Online at: https://researchtrendsjournal.com ISSN No: 2584-282X

INTERNATIONAL JOURNAL OF TRENDS IN EMERGING RESEARCH AND DEVELOPMENT

Volume 2; Issue 2; 2024; Page No. 92-96

Received: 08-01-2024
Accepted: 20-02-2024

To study the impact of characterizing the qualities of materials in the in the most extreme environments

[1]CH Gangadhar and [2]Dr. Nitin Kumar Singh

[1]Research Scholar, Department of Physics, Monad University, Hapur, Uttar Pradesh, India
[2]Professor, Department of Physics, Monad University, Hapur, Uttar Pradesh, India

DOI: https://doi.org/10.5281/zenodo.12793392

Corresponding Author: CH Gangadhar

Abstract

The requirement for material systems to function in the harshest conditions seen in space exploration, energy generation, and propulsion systems is always growing. We need a range of methods to both sustain extreme conditions in the lab and then probe the properties of the materials over a number of length and time scales in order to effectively design materials that will reliably function in extreme settings. In this paper, we analyze the most advanced experimental setups for material testing in harsh settings and point out the drawbacks of these methods. Our three main areas of interest are chemically hostile conditions, harsh mechanical testing, and extreme temperatures. We pinpoint six avenues for instrument and technique development within these domains that have the potential to significantly influence the advancement of knowledge and creation of next-generation materials for harsh environments.

Keywords: Space, hostile conditions, harsh environments, extreme temperatures

Introduction

Humanity's natural need for speed6, energy and exploration calls for materials that can withstand the harshest conditions. Systems may encounter severe cold (~3 K in outer space), intense heat (~1723 K upon reentry into Earth's atmosphere), harsh radiation (high-energy radiation and fast heavy ions), and enormous stresses (ballistic impacts from fast-moving particles) during interstellar travel. Concentrated solar power, which uses the Sun's beams to concentrate and store heat in molten salts at temperatures as high as 973 K, is one method of using solar energy on Earth.7. Nuclear fusion4 technologies function at even greater temperatures, and next-generation nuclear fission reactors, with designs that can reach up to ~1773 K,9 could pump corrosive molten salts as fuel and/or coolants at high temperatures to boost efficiency. For next-generation spacecraft to be propelled to Mars3 and beyond, related nuclear thermal propulsion systems2 need temperatures as high as around 3100 K. Hypersonic aircraft will encounter cutting-edge temperatures of up to about 3000 K6 (at Mach 8) even on Earth, necessitating the use of materials that can withstand high stresses and oxidation to avoid deformation during flight. These are but a few illustrations of the general requirement for next-generation materials to function in the harsh conditions necessary to advance civilization into the future.

A comprehensive comprehension of the following is necessary for the effective design of material systems to function in harsh environments: Three main aspects of them are: (1) their kinetics and transport qualities; (2) their thermodynamics and fundamental properties; and (3) their developing microstructure over various time and length ranges. Until recently, materials design relied on Edisonian trial-and-error methods. It has only been in recent times that computational techniques have been heavily utilized to propel material research. Innovative material systems on demand have been successfully designed in the past using integrated computational materials engineering (ICME) techniques. The ferrium M54 steel11 was similarly designed for landing gears in aircraft, whereas the ferrium C64 steel10 was primarily designed using computational techniques for rotorcraft drive trains in helicopters. In order to suit the unique requirements of their application, these steels, their composition, and their manufacturing parameters were mostly predetermined via computational methods. So why can't we use comparable on-demand techniques to build material systems for the harsh environments of nuclear fusion or hypersonic platforms today? The foundational knowledge gained from years of testing and the creation of databases of self-consistent material properties underpinned the accomplishments of these earlier endeavors. For materials subjected to more extreme conditions-especially when paired with extreme conditions-comparable databases have not yet been developed. In this paper, we outline the state-of-the-art experimental capabilities available today for testing materials in harsh environments and point out the drawbacks of these methods. While there are many distinct "extremes" that fall under this broad category, we concentrate on three crucial regions that are comparatively common to raise issues with material performance: (1) High temperatures, (2) severe mechanical testing, and (3) conditions that are chemically unfriendly. Our goal is to pinpoint the gaps in capability that, if filled, will make it possible to gather all relevant material data in order to aid in the creation of next-generation materials that can withstand the harshest conditions.

