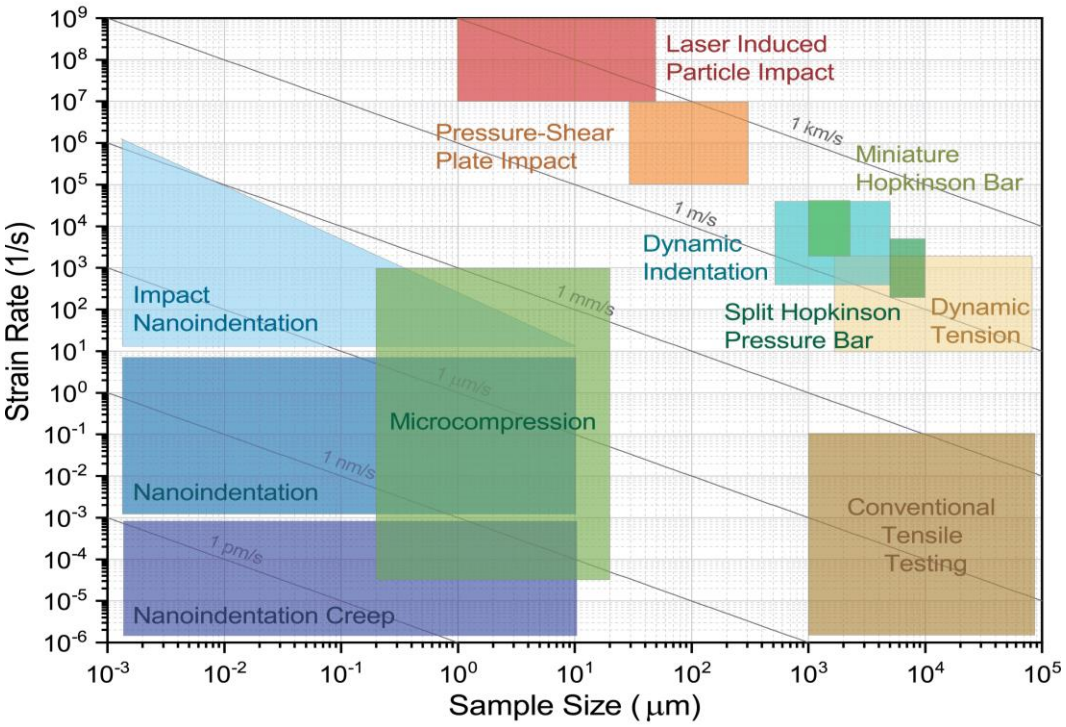

Figure 2 – Schematic strain rate and sample length scale relationships for various mechanical testing techniques (*19–26*)**.**

The primary technique used for small scale investigation is nanoindentation. The 3-sided, Berkovich pyramidal geometry typically used for nanoindentation has variable strain rate testing intrinsically incorporated into it due to the increasing contact area with greater penetration depths. Maintaining a constant strain rate during nanoindentation requires a proportionally increasing displacement or loading rate: $\dot{\varepsilon} = \frac{\dot{h}}{h} \approx \frac{\dot{P}}{2P}$ (*27*). Using this relationship, indentation has been widely used to probe strain rate sensitivity using monotonic (*28*) and jump (*29*) tests. The typical range of strain rates interrogated using these tests is $10^{-3}\,s^{-1}$ until $10^{1}\,s^{-1}$. Lower strain rate regimes can also be probed using indentation creep tests, where the load is kept constant while the slowly increasing penetration depth is measured. Together, this range of strain rates, while quite wide already, is primarily limited by the system's mechanical design and data acquisition/control electronics.

To probe higher strain rates with nanoindentation, a few different systems have been developed. MicroMaterials Ltd. (*23*) has developed a nano-impact system

where the indenter is accelerated to perform a ballistic impact on the surface, achieving very high strain rates (*30*) during the initial penetration and being rapidly decelerated during the indentation. A similar test, using step loading to accelerate the indenter, has been developed by Nanomechanics Inc./KLA Tencor (*24*), allowing hardness measurements at very high strain rate (4000 s^{-1}) by accounting for the dynamic behavior of the system.

Another technique from Alemnis AG (*25*), using high speed piezoelectric transducers, allows rapid displacement and load sensing to be achieved in both indentation and microcompression over a wide range of strain rates (10^{-4} to 10^{3} s^{-1}). This has the benefit over ballistic impacts of producing controlled levels of displacement at the specified rates. For example, a micropillar could be precisely deformed to 2% strain and then sectioned for later investigation by transmission electron microscopy.

