

ODS IRON ALUMINIDES

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## ABSTRACT

Since oxide dispersion-strengthened (ODS) FeCrAl-based alloys have sufficient creep strength and good oxidation resistance at the very high temperatures of interest for the primary heat exchanger in advanced, closed-cycle gas turbine systems, they constitute viable alternative candidates to ceramics. A major life-limiting factor of these alloys is the ability to continue to form a protective scale of aluminum oxide, a factor proportional to the total amount of aluminum contained in the alloy. Fe<sub>3</sub>Al has oxidation resistance comparable to that of the FeCrAl-based alloys, and significantly superior sulfidation resistance. Also, because of its larger reservoir of aluminum, Fe<sub>3</sub>Al would be expected to exhibit longer lifetimes at the temperatures of interest. Since the strengthening effects of ODS processing are expected to confer similar high-temperature creep properties to those found for the FeCrAl-based alloys, ODS-Fe<sub>3</sub>Al is considered to have excellent potential for the very high-temperature heat exchanger application. The program effort on ODS Fe<sub>3</sub>Al includes examination of the properties of available ODS-FeCrAl alloys; development of mechanical alloying parameters for ODS-Fe<sub>3</sub>Al; determination of the effects of a dispersion of reactive element oxides on the high-temperature oxidation behavior of Fe<sub>3</sub>Al; and evaluation of methods for joining them.

## INTRODUCTION

There has been a recent increase of interest in advanced cycles that involve indirectly-fired gas turbines, in which coal- or gas-fired high-temperature heat exchangers are used to heat a working fluid in a closed system<sup>1</sup>. In a program conducted as part of the European COST-501 Concerted Action Project (Round 2, Work Package 4), available alloys based on FeCrAl-Y<sub>2</sub>O<sub>3</sub> (listed in Table 1) were evaluated for use in the main heat exchanger in a similar closed-cycle gas turbine application<sup>2,3,4,5,6,7,8,9,10,11,12</sup>. One of the currently available ODS FeCrAl alloys was found to meet the strength requirements for this application, in which the working fluid at 0.9 MPa (131 psi) flowing at 5,889 kg/hr (12,955 lb/hr) was to be heated from 800 to 1100°C (1472 to 2012°F) over a tube length of 4 m (13 ft).

FeCrAl-based alloys have long been regarded as having excellent resistance to high-temperature oxidation, with reactive metal or reactive metal oxide additions playing some beneficial role<sup>13,14,15</sup>. However, much of the information on oxidation behavior was generated from relatively short-term exposures. In the very high-temperature heat exchanger application, an area of unexpected

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but major concern with these alloys is their ability to maintain a protective oxide scale for the extended times necessary for commercial application. After extended exposure times, these alloys

Table 1. Nominal Compositions of Available Ferritic ODS Alloys (weight percent)

Alloy	Fe	Cr	Al	Mo	Ti	Y <sub>2</sub> O <sub>3</sub>
MA 956	Bal	20.0	4.5	—	0.5	0.5
PM 2000	Bal	20.0	5.5	—	0.5	0.5
ODM 751	Bal	16.5	4.5	1.5	0.6	0.5
ODM 331	Bal	13.0	3.0	1.5	0.6	0.5

suffer increasing loss of scale through spallation, as indicated by the trend to weight losses as shown in Fig. 1. The oxidation life of these alloys is determined by their ability to form or reform a protective alumina scale, and can be related to the time for the aluminum content of the alloy to be depleted to some minimum level; this, in turn, is a function of the total aluminum content of the alloy. This dependence on total available aluminum content is illustrated by results<sup>8</sup> from the COST-501 project as summarized in Fig. 2, which relates the effective life of the alloy tube (represented as 'time to breakaway') to its wall thickness.

The major factors that result in depletion of the reservoir of aluminum in the alloy are the inherent growth rate of the oxide, a process that consumes aluminum, and the tendency for the scale to spall, which also results in a net loss of aluminum from the alloy together with a (temporary) increase in oxide growth rate in the area affected by spallation. When the alloy aluminum reservoir falls below the minimum level at which an alumina layer can be reformed, less-protective oxides of the other alloy

