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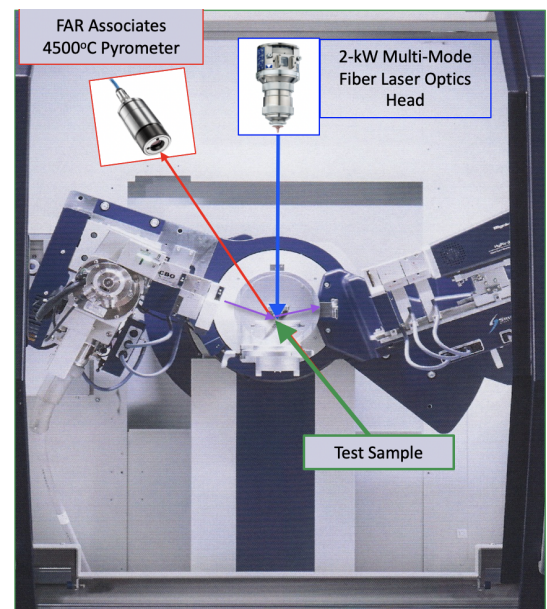
Tracking Melting Points Above 4000 Degrees Celsius

With a new grant, a UC San Diego engineer is developing a better platform to study new materials that melt above 4000 degrees Celsius

A materials engineer at the University of California San Diego is leading the development of a new research platform for studying high-performance materials, in particular new materials that melt above 4000 degrees Celsius (C). UC San Diego nanoengineering professor Kenneth Vecchio is leading the project, which is funded by a new \$800,000 grant from the US Office of Naval Research (ONR), through the Defense University Research Instrumentation Program (DURIP).

The research platform will be built to specifically address the challenges of studying new materials that melt at temperatures higher than 4000C, which is approximately 80 percent of the temperature of the surface of the sun.

This platform will become a national resource for engineers and other researchers who are pushing the limits of materials science. There are many industrial, energy, space and defense applications that would benefit from new solid materials that perform reliably at record-breaking, ultra-high temperatures. Applications include the containment walls for fusion reactors, leading edges of air vehicles that travel five or more times the speed of sound, and new industrial material machining methods. (The tips of cutting tools, for example, can experience temperatures above 3000C at high machining speeds. New cutting tool materials with much higher melting temperatures would enable faster manufacturing.)



Creating and then characterizing materials that don't melt until exposed to ultra-high temperatures presents multiple challenges. In addition to creating candidate materials, researchers need to be able to characterize these materials to demonstrate performance. One of the key metrics is the temperature at which materials melt.

When working at temperatures around 4000C, nothing is simple. For example, how do you test the melting point of new materials that melt at temperatures higher than any containers you might try to hold them in? How do you heat a sample to temperatures in the 4000C range while precisely controlling the temperature? Or how do you determine exactly when a sample at such extreme conditions actually melts?

These are some of the issues that Vecchio and his team plan to solve via an experimental platform he designed and will build thanks to \$800K in new funding from the ONR DURIP grant.

New Materials that melt at temperatures higher than 4000C

How do you increase the melting point of materials that already melt at ultra-high temperatures? For the past five years, Vecchio has led a UC San Diego project funded by the ONR Multidisciplinary University Research Initiatives (MURI) Program to focus on this.

The team is taking a new approach to this difficult task. To make sense of their strategy, you first need to know that there are two physical phenomena that largely determine the temperature at which a solid material melts into a liquid.

The first phenomenon is enthalpy. Enthalpy describes the energy associated with keeping a material in its solid form. The higher the enthalpy, the stronger the bond between the two atoms will be, and the higher you have to heat the material to melt it by breaking those bonds.

The second phenomenon is entropy. Entropy is associated with disorder and describes the energy driving the atoms to separate.

"Melting in a solid occurs when the entropy becomes high enough to exceed the enthalpy," said Vecchio.

