Preparation of plan-view Co-doped FeSi thin film TEM specimens using FIB

M B Ward, N A Porter, P Sinha, R Brydson and C H Marrows

Institute for Materials Research, University of Leeds, Leeds, LS2 9JT
Department of Physics and Astronomy, University of Leeds, Leeds, LS2 9JT

m.b.ward@leeds.ac.uk

Abstract. When examining thin films using transmission electron microscopy (TEM), it is usually necessary to image a cross-section of the film (i.e. parallel to the film). However sometimes it is favourable to image thin films in plan-view (i.e. perpendicular to the film). This is the case for Co-doped FeSi thin films, which possess chiral symmetry along certain zone axes. In order to view these zone axes it is necessary to prepare the films in plan-view. There exist various ways to produce plan-view TEM specimens of thin films, such as back etching, ion milling and mechanical polishing. Here, a method using focused ion beam (FIB) is described in detail. Benefits of using FIB are that it is a quick process, there are no limitations in terms of substrate material, and samples can be produced from sections of substrate/film that may be too small to prepare any other way. The effectiveness of the preparation technique is also discussed here, with some preliminary energy dispersive X-ray (EDX) mapping data.

1. Introduction

Focused ion beam (FIB) is a well-established technique that has found applications in a number of areas due to its versatile fabrication capabilities and ability to image (either using secondary electrons generated by the ion beam or using an integrated scanning electron microscope (SEM)). One area that has become routine is in the preparation of samples for transmission electron microscopy (TEM) [1, 2].

The main benefits of using FIB to prepare samples for TEM are the ability to be site specific and the ability to apply the technique to a wide range of materials. The technique has uses in both the preparation of bulk materials and also thin film/multilayer materials. With respect to the latter, there is usually a requirement to view films in cross section using TEM. This allows for the determination of factors such as film thickness, epitaxy, de-bonding and elemental segregation. However in some cases it is favorable to view thin films in plan-view, and this is the case for the thin film Co-doped FeSi discussed in this paper.

Co-doped FeSi thin films grown in the 111 direction on a Si substrate are being investigated in order to establish a relationship between their structure and magnetic properties. Films composed of MnSi have been shown to form chiral domains, where reversals in the chiral symmetry in the [221] direction occur between regions of the order of a few microns [3]. In order to carry out similar investigations into Co-doped FeSi thin films, plan-view specimens need to be produced so that they can be analysed using TEM.

