Studies of crystalline phases in amorphous magnetic alloys – 28-01-856

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Introduction

The purpose of this work is to exploit the kinetic diffraction technique to investigate phase formation and transformation in $Co_xFe_{(80-x)}B_{20}$ alloys.

Alloys based on $Fe_{80}B_{20}$ have been shown to exhibit interesting magnetic properties, including variations in their magnetic phases with applied field and temperature [1]. These materials have recently been used as the components in magnetic tunnel junctions (MTJ's). These consist of ferromagnetic/insulating oxide/ferromagnetic trilayers – materials for which room temperature the tunnelling magneto resistance ratio (TMR) can be as high as 200%. This ratio is greatest with crystalline components, but fabrication in a crystalline state requires an expensive single crystal substrate. Alternatively the correct crystalline orientations can be established by sputtering an amorphous ferromagnetic layer onto a crystalline insulating oxide layer, and then annealing the entire trilayer at a given temperature. Previous XRD and TEM studies indicate that the benefit of the CoFeB as the ferromagnetic layer lies in its ability to induce crystallisation and lattice matching with a MgO barrier layer [2]. However to date there appear to have been no reports of the annealing profiles of the amorphous alloys.

It is the intention of this work to investigate using kinetic synchrotron diffraction the routes of recrystallisation in $Co_xFe_{(80-x)}B_{20}$, x = 40,60 alloys, and ultimately through the use of complimentary in-house techniques determine how variation in alloy composition and annealing times has an effect on the magnetic and crystallographic properties.

Previous work

Previously on XMaS (28-02-813) we performed linear heating (500-800K at a rate of 2K min^{-1}) experiments on $Co_xFe_{(80-x)}B_{20}$, x = 40,60, collecting diffraction spectra every 2K.
Analysis of these results illustrate that in changing the chemical composition of the alloy the phase formation and transformation in the alloy varies markedly with only a single CoFe solid solution phase common to both alloys.

This work

To complement the previous experiment we used XMaS to determine the kinetics of the CoFe solid solution phase, by undertaking a series of isothermal anneals on each of the alloys.

Melt spun ribbons of $Co_xFe_{(80-x)}B_{20}$ were mounted in the heating stage of a Displex temperature controller and were rapidly heated to an annealing temperature and were then held isothermally until the phase was deemed to be completely transformed. Diffraction spectra were collected every 30 seconds on the MAR CCD detector . Using the software ESA Project [3] the images were integrated over the entire pixel range yielding intensity versus 2θ as a function of time.

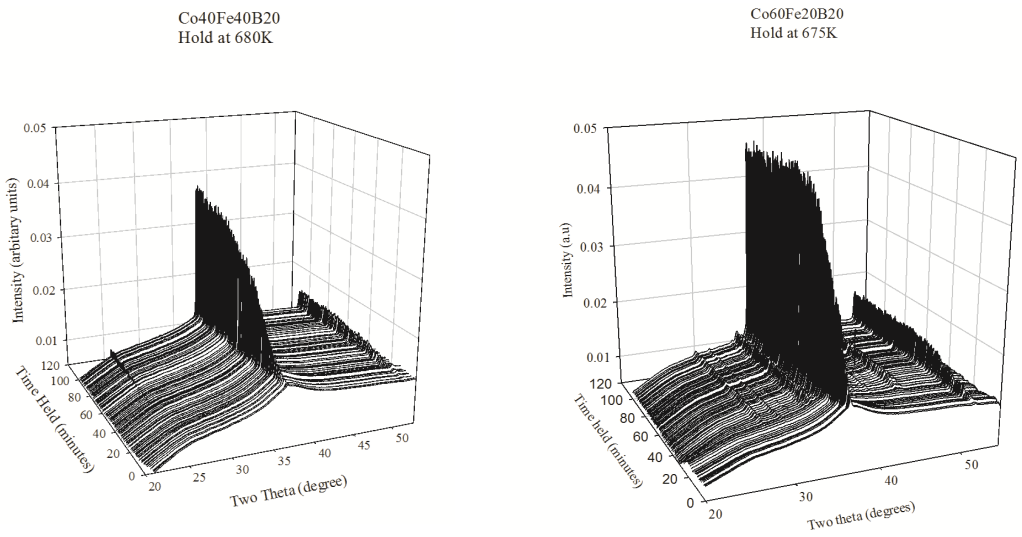


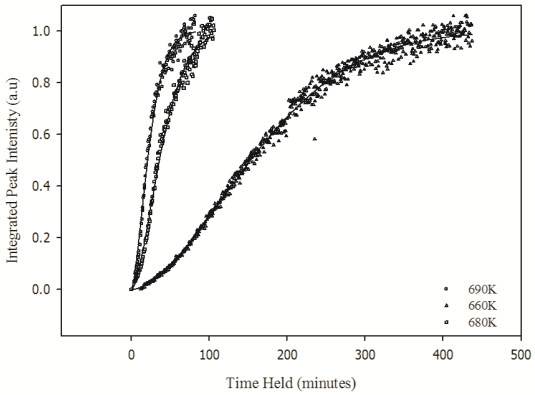
Figure 1: *Typical X-Ray thermograms of $Co_{40}Fe_{40}B_{20}$ and $Co_{60}Fe_{20}B_{20}$ alloys isothermally held at 680K and 675K respectively*

