FOSSIL ENERGY APPLICATIONS OF INTERMETALLIC ALLOYS

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ABSTRACT

This paper presents the history and results of the U.S. Department of Energy, Office of Fossil Energy (FE), development effort on iron aluminide alloys and the status of research and development on ultrahigh-temperature (well above 1000°C) intermetallic alloys.

The outstanding (perhaps unequaled) sulfidation resistance of iron aluminide alloys, based on the Fe_3Al composition, was the basis for initiation of an exploratory project to determine whether improvements in mechanical properties of iron aluminide alloys could be achieved. This was a high-risk venture but with significant payoff if successful, because sulfidation was a critical problem for applications of alloys in coal gasification systems. The exploratory project, which was conducted at Oak Ridge National Laboratory (ORNL), was successful, and the program was expanded in both scope and participation. Several other national laboratories, industrial research organizations, and universities were involved. Iron aluminide development

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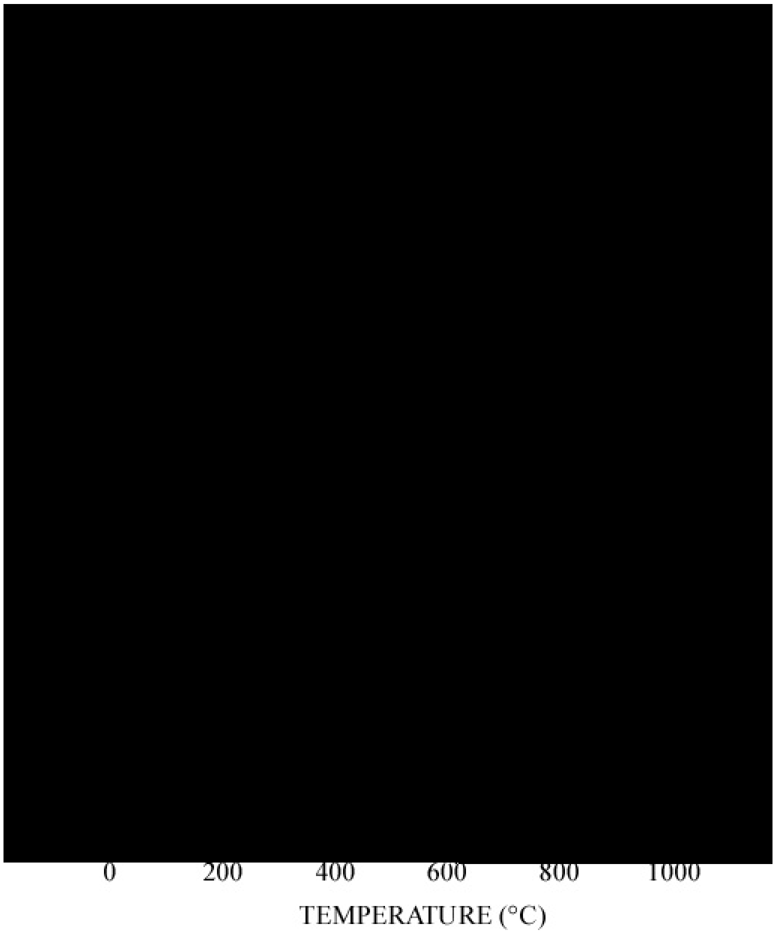

Fig. 1. Comparison of high-temperature strength of advanced aluminide alloys with conventional alloys.

IRON-MODIFIED NICKEL ALUMINIDES, $(Ni,Fe)_3Al$

The initiation of FE-sponsored work on $(Ni,Fe)_3Al$ was due primarily to the success of C. T. Liu and co-workers at ORNL in developing ductile nickel aluminide alloys based on Ni_3Al (Liu and Stiegler 1984; Liu and White 1985). The discovery that low-level (200 ppm) boron additions to Ni_3Al dramatically improved ambient-temperature ductility generated great interest in this class of alloys for high- temperature structural applications. However, these Ni_3Al alloys did not have adequate sulfidation resistance (Liu et al 1987) for many of the fossil energy applications of interest (particularly coal gasification). Consequently, an investigation was initiated on Ni_3Al alloys with up to 19% iron additions to attempt to sufficiently improve the sulfidation resistance of the alloys so they could be used in a broad range of fossil energy applications. The motivation for this effort was the potential for very-high-strength alloys (Fig. 1) at the high temperatures typical of many coal-fueled systems.

