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Magnetization reversal processes in hot-extruded τ-MnAl-C

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The magnetic domain structure of hot-extruded bulk τ-$Mn_{53}Al_{45}C_2$ was studied by Kerr microscopy under application of a magnetic field in-situ. The microstructure consists of recrystallized, fine-grained regions and large non-recrystallized grains which contain a high density of twins. Within these large polytwinned grains, a clear pinning interaction of magnetic domain walls at twin boundaries was observed but with a rather small pinning force. The smaller, recrystallized grains show a higher resistance to magnetization reversal. The critical single domain particle size of this material was estimated at 773 nm and the fine, recrystallized grains are in the range of this size. Demagnetizing the sample following saturation using a 3 T field pulse revealed that individual fine grains reverse independently from their neighbours.

1. Introduction

Rare earth free permanent magnets have recently drawn much attention because of their potential to exhibit energy densities between those of ferrites and rare earth based magnets [1–3]. MnAl is a highly promising candidate. In order to obtain a high magnetic remanence, a strong crystallographic texture is required and in MnAl-C, a uniaxial texture can be introduced by hot-extrusion [4]. The addition of C is beneficial in order to improve the resistance to thermal decomposition of the metastable τ-MnAl phase [4]. In spite of the promising attributes, the coercivity achieved in practice in MnAl-C is rather low [4] and must be improved before the full potential of the material can be realised.

Several aspects are thought to be important in determining the coercivity in this material system. These are, firstly, the pinning and/or nucleation of domain walls at intragranular defects, such as antiphase boundaries, stacking faults, twin boundaries or precipitates [5–8], and secondly, the influence of grain refinement due to dynamic recrystallization [9–12]. The fine grains possibly contribute to a high coercivity due to pinning of domain walls at grain boundaries [10,12,13]. Bittner *et al.* recently showed that three different types of twin boundaries are present in hot extruded MnAl-C and that the proportion of the three types changes according to the processing conditions [14]. In order to understand the influence of different microstructural features on coercivity, it is beneficial to study the behaviour of magnetic domains when applying a magnetic field to the sample. The magnetic microstructure of MnAl has been mainly studied with Lorentz Microscopy in the Transmission Electron Microscope (TEM) so far [5–7,11,12]. Due to the limited specimen thickness for TEM observations, it is difficult to draw conclusions from Lorentz microscopy that are applicable to bulk systems [15]. The current paper presents magnetic domain studies using Kerr microscopy and Magnetic Force Microscopy (MFM) of bulk, hot-extruded MnAl-C for the first time. Applying a magnetic field during the observation was used in order to reveal the strength and importance of the effect of different microstructural features on coercivity.

2. Experimental

The investigated sample was prepared by induction melting of 99.99% pure Mn and Al in addition to 99.9% pure C under argon atmosphere with a final nominal composition of $Mn_{53}Al_{45}C_2$ (at%). In order to homogenize the material, it was heat treated at 1100 °C for 2 days and then quenched into water. One part of the homogenized alloy was hot extruded at 680 °C with an area reduction ratio of 4.

The microstructure of the sample was studied with a Gemini Leo 1530 Scanning Electron Microscope (SEM). For the determination of the local crystallographic orientation Electron Backscattered Diffraction (EBSD) was carried out. Magnetic hysteresis loops were obtained by using a Vibrating Sample Magnetometer (VSM) and a Superconducting Quantum Interference Device (SQUID), in which different maximum applied magnetic fields were used. The Curie temperature, T_C, of the sample was determined by thermomagnetic measurements using a VSM.

The magnetic microstructure was studied with a Kerr microscope (polar contrast) and MFM using a high coercivity MFM tip

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(ASYMFMHC, Asylum Research). The MFM data was analysed using the software WSxM [16]. During the observation of the magnetic microstructure in the Kerr microscope, an out-of-plane magnetic field of up to 740 mT was applied in-situ.

In order to extract details of the magnetization reversal process, digital processing and analysis of the Kerr images was carried out using functions available in Python. The raw images taken at different applied field values were aligned using cross correlation in order to remove any lateral shifts of the analysis area. Low spatial frequency contrast variations were removed from the images and the contrast and brightness were set to similar values. Difference images were then calculated by subtracting the next image in the sequence from the previous one. Thresholding was carried out using a manually determined cutoff value in order to produce binary difference images. The bright regions in these binary difference images were then separated using a 4-fold connectivity and a size filter was applied to exclude any regions smaller than 15 pixels, which were considered to be noise. An ellipse was fitted to each of the remaining regions and the length of the major and minor axes of the ellipse were taken as a measure of the size of the regions detected. The parameters in the procedure were optimized for detection of differences in the fine recrystallized grains.

3. Results and discussion

The hot extruded MnAl-C material has been previously characterized using x-ray diffraction and SEM with EBSD [14], and was shown to consist primarily of the τ phase ($L1_0$, P4/*mmm*) with some precipitates of the equilibrium phase, β-Mn (Mn, P4132). Fig. 1(a) shows major hysteresis loops of the sample measured parallel (solid line) and perpendicular (dashed line) to the extrusion direction with a maximum field of 6 T. The sample exhibits remanence values of 0.47 T and 0.26 T, when measured parallel and perpendicular to the extrusion direction, respectively. From the difference in remanences, it can be deduced that an easy axis texture parallel to the extrusion direction has been formed during the processing. The degree of texture was calculated elsewhere [14] and was shown to be 0.45 which corresponds to a medium texture quality. The coercivity parallel to the extrusion direction is 0.16 T, while measured along the radial direction it is 0.20 T.

The Kerr microscope images of magnetic domains under in-situ application of a magnetic field are necessarily recorded in an open magnetic circuit. In order to be able to correlate the magnetic properties with the domain images from Kerr microscopy, two minor hysteresis loops were measured parallel to the extrusion direction up to maximum fields of 740 mT and 3 T, and these were not corrected for the effect of demagnetising fields (i.e. open magnetic circuit conditions). The former field value represents the maximum applied field during Kerr investigations (dashed line in Fig. 1(b)). An additional hysteresis loop is shown with a maximum applied field of 3 T (solid line in Fig. 1(b)) since for a particular experiment the sample was magnetized ex-situ with a 3 T field pulse before being transferred to the Kerr microscope. The coercivity values for the two minor hysteresis loops are 129 mT and 145 mT for the 740 mT loop and 3 T loop, respectively.

The microstructure of the sample can be seen in Fig. 2. The extrusion direction, which represents the easy axis of magnetization, is parallel to the image normal in all cases. It is clear from Fig. 2 that the microstructure contains two different constituents, namely fine-grained regions and large grains with a size of up to 50 µm. Hot extrusion leads to dynamic recrystallization which is accompanied by a drastic grain size reduction [4,9,10,12,14]. The remaining large, non-recrystallized grains reveal that the recrystallization is not fully completed during the extrusion process [14]. The remaining large grains show a high twin density, as can be seen clearly in Fig. 2(a). These twins are so fine that they are not fully resolved in the orientation map (Fig. 2(b)) derived from EBSD data with step size of 0.25 µm. Despite the high twin density, the large, non-recrystallized grains tend to show a single effective magnetic orientation, which can be deduced from the Kerr images (Fig. 2(c), (d)). The dimensions of the magnetic domains in the large grain on the right of the images are much larger than the twin structure and though a clear alternating domain contrast due to the twin structure can be seen, the formation of a global domain structure is not disrupted. The orientation of the effective magnetic easy axis of the grain can be estimated from the irregular and jagged global domain structure observed. If the magnetic easy axis were parallel to the image plane, stripe domains would be expected. In the case of the magnetic easy axis being perfectly perpendicular to the image plane, branched fractal-like domain patterns appear. The appearance of the magnetic microstructure visualized by Kerr (Fig. 2(c), (d)) presents a mixture of both scenarios. From that, it can be derived that the in-plane component of the effective easy axis lies as marked by the dashed line in the image but also that the effective easy axis has a certain tilt to the image plane.

It appears from the Kerr images as if the fine recrystallized grains are single domain particles. Only slightly larger grains appear to contain more than one domain. To prove that it is a real magnetic contrast, the orientation of the analyser within the light path of the Kerr microscope was changed, thus reversing the magnetic contrast. Light domains in the left image should therefore appear dark in the right one and vice versa. Examination of Fig. 2(c) and (d) shows this to

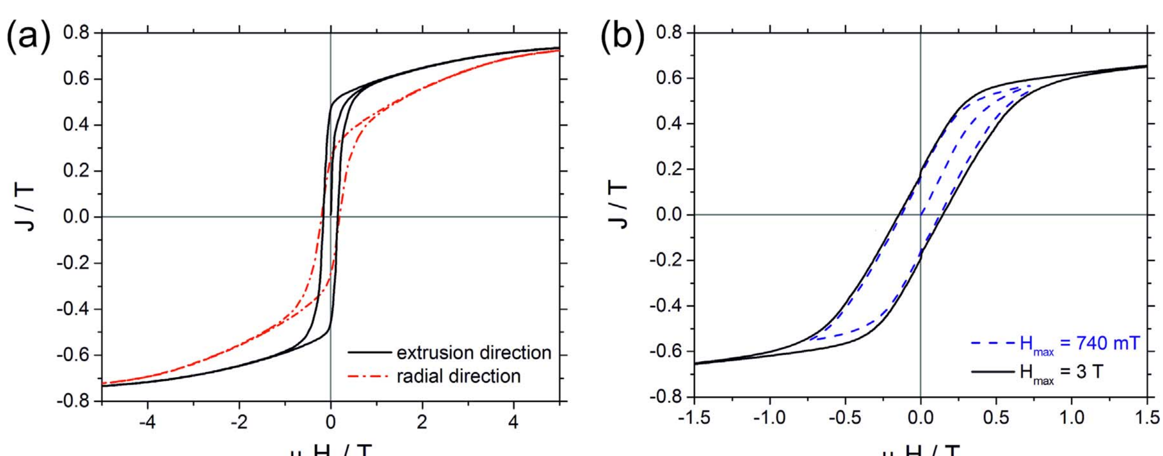

Fig. 1. (a) Hysteresis loops of hot-extruded MnAl-C measured parallel (solid line) and perpendicular (dashed line) to the extrusion direction (maximum applied field = 6T). The curves were corrected for demagnetizing effects. (b) Minor hysteresis loops with maximum fields of 740 mT (dashed line) and 3 T (solid line). The curves were not corrected for demagnetizing effects in order to show the situation in an open magnetic circuit, similar to that in the Kerr microscope.