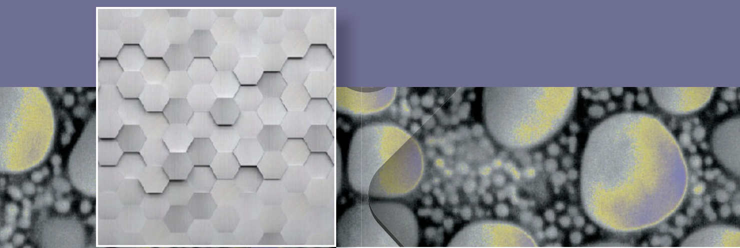
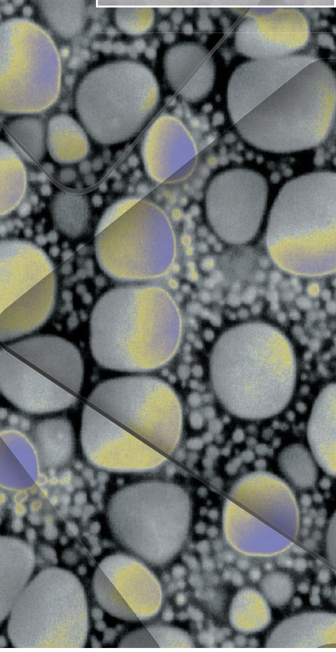

High-temperature materials for structural applications: New perspectives on high-entropy alloys, bulk metallic glasses, and nanomaterials

E-Wen Huang and Peter K. Liaw, Guest Editors

Tougher, lighter, and more formable and machinable metals for broader ranges of applications at higher temperatures are needed now more than ever. High-performance computing, high-resolution microscopy, and advanced spectroscopy methods, including neutrons and synchrotron x-rays, together with advances in metallurgy and metal mixology, reveal the potential of multicomponent advanced metals, such as multicomponent bulk metallic glasses and advanced high-entropy alloys. The development of new experimental approaches relates bulk properties and voxel-associated optimized properties throughout structures with high resolution. The correlations from *in situ* measurements greatly improve crystal plasticity-based models. This issue of *MRS Bulletin* overviews recent progress in the field, and this article highlights the importance of these new perspectives. The latest progress and directions in the science and technology for prospective high-temperature metals for structural applications are reported.

Introduction

Advances in engines and other applications with high-temperature requirements have been constrained by our limited ability to garner heat losses and improve performance metrics by optimization. In addition, materials researchers are pursuing advanced metallic materials to answer the challenging strength–ductility dilemma. Due to the advantages of their higher toughness and predictable fracture behavior, metals, such as aluminum and titanium alloys, have been used in aerospace applications.[1] Recent elevated-temperature alloy developments have raised the bar on high-temperature stability through advances in precipitation hardening and microstructure design.

Besides the conventional perspectives for high-temperature metals for structural applications,[2–6] new demands for emergent businesses and industries have arisen. For example, while new power technologies have surpassed coal in addressing global warming,[7–9] the development of high-temperature alloys for structural applications directly improves thermal efficiency and reduces greenhouse gas emissions, such as those alloys used in ultra-supercritical coal plants that utilize supercritical fluids and reduce air pollution.[10–12] Because increased operating pressures and temperatures are needed, the US Department of Energy Fossil Energy Program requires an increase in the steam temperature for ultra-supercritical steam turbines[13] of the coal plants. The increasing need for boilers and other high-temperature metals demands the development of superalloys for elevated-temperature applications.[14]

New high-temperature materials are currently approaching 70% or higher of their absolute melting temperatures, which is the upper limit to resist creep and vacancy formation. In structural applications, materials capable of performing under these temperatures, as defined by their percentage of their absolute melting temperatures, are designated “superalloys.” Advances in combustion performance, structural performance, or other performance metrics for a variety of applications are hampered by the lack of availability of alloys that are stable at high temperatures. Although researchers have pushed the envelope on these new alloys, and the world needs to further push for the next generation of alloys.