Although the mechanical properties of these $(Ni,Fe)_3Al$ alloys were excellent, it was not possible to improve sulfidation resistance sufficiently to use them in sulfidizing environments. Nonetheless, the alloys were patented (U.S. Patent No. 4,731,221) and licensed for other high-temperature structural applications. The AR&TD Materials Program development on $(Ni,Fe)_3Al$ was transitioned to the development of alloys based on the binary compound, Fe_3Al.

IRON ALUMINIDES BASED ON Fe_3Al

An exploratory investigation of Fe_3Al-based alloys was begun in 1986 (McKamey et al 1991). DeVan (1989) obtained oxidation and sulfidation data that indicated great potential for use of this class of iron aluminide alloys in coal gasification environments (Fig. 2). Conventional wisdom at the time was that chromia formers such as type 310 stainless steel were most appropriate for applications in high-temperature sulfidizing environments. The data presented in Fig. 2 indicate an order-of-magnitude advantage for iron aluminides, which form protective alumina, over chromia formers like type 310 stainless steel.

Several issues had to be addressed, however, before these alloys could be used in high-temperature applications. They had poor ambient-temperature ductility, were not weldable, and were subject to embrittlement in many environments. Furthermore, the alloys were inherently much weaker than other alloys, such as those based on Ni_3Al, for example. The challenge was then to improve the properties of alloys based on Fe_3Al to enable them to be used and to take advantage of their outstanding oxidation and sulfidation resistance.

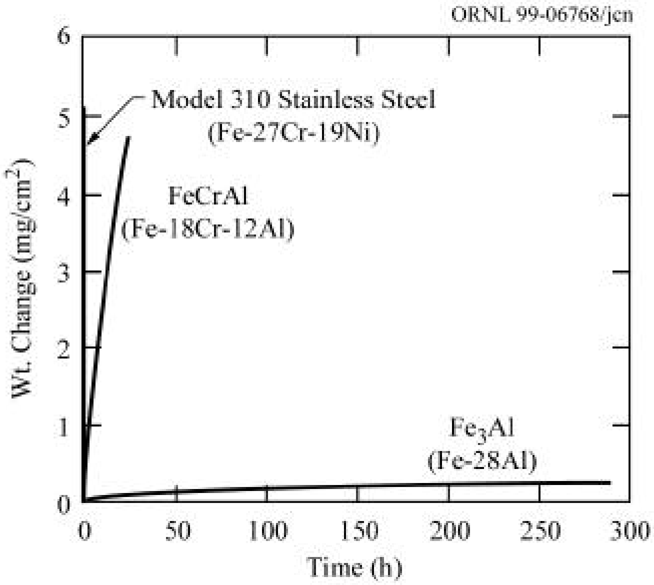

Fig. 2. Comparison of sulfidation of Fe_3Al with type 310 stainless steel and a FeCrAl alloy.

Poor ambient-temperature ductility was addressed primarily through additions of chromium to the Fe_3Al-based alloys. Additions of 2 to 5 wt% of chromium improved the ductility of the alloys considerably, with optimum improvements at 5% chromium (McKamey and Liu 1990). Further improvements in ductility were achieved by incorporating an oil quenching or oil coating operation in thermomechanical forming of the alloys (Sikka, Viswanathan, and McKamey 1993; Sikka 1997). Figures 3 and 4 demonstrate the effects of alloy additions and oil coating, respectively, on ductility.

