The role of active elements in Fe–Cr–Al alloys for heating element applications

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Abstract. The role of active elements (zirconium, cerium and yttrium) in grain growth and its subsequent effect on room-temperature tensile fracture behaviour of Fe–Cr–Al alloys has been investigated. The alloys containing active elements exhibited improved resistance to grain growth. Yttrium-containing alloy retained higher room-temperature tensile ductility even after heating at higher temperatures because of its superior resistance to grain coarsening. All the alloys, with and without active elements, exhibited a sudden drop in room-temperature tensile ductility, with concomitant change in fracture mode from dimple rupture to cleavage, in a narrow band of 39–44 μm grain size. The ductile–brittle transition temperatures (DBTT) of these alloys were found to be 150°C and 215°C for grain sizes 25 μm and 100 μm respectively. The accelerated life test showed that the alloys containing active elements exhibited longer lives.

Keywords. Ferritic stainless steel; heating element; active element; ductile brittle transition temperature; grain growth; fracture mode.

1. Introduction

Iron–chromium–aluminium alloys are essentially ferritic stainless steels and are extensively used for heating element and other applications where high-temperature oxidation resistance is the most important requirement. These alloys are available in the market with trade names Kanthal (Sweden), Gilphal (France), Fecralloy (UK). A variety of heating element materials are available; however, bulk of the requirement is met by Ni–Cr, Ni–Fe–Cr and Fe–Cr–Al alloys because of their easy processing coupled with good performance over a wide range of temperatures in varied environments. Of these, Ni–20Cr and Ni–Fe–Cr alloys are being produced at MIDHANI, Hyderabad. However, technology for indigenous production of Fe–Cr–Al alloys is not available and these are being imported. In view of this, a developmental programme was initiated at DMRL and the production technology has been established on a laboratory scale.

Fe–Cr–Al alloys have been in use for heating element applications for a fairly long time. However, there is little information published on the physical metallurgy of these alloys because of its technological significance (Keown 1987). These alloys have outstanding oxidation resistance due to the formation of a very compact, adherent and stable Al_2O_3 layer at the surface. The details concerning the oxidation behaviour of these alloys with and without addition of rare earth elements such as Ce, La, Y, Er, etc are well documented (Tien and Pettit 1972; Golightly *et al* 1976; Amano *et al* 1979, 1983; Ramanarayan *et al* 1984; Beranger *et al* 1989). This paper presents the effect of active elements in Fe–Cr–Al alloys on their grain growth and its subsequent effect on their tensile fracture behaviour (at room temperature) and impact properties. Also, the role of active elements in the performance of Fe–Cr–Al alloys during accelerated life test is presented.

2. Experimental

The chemical composition of three alloys melted at DMRL and designated K-1, K-7 and K-8 is given in table 1. Of these, alloys K-1 and K-8 were melted in an air induction furnace and alloy K-7 in a vacuum induction furnace and cast into ingot moulds. All the ingots were hot-transferred to a furnace and homogenized at 1100°C for 8 h. The ingots were subsequently processed according to the details given in the flow chart (figure 1). In order to evaluate the role of active elements on the grain growth behaviour, samples of all the three alloys with initial grain size of 15 μm were heated at 1050 and 1200°C for 100 and 25 h respectively. Test bars of alloys K-1, K-7 and K-8 were heated at 750, 850, 950, 1050 and 1150°C for 2 h to determine the effect of temperature on grain growth and its subsequent effect on their tensile fracture behaviour at room temperature. Heat-treated test bars were machined to the dimensions of 4 mm gauge diameter and 25 mm gauge length and tensile tests were carried out on an Instron machine. Impact properties of alloys K-7 and K-8 were determined as a function of temperature using 6-mm-thick substandard charpy specimens. Metallographic samples of heat-treated and tensile tested specimens were prepared. Grain size was measured by the linear intercept method. A scanning electron microscope with energy dispersive X-ray microanalysis facility was used to detect and identify the grain boundary particles and also to examine the microcracks in the tensile tested specimens.

2.1 Accelerated life test

Accelerated life test was carried out according to ASTM specification No. B78 (1981) to determine the resistance to oxidation of heating element materials under cyclic heating and cooling conditions. In this test, a 0.6 mm diameter wire of 250 mm length was bent to a U-shape and was intermittently heated by resistance heating (by passing current through the wire) between room temperature and 1200°C with a hold time of 2 min. Temperature of the wire was measured using an optical pyrometer once in 24 h and adjusted, if necessary. Time elapsed for first appearance of the hot spot was considered to be life of the wire.

3. Results and discussion

3.1 Grain growth behaviour

Figure 2 shows the microstructures of the three alloys K-1, K-7 and K-8 obtained after heating at 1050°C for 100 h. Grain size of the three alloys after heating at 1050

Table 1. Chemical composition of the three alloys (wt%).