Finally, a third strategy is also being pursued to allow investigation of materials properties at the highest rates: 10^{7}-10^{9} s^{-1}. This involves laser-induced particle impact testing (LIPIT), where a ~10-50 μm particle of any material is rapidly accelerated to speeds on the order of 100-1000 m/s by a laser-induced expansion of a thin film (*26*). The particle then impacts locally on a target substrate, producing an indentation or an adhering splat. The incident and rebound velocities are measured by a high speed camera, allowing for the work of indentation to be determined similarly to the ballistic techniques described above (*30*).

Together, all these techniques have greatly extended the range of strain rates accessible by SSMT to ranges similar to, and in some cases higher than, their large scale counterparts. Combining variable strain rate methods with high/low temperature methods from the previous sections creates enormous potential to probe materials' plasticity and activation behavior (*15*), and some authors (*31*) have already begun extending this to the high strain rate regimes, as well. Future efforts are expected to allow routine SSMT at even higher strain rates and bridge the gaps with conventional testing to achieve a holistic understanding of materials deformation over a wide, continuous range of length and time scales.

Hydrogen Environments

Hydrogen has a deleterious effect on the mechanical properties of metals in the form of a severe degradation of strength and toughness, commonly known as hydrogen embrittlement (HE). Traditionally, macro-scale mechanical tests have been used to study the effect of dissolved hydrogen on the mechanical behaviour of metals and alloys. However, the response of a hydrogen-charged macro-scale sample to a mechanical load is affected by discrete spatiotemporal events caused by H interaction with the materials. At this scale, the effects of these discrete events can only be seen as their average aggregate behavior, due to the large number of them, so only the sum of the H effect on mechanical properties in the form of reduced ductility is registered. Certainly, useful information and design parameters can be extracted from macroscale tests; however, a mechanistic understanding of the HE phenomenon requires new *in situ* environmental SSMT tools with higher spatial and temporal resolution to resolve and elucidate individual HE events (*32*).

Once the size of the tested material is reduced, the most challenging task is to retain the H atoms in such small dimensions. Except for special alloys and metals (*33*, *34*), it is impossible to stop hydrogen outgassing from a small sample, which can also have a significant effect on mechanical behavior (*35*). Therefore, small-scale mechanical testing of HE must be combined with *in situ* H charging. As the most versatile method of H charging, electrochemical techniques are used and combined with small scale testing. Recently, commercial electrochemical cells have become available for performing *in situ* electrochemical nanoindentation. The most crucial and challenging aspect of this type of testing is maintaining local control of the electrochemistry at the surface to maintain a constant chemical potential or H charging level during the test. Even though electrochemical H charging might be thought to protect the metal surface through cathodic protection, for sensitive metals like low alloy steels, keeping the surface stable can be challenging. Local variation of the electrical field and pH at the surface can result in unacceptable levels of corrosion and loss of surface integrity for small scale mechanical testing. To combat this problem, Hajilou et al. (*36*) developed a glycerol-based electrolyte to stabilize surface electrochemistry, and

Wang et al. (*37*) have successfully applied this electrolyte to perform *in situ* electrochemical nanoindentation on a very corrosion sensitive TWIP steel. In another novel approach, Kim and Tasan used a miniaturized electrochemical permeation setup inside the SEM where the hydrogen exit site was exposed to electron beam for high resolution SEM and *in situ* nanoindentation (*38*). This approach assures an intact surface for whole period of the testing while raising some concern about the amount of the hydrogen in the tested volume in vicinity of the surface in contact with the vacuum.

Nanoindentation, as the most popular small-scale testing method, has been the main technique used in combination with electrochemical hydrogen charging to study the effect of hydrogen on mechanical properties (*38–40*).

To extend this electrochemical understanding, it can be desirable to avoid the complex stress state underneath a nanoindenter tip by using an alternative micromechanical test geometry: microcompression or microcantilever bending. In these geometries, again, surface stability during the experiment is highly decisive for the validity of the test results. Additionally, the thermal stability and control of thermal drift becomes very important due to the longer experiment durations. Several researchers have performed electrochemical pillar compression tests during electrolytic charging (*41*). However, the most conclusive works so far have been the electrochemical microcantilever bending tests performed in the electrolyte by Hajilou et al.(*42*), where a clear change in the cracking process was directly observable in the presence of the hydrogen.

Recent work has started to move beyond electrochemistry to investigate hydrogen embrittlement. Normally, the other alternative to electrolytic charging with H is charging with H_2 Since the process of H_2 uptake in metals typically requires the slow process of H_2 chemisorption and its thermal dissociation into H on the surface, tests are usually performed in high-pressure H_2 or high pressure combined with elevated temperature to enhance H_2 uptake. Either of these is far more technically demanding for *in situ* testing than electrochemical charging. Thus, alternative approaches for enhancing H charging are very attractive.

