

'Keiser rigs' stress materials to the max to improve products for power, propulsion

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ORNL's Jim Keiser and Mike Stephens (on stepladder) prepare to install samples in a Keiser rig, a furnace for exposing materials to corrosive gases, crushing pressures and calamitous heat. Credit: Oak Ridge National Laboratory, U.S. Dept. of Energy; photographer Carlos Jones

The global marketplace demands constant improvements in performance



and efficiency of aircraft engines, power turbines and other modern mainstays of energy technology. This progress requires advanced structural materials, such as ceramic composites and metal alloys with higher-temperature capabilities. Understanding and predicting the performance of such cutting-edge materials in extreme environments have long been signature strengths at the Department of Energy's Oak Ridge National Laboratory. For decades, environmental exposure chambers at ORNL, including some called Keiser rigs, have subjected materials to corrosive gases, crushing pressures and calamitous heat. The extreme environments created in the Keiser rigs have spurred advances and continue to do so by providing insight into the conditions under which materials fail so researchers can apply the lessons learned to design better materials for power and propulsion applications.

"We've been able to conduct tests in a wide range of high-pressure gaseous atmospheres—pure steam, pure hydrogen, pure oxygen and a very wide range of corrosive mixed gases," said ORNL's Jim Keiser. In 1991 Keiser, along with Irv Federer, built two identical systems with unique capabilities that could generate temperature and pressure conditions difficult to recreate in laboratory settings—so difficult in fact that at the outset of the endeavor many thought it couldn't be done. The systems they built subjected <u>materials</u> to temperatures (up to 1,400 degrees C/2,550 degrees F) and pressures (up to 34 bar/500 psi) more severe than those characteristic of typical methane reformers, which react steam with methane to produce hydrogen and carbon monoxide for the chemical process industry. The furnaces—packed into shiny metal cubes as tall as refrigerators—came to be known as the "Keiser rigs."

Keiser and Federer built the test chambers when Stone & Webster, a Massachusetts-based engineering services company, asked ORNL scientists for help evaluating the durability of ceramic tubes for heat exchangers. If ceramic tubes could endure <u>extreme environments</u> that severely corroded tubes made with other materials, they could increase



the efficiency of industrial operations. It turned out that the joints connecting the ceramic tubes to the metal header of the heat exchanger failed much sooner than the tubes did, so the project was brought to an end. The Keiser rigs, in contrast, lived on and even multiplied. Three now operate at ORNL.

The Keiser rigs then loomed large in a major project to find the best combustor materials for a gas turbine that would be installed at the Malden Mills industrial plant in Massachusetts. Between 1997 and 1999, the collaborative effort of Solar Turbines, ORNL, Pratt & Whitney, DOE's Argonne National Laboratory, B. F. Goodrich and Honeywell Advanced Composites culminated in the creation of a Solar Turbines natural gas–powered turbine engine with combustor liners made of continuous fiber-reinforced ceramic composites, or CFCCs. When it went into operation in 1999, it had the lowest emissions of any commercial combined heat and electric power facility in the United States.

In CFCCs, ceramic fibers are surrounded by a protective coating and embedded in a ceramic matrix. To identify the best materials for Malden Mills and other gas turbine applications, the ORNL researchers and the community in general had pushed CFCCs based on silicon carbide, or SiC, to their limits. These CFCCs handled heat better than most metal alloys, and environmental barrier coatings protected them from the highpressure combustion gas that turned a turbine to generate power.

With funding from DOE's Office of Energy Efficiency and Renewable Energy in the 1990s and early 2000s, researchers from what was then ORNL's Metals and Ceramics Division screened many candidate CFCCs, most of which were produced by partner companies. These promising materials included SiC fibers embedded in a SiC matrix (called "SiC–SiC" composites) and oxide-based composites. Moreover, using ORNL's forced-flow chemical vapor deposition process, Rick



Lowden made composites with a variety of fibers and fiber coatings. Edgar Lara-Curzio evaluated the mechanical performance of the materials—such as strength during stretching and flexing, and stress responses including fatigue, creep and rupture. Then Pete Tortorelli put the materials through extremely high temperatures and pressures in the Keiser rigs and other test environments.

Typically, the team would test coupon-sized material samples in the rig for 500 to 2,000 hours and extrapolate data about degradation rates to predict when materials would fail. The data plots showed that without protective coatings, most of the SiC-based CFCC materials failed within 5,000 hours.

"It was our job to understand how the CFCCs degraded," said Karren More, who performed microstructural characterization of CFCCs prepared with different materials and methods. Comparing identical materials exposed to harsh conditions in either the Keiser rigs or engine tests, More found the mechanism of degradation was the same. "Across the gas flow path you would start to see recession, or loss of material, due to the material's volatilization," she explained.

Nearly two decades after record-low emissions were achieved at Malden Mills, Keiser rigs are still going strong. "Very few similar facilities are available, so Jim is regularly getting industrial requests to use the equipment," said Bruce Pint, leader of ORNL's Corrosion Science & Technology group.

Because increasing steam temperature and pressure improves the efficiency of boilers and turbines, Keiser rigs were also used extensively by a consortium developing materials for the next generation of high-efficiency coal-fired boilers, Pint said. Typical operations will occur at temperatures ranging from 500–800 degrees C (930–1,470 degrees F) and pressures reaching 30 times that of Earth's atmosphere at sea level.



Less than 6 months after the Fukushima Daiichi nuclear disaster of 2011, during which loss of reactor coolant led to three nuclear meltdowns, a Keiser rig was employed to simulate temperature and pressure conditions reached during the accident and screen new candidate fuel claddings up to 1,350 degrees C (2,460 degrees F). The publication that resulted won the 2013–2014 Best Paper Award of the *Journal of Nuclear Materials*.

Currently one Keiser rig is steam-testing materials for the Electric Power Research Institute. For a DOE Office of Fossil Energy study, another Keiser rig is evaluating structural alloys for a new combustion concept, called staged pressurized oxy-combustion, aimed at cleaner use of coal.

Renewed interest in ceramic composite materials may soon call a Keiser rig into service again—this time to evaluate their potential in newer turbine engines.

More information: B.A. Pint et al. High temperature oxidation of fuel cladding candidate materials in steam–hydrogen environments, *Journal of Nuclear Materials* (2013). DOI: 10.1016/j.jnucmat.2013.05.047

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