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FACTORS AFFECTING CORROSION RESISTANCE OF RECUPERATOR ALLOYS

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ABSTRACT

As microturbine recuperators are needed to operate at higher temperatures in order to achieve higher engine operating efficiencies, a recurring problem encountered is a severe degradation in environmental resistance of type 347 stainless steel. Above 600°C, stainless steels can experience accelerated attack caused by water vapor, which is often present as a combustion product. Nickel-base alloys are less susceptible to this accelerated attack but their cost is prohibitive. In order to provide a clear, fundamental understanding of alloy composition effects on corrosion resistance of stainless steel components used in recuperators, the oxidation behavior of model alloys is being studied. A composition range of Cr and Ni contents has been identified with better corrosion resistance than type 347 stainless steel. Finer-grained alloys showed improved corrosion resistance compared to coarse-grained alloys with the same composition. It also has been demonstrated that minor alloy additions of Mn and Si are beneficial to corrosion resistance in these environments. This type of information will aid in the development of a corrosion-resistant and cost-effective recuperator material for operation at 650°-700°C.

INTRODUCTION

One goal of the U.S. Department of Energy's Distributed Energy Resources program[1,2] is to improve the efficiency of small (25-200kW) gas turbines or microturbines.[3-4] These small engines have relatively low (<30%) efficiencies compared to larger turbines which use higher turbine inlet temperatures. A common strategy to increase their efficiency is to add a recuperator to use exhaust heat to preheat the inlet air to the combustor.[3-6] Several recuperator designs are being tested but most employ some type of corrugated metal foil in order to maximize heat transfer. Traditionally, primary surface recuperators use type 347 stainless steel foil which offers an excellent combination of creep strength and corrosion resistance at a reasonable cost. However, creep and corrosion limit the recuperator operating temperature to 600°C (1100°F). Despite efforts to improve the creep strength of type

347 stainless steel,[7,8] this material is still subject to accelerated corrosion attack in exhaust gas at 650°C due to the presence of water vapor.[9,10] As a combustion product, water vapor has long been known as a potential problem for both metals and ceramics.[9-18] The effect of water vapor is being widely studied but there is no widely accepted mechanism for its role in reducing the corrosion resistance of chromia-forming stainless steels. Therefore, in order to increase the recuperator operating temperature to further increase the microturbine's efficiency, new materials will be needed.

Previous work has looked at Ni-base and alumina-forming alloys for primary surface recuperators[19,20]. However, these alloys may be prohibitively expensive for microturbines trying to compete with low-cost reciprocating engines. Thus, one current research focus is to develop an improved stainless steel composition with better corrosion resistance while maintaining some degree of creep resistance. The objective of this paper is to outline some ongoing work in examining the effects of Cr and Ni contents, minor alloy additions such as Mn and Si, and alloy grain size on the corrosion resistance of model alloys.

EXPERIMENTAL PROCEDURE

The chemical compositions of selected alloys are listed in Table I. The model alloys typically had impurity levels of 10-20ppm C, 10-20ppm N and 35-45ppm S. The model alloys were vacuum induction melted at ORNL and hot and cold rolled to 1.25mm sheet. After the final cold rolling step, the sheets were annealed in Ar for 2min at 1000°C for the austenitic alloys and 2min at 900°C for the ferritic alloys. The austenitic alloys generally had a much smaller grain size than the ferritic alloys, Table I, typical of that found in foils. Foil specimens (12mm x 18mm x 100µm) were tested in the as-rolled conditions and similar-sized sheet specimens were polished to a 600 SiC grit finish. The specimens were cleaned in acetone and methanol prior to oxidation and mass changes were measured using a Mettler-Toledo model AG245 balance. Exposures were either 1h cycles at 700°C or 100h cycles at 650°, 700° or 800°C.

Oxidation exposures in humid air were conducted by flowing the

Table I. Alloy chemical compositions (weight %) and average grain sizes (μm).