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New perspectives

The Gartner Hype Cycle,[15] which helps industries and companies predict the maturity and adoption of technologies and applications, reports several new applications that need structural materials for higher operational temperatures, such as additive manufacturing and the Unmanned Aircraft System. In general, metallic systems in aerospace applications need to balance specific strength, creep resistance, and environmental stability.[16] The Leading Edge Aviation Propulsion (LEAP) is the first jet engine that includes three-dimensional (3D) printed fuel nozzles of a Co-Cr superalloy.[17,18] On June 14, 2017, the Airbus corporation delivered the first LEAP-1A-powered A320neo aircraft to EasyJet.[19] Compared to its predecessor, the A320neo saves up to 15% in fuel and reduces up to 15% CO_2 emissions.[20] Because of the new integrated unibody geometry of the LEAP-1A, the noise of A320neo on takeoff and landing decreased by approximately 50%.

Today, a science-based systems-engineering approach is necessary to achieve such design objectives for advanced high-temperature materials. A hierarchy of computational and experimental tools needs to be integrated for the design of prototype alloys.[21] As an example, in 2019, ANSYS, which develops and markets the engineering simulation software, acquired Granta Design,[22] co-founded by Ashby and Cebon, a provider of the materials information technology using professional big data to develop such hierarchical computational tools to integrate materials properties into spatially resolved system simulations.

Why metal mixology?

In 2014, Gludovatz et al. reported that the CoCrFeMnNi high-entropy alloy (HEA),[24] which contain late transition metals such as Fe, Ni, Co, and Cu, was one of the best fracture-resistant materials for cryogenic applications.[24] Senkov et al. later designed refractory HEAs, which primarily consist of five or more refractory elements, and showed superior properties for high-temperature applications.[25] The high-temperature strengths of several refractory HEAs and conventional stainless steel, aluminum, titanium, and nickel-based alloys are compared in **Figure 1**. Most of the refractory HEAs are significantly higher in strength (**Table I**). Senkov et al. suggest the solid solution strengthens the metallic systems.[25]

Continued improvements in yield strength (Figure 1) require a systematic approach to facilitate age-hardening effects for HEAs. Age hardening is the most widely used means for strengthening metal alloys. This strengthening mechanism relies more on the precipitation of desirable phases than on hardening from uniform dispersions. Strain fields introduced by the lattice mismatch between the precipitates and homogeneous matrix pin dislocations and act as a barrier to dislocation motion.[41]

