

MICROSTRUCTURE OF HEAT TREATED NdFeB-BASED ALLOYS

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ABSTRACT

Alloys of composition closed to the stoichiometric require a lengthy heat treatment to dissolve α -Fe within the microstructure. Microstructural comparisons of alloys before and after heat treatment revealed increasing amount of the Nd₂Fe₁₄B phase but proportionally decreasing the secondary phase (Nd-rich and Nd_{1.1}Fe₄B₄). Grain size of hard magnetic phase increased substantially with time and temperature of treatment. Such microstructural conditions are generally attributed to the lower rate of hydrogen reaction.

INTRODUCTION

The Nd-Fe-B alloys are normally prepared by melting and casting in a high vacuum melting furnace. Three different intermetallic phases coexist in the alloy of 'Neomax' (Nd₁₅Fe₇₇B₈) composition: Nd₂Fe₁₄B (a ferromagnetic), Nd_{1.1}Fe₄B₄ and Nd-rich (non-ferromagnetic) phase. For compositions that are closed to stoichiometric (Nd_{11.8}Fe_{82.3}B_{5.9}), a soft ferromagnetic α -Fe phase usually precipitated within the microstructure as a result of incomplete peritectic reaction [1]. A proportion of these phases depend on the cooling rate of casting [2]. A significant amount of α -Fe phase in the Nd_{11.8}Fe_{82.3}B_{5.9} ingot was disappeared when cast by strip casting [3]. The Fe-dendrites can be avoided also by shifting the alloy away from the stoichiometric composition and/or to modify the composition as suggested by Sagawa et al (1984). It has been reported that it is possible to reduce the amount of dendrites with the addition of zirconium [4]. Thus the present work was undertaken to investigate effects of heat treatment in dissolving the α -Fe from the Zr and non-Zr containing ingots.

EXPERIMENTAL

The near stoichiometric Nd-Fe-B alloys used in the present work had a nominal composition of Nd_{12.74}Fe_{81.25}B_{6.01}, (named C-1) and Nd_{12.77}Fe_{81.06}B_{6.08}Zr_{0.09}, (named C-2). The ingots were cut parallel to the cooling direction. Isothermal heat treatment or homogenization was done using a high vacuum tube furnace. The samples, covered by Fe foil, were heated under vacuum up to 600°C followed by heated under argon partial pressure to 1090°C. The treatment was between 5 to 36 hrs. The microstructure and phase composition were characterized by the Scanning Electron Microscopy (SEM) combined with an energy dispersive X-ray (EDX).

RESULTS AND DISCUSSION

Figure 1 and 2 shows the microstructure at central part of sample C-1 and C-2 respectively. As seen, both alloys consist of four main phases: $\text{Nd}_2\text{Fe}_{14}\text{B}$, $\alpha\text{-Fe}$, Nd-rich and $\text{Nd}_{1.1}\text{Fe}_4\text{B}_4$ phase, which are labeled as A, B, C and D respectively. The composition of these phases was determined by EDX analysis. A feather-like structure (labeled E) was seen embedded in the Nd-rich phase at the grain boundaries of C-2. The EDX analysis revealed that these are a Zr-containing phase.

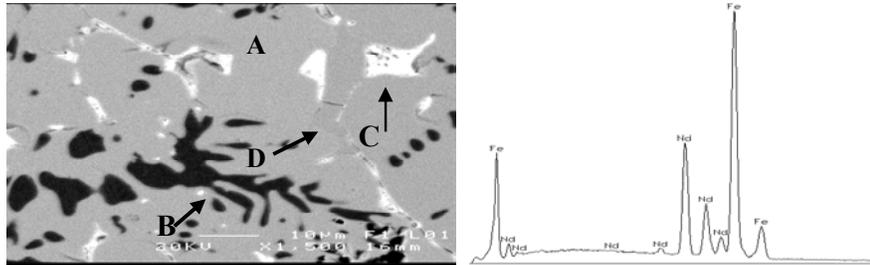


Figure 1: BSE-SEM micrograph of C-1 sample with a typical EDX spectrum pointed on the $\text{Nd}_{1.1}\text{Fe}_4\text{B}_4$ phase.

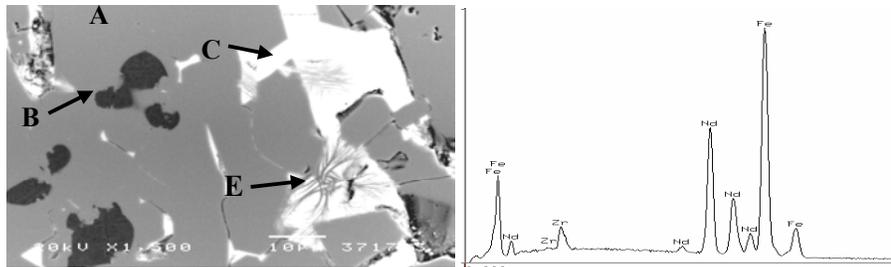


Figure 2: BSE-SEM micrograph of C-2 alloy, showing the appearance of feather-like structure (E), with a typical EDX spectrum pointed on the structure.

