ScienceWatch Home
Inside This Month...
Interviews

Featured Interviews
Author Commentaries
Institutional Interviews
Journal Interviews
Podcasts

Analyses

Featured Analyses
What's Hot In...
Special Topics

Data & Rankings

Sci-Bytes
Fast Breaking Papers
New Hot Papers
Emerging Research Fronts
Fast Moving Fronts
Corporate Research Fronts
Research Front Maps
Current Classics
Top Topics
Rising Stars
New Entrants
Country Profiles

About Science Watch

Methodology
Archives
Contact Us
RSS Feeds

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TRACKING TRENDS & PERFORMANCE IN BASIC RESEARCH

FAST MOVING FRONTS - 2009

November 2009

PDF

Ronald L. Klueh talks with *ScienceWatch.com* and answers a few questions about this month's Fast Moving Front in the field of Engineering. The author has also sent along images of his work.

Article: Oxide dispersion-strengthened steels: A comparison of some commercial and experimental alloys
Authors: Klueh, RL;Shingledecker, JP;Swindeman, RW;Hoelzer, DT
Journal: J NUCL MATER, 341 (2-3): 103-114 MAY 15 2005
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SW: Why do you think your paper is highly cited?

Engineers and designers of conventional fossil-fuel-fired power-generation plants constantly push for increased efficiency in an effort to produce more power with less pollution. To increase efficiency, higher operating temperatures are required. In the case of nuclear fission and nuclear fusion power plants, not only are high temperatures required, but the structural materials of these plants will also be subjected to high neutron fluences and corrosive environments.

For most new designs, therefore, available materials can place limitations on designers and engineers, and when a new material is unveiled that shows significantly improved properties, there is a scramble to advance that material to the point where it can be incorporated into the new designs.

In the fusion materials program at Oak Ridge National Laboratory (ORNL) around 2000, we performed research on a Japanese oxide dispersion-strengthened (ODS) steel that had remarkable short-term tensile properties (yield stress and tensile strength) as a function of temperature out to >800ºC.

It also had unusual microstructural features on the nanoscale level, as determined by transmission electron microscopy and atom-probe tomography. Excitement raised by observations on this ODS steel (labeled 12YWT) sent numerous investigators throughout the world on a search for the next great structural material for fusion. These steels were soon seen also as future materials for improved fossil-fired and nuclear fission power plants.

In reality, ODS steels were not new. They were first investigated by Belgian researchers for fuel cladding for nuclear reactors in the 1960s, but problems were encountered, and the steels were not perfected for the cladding application. International Nickel obtained a patent on an ODS steel in the 1970s, and eventually ODS products became available commercially with small quantities being used in niche applications. During the 1980s, Japanese researchers again began investigating them as possible fuel-cladding materials, and the 12YWT material ORNL investigated came from that source.

I believe our paper (co-authors: John Shingldecker, Robert Swindeman, and David Hoelzer) is highly cited because we were the first investigators to conduct creep tests on 12YWT and on one commercial ODS steel and compare the tensile and creep-rupture properties of 12YWT with other experimental and commercial ODS steels.

The paper compared elevated-temperature creep-rupture properties for five different steels, and an effort was made to explain the mechanical properties behavior in terms of microstructural characteristics to explain why some ODS steels are stronger than others.

Based on our tests, we postulated microstructures for some of the commercial steels that were later verified by atom-probe studies. Our work therefore provided a roadmap with which later investigators and alloy developers could compare and analyze the microstructure and mechanical properties of their ODS material.

SW: Does it describe a new discovery, methodology, or synthesis of knowledge?

I would say it describes a synthesis of knowledge in that it offers an explanation for the variation in the elevated-temperature strength properties of the commercial and experimental ODS steels. Based on our observations, we suggested avenues of research that should be pursued to verify our hypotheses on the origin of the elevated-temperature strength of the steels.

SW: Would you summarize the significance of your paper in layman's terms?

To explain our work, it is necessary to provide a little background on the nature of ODS and conventional steels. Properties of conventional steels depend on the alloy content and heat treatment. Steels are iron-based alloys, and conventional high-temperature steels contain 2-12% chromium and lesser amounts of a combination of elements such as carbon, nitrogen, tungsten, molybdenum, vanadium, niobium, etc.

Conventional steels are produced by melting and casting. The casting is then fabricated into the desired structural component, after which it is heat treated to optimize the desired properties.

When the steel is viewed under a light microscope up to 1,000X, it is seen that there is a matrix phase that contains a distribution of relatively small particles of other phases that precipitated during the heat treatment. The precipitates are usually carbides or nitrides of chromium, vanadium, and/or niobium, etc., depending on the elements in the steel composition.