	Cr	Ni	Mn	Si	Other	Grain Size (μm)
Type 347	17	11	1.6	0.6	0.6Nb	40
20/25/Nb	20	25	1.0	0.4	1.6Mo, 0.14Nb	10
Fe-16Cr-15Ni	15.8	14.8	<	<		n.d.
Fe-16Cr-20Ni	15.6	19.5	<	<		19
Fe-20Cr-15Ni	19.4	15.3	<	<		n.d.
Fe-20Cr-20Ni	19.7	20.1	<	0.01		14
Fe-20Cr-30Ni	19.6	30.1	<	0.01		13
Fe-16/15+MS	15.9	14.9	1.74	0.24		~15
Fe-16/20+MS	15.8	19.7	1.72	0.24		~15
Fe-20/15+MS	19.8	14.9	1.70	0.24		~15
Fe-20/20+MS	19.8	19.8	1.69	0.25	0.01Ce	~15
Fe-20/20+Etc	20.9	20.8	>2	0.24	Nb	n.d.
Fe-12Cr	12.6	<	<	<		81
Fe-16Cr	15.7	<	<	<		97
Fe-20Cr	19.6	<	<	<		84
Fe-16Cr+Si	16.1	<	<	0.17		100
Fe-16Cr-1Mn	16.0	0.01	1.1	0.01	<	88
Fe-16Cr+TiB ₂	16.1	<	<	<	0.3Ti, 0.1B	~20

< indicates below the detectability limit of <0.01%

gas at 450cc/min through an alumina tube that was inside a resistively-heated tube furnace. Distilled water was atomized into the flowing gas stream above its condensation temperature and heated to the reaction temperature within the alumina tube. Water was collected and measured after flowing through the tube to calculate its concentration and calibrate the amount of injected water. A water content of 10 vol.% was used for these experiments. For testing in air, the alumina furnace tubes were not sealed. For 1h cycle tests, specimens were attached with Pt-Rh wire to alumina rods. For 100h cycle tests, up to 40 specimens were positioned in alumina boats in the furnace hot zone so as to expose the specimen faces to the flowing gas. After oxidation, selected specimens were Cu-plated and sectioned for metallographic analysis.

RESULTS

Foil specimens

To demonstrate the extent of accelerated attack observed on type 347 in air + 10vol.%H₂O, foil specimens were exposed in 100h cycles at 650°-800°C, Figure 1. Two types of foil were exposed, one rolled at ORNL with relatively coarse grains (40 μm) and a commercial product made by Allegheny-Ludlum with a smaller grain size (10 μm). At each temperature, the coarse-grained alloy immediately exhibited accelerated mass gains. Figures 2a and 2b show cross-sections of the foils after 1000h at 650° and 800°C, respectively. At 650°C, regions of thicker Fe-rich oxide can be seen along with thinner regions that are primarily Cr-rich. At 800°C, a thick duplex oxide has consumed a large fraction of the metal foil with the outer layer being primarily iron oxide and the inner layer containing a mixture of Fe and Cr, Figure 2b.

In contrast to the performance of the coarse-grained foil, the fine-grained foil showed a delayed response or incubation period with relatively low mass gains prior to the onset of accelerated attack at 650°C and 800°C. However, when accelerated attack occurred, it was very similar in extent to that observed for the coarse-grained foil. Thus while there was a delay in onset there was no significant difference in

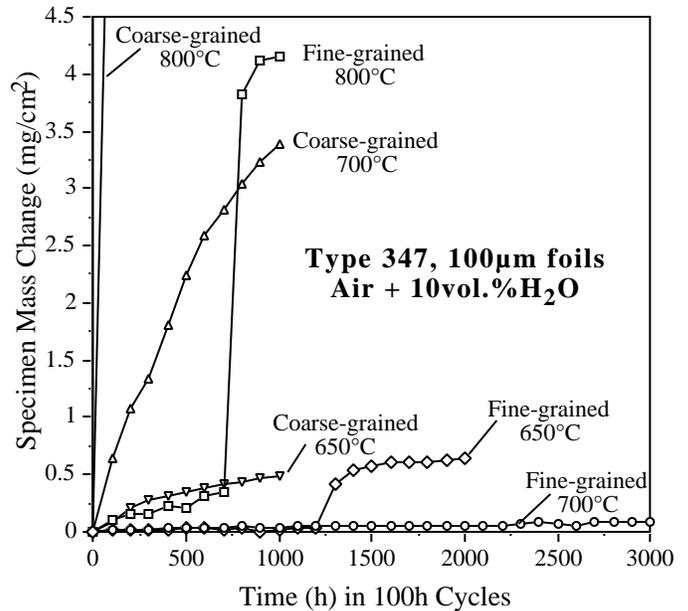


Figure 1. Mass change of stainless steel foils during 100h cycles at 700° and 800° in dry air or air plus 10% water vapor.

the degree of accelerated attack when it occurred. At 700°C, the fine-grained commercial foil has shown no accelerated attack after 3000h of exposure in humid air. The results for foil materials point to a role of alloy microstructure as well as composition on oxidation resistance under these conditions.