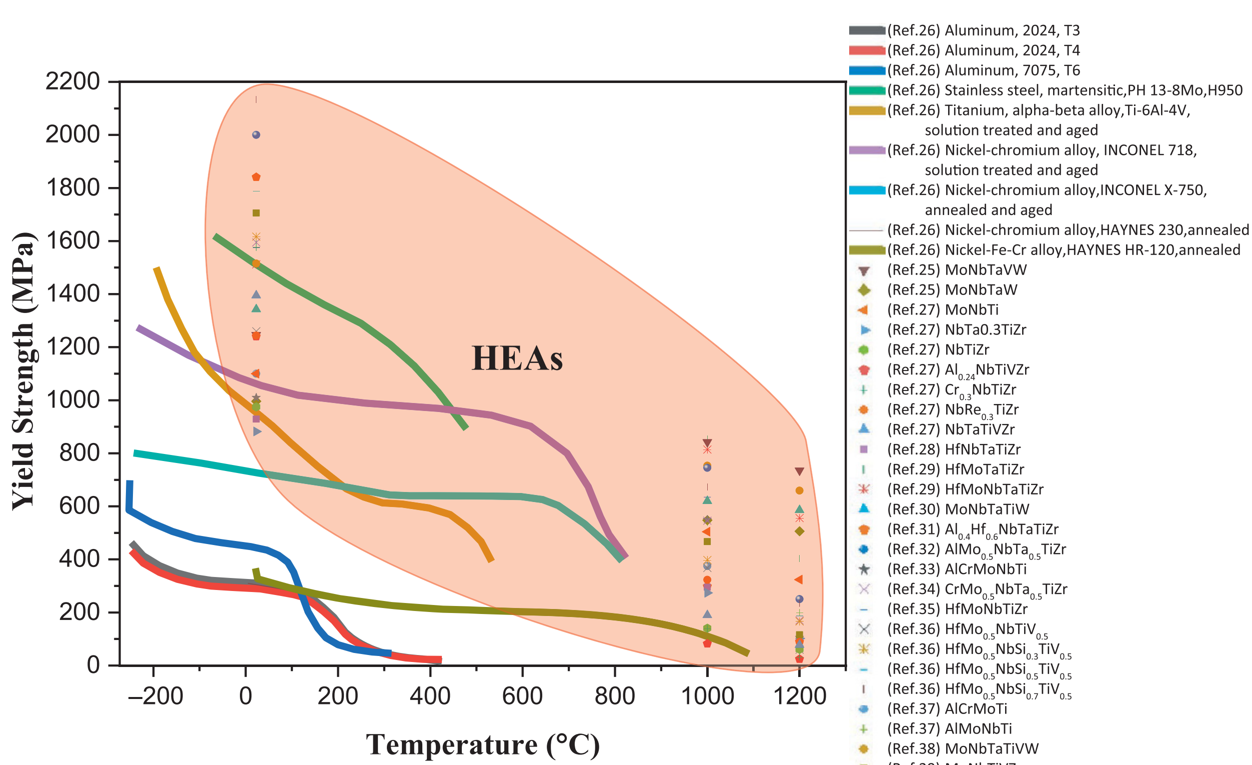

Figure 1. Yield strength ($\sigma_{0.2}$) of several refractory high-entropy alloys[25–39] (HEAs) and conventional stainless steel, aluminum, titanium, and nickel-based alloys. As potential high-temperature materials for structural applications, the HEAs show greater strength. Thermodynamic and mechanical properties for these single-phase and multiphase HEAs are summarized in Reference 40.

In their article in this issue, Cao et al. report[42] the application of precipitation strengthening in HEAs. HEAs with multiple principal elements[23,43] have shown great potential as a new class of metal mixology for high-temperature applications.[25,44] In their article, Inoue et al.[45] describe the latest progress in metallic alloys with elevated-temperature resistance in the form of glassy/amorphous structures, such as bulk metallic glass materials, rather than a crystalline structure. By introducing specific crystalline phases in an amorphous matrix (**Figure 2**),[46] BMG-composite materials demonstrate improved plasticity and toughness when compared to monolithic amorphous materials.[47–49]

In situ high-temperature measurements using neutrons and synchrotron x-rays

Spallation neutron sources have the advantage of illuminating multiple diffractions for texture-sensitive studies of materials as well as nano-precipitate diffraction. In particular, a main advantage of the *in situ* neutron environment is the possibility of investigating the evolution of microstructures since identical specimens are monitored during the entire experimental time and are subjected to the changes of the control parameters. Moreover, most of the neutron-diffraction instruments are equipped with the strobing software, such as the VULCAN Data Reduction and Interactive Visualization softwarE (VDRIVE)[50] of VULCAN at the Spallation Neutron Source (SNS) of Oak Ridge National Laboratory (ORNL), which can reduce the data at the end of each test and is much more useful. The strobing system is a continuously run software utilizing event-based data acquisition where each neutron carries a time stamp. In this case, diffraction data can be collected continuously and binned later according to the desired time scale. Besides studying conventional materials, advanced *in situ* neutron diffraction would be useful in studying HEAs.[51–53]

TAKUMI,[54] a materials engineering diffractometer located in Japan at the Japan Proton Accelerator Research Complex, is another spallation-neutron-source diffractometer that is capable of various *in situ* environments, including elevated-temperature measurements. TAKUMI's orientation-dependent data-acquisition

Table I. Yield strength (YS) values for alloys listed in Figure 1.