The proportion of $\alpha\text{-Fe}$, Nd-rich and $\text{Nd}_2\text{Fe}_{14}\text{B}$ with treatment time are shown in Figure 3. In as-cast state, the C-1 alloy contains higher content of $\alpha\text{-Fe}$ compared to the C-2 alloy. At the early stage of homogenization a rapid initial drop in the volume of $\alpha\text{-Fe}$ was observed in both alloys. After 12hrs of the treatment the dendrite structure is quite stable with a slight decrease in its content. The amount of the Nd-rich phase also decreases gradually. The atomic diffusion process slows down after 12hrs probably due to the less content of the secondary phase, and the structure and location of $\alpha\text{-Fe}$. As a result of homogenization at 36hrs, the amount of $\text{Nd}_2\text{Fe}_{14}\text{B}$ was increased to 98 % of the volume fraction of the alloy.

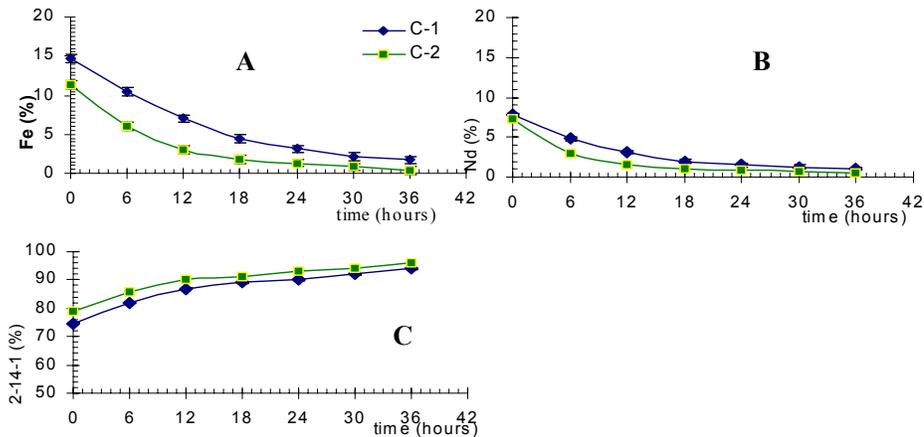


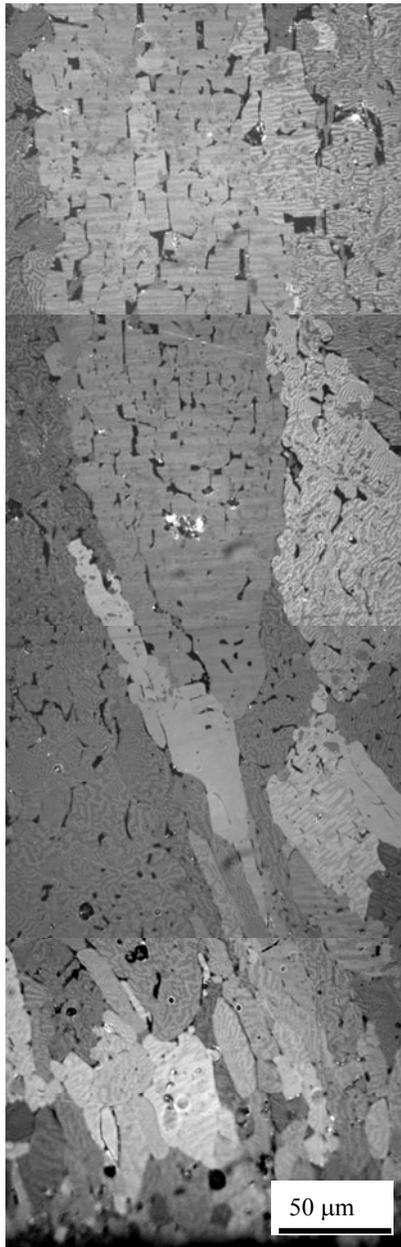
Figure 3: The proportion of Fe (A), Nd-rich (B) and Nd₂Fe₁₄B (C) with homogenization time

Grains of various size and shape were observed from a series of optical micrographs taken on each ingot, from the edge towards the central area as shown in Figure 4(A) and (B) for C-1 and C-2 ingot respectively. Smaller grains (10 -30 μ m) within a very small zone near the edge, which was in contact with the mould were observed. The sizes are gradually increased (up to 200 μ m) with elongated shapes when located further away from the edge. When comparing these as-cast alloys, the C-2 appears to have a smaller grain structure than the C-1.