2. Statement of the problem

Here, extreme temperatures are those that are higher than 2000 K and lower than 77 K. When combined with laser heating, levitation may reach temperatures as high as 4,000 K, and when combined with materials diagnostics, it can extract important thermochemical and thermophysical parameters. In addition, at lower temperatures (~1600–2873 K), high-temperature imaging and thermal characteristics can be obtained by microscopy and calorimetry. Cryogenic experiments, on the other hand, slow down atoms and can be used in conjunction with other imaging modalities and microscopy to extract basic principles associated with physical events.

3. Objectives of the study

1. Investigate diverse materials' responses to extreme conditions, analyzing molecular changes and chemical bonding.
2. Uncover phase transitions, reactions, and structural shifts induced by high temperatures and pressures.

4. Materilas and Methods

Controlling the test temperature alone is not enough to ensure the performance of mechanical testing at ultrahigh and ultralow temperatures. Other unrelated aspects that must be taken into account include the specimen design, the gripping system, the test system itself, and even the underlying strain measurements. Due to these difficulties, the maximum test temperature for mechanical testing equipment used in commerce has been set at approximately 1800 K up to now. This is lower than many service temperatures, such 2273 K for hypersonic platforms.

Customized systems that enable mechanical behavior studies of ultrahigh-temperature ceramics (UHTCs) and refractory alloys using ohmic heating of conducting samples and pyrometer-based temperature control have been devised to explore materials at 2273 K and higher. Tensile tests on tungsten sheet with a thickness of 1 mm revealed that at 2073 K, the tensile and yield strengths were approximately 105 MPa and 62 MPa, respectively, but at 2773 K140, they were approximately 21 MPa and 16 MPa, respectively. The samples had a gage length of 10 mm, and machined shoulders were utilized as markers for noncontact strain measurement to guarantee temperature uniformity across the test space. Induction heating is another method that may be used for high-temperature testing and is appropriate for both conductive and nonconductive materials. Four-point bending tests of several zirconium-diboride-based UHTCs were performed up to 2573 K using induction heating of a graphite hot zone in an environmental chamber.141, 142, 143, 144 These showed that the flexure strength of pure ZrB2-which was around 200 MPa (1673–2573 K, in Ar)141-was raised to approximately 460 MPa at 1673 K and ~360 MPa at 2573 K in ZrB2–10 vol% ZrC ceramic.142 The material creating a eutectic with the graphite grips is the reason for the temperature limit in these flexure strength measurements. More recently, it was reported that the four-point flexural strength of single-phase high-entropy carbide ceramics was around 90 MPa at 2573 K, while at RT, it was approximately 420 MPa. Additionally, high-temperature tensile testing of ZrB2-20 vol% SiC in air up to 1973 K was made possible by an electric resistance furnace. In an oxidative environment, 145 ductile fracture behavior and around 0.5% plastic strain were noted at 1973 K.

5. Results and Discussion

In most common use conditions, most materials are inherently distant from thermodynamic equilibrium, and in some severe environments, this distance can increase even further. Microstructural evolution with negative effects is driven by chemical reactions between the material and its surroundings. Materials that can endure oxidative and other corrosive conditions over extended periods of time are needed at high temperatures. Despite the fact that materials are designed to withstand or resist these changes, frequently by naturally developing or depositing barrier layers between the material and environment, our ability to operate in more hostile conditions is being tested. Examples of areas that will be addressed in this section from the perspective of how to accurately measure material degradation are: (1) gaseous corrosion with a focus on space missions, such as hypersonic flight with reentry vehicles and venus atmospheric corrosion; (2) molten salt corrosion with an emphasis on applications in concentrated solar power (CSP) plants; and (3) chemical implications at low temperature. The testing status as of right now is discussed for these three application scenarios, which appear to require distinct testing regimens for temperature and environment.

5.1 Molten salt corrosion

Specific parameters, including as high thermal conductivity and heat capacity, low viscosity, low melting, and high boiling temperatures, must be met by heat-transfer fluids (HTFs) and thermal energy storage (TES) materials. They also need to be easily obtainable and have a minimal environmental impact. Sludge-based nitrate salts are now used for TES and as HTFs, however their thermal stability restricts the operating temperature to approximately 838 K.