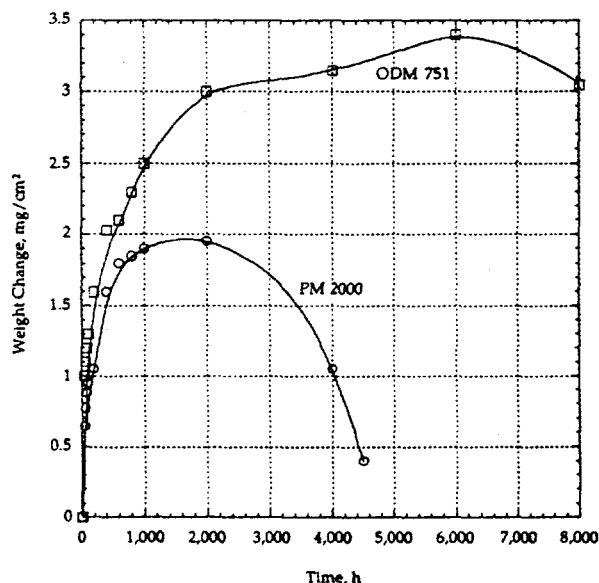


Figure 1. Oxidation kinetics for ODS-FeCrAl-Y<sub>2</sub>O<sub>3</sub> alloys in air at 1200°C cycled to room temperature every 48 hr (after ref. 8).

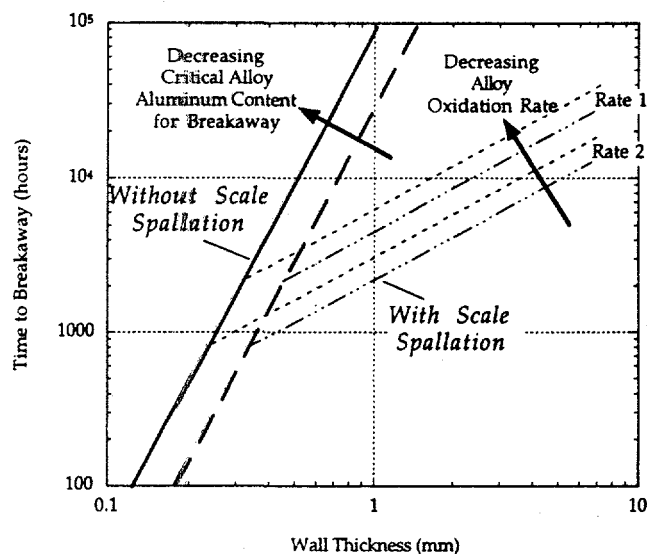


Figure 2. Effects of alloy oxidation rate, oxide spallation, and tube wall thickness on lifetime (time to onset of breakaway oxidation) of ODS-FeCrAl alloys (after ref. 8).

constituents will form in areas where the initial scale spalls, and the rate of oxidation may rapidly increase as the alloy undergoes breakaway oxidation. A further complication is that, according to results from the COST-501 project, differences among alloys that are insignificant for oxidation growth rate and tendency to spall can dramatically affect the time to breakaway. Hence, similar components manufactured from alloys of nominally similar compositions may fail at quite different times.

Iron-aluminum alloys have the obvious potential to provide longer service lives because of their inherently larger aluminum reservoirs (15.8 vs 3 to 5.5 wt.% Al), provided that their oxidation and mechanical properties are similar to the FeCrAl-based alloys. Iron-aluminum alloys have been regarded for many years as having excellent resistance to high-temperature oxidation<sup>16, 17, 18, 19</sup>, and this has been confirmed by the recent work at ORNL on Fe<sub>3</sub>Al alloys<sup>20</sup>. However, until this recent work, the development of usable ferritic alloys was restricted by their poor mechanical properties, especially the lack of ambient-temperature ductility which impaired fabricability. The lack of strength at temperatures above about 600°C is still a problem for the conventionally-cast alloys. The potential for increasing the creep rupture strength of iron aluminides by ODS processing was demonstrated in the 1960's<sup>21</sup>: the incorporation of 12 volume percent of approximately 1  $\mu\text{m}$  Al<sub>2</sub>O<sub>3</sub> to Fe-40 at. percent Al improved the high-temperature (732°C) creep strength by a factor of 4, compared to the dispersion-free, cast alloy.

Overall, there appears to be a niche application for an ODS version of  $\text{Fe}_3\text{Al}$  in components such as heat exchanger tubes that must operate at very high temperatures and for which the obvious competition is from ceramic materials. The work in this program is aimed at evaluating this potential.

#### PRELIMINARY EXAMINATION OF AVAILABLE FERRITIC ODS ALLOYS

A sample of an ODS FeCrAl alloy in the form of a round bar approximately 13 mm (0.5 in.) diameter was obtained from Dour Metal for characterization. Chemical analysis of the alloy showed that it was probably Dour Metal alloy ODM 331, rather than ODM 751 (see Table 1). The grain size of the as-received alloy was determined by metallographic techniques to be approximately  $2\text{ }\mu\text{m}$ , as shown in Fig. 3. Round specimens with a gage diameter of approximately 3 mm (0.12 in.) and a gage length of 12.7 mm (0.50 in.) were machined from the as-received bar, and subjected to creep-rupture testing in air. The first creep test at  $1000^\circ\text{C}$  ( $1832^\circ\text{F}$ ) and 55 MPa (8 ksi) failed in only a few minutes, before sufficient data could be recorded. A subsequent test at  $816^\circ\text{C}$  ( $1500^\circ\text{F}$ ) with a stress of 34.5 MPa (5 ksi) produced the data plotted in Fig. 4; the rupture-life was less than 5 hours, with a final elongation of 119.3 percent, and the minimum creep rate (MCR) was approximately 7.75%/h. This unexpectedly-low creep-rupture resistance was attributed to the small grain size of the as-received material.