Identification	Cr	Al	Co	Zr	Si	Mn	C	Ce	Y	Fe
K-1	21.8	5.1	0.98	—	0.4	0.3	0.06	—	—	Bal.
K-7	22.0	4.98	1.0	0.15	0.4	0.3	0.014	—	0.47	Bal.
K-8	21.6	5.0	1.0	0.15	0.5	0.3	0.08	0.03	—	Bal.

Figure 1. Process flow chart of iron–chromium–aluminium alloys.

and 1200°C for 100 and 25 h respectively is recorded in table 2. Considerable grain coarsening can be noted in the alloy which did not contain any active element (K-1). In contrast, grain coarsening was restricted in the alloy which contained about 0.5% yttrium (K-7). Figure 3a shows the presence of yttrium-rich particles both at the grain boundaries and within grains of alloy K-7 (figure 3b). A number of yttrium-rich phases have been reported in Fe–Cr–Al alloys containing yttrium (Keown 1987), but the major phases identified are YFe_9 and Y_2O_3 . Both the phases are extremely stable up to very high temperatures and they are considered to be responsible for the remarkable grain growth resistance of yttrium-containing Fe–Cr–Al alloys. Grain coarsening was also restricted to some extent in the alloy which contained Zr and Ce (K-8). Precipitates of chromium carbide were formed in both K-8 and K-1 (figure 4). In addition, there were a few alumina inclusions as melting of K-1 and K-8 was carried out in an air induction furnace. Although these carbide particles were present in both the alloys, grain coarsening was restricted only in alloy K-8. Considerable grain growth in alloy K-1 indicates that chromium-rich carbide was not effective in inhibiting grain coarsening, because of its instability at temperatures greater than 1000°C (Satyanarayana and Pandey 1993). It appears therefore that grain coarsening in alloy K-8 was restricted due to the presence of

Figure 2. Microstructures of the alloys K-1, K-7 and K-8 after heating at 1050°C for 100 h.

zirconium and/or cerium, in the form of some intermetallic compound. Figure 5 shows the variation of grain size in all the three alloys as a function of temperature. It can be seen that grain coarsening, above 1000°C, is very rapid for alloys K-1 and K-8 compared to alloy K-7. This clearly demonstrates that addition of yttrium to Fe–Cr–Al alloy is very effective in inhibiting grain growth up to very high temperatures.

3.2 Tensile fracture behaviour

Figure 6 shows the effect of exposure temperature on the room-temperature ductility of alloys K-1, K-7 and K-8. It can be seen that ductility is reduced quite steeply for

Table 2. Grain size of alloys K-1, K-7 and K-8 obtained after heating at 1050°C and 1200°C (initial grain size 15 μm).

Identification	Exposure temperature (°C)	Duration (h)	Grain size (μm)
K-1	1050	100	100
K-7	1050	100	30
K-8	1050	100	50
K-1	1200	25	370
K-7	1200	25	90
K-8	1200	25	140

Figure 3. a.

Figure 3. a. Scanning electron micrograph showing the presence of yttrium-rich particles, both at the grain boundaries and within the grains in alloy K-7 and b. EDAX spectrum showing presence of yttrium in the grain boundary particles.

Figure 4. a. Backscattered electron image showing the presence of chromium carbide and alumina particles both at grain boundary and within the grains; X-ray images of b. chromium, c. aluminium and d. carbon.

exposure temperature of 950°C and above for alloys K-1 and K-8, and for 1050°C and above for alloy K-7. Also, ductility values are quite high for alloy K-7 for all exposure temperatures above 900°C. This indicates that alloy K-7 has ability to retain a good amount of ductility after exposure to higher temperatures in contrast to alloys K-1 and K-8. This can be attributed to its better resistance to grain coarsening (figures 2 and 5). Figure 7 shows the effect of grain size on tensile ductility of alloys K-1, K-7 and K-8. A sudden drop in ductility, with concomitant change in the fracture mode from

Figure 5. Variation of grain size for alloys K-1, K-7 and K-8 as a function of temperature.

Figure 6. Effect of annealing temperature on the room-temperature tensile ductility of the alloys K-1, K-7 and K-8.

dimple rupture to cleavage, can be noted for all the three alloys in a narrow band of 39–44 μm grain size. This shows that there is a ductile-to-brittle transition at room temperature in these alloys at grain size close to 40 μm . Embrittlement of Fe–Cr–Al alloys following high-temperature exposure has also been reported (Dubey *et al* 1982). Fractographic studies revealed that the fracture surface of tested specimens of all the three alloys exhibited dimples when grain size was below about 40 μm (figure 8a) and cleavage when grain size was above about 40 μm (figure 8b). Figures 9a and 9b show nucleation of microvoids at the particle–matrix interface near the fracture end of specimens with fine grain size below 40 μm of alloys K-7 and K-8. Nucleation of microvoids occurred either by decohesion of smaller particles or cracking of coarser particles. Final fracture occurred by subsequent growth and coalescence of these microvoids resulting in dimple rupture. Figures 10a and 10b show cleavage microcracks in the tensile tested specimens of alloys K-7 and K-8 with coarse grain structure ($> 40 \mu\text{m}$). Microscopic examination of tensile tested specimens revealed that all the cleavage microcracks were not associated with particles, indicating that the presence of particles is not mandatory for nucleation of microcracks. It has been shown in some bcc metals (Zener 1948) that nucleation of cleavage cracks occurs due to stress concentration ahead of slip band blocked either by particles or by grain boundaries. The stress concentration at the tip of the slip band depends on slip distance which increases with grain size. This indicates that once grain size reaches a critical value, sufficient stress concentration is built up to cause nucleation of cleavage cracks. In the present alloys the critical grain size was found to be about 40 μm .

3.3 Impact properties

Figure 11 shows variation of impact energy of alloy K-8 as a function of temperature for 25 and 100 μm grain sizes. It can be seen that ductile–brittle transition temperature

Figure 7. Effect of grain size on the tensile ductility of the alloys K-1, K-7 and K-8.

Figure 8. Fractographs of Fe–Cr–Al alloys showing **a.** dimple rupture for specimens with grain sizes below about 40 μm and **b.** cleavage mode of fracture for specimens with grain sizes above about 40 μm .

(DBTT) is about 150°C for grain size of 25 μm and about 215°C for 100 μm . DBTT was also determined for alloy K-7 at a grain size of about 100 μm and was found to be about the same as that for alloy K-8, indicating that addition of yttrium and the vacuum melting route had no effect on DBTT of these alloys. The high DBTT of this family of alloys indicates that these alloys are very notch-sensitive at room temperature and therefore appropriate care needs to be taken while handling (shifting from

Figure 9. Scanning electron micrographs showing nucleation of microvoids at particle-matrix interface in tensile tested specimens of **a.** alloy K-7 and **b.** alloy K-8.

one place to other), conditioning (grinding and/or machining to remove surface defects) and room-temperature processing of large sections of these alloys.

3.4 Accelerated life test

The life test was carried out to evaluate the ability of the heating element materials to form and retain a protective oxide film under severe cyclic heating and cooling

Figure 10a.

Figure 10. Scanning electron micrographs showing nucleation of cleavage microcracks in tensile tested specimens of a. alloy K-7 and b. alloy K-8.

Figure 11. Variation of impact energy as a function of temperature for alloy K-8 for grain sizes a. 25 μm and b. 100 μm.

conditions. The life and performance of a heating element, therefore, depends on the nature of oxide film formed under these conditions. Table 3 shows the life of heating elements of the three alloys obtained from the accelerated life test. Alloy K-1 showed very poor life compared to alloys K-7 and K-8. Protective film formed in all three alloys was Al_2O_3 (figure 12). However, adherence of the oxide film formed on the heating element of alloy K-1 was very poor and it spalled off over almost the whole length of the wire after a few cycles. In case of alloys K-7 and K-8 the film was very compact and strongly adherent over the whole length of the heating element wire and appearance of bright spot occurred in localized

Table 3. Accelerated life test data.

Identification	Life (h)
K-1	50
K-7	300
K-8	220

Figure 12. a. Backscattered electron image showing aluminium oxide film formed at the surface of heating element during accelerated life test of Fe–Cr–Al alloys; X-ray images of b. aluminium, c. chromium and d. oxygen.

regions where oxide film spalled off after large number of cycles. It appears that better performance of alloys containing active elements is due to the improved adherence of the oxide film formed at the heating element surface. Active elements improve the adherence of Al_2O_3 film in Fe–Cr–Al alloys either by preventing

the formation of voids at oxide–alloy interface (vacancy–sink mechanism, Tien and Pettit 1972) or by changing growth mechanism and morphology of oxide scale (Golightly *et al* 1976) or by forming oxide pegs along the grain boundaries (Amano *et al* 1979).

4. Conclusions

- (i) Fe–Cr–Al alloys containing active elements exhibited better resistance to grain coarsening. The effect was more pronounced in the case of alloy containing yttrium.
- (ii) The alloy with yttrium was able to retain higher room-temperature tensile ductility even after heating at higher temperatures due to its better resistance to grain coarsening.
- (iii) A sudden drop in ductility, with concomitant change in fracture mode from dimple rupture to cleavage, was observed in all the three alloys in a narrow band of 39–44 μm grain size.
- (iv) The ductile–brittle transition temperatures of these alloys were found to be 150 and 215°C for 25 and 100 μm grain sizes respectively.
- (v) Alloys containing active elements exhibited longer lives in accelerated life test.

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