

Article

Morphological and Crystallographic Characterization of Primary Zinc-Rich Crystals in a Ternary Sn-Zn-Bi Alloy under a High Magnetic Field

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Abstract: Due to the unique capacity for structural control, high magnetic fields (HMFs) have been widely applied to the solidification process of alloys. In zinc-based alloys, the primary zinc-rich crystals can be dendritic or needle-like in two dimensions. For the dendritic crystals, their growth pattern and orientation behaviors under HMFs have been investigated. However, the three-dimensional crystallographic growth pattern and the orientation behaviors of the needle-like primary zinc-rich crystals under a high magnetic field have not been studied. In this work, a ternary Sn-Zn-Bi alloy was solidified under different HMFs. The above-mentioned two aspects of the needle-like primary zinc-rich crystals were characterized using the Electron Backscattered Diffraction (EBSD) technique. The results show that the primary zinc-rich crystals are characterized by the plate-shaped faceted growth in three dimensions. They grow in the following manner: spreading rapidly in the {0001} basal plane with a gradual decrease in thickness at the edges. The application of HMFs has no effect on the growth form of the primary zinc-rich crystals, but induces their vertical alignment. Crystallographic analysis indicates that the vertically aligned primary zinc-rich crystals orient preferentially with the *c*-axis perpendicular to the direction of the magnetic field.

Keywords: high magnetic field; solidification; zinc-rich crystal; characterization; crystallography; EBSD

1. Introduction

Since the last century, great attention has been devoted to the application of a magnetic field to the solidification process of alloys due to its unique capacity for structural control [1–23]. Before the development of superconducting magnets, a conventional magnetic field (usually smaller than 2 T) was the most extensively used in research and practice. Owing to the low intensities, conventional magnetic fields mainly affect the structures of alloys through suppressing or driving the fluids by the induced Lorentz force [1–5]. With the recent development of the superconducting magnet, high magnetic fields (HMFs) (usually larger than 2 T) have become readily available and have been widely introduced in the solidification process of alloys. When compared to the conventional magnetic fields, the HMFs significantly enhance the Lorentz force and the magnetization force (usually negligible in conventional magnetic fields), thereby imposing more abundant effects on the structures of the alloys [6–23]. It has been found that the HMFs could orient and align the structures [13–17], increase the

phase transformation temperature [19], enhance the magnetic coercivities [16], suppress the diffusion of solute elements [21], modify the orientation relationship between the eutectics [22], and change the solid-liquid interface morphologies [23].

Hexagonal close-packed zinc is characterized by large solid-liquid interfacial energy anisotropy [24,25] and magnetocrystalline anisotropy [13,14]. Therefore, it is of fundamental interest to study its growth under HMFs. In other work, it has been shown that the primary zinc-rich crystals in Zn-Al [13] and Zn-Sn [14,26] alloys have a dendritic form (preferentially grow along $\langle 10\bar{1}0 \rangle$ and/or $\langle 0001 \rangle$) and can be preferentially aligned and oriented by HMFs. According to some recent work, the primary zinc-rich crystals in ternary Sn-Zn-Bi solder alloys usually exhibit a needle-like morphology in two dimensions [27]. However, their crystallographic growth pattern in three dimensions has not been addressed. The crystallographic effects of HMFs on such crystals are also not clear. Based on this context, a ternary Sn-12Zn-6Bi alloy (nominal composition: wt %) was solidified under various HMFs in this work. The three-dimensional growth form, the alignments, and the orientations of the primary zinc-rich crystals in the alloy were characterized using Electron Backscattered Diffraction (EBSD). The affecting mechanism of the HMFs on the alignments and orientations of the primary zinc-rich crystals was discussed briefly.