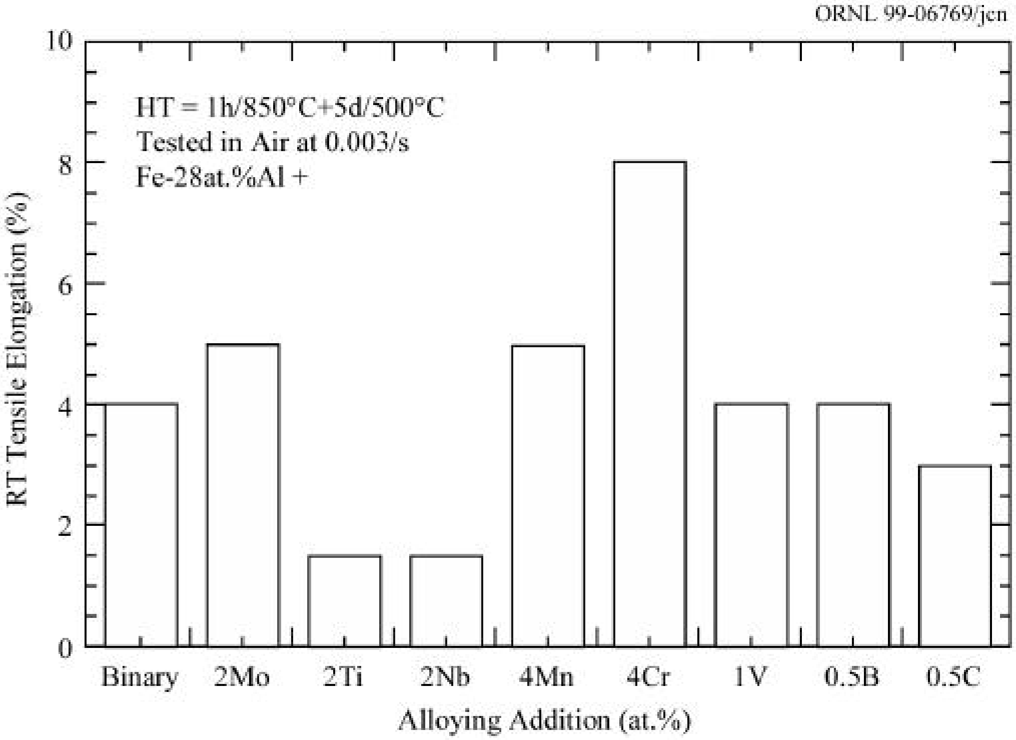

Fig. 3. Effect of minor alloying elements on the room-temperature tensile ductility of Fe_3Al.

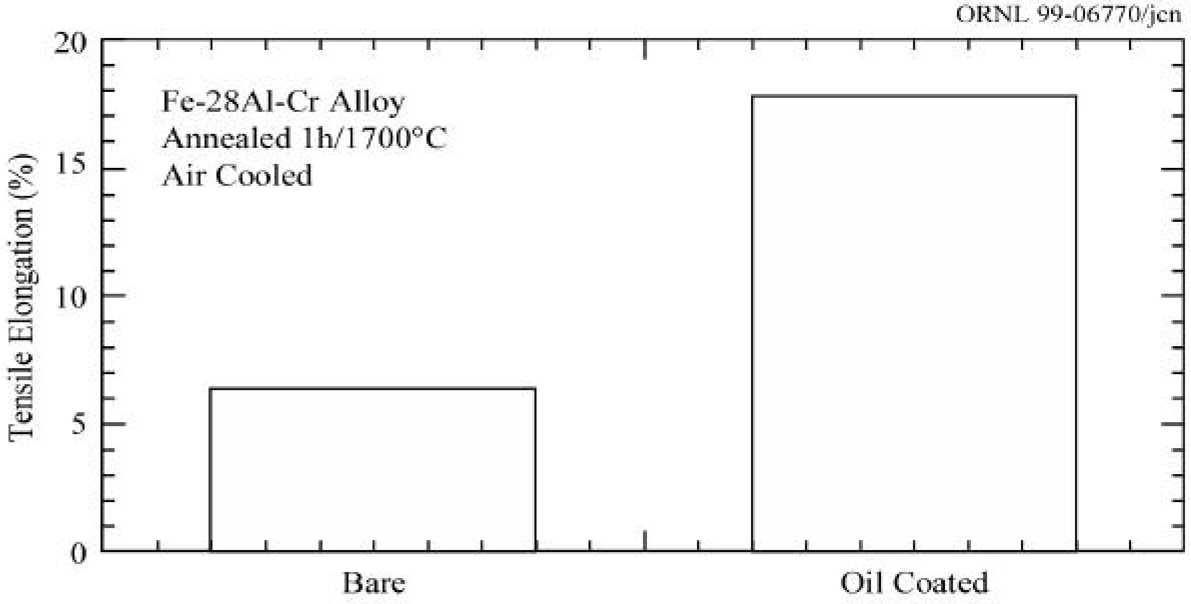

Fig. 4. Effect of oil coating on tensile ductility of a Fe-28Al-2Cr(at.%) alloy.