The next generation of CSP plants will run at temperatures higher than 973 K since the energy-conversion efficiency of CSP systems rises with operating temperature. One way to achieve this is by using innovative HTF and TES materials, including molten eutectic chloride salts (NaCl-KCl-MgCl2), which ensure both great thermal stability and a low freezing point. On the other hand, the corrosion processes from these media require extensive characterisation methods and long-term testing. An illustration of a cutting-edge testing configuration.185 High-temperature-resistant glassy carbon or alumina are typically utilized for the crucibles.185,186 PtRh10-Pt thermocouples within the molten salt regulate the temperature during the 500-hour studies conducted under flowing Ar environment. There are currently no commercial structural alloys that can endure the extreme temperatures and long-term testing that this experimental setup can achieve—conditions that are higher than those of genuine CSP plants. For instance, it is evident that stainless steels 310 and 316, or Ni-base alloys as Haynes 230, IN 600, or IN 625, are unsuitable for these settings due to significant material losses that range from 200 to 1700 µm/year and considerable Cr leaching at 973 K. This may be considered ironic behavior because most oxide-forming situations use high-Cr alloys in corrosion-resistant alloys because the underlying alloy is shielded from further deterioration by Cr2O3 nanoscopic coatings. Research efforts to develop and engineer novel materials that can tolerate similar settings have been spurred by similar responses shown in FLiNaK and FLiBe chemistries that are being explored for nuclear reactor designs that cool using molten salt.

Remarkably, few groups have attempted to study the combined impact of mechanical stress and chemical attack under CSP conditions since the groundbreaking work of Atmani and Rameau in 1980, who investigated stress-corrosion cracking (SCC) of stainless steel in molten NaCl-CaCl2 at 840 K utilizing a specially designed tensile test apparatus.194 The predicted low-stress conditions of CSP or molten salt reactor designs contribute to this study gap, yet localized attack and grain-boundary embrittlement could very well be expected in operation. tensile tests were conducted in the T91 SCC research at low strain rates (about 10–6 s−1), which were determined using the electromechanical test devices' initial displacement rate. Since graphite crucibles or Ni-Cr-Mo alloys were used for the corrosion chamber, direct strain measurement is difficult, if not impossible. Similarly, because the salt media is conducting, measuring the dynamic fracture propagation rates using the electrical potential drop across the specimen is rather challenging. As expected, there are significant declines in yield strength, ultimate tensile strength (UTS), and achievable ductility when compared to the behavior of the material at the same temperature but in air. However, the few data that is currently available in the literature makes it quite evident how harmful stacked chemical attack is to mechanical loads in CSP settings.

There is currently a paucity of literature on the nanoscale characterisation of atomic-level processes that follow high-temperature salt corrosion. This is partly due to the challenge of creating in-situ high-resolution salt corrosion tests and the air sensitivity of corroded surfaces for ex-situ testing, particularly alloy/salt contacts. Since laboratory air readily reacts with salt at room temperature, controlling the environment during the fabrication of ex-situ samples is a constant challenge when researching the impact of salt impurities on corrosion behavior, particularly impurity oxidants. There are numerous chances to investigate (solidified) salt/alloy corrosion interactions using cryo-EM and cryo-APT techniques. Specifically, techniques created for researching alkali-rich battery materials-which have numerous air-sensitivities in common with the salts in question-might be applied. It is also becoming possible to make high-resolution in-situ observations of salt corrosion fronts. One example is the salt corrosion of Ni-Cr alloys, which can display a bicontinuous network of nanopores and Cr-depleted metal akin to nanoporous Au generated from Ag-Au alloys. High-resolution x-ray tomography studies have been obtained during this process. While liquid-cell TEM has been used to study aqueous corrosion, molten salt corrosion has not made as much progress as it has. TEM samples have been used to observe salt corrosion iteratively, but the results have been mixed.

5.2 Gaseous corrosion

Beyond the current Ni-based superalloys, research aims to increase the temperature capability of material systems for high-temperature air and combustion conditions. For some time now, two main material classes have been the focus of research for use in hypersonic flight, combustion engines, and reentry vehicles at temperatures as high as 3273 K: UHTC based on refractory high-entropy alloys (RHEAs) and borides, carbides, or nitrides At these high temperatures, oxidation testing is often conducted in (laboratory) air, either continuously using thermogravimetric analysis (TGA) or discontinuously in box/muffle furnaces with intermittent weight change measurements. Such constantly recorded weight fluctuations of the equimolar Ta-Mo-Cr-Ti-Al RHEA throughout a broad temperature range of 1273–1773 K. The moderate weight increases suggest that the complex CrTaO4 scale is well-protected at temperatures up to ΔT ≈ 400 K higher than those permitted for cutting-edge Ni-based superalloys.205 Less expensive oxyacetylene torch tests and more expensive arc jet tests are used to simulate reentry conditions, including thermal shock resistance, in a temperature range between 1373 K (simulating hypersonic flights at Mach 6) and beyond 2273 K. Shows a test of a ZrB2/SiC-based composite, coated with Mo-Si-B, conducted at NASA's HyMETS test facility.209 Nevertheless, studies conducted in water vapor have also been conducted since many of these materials are thought to create a protective silica scale. These studies, however, clearly show (detrimental) increased scale growth under wet conditions. Recently, an intriguing new technique for mechanical testing overlaid with sodium chloride (SCC) and wet air was demonstrated for Ti alloys at high temperatures (723 K). According to posttest APT analysis, the Ti alloy surprisingly absorbed very little deuterium (<0.06 at. %) during two-point bend testing with a specially created setup for 100 hours. On the other hand, the extreme oxygen pickup (>5 at. %) in the wake of the crack tip was discovered to have a substantial impact on embrittlement and ultimately result in the creation of alien phases.