2. Results and Discussion

Figure 1 shows the differential scanning calorimetry (DSC) curve of the Sn-12Zn-6Bi alloy upon cooling, where two exothermic peaks are detected. This is similar to the case of the Sn-8Zn-6Bi alloy [27], according to which the reaction orders should be as follows: $L \rightarrow L + \text{primary Zn} \rightarrow L + \text{primary Zn} + \text{secondary Sn} \rightarrow \text{primary Zn} + \text{secondary Sn} + \text{eutectic (Sn + Zn)}$. Furthermore, as the solubility of Bi in Sn decreases with the drop in the temperature, Bi crystals will be precipitated from the Sn matrix. The small peak in the curve corresponds to the crystallization of the primary Zn in the melt.

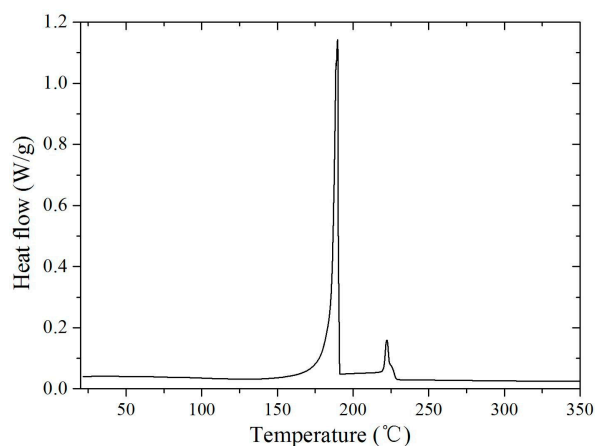


Figure 1. DSC curve of the ternary Sn-12Zn-6Bi alloy upon cooling.

Figure 2 shows the longitudinal macrostructures of the Sn-Zn-Bi specimens under various HMFs. As can be observed, the large dark primary zinc-rich crystals reveal a typical needle-like form. Obviously, the alignments of these primary crystals are heavily affected by the application of HMFs. Without and with a 0.6 T HMF, they are randomly aligned (Figure 2a,b). When increasing the HMF to 1.5 and 3 T, some crystals in the central regions of the specimens tend to align vertically, i.e., with the longer axis parallel to HMF direction *B* (Figure 2c,d). When the HMF is increased to 5 T, almost all of the crystals in the central regions align vertically. However, a further increase of the HMF to 12 T does not further enhance the alignment tendency. Moreover, it should be noted that the needle-like zinc-rich crystals in the peripheral regions are less affected by the application of the HMFs, i.e., they align randomly in all cases.