Welding of iron aluminides was especially difficult due to both hot-cracking and cold-cracking. These problems were addressed and welding procedures were established involving pre- and post-weld heat- treating schedules that enable these alloys to be welded (Goodwin et al. 1994; Goodwin 1997). Welding R&D has been extended to weld-overlay techniques that are used to apply protective Fe_3Al surfaces to conventional steels and superalloys to provide corrosion protection in the often-hostile coal-combustion and gasification environments (Tortorelli et al. 1996). Work at Lehigh University (Banovic et al. 1997) has been directed toward weld overlays for use in low-NO_x burner systems of advanced coal-based power systems being developed to limit emissions of oxides of nitrogen, which are pollutants contributing to smog and possibly to climate change. In these systems, conditions are often locally reducing, and conventional alloys have experienced increased corrosion, presumably because the localized oxygen potential is too low for these alloys to achieve and maintain protective oxide surfaces. This is not necessarily the case for iron aluminides, and initial results have indicated a potential for using Fe-Al alloys as corrosion-resistant coatings under low- NO_x conditions (Banovic et al.1998: Tortorelli, Pint, and Wright, 1998). Coatings produced by electrospark deposition (Johnson 1995) have also exhibited good corrosion resistance under sulfidizing conditions (Natesan and Johnson 1995).

Alloys based on Fe_3Al are susceptible to a form of hydrogen embrittlement that occurs in moisture-containing environments. This is a strain-rate sensitive phenomenon that can be mitigated by chromium additions, control of microstructure, preoxidation, and environmental control (Liu, Lee, and McKamey 1989; Liu and McKamey 1990; Liu, McKamey, and Lee 1990). Because of all the advancements made in understanding the properties and degradation mechanisms for iron aluminides, it is now possible to produce Fe_3Al-based alloys with ambient temperature tensile ductilities of 10 to 20% and tensile yield strengths up to 500 MPa.

Powder-metallurgy processing techniques for both dense and porous articles have been investigated. Pall Corporation of Cortland, New York, ORNL, and the DOE Federal Energy Technology Center (FETC) have collaborated on the development of hot-gas filters made of iron aluminide alloys (June and Sawyer 1998, Tortorelli et al. 1998, 1999). A modification of Pall's conventional production methods is employed in fabricating the filters (Fig. 5). The first two commercial Fe_3Al alloy products are the powder, which is produced under an ORNL license by Ametek Specialty Metals Division of Eighty Four, Pennsylvania, and the iron aluminide filters produced by Pall using Ametek-produced powder. Performance of the filters has been tested and demonstrated in several test beds and plants, including the FETC gasifier in Morgantown, West Virginia; the Power Systems Development Facility in Wilsonville, Alabama; the Transport Reactor Demonstration Unit at the University of North Dakota Energy and Environmental Research Center (EERC); the Foster Wheeler Circulating Pressurized Fluidized Bed Combustion Plant in Karhula, Finland; the Dynegy Wabash Coal Gasification Plant in West Terre Haute, Indiana; and in fluidized-bed combustion plants in Japan. To date, these filters have performed well.

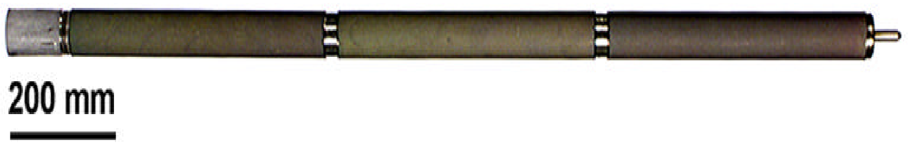

Fig. 5. Iron aluminide hot-gas filter manufactured by Pall Corporation.