Figure 3. Secondary scanning electron image of a transverse cross section of ODM-331 showing the small grain size and the presence of cavities and oxide particles.

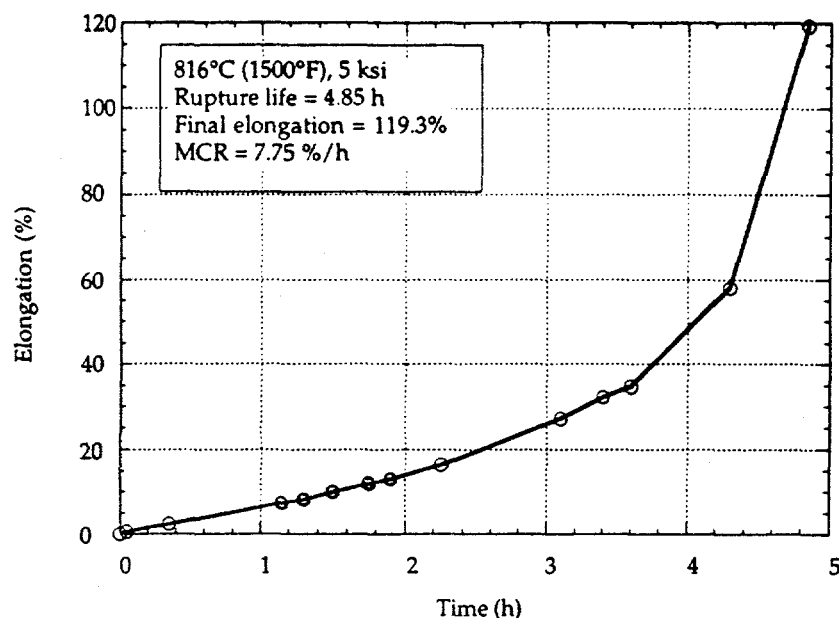


Figure 4. Creep-rupture curve for ODM-331 in the as-received and machined condition, tested at 816°C at a stress of 34.5 MPa (5 ksi).

In an attempt to produce larger, elongated grains to increase the creep strength, a heat treatment of 25 hours at 1350°C (2462°F) in vacuum was performed on a small sample of the alloy. However, no etchant could be found that would satisfactorily reveal the grain structure resulting from the heat treatment. A drawback of this heat treatment was obvious evaporation of chromium from the outer surface of the specimen, and an observed increase in the size of some of the oxide particles in the matrix. In the absence of a means of measuring the alloy grain size, further testing of this alloy was postponed.

A second series of tests was conducted on a sample of an ODS Fe<sub>3</sub>Al-based alloy produced at ORNL (designated FAS-3Y). The nominal composition of this alloy was Fe-28Al-2Cr-0.05B (at.%), to which 0.2 cation% Y<sub>2</sub>O<sub>3</sub> was added by mechanical alloying (see following section). Tensile specimens with gage dimensions of 12.7 x 0.75 x 3.2 mm (0.50 x 0.030 x 0.126 in.) were punched from the as-rolled sheet and heat treated for 8 h at 1275°C (2327°F) in argon to produce the large-grained banded grain structure shown in Fig. 5. The results of tensile tests conducted in air at room temperature, 800, and 1000°C (1472 and 1832°F) using a strain rate of  $3.3 \times 10^{-3} \text{ s}^{-1}$  are shown in Table 2. Typical data for monolithic FAS, two other monolithic ORNL-developed iron aluminide alloys, and the Dour Metal alloy ODM-331, are included for comparison. At room temperature, the FAS-3Y alloy had a similar tensile strength but a lower ductility compared to the FA-180 alloy, as

