



High-resolution chemical analysis by STEM-EELS of nanosized oxide particles in a mechanically-alloyed FeCrAl intermetallic



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ABSTRACT

The chemical composition of nanosized oxides has been analysed in a mechanically-alloyed (MA) iron–chromium–aluminium intermetallic containing yttria additions using an aberration-corrected, high-resolution scanning transmission electron microscope (STEM). The oxide particles are seen to contain yttrium and oxygen only, but very little of the matrix metallic elements, while the matrix in the immediate vicinity shows a very low iron content. Possible reasons for the change of matrix composition outside the particle–matrix interface are discussed.

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1. Introduction

Recent studies [1–7] of powder-consolidated FeAl(Cr) intermetallics, dispersion strengthened by nanoscale oxide particles, have shown that very good strength can be achieved with good ductility at room temperature, while at high temperature both flow stress and creep stress remain good. The most recent studies [6,7] deduced that many of the nanosized disk-shaped oxide particles had a simple fcc crystal structure while others had a monoclinic yttrium–aluminium (YAM) structure. Chemical analysis carried out by transmission electron microscopy (TEM) using Energy Dispersive X-ray Spectroscopy (EDS) showed that these oxide particles contained significant amounts of Al in addition to the Y–O introduced by mechanical alloying to the iron aluminide matrix.

The mechanical alloying process (MA) has been developed as a way to introduce stable oxides for strengthening, especially at high temperatures, not only in iron aluminides, as mentioned above, but also in a range of Fe–Cr–base steels [8–11]. It is seen as a way to obtain suitable intermetallic matrices of various aluminides or silicides, for example [12–14], and as a better way to introduce fine and stable oxides than methods involving lower-energy mixing of powders or reactive mixing of elemental components [15]. While the MA process typically mixes yttria particles into the alloy or intermetallic matrix, subsequent analysis of the milled-consolidated product generally shows that the chemical composition and crystal structure of the oxide particles have changed. For iron aluminide matrices the oxides typically contain large amounts of Al [4,6,7,16,17] while for Fe–Cr steel matrices they

typically contain large amounts of Cr and other elements [8,9,11, 18–21], and crystal structures change from that of the initial yttria to more complex structures. Indeed it has been argued that the yttria dissolves in the matrix during milling and subsequently precipitates out as the complex oxide containing many elemental components [22]. These analyses have generally been carried out by TEM with EDS, and sometimes by atom probe tomography techniques, with different degrees of spatial and chemical resolution, depending on the instrumentation used. The present study repeats the analysis of the oxide particles found in the FeAl(Cr) intermetallics which have been previously reported [6,7] using an aberration-corrected (scanning) transmission electron microscope and Electron Energy-Loss Spectroscopy (EELS) in order to achieve better spatial chemical analysis.

2. Experimental

The iron aluminides examined here were the same as reported earlier [6,7] and were prepared by industrial pilot-scale milling and consolidation by Plansee AG from pre-alloyed metallic powders and yttria additions. The chemical composition of the material specifically shown below is that with 39% Al and 10% Cr (atomic percent) containing a nominal 1% by volume of yttria addition. Some of the other iron aluminides reported earlier [6,7] were also examined in the present study and similar results were found.

Thin foil samples were prepared from the bulk materials after machining bars with a diameter of 3 mm and cutting thin disks using a diamond saw. Thin regions suitable for examination by TEM were prepared in two ways – by electropolishing and by ion milling. Electropolishing was carried out in a twin-jet polisher using a 20% nitric acid in methanol mixture at 30 °C and about 16 V. Other samples were

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prepared by first electropolishing for about half the time for expected penetration and were then milled in a dual-ion system at liquid nitrogen temperature with a 70° take-off angle. Some of the thin foils were given a mild ion beam clean immediately prior to microscope examination. Particles inside thin foil samples were examined using a JEOL 3000F for high resolution imaging and a JEOL ARM 200cF, with aberration correction of the condenser lens, for chemical analysis and elemental mapping. This microscope has a cold field emission electron source, operates at 200 kV, and has quoted spatial resolutions of 0.2 nm in TEM mode and 0.08 nm in STEM (scanning transmission electron microscopy) mode. Chemical analysis was carried out in the STEM mode using an EELS system, with energy resolution quoted as 0.3 eV, obtaining elemental maps of complete particles with their surrounding matrix. The relevant energy-loss lines are listed in Table 1, together with the energy windows used for mapping. Background radiation was removed from the relevant elemental line spectra using standard correction software.

3. Results and discussion

3.1. Oxide particle morphology and structure

The oxide particles found in all the Fe–Al–Cr intermetallics examined were disk particles of typical thickness of about 5 nm and diameter of 15–25 nm, lying on the cube planes of the B2 matrix [6,7]. Figs. 1 and 2 show the general shape and distribution of these particles, confirming the generally homogeneous distribution of particles throughout all the grains. In addition a small number of larger particles were found, often sitting on the grain boundaries. The observation of such uniform distribution and occasional larger particles is typical of Oxide Dispersion Strengthened (ODS) materials, as reported many times before [2–11, 16–22]. One difference noted here is that the present particles are disk-like with two major planar interfaces, while the oxide particles are generally found to be near-spherical. Oxide particles are often found to be coherent with the matrix, especially when present as very fine, nanosized particles [10,11,18–22], and on some occasions they have reported to be faceted [23,24]. The particles here were identified as generally having the fcc structure with lattice parameter near 0.525 nm, while some had the YAM structure [6,7]. Fig. 3 shows part of one such particle, with the [110] fcc orientation, in a [001] oriented matrix, and the partial coherence of {110} planes in the matrix and {111} planes in the oxide is clear. Such partial coherence is presumed to lead to a relatively low oxide–matrix interface energy which can make particle nucleation easier from a supersaturated matrix during the high temperature powder-consolidation stage.

3.2. Chemical composition of oxide particles

Particles such as those shown in Fig. 3 were imaged by high resolution STEM with parallel EELS elemental mapping, as illustrated in Fig. 4. Very similar images were obtained for all the Fe–Al–Cr intermetallics examined and both for those particles showing the fcc structure and those with the YAM structure. In all cases, particles were examined end-on in foil regions of thickness of approximately 15–20 nm such that the particle disks are expected to traverse the entire foil thickness, allowing determination of the true particle composition. Fig. 4a shows a HAADF (high-angle, annular dark field) image of a disk particle, seen

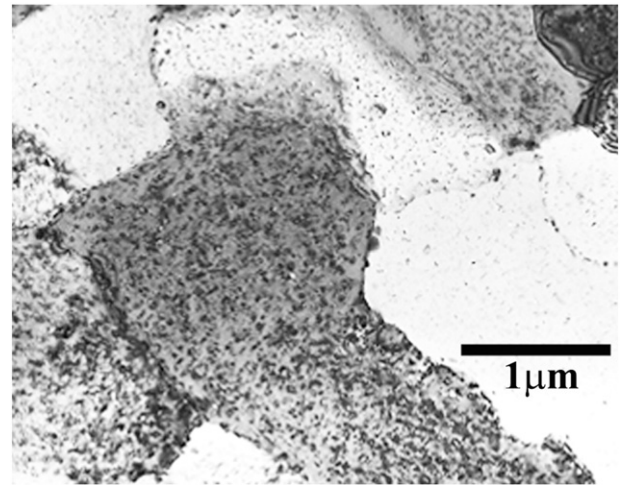


Fig. 1. Low magnification TEM image showing the general morphology and distribution of oxide particles in several grains of the Fe-39% Al-10% Cr-1% yttria material.

end-on, where some internal crystal structures can be distinguished within the particle, and a dark band surrounding the particle may be seen. This band was visible in light contrast when seen in the bright field image. Elemental maps indicate the changes of chemical composition occurring from the matrix, across the interface, and into the oxide particle, with line-scan plots deduced from the elemental maps confirming the mapping analyses. The maps (Fig. 4) confirm that the oxide particles were very rich in Y and O, with essentially no Al, and a low Fe–Cr content. While it is not possible to deduce accurate content levels for these different elements, it is clear that the particles were essentially a yttrium oxide, with both the Al and the Cr contents below about 2%, and the Fe content below 5%. Instead of being the Y–Al–O composition previously indicated [4,6,7,16,17] the present higher-resolution chemical analysis has confirmed that the oxides are essentially binary Y–O composition, with only a small Fe(Cr) content. Under these conditions, the oxide present is deduced to be the fcc Y_2O_3 phase (X-ray diffraction pattern 00-043-0661 of the ICDD (International Centre for Diffraction Data) files).

A second feature visible in Fig. 4 is the wider region of Fe depletion than the oxide thickness as indicated by the Y-rich and O-rich regions. The Cr-depleted region appears to have the same width as the Fe-



Fig. 2. Bright field TEM image showing the disk-like morphology of the oxide particles, arranged on the cube planes of the matrix of the Fe-39% Al-10% Cr-1% yttria material. Some larger particles are seen near the grain boundary, near bottom. The matrix is oriented at the [001] zone axis, and most disk particles are seen end-on aligned in the [100] and [010] directions.

Table 1

Energy loss parameters used for mapping. Width of precipitate or hole shown in Fig. 4.

Element	Line	Line edge energy (eV)	Window used for mapping (eV)	Hole or precipitate width (nm)
Al	L _{2,3}	73	75–130	4.7
Y	M _{4,5}	157	160–230	4.7
O	K	532	515–560	4.8
Cr	L _{2,3}	575	560–610	6.0
Fe	L _{2,3}	703	700–755	6.1

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