There are two classes of elevated-temperature steels, known as ferritic and austenitic steels. The matrix phase of ferritic steels has a body-centered cubic structure. That is, on the atomic scale, atoms are arranged on a cubic lattice with atoms on each of the corners of the cube and one atom in the cube center. Austenitic steels have a face-centered cubic lattice structure with atoms on each corner of the cube and one in each cube face.

The ODS steels of this discussion are ferritic steels, which are preferred for elevated temperature applications because of their better thermal properties—higher thermal conductivity and lower thermal-expansion coefficient. They are also more irradiation resistant for nuclear applications in fission and fusion reactors.

We conducted two types of mechanical properties tests in our studies: a short-time tensile test and a long-time elevated temperature creep-rupture test. In a tensile test, a rod specimen is deformed by pulling at a constant rate until it ruptures. A measure of strength is the yield stress, the stress at which plastic or permanent deformation first occurs. Tensile tests are conducted at room temperature and at intervals up to 800-900ºC to determine the temperature effect.

If a rod specimen is heated to an elevated temperature and a weight hung on the specimen such that there is no immediate deformation, with time at temperature, the specimen will elongate—it will "creep" which is defined as time-dependent deformation. A creep-rupture test is a creep test that is run until the specimen ruptures.

Deformation of a metal alloy occurs by the movement of line defects—called "dislocations"— through the alloy matrix. Dislocations can be imaged by transmission electron microscopy (TEM) at a magnification of several thousand times. One way to slow the movement of dislocations is to introduce a high number density (number per unit volume) of small precipitate particles into the matrix. In conventional steels, precipitates become unstable at elevated temperature, and they grow into a smaller number of large particles, thus limiting their strengthening effect and limiting the upper-use temperature of the steel.

A solution to this problem is to produce steel with particles that remain stable to higher temperatures. It turns out that oxides are much more stable than most carbides and nitrides that form in conventional steels. Unfortunately, it is not possible to precipitate stable oxides in the lattice, so fabrication processes other than melting and casting are required.

Powder metallurgy techniques are used to produce ODS steels with a fine distribution of stable oxide particles. These ODS steels are produced from a fine powder of a steel alloy composition that serves as the matrix, such as an iron-chromium-tungsten composition, and mixing it with an oxide powder. Yttrium and titanium oxides are commonly used. After the powders are mixed, they are compacted and extruded at an elevated temperature to form a solid. Finally, the extruded product is rolled into bar, plate, or sheet, or extruded into rods or tubes.

To understand the microstructural differences between a strong and a weak ODS steel, ORNL investigators conducted optical microscopy, TEM, and atom probe field ion microscopy studies on two ODS steels. The first, labeled 12Y1, was fabricated with powders of an iron-12% chromium alloy and yttrium oxide (Y_2O_3), and the second, labeled 12YWT, was obtained from powders of an iron-12% chromium-tungsten-titanium alloy and Y_2O_3.

Figure 1

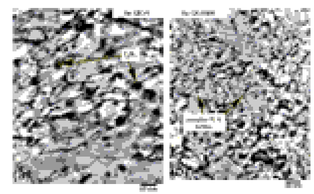


Figure 1: Transmission electron micrographs of experimental ODS steels (a) 12Y1 (Fe–12Cr–0.25Y_2O_3) and (b) 12YWT (Fe–12Cr–2.5W–0.4Ti–0.25Y_2O_3).

[+] enlarge

The results produced very different microstructures in the two steels (Fig. 1). For 12Y1 [Fig. 1(a)], particles were estimated to be 10–40 nm in diameter at a number density of 10^{20}-10^{21} m^{-3}.

Diffraction studies indicated the particles in 12Y1 were essentially pure, crystalline Y_2O_3. For 12YWT [Fig. 1(b)], particle size and particle number density were estimated at 3–5 nm diameter and about 10^{23} m^{-3}, respectively. For this alloy, three-dimensional atom probe analysis revealed compositionally distinct nano-sized clusters enriched in yttrium, titanium, and oxygen, slightly enriched in Cr, and slightly depleted in Fe and W.

The difference in oxide particle size in the two steels is evident. The lines between particles are dislocations. Work reported in our paper involved the study of the mechanical properties as a function of temperature of these two experimental steels and three commercial ODS steels and demonstrated the difference in properties in the two types of ODS microstructures. As expected from the microstructural differences observed in Fig. 1, the 12YWT was much stronger than 12Y1 in a short-time tensile test and a long-time creep-rupture test.

Comparison of 12YWT with the commercial steels gave different results. One of them (MA 957) had comparable short-time tensile and long-time creep properties. This steel had been shown to have the nano-sized clusters similar to those of 12YWT.