Takahashi et al. studied the fracture behavior of bi-crystalline nickel-aluminide micro-cantilevers inside a high-voltage environmental transmission electron microscope (*43*). The tests were conducted either in vacuum or N_2-20 vol%H_2 gas (H_2 partial pressure of 1 kPa). They used the high acceleration voltage of 1 MeV to facilitate the dissociation and ionization of gas molecules. Recently, Wan et al. (*44*) used low pressure plasma to enhance the H uptake from H_2 for *in situ* mechanical testing in an environmental scanning electron microscope (ESEM). Deng et al. (*45*) and Rogne et al. (*46*) used the high reactivity of FeAl and Fe_3Al intermetallic alloys with water vapour, which produces H uptake through surface reduction of the water, to perform microcantilever bending tests *in situ* in an ESEM - Figure 3. They were able to observe the H enhanced crack propagation process in real time. Testing in the SEM typically allows high resolution visualization for positioning prior to environmental exposure, as well as precise measurement of the crack mouth opening and/or use of digital image correlation for local strain measurement. However, upon increasing the chamber pressure in an ESEM, the imaging resolution is reduced (Figure 3), precluding high resolution, local visualization of the deformation processes, but enabling the acquisition of local mechanical behavior in the selected environment.

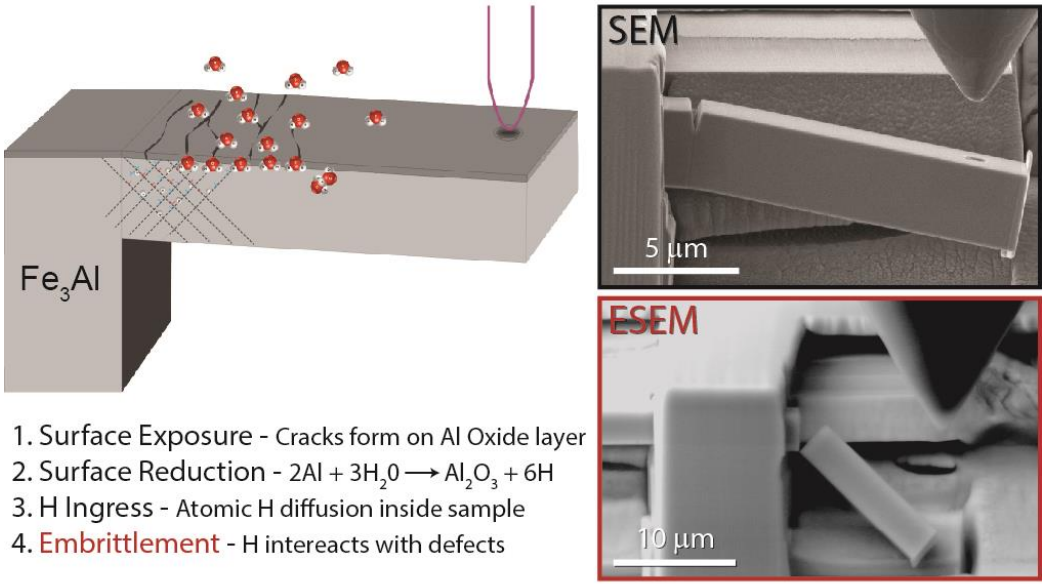

Figure 3 – Schematic showing hydrogen ingress and embrittlement of a FeAl sample with *in situ* secondary electron micrographs showing the relatively tough behavior in vacuum in contrast with the hydrogen embrittled behavior in the ESEM (*45*)**.**

In situ SSMT in combination with electrochemical H charging is a powerful tool for investigating the underlying mechanisms of hydrogen embrittlement. In the future, we expect to see the increasing application of these environmental techniques to address the HE problem. Future efforts are expected to combine local hydrogen measurements with small scale mechanical testing using special, functionalized nanoindenter tips.

Radiation

Nuclear environments are among the most challenging extreme environments for materials. Materials are exposed to high temperatures, corrosion, and radiation simultaneously for extended periods of time, sometimes even for decades (*47*, *48*). Associated with these environments are materials degradation mechanisms that can lead to mechanical failure, such as radiation induced embrittlement, increase in yield strength, irradiation induced creep, and radiation induced stress corrosion cracking to name a few. Small scale mechanical testing has been increasingly utilized on materials exposed to these environments due to the fact

that the small volumes of radioactive material allow researchers to increase the safety of the worker, to access specific regions of interest, to target specific phenomena and separate different radiation effects, and to directly assess mechanical property degradation using ion beam irradiations instead of neutrons. This final advantage has gained popularity due to its specificity and higher dose rates allowing shorter turnaround times (*49*). Numerous small scale mechanical testing techniques are currently deployed on irradiated model or engineering materials including nanoindentation (*50–52*), microcompression (*53–55*), and micro tensile testing (*56–58*), and these have been employed both at room temperature and at elevated temperatures, mostly *ex situ* but also during irradiation - Figure 4.