Ultra-high-temperature materials development is usually focused on enthalpy. But researchers seem to have reached the limits of the enthalpy approach, at least for materials that do not

react with oxygen. The roadblock is due to the fact that the enthalpy of bonding between atoms is largely fixed. That means that once you find materials with the highest enthalpy of bonding, there is little room for increasing the melting temperature of that material via enthalpy.

In their quest for materials that melt at ever higher temperatures, Vecchio and his team at the UC San Diego Jacobs School of Engineering have turned to the other phenomenon controlling melting temperature: entropy.

Most people have a vague sense that entropy has something to do with chaos or disorder. That notion will come in handy for understanding this new approach to developing materials with record-breaking melting temperatures.

"A liquid has a very high level of entropy. Atoms are moving all around in a liquid, that's entropy. If we can make a solid look like a liquid, in terms of its free energy, then there will be less driving force to change from the solid to the liquid state," said Vecchio.

Making a solid "look" like a liquid means increasing the inherent entropy of the material when it's a solid. That's what Vecchio has set out to do. Their strategy is to increase the disorder in new high-temperature solid materials by mixing large numbers of different atoms together.

"We create disorder through atomic mixing," said Vecchio.

By taking this approach, the researchers are making their solid materials structurally appear more similar to the atomic state of that material's liquid form. This reduces the driving force for the solid to "want" to melt, which can lead to an increase in the temperature at which it melts.

For example, silicon carbide (SiC) is a well-known material with an ultra-high melting point of 2730C. But it would be useful to have related materials that melt at even high temperatures.

Following the entropy-focused approach in previous research, Vecchio and his collaborators replaced the Si atoms with equal amounts of five different metals, which led to new materials with even higher melting temperatures. These new materials, called, high-entropy carbides, or more broadly high-entropy ceramics, are discussed in a research article published in the journal *Acta Materialia* in 2019.

Vecchio and his team are pursuing another use of entropy as well. More entropy can be added

to a solid-material system by adding other molecular and atomic-bonding variations that alone would not increase the melting temperature. But when you add enough of them into the system, the diversity of molecular elements and bonding scenarios further increases entropy, which can increase the temperature at which the solid melts into a liquid.

Researchers must be able to experimentally validate melting temperature and other properties for these kinds of approaches to materials development to lead to new useful materials. That's where the new platform comes in.

High Temperature-X-ray Diffraction platform

The new High Temperature-X-ray Diffraction platform being funded by the ONR DURIP grant to UC San Diego is designed to enable the heating of a sample region to 4500C, while measuring its temperature very accurately, and detecting the onset of melting.

The system components include: a platform that enables high-speed measurements called a high brightness X-ray diffraction platform; a high power laser to locally heat a small region of a material sample; and a high-tech thermometer (a pyrometer) that records temperatures up to 4500C by measuring the wavelength of light emitted from the heat source.

"The integration of these three tools is a fascinating engineering challenge," said Vecchio.

In order to overcome the 'container problem' in which a container melts before the experimental sample it is supposed to be holding, Vecchio plans to use sample materials as their own containers.

"We will only heat a small circular region in the middle of the sample using the laser," he said.

The remaining perimeter of the sample that is not exposed to the laser will serve as the container that holds the sample as it melts from solid to liquid.

"I look forward to being able to share this unique platform with researchers across the country," said Vecchio. "People have been trying to devise systems to measure melting temperatures of these types of materials, but their biggest scientific problem is they have no method to verify what structure the sample has at the perceived onset of melting. My design will solve this problem," explained Vecchio. "By building our melting system inside an x-ray diffraction platform, we will be able to exactly know the structure and type of material present right at the

point of melting, and we will be able to completely verify melting as it has a completely different X-ray diffraction result compared to a solid sample."

He estimates that the facility will be up and running by the end of 2022.

The funded ONR DURIP proposal is entitled "Thermodynamic Measurements of Entropy-Stabilized Ultra-high Temperature Materials." University of California San Diego NanoEngineering Professor Kenneth Vecchio is the Principal Investigator (PI) on the grant. (ONR award number: N00014-20-1-2872).

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