Neutron scattering research with superalloys is helping scientists develop better materials for engineering and industrial applications.

Superalloy Studies Heat Up

Nickel-based superalloys are attractive structural materials for buildings, precision machinery, and vessels for nuclear power reactors. These remarkable alloys are as strong as steel and possess excellent dimensional stability—that is, they do not expand over a desirable range of operating temperatures.

One type of nickel-based superalloy studied at HFIR is Waspaloy®. This heat-resistant material is used in hot-section components of turbine engines that generate electricity. The constituents of Waspaloy are nickel, chromium, cobalt, molybdenum, titanium, iron, and aluminum. During its manufacture, this alloy is heat-treated to better distribute its precipitates, thus increasing the material's localized "microhardness." The improved precipitate distribution is responsible for the superalloy's excellent mechanical and electrical properties.

Failed tests to drive a pin through Waspaloy samples demonstrate the material's microhardness. The localized spherical inclusions, or solid precipitates, in the polycrystalline material serve as "blocking points" to prevent crack propagation. The heat treatment makes the superalloy more uniform, conferring high electrical conductivity.

The electrical conductivity could be used as a nondestructive measure of performance. If the conductivity suddenly decreases after a long time, the change may indicate that a small crack is about to form in the superalloy, marking a precondition for mechanical failure.

Ken Littrell, lead scientist for the GP-SANS instrument at HFIR, and Rosario Gerhardt and Ricky Whelchel, both of the Georgia Institute of Technology, are using SANS to investigate whether the distribution of precipitates changes in Waspaloy components in turbine engines as a result of repeated thermal exposure and cycling during service. SANS uses beams of cold neutrons, cooled with liquid hydrogen, to produce slower, longer-wavelength neutrons.

The researchers used cold neutrons to strike, at an angle of less than 1 degree, a previously heated Waspaloy sample measuring about 1 square centimeter in area and 1 millimeter in thickness. The results showed that the size of and separation between the inclusions depended on how hot the sample became and how long it was heat treated. Because the low-energy neutrons used had a wavelength about the same size as an inclusion (0.4 to 2 nanometers), these measurements could be made.

"By studying the SANS diffraction pattern, we found that heating time and temperature could be correlated with inclusion size and spacing between these particles," Littrell says. "Smaller inclusions are formed when the Waspaloy sample is heated at low temperatures, and larger inclusions are formed at higher temperatures."

The researchers obtained a previously prepared Waspaloy sample and measured the distances between the inclusions using SANS. Then they heat treated it outside the SANS instrument (ex situ heating) for a

nominal two minutes. When the heat-treated sample cooled to its stable configuration at room temperature, the scientists placed the specimen in the SANS instrument. "It took less than five minutes in room temperature to see the effects of heat treatment," Littrell says, indicating that the spacing between the inclusions had increased and become more uniform.

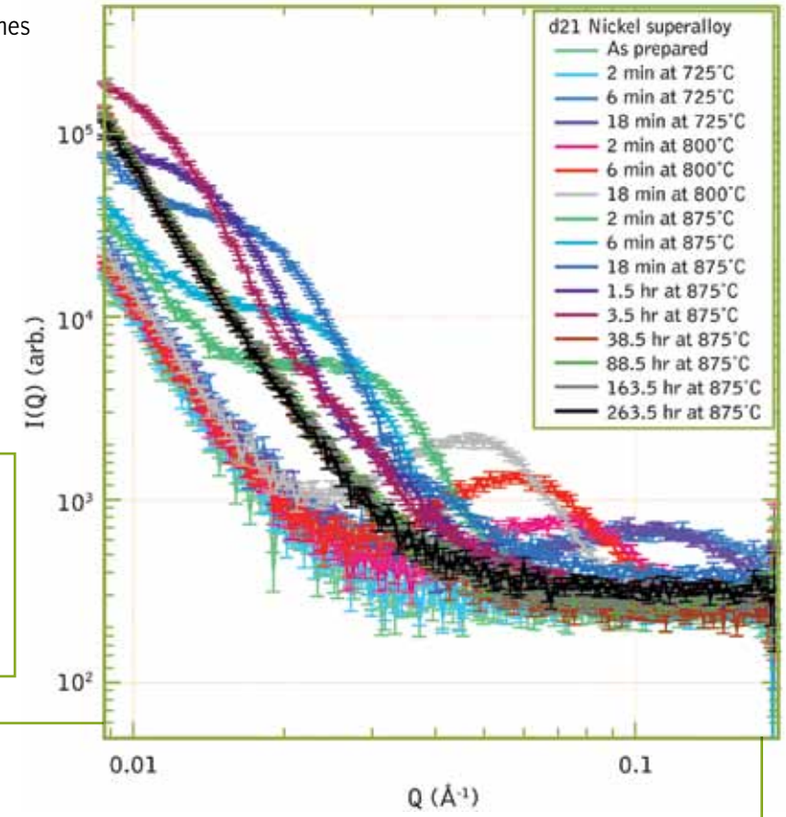
"The diffractions from these correlations show up as a beautiful ring that is faint and broad, spreading to the edges of the detector," Littrell says. "The center of the ring becomes sharper and brighter. Our measurements clearly showed that the distance between the precipitates increases with a higher heat treatment temperature or a longer treatment time."

The peaks in the neutron scattering curves (see image) represent the scattering from correlated spherical inclusions; the positions of the peaks give the average separation between inclusions. The number and density of the inclusions are related to the hardness of the material. Changes in hardness that signal imminent failure of the part can be confirmed by detecting a decrease in electrical conductivity.

The statistical results and measurement times demonstrate that the GP-SANS system at HFIR is suitable for more complex experiments in which superalloy samples are heated while being probed by neutrons.

"We want to do real time in situ heating of the Waspaloy sample while probing it with SANS," Littrell says. "Instead of frozen snapshots of the sample before and after heating outside the instrument, we hope to observe the rate of changes in the sample's spacing between precipitates and its composition during the heating process. It will be sort of like making a movie."

Ken Littrell discusses the GP-SANS capabilities with Neutron and X-Ray School students.



The peaks in the neutron scattering curves represent the scattering from correlated spherical inclusions; the positions of the peaks give the average separation between inclusions. The number and density of the inclusions is related to the hardness of the material. Changes in hardness that are signs of imminent failure of the part can be confirmed by detecting a decline in electrical conductivity.