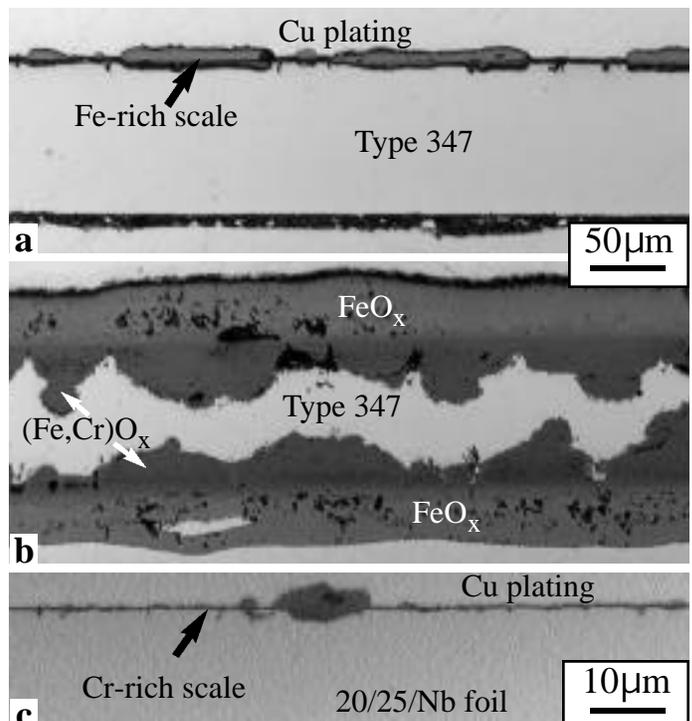


Figure 2. Light microscopy of polished cross-sections of ORNL-rolled 100 μm foils: Type 347 after 1000h (10, 100h cycles) in humid air at (a) 650°C and (b) 800°C; (c) 20/25/Nb foil after 1000h at 700°C.

Role of Cr and Ni contents

The first series of model alloys contained various Cr and Ni contents. They were tested in both the as-cast and rolled forms. Rolling the alloys resulted in a much finer grain size (Table I) than the large grains (>100µm) of the casting. Testing in dry and humid air was performed at 650°-800°C in order to isolate the role of Cr and Ni on resistance to accelerated attack (AA). Previous corrosion testing on stainless steels had suggested that both the Cr and Ni contents affected corrosion resistance but only a few compositions had been examined.[9] More detailed results of these studies are being reported elsewhere.[21,22]

Figure 3 gives a general summary of the results of this study found for exposures in air + 10%H₂O at 700°C. The dashed lines in Figure 3 denote boundaries between protective oxide scale (POS) formation and accelerated attack (AA) for as-cast, coarse-grained specimens and rolled, fine-grained specimens of the same composition. Figure 2c gives an example of a thin POS, similar to the scale formed in dry air. As with the foil materials, alloy grain size was a significant factor and had a major effect on the critical Cr and Ni contents needed to prevent AA. The fine-grained specimens formed a POS at much lower Cr and Ni contents than the coarse-grained specimens. This effect of grain size is attributed to the importance of short-circuit Cr diffusion in these alloys[23] which may prevent Cr depletion at the metal-scale interface. This effect appears to be especially important at 650°-700°C but plays less of a role at 800°C.[24]

The positive slope of the boundary lines indicates that increasing the Ni content has a beneficial effect on improving resistance to AA. To improve the performance of type 347 stainless steel, merely increasing the Cr content would likely reduce the creep strength and lead to sigma phase formation which could embrittle the foil.[8] These results indicate that modest increases in both the Cr or Ni contents could lead to a more corrosion resistant composition.

