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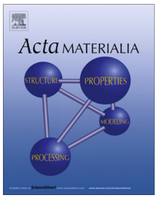

Twin-like defects in $L1_0$ ordered τ-MnAl-C studied by EBSD

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ABSTRACT

Twin-like defects in τ-MnAl-C, which has the $L1_0$ structure, have been studied using electron backscattered diffraction (EBSD) in as-transformed and in subsequently hot extruded samples. In both states, three distinct twin-like defects were found, whose misorientations were described by rotations of 62°, 118° and 180° about the normal to {1 1 1}. These are denoted as pseudo twins, order twins and true twins, respectively. The true twins are often observed in this type of material. The order twins formed the boundaries between regions where the *c*-axes were almost perpendicular to each other and these were thought to form due to the accumulation of strains during the transformation to τ from the hexagonal parent phase, ε. Due to symmetry, pseudo twins necessarily appeared at points where order twins interacted with true twins. The frequency of the different defects was very sensitive to the sample state. As the parent phase ε is not involved in the dynamic recrystallization which occurred during hot extrusion, there was a greatly reduced fraction of order twins and pseudo twins in the hot extruded state. The misorientation angle of the magnetically easy ⟨0 0 1⟩ axis across the three twin-like defects was 48°, 86° and 75° for pseudo, order and true twins, respectively. The interaction of the three twin-like defects with 180° magnetic domain walls and the resulting effect on the magnetic properties of the material may therefore be different.

1. Introduction

The ferromagnetic $L1_0$ phase (P4/mmm, AuCu I type) in the Mn–Al system, known as τ, has intrinsic magnetic properties (anisotropy constant $K_1 > 1$ MJ/m^3 [1], saturation magnetisation $\mu_0 M_s > 0.7$ T [1,2] and Curie temperature $T_c > 300$ °C [1,3]), which make it highly interesting as a possible rare earth free permanent magnet with an energy density between those of ferrites and rare earth based magnets [4–6].

The τ phase is metastable and is formed in the miscibility gap between β-Mn (P4132, Mn *cP*20 type) and the intermetallic γ_2 (R3 m, Al_8Cr_5 type) by undercooling the high temperature hcp ε phase ($P6_3$/mmc, Mg type) [3]. Early investigations suggested that the ε → τ transformation is martensitic [7,8] but more recent studies established a massive transformation mode [9–11] with additional features of a displacive transformation [12]. The addition of C on interstitial lattice sites has been shown to hinder the decomposition of the τ phase, thus facilitating processing at elevated temperatures [13].

Hot extrusion of as-transformed τ-MnAl-C followed by an aging treatment was demonstrated, using an alloy of composition $Mn_{52.7}Al_{44.9}C_{2.4}$ (at.%), to yield a uniaxially textured bulk magnet with remanence of $\mu_0 M_r = 0.61$ T, coercivity of $\mu_0 H_c = 0.32$ T and energy density of $(BH)_{max} = 56$ kJ/m^3 [13]. These extrinsic magnetic properties are greatly improved compared to the as-transformed state [13] but further improvements, particularly in coercivity, are required in order to make MnAl-C magnets more attractive for applications. An increased coercivity of $\mu_0 H_c = 0.48$ T was demonstrated in mechanically milled MnAl powder [14] and even $\mu_0 H_c = 1$ T was achieved in MnAl thin films [15] and therefore it seems likely that higher coercivities in bulk materials are also possible. Coercivity is very sensitive to the microstructure [16] and improvements may be achieved if detailed knowledge of the microstructural features and their formation mechanisms during processing can be obtained and if the type of interaction of these features with magnetic domain walls can be ascertained. Processing routes can then be optimised to favour the formation of microstructural features which are beneficial for coercivity, for example.

Several studies using transmission electron microscopy (TEM) over the last decades have reported the presence of defects such as stacking faults, antiphase boundaries and twins in τ-MnAl and

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MnAl-C [17–23]. The population of some defects is known to be sensitive to the sample state, for example antiphase boundaries were only observed in as-transformed samples but not after hot extrusion. In as-transformed samples, antiphase boundaries were reported to be often associated with magnetic domain walls [18–21] and therefore the higher coercivity of hot extruded samples has been partly attributed to the absence of antiphase boundaries [20,22]. Grain refinement, which occurs via dynamic recrystallization during hot extrusion [20,22], has also been reported to contribute to the increase in coercivity [20,24], possibly due to pinning of domain walls at grain boundaries [25]. Twins are common in both as-transformed and hot extruded conditions [18,20,21,23] and domain walls have been observed to be present at twin boundaries in both states [18,21,22]. From these results, an assessment of whether twin boundaries act as nucleation or pinning sites for magnetic domain walls cannot be made and therefore the effect of such boundaries on the coercivity is unclear.

The $L1_0$ structured γ-TiAl is analogous to τ-MnAl-C in that the parent phase has hexagonal symmetry in both cases. Three crystallographically different types of twin-like defects have been distinguished in γ-TiAl ($c/a = 1.02$ [26]) and the misorientation across the defect boundaries can be conveniently described by rotations of multiples of 60° about the normals to $\{1\,1\,1\}$ planes [27,28] (see also Table 1). The $L1_0$ crystal structure can be described by two different unit cells: one contains 4 atoms (Pearson symbol *tP*4) and is related to fcc and the other contains 2 atoms (*tP*2) and is related to bcc. The lattice parameters of the two cells are related thus: $c_{tP2} = c_{tP4}$ and $a_{tP2} = \sqrt{2}\, a_{tP4}$. In this study the *tP*4 cell will be used. The existence of the three twin-like defects in $L1_0$ structured γ-TiAl can be attributed to two different twinning modes, which are similar to those found in the related fcc and bcc structures. Defects described by rotations of 60° or 180° about $\{1\,1\,1\}$ derive from the fcc-based twinning mode $\langle 1\,1\,\bar{2}\rangle\ \{1\,1\,1\}$, where the twinning plane is $\{1\,1\,1\}$ and the shear direction is $\langle 1\,1\,\bar{2}\rangle$. The symmetry reduction of the $L1_0$ structure compared to fcc leads to a splitting of this twinning mode into two crystallographically different defects. Those described by a rotation of 180° about $\{1\,1\,1\}$ are known as true twins and those described by a rotation of 60° about $\{1\,1\,1\}$ are known as pseudo twins. The bcc-based twinning mode can be alternatively expressed using the *tP*4 cell as $\langle 1\,0\,1\rangle\ \{1\,0\,\bar{1}\}$. This can be described by a rotation of 120° about $\{1\,1\,1\}$ and such defects are referred to as order twins [29]. The order twins observed in γ-TiAl separated regions in which the *c*-axes were perpendicular to each other and the formation of these so-called *c*-domains was attributed to the accommodation of strain during the transformation from the hexagonal parent phase [28]. Such *c*-domains and the associated boundaries are also observed in systems where the parent phase is fcc [28,29].

To date only the existence of boundaries described by 180° rotations about $\{1\,1\,1\}$, *i.e.* true twins, has been shown in as-transformed and hot worked τ-MnAl using TEM [22,23]. The limitation of such high resolution studies is that only a relatively small area is available for analysis. Electron backscattered diffraction (EBSD) is an excellent alternative because it allows the investigation of much larger areas, albeit at a lower spatial resolution. In the current work, a detailed microstructural study of twin-like defects in MnAl-C samples in the as-transformed and hot extruded states has been carried out using EBSD for the first time. The aim of the work was to investigate whether all three twin-like defects are present in as-transformed and in subsequently hot extruded τ-MnAl-C and if so, whether the fractions of the three types are affected by the extrusion process. As the effect of twin boundaries on coercivity in MnAl-C is currently unknown, the results of this study will help to accelerate the development of MnAl-C magnets with improved properties.

Table 1
Summary of the 3 different twin-like defects in γ-TiAl $L1_0$ ($c/a = 1.02$ [26]), which are distinguished by rotations of multiples of 60° about the crystallographic $\{1\,1\,1\}$ pole. Note that the twinning plane and shear direction of the order twin are given in *tP*4 notation.

Rotation angle about $\{1\,1\,1\}$	Denotation	Related twinning mode	Twinning plane	Shear direction
60°	Pseudo twin	fcc	$\{1\,1\,1\}$	$\langle 1\,\bar{2}\,1\rangle$
120°	Order twin	bcc	$\{1\,0\,\bar{1}\}$	$\langle 1\,0\,1\rangle$
180°	True twin	fcc	$\{1\,1\,1\}$	$\langle 1\,1\,\bar{2}\rangle$

2. Experimental procedure

An alloy with nominal composition $Mn_{53}Al_{45}C_2$ (at.%) was prepared by induction melting 99.99% pure Mn and Al with 99.9% pure C under argon atmosphere and was then cast in a cylindrical Cu mould of 10 mm diameter. The as-cast material was encapsulated in a glass tube which was evacuated to 10^{-4} mbar and then filled with 150 mbar of pure Ar. The tube was then placed into a furnace at 1100 °C and was left there for 2 days in order to homogenise the material. The tube was then removed from the furnace and quenched into water. Part of the homogenised material was hot extruded at 680 °C with an area reduction ratio of 4.

X-ray diffraction (XRD) was carried out using a Bruker diffractometer with Co-Kα radiation. The microstructure was studied using a Gemini Leo 1530 Scanning Electron microscope (SEM). The local crystallographic orientation was determined by electron backscattered diffraction (EBSD) measurements. An evenly spaced grid of measurement points with a spacing of 250 nm or 100 nm was chosen in the analysis region, yielding in total 40,000 data points for each measurement.

The magnetic properties were measured using a Quantum Design SQUID with applied fields up to 5 T at room temperature. The data were corrected for demagnetisation effects.

3. Results and discussion

3.1. General microstructure, texture and magnetic properties

The XRD pattern of the as-transformed sample is shown in Fig. 1. All reflexes can be attributed to τ. The inverse pole figure map of the as-transformed sample (Fig. 2a) is derived from EBSD data and shows the orientation of the sample normal direction with respect to the crystallographic directions. The as-transformed sample consisted of irregularly shaped grains of the τ phase, several tens of μm in size (Fig. 2a). In agreement with

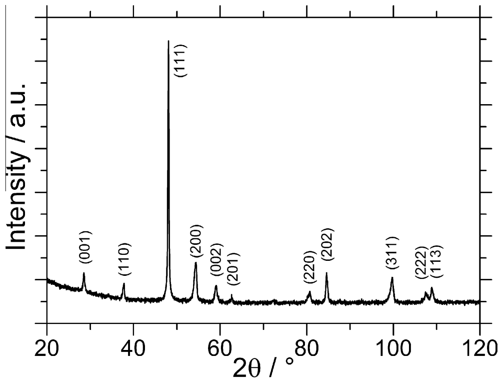

Fig. 1. XRD pattern of the as-transformed sample after quenching.