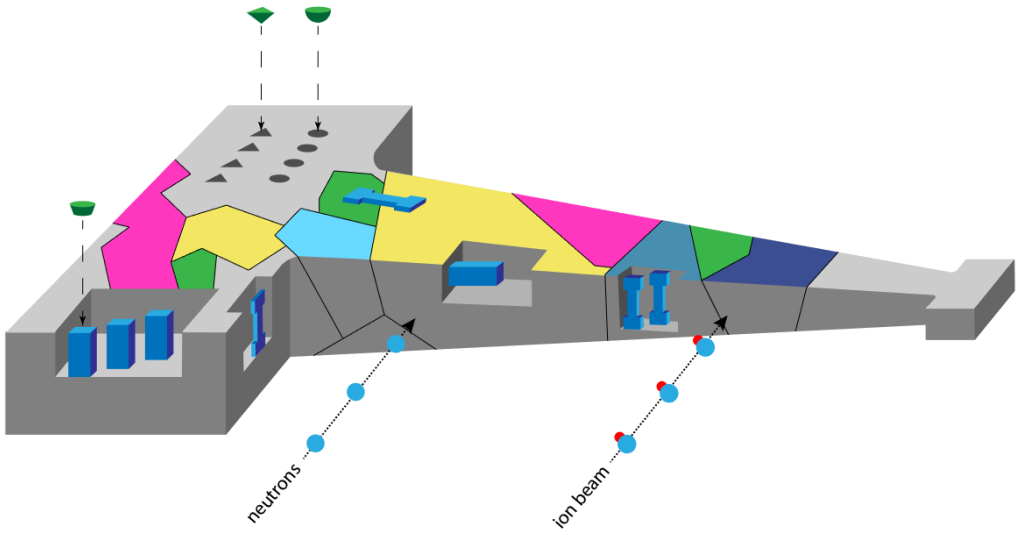

Figure 4 – Schematic of radiation penetration and specimen geometry locations by type.

The main aim of this area of research is to assess the materials' property change at small scales after/during irradiation and then to relate this to bulk property changes. While assessing the mechanical effects of radiation on materials on a phenomenological level allows the community to learn about the importance of specific effects, it is the ultimate goal to translate the small scale results into bulk property assessments. This goal is also the main challenge, since scaling effects, dose gradients, implantation effects, surface effects, and microstructure effects all need to be addressed in order to accurately infer bulk properties. Recent studies

have focused on indentation of neutron and ion beam irradiated materials (*59–61*), and this has fostered a more general understanding of scaling effects associated with specific defects in the material. Indentation lends itself well to statistical testing and therefore allows investigation of numerous effects, such as dose gradients in ion beam irradiated materials. In contrast, microcompression testing allows the assessment of phenomenological materials' failures, such as highly localized slip from radiation-induced creep. Recent work by K. Tai et al. (*62*) undertook the extremely difficult task of performing microcompression testing *in situ* during ion beam irradiation to investigate radiation-induced creep as a function of dose rate, while other investigator, e.g. Reichardt et al (*63*), have addressed localized slip and therefore sudden failure in irradiated materials.

Most studies have previously focused on the stress-related aspects of irradiated materials deformation, but recently strain-to-failure and ductility has been investigated utilizing micro-tensile testing and micro-cantilever testing (*64*, *65*). It was found that reduced strain-to-failure can be accurately assessed using these approaches, and further work in this area is greatly needed. Recently, grain boundary failure due to transmutation products agglomerating at grain boundaries has gained interest in the community, but so far only a very limited number of studies have attempted to investigate grain boundary failure using SSMT techniques (*66*). This exciting field of research is still currently wide open.

Despite the recent efforts, it is still vital to find a way to transfer the small scale mechanical properties measured and phenomena observed to the macroscopic world, and this can only be achieved by pairing small scale investigations with crystal plasticity modeling. Despite the challenges mentioned above, utilizing small scale mechanical testing for irradiated materials research will be a key component to fully understanding the materials degradation phenomena under irradiation and enabling the prediction and prevention of materials' failures in service.

Summary

We have highlighted the numerous advances made towards deploying small scale mechanical testing of materials *in situ* in extreme environments. Significant progress has been made in the community, but in our opinion, the community is still only in the inception phase. New, refined, and synergistic approaches in coming years are expected to provide fundamental insight into materials behavior at extreme conditions.

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Figure Captions

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from composites to light and high temperature metallic alloys, with the aim of understanding deformation mechanisms and informing microstructurally based multiscale materials models.

Peter Hosemann is the leader of the nuclear materials group at the University of California at Berkeley's nuclear engineering department and currently department chair. He obtained his PhD degree from the Montanuniversität Leoben in Austria in 2008 while performing his research at Los Alamos National Laboratory. His research focuses on materials evaluation in extreme environments with a focus on nuclear environments and spearheads small scale mechanical testing on nuclear materials.

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