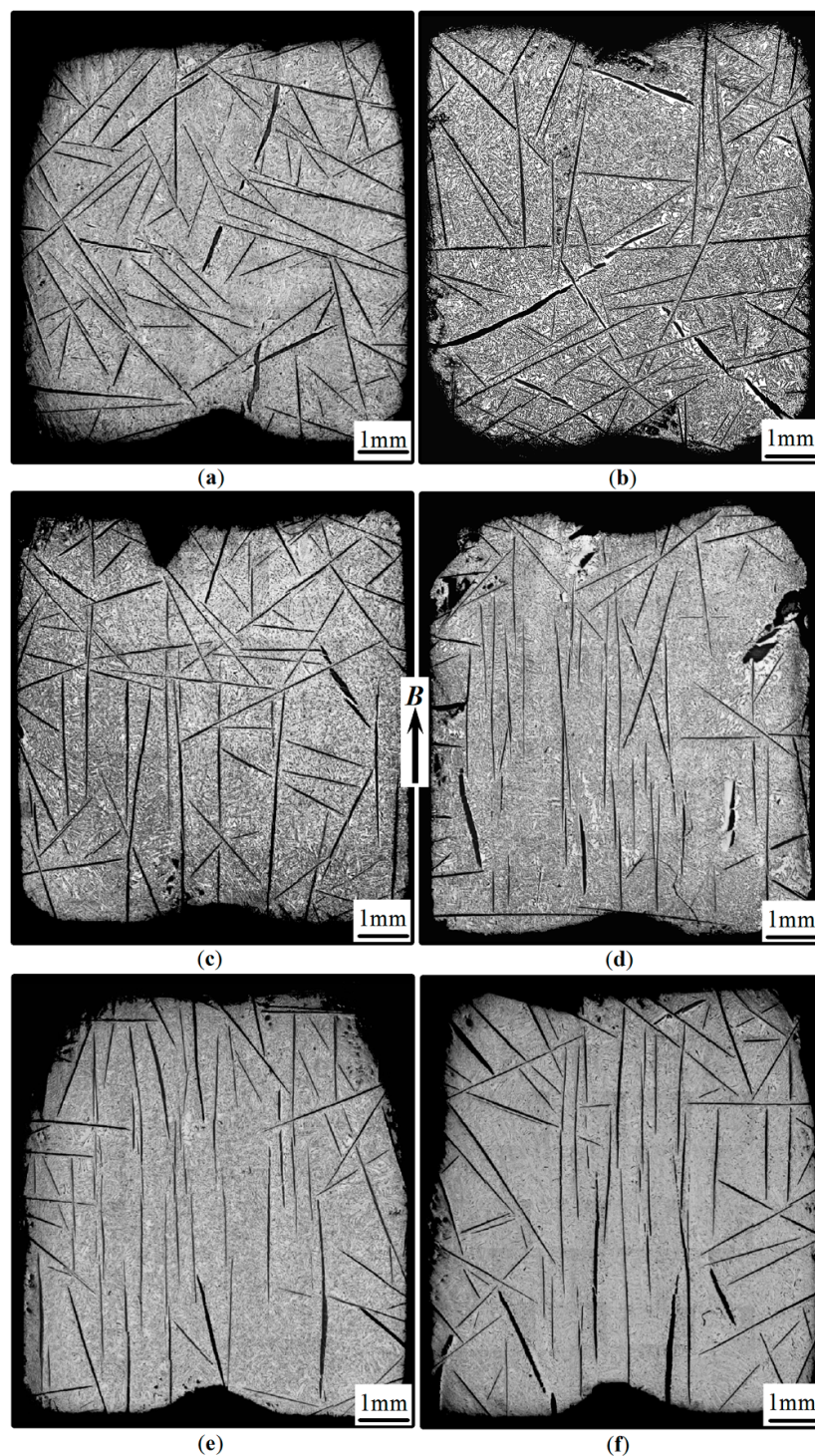


Figure 2. Longitudinal macrostructures of the Zn-Sn-Bi alloy under various magnetic fields: (a) 0 T; (b) 0.6 T; (c) 1.5 T; (d) 3 T; (e) 5 T; (f) 12 T. The arrow indicates the direction of B .

For more details, Figure 3 shows the microstructures in the central regions of the specimens under various HMFs, in which the regular alignments of the needle-like zinc-rich crystals can be more clearly observed. Other than this, it can also be seen that many fine dark-gray fibers are distributed randomly in the white-gray matrix surrounding the large primary zinc-rich crystals. Some small medium-gray globular crystals also exist and attach to each fiber to form a string-of-pearls-like morphology, as shown in the magnified insert in Figure 3a. A microstructural analysis indicates that the dark-gray fibers,

the white-gray matrix, and the medium-gray globes are the eutectic zinc-rich phase, β -Sn matrix, and Bi crystals, respectively. Here, it should be mentioned that the various contrasts and morphologies of the eutectics are related to the polishing time: as the polishing solution is corrosive, different polishing times will result in different appearances (e.g., Figure 3b,c correspond to a longer polishing time so that many globular Bi crystals are revealed). However, it is certain that the random alignments of the fiber-like eutectic zinc-rich phase are unaffected by the HMF.

