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Trace analysis for magnetic domain images of $L1_0$ polytwinned structures

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Abstract

The polytwinned structure in $L1_0$ FePd alloy is studied by using both conventional TEM and Lorentz microscopy methods. Herein we develop a trace analysis method to determine the surface orientation of a magnetic domain image without the use of electron diffraction patterns. This allows us to analyze magnetization directions.

Keywords: $L1_0$; Magnetic domain; Twin; Transmission electron microscopy; Lorentz microscopy

1. Introduction

The equi-atomic FePd and FePt alloy can develop a polytwinned microstructure after it undergoes the atomic ordering transformation. During the atomic ordering, the high temperature FCC phase transforms into the low temperature $L1_0$ phase which has a c/a ratio equal to $\sim$0.966. Because of this tetragonal lattice misfit of the tetragonal phase with the cubic phase matrix, a large amount of strain energy is induced. The formation of the polytwinned structure is due to the minimization of this atomic ordering strain energy [1]. In the polytwinned structure, a macro-twin variant is composed of elemental $L1_0$ twin variants [1,2]. At room temperature, each ordered $L1_0$ twin variant is ferromagnetic and has the magnetization direction along its c-axis. Therefore, the corresponding magnetic domain structure is very complicated.

The morphology of the magnetic domain structure in FePd polytwinned structure has been studied extensively by using both optical methods [2] and Lorentz microscopy methods [3]. It has been shown that the magnetic domain structure is sensitive to the specimen surface orientation. Sokolovskaya et al. studied the magnetic domain pattern on two low index surfaces, plane (101) and (010) in a single crystal FePt alloy. For polycrystalline FePd or/and FePt, the specimen surface may not be low index plane. In this case, electron diffraction methods are often used to find out the surface orientation. However, when imaging the magnetic domain pattern, the electron diffraction method is not appropriate, because the sample is acted upon by the large magnetic field of the objective lens. Thus the TEM sample could easily change its arrangement of the magnetic domains. In this study, we develop a trace analysis

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method to determine the surface orientation of a polytwinned FePd thin film. It is a method of stereographic analysis to determine the traces of twin planes in an ordered phase [4]. The surface normal can be calculated by knowing the type of planes that show their traces on the specimen surface, and by measuring the angles between these traces. This method works not only for low index specimen surface normal, but also gives high accuracy for high index specimen surface.

2. Experimental procedure

The Fe–55at.%Pd alloy used in this study was prepared by using an arc-melting process. The bulk Fe–Pd alloy was homogenized at 860 °C for 43 h. It was further annealed at 500 °C for 36 h, followed by water quench. The annealing temperature used here is slightly above the Curie temperature ($T_c = 730$ K) [1]. From the X-ray diffraction scan, we know the alloy was chemically ordered after the annealing. Thin foils for TEM were electro-polished in a twin-jet polisher using 25 V at 0 °C. The solution used was 82 vol.% glacial acetic acid, 9 vol.% perchloric acid and 9 vol.% ethanol [3].

The TEM study was carried out at 400 kV using a JEOL 4000 TEM. The detailed construction of this TEM can be found elsewhere [5]. During the Lorentz microscopy imaging, the objective lens is turned off, and the sample is mounted in an almost field-free region. The objective mini-lens focuses the electrons into a diffraction pattern at the selected area (SA) aperture plane. The maximum magnification is about 3000×, with an additional factor of 20× achieved by using a Gatan imaging filter. The Fresnel imaging mode was used in Lorentz microscopy imaging. In this mode, the SA aperture is taken away from the back focal plane. The objective mini-lens is over-focused or under-focused, and the magnetic domain walls are imaged as alternate bright and dark lines.

3. Result and discussion

Fig. 1a and c show the Fresnel magnetic domain wall image of the annealed FePd alloy. The polytwinned structure indicates that it is the ordered phase with $L1_0$ structure. The bright and dark lines are the magnetic domain walls. We can see some of the magnetic domain walls coincide with the twin planes and some of the domain walls run across many twin variants. The bright lines and dark lines change their contrast when imaged from under-focus Fresnel mode to over-focus Fresnel mode. The magnetic domain walls and twin planes interact with each other and form a complicated magnetic domain structure. To analyze these magnetic domain images, we first need to know the surface orientation. We use trace analysis to obtain the surface orientation, which is described below.

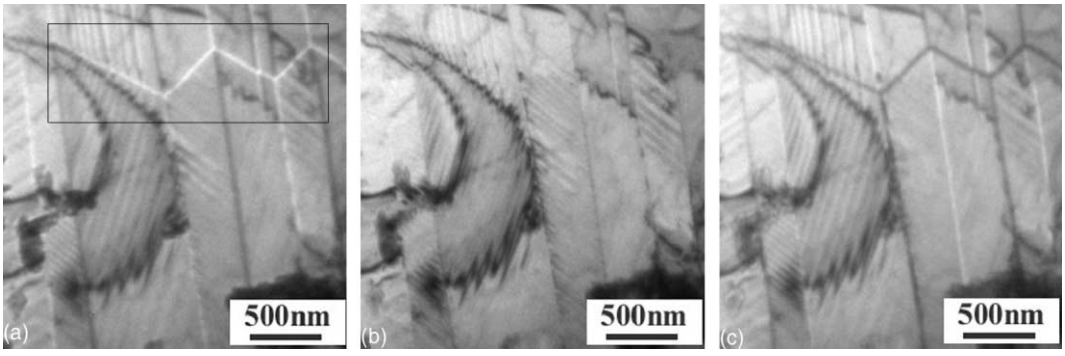

Fig. 1. Lorentz images of ordered Fe–55at.%Pd Alloy, the image was taken at 0° tilting. (a) Fresnel under-focus magnetic domain wall image. (b) Fresnel in-focus image magnetic domain wall image. (c) Fresnel over-focus magnetic domain wall image, note the changes of domain wall contrast compared with (a).

3.1. Trace analysis

Due to the twinning mechanism of $L1_0$ variants [6], the twin planes are the equivalent FCC $\{1\,1\,0\}$ planes in the FePd $L1_0$ structure. These twin planes intersect with the surface and leave their traces on the surface. The directions of traces and the angles between the twin plane traces are unique for a certain surface orientation. Table 1 lists the angles between twin plane traces on several low index surfaces.

This table was obtained as following: we first calculate the traces of $\{1\,1\,0\}$ planes on the listed low index surfaces in a FCC structure; then we calculate the angles between these traces and the $(1\,1\,0)$ plane trace. The plus sign indicates counterclockwise rotation with respect to the $(1\,1\,0)$ plane trace on the surface. The minus sign indicates the clockwise rotation with respect to the $(1\,1\,0)$ plane trace. By comparing the measured angles with the angles in Table 1, we can find the surface orientation for low index surfaces. For high index surfaces, we use a computer program to do the search. The inputs for the computer program are three unique angles between the twin plane traces measured from the magnetic domain image, the accuracy of the angle measured, and the range of the index needed.

Fig. 2 shows the schematic diagram of the measured angles from the Fig. 1. There are four types of twin plane traces, A, B, C and D. For each twin plane trace, the two lines (a and b) are the traces of the twin plane's intersection with the top and bottom surface of the thin sample.

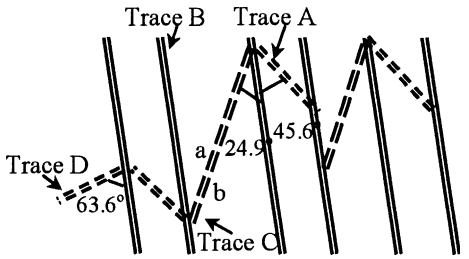

Fig. 2. The schematic diagram of the measured angles from Fig. 1. A, B, C, D are the traces of the $\{1\,1\,0\}$ planes.

In Fig. 2, each angle is measured by using image-processing software. The accuracy of the measurement can reach 0.5°. The range of index is chosen to be 20, that is, searching from $\{-20,-20,-20\}$ to $\{20,20,20\}$. It is not surprising that during the calculation, many other surface orientations also satisfy the measured angles. However, by carefully comparing the plus and minus signs of these angles, we limit the surface normal to the following indices: $\{-19,1,7\}$, $\{-18,1,7\}$ and $\{-16,1,6\}$. The maximum deviation among these three plane normals is 0.59°. More possible indices for the surface normal can be found when the range of index is increased. But these indices are so close to each other, that it becomes unnecessary to go to higher index given the accuracy of the angle measurement is 0.5°.

We also used a conventional TEM method to determine the surface orientation for this polytwinned structure. We kept the image at the same sample position and tilted the specimen to two known zone axes, namely $[0\bar{1}0]_1/[\bar{1}00]_2$ and

Table 1
A list of the angles between the traces on different surface planes

Surface	The angles of the plane traces with $(1\,1\,0)$ plane trace							
(h,k,l)	$(1,0,1)$	$(0,1,1)$	$(1,-1,0)$	$(1,0,-1)$	$(0,1,-1)$	$(1,0,0)$	$(0,1,0)$	$(0,0,1)$
$(0,0,1)$	−45.00	45.00	−90.00	−45.00	45.00	−45.00	45.00	–
$(0,1,0)$	−45.00	−90	0.00	45.00	90.00	0.00	–	−90.00
$(1,-1,0)$	54.74	54.74	–	−54.74	−54.74	0.00	0.00	90.00
$(1,-1,-1)$	60.00	30.00	90.00	−30.00	−60.00	30.00	−30.00	90.00
$(1,-2,0)$	48.19	65.91	0.00	−48.19	−65.91	0.00	0.00	90.00
$(1,-2,-1)$	58.52	58.52	58.52	−31.48	−62.96	19.29	−31.48	97.75
$(1,-2,-2)$	61.93	30.96	75.96	−14.04	−59.04	30.96	−40.60	102.53
$(1,-3,0)$	46.51	72.45	0.00	−46.51	−72.45	0.00	0.00	90.00
$(1,-3,-1)$	56.44	73.22	39.66	−33.56	−67.11	14.31	−33.58	98.57

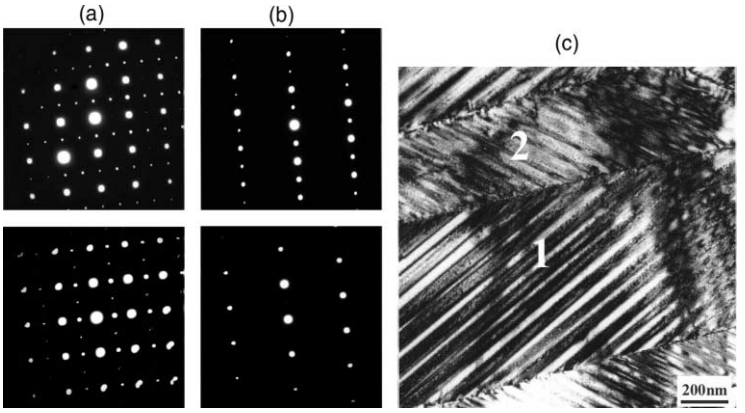

Fig. 3. The diffraction pattern of two zone axis at two different regions. (a) $[0\bar{1}0]_1/[\bar{1}00]_2$ zone axis diffraction pattern. (b) $[1\bar{3}0]_1/[\bar{3}01]_2$ zone axis diffraction pattern. (c) The bright field image of the same area as shown in Fig. 1. The subscripts 1 and 2 indicate where the diffraction patterns are taken from, region 1 or 2.

$[1\bar{3}0]_1/[\bar{3}01]_2$ axes (Fig. 3a and b). The subscripts 1 and 2 indicate the diffraction patterns are taken from different regions (Fig. 3c). The diffraction patterns are indexed with respect to an ordered $L1_0$ grain. From the tilting angle, we calculated the surface orientation to be $[1, -3.096, 0.106]$ (region 1) and $[-3.096, 0.106, 1]$ (region 2) respectively. These two surface orientations are equivalent in the FCC reference frame. We can conclude that regions 1 and 2 were from the same FCC grain, and the surface orientation is $\langle -3.096, 0.106, 1\rangle$ in this FCC grain. This is close to the ones we obtained from the trace analysis. The deviation between the surface orientations obtained by trace analysis and the electron diffraction is below 3.5°.

When using the electron diffraction method, errors can be introduced from many sources, such as tilting angle goniometer readings and deviation from the exact zone axis. But in the trace analysis, the accuracy of the surface orientation is mainly dependent on the accuracy of angle measurement. This leads us to believe that the trace analysis can be more accurate in determining the surface orientation in this polytwinned microstructure. By using the image-processing software and statistical analysis, this error can be limited to 0.5°.

3.2. Analysis of magnetic domain image

Although trace analysis gives only the equivalent family of the surface normals, it still can be used to find out the *c*-axis orientation for different $L1_0$ variants (Fig. 4a). We take a FCC grain as the reference, during the ordering, the $L1_0$ variant *c*-axis can take any of the three $\langle 100\rangle$ axis orientations. Therefore, by calculating the angle between the three $\langle 100\rangle$ orientations with respect to any of the equivalent image surface normal, we can determine the orientation of the *c*-axis in the space for different $L1_0$ variants. This method is only valid when the $L1_0$ variants are ordered within the same FCC grain.

Since the *c*-axis is the easy axis of magnetization in FePd tetragonal variants, the magnetization vector will be along this direction. From the trace calculation as shown in Table 1, the twin plane traces (A, B, C, D) and the $\langle 100\rangle$ direction projections (p) on the surface plane can be plotted together onto the surface plane (Fig. 4b). DW1 and DW2 are the directions of the zigzag domain walls measured from Lorentz micrograph of Fig. 1a. Fig. 4c shows a schematic magnetization induction mapping corresponding to the enclosed area in Fig. 1a. In this diagram, small arrows indicate the projection of the *c*-axis of the $L1_0$ variants on the surface plane. Large arrows are parallel to the zigzag domain wall DW1 and DW2; the directions of which are the combination of the small arrows. These large arrows represent for the average magnetization direction in magnetic macro-domains. The magnetic macro-domain walls are illustrated as bright and dark solid lines. The

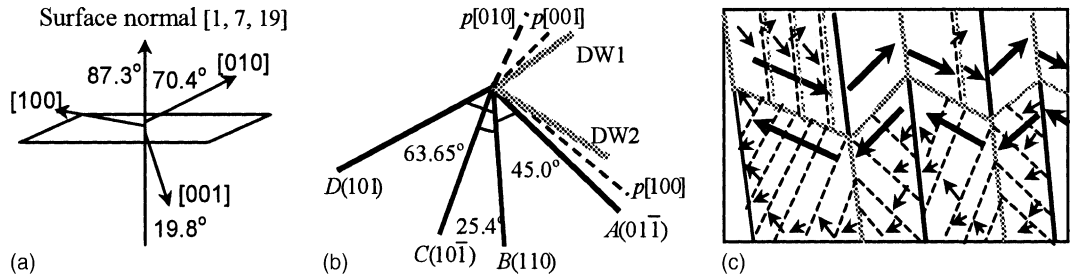

Fig. 4. (a) The c-axis orientation for $L1_0$ variants. The surface normal is [1,7,19]. (b) The twin plane traces $\{1\,1\,0\}$ and $\langle 1\,0\,0\rangle$ direction projection on surface plane. DW1 and DW2 are directions of zig-zag domain walls. (c) Schematic magnetic domain wall image corresponds to the enclosed area in Fig. 1a. Small arrows indicate the magnetization direction of twin variants projected on the surface plane. Large arrows are the combination of small arrows and are parallel to the zigzag domain walls. The dashed lines are the $\{1\,1\,0\}$ twin plane traces.

dashed lines are the $\{1\,1\,0\}$ twin plane traces. Since the easy axes of the adjacent twin variants are 90° apart from each other, the magnetic micro-domain walls coincide with the twin planes. In Fig. 1, some of the twin plane traces show the bright and dark contrast of magnetic domain walls, while others do not. It is possible that the contrast of the later is dominated by the diffraction contrast, since the c-axis of one of the variants is nearly parallel to the beam direction.

4. Conclusions

In this work, we developed a trace analysis method, which determines the surface orientation of the magnetic domain image of FePd polytwinned structure. This method can help us gain the crystallographic information about the structure without taking electron diffraction patterns and therefore without altering the magnetic domain arrangements. In order to achieve accurate index, three unique angles need to be measured. The accuracy of the angle measurement directly influences the accuracy of the analysis. Therefore, it is recommended to use image-processing software to measure the angles. After the trace analysis, conventional transmission electron microscopy is used to verify the surface orientation. We find out that the trace analysis provides high accuracy of within 5° for both low and high index surface normal. Compared with the electron diffraction method, the trace analysis is easier, more convenient and can be more accurate.

By knowing the surface orientation, we can further determine the magnetization direction in the structure and qualitatively construct the magnetic induction mapping.

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