Symbols/Materials	YS at 23°C (MPa)	YS at 1000°C (MPa)	YS at 1200°C (MPa)	Ref.
MoNbTaVW	1246	842	735	25
MoNbTaW	996	548	506	25
MoNbTi	1100	504	324	27
$NbTa_{0.3}TiZr$	882	274	102	27
NbTiZr	975	141	61	27
$Al_{0.24}NbTiVZr$	1240	82	24	27
$Cr_{0.3}NbTiZr$	1576	139	32	27
$NbRe_{0.3}TiZr$	1244	323	89	27
NbTaTiVZr	1395	190	78	27
HfNbTaTiZr	929	295	92	28
HfMoTaTiZr	1600	855	404	29
HfMoNbTaTiZr	1512	814	556	29
MoNbTaTiW	1343	620	586	30
$Al_{0.4}Hf_{0.6}NbTaTiZr$	1841	298	89	31
$AlMo_{0.5}NbTa_{0.5}TiZr$	2000	745	250	32
AlCrMoNbTi	1010	550	105	33
$CrMo_{0.5}NbTa_{0.5}TiZr$	1595	546	170	34
HfMoNbTiZr	1575	635	187	35
$HfMo_{0.5}NbTiV_{0.5}$	1260	368	60	36
$HfMo_{0.5}NbSi_{0.3}TiV_{0.5}$	1617	398	166	36
$HfMo_{0.5}NbSi_{0.5}TiV_{0.5}$	1787	614	188	36
$HfMo_{0.5}NbSi_{0.7}TiV_{0.5}$	2134	673	235	36
AlCrMoTi	1100	375	100	37
AlMoNbTi	1100	540	200	37
MoNbTaTiVW	1515	753	659	38
MoNbTiVZr	1706	467	116	39

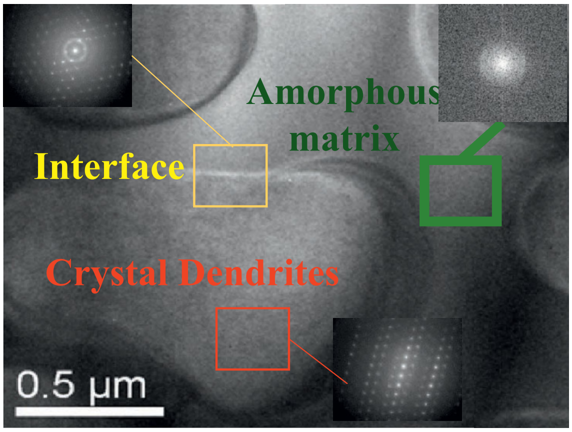

Figure 2. $Zr_{58.5}Ti_{14.3}Nb_{5.2}Cu_{6.1}Ni_{4.9}Be_{11}$ bulk metallic glass matrix composite. (Insets) Diffraction patterns corresponding to the respective boxed areas.

function can couple various detectors (**Figure 3**a). Figure 3b shows the high-temperature heating setup for TAKUMI for *in situ* loading. Figure 3c shows the spatially resolved temperature mapping for the sample and the environment; meanwhile, additional thermocouples are attached on the sample to measure the temperatures.

Besides VULCAN and TAKUMI, the Spectrometer for Materials Research at Temperature and Stress at the Los Alamos Neutron Science Center[55] in the United States, and ENGIN-X[56] of the ISIS at the Rutherford Appleton Laboratory in the United Kingdom are also capable of similar types of measurements. For a reactor-based neutron diffractometer, the Residual Stress Instrument (RSI) installed at the High-Flux Advanced Neutron Application Reactor (HANARO) of the Korea Atomic Energy Research Institute (KAERI), is a wide-angle neutron diffractometer optimized for strain–stress scanning and deformation behavior studies for polycrystalline metals and alloys[57] (**Figure 4**).