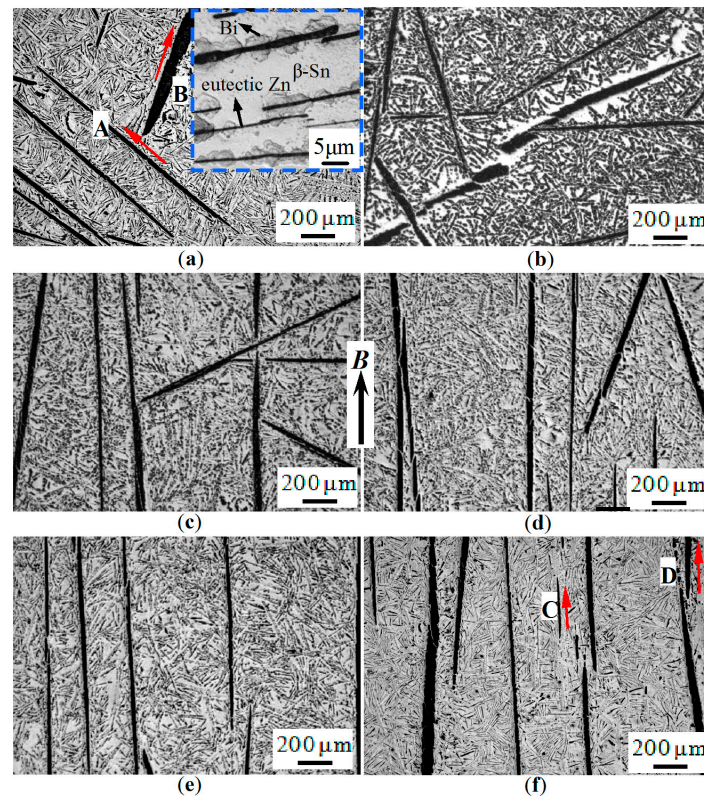


Figure 3. Microstructures corresponding to the central regions of the macrostructures in Figure 2: (a) 0, (b) 0.6, (c) 1.5, (d) 3, (e) 5, and (f) 8.8 T, respectively. The insert in (a) shows the magnified microstructure consisting of eutectic zinc-rich phase, β -Sn matrix, and Bi crystals, respectively. A, B, C, and D denote the crystals that will be further analyzed in Figure 6. The arrow indicates the direction of B .

To further understand the effects of the HMFs, Figure 4 shows the transverse macrostructures of the specimens. As can be observed, the large primary zinc-rich crystals still reveal a needle-like form, but align randomly in all cases. However, the unchanged needle-like form indicates that the large zinc-rich crystals may be plate-shaped in three dimensions. To identify this, Figure 5a,b show the microstructures of the cubes cut from the 0 and 12 T specimens, respectively. It can be seen that the primary zinc-rich crystals in surfaces 1, 2, and 3 connect perfectly at the edges of both of the cubes, confirming a plate shape in three dimensions, irrespective of whether the HMF is applied. Nevertheless, the needle-shaped form (with sharp ends) in two dimensions suggests that the edges of these plates should have a tapered transition in thickness. Furthermore, the primary zinc-rich plates exhibit some faceted growth character, as can be seen from the relatively smooth interface traces. A crystallographic interface calculation indicates that the large surfaces of the plates correspond to the $\{0001\}$ basal plane. This result can also be indirectly proven by the $\langle 0001 \rangle$ pole figures in Figure 6a,b, corresponding to the crystals A & B in Figure 3a and C & D in Figure 3f, respectively. The projection lines OA, OB, OC, and OD are approximately perpendicular to the interface traces of the crystals A, B, C, and D in Figure 3 (see the arrows denoting the extension directions of the interfaces), respectively.

Furthermore, it should be mentioned that crystal B in Figure 3a is coarser than the others. This is because that it approximately exposes the {0001} basal plane in the longitudinal section, as evidenced by pole A near the center point O in Figure 6a. From the analysis above, it can be concluded that the plate-shaped primary zinc-rich crystals should grow in three dimensions, as follows: spreading rapidly in the {0001} basal plane and then decreasing gradually in thickness at the edges. Figure 7 schematically shows the three-dimensional morphology of a primary zinc-rich crystal, in conjunction with the hexagonal unit cells, denoting its orientations relative to the observation plane. It should be mentioned here that the growth manner of the primary zinc-rich crystals in the present alloy is quite different from that of the crystals in Zn-Sn and Zn-Al alloys, which, as mentioned previously, grow along the $\langle 10\bar{1}0 \rangle$ and/or $\langle 0001 \rangle$ directions to form complex dendritic morphologies [13,14,26]. Such a growth manner may be related to the types and amounts of alloying elements, which affect the growth kinetics of zinc-rich crystals. This is open to future research.