Grain growth usually occurred during heat treatment but may control by additives like Nb [5]. Homogenization at 36 hours (Figure 5) shows much larger grains than those in the as-cast state (Figure 4). In comparing the grain growth of two alloys the C-1 (non-Zr containing) alloy appeared to be much more significant than that in the C-2 (zirconium containing) alloy. This observation suggested that Zr appears to be effective in inhibiting grain growth during homogenization. This effect of Zr is similar to that observed on the Hydrogenation Disproportionation Recombination Desorption (HDDR) processed NdFeBZr alloy [6].

Microstructure changes due to the atomic diffusion during homogenization are displayed in Figure 6 and 7. The Nd-rich phase was redistributed along the grain boundaries and formed big pockets at the grain junctions. It is noted that the shape of the dendrites was changed from an elongated shape to a more spherical shape, mainly allocated near the grain boundaries. For the C-1 (non-Zr containing) alloy a small proportion of Fe still existed after 36 hours heat treatment, meaning that a longer homogenization time is required in order to dissolve the Fe completely. Unlike the C-2 (Zr-containing) alloy, the heat treatment for 36 hours results in a fully dissolved α -Fe phase, as examined by the SEM.

(a) Grains structure on C-1 alloy



(b) Grains structure on C-2 alloy

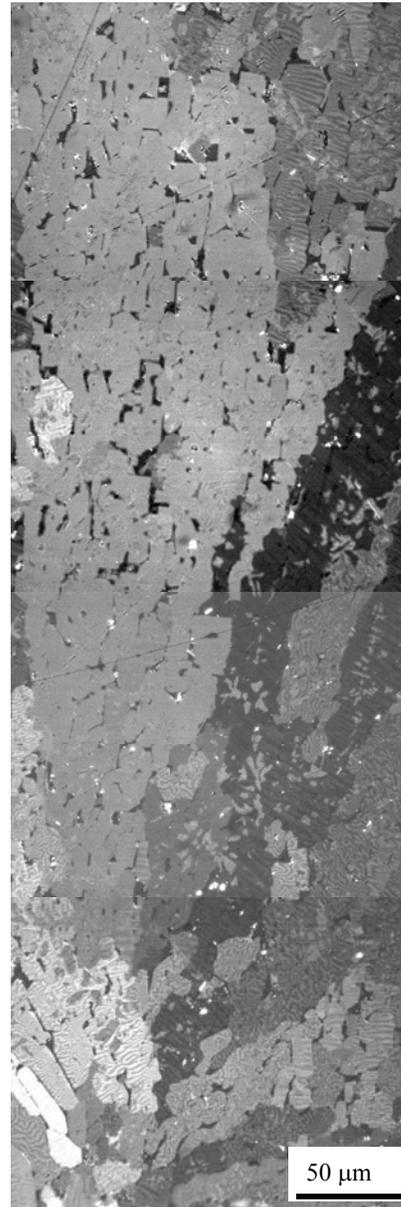
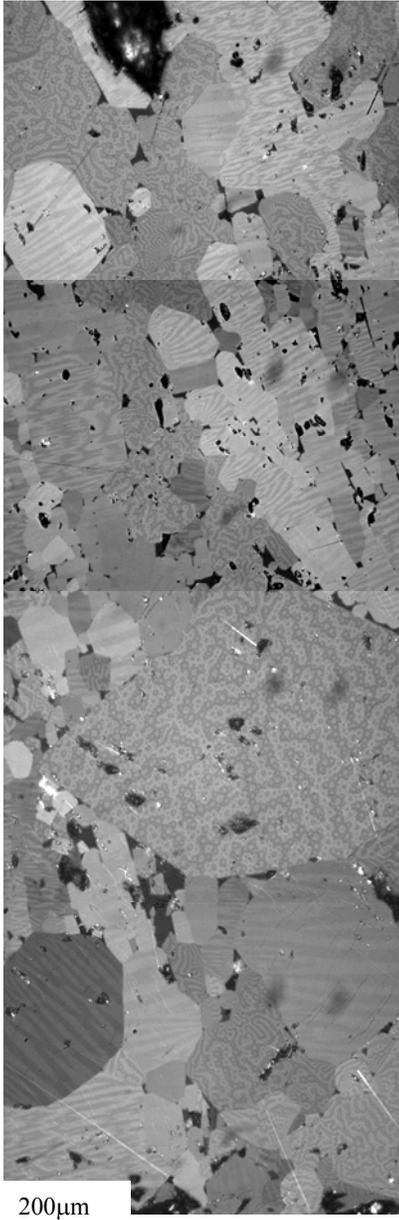


Figure 4: Optical micrographs of the cross-section on alloy of (a) C-1, (b) C-2 viewed under polarized light.