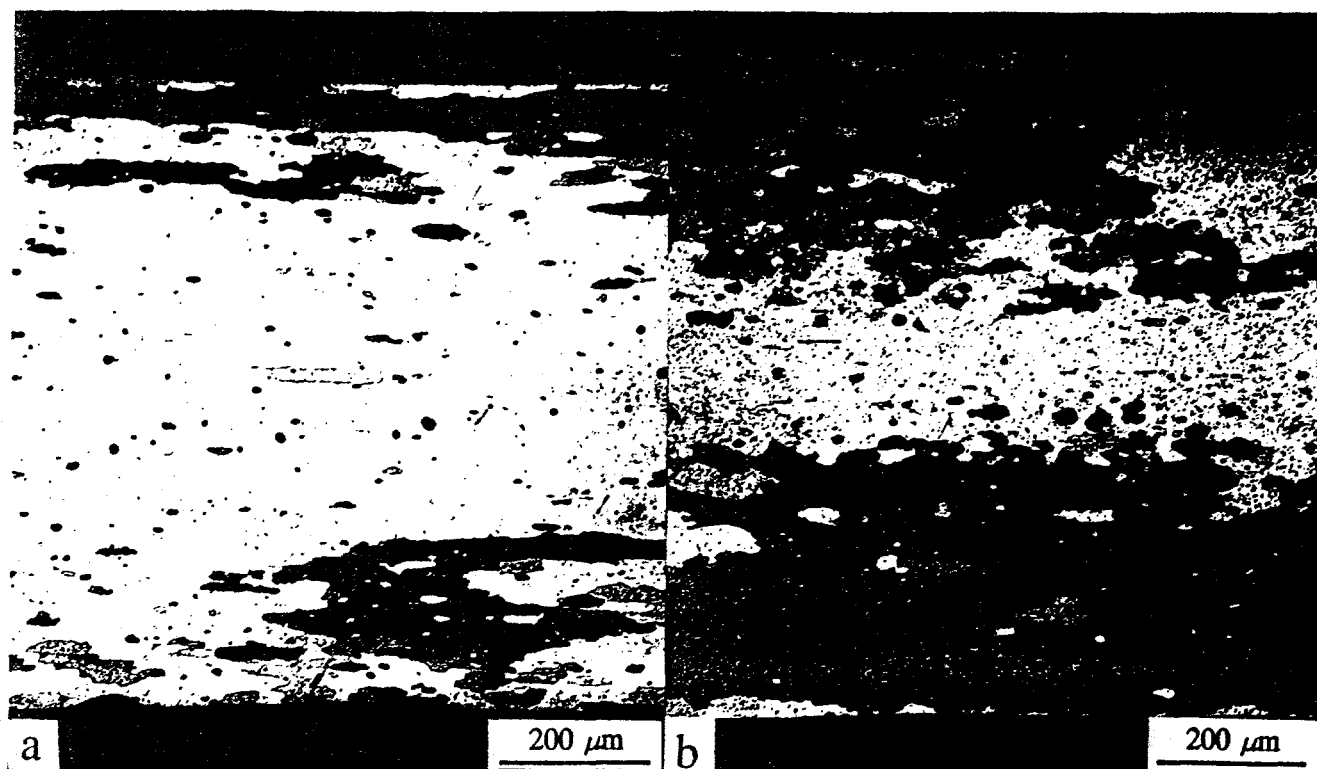


Figure 5. Optical micrographs of FAS-3Y after a heat treatment of 8 h at 1275°C in air; shown is the through-thickness direction (a) parallel to and (b) perpendicular to the rolling direction.

Table 2. Comparison of Tensile Properties of Alloy FAS-3Y With Other Iron-Based Alloys

Property	FAS-3Y	FAS	FA-129	FA-180	ODM-331
<u>20°C</u>					
YS (MPa)	643	394	380	620	
UTS (MPa)	959	679	900	900	
Elongation (%)	2	9	15	8	
<u>800°C</u>					
YS (MPa)	110			140	
UTS (MPa)	120			148	
Elongation (%)	36			79	
<u>900°C</u>					
YS (MPa)					160
UTS (MPa)					170
Elongation (%)					10
<u>1000°C</u>					
YS (MPa)	43			27	
UTS (MPa)	49			27	
Elongation (%)	12			137	

FA-129 = Fe-28Al-5Cr-0.5Nb-0.2C (at.%)

FA-180 = Fe-28Al-5Cr-0.5Nb-0.8Mo-0.025Zr-0.05C-0.005B (at.%)

expected for an oxide-dispersion-strengthened alloy. The FAS-3Y specimen failed in a ductile-dimple manner, with particles at the base of the dimples. Since tensile properties are usually better in iron aluminides with finer-grained microstructures, it is anticipated that the strength of this alloy will improve with heat treatment at a lower temperature.