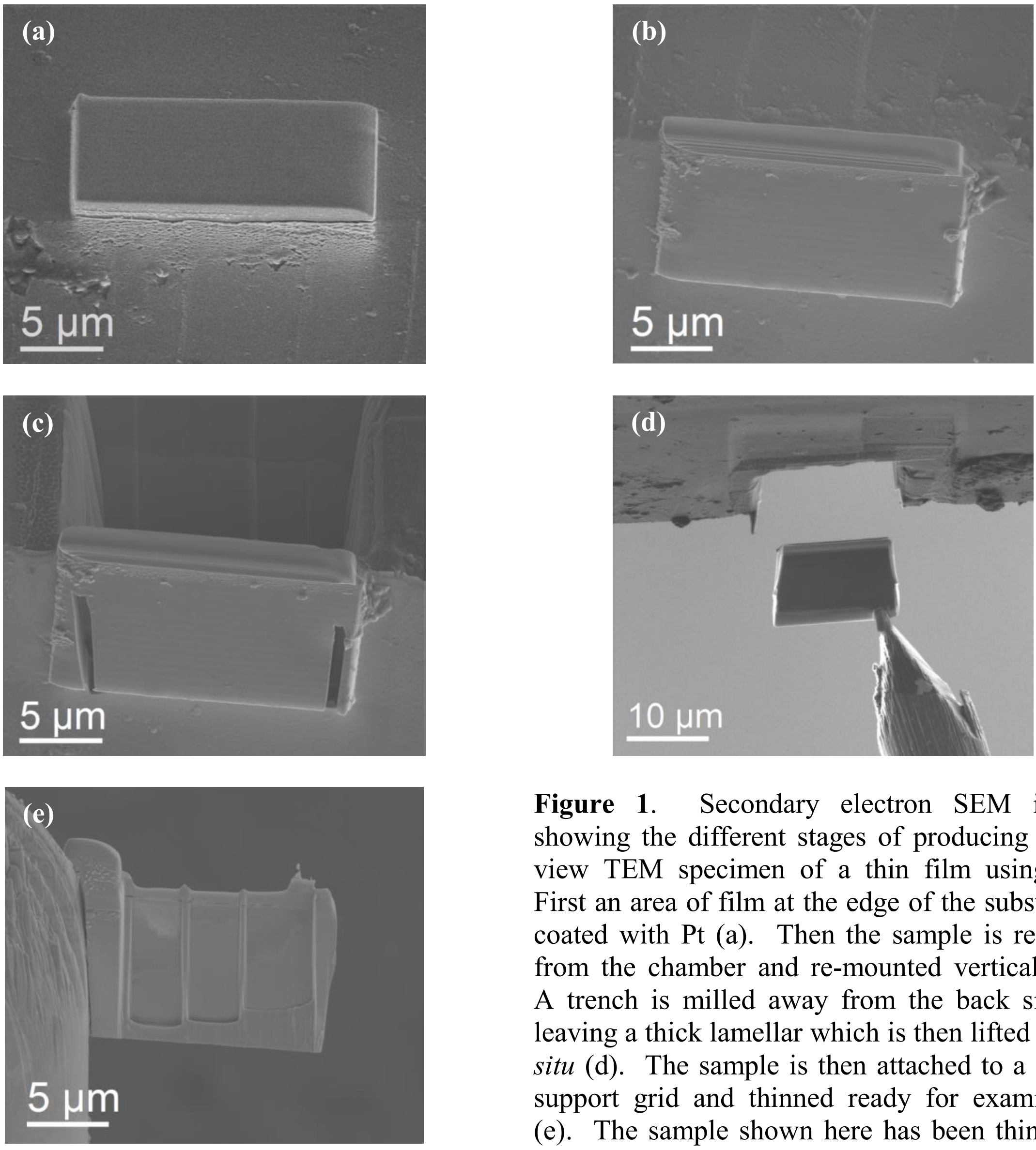

Figure 1. Secondary electron SEM images showing the different stages of producing a plan view TEM specimen of a thin film using FIB. First an area of film at the edge of the substrate is coated with Pt (a). Then the sample is removed from the chamber and re-mounted vertically (b). A trench is milled away from the back side (c) leaving a thick lamellar which is then lifted out *in-situ* (d). The sample is then attached to a copper support grid and thinned ready for examination (e). The sample shown here has been thinned in three separate windows for stability.

It has been demonstrated that plan-view thin film specimens can be produced via mechanical polishing [3], back etching [4] and ion milling [5]. It has also been shown that FIB can be used to produce plan-view TEM specimens [6, 7], but here a new FIB method for specifically producing plan-view TEM specimens of thin films is described. The reason for using FIB as an alternative method is that samples can be made from very small pieces of film on substrate (too small for fabrication by any other technique). This may be the case if samples can only be made in small dimensions and/or they need to be divided for characterisation by different techniques. Additionally it may be that FIB is the only technique that is available or the only that expertise exists for. Finally, if the films have been grown on a substrate material that is not compatible with any other sample preparation technique then FIB may be an alternative.

2. Methods

For this investigation a 50 nm thin film of Co-doped FeSi was grown via MBE on a <111> oriented Si substrate as described in [8]. TEM specimens were prepared using a FEI Nova200 Nanolab dual beam SEM FIB fitted with a Kleindiek micromanipulator for *in situ* lift-out. The ion column was operated at 30 kV for all steps except final cleaning of the specimen, where a voltage of 5 kV was used. Beam currents varied between 100 and 5000 pA. TEM was carried out using a Philips CM200 FEGTEM fitted with an Oxford Instruments X-Max 80mm^2 SDD EDX detector running Aztec software.

Figure 1 shows the main stages in the production of plan-view TEM specimens of thin films. The sample was first mounted horizontally in the chamber (that is with the thin film perpendicular to the axis of the SEM column) and a region at the edge of the thin film located. The reason for choosing an area at the edge of the film was so that the edge could serve as the top surface for the final prepared specimen and it could be easily lifted out. The electron beam was used to deposit a layer of Pt 200 nm thick over an area equal to the proposed specimen size (approximately 18 μm wide and 10 μm tall). The ion beam was then used to deposit a layer 1 μm thick over the same area (figure 1a). Next the sample was removed from the chamber and re-mounted vertically (with the thin film parallel to the axis of the SEM column) with the edge of the film that was identified in the previous step now topmost. The same area that was coated previously was located and an 18 μm × 2 μm layer of ion beam deposited Pt 1 μm thick was deposited on top (figure 1b). Preparation could now proceed in a similar fashion to standard TEM specimen preparation [1, 2] except material only had to be removed from one side of the specimen. This was done using a standard cross-section milling pattern, to reach a final depth of around 10 μm. The specimen was then partially cut-out (figure 1c). The sample was lifted out *in situ* (figure 1d) and then attached to a copper support grid. Thinning of the specimen could then proceed. This was first performed on the front face of the specimen that was covered in platinum during the first stage of preparation. The sample was oriented so that this front face was in-

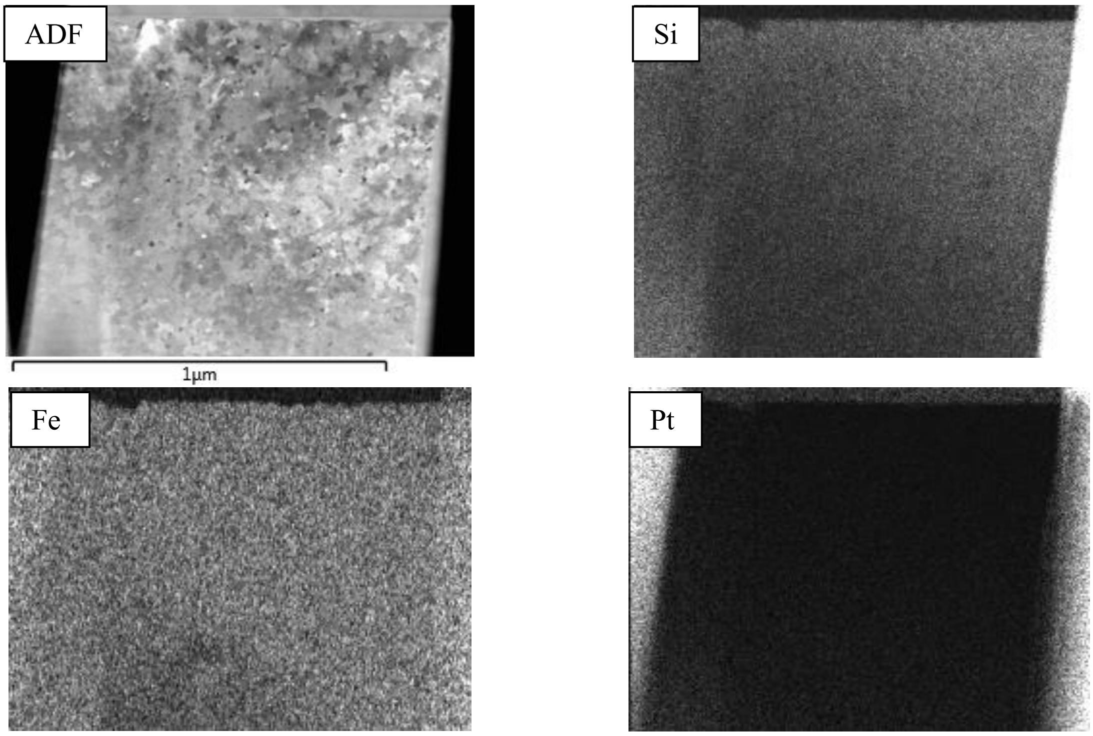

Figure 2. An annular dark-field (ADF) STEM image and corresponding EDX maps of Si, Fe and Pt of one of the thinned windows in the specimen. The Fe map confirms that the Co-doped FeSi film has largely remained intact over the entire window, and the absence of Pt signal at the very top of the window suggests that the protective Pt layer on the face of the specimen has mostly been removed by the thinning process.

line with the ion-column. This was checked by imaging the face with the ion column whilst varying the tilt of the specimen in increments of 1º. At the point where the face disappeared from view, it was assumed to be in-line with the column. Thinning then proceeded using a gradual cross-section cleaning milling pattern, with a final milling beam current of 100 pA. The tendency for TEM specimens to be left thicker at the base during the thinning process [1] meant that the Co-doped FeSi film was first revealed at the top of the specimen, and this could be easily observed through a change in contrast between the thinned region at the top and that below, with an abrupt interface between (this can be seen in the left-hand thinned window in figure 1e). At this point, thinning was halted so that the Co-doped FeSi was not completely milled away. Then thinning of the back-side was carried out in the same way with a counter-tilt of 1-2º to ensure the specimen had parallel sides [1]. Finally a 5 kV cleaning step was carried out on each side.

3. Results and discussion

Once the sample had been prepared, it was characterised via Scanning (S)TEM energy dispersive X-ray (EDX) mapping in order to check that the Co-doped FeSi film still remained and that the specimen was free from the protective platinum layer. STEM-EDX maps were collected for Si, Fe and Pt (figure 2). The Pt map shows that the protective layer has been mostly removed, which was important so that the Pt did not affect any subsequent TEM data (imaging of spectroscopic) collected from the specimen. The Fe map shows the coverage of the Co-doped FeSi map, which is largely complete across the entire thinned window. The Si map will contain signal from the Co-doped FeSi film, but also contributions from any substrate material that remains. Therefore, it is difficult to assess how effectively it has been removed by the thinning process. The presence of some Si substrate material should not affect the analysis in this case however, as any chiral domains (which are the features of interest in this sample) should still be visible via diffraction contrast.

4. Conclusions

A plan-view TEM specimen of a 50nm film of Co-doped FeSi on a Si substrate has been produced using a new FIB method. In principle the method can be used for any film/substrate materials. STEM-EDX mapping of the specimen has shown that the Co-doped FeSi film remains largely intact, and free from the deposited platinum used to protect the film during the process. The technique could certainly be used to produce specimens from thicker films (>50 nm), however great care would have to be taken when making specimens from thinner films so that the film was not lost during the thinning process.

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