Role of Minor Alloying Additions

Minor alloying additions can have a major effect on oxidation performance. In order to investigate the relative importance of minor additions on the formation of chromia scales in humid air, additions were made to a base Fe-16Cr ferritic alloy. (A ferritic base alloy that experiences AA was chosen to simplify the study.) Figure 4 provides a

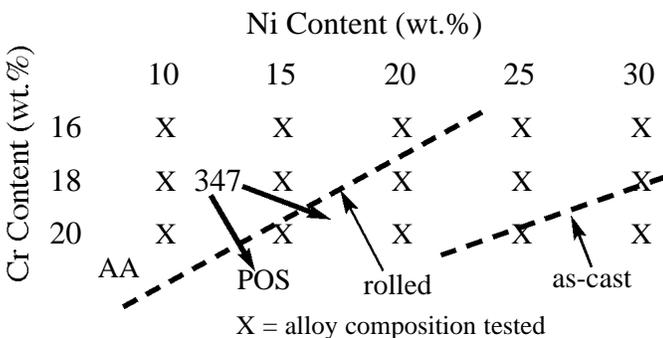


Figure 3. Schematic of the performance of the model Fe-Cr-Ni alloys at 700°C in 10% H₂O showing the composition line between the formation of a protective oxide scale (POS) and accelerated attack (AA) for the coarse-grained, as-cast specimens and the fine-grained, rolled specimens of the same composition.

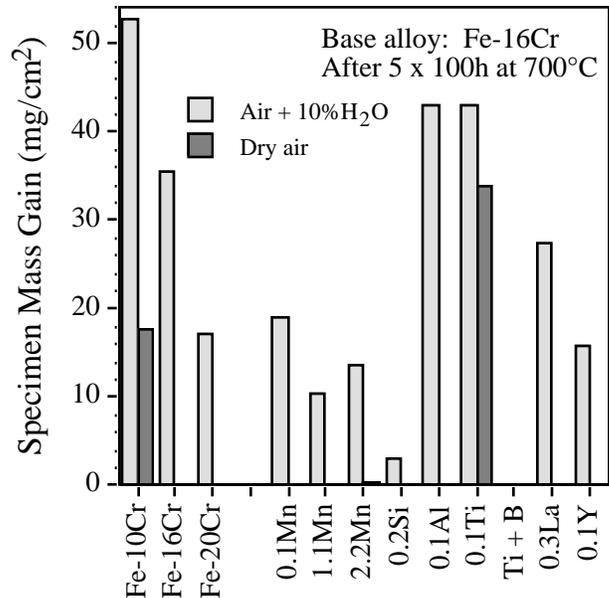


Figure 4. Specimen mass gains for ferritic model alloys after 5, 100h cycles in dry and humid air at 700°C. The minor alloy additions were made to a base alloy of Fe-16Cr.

summary of the results of this study at 700°C. In dry air, most of the model alloys (except for Fe-10Cr and Fe-16Cr-0.1Ti) showed only small mass gains after 5, 100h cycles at 700°C. In humid air, most of the alloys showed AA. For reference, the performance of three binary Fe-Cr alloys from 10-20%Cr is shown. Additions of Al, Ti and La to Fe-16Cr showed similar levels of accelerated attack as the base alloy. Various levels of Mn were added to Fe-16Cr because of a study which suggested Mn reduced the evaporation of Cr₂O₃. [25] The alloys with Mn showed a slight drop in total mass gain but AA was still observed at this temperature. The alloy with 0.2Si showed a stronger beneficial effect with a much lower mass gain than the base alloy, Figure 4. Previous work also has indicated a strong beneficial effect of Si under similar environments.[16,26]

Most of the additions had only a minimal effect on the alloy grain size except for the TiB₂ addition (Table I) and this alloy also had the lowest mass gain after 500h at 700°C in humid air with no indication of accelerated attack, Figure 4. Titanium and boron were added to pin alloy grain boundaries in ferritic alloys.[27] This strategy was successful as the alloy grain size of this material (Figure 5b) was significantly less than the base alloy, (Figure 5a) or the other ferritic model alloys, Table I. This alloy also showed excellent behavior during 1h cycles at 700°C in humid air, Figure 6. Most of the alloys underwent AA in less than 100 cycles. The alloy with Si showed AA after 100h but the alloy with TiB₂ showed a POS after 500 cycles. The slight mass loss during the test was attributed to the evaporation of CrO₂(OH)₂. [28] The rate of mass loss was identical to that of an oxide dispersion strengthened Fe-13Cr-3W alloy which has a fine subgrain structure and excellent oxidation resistance.[29,30] Again alloy grain size appeared to have a dominant effect, but Si and possibly Mn appeared to be the most promising minor alloying additions.