A creep-rupture test of this alloy was initiated at 800°C (1472°F) with an initial stress of 34.5 MPa (5 ksi). At this stress, the minimum creep rate appeared to be almost zero. After approximately 240 hours, the stress was increased to 48.3 MPa (7 ksi) and the test was allowed to proceed to rupture. As shown in Fig. 6, rupture occurred suddenly after 383.45 hours, at a specimen elongation of about 3 percent. Another test at 1000°C (1832°F) started at 13.8 MPa (2 ksi). Since there was no discernible creep after 240 hours, the stress was increased to 20.7 MPa (3 ksi), and then to 27.6 MPa (4 ksi) before failure occurred after 477 hours, with a final elongation of 6.9 percent. Further creep tests on FAS-3Y will be initiated following the fabrication of a new sample of FAS-3Y material and the development of thermomechanical processing to produce the desired large, elongated grain structure.

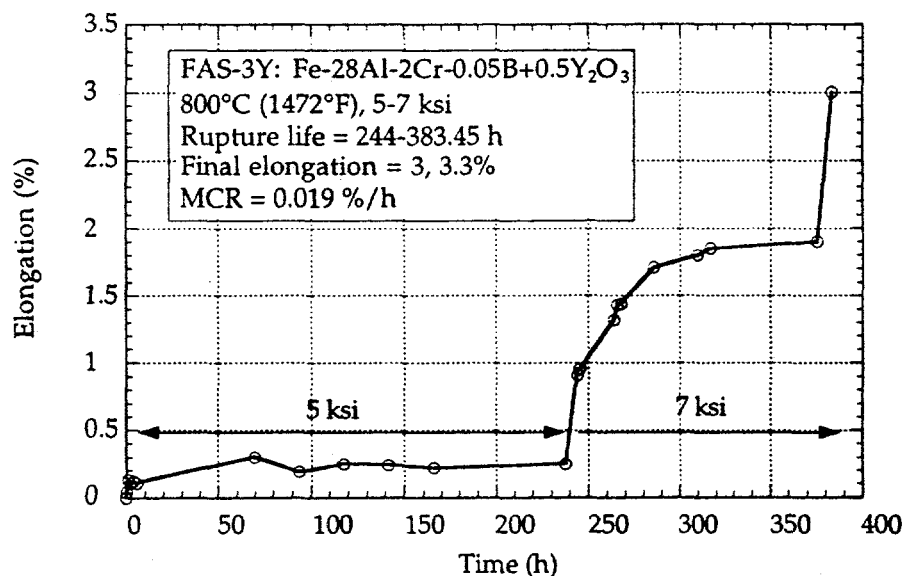


Figure 6. Creep-rupture curve of FAS-3Y heat treated 8 h at 1275°C and tested at 800°C.

The limited creep-rupture data generated so far on the FAS-3Y material suggests a lower creep resistance than the ODM alloys produced by Dour Metal. However, with the limited number of samples available, the best fabrication and heat treating conditions to produce the desired



microstructure for maximum strength have not been determined. Plans have been made to obtain a larger sample of this alloy from a commercial vendor.

#### DEVELOPMENT OF MECHANICAL ALLOYING PARAMETERS FOR ODS Fe<sub>3</sub>Al

Initial investigation of mechanical alloying parameters used a Svegvari Type 01STD attritor mill (purchased from Union Process Inc., Akron, Ohio), which has a capacity of 0.75 litres (0.26 ft<sup>3</sup>). This mill was top-loaded (and unloaded) and was fitted with a water-cooled lid and paddle bearing. A typical charge consisted of 150 g (0.33 lb) of powder, and 1500 g (3.3 lb) of stainless steel balls (powder:ball ratio of 1:10). Prealloyed Fe<sub>3</sub>Al (FAS) powder was used, together with reactive metal oxide powder which was obtained with an average particle size of less than 1 µm. The FAS powder was obtained from Ametek, and consisted of spherical particles having a particle size range of 150 µm to 45 µm, (mesh size -100/+325). In use, the loaded and assembled mill was purged with argon for 5 min. prior to the start of milling; a continuous flow of argon was maintained during milling. At the end of a run, the lid was removed and the cooled powder exposed to the ambient laboratory air. The effect of increasing milling time was evaluated through examination of the powder size and shape by scanning electron microscopy; the extent of mechanical alloying was assessed by light microscopy of metallographically-prepared samples of the powder. The mechanical alloying process incorporates the oxide dispersoid into the alloy structure by a repetitive process of welding and smearing of the alloy powder particles onto the mill walls and the balls, followed by detachment of fragments of the powder. A layered structure is developed which contains entrapped particles of the dispersoid; with time, the layers become sufficiently fine that the alloy powder particles attain a homogeneous composition, with a uniform distribution of the dispersoid particles. Figure 7 shows the change in powder particle size as a function of milling time. The most obvious initial effect of milling is flattening of the original spherical particles into discs, which assume irregular shapes with increased milling time. Further milling results in the transformation from flattened plates to agglomerates of much smaller particles. Once the powder structure observed after 50 hours of milling is attained, further milling produces little alteration in alloy structure or actual particle size.