OXIDE-DISPERSION-STRENGTHENED (ODS) IRON ALUMINIDE ALLOYS

Mechanical alloying of iron aluminide alloy powder with yttria to provide oxide-dispersion strengthening is being developed to increase strength and to extend the operating temperature to >1000EC while maintaining the outstanding high-temperature corrosion resistance. The milling process employed results in a uniform, fine dispersion of inert oxide particles within the alloy structure. Thermomechanical processing, such as extrusion of the powder followed by heat-treating results in the growth of very large grains of the alloy (Fig 6). The combination of these large grains and the grain boundaries (which are pinned by the oxide dispersions) together with the general impedance to dislocation movement caused by the oxides dispersed within the grains results in extremely high creep strength. Figure 7 compares the creep strength of these alloys with conventional high-creep-strength alloys. The alloy development is being conducted by ORNL, Metallwerke Plansee, the University of Liverpool, and the University of California, San Diego. Welding process development is being performed at The Welding Institute (TWI) in Cambridge, England. These ODS alloys have great potential as alternatives to high-temperature ceramics in heat exchangers and recuperator applications (Wright et al., 1998). These alloys would be used in applications that require strength and environmental resistance at temperatures above the capabilities of conventional alloys. For example, they could be used in heat exchangers in indirectly fired turbine systems such as a High- Performance Power System (HiPPS), in which the hot combustion gases are used to heat air in the heat exchanger tubes and the hot air is the working fluid for a gas turbine. They could also be used to fabricate combustor components in engines required in various Vision 21 configurations.

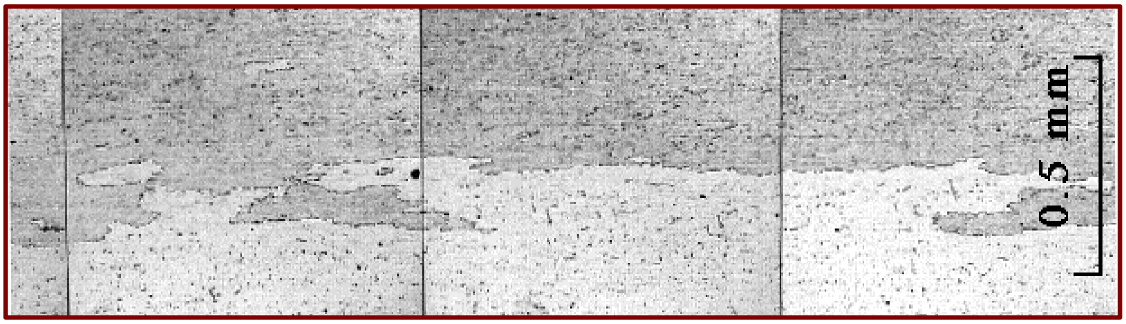

Fig. 6. Photomicrograph of an oxide-dispersion-strengthened iron aluminide alloy showing large grain sizes.