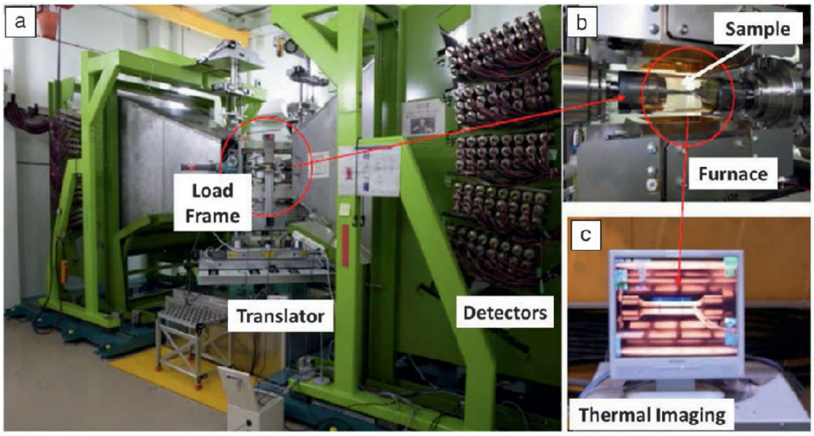

Figure 3. (a) The TAKUMI instrument (Republic of Korea); (b) load frame equipped with a high-temperature chamber; and (c) temperature-mapping monitor.[54]

Similarly, synchrotron x-rays can also illuminate the microstructure with high penetration. For *in situ* heating experiments, the Taiwan Photon Source (TPS) in Taiwan has two stations.[58] The x-ray nano-diffraction (TPS-21A) provides spatially resolved mapping for elements, phases, orientation, residual strain–stress, and dislocations at a resolution of 100 × 100 × 50 nm.[59] For high-temperature heating, temporally coherent x-ray diffraction (TPS-09A) can heat the samples up to almost 1200 K.[60] In addition to studying conventional alloys, advanced synchrotron x-ray diffraction can be effective in investigating HEAs.[61,62]

Several neutron and synchrotron x-ray instruments have been reviewed elsewhere.[63] Based on the different perspectives of heating effects, various *in situ* measurements can be applied. Crystallographic slip and associated dislocation activities during deformation at different temperatures can be revealed using these instruments. By refining the diffraction profiles with the general structure analysis software, peak components enable the investigation of dislocations and the average distance between patterned-dislocation structures,[64] which will be useful for probing the plastic behavior of HEAs.

Besides advancements in high-temperature experimental environments, Agnew et al. and Tome et al.'s self-consistent model couples bulk properties and diffraction data, enabling crystal-plasticity-based investigations.[65] A self-consistent model compares the internal strains within a polycrystalline alloy measured during deformation testing by *in situ* neutron diffraction. Using Tome's self-consistent simulation code, information about the operation of slip and mechanical twinning modes as a function of strain can be obtained. In their article in this issue, Wang et al. report[66] modeling twinning, detwinning, and dynamic recrystallization of magnesium alloys.

In the age of artificial intelligence and machine learning

A group from Rolls-Royce and the University of Cambridge recently reported the successful use of a neural network to design high-temperature molybdenum-based HEAs.[67] Their artificial intelligence (AI) tool evaluated the cost, phase stability, precipitate content, yield stress, and hardness simultaneously, and their experimental results validated that the predicted alloy fulfills the computational predictions. Tshitoyan et al. report a machine-learning algorithm that successfully identifies and predicts emerging fields within the broad materials field without guidance from humans.[68] Their material informatics system integrates high-throughput experiments, computations, and data-driven methods from 3.3 million published abstracts, which enable new materials design. The AI technology thus effectively saves time when compared to traditional methods used by humans. For materials development, the Materials Genome Initiative has shown significant progress in the computational simulation and modeling of materials.[69]