A second steel (PM 2000) had short-time tensile properties comparable to 12Y1 at low temperatures, but contrary to 12Y1, it had elevated-temperature creep-rupture properties comparable or better than those of 12YWT, which was unexpected.

Finally, and also unexpected, the third commercial ODS steel (MA 956) had inferior tensile properties relative to those of 12Y1, but nevertheless, its long-time creep-rupture properties were comparable to those of 12YWT.

These results indicated that differences involved more that just the sizes and number densities of the oxide particles. Among the possibilities discussed were grain size and the amount of recrystallization that can affect tensile behavior at low and intermediate temperatures.

SW: How did you become involved in this research and were any particular problems encountered along the way?

My work in recent years has involved the physical metallurgy of steel, studies of the relationship of mechanical properties to microstructure, and the development of new steels. It was therefore logical for us to study the relationship of these unique microstructures to their mechanical properties. A major difficulty was the availability of ODS material to test.

It is ironic that, although ODS steels are at present a hot scientific topic, there are no longer any commercial manufacturers of the product, since the market for ODS steels is limited, and it is not cost-effective for the manufacturers to continue to support manufacturing facilities for limited sales.

SW: Where do you see your research leading in the future?

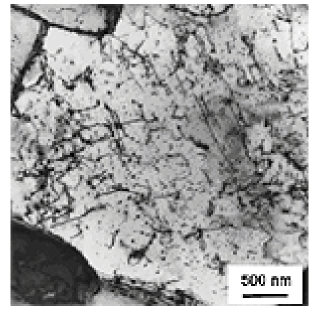
In recent years, my work has been associated more with conventional steels, those produced by melting, casting, fabricating, and heat treating. The ODS steels have numerous problems to overcome. That is why they have been in a development stage since the 1960s.

Because of the powder metallurgy processes used to produce them, their mechanical properties are anisotropic. That is the reason they were abandoned in the 1970s as possible nuclear fuel cladding materials for fast reactors. Their high strength is in the direction in which they are worked—rolled or extruded—and they are relatively weak in the direction orthogonal to the working direction (the niche applications mentioned above exploited the anisotropy). Much effort has been expended over the years to solve this problem.

In addition, they cannot be welded by the usual fusion-welding techniques, since melting destroys the fine oxide distribution and thus produces a weld of inferior strength. Should those problems be solved, a possible bigger impediment to their eventual widespread use is economics. The complicated manufacturing process is extremely expensive relative to conventional steels.

In recent years I have been involved in trying to develop improved high-temperature steels using conventional steelmaking techniques to produce a high number density of fine precipitates into the steel matrix. We have had some success in producing steels using conventional processing techniques that contain a high number density of small nitride precipitates similar to those in 12YWT (Fig. 2). This was accomplished by a thermo-mechanical treatment, wherein hot working was introduced into the normal heat treatment sequence typically used for conventional steels.

Figure 2



Transmission electron microscopy photomicrograph of a 9Cr-1Mo steel after a thermo-mechanical treatment. The steel contains a high number density of fine nitride precipitates that formed on dislocations (lines in the photo) during the treatment.

[+]enlarge

The normal heat treatment for conventional elevated-temperature steels produces similar nitrides as those shown in Figure 2. However, in conventional steels they are present in a much smaller number density and have a much larger size. Thermo-mechanical treatment (TMT) that is used to produce the microstructure in the figure is hot working, which produces a high number of dislocations into the matrix. These dislocations, some of which can be seen as lines in the figure, act as heterogeneous nucleation sites for the nitride precipitates.

Although these precipitates are not as stable as oxides in ODS steels and the new steels could not be of used to 800ºC and beyond, as visualized for ODS steel, they could push the use temperature well above the 600-620ºC limit of present high-temperature ferritic steels. Such an increased service temperature could have a significant effect on the efficiency of a power plant. It needs to be emphasized that, just as the ODS steels are still in a development stage, the same must be said for these thermo-mechanical treated steels.

Of course, the best approach to better elevated temperature steels would be to improve the properties by alloying and using conventional heat treatment processes used in the steel industry at present. Today, computational thermodynamics programs are available that allow us to obtain insight into how we might accomplish that. Based on thermodynamics calculations, I have developed some ideas on how improved steels using present-day steelmaking processes might be produced, ideas which I would like to pursue should the opportunity arise.

SW: Do you foresee any social or political implications for your research?

If ODS steels or improved conventional steels discussed here can be used in the future, they could have considerable effect on energy production for electricity, which could, in turn, have major social and political implications, since energy plays such a large part in our everyday lives.

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