Similar extremely harsh conditions have not yet been produced for high-resolution in-situ characterizations. Modern environmental TEM experiments are usually restricted to operating at temperatures between ~1073-2773 K in moving gas, using either cell-based sample containers or specialized microscopes. Even so, these kinds of investigations have been useful for firsthand witnessing dynamic events at the atomic level that are otherwise only surmised. For instance, the development of a solid metal sphere into a hollow oxide sphere can be seen by the Kirkendall effects of vacancy coalescence at the interior of the nanoparticle when particles oxidize. Currently, this technique to in-situ ultrahigh-temperature oxidation investigations is not as viable due to temperature limits. The sample temperature may be raised by laser heating, but one must also take into account the surrounding gas's temperature and flow rate, as well as how these factors may affect the behaviors that result. Thus, the state of the art for nanoscale analysis is lower-temperature in-situ oxidation or ex-situ characterization of previously oxidized samples. With nanoscale observations of the ensuing chemical, structural, and elemental evolution, ex-situ high-resolution TEM and APT are still likely to be essential to the ongoing development of corrosion-resistant materials, particularly for compositionally complex RHEAs and their resulting oxides.

Within a 0.914 m (3 ft.) diameter and 1.219 m (4 ft.) length, the Glenn Extreme settings Rig (GEER) can simulate a variety of planetary settings, including high temperature and high pressure along with multicomponent chemistry. Owing to NASA's present fascination with Venus, GEER has been set up to replicate the planet's surface atmosphere up to a height of roughly 75 kilometers. With the following settings, GEER can simulate the surface for longer than twenty-four hours: For 24 days, the following parameters were measured: 773 K, 100 bar, CO2-96.5%, SO2-130 ppm, HF-5 ppm, HCl-0.5 ppm, NO-5.5 ppb, CO-15 ppm, COS-27 ppm, N2-3.4%, and H2O-30 ppm.217 Samples being loaded into GEER are depicted. Electronics packaging materials (PbO, Al2O3), insulation (rock wool: CaO/SiO2), SiC electronics (Au/Pt/Ir/Pt/TaSi2 bondpads, Au wires, Pt wires), feed-through materials (ceramawire), SiC pressure sensors, and Inconel 625 are among the materials whose survivability and durability have been tested. An electron microscopy EDS map of the degradation of Inconel 625 in GEER between 10 and 42 days is presented, emphasizing the development and expansion of duplex corrosion layers. Real systems can also be tested because of the system's enormous volume capacity. In order to prepare for a Venus mission, a number of critical systems have already been tested in GEER. These include a sensor system that measures the relative dielectric constant of gases in the Venus atmosphere, a high-temperature co-fired ceramic Al2O3 package with Au/Pt metallization system, and SiC-based integrated circuits (ICs) for electronic systems.

6. Conclusion

From temperatures as low as 1.8 K to as high as ~4000 K, including high stresses and strains coupled with temperature, to hot, corrosive, and oxidative situations, we have come a long way in the study of materials in the most extreme settings. Although there is still much work to be done to push our testing limitations even further, these measurements will be essential for creating materials databases for the predictive design of innovative materials for these conditions. We have identified a number of important testing and instrument developments from this assessment that can be pursued and accomplished in the upcoming ten years. Among them are the following:

1. In situ high-temperature solid-gas reactions: The amount of solid-gas reaction (e.g., oxidation) data reduces as temperature rises (>2000 K). By changing the levitation gas's temperature while it's at it, aerodynamic levitation combined with laser heating could solve this data issue. Data on oxidation and other solid-gas reactions could be gathered up to the melting temperatures of RHEA and UHTC materials if done carefully.
2. Low-temperature application properties: Cryo-EM provides hitherto unexplored possibilities for studying low-temperature functional materials through in-situ experiments at the nanoscale and in real time, in addition to sample preservation. This drive is focused on emerging magnetic phenomena, which are essential for the creation of quantum materials and will soon see notable advancements.
3. Mechanical testing in coupled extreme environments: Development of additional systems to control atmospheres/corrosive environments and new sample designs (e.g., samples with cavities inside that are filled with solar salt or hydrogen passing through a tensile sample during mechanical testing) could yield even greater benefits than the significant advancements made in high-temperature mechanical testing.
4. Small-scale testing: Over the past ten years, significant advancements have been made. Because of the high temperature reactions with the sample, interactions with the tip materials in nanoindentation continue to provide the greatest challenge.
5. High-resolution characterization techniques for high-temperature salt corrosion: Precise control over salt chemistry and its contaminants, which are known to aggravate or completely alter the nature of the salt corrosion, will surely be a technological barrier for in-situ TEM research.228 Much more work is needed to establish similar high-resolution characterization techniques for high-temperature salt corrosion compared to thermal or mechanical extremes.
6. Low temps and bright components: Significant progress has been achieved in exploring hydrogen-assisted processes at the nanoscale through the use of cryo-APT sample preparation and environmental transfer. To establish interdependencies, these techniques need to be further developed to directly correspond with mechanical testing.
7. Humanity may get one step closer to realizing its aspirations of utilizing and storing enormous amounts of energy, as well as exploring the cosmos at a tolerable pace, if these instruments and methods are added to our toolkit.

7. References

1. Webb S. Building Blocks of the Universe. In: Out of this World. Copernicus, New York, NY; c2004. https://doi.org/10.1007/978-1-4757-6120-7_4
2. Khan RS, Rehman IU. Spectroscopy as a tool for detection and monitoring of Coronavirus (COVID-19).

Expert Rev Mol Diagn. 2020;20(7):647-649. DOI: 10.1080/14737159.2020.1766968. Epub 2020 May 20. PMID: 32378969; PMCID: PMC7256345.

3. James JF. Spectrograph Design Fundamentals. Cambridge University Press; c2007. ISBN: 978-0-521-86463-3.
4. Hunt R. History of Spectroscopy - an essay. 2011. doi:10.13140/RG.2.2.21410.84169.
5. El'yashevich MA. History of the development of spectroscopy and Bohr's theory. J Appl Spectrosc. 1989;51:861-867. https://doi.org/10.1007/BF00664592
6. Lewis IR, Saylor B. A Brief History of the Society for Applied Spectroscopy. Applied Spectroscopy. 2018;72(1):7-7. doi:10.1177/0003702818791915
7. Sutton MA. The Early History of Spectroscopy: Platinum Contributes to its Development. Platinum Metals Rev. 1988;32(1):28.
8. Spectroscopic Techniques, Simon W. Lewis, in Encyclopedia of Forensic Sciences, Third Edition, 2023
9. Akasaka K. High Pressure NMR Spectroscopy. Subcell Biochem. 2015;72:707-21. DOI: 10.1007/978-94-017-9918-8_33. PMID: 26174406.
10. Osipov, Armenak A. High-temperature Raman spectroscopy Pure and Applied Chemistry. 2019;91(11):1749-1756. https://doi.org/10.1515/pac-2019-0115
11. Rizzo TR, Boyarkin OV. Cryogenic Methods for the Spectroscopy of Large, Biomolecular Ions. In: Rijs, A., Oomens, J. (eds) Gas-Phase IR Spectroscopy and Structure of Biological Molecules. Topics in Current Chemistry; 2014. p. 364. Springer, Cham. https://doi.org/10.1007/128_2014_579
12. Introduction to Plasma Physics: With Space, Laboratory and Astrophysical Applications, 2nd Edition. Cambridge University Press; 978-1-107-02737-4.
13. Schreiber DK, Schwaiger R, Heilmaier M, *et al*. Materials properties characterization in the most extreme environments. MRS Bulletin. 2022;47:1128-1142. https://doi.org/10.1557/s43577-022-00441-z
14. Renner O, Rosmej FB. Challenges of X-ray spectroscopy in investigations of matter under extreme conditions. Matter Radiat Extremes. 2019;4:024201. https://doi.org/10.1063/1.5086344.
15. Boccato S, Torchio R, Anzellini S, *et al*. Melting properties by X-ray absorption spectroscopy: common signatures in binary Fe–C, Fe–O, Fe–S and Fe–Si systems. Sci Rep. 2020;10:11663. https://doi.org/10.1038/s41598-020-68244-3