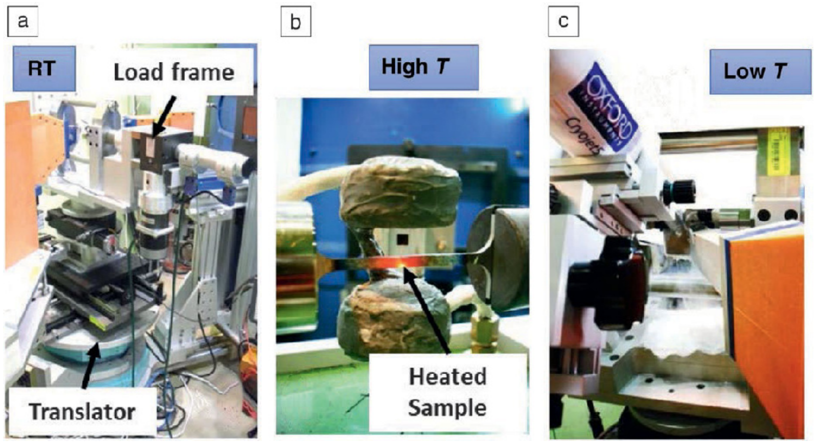

Figure 4. Three different environmental temperature setups for measuring the residual stress in the High-Flux Advanced Neutron Application Reactor (HANARO): (a) room-temperature, (b) high-temperature, and (c) low-temperature *in situ* measurements. Note: *T*, temperature.

Although text mining does demonstrate superior capability for materials discovery,[68] most data are from traditional experiments. Hence, the data domain is limited by conventional measurements, such as microstructure images. Meanwhile, the types of data collected from various instruments and with different resolutions, such as neutrons and high-energy synchrotron x-rays, are different from what is collected from traditional routine measurements. An advanced photon source can penetrate bulk materials, which can yield complementary ensemble average with a much greater sample size.

With the advances in synchrotron x-ray and neutron facilities, the neutron and synchrotron x-ray diffractometers are being equipped with load frames, a furnace, and other various sample environments. Hence, real-time *in situ* diffraction measurements are possible using these facilities. For example, VULCAN[70–72] is dedicated to studying the mechanical behavior of materials at the SNS of ORNL. The special feature of this diverse sample environment bridges traditional mechanical tests and the underlying microstructure characterizations. In their article in this issue, An et al.[73] demonstrate several unique examples of *in situ* measurements, which now are routine operations of VULCAN.

Extension of the AI technology to correlate *in situ* advanced light-source results, traditional protocols, and high-throughput examinations is expected in future elevated-temperature materials development, including HEAs.[74]

Further advances and opportunities

New metallurgical routes facilitating nanotechnology to explore the new territory of high specific strength for elevated-temperature metals are expected for structural applications. Researchers fabricated bulk ultrafine-grained (UFG)/nanocrystalline metals via slow cooling by instilling a continuous nucleation and growth control mechanism during slow cooling.[75] The bulk UFG/nanocrystalline metal with nanoparticles (bulk Cu ingots with WC nanoparticles) also reveals unprecedented thermal stability up to 1023 K, which is 75% of the melting temperature of Cu. Humphry-Baker et al. established a mechanochemical reaction between solid phases for 3D printing high-specific strength metals to reduce specific strengths.[76] Meanwhile, Lin et al. strengthen metals using UFG/nanocrystalline phases, unlike conventional microstructure-refinement methods.[77]

Summary

This issue explores the state of the latest metal mixology for high-temperature applications. The microstructural degradation and failure mechanisms of the materials subjected to heating are reported for future applications associated with complex mechanical and thermal-loading conditions during service. The objective of this issue is to review modern tools, integrated with focused materials, to report innovative metallic systems, mainly HEAs for high-temperature applications. The issue explores advanced metallic systems of HEAs and BMGs, modern characterization, and computational tools for the development of the high-temperature metals, and creep behavior and thermal stability of some HEAs. Specifically, at higher temperatures, vacancy-induced behavior becomes critical, especially for HEAS.[78] Lin et al.[79] review some of the latest results of the creep for HEAS for this emergent field via nanoindentation technology. The content also includes alloy design, microstructure engineering, process development, machine-learning, high-throughput technology, and mechanistic modeling for deformation.

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