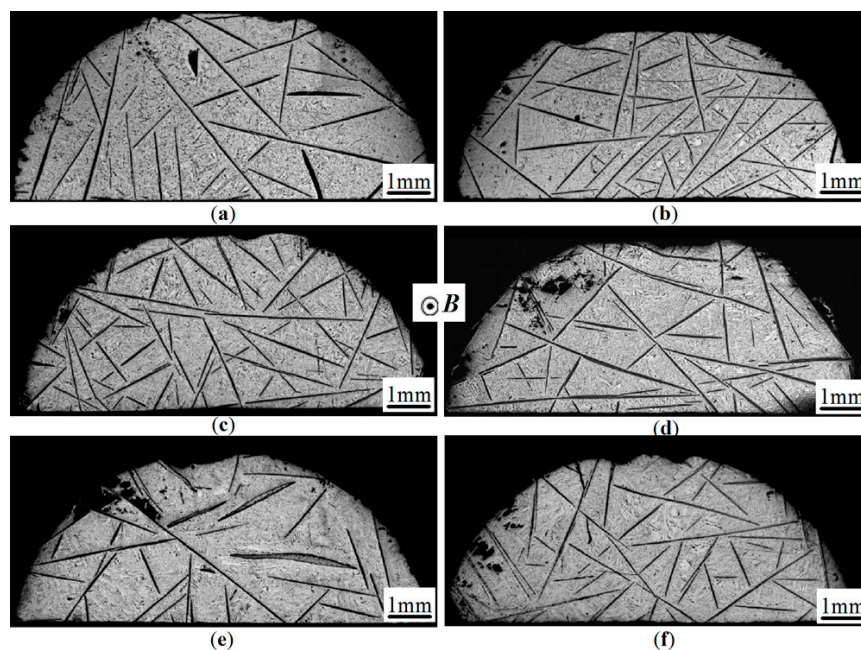


Figure 4. Transverse macrostructures of the specimens under various HMFs: (a) 0 T; (b) 0.6 T; (c) 1.5 T; (d) 3 T; (e) 5 T; (f) 12 T.

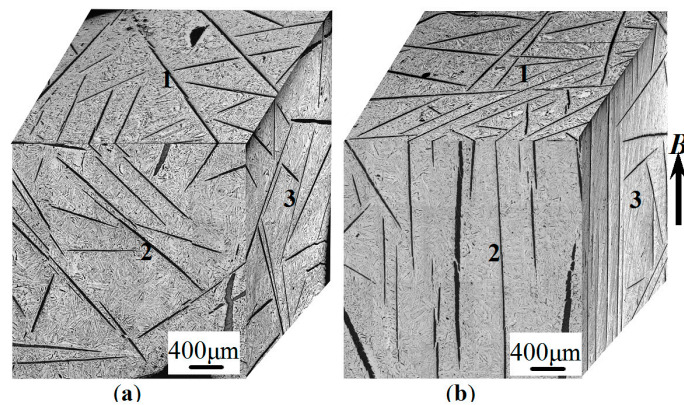


Figure 5. Microstructures of the cubes cut from the 0 and 12 T specimens. 1, 2, and 3 indicate the different surfaces of the cubes. The arrow indicates the direction of B .