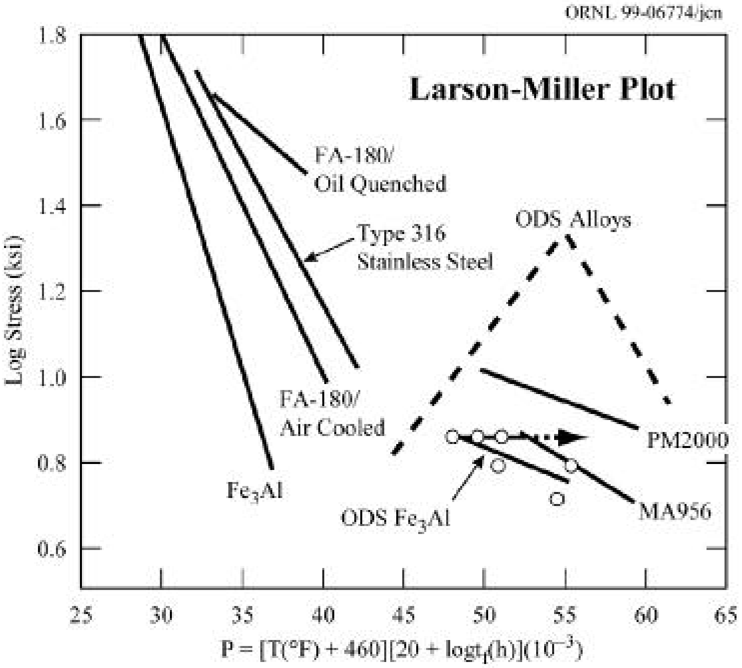

Fig. 7. Comparison of the creep strength of oxide-dispersion-strengthened alloys with conventional alloys.

ULTRAHIGH-TEMPERATURE INTERMETALLICS

The practical application of metals as structural materials at ultrahigh temperature (well above 1000EC) is a formidable challenge. Such high-temperature applications are necessary to achieve the high- performance goals of Vision 21. Vision 21 is the DOE FE concept of very-high- efficiency (60–65%) power generation systems with dramatically reduced pollutant emissions, including little to no CO_2, which implies the necessity for carbon sequestration. Generally, the melting temperature of a material for structural applications at such temperatures should be in the range of at least 1600 to 2000EC, and the only metals capable of withstanding such high temperatures are refractory metals such as molybdenum and tungsten and a few intermetallics and intermetallic-strengthened alloys. However, poor mechanical properties and/or proclivity to catastrophic oxidation severely limit their use. Several ultrahigh-temperature alloy systems have been investigated under sponsorship of the AR&TD Materials Program to determine whether the requisite properties can be obtained. These include alloys based on Cr(Nb)-Cr_2Nb, Cr(Ta)-Cr_2Ta, and Mo_5Si_3.

Intermetallic-Reinforced Cr Alloys

Intermetallic-reinforced Cr-based alloys are targeted for use as hot components in advanced fossil energy conversion and combustion systems envisioned for the DOE-FE Vision 21 Concept. They are intended for structural use in oxidizing and aggressive, high-temperature corrosion environments in the 900 to 1300°C temperature range. Potential applications include thermowells, splash plates, and hot-gas filters, and eventually (assuming sufficient continued progress is made) first-stage turbine components such as vanes, seals, and nozzles.

The initial alloy property goals of this project are oxidation resistance comparable to conventional chromia-forming alloys at 1100°C in air, 350-MPa tensile fracture strength and creep resistance comparable to superalloys in the 1000 to 1200°C temperature range, and ambient-temperature fracture toughness in the 15 to 20-Mpa / m range. Efforts first focused on Cr(Nb)-Cr_2Nb alloys (Liu et al 1996) but the best properties were obtained in the Cr(Ta)-Cr_2Ta

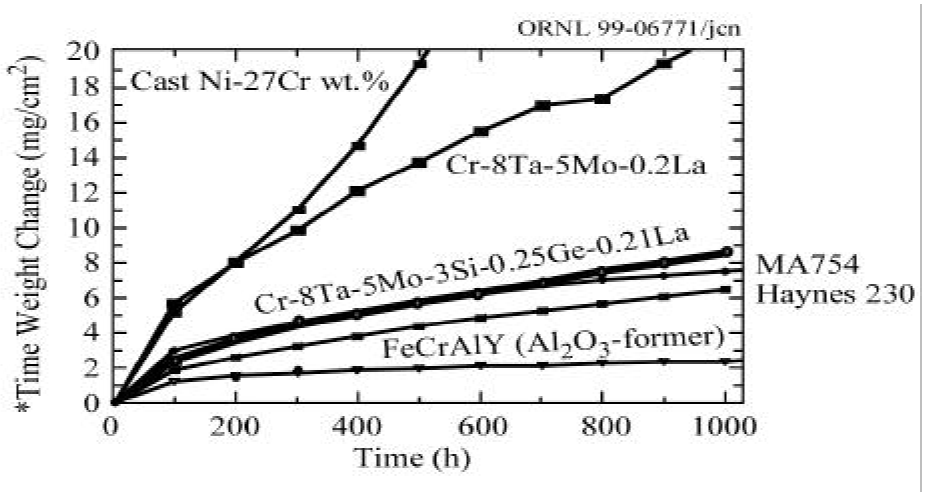

Fig. 8. Cyclic oxidation behavior of several alloys at 1100°C in humid air (comparison date of B. A. Pint and I. G. Wright, unpublished, 1999). (*Includes scale volatility and spallation.)

