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The influence of melt flow on grain structure of tin and its alloys produced by ultrafast quenching from the melt

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Abstract. The results on the grain structure and texture of tin and tin-based foils produced by ultrafast quenching from the melt are presented in this work. The formation of fine equiaxed and large grains elongated along the spreading direction of the foil was observed. Detailed studies of subgrain structure showed that the crystallographic orientation of elongated grains changes monotonically within the grain. It can be assumed that the reason for the change of crystallographic orientation is the deformation of solidified material. The deformation is caused by the viscous force of undercooled melt which moves above the crystallization front in the direction of the foil spreading.

1. Introduction

The classical methods to improve mechanical properties of material have already been well studied. Currently, intense research work on the materials production in non-equilibrium solidification conditions is conducted. Strongly nonequilibrium crystallization conditions are implemented in such techniques as: melt spinning technique, ultrafast quenching from the melt, laser, ion-plasma, compression-plasma surface treatment. These techniques allow reaching the purposeful influence on the functional material properties. In these techniques, the cooling rate of the melt reaches 10^4–10^7 K/s [1–2]. Solidification begins at deep undercooling and proceeds with a high liquid – solid interface velocity in the range 1–10 m/s [3–4]. In this case, the melt which is above the solidification front is not stationary but moves in the tangential direction relative to the interface. Thus, during ultra-fast quenching from the melt, velocity is comparable to the linear crystallizer velocity and is a few meters per second. It is known that the viscosity of the melt decreases when its temperature increases, rising by fifteen orders from the melting point to the glass transition temperature [5]. The directional motion of the viscous supercooled melt causes the shear stress on the surface of the solidified layer, leads to the material deformation and the microstrain appearance in the grain volume, i.e. strain hardening. In the present study grain structure of foils of tin and its alloys with Bi, Pb, Zn and In obtained by ultrafast quenching from the melt is investigated.

2. Experimental

Foils were prepared by splashing of melt drops of mass 0.2–0.3 g on a polished inner surface of a rotating hollow copper cylinder (mold). The diameter of the mold was 25 cm and the linear velocity of rotation was 15 m/s. Melt temperature was controlled by a thermocouple placed in a furnace in the

vicinity of the melt, and was set in the range 550–570 K. To homogenize the melt, it was kept in the furnace for 60 seconds. Foils were prepared from tin, with purity better than 99.99%.

Studies of the grain structure and texture of the foils were performed by electron backscatter diffraction technique (EBSD), which was implemented using the phase analysis attachment «HKL CHANNEL 5» (Oxford Instruments, UK) to the scanning electron microscope LEO 1455 VP (Karl Zeiss, Germany). Grain structure was analyzed along the foil by scanning of the side adjacent to the mold, the inner layer, freely solidified side, as well as longitudinal and cross sections of the foil. Slices were prepared by grinding and polishing machine TegraPol by Struers technique.

3. Results

The foils obtained by ultrafast quenching from the melt were characterized by width ranging from 5 to 10 mm, thickness ranging from 40 to 50 µm and by the length up to 10 cm. Further, it will be assumed that the direction of the foils spreading is OX, direction perpendicular to it in plane parallel to the foil surface is OY and the direction perpendicular to the foil surface is OZ (figure 1).
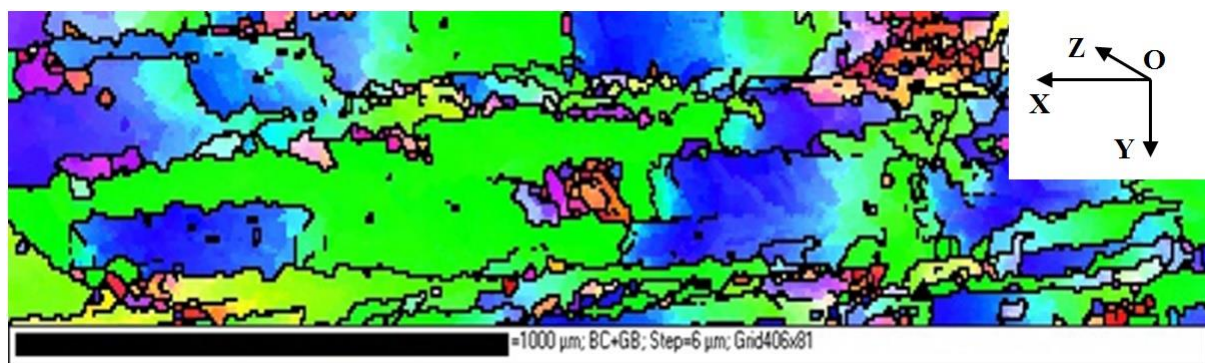

Figure 1. A typical view of the grain structure of the middle foil part.

As it was shown in [6], two types of grains are formed in the major part of the foil: large elongated along the spreading direction grains, and fine equiaxed grains located at the boundaries of large ones. The size of small grains is in the range of 10 - 80 microns, and their average diameter is 35 µm. The volume fraction of small equiaxed grains is 20 - 25%. Large elongated grains make up about 80% of the foil's volume. Both large and small grains have a columnar structure. The orientation of the elongated grains have similar character on the side adjacent to the mold, in the middle of the foil and at freely solidified side because of the columnar structure [7]. Thus, the foils have a bimodal grain structure consisting of small grains and large elongated grains.

It was revealed by EBSD technique that grains oriented so that the deviation of axis C_4 of β-Sn from foil spreading direction (OX) is less than 20 degrees occupy up to 80% of the total area. The grains of such orientation are shown in the figure 2a. Figure 2d shows the unit cell of β-Sn with it's C_4 axis of symmetry and figure 2b shows scheme of grains colouring according to the crystal orientation relative to the surface of the foil. According to the crystal orientation colouring such grains are colored in green or blue colors.

a)

b)   c)

d)   e)
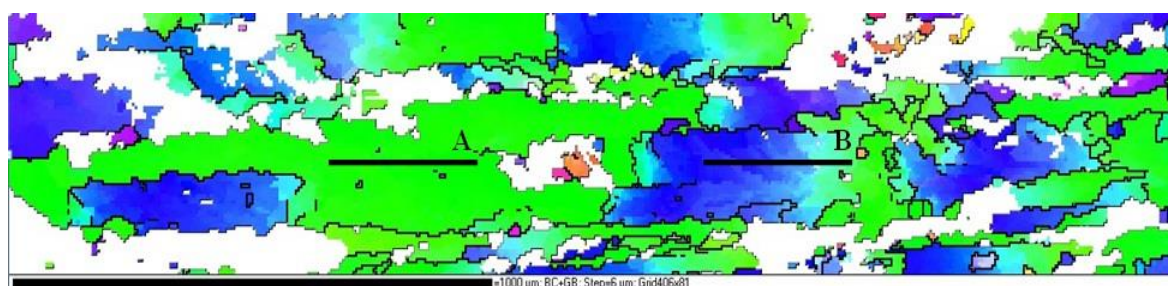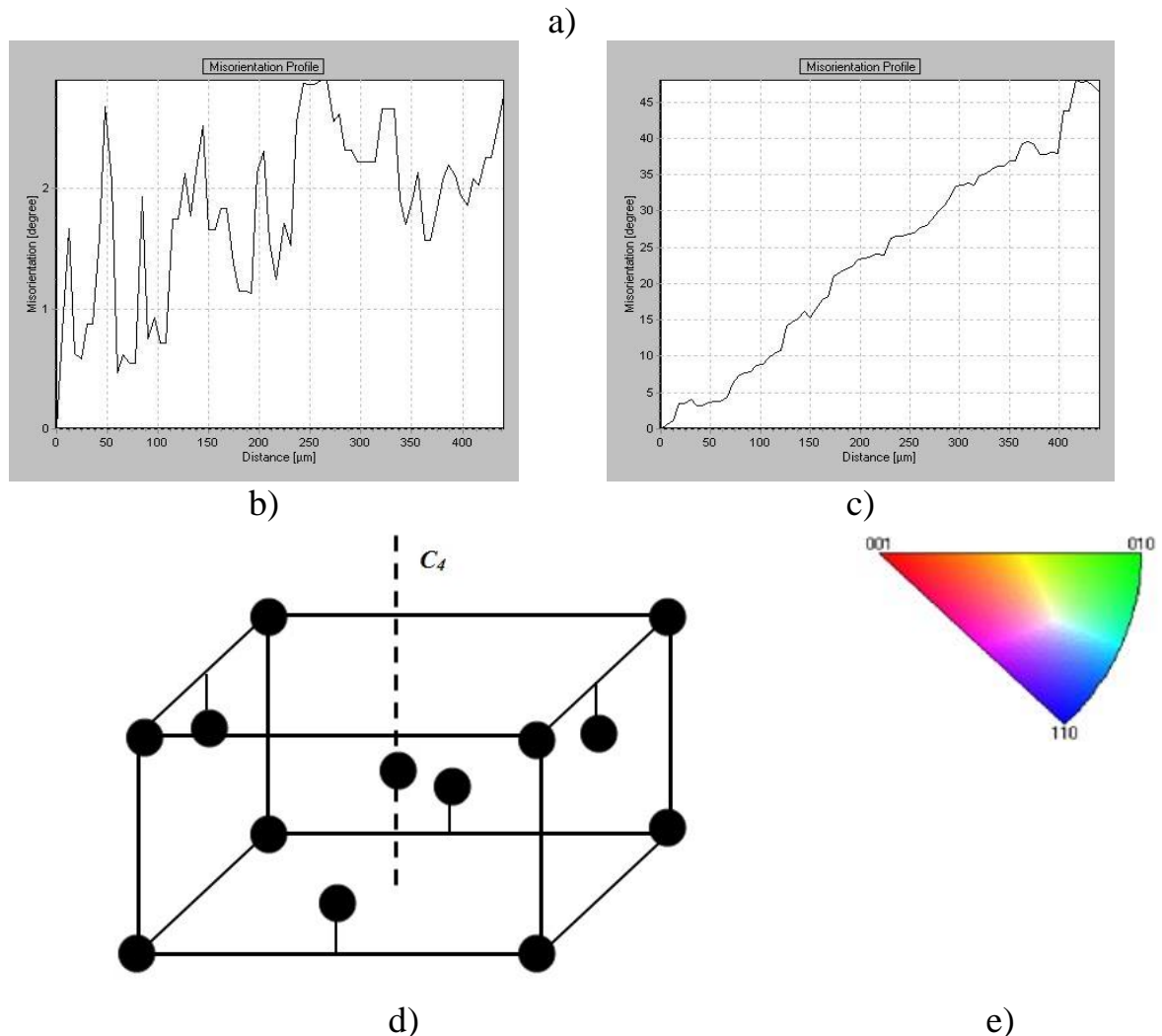
Figure 2. The elongated grains (a), changes of the angle between the identical crystallographic planes along the line at A-type grain (b) and B-type grain (c), unit cell of β-Sn (d), colouring scheme (e).

At figure 2a high-angle grain boundaries (the angle between the grains orientation of more than 10 degrees) are outlined by black lines. As it seen from figure 2a elongated grains are of two types of colouring: uniform (grain of A-type) and non-uniform (grain of B-type). Non-uniform coloration of B-type grains means that the crystal orientation is changed within the grain.

Figures 2 d and e shows changes of the angle between the identical crystallographic planes along the line which is parallel the spreading direction of the foil. Scanning grains of A- type shows that the adjacent parts of the grain are randomly disoriented by angle which is not higher than error bar of the EBSD-measurements. That means that there are now high-angle boundaries in such grains. A different kind of behavior of the angle between the same crystallographic planes along the line is observed for grains of B-type (figure 2c). In this case, the angle between the same crystallographic planes changes monotonically within a grain. In the given example the final angle change is 45 degrees (figure 2c).

It was found that the subgrain structure of large elongated grains depends on the crystallographic orientation of nucleation center. Uniformly coloured grains (A- type) grow in the case if (100) plane parallel to the foil surface or deviated by a small angle and the C_4 crystallographic axis is parallel to the spreading direction. B-type grains grow if the nucleation center is oriented so that the (100) plane parallel to the foil surface or deviated by a small angle but the C_4 crystallographic axis is not parallel

to the spreading direction. In this case, the lattice orientation monotonically changes during the growth process. A sequence of low-angle boundaries (outlined by white lines) is revealed in the grains of type B by the EBSD technique. The lattice orientation changes at the points 1, 2, 3, 4 and 5 of the B-type grain showed at the figure 3. It was found that the unit cell orientation changes in a way that it is "rotated" around the C_4 axis. The unit cell "rotates" from the initial position until (110) plane becomes parallel to the foil surface.
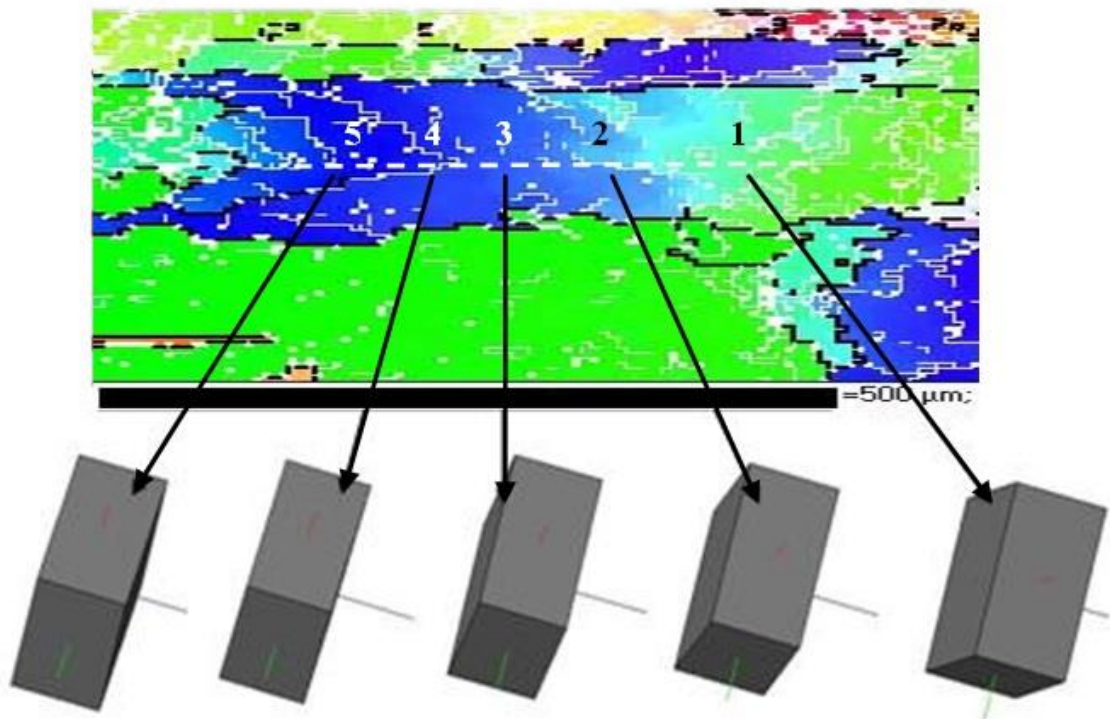
Figure 3. Changes of the lattice orientation in the grain of B-type.

Studies of foils grain structure of binary alloys of tin with lead and bismuth have shown that the addition of the alloying element with amount not higher than its solubility at ultrarapid quenching from the melt (up to 8 wt. % Bi, and 5 wt. % Pb) has no significant effect on the characteristics of the grain structure. Figure 4a and 4b shows the grain structure of foils of alloys Sn - 4 wt. % Bi, Sn- 2 wt. % Pb. It was also found that the elongated grains observed in the foils of ternary eutectic Sn - 8 wt. % Zn -3 wt. % Bi alloyed additionally by 5 wt.% In (figure 4c).

a)   b)   c)
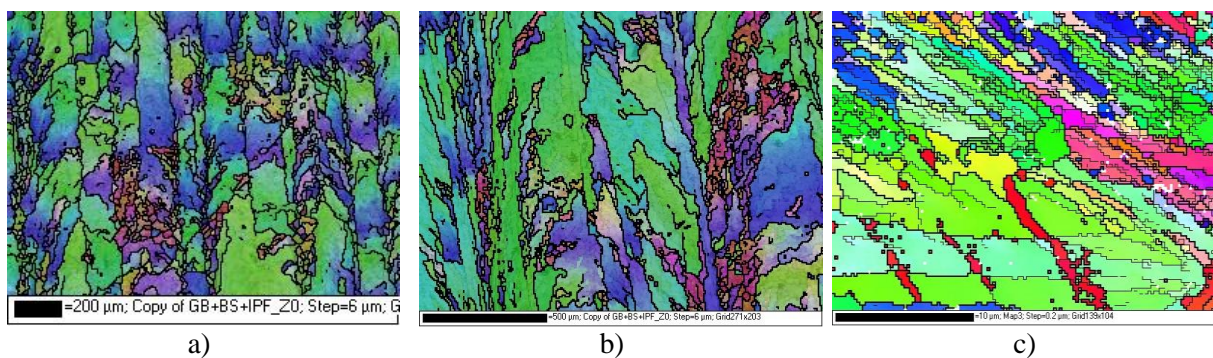
Figure 4. Grain structure of foils of alloys Sn - 4 wt. % Bi (a), Sn- 2 wt. % Pb (b) and
        Sn - 8 wt. % Zn -3 wt. % Bi -5 wt.% In.

4. Discussion

The above-described elongated grains shape is formed because the grain grows both in the direction of the heat extraction (perpendicular to the mold) and in the direction of melt flow. This is possible in the case of relatively slow rotation of the mold and the high velocity of the liquid - solid interface motion [7]. According to our previous studies, an increase of the mold rotating velocity leads to the equiaxed shape of grains [6 - 7].

It was found that during ultra-fast quenching of the Sn from the melt the nucleation of the grain oriented by the (100) plane parallel to the surface of the mold is the most likely one. Since the (100) plane of β-Sn has the highest reticular density, the formation of nuclei in which this plane is parallel to the substrate provides the lowest surface energy.

The observed "rotation" of grain orientation is caused by the features of crystallographic structure of the tin and its mechanical properties. The distinctive feature of the tin is large number of sliding systems and high plasticity. According to the Ref. [8], the following sliding systems are realized in tin at room temperature (110) [$\bar{1}11$], (110) [001], (100) [010], (100) [001], (101) [$\bar{1}01$] и (121) [$\bar{1}01$]. The sliding systems (110) [$\bar{1}11$] and (110) [001] are the most probable.

It can be assumed that the reason for the change of crystallographic orientation of the B-type grains is the deformation of just crystallized material by the viscosity force of the supercooled melt which moves above the crystallization front in the direction of foil spreading.

According to the Schmid-Boas equation a shear stress at sliding plane in the direction of the sliding can be calculated by following equation:

$$\tau = \sigma \, sin(\chi) \, cos(\lambda), \tag{1}$$

where σ is applied stress, χ is angle between applied stress direction (OX direction of foil spreading) and sliding plane, λ is angle between stress direction and sliding axis.

If the grain is positioned so that the projection of the shear stress on the plane (110) has a component along the [111] and [001] directions, the crystal «rotates» until the sliding direction is parallel the axis of stretching. Shear strain persists as long as the stress τ is zero, that is, until the (110) plane is not parallel to the direction of flow of the melt.

The fact that the above-described binary and multicomponent alloys based on tin have a similar grain structure with the grain structure of pure tin is caused by the formation of a supersaturated solid tin-based solution at the crystallization

5. Conclusions

It was found that the grains of two types are formed at the main foil body: large grains elongated in the direction of foil spreading and fine equiaxed grains localized on the border of the large ones. Subgrain structure of large elongated grains depends on the crystallographic orientation of the nucleus. If the nucleation center is oriented so that the (100) plane parallel to the foil surface or deviated by a small angle and the C_4 axis deviated from to the spreading direction by any angle, the lattice orientation monotonically changes during the growth process. It can be assumed that the reason for the change of the crystallographic orientation of type B grains is the deformation of solidified material by the viscosity force of undercooled melt, which moves above the crystallization front in the direction of the foil spreading. The easy slip systems of tin are (110) [111] and (110) [001]. Therefore, if a grain is oriented so that the projection of the shear stress on the plane (110) has a component along the [111] and [001] directions, the crystal rotates until the sliding direction becomes parallel to the foil spreading direction. Shear deformation persist until the shear stress is not zero, that is, until the (110) plane is parallel to the melt flow direction.

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