The milling parameters chosen for the processing of powder intended for subsequent consolidation and preparation of test specimens were 500 rpm for 48 hours. A larger Svegvari attritor mill, Type 01HD with a capacity of 1.4 litres (0.49 ft<sup>3</sup>), was used to prepare these materials. The milling conditions were the same as those used in the smaller mill. The amount of powder per batch was increased to 300g (0.66 lb) and the larger mill is capable of running 24 hours per day.

A search was made for facilities capable of producing larger batches of mechanically-alloyed powder, of the order of several kilograms to commercial alloy-production sizes. The following possible sources were identified: INCO, Dour Metal of Belgium; PM Hochtemperatur-Metall of Germany; AEA Technology of Risley, U. K.; and CRM of Italy. INCO (Huntington, West Virginia) uses ball milling to produce Ni-based ODS-alloys only and has no current capability that could address the needs of the iron aluminide program; there was also concern about contamination of any mill that was used. INCO International (Hereford, U.K.) produces only Fe-based ODS alloys, but it also has no available mill capacity. Dour Metal has produced tubes of ODS-FeCrAl-Y<sub>2</sub>O<sub>3</sub> using in-house

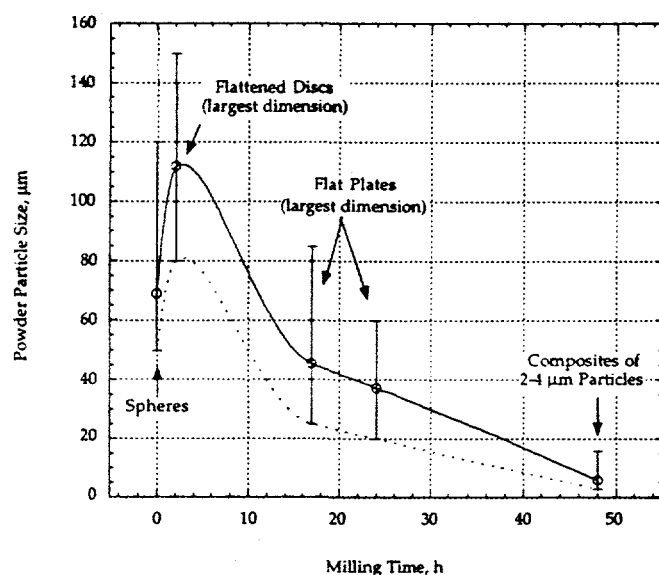


Figure 7. Plot of the average particle size of FAS-3Y alloy powder as a function of milling time.

processing that starts with ball milling. They also have made an ODS version of Fe-40 at.% Al using alumina as the dispersoid. PM Hochtemperatur-Metall uses ball milling to produce ODS-FeCrAl-Y<sub>2</sub>O<sub>3</sub> on a commercial basis, but has not made tubing. AEA, Risley, U.K. has a small-capacity centrifugal mill of new design; this equipment has the potential for better processing control than conventional attritor or ball milling, but is as yet not available in a commercial size. CRM has a small-capacity ball mill, and has been involved with ODS-Fe-40Al. Arrangements currently are being made to work with both Dour Metal and PM Hochtemperatur-Metall to produce several 20 kg (44 lb) batches of ODS-Fe<sub>3</sub>Al powder from prealloyed FAS powder made by Ametek corp. The mechanically-alloyed powder will be used by ORNL to optimize the thermomechanical processing required to produce the required grain size and structure and to measure the properties relevant to the envisioned application.