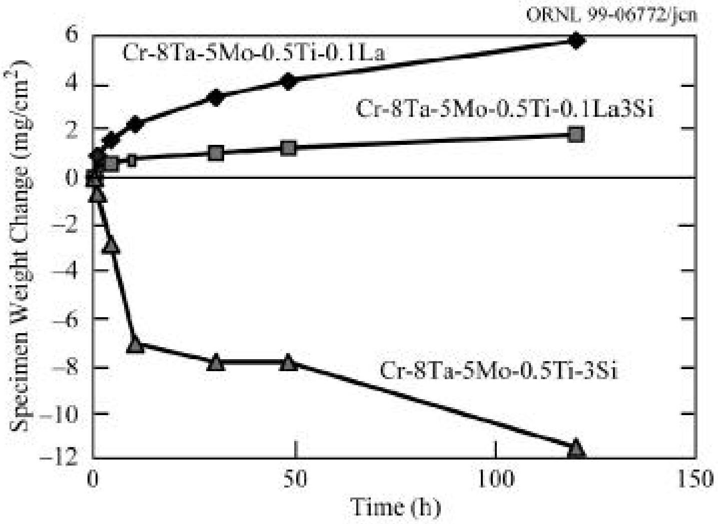

Fig. 9. Cyclic oxidation data for some Cr(Ta)-Cr_2Ta alloys at 1100°C in humid air.

High-temperature (1000EC) tensile creep testing was performed in air on Cr-8Ta-5Mo-0.5Ti-0.01Ce in two microstructural conditions, dispersed Laves phase particles in a Cr matrix and lamellar Cr/Laves phase (Fig. 10). Creep resistance (load stress of 138 MPa) was outstanding for the lamellar microstructure; in fact, the test had to be stopped after nearly 1500h because the grip rods (MAR 246) and plate (Ni_3Al) were severely deformed. The Laves-dispersed microstructure also showed excellent creep resistance and failed in a ductile manner after about 700h. These preliminary results indicate that the creep resistance of these alloys in both microstructural conditions is comparable to that of even some single-crystal superalloys and therefore also meets the initial property goals of this project. (Due to the deformation of the test equipment, the data in Fig. 10 should be considered semiquantitative).

Ambient temperature fracture toughness of the Cr(Ta)-Cr_2Ta alloys was evaluated using chevron notched three-point bend samples. The current alloys have fracture toughness values in the 12-14 MPa **/**m range (Brady et al. 1999); thus, although they come close, the alloys currently fall short of the fracture- toughness goal of at least 15 to 20MPa **/**m at room temperature. Strategies to increase ambient-temperature fracture toughness involving tailoring of the Cr(Ta)/Cr_2Ta lamellar structure (including directional solidification) and ductilization of the Cr(Ta) matrix phase via alloying with MgO (Scruggs, Van Vlack, and Spurgeon 1968, Brady et al. 1999) are being pursued.

Molybdenum Silicide Intermetallics Containing Boron

The objectives of this project are to develop corrosion-resistant Mo-Si alloys for structural applications at temperatures as high as 1600EC. This is, of course, a monumental challenge today and would have been considered impossible (or perhaps foolish) only a few years ago. This project is linked to a DOE-Office of Science activity under the Basic Energy Sciences, Division of Materials Sciences. That activity, entitled "Design and Synthesis of Ultrahigh-Temperature Intermetallics," is conducted under the Center of Excellence for Synthesis and Processing (CSP) of Advanced Materials. The broad objective of the project is to generate the knowledge required to establish a scientific basis for the design and processing of transition metal silicides with improved metallurgical and mechanical properties at ambient and high temperatures. Its ultimate goal is to develop scientific