Mn and Si Additions in Austenitic Alloys

The most recent work has been to focus testing on alloys in the 16-

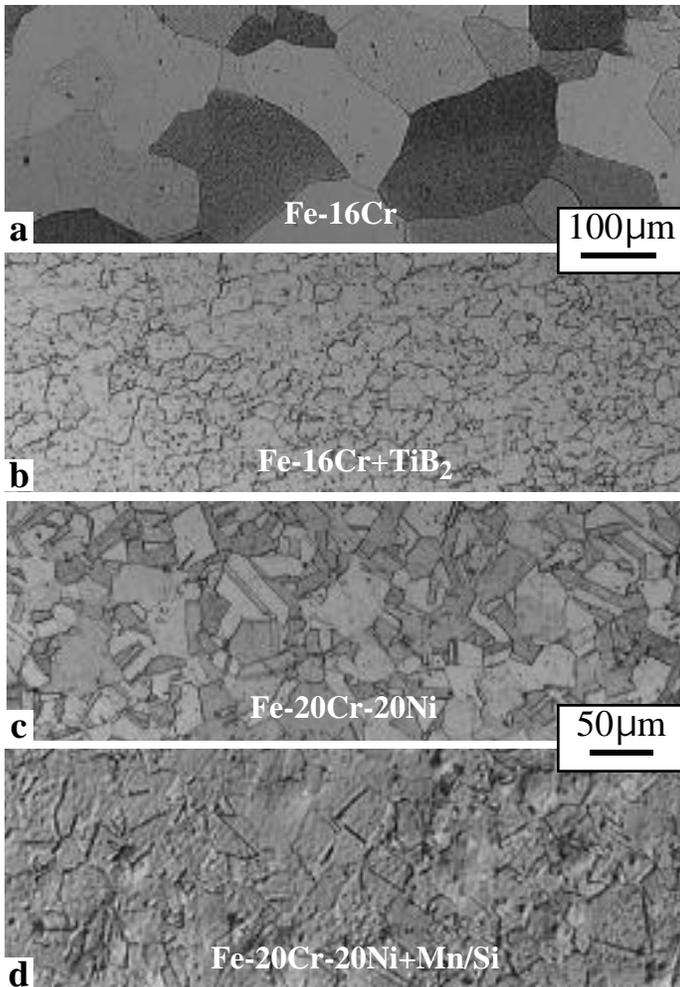


Figure 5. Light microscopy of rolled model alloys (a) Fe-16Cr (b) Fe-16Cr + TiB₂, (c) Fe-20Cr-20Ni and (d) Fe-20Cr-20Ni+Mn/Si.

20%Cr and 15-20%Ni range. These compositions have only a modest increase in alloying elements compared to type 347 stainless steel and thus should represent only a modest cost increase. Additions of Mn and Si were made to alloys in this composition range and short-term testing performed in humid air at 650°-800°C. Figure 7 shows specimen mass changes during 100h cycles at 650°C in humid air. Without the addition of Mn and Si, the rolled and cast specimens show AA. The mass losses indicate that the thick, Fe-rich oxide spalled at the end of each cycle. The mass losses expected from scale evaporation are much lower, e.g. Figure 6. Mass gains for the Fe-20Cr-20Ni specimens indicate that the Fe-rich oxide did not spall from these specimens. In general, the as-cast (dashed line) specimens showed similar behavior as the rolled (solid line) specimens, indicating that alloy grain size had less of an effect at this temperature. The specimens with Mn and Si additions (labeled +MS in Figure 7) performed significantly better than the specimens without these additions. The specimens with Mn and Si were only tested in the rolled condition. The beneficial effect of Mn and Si cannot be attributed to an alloy grain size effect as all of the rolled specimens had similar grain sizes. For example, Figures 5c and 5d show a similar grain size for Fe-20Cr-20Ni with and without the additions. All of the specimens with Mn and Si showed small mass