Figure 1 illustrates typical thermograms for each alloy composition investigated. It can be seen that in changing the alloy composition the phase formation process differs, however, when investigating the effect of temperature of the isothermal hold, it was found that within the same alloy composition the phases formed do not differ.
In the $Co_{40}Fe_{40}B_{20}$ alloy a sole bcc CoFe solid solution is formed. Rietveld refinements have indicated that the lattice parameters are a = (2.88824; 2.880247; 2.88725)± 0.0009 Å for holds at 660K, 680K and 690K respectively.
Alternatively for the $Co_{60}Fe_{20}B_{20}$ the major peaks have been confirmed to be CoFe solid solution and $(Co,Fe)_2B$, there are however low intensity peaks which account for possibly >10% of the sample. Initial phase identification suggests that these are associated with α-Fe and $(Co,Fe)_{23}B_6$.

In addition to the phase formation analysis, the kinetics of the phase growth can be studied using the Johnson-Mehl-Avrami-Kolmogorov (JMAK) model.
The JMAK model for isothermal heating conditions describes the amount of transformed volume during a phase transformation as:

$$V(t) = 1 - exp\left(-\left(\frac{t}{\tau}\right)^n\right) \quad (1)$$

Where *V(t)* is the fraction of transformed volume in time *t*, the JMAK parameters *n and* τ represent the dimensionality of growth (Avrami exponent), and the time constant of phase formation respectively [4].
Figure 2 shows the integrated peak intensity of the 110 CoFe solid solution peak as a function of time. The solid line fitted to the data is the JMAK curve (Eq. 1); the parameters associated with this fit are given in the table alongside the graph. An Avrami parameter that lies between 1 and 2 suggest 1D or rod-like growth at all temperatures.



Temperature of Hold	Time constant (mins)	Avrami Exponent
660K	8717 ± 400	1.731 ±0.009
680K	460 ± 12	1.61 ±0.02
690K	142 ± 5	1.51 ±0.02

Figure 2 *Integrated peak intensity vs time of isothermally held for $Co_{40}Fe_{40}B_{20}$ alloy. The table lists the Avrami exponents determined from the curve fit.*

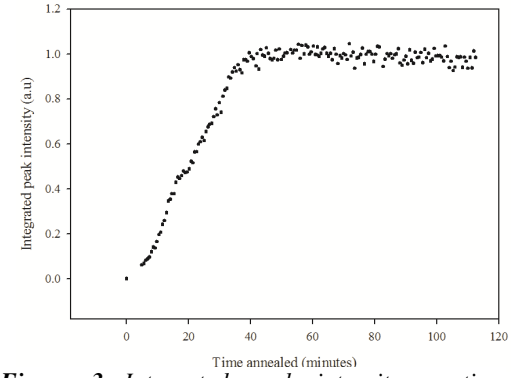


Figure 3 *Integrated peak intensity vs time isothermally held at 675K for $Co_{60}Fe_{20}B_{20}$.*

A similar analysis has been applied to the $Co_{60}Fe_{20}B_{20}$ alloy, however, as the phase formation is more complicated it has not been possible to isolate a single CoFe peak in the diffraction pattern.

This is clear from Figure 3 which shows that a second phase starts to grow at approximately 18 minutes. Since one of the assumptions for the JMAK model is that there is only a single phase being treated it cannot be applied to this set of data. Alternative models are being sought to determine the Avrami parameters for this alloy.

Conclusion

Synchrotron x-ray diffraction has been successfully used to investigate the crystallisation of $Co_xFe_{(80-x)}B_{20}$ alloys. It has been shown that the alloy composition has a large impact on the ultimate phase formation and transformation processes that the alloys undergo. Futhermore synchrotron x-rays have been used to follow the kinetic processes of a single phase formation. In order to complete this work a series of in-house techniques are being used; SEM and TEM pictures will confirm the JMAK parameters. Also DSC will be used to aid the JMAK analysis in the $Co_{60}Fe_{20}B_{20}$ alloy by employing the Kissinger method of analysis.

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References

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