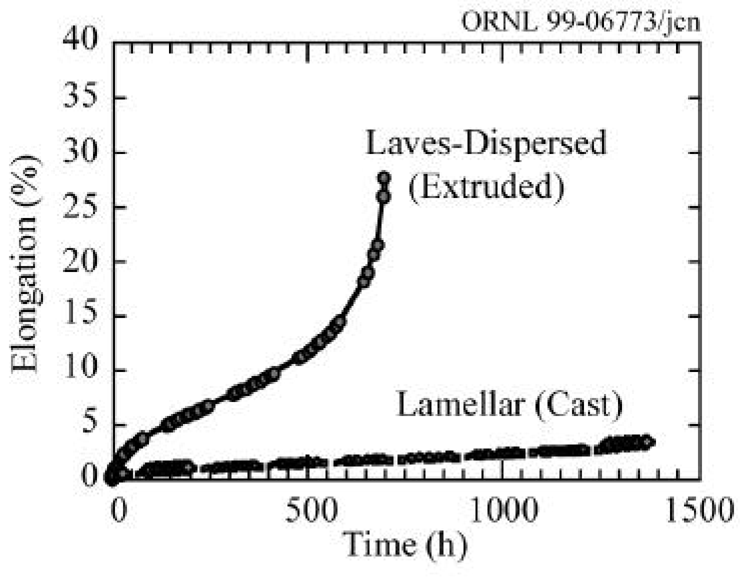

Fig. 10. Creep data obtained at 1000°C and 138 Mpa in humid air for Cr-8Ta-5Mo-0.5Ti-0.01Ce with Laves phase dispersed or lamellar microstructures. Note that the test of the lamellar microstructure alloy was stopped due to failure of the grip rods and plate

principles to design new-generation materials based on silicides for structural applications at and above 1400EC. The project involves Ames Laboratory, Argonne National Laboratory, Idaho National Engineering and Environmental Laboratory, Lawrence Berkeley National Laboratory, Lawrence Livermore National Laboratory, Los Alamos National Laboratory, ORNL, Sandia National Laboratory (California) and the University of Illinois Materials Research Laboratory (Judkins and Thompson 1997). The balance of the discussion here addresses work at ORNL, but it is clearly the combined expertise and efforts of the researchers at all the participating laboratories that will make success on this project possible.

Molybdenum silicides of particular interest at the present time are based either on the phase Mo_5Si_3 (also called T1) or on a-Mo. The Mo_5Si_3-based materials also contain the phases Mo_3Si and Mo_5SiB_2 (also called T2) and have compositions such as Mo-31Si-8B (at. %). While Mo_5Si_3 itself is not oxidation resistant, the B additions improve the oxidation resistance dramatically due to the formation of a protective borosilicate glass (Meyer, Kramer, and Akinc 1996a). These types of materials are best processed via powder-metallurgical routes (Meyer, Kramer, and Akinc 1996b, Schneibel et al. 1998). Attractive high- temperature creep strengths (e.g., $10^{-6}s^{-1}$ at 148 MPa and 1302°C; Meyer, Kramer, and Akinc 1996b) and flexure strengths (e.g., 500 MPa at 1200°C; Schneibel et al. 1998) have been reported. However, since the three phases present in the Mo_5Si_3-based materials are brittle, the fracture toughness is expected to be low.

The a-Mo-based materials contain the phases Mo_3Si and Mo_5SiB_2 (T2). They were originally developed by Berczik (1997). The phase relationships in these materials have been extensively investigated by Perepezko and co-workers (Nunes, Sakijda, and Perepezko 1997). A typical composition, which cann e

Significant progress has been made in improving those properties of ultrahigh temperature intermetallics based on the intermetallic phase-strengthened alloys, Cr (Nb)-Cr_2Nb, Cr(Ta)-Cr_2Ta, and the boron-containing alloys based on Mo_5Si_3 that would restrict them to being intellectual curiosities. The use of Cr(Ta)-Cr_2Ta for thermowells and other static components of slagging coal gasification and combustion now seems a near-term possibility. Hot-path components, for example, in advanced gas turbines is a longer-term, but very real possibility. Materials have been identified as a critical supporting technology for Vision 21 systems, and intermetallics will be an important element of that critical supporting technology because of their unique combination of properties.

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