(A) Homogenised C-1 sample



(B) Homogenised C-2 sample

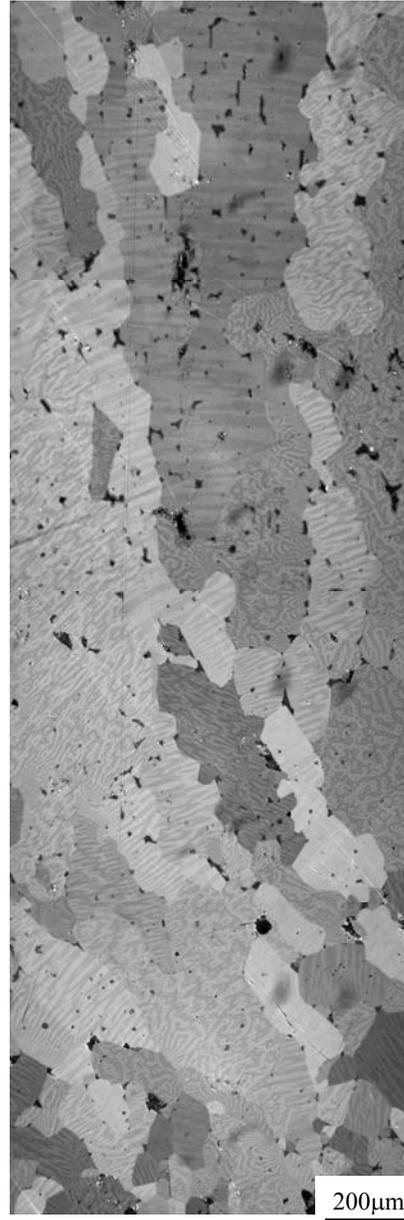


Figure 5: Optical micrographs of heat treated alloys (A) C-1, (B) C-2 at 1090°C for 36 hours

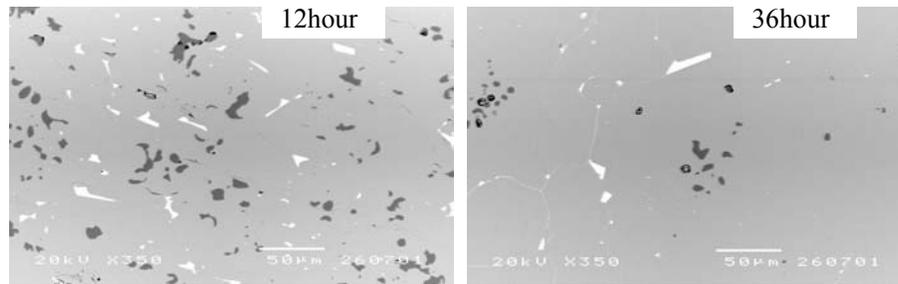


Figure 6: BSE-SEM image on heat-treated C-1 sample after 12 and 36hours

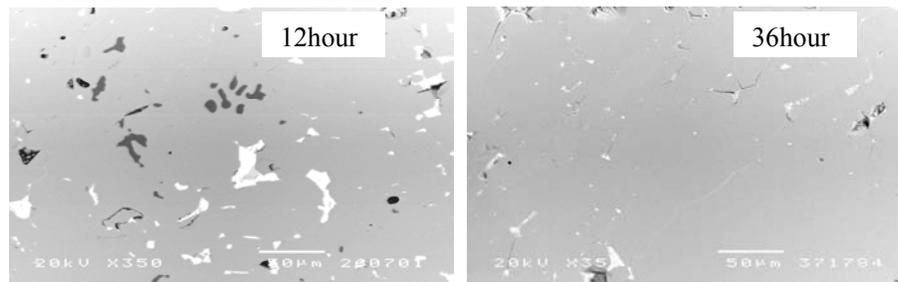


Figure 7: The BSE-SEM image on C-2 sample after 12 and 36 hours at 1090°C

CONCLUSIONS

Distribution of the α -Fe, Nd-rich and boride phases before homogenization determines the required time for a complete removal of α -Fe. Samples homogenized for more than 36 hours were believed to still contain a small amount of α -Fe in some regions that had less Nd-rich at the earlier stage.

Due to the Nd losses at high temperature, a complete removal of α -Fe from the stoichiometric NdFeB that had been cast conventionally is extremely difficult.

A reduction of α -Fe and Nd-rich content after the heat treatment gave rise to an increase in the proportion of Nd₂Fe₁₄B phase in all NdFeB-based alloys

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