## EFFECTS OF A DISPERSION OF OXIDES ON THE HIGH-TEMPERATURE OXIDATION BEHAVIOR OF $\text{Fe}_3\text{Al}$

Mechanically-alloyed powders of  $\text{Fe}_3\text{Al}$  containing additions of the following reactive element oxides:  $\text{Y}_2\text{O}_3$  (0.05, 0.1, 0.2, 0.3, 0.5 cation %),  $\text{ZrO}_2$  (0.2 cat. %),  $\text{La}_2\text{O}_3$  (0.05, 0.2 cat. %),  $\text{Al}_2\text{O}_3$  (0.2 cat. %),  $\text{CeO}_2$  (0.2 cat. %),  $\text{Nd}_2\text{O}_3$  (0.2 cat. %), and  $\text{Yb}_2\text{O}_3$  (0.2 cat. %) were made at ORNL using nitrogen-atomized prealloyed FAS powder in the Svevari 01HD mill. These powders were canned in mild steel, and extruded at  $1050^\circ\text{C}$  ( $1922^\circ\text{F}$ ) with a reduction ratio of 12:1 to produce rods approximately 25 mm (1.0 in.) in diameter. After decanning, disc-shaped specimens were cut from these rods and subjected to oxidation testing with no further heat treatment, that is, with a relatively small alloy grain size. The specimen surfaces were polished with  $0.3\ \mu\text{m}$  ( $12 \times 10^{-6}$  in.) alumina and washed before exposure. The details of the test procedures and some of the cyclic oxidation results are presented elsewhere<sup>22</sup>. Figures 8 and 9 summarize the kinetics of isothermal oxidation in air, and cyclic oxidation in oxygen, respectively, both at  $1200^\circ\text{C}$  ( $2192^\circ\text{F}$ ), for the FAS alloy with and without a dispersion of  $\text{Y}_2\text{O}_3$ . Ignoring the initial rapid

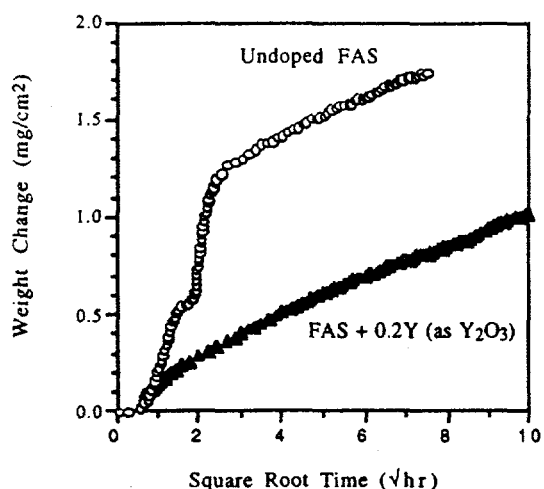


Figure 8. Comparison of the isothermal oxidation kinetics of conventionally-produced and ODS-FAS (FAS-3Y);  $1200^\circ\text{C}$  in oxygen.

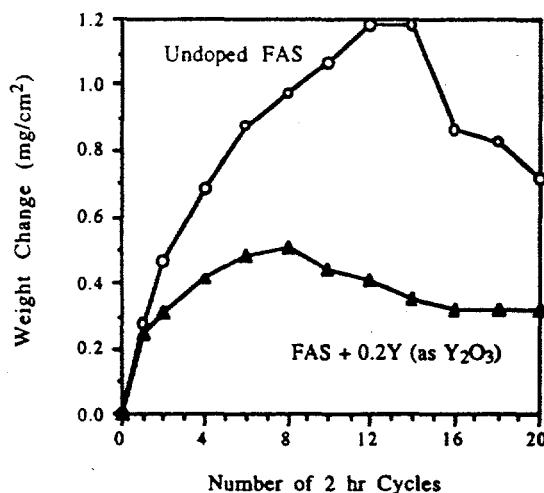


Figure 9. Comparison of the cyclic oxidation kinetics of conventionally-produced ODS-versions of Alloy FAS (FAS-3Y);  $1200^\circ\text{C}$  in air.

oxidation of the undoped alloy during the first 4 hours of isothermal exposure, the oxidation rates of the two alloys at longer times appear quite similar; both alloys exhibited oxidation rates that are slightly slower than parabolic, suggesting that the oxide scales become more protective with time. In the cyclic oxidation test, the weight gain of the ODS alloy was very similar to that in the isothermal test up to approximately 16 hours (and obviously slower than that of the undoped alloy), after which it

apparently suffered some degree of scale spallation. The reason for the increased weight gain of the undoped alloy in this test was because it suffered from scale spallation at the end of each cycle. The areas over which spallation occurred were extremely small and, since the spalled scale did not fully detach, a net increase in weight was measured up to the 14th cycle, or 28 hours, after which actual scale loss apparently occurred.

Figures 10 and 11 compare the isothermal and cyclic oxidation behavior of the FAS-3Y alloy with a commercially-available ODS-FeCrAl-Y<sub>2</sub>O<sub>3</sub> alloy, MA956 (see Table 1) at 1200°C (2192°F) in air, and in oxygen, respectively. The kinetics of isothermal oxidation to 100 hours appear essentially