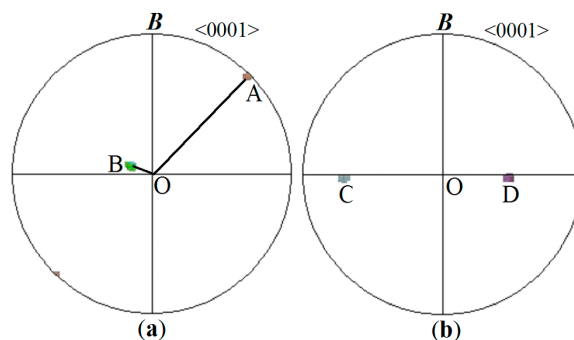


Figure 6. $\langle 0001 \rangle$ pole figures corresponding to the primary zinc-rich crystals (a) A & B in Figure 3a and (b) C & D in Figure 3f, respectively.

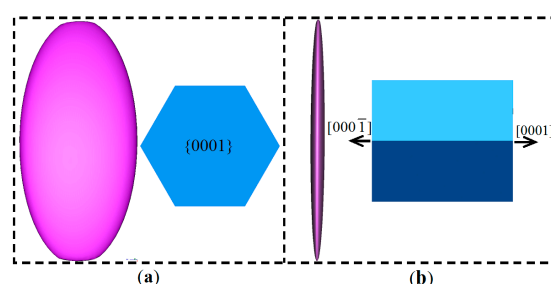


Figure 7. Schematic morphology of a primary zinc-rich crystal in three dimensions: (a) front and (b) side views. The hexagonal unit cells denote its orientations relative to the observation plane.

It has been optically observed that the primary zinc-rich crystals in the Sn-Zn-Bi alloy are highly aligned owing to the application of the HMFs. To discover more structural transformation information, further crystallographic analysis was conducted in this work. Figure 8a,b show the phase maps (zinc-rich crystals—red; β -Sn matrix—blue) and the all-Euler orientation micrographs corresponding to the longitudinal structures without and with a 12 T HMF, respectively. It can be seen that the large primary zinc-rich crystals are distributed in the fine β -Sn grains. Irrespective of whether the HMF is applied, the primary zinc-rich crystals show different colors, indicating that they have different orientations. However, the crystallographic calculation suggests that the HMF tends to orient its c -axes perpendicular to B . Figure 9a,b show the scattered $\langle 0001 \rangle$ pole figures corresponding to the primary zinc-rich crystals in Figure 8a,b, respectively. The $\langle 0001 \rangle$ poles are randomly distributed in the absence of the HMF, whereas they reveal a linear distribution in the presence of the HMF. This further proves that the primary zinc-rich crystals tend to orient preferentially with the $\langle 0001 \rangle$ direction, i.e., the c -axis, perpendicular to B . This orientation feature is consistent with that of the dendritic primary zinc-rich crystals under a HMF [13]. A detailed crystallographic analysis was also conducted on the β -Sn matrix, implying no preferential orientation, even with a 12 T HMF.

As analysed in other work [13], the preferential orientation of the hexagonal primary zinc-rich crystals should be related to their magnetocrystalline anisotropy. According to the magnetization energy theory, the easy magnetization axis should be parallel to the magnetic field for paramagnetic materials and perpendicular to the magnetic field for diamagnetic materials [18,28]. Zinc is a diamagnetic material with a smaller magnetic susceptibility along the c -axis ($-0.169 \times 10^{-6} \text{ cm}^3/\text{g}$) than that in the direction perpendicular to the c -axis ($0.124 \times 10^{-6} \text{ cm}^3/\text{g}$) [29]. To minimize the magnetization energy, the c -axis of the primary zinc-rich crystals tends to be rotated to the direction perpendicular to B under the HMFs. The driving force to start the rotation is the magnetic torque, as analysed in detail elsewhere [17,18,30]. However, to complete this rotation, a weak constraint medium is indispensable for the crystals [28]. For the present Sn-Zn-Bi alloy in this work, the zinc-rich crystals are primarily crystallized from the melt during the solidification process. The surrounding liquid

provides a free rotation environment for them. However, for the crystals in the peripheral regions of the specimens, their rotations may be hindered by the crucible walls. It is known that the melt solidifies inward from the crucible wall in a non-directional solidification way. This means that the crucible walls may act as the effective nucleation sites for the primary zinc-rich crystals, as a result of which their rotations are prevented by the crucible walls. After nucleation, these crystals grow towards the central regions of the specimens, and finally reveal random alignments in all cases.