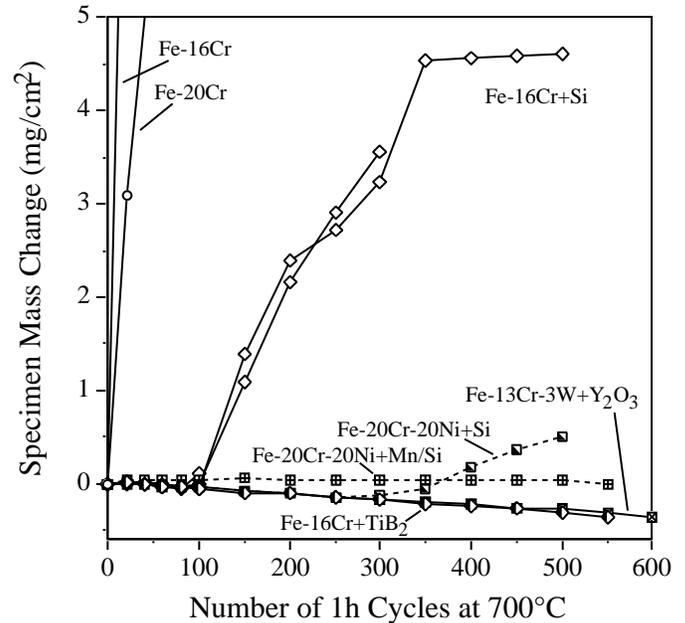


Figure 6. Specimen mass change for model ferritic and austenitic alloys during 1h cycles at 700°C in air +10%H₂O.

changes indicating that a POS formed except for the Fe-16Cr-15Ni+Mn/Si specimen, which initially showed a mass gain but then began to lose mass. This behavior was significantly better than the Fe-16Cr-15Ni specimens without additions indicating some benefit of the Mn and Si additions. This specimen contained the lowest Cr and Ni contents in this group, indicating that the beneficial effect of Mn and Si was limited. During 100h cycles at 700°C, aPOS also was observed to form on the alloys with Mn and Si except for the specimen with 16Cr

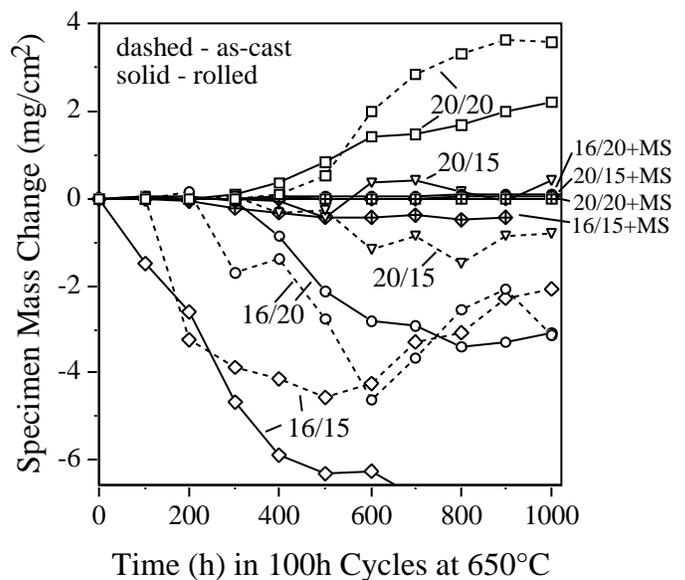


Figure 7. Specimen mass changes for as-cast (dashed lines) and rolled (solid lines) model Fe-Cr-Ni alloys (specified by their Cr/Ni contents) some of which contain Mn and Si (MS) during 100h cycles at 650°C in air plus 10%H₂O.