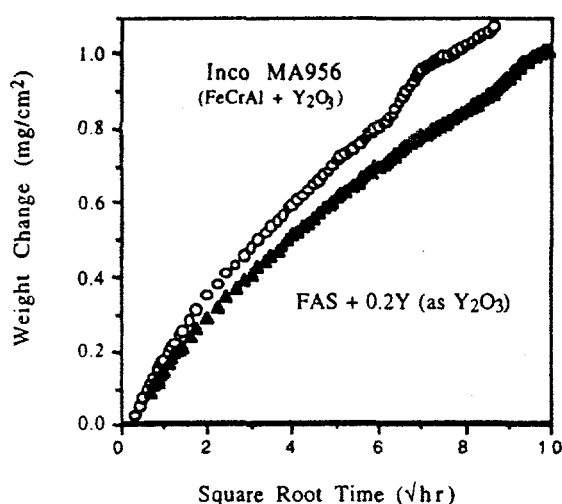


Figure 10. Comparison of the isothermal oxidation kinetics of ODS-FAS (FAS-3Y) with a commercial ODS-FeCrAl-Y<sub>2</sub>O<sub>3</sub>; 1200°C in air.

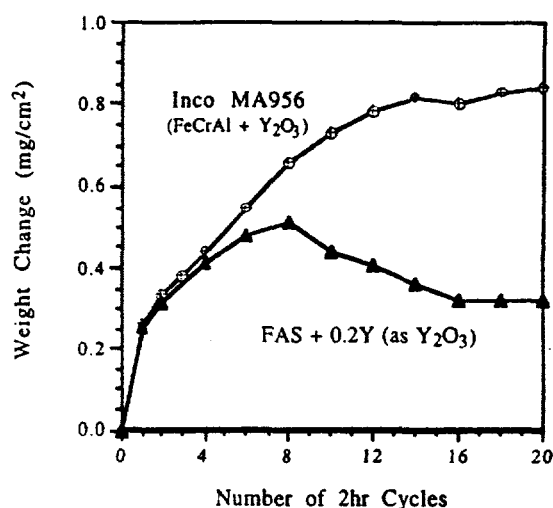


Figure 11. Comparison of cyclic oxidation kinetics of ODS-FAS (FAS-3Y) with a commercial ODS-FeCrAl-Y<sub>2</sub>O<sub>3</sub>; 1200°C in oxygen.

the same, as might be expected for alloys that are intended to form a protective alumina scale. Under isothermal conditions, the rate of consumption of aluminum to form the protective scale would be very similar for the two alloys. In the cyclic oxidation test, the weight gain of the MA956 after 40 hours was almost the same as that measured in the isothermal test, indicating no scale spallation. As noted above, the weight gains measured for the FAS-3Y indicated some scale spallation. This is a surprising result, since a dispersion of a reactive element oxide with the appropriate particle size and distribution generally has been found to improve the overall scale adherence on alumina-forming alloys. Such spallation would obviously increase the rate of depletion of the aluminum reservoir of the FAS-3Y alloy. Although this reservoir is much larger than that in alloy MA956, the rate of spallation must be minimized to maintain the potential service lifetime advantage of FAS-3Y<sup>22</sup>.

There are several possible reasons for the difference in scale spallation behavior between the two alloy types, ranging from inhomogeneity in FAS-3Y to a fundamental difference in the functioning of the reactive element effect in  $\text{Fe}_3\text{Al}$  compared to  $\text{FeCrAl-Y}_2\text{O}_3$ . The possibility that there are inherent differences in scale spallation behavior between FeCrAl-based and iron aluminide-based alloys<sup>20</sup> and the effects of different types and levels of reactive element oxides on the oxidation behavior of  $\text{Fe}_3\text{Al}$  are the subject of ongoing research.

## SUMMARY AND CONCLUSIONS

Heat exchangers for cycles which require a working fluid to be heated to temperatures of 1100-1200°C (2012-2192°F) provide a potential application in which the exceptional high-temperature oxidation and sulfidation resistance of iron aluminides can be exploited. The alternative materials choices for such an application are ODS-FeCrAl-based alloys and ceramics. An ODS-version of  $\text{Fe}_3\text{Al}$  has the obvious advantage over ceramics that it has metallic characteristics, and is expected to exhibit a significantly longer service life than ODS-FeCrAl alloys. Creep tests with a non-optimized ODS- $\text{Fe}_3\text{Al}$  indicated significantly improved strength compared to non-ODS iron aluminides, but lower creep resistance compared to a commercial ODS-FeCrAl alloy. The isothermal oxidation rate of the ODS- $\text{Fe}_3\text{Al}$  was essentially the same as that of a commercial ODS-FeCrAl alloy at 1200°C (2192°F), but there was evidence of inferior resistance to cyclic oxidation. The main issues to be resolved are the ability of optimized ODS processing of  $\text{Fe}_3\text{Al}$  to achieve an equivalent improvement in high-temperature creep strength to that found with ODS-FeCrAl alloys and a method for improving the resistance to scale spallation of  $\text{Fe}_3\text{Al}$ .

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