Thesis Abstract: Microstructural Characterization of Nanocrystalline Fe-Zr-B(-Cu and/or Ge) Soft Magnetic Alloys

DEMING ZHU


In the research presented in this thesis, the origins of the microstructure and solute partitioning behaviour are characterized in nanocrystalline Fe-Zr-B(-Cu and/or Ge) alloys in order to design new experimental soft magnetic alloys. The structural and microstructural evolution of nanocrystalline Fe\(_{93-x-y}\)Zr\(_y\)B\(_x\)Cu\(_y\) alloys (\(x = 3\) and \(9\) at.\%\(; y = 0\) and \(1\) at.\%) was examined, with the aim of investigating the effect of B and Cu additions. The effect of Ge on the microstructural and magnetic properties of Fe\(_{89-x}\)Zr\(_3\)Cu\(_1\)Ge\(_x\) (\(x = 0\) to \(5\) at.\%) alloys was also examined. The experimental techniques used in this research were differential scanning calorimetry (DSC), thermo-gravimetric analysis (TGA), X-ray diffraction (XRD), transmission electron microscopy (TEM), field emission scanning transmission electron microscopy (FES-TEM) with nanoprobe energy-dispersive X-ray spectroscopy (NanoEDXS), and dc B-H loop tracer analysis.

In the investigation of the structural and microstructural evolution of nanocrystalline Fe\(_{93-x-y}\)Zr\(_7\)B\(_x\)Cu\(_y\) alloys, the following findings were made. B and Cu play opposite roles in stabilising the amorphous phase. While the addition of B stabilises the amorphous phase, the addition of Cu destabilises it. Cu atoms act as heterogeneous nucleation sites for the primary crystallization and the enhanced nucleation rate leads to nanocrystallites of bcc Fe in both low and high B content alloys. In high B content alloys, the crystallization of the amorphous phase occurs through two distinct reaction stages of amorphous \(\rightarrow\) bcc Fe + residual amorphous \(\rightarrow\) \(\alpha\)-Fe + Fe\(_2\)Zr + Fe\(_3\)Zr. The ease with which the residual amorphous phase transforms into Fe-Zr based compounds is dependent upon Cu content. In Cu-free high B alloy, both the primary crystallization and secondary crystallization have similar kinetics, and the primary crystallization is immediately followed by the secondary crystallization. However, in the presence of Cu, the enhanced nucleation of bcc Fe alters the composition of the residual amorphous phase and thus delays the onset of secondary crystallization. In Cu-free high B alloy, the grain growth of Fe crystallites was observed. This is attributed to the formation of Fe-Zr based compounds, which consumes Zr atoms. In Cu-containing high B alloy, due to the absence of such a reaction, Zr atoms are effective in pinning the bcc Fe grain boundaries giving rise to the nanocrystalline microstructure. NanoEDXS studies show the enrichment of Zr at bcc Fe grain boundaries confirming the pro-
posed grain refinement process.

In the investigation of the microstructural and magnetic properties of Fe$_{89-x}$Zr$_7$B$_3$Cu$_1$Ge$_x$ alloys, the following findings were made. The onset temperatures of primary crystallization for amorphous Fe$_{89-x}$Zr$_7$B$_3$Cu$_1$Ge$_x$ alloys are independent of the Ge content, but the Curie temperatures of these alloys are close to room temperature and increase with increasing Ge content. The crystallization takes place through two stages of amorphous $\rightarrow$ bcc Fe + residual amorphous $\rightarrow$ $\alpha$-Fe + Fe$_3$Zr regardless of Ge content. When the alloys are annealed for 3.6ks at temperatures between 773 and 923 K, the microstructure consists mainly of bcc Fe crystallites with an average grain size of 11 to 13 nm embedded in the amorphous matrix. Ge is enriched in the residual amorphous matrix together with Zr. This increases the Curie temperature and the exchange stiffness in the intergranular region, thereby improving the soft magnetic properties. The coercivity of Fe$_{89-x}$Zr$_7$B$_3$Cu$_1$Ge$_x$ alloys annealed for 3.6ks at 773 and 823 K exhibits a tendency to decrease with increasing Ge content, but this effect is not evident at 873 K and 923 K due to the relatively small volume of the residual amorphous phase. The coercivity of Fe$_{89-x}$Zr$_7$B$_3$Cu$_1$Ge$_x$ alloys annealed for 24h at 923 K exhibits a tendency to increase rapidly, due to the formation of the more strongly anisotropic Fe$_3$Zr compound and the enhancement of the grain size (55–60 nm).

Deming Zhu
School of Physics and Materials Engineering, Monash University
Victoria 3800
Australia
email: deming.zhu@spme.monash.edu.au

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