The effect of rotating magnetic field on the solidified structure of Sn–Cd peritectic alloys

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Abstract. The peritectic alloys, such as some types of steel, Ni-Al, Fe-Ni, Ti-Al, Cu-Sn, are commercially important. In contrast to other types of alloys, many unique structures (e.g. banded or island ones) can form when peritectic alloys are directionally solidified under various solidification conditions. It can be observed in the course of the directional solidification experiments performed in a rotating magnetic field (RMF) that the melt flow has a significant effect on the solidified structure of Sn-Cd alloys. This effect was investigated experimentally for the case of Sn–1.6 wt% Cd peritectic alloy. For this purpose, a Bridgman-type gradient furnace was equipped with an inductor, which generates a rotating magnetic field in order to induce a flow in the melt. As a result, the forced melt flow substantially changes the solidified cellular microstructure. The cell size and the volume fraction of the primary tin phase were measured by an image analyzer on the longitudinal polished sections along the entire length of the samples. The microstructure was investigated by scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS).

Introduction

During solidification of alloys uncontrolled flow can develop under earth conditions due to density difference in the liquid phase caused by temperature and concentration differences. The intensity of melt flow affects the solidified microstructure and therefore the physical and mechanical properties of the alloys. The melt flow can be controlled by rotating magnetic field (RMF). During controlled solidification, the melt flow induced by RMF leads to macrosegregation in radial and axial directions, which results in a change of phase ratio and distribution [1-8]. If macrosegregation is substantial, new phases (which are not found in the original alloy) also appear in the microstructure. The majority of these investigations were performed in Al [3] or Ni-based [4] solid solutions and eutectic alloys [8, 10, 11]; only a few reports are devoted to the effect of melt flow induced by RMF on the microstructure of peritectic alloys [12, 13]. The peritectic alloys are commercially important; these are, for example, some types of steel, Ni-Al, Fe-Ni, Ti-Al, Cu-Sn alloys.

The aims of this paper are to understand the effect of the rotating magnetic field on the Sn-Cd alloy solidification and acquiring new knowledge about the stirring effect of magnetic field.

Experimental procedures

The binary Sn-Cd 1.6 wt % alloy used for solidification experiments was prepared by induction melting, from pure Sn (99.99 wt%) and Cd (99.95 wt%), of which 8 mm diameter rod was drawn in several steps. The solidified probes were 8 mm in diameter and 100 mm long. The unidirectional solidification experiments were carried out on the Crystallizer with High Rotating Magnetic Field

(CHRMF) equipment developed by the MTA-ME Materials Science Research Group [10, 11, 12]. The molten alloy was continuously stirred by a constant rotating magnetic field until solidification was complete. The first 40 mm of the specimen solidified without magnetic stirring, and then the magnetic field was switched on, which stirred all the remaining melt during solidification. The following experimental parameters were applied: magnetic induction 3, 10, 20 and 60 mT, frequency 50 Hz, magnetic Taylor number Ta = $1.45x10^3$, $1.6x10^4$, $6.4x10^4$, $5.8x10^5$, temperature gradient G = 6 K/mm, specimen moving velocity v = 10^{-2} mm ^{2}/ s (G/v = $3 \cdot 10^2$ Ks/mm^2). A longitudinal section of the solidified specimen was made, then grinded, polished and etched in a 4% Nital.

The specimens were studied by Light and Scanning Electron Microscopy, and X-ray diffraction. The Cd concentration was measured by a BRUKER AXS type EDS coupled with a Hitachi S-4800 scanning electron microscope without standard.

Results and discussion

Microstructure

Fig.1 shows longitudinal sections of the Sn-1.6 wt% Cd alloy solidified under different magnetic induction. It can be seen that in the first (unstirred) 40 mm part of the specimen columnar (cellular) structure is formed, similarly to results found in literature [16].

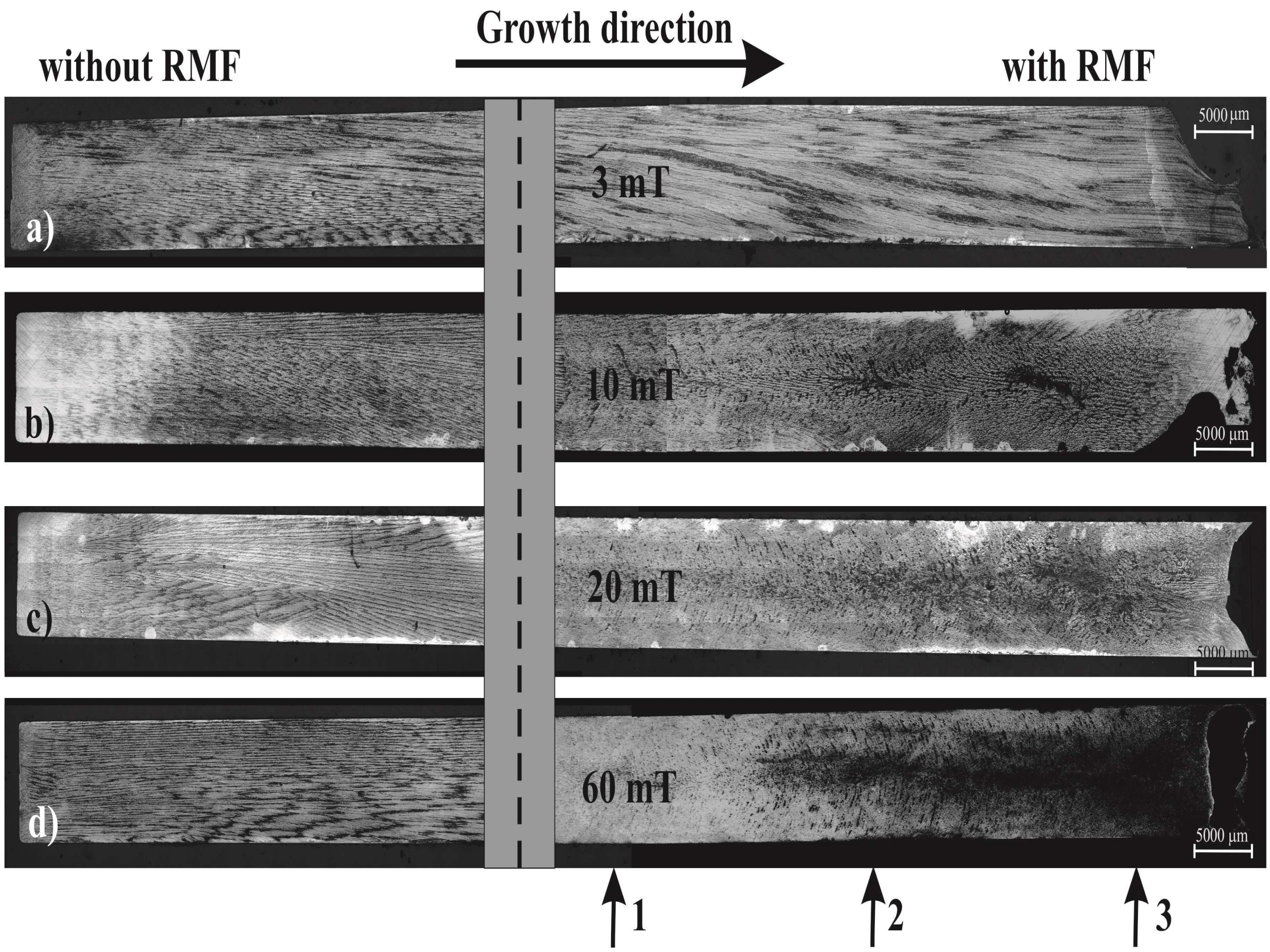

Fig. 1. Longitudinal section of Sn-1.6 wt% Cd alloy solidified at G = 6 K/mm, v = 0.02 mm/s, a) 3 mT; b) 10 mT; c) 20 mT; d) 60 mT

Primarily, α phase (Sn-based primary solid solution) cells (light areas in the figures) began to solidify from the melt (L), and then – in between the cells – directly