EBSD Characterization of the Twinning Microstructure in a High-Damping Mn–Cu Alloy

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The (101) transformation twinning bands in Mn–Cu alloys are considered to be the dominant microstructural feature contributing to the high damping capacity of the alloys. Because the face-centered-tetragonal (fct) phase of Mn–Cu alloys has an axis ratio (c/a) of nearly 1, an angular resolution for Kikuchi diffraction bands of better than 0.5° is necessary to identify the twin boundaries by the electron backscattering diffraction (EBSD) method. At some angular resolutions, (100) and (001) orientations of the fct phase may be randomly recorded within the single twinning band. In contrast, the image-quality signal of EBSD shows a reflection that is sensitive to the twinning bands. The image-quality contrast is produced by the average orientation difference between the twinning bands. Therefore, the twinning bands of Mn–Cu alloys can be identified by partitioning the orientation distribution of the normal direction for the observed sample section in a range of about 1°. With the aid of (101) stereographic projection, the width of the twinning bands and the intersection morphology were analyzed for a Mn–Cu alloy. The observed intersection morphology was related to a 60° or 90° spatial intersection configuration of different groups of twinning bands. In addition, sharpened ends of twinning bands at the junction regions and secondary twinning phenomena were also characterized.

Keywords: Manganese–copper alloy, Electron backscattering diffraction, Twin boundary, Twinning bands, phase transformation.

1. Introduction

The high damping capacity and magnetic properties of Mn–Cu alloys have attracted the interest of many researchers.1,2 The most important feature of those alloys is the fcc–fct phase transformation, which is closely associated with the paramagnetic–antiferromagnetic transition at the temperature $T_N$. In the temperature region below this phase-transformation temperature, the characteristic microstructure of those alloys is the [101] phase-transformation twin in the fct structured product phase. According to the double-shear model developed by Bowles et al.,3) the fct lattice with $c/a = 1 + 3/2c$ is transformed from an fcc lattice by successive shears of $\varepsilon(110)(110)$: this double-shear transformation model is illustrated schematically in Fig. 1. When the first shear has occurred on a (101) plane, the secondary shear necessary for the lattice transformation can proceed on two equivalent [110] planes. As shown in Fig. 1, as a result of the different secondary shears, two fct lattices appear, designated $(a', b', c')$ and $(a'', b'', c''$, respectively. The common (101) plane shared by the two fct lattices, i.e. the first shear plane in the parent fcc lattice, will become the twin boundary separating the two corresponding fct lattices by minimizing the phase-transformation strain energy. Consequently, the misorientation between the two fct lattices can be expressed as $\theta_{(\text{MT})} = 90° - 2\tan^{-1}(c/a)$. $M$ and $T$ represent the two twinning bands that share the (101) twin boundary. As in the configuration shown in Fig. 2, four equivalent {101} twin boundaries that appear in the intersecting or parallel arrangement within each crystal grain have been observed in Mn–Cu alloys.4,5)

On the other hand, the main damping peak observed in Mn–Cu alloys occurs at nearly the same temperature irrespective of the composition of the alloy and it shifts to higher temperatures at higher vibration frequencies.6) The damping peak has therefore been attributed to a relaxation process associated with the movement of the {101} twin boundaries in the fct phase of Mn–Cu alloys. The amount and configuration of the twin boundaries and the boundary orientation relative to the direction of vibrational stress should therefore have a large effect on the damping behavior. In a recent study, both the peak damping capacity and the activation energy of Mn–30 at% Cu alloy were found to be markedly changed by treating the alloy in different ways while holding the same phase-transformation temperature.5) It was suggested that the twinning microstructure of the alloy was changed by the different treatment methods and that the variation in twin-band configuration caused the change in the twin boundary-related damping behavior. The twinning microstructure of Mn–Cu alloys has generally been observed and analyzed by means of transmission electron microscopy (TEM).4,7,8) However, it appears to be difficult to apply TEM to obtain statistical results on the spatial configuration of the
twin boundaries in order to relate the microstructural characteristics of the alloys to their damping behavior. The electron-backscattering diffraction technique (EBSD) has been developed for characterizing microstructures from information on the spatial crystallographic orientation of probed points; it should, therefore, be feasible to identify the \{101\} twin boundaries in Mn–Cu alloys by applying EBSD. In the present work, EBSD analysis was applied to a Mn–Cu alloy that shows the typical twinning microstructure at room temperature to identify the statistical features of the twin boundaries in the alloy. The misorientation between the corresponding twinning bands is only about $1/14^\circ$, so this is a challenge to EBSD analysis, although the orientation accuracy of EBSD is claimed to be about $0.5/14^\circ$.

2. Experimental Procedure

A Mn–22.6Cu–5.42Ni–2.06Fe (mass\%) alloy was used in the present work. An induction-melted ingot was forged and rolled at 1173 K, and sheets of 1-mm thick were produced by cold rolling. After solid-solution treatment at 1173 K for $3.6 \times 10^3$ s, the sheet specimens were water-quenched and then aged at 723 K for $4.3 \times 10^4$ s. The transformation temperature of the aged sample was found to be 353.7 K from the minimum Young’s modulus variation measured by dynamic mechanical analysis. The lattice constants of the \textit{fcc} phase at room temperature were found to be $a = 0.374$ nm, $c = 0.368$ nm ($c/a = 0.984$) by x-ray diffraction measurements.

An electron backscattering diffraction (EBSD) system (TSL Inc.) attached to a LEO-1550 scanning electron microscope (SEM) with a Schottky-type field-emission gun (Carl Zeiss Inc.) was used at an operating voltage of 20 kV and a probe current of about 500 pA.

3. Results and Discussion

3.1 Analysis of the \{101\} Twinning Boundary in Mn–Cu Alloy by EBSD

Figure 3(a) shows the twinning microstructure in the aged alloy obtained by image-quality (IQ) contrast in EBSD. The average grain size of the parent \textit{fcc} phase in the alloy is about 100 µm and the observed grain shows an orientation with a lattice axis (e.g., $a$) normal to the sectioned plane. Two kinds of twin band in the grain are observed as a result of the IQ contrast shown by the coupled twinning bands. The two groups of twinning bands intersect almost perpendicularly and show different band widths. To confirm the twinning orientation relationship between the \{101\} twin boundaries, the misorientation distribution of the observed grain was determined and is shown in Fig. 3(b). Misorientations at both a low angle ($\approx 5/14^\circ$) and near $90^\circ$ are found to occupy the main fraction. As described in the Introduction, a difference in orientation occurring at the twinning boundaries should be characteristic of the two kinds of misorientation, taking into consideration the 90° rotation symmetry of the \textit{fcc}-structured crystals. In other words, the twinning boundaries (at least those near 90°) should be the corresponding sites showing this type of characteristic misorientation. Figures 4(a) and 4(b) show the boundary trace images of the observed grain with the two characteristic misorientations, $\approx 0.5^\circ$ and near...
Note that the twin boundary traces contrasted by the IQ image are not reflected by boundaries with two characteristic misorientations. Both kinds of boundaries show a random distribution in the observed grain.

Because the observed grain has an aligned orientation with one lattice axis parallel to the normal direction of the sectioned plane, twin boundaries can be found by distinguishing the parallel \( a \) and \( c \) axes in the coupling crystals. Figure 5 shows the simulated Kikuchi diffraction patterns of the \( fct \) phase at (001)[010] and (100)[010] orientations, respectively. The angles between the \{113\} Kikuchi bands provide the basic information that is required to differentiate the two orientations during EBSD analysis. As shown in the figure, the two characteristic angles between the two sets of intersecting \{113\} bands are 144.4° and 144.95° for the (001)[010] orientation and 145.35° and 144.4° for the (100)[010] orientation, respectively. According to the \( c/a \) parameter of the \( fct \) phase, the misorientation between the twinning bands \( \theta_{MT} \) is 0.924° in the investigated alloy. However, to distinguish the two characteristic orientations, i.e. the \( a \) or \( c \) axis of the \( fct \) phase, a Kikuchi band angular resolution of less than 0.5° is required for the present alloy. It appears that the present EBSD measurement conditions, such as the scan step and the exposure time, are not suitable for producing the high angular resolution necessary for the \( fct \) phase. As a result, the \( a \) or \( c \) axis is randomly detected and recorded within one twinning band that is just a single crystal.

The contrast shown by the image quality is mainly influenced by the strain state and orientation of the detected areas. In aged Mn–Cu alloys, the twinning bands contrasted by the IQ graph shown in Fig. 3(a) are considered to be the result of a difference in orientation between the twinning bands. Figure 6 shows the orientation distribution in the normal direction of the observed section in a similar fashion to that shown in Fig. 3(a). Both (100) and (001) orientations are detected for the observed section. In the ideal case where the resolution of EBSD is sufficiently high to distinguish the \( a \) or \( c \) axis of the \( fct \) phase, the detected (100) and (001) orientations correspond to the respective twinning bands. As discussed above, the boundaries at either a low angle or near 90° should coincide with the twin boundaries. It is the inadequate EBSD resolution that provides a (100) orientation...
with contributions from all the observed area. However, twinning bands with an average difference in orientation of about 1° could be identified by partitioning the dispersive (100) orientation on the stereographic projection of (100) enlarged to 5°, as shown in Figs. 6(a) and 6(b). Figures 6(c) and 6(d) show the (001) orientation distribution in the same observed grain. Note that one of the twinning bands dominates the (001) orientation and secondary twinning is observed within the same bands with an average difference in orientation of about 1°. Figure 7 shows another grain that has a random orientation to the observed section. Both {15 5 3} and (6 4 19) orientations are found; these are orientations near (100) and (001), respectively. The {15 5 3} orientation includes both {15 5 3} and (5 15 3) orientations, and most of those orientations are dominated by one of the twinning bands. On the other hand, both bands contribute to (6 4 19) orientations, and the average orientation difference of about 1° corresponds to the contrast of the twinning bands. The two orientations are not contributed distinctly by the two twinning bands; however, the dominant fractions of the two orientations correspond to the twinning bands, i.e. the band shown in red for the (15 5 3) orientation and band shown in blue in the (6 4 19) orientation. Figure 8 shows a stereographic projection of the (6 4 19) orientation of the observed grain shown in Fig. 7. The (15 5 3) orientation relationship reflects the crystallographic twinning relation of the fct phase. In other words, rotating the (15 5 3) orientation to the center position of the (6 4 19) projection will leave at least one {101} plane shared by both crystals. The deviations of (15 5 3) from (100) and of (6 4 19) from (001) are found to be 21.33° and 20.48°, respectively. Therefore the deviation in the orientation of the twinning bands can be calculated from the two deviation angles to be 0.85°, which is similar to the value of $\theta_{(M,T)} = 0.924°$ deduced from the lattice constants.

The [011] twinning bands in Mn-Cu alloys is intrinsically characteristic of the (001)$_M$//(100)$_T$ with $\sim 1°$ deviation orientation relationship. In other words, the misorientation between (001)$_M$ and (001)$_T$ is near 90°. In the random sections for EBSD observation the twinning relationship of the bands is revealed with the $h_1k_1l_1$/$h_2k_2l_2$ orientation relationship. Even though the misorientation between $(h_1k_1l_1)_M$ and $(h_2k_2l_2)_M$ or $(h_1k_1l_1)_T$ and $(h_2k_2l_2)_T$ varies from 90 to 0° depending on the hkl values, the same {011} twinning relationship between the bands can be characterized.

The twinning bands in Mn–Cu alloys can be characterized in terms of the average orientation difference by partitioning two stereographic projections of the fct phase. For statistical calculation of the orientation difference between the twinning bands, it is feasible to use an fcc crystal structure for EBSD scanning of the fct phase. In this case, all the deviations in orientation can be reflected on one stereographic projection. Figure 9 shows the EBSD image and the twinning bands partitioned by the average orientation difference for the aged Mn–Cu alloy. The normal direction of the observed section is found to be (13 2 23) from the standard triangle inverse pole figure, as shown in Fig. 9(b). With the stereographic projection of (13 2 23), the deviation in orientation of the observed area is shown in the 5° circle of the stereographic projection (Fig. 9(c)). By partitioning the dispersive orientations, the twinning bands then become contrasted and the deviation in orientation between the twinning bands can be easily estimated.

3.2 Characterization of the Twinning Microstructure in Aged Mn–Cu Alloy

Besides the deviation in orientation between the twinning bands, which reflects the structure of twin boundaries, the width of twin bands and the intersecting configuration of difference groups of twinning bands are also important features of the twinning microstructure in Mn–Cu alloys.

Fig. 7 EBSD twinning band microstructure within a grain with a random orientation to the normal direction of the sample section, contrasted by the average orientation difference in the enlarged 100 stereographic projection (a, b), and 001 projection (c,d), respectively.
three factors are believed to affect the damping behavior of Mn–Cu damping alloys. Figure 10 is a stereographic projection showing the orientation relationship between the direction of observation and the intersecting twinning variants. Six {011} projections can be found in the stereographic projection: among these, only four poles possibly correspond to the {011} transformation twin boundaries. Different {011} twin boundaries could constitute a spatial configuration with a 60° or 90° intersection within one grain; the former has four combinations and the latter has two combinations. Two-section observation for each grain is required to confirm the spatial configuration of different groups of twinning bands; nevertheless, with the help of {011} stereographic projection, the configuration can also be determined from a one-section observation.

Figure 11 shows the twinning microstructure of the aged Mn–Cu alloy analyzed by image-quality contrast and by {011} stereographic projection. EBSD measurement for the aged alloy is conducted by fcc phase setting for the simple analysis. Based on the alignment of the twin boundary traces shown by the image-quality graphs, the four possible twin-boundary poles are shown by solid line circles in the stereographic projection. Figure 11(a) and 11(b) show twinning microstructures containing two groups of twinning bands in an aligned morphology. From the normal direction of the observed section and the projection poles of [101], it is inferred that both cases are the products of a 60° spatial intersection configuration. When the normal direction of the observed section is close to that of the trace of the two [101] twin-boundary projections, two groups of twinning bands appear in a parallel configuration. On the basis that the width of the twinning bands is the same for the four possible variants, the observed widths of the twinning bands are found to depend on the relative orientation of the observation direction. Therefore, the widths of the two groups of twinning bands are quite similar in the case of Fig. 11(a), but show a large difference in the case of Fig. 11(b). Figure 11(c) shows an intersecting configuration of two groups of twinning bands, which are found to be twins spatially intersecting at 60°. Because the observation direction of [10 5 22] deviates from the trace of (−101) and (0−11), the intersecting configuration is revealed in the observed section. As shown in Fig. 10, twins spatially intersecting at 60°, e.g. (101)−(011), will appear in a different morphology depending on the normal direction of the observed section. The parallel configuration changes to one intersecting at 60° when the normal direction shifts from [112], [001] to [−1−11]. Furthermore, the intersection angle of the 60° spatially intersecting twins at a random observation section can be calculated from the angle of the two lines of intersection resulting from (uvw)x(101) and (uvw)x(011), respectively. In the case of Fig. 11(c), the two lines of intersection of the two groups of twin boundaries with a normal direction of [10 5 22] are [5 12 5] and [17 10 10], respectively. The observed angle of intersection of the twin boundaries is confirmed to be 34.12°.

Figures 11(d), 11(e), and 11(f) show the twinning microstructure of the aged Mn–Cu alloy, which is typical of 90° spatially intersecting twins. As shown in Fig. 10, (101)−(−101), and (0−11)−(011) twin boundary combinations are responsible for the 90° spatially intersecting twins. In the case of (101)−(−101), when the normal direction of the observation section shifts from [001], [011] to [010], the parallel morphology of two groups of twinning bands changes to a perpendicularly intersecting morphology. On the other hand, when the normal direction shifts on the trace of (101), (−101) projection poles, the same band width can be observed at [001], and only one group of twinning bands is retained in the [−101] or [101] direction. In agreement with
the above analysis, Fig. 11(d) shows only one variant of twinning bands, since the normal direction is near \([-10–1]\). In the case of Fig. 11(f), nearly perpendicularly intersecting twinning bands are observed at the normal direction of \([8\ 16\ 5]\). The intersection lines of the \((10–1)\) and \((101)\) twin boundaries with the observation section are \([16\ -13\ 16]\) and \([-16\ 3\ 16]\), respectively. The intersection angle of the two lines is calculated to be 93.75°. Meanwhile, perfectly perpendicularly intersecting twinning bands are shown in Fig. 3, where the normal direction of observation is, fortuitously, exactly \([100]\).

Fig. 11(e) shows a complicated case involving the intersection of three groups of twinning bands. Whereas the \((0–11)\) and \((-10–1)\) twinning bands can be identified as 60° spatially intersecting twins, the \((-101)\) and \((-10–1)\) twinning bands constitute 90° spatially intersecting twins. The intersection lines of the two latter boundaries are \([13\ -5\ 13]\) and \([-111]\), respectively, and the intersection angle is therefore calculated to be 98.71°. However, note that the \((-101)\) twinning bands that occur within the \((-10–1)\) twinning bands, i.e. the so-called secondary twinning, are different from the spatially intersecting configuration. Such a secondary twinning phenomenon is more easily observed in binary Mn–Cu alloys, and the width of the secondary twinning bands is almost 1/10th of that of the normal twinning bands.4)
From EBSD characterization of the twinning microstructure of Mn–Cu alloys, it is found that at least two groups of twinning bands will appear in each grain, producing a spatially intersecting configuration. The intersection of different groups of twinning bands produces a junction region where the twinning bands become sharpened. The constrained twinning bands at the junction region, as shown in Fig. 11(c), may eliminate the damping capacity of the alloy by hindering the reversible movement of the twin boundaries. It appears that the 60° spatially intersecting twins intersect each other and contain a larger junction region. Control of the intersection configuration of twinning bands and of the character of the grain boundaries and a decrease in the constrained twinning band terminals should be effective in improving the damping behavior of Mn–Cu alloys. On the other hand, within each group of twinning bands, variation in the width of bands and the occurrence of secondary twinning are usually observed, probably as a result of changes in the axis ratio and lattice volume with temperature. Such changes in twinning features have also been found in the plastic strained alloy and have been attributed to the stress-induced de-twinning and reorientation processes.9)

4. Conclusions

The twinning microstructure of aged Mn–Cu alloy is the product of phase transformation from an fcc phase to an fct phase. The detection of the {101} twin boundaries in Mn–Cu alloys by the EBSD method requires an angular resolution for Kikuchi diffraction bands of better than 0.5°, because the fct phase of Mn–Cu alloy has an axis ratio (c/a) approaching 1. The present EBSD measurement conditions were inadequate for correctly distinguishing the [100] and [001] orientations that cause a mismatch in the misorientation boundaries with the [101] twin boundaries. However, image-quality contrast during EBSD measurement was found to be sensitive to the average difference in orientation between the twinning bands, which was found to be about 1° by partitioning the orientation distribution on the stereographic projection of the normal direction for the observed sample section.

On the basis of the image-quality contrast and {101} stereographic projection obtained by EBSD measurements, the twinning microstructure of Mn–Cu alloy can be characterized in terms of the spatial configuration of the twinning bands. The observed width of the twinning bands and the intersection morphology were found to depend on the normal direction of the observation sample section. Both 60° and 90° spatially intersecting twins were found in the alloy, and a relationship between the observed morphology and the spatial intersection configuration of different groups of twinning bands was established. The 60° spatially intersecting twins usually have a large junction region with sharpened ends. It was also observed that secondary twinning occurs within the twinning bands in the 90° spatially intersecting configuration. All those microstructural features are considered to affect the damping behavior of Mn–Cu alloys.

REFERENCES