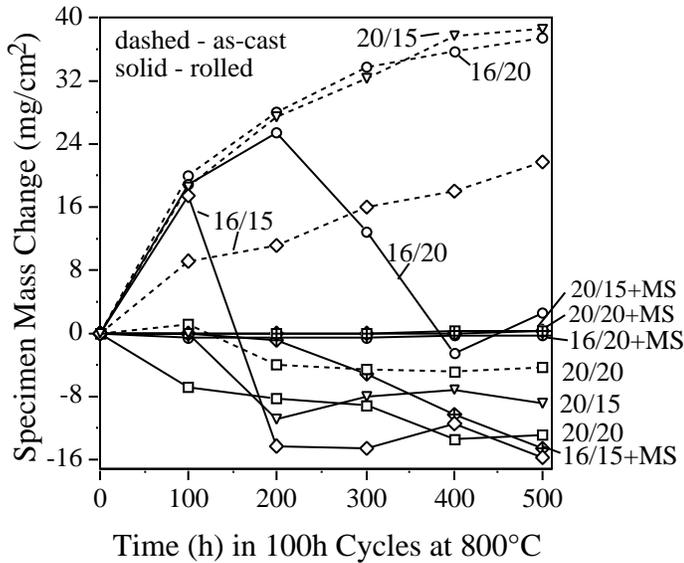


Figure 8. Specimen mass changes for as-cast (dashed lines) and rolled (solid lines) model Fe-Cr-Ni alloys (specified by their Cr/Ni contents) some of which contain Mn and Si (MS) during 100h cycles at 800°C in air plus 10% H₂O.

and 15Ni.

At 800°C, similar trends were observed, Figure 8. At this temperature, the mass gains and losses are higher for the alloys showing AA. All of the as-cast specimens showed large mass gains except for the Fe-20Cr-20Ni specimen. The rolled specimens without additions also showed high mass gains or losses. As at the lower temperatures, the addition of Mn and Si to each of the base compositions resulted in a POS, except for the 16Cr-15Ni specimen which showed a large mass loss after 5 100h cycles, Figure 8. At all temperatures, longer test times are needed to better define the benefits of these compositions.

An additional Fe-20Cr-20Ni alloy was made with only a Si addition in order to assess its effect without Mn. Figure 9 compares the mass changes of the various Fe-20Cr-20Ni alloys at 650°-800°C with an expanded y-axis compared to Figures 7 and 8. In general, the specimens with only Si resulted in POS formation at each temperature. However, compared to the specimens with both Mn and Si, the addition of Si resulted in slight mass losses. Based on visual observations, the small mass losses were attributed to scale evaporation and not to scale spallation. In 1h cycles at 700°C, Figure 6, the specimen with only Si began to show a mass gain that may indicate the onset of AA while the specimen with Mn and Si showed only a small mass change. This may indicate that the Mn addition did have a beneficial effect such as limiting scale evaporation as suggested previously.[25] Testing for longer times may further differentiate these effects.

The oxidation behavior of one additional Fe-20Cr-20Ni alloy also has been evaluated. This laboratory-made alloy has been modified with additional elements to increase its creep strength and improve its austenite stability. (The exact composition is the subject of an invention disclosure.[31]) This alloy (marked Etc. in Figure 9) also showed POS formation at each temperature. It showed slightly higher mass gains than the model alloy with just Mn and Si. However, this was not an indication of the onset of AA as its mass gain in dry air

(dashed line in Figure 9) was almost identical.

DISCUSSION

Based on this work, the most important factors for good corrosion resistance in humid air at 650°-800°C are (1) high Cr and Ni contents, (2) small alloy grain size, and (3) Mn and Si additions. Considering type 347 stainless steel, it already contains Mn and Si and, in foil form, is usually fabricated with a fine grain size. Therefore, its major problem is that it does not contain sufficient Cr and Ni to be corrosion resistant under these conditions. Therefore, type 347 stainless steel is inherently limited and if a recuperator is needed to operate in this temperature range, it appears necessary to develop a new alloy with higher Cr and Ni contents. There does not appear to be any possibility of improving the oxidation resistance of type 347 stainless steel without significantly changing its Cr and Ni contents.

More highly alloyed steels are available such as Nb-modified Fe-20Cr-25Ni alloys (i.e. NF709) and Fe-25Cr-35Ni (i.e. HR120). However, increasing the Cr and Ni contents increases the cost of the alloy thereby increasing the cost of the recuperator. These results suggest that sufficient corrosion resistance may be attainable with only a modest increase in the Cr and Ni contents resulting in a more affordable alloy. However, more extensive long-term laboratory work is needed to confirm these results, including testing of foils. Positive results would need to be followed by commercial foil fabrication and engine testing. It is difficult to truly assess the combined effects of high gas velocity and pressure, along with stress, on the alloy in laboratory testing.

One of the ultimate goals of this work is to obtain a better understanding of the role of water vapor in initiating accelerated attack. There is still disagreement in the oxidation literature about the mechanism by which water vapor affects the POS. These results about the importance of grain size and Si and Mn additions tend to support a mechanism where AA results from a combination of Cr depletion in the

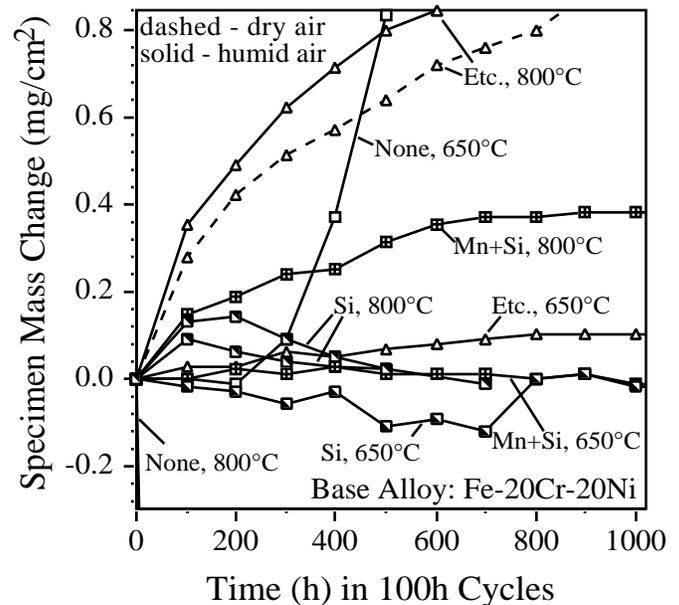


Figure 9. Specimen mass gains for austenitic model Fe-20Cr-20Ni alloys with various additions during 100h cycles in humid air at 650° and 800°C. The specimens marked Etc. has additional elements besides Mn and Si to improve mechanical properties.

metal, which drops the interfacial Cr content below the critical level needed to reform a POS, and evaporation of the thin chromia scale which is normally protective in dry air. With this mechanism, increasing the Cr content of the alloy would obviously help prevent the interfacial Cr content from dropping below the critical value. It is not as clear why increasing the Ni content also was observed to be beneficial. The Ni content may affect diffusion in the alloy but could also have other effects such as decreasing the solubility of oxygen in the metal.[21] The finer grain size increases the Cr diffusion rate in the alloy thus reducing the possibility of the interfacial Cr depletion. Minor additions of Mn have long been known to form an outer Mn,Cr oxide layer [32] which may reduce the rate of evaporation. Likewise, minor Si additions are well known to form silica inner layers, even with extremely low (0.05wt%) Si contents.[29] Formation of an inner silica layer may provide sufficient protection such that AA is avoided. A thin silica layer was observed to form on type 321 stainless steel oxidized in dry air at 700°C but not in humid air.[33] These minor alloy additions may also play other roles such as affecting diffusion rates in the alloy.

There is still debate regarding whether water vapor has an effect on the growth rate and/or oxidation mechanism of chromia scales. That has not been investigated in the current work but will be more fully examined on these model alloys. Previous work showed only minor effects of water vapor on the chromia scale microstructure of commercial alloys.[20]

SUMMARY

The oxidation behavior of type 347 stainless steel foils and various austenitic and ferritic model alloys was examined in dry and humid (10% water vapor) air at 650°-800°C in order to better understand performance in exhaust gas environments in recuperators. Alloys with coarse grain structures and low Cr and Ni contents are subject to accelerated attack in water vapor that is not observed in dry air. Oxidation resistance in these environments is improved by increasing the Cr and/or Ni contents of the alloy, decreasing the grain size and adding minor additions such as Mn and Si. Unfortunately, commercial type 347 stainless steel foil already has a fine grain size and additions of Mn and Si but still is subject to accelerated attack in humid air at 650°-800°C. Therefore, if recuperators are needed for operation at 650°C-700°C, an alloy with higher Cr and/or Ni contents will be needed. Initial testing on model alloys with 20Cr and 20Ni show good oxidation resistance in humid air at 650° and 700°C suggesting that an improved performance stainless steel may be developed with only a modest increase in cost compared to type 347 stainless steel foil. Further laboratory testing and eventual engine testing will be needed to confirm these results.

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