Microstructure and mechanical properties of Fe₃Al alloys with chromium

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Alloys based on Fe₃Al have an equilibrium DO₃ structure at low temperatures and transform to a B2 structure above about 550 °C. The influence of different rates of quenching from the B2 region to room temperature and of subsequent heat treatments on the microstructure and mechanical properties of powder metallurgy (P/M) alloys with two different chromium contents have been examined. Optimizing the processing to maximize the amount of B2 order, without eliminating dislocations that enhance both strength and ductility, yields room-temperature ductility approaching 20%, although the fracture mode is primarily brittle cleavage. The B2 structure generally has lower flow stress than the DO₃ structure because of its lower strain-hardening rate, although B2 order actually has higher yield strength when the structure is free of dislocations. Increasing the chromium content from 2% to 5% has little effect on ductility, although the 2% Cr alloys generally have higher yield strengths and larger order parameters.

1. Introduction
Fe₃Al-based alloys exhibit good oxidation and sulphidation resistance as well as excellent resistance to abrasive wear and erosion [1], but until recently interest in these alloys has been limited because of their low room-temperature ductility. Liu et al. [2-4] and others [5, 6] have recently discovered that the limited ductility of iron aluminides is largely the result of environmental embrittlement. Tensile elongations of specimens tested in air or H₂O + argon are significantly less than those of specimens tested in dry oxygen [4], vacuum [6] or even hydrogen + argon [2, 3]. This is attributed to embrittlement by atomic hydrogen generated by a reaction with water vapour, similar to that found in many aluminium alloys. Tensile testing under various conditions of controlled pH and electrochemical potential indicate that atomic hydrogen is particularly detrimental to ductility, but that non-dissociated water molecules also cause embrittlement [3].

Fe₃Al alloys with higher strength and improved ductility have resulted from increased understanding of alloying and thermomechanical processing effects [7, 8]. Chromium is added principally to improve the room-temperature ductility [7, 8], while other alloying elements such as niobium, molybdenum and silicon appear to improve the elevated temperature strength but are deleterious to room-temperature ductility [9-11]. Warm working the material to create a high defect density at room temperature and appropriate heat treatments are thought to markedly increase the room-temperature ductility [12]. Powder metallurgy (P/M) methods offer the potential of a refined grain size in the final product, thus increasing the strength without significantly reducing the ductility.

A portion of the Fe-Al phase diagram is shown in Fig. 1. Compositions with stoichiometry of approximately 3 iron to 1 aluminium have three possible crystal structures. Above about 900 °C, a solid solution on a bcc lattice is the equilibrium structure. Two ordered phases exist at lower temperatures. The FeAl phase field shown in Fig. 1 is present above a critical temperature of about 550 °C and has the B2 structure. For compositions with an equiatomic stoichiometry the B2 structure has one type of atom at the corner sites of a cube and the other type at the centre; however, for Fe₃Al stoichiometry, iron atoms occupy all of one type of site, and the other sites are occupied by iron atoms and aluminium atoms with equal probability. Below the critical temperature, DO₃ is the equilibrium structure (labelled Fe₃Al in Fig. 1). Fig. 2 illustrates the DO₃ structure. For both B2 and DO₃ order it is assumed that chromium, the alloying element used in this study, occupies iron and aluminium sites with equal probability. Below the critical temperature, the B2 structure generally has lower flow stress than the DO₃ structure because of its lower strain-hardening rate, although B2 order actually has higher yield strength when the structure is free of dislocations. Increasing the chromium content from 2% to 5% has little effect on ductility, although the 2% Cr alloys generally have higher yield strengths and larger order parameters.
transformation is sluggish [13, 14]. By quenching from different temperatures and with different media, the relative volume fractions of the two ordered phases can be controlled. This is not the case for binary iron aluminides that have a rapid B$_2$--$\rightarrow$DO$_3$ transformation [15, 16]. The amount of each type of order can be described by the Bragg–Williams long-range order parameter, $S$, defined as

$$S = \frac{r_A - f_A}{(1 - f_A)} \quad (1)$$

where $r_A$ is the fraction of A sites occupied by the “right” atoms and $f_A$ is the fraction of A atoms in the alloy. An $S$ of 1.0 indicates perfect order of a certain type, and values less than 1.0 indicate a departure of the bulk sample from this perfect order. This parameter is determined by measuring the integrated intensity of the X-ray diffraction (XRD) peaks and comparing the measured values with calculated values for perfect order of the different phases.

In the present work, the effect of different heat treatments on the microstructure of two chromium-containing Fe$_3$Al alloys was examined by the use of XRD, transmission electron microscopy and optical metallography. The mechanical properties resulting from the various structures have been determined by room-temperature tensile testing of extruded and hot-rolled materials produced by P/M processing.

2. Experimental procedure

Two batches of powder with stoichiometries designed to yield Fe$_3$Al containing nominally 2% or 5% Cr by weight were produced by inert gas atomization with argon. The alloys were melted in zirconia crucibles prior to atomization; there was no indication of gross chemical interaction between the melt and the ceramic material.

The powders were canned in mild steel, evacuated and hot extruded at 1000 °C to an area reduction ratio of 9:1. The as-extruded structure was examined using standard optical metallography techniques. Round tensile bars were machined by centreless grinding from a portion of the extrusions. Tensile bars were tested in both the as-extruded and heat-treated conditions as described in Table I. A portion of the 5% Cr alloy extrusion was squared, annealed for 1 h at 1100 °C, clad in mild steel, and rolled to 10% and 30% reductions in thickness at 650 °C to introduce two different densities of defects. Round tensile specimens machined from these materials were tested in the as-rolled condition and after heat treatment as outlined in Table I.

After removing the steel can, another portion of each alloy extrusion was forged at 1000 °C to 8.6 mm thick and then rolled at 800 °C to sheet approximately 2.54 mm thick. The sheet was finish rolled at 650 °C to 0.76 mm. Tensile specimens were punched from the sheet and heat treated at temperatures from 700–850 °C before quenching in oil or air. All tensile testing of these punched sheet specimens was performed at a strain rate of $3.3 \times 10^{-3}$ s$^{-1}$. Three additional rolled specimens were cut from the 2% Cr sheet before heat treating, carefully polished, and tensile tested at room temperature at a strain rate of $6.6 \times 10^{-4}$ s$^{-1}$. The heat treatments for these samples were designed to result in varying amounts of the DO$_3$ ordered phase. The heat treatments for all sheet specimens described above are summarized in Table I.

After testing, the fracture surfaces of some of the samples were examined using SEM. The grip sections of the tensile bars were examined by standard optical metallography techniques to study the as-rolled structure and measure the grain size. The grain size was determined by the line intercept method. For a given specimen, four areas were photographed and three grain size measurements made on each area. Thin foils for TEM were prepared by electropolishing extruded transverse sections and by core drilling polished material from sheet short-transverse sections.

XRD was used to determine phases present and measure the order parameters in as-atomized powders, as-extruded material and heat-treated rolled specimens. Order parameters of the powder samples were determined by measuring the DO$_3$, B$_2$ and fundamental peaks that occur at the lowest diffraction angle. Samples from the grips of tensile specimens were repeatedly etched and lightly repolished to remove any flowed metal on the surface that might affect the