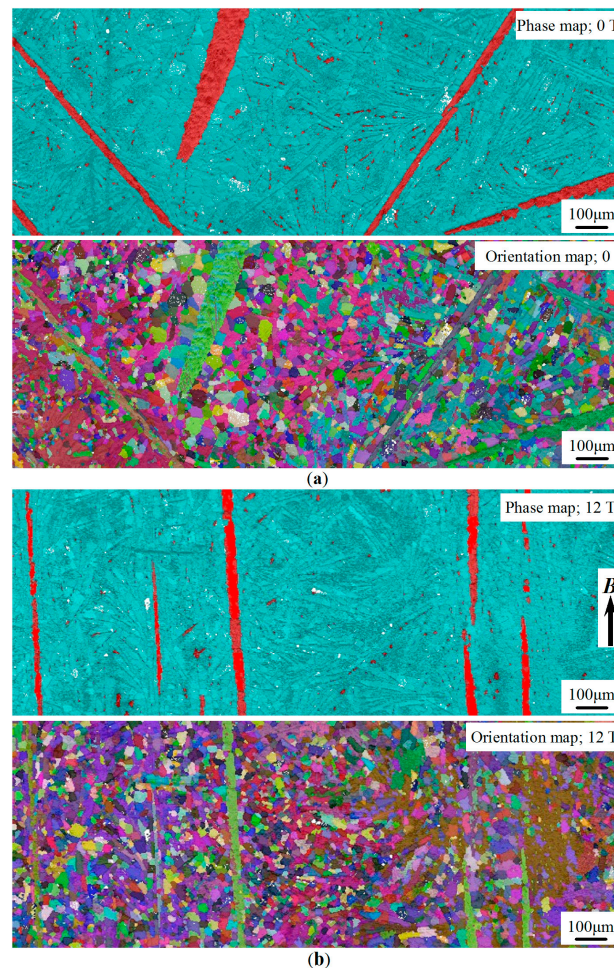


Figure 8. Phase maps and all-Euler orientation micrographs corresponding to the longitudinal structures (a) without and (b) with a 12 T HMF, respectively. The arrow indicates the direction of B .

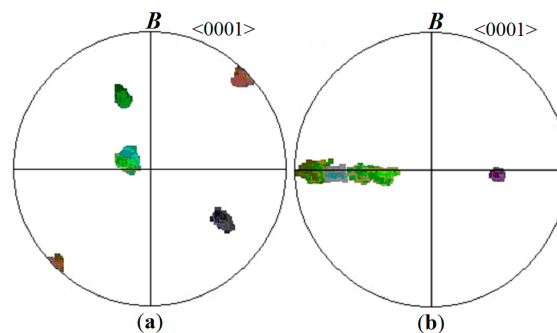


Figure 9. Scattered $\langle 0001 \rangle$ pole figures corresponding to the primary zinc-rich crystals in (a) Figure 8a and (b) in Figure 8b, respectively.

Now it can be understood that in the absence of the HMFs, all the plate-shaped primary zinc-rich crystals align randomly, as schematically shown in Figure 10a. When the HMFs are applied, the c -axes of the crystals in the central regions of specimens are fixed in the plane perpendicular to B . As the {0001} basal plane corresponds to the large surfaces of the plate-shaped crystals, they finally exhibit a vertical alignment, as schematically shown in Figure 10b.

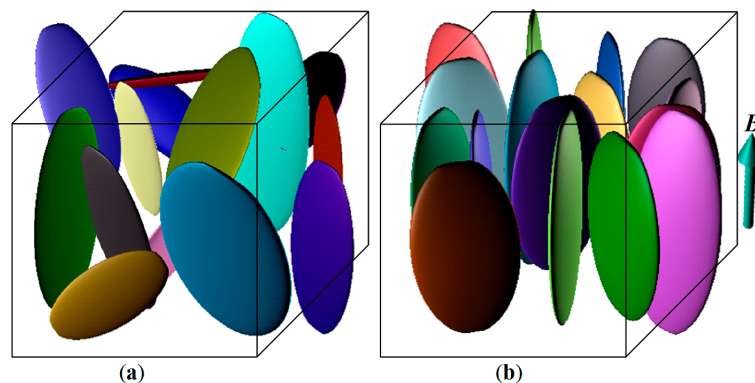


Figure 10. Schematic diagrams showing the alignments of the plate-shaped primary zinc-rich crystals (a) without and (b) with a HMF, respectively. The arrow in (b) indicates the direction of B .

3. Materials and Methods

A ternary Sn-12 wt % Zn-6 wt % Bi (nominal composition) alloy was prepared by melting pure zinc (99.9 wt %), tin (99.95 wt %), and bismuth (99.9 wt %) in an induction furnace. The alloy was reheated to 673 K (400 °C) at a rate of 5 K/min in an electric resistance furnace under an argon atmosphere and held at this temperature for 25 min to ensure a homogeneous composition. Afterwards, the melt was cooled to room temperature at a rate of about 10 K/min. From the start of the reheating until the end of the cooling, different uniform-distributed HMFs were applied, as introduced in detail elsewhere [13].

The HMF-treated as-cast ingots (Φ 10 × 12 mm) were cut longitudinally or transversely (parallel or perpendicular to the HMF direction B), following a standard mechanical polishing. The macro/microstructures were observed with a Leica DMR optical microscope (Leica Microsystems, Wetzlar, Germany). Moreover, two small cubes were also cut from the 0 and 12 T specimens for three-dimensional microstructure observations. After further polishing plus Ar ion beam cleaning, EBSD auto scanning was performed on the specimens for crystallographic analysis on a Zeiss ULTRA PLUS FE-SEM (Carl Zeiss, Jena, Germany), equipped with an Oxford-HKL Channel 5 system (Oxford Instruments, Abingdon, Oxfordshire, UK). Differential scanning calorimetry (DSC) was carried out on a Q100 TA Instrument (TA Instruments, Newcastle, DE, USA) to understand the cooling process of the alloy: a 20 mg specimen was cooled from 623 K (350 °C) to 293 K (20 °C) at a constant rate of 5 K/min in Ar flow.

The large faceted interface of the primary zinc-rich crystals exposed to the Sn matrix was calculated by an indirect two-trace method [31,32]. The preferential direction of the primary zinc-rich crystals relative to the HMF direction is determined as follows: first, the B vector in the sample coordinate system is transformed to its equivalent vector in the crystal coordinate system with the measured orientation data of the crystals; then, a plane is defined by the cross product of these two differently oriented vectors; finally, the statistically representative crystalline direction close to the normal vector of the plane is determined as the preferential direction [13].

4. Conclusions

A ternary Sn-12Zn-6Bi alloy was solidified under different HMFs, and it was found that:

- a. Irrespective of whether the HMF is applied, the primary zinc-rich crystals reveal a plate-shaped form and grow in three dimensions, as follows: spreading rapidly in the {0001} basal plane and then decreasing the thickness at the edges gradually;
- b. In the absence of the HMF, the plate-shaped primary zinc-rich crystals align randomly, whereas in the presence of the HMF, those in the central regions of the specimens tend to align vertically;
- c. Crystallographic analyses indicate that the vertically aligned primary zinc-rich crystals orient preferentially with the *c*-axis perpendicular to *B*;
- d. The preferential orientation and thus the regular vertical alignment should be related to the magnetocrystalline anisotropy of the diamagnetic primary zinc-rich crystals.

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Conflicts of Interest: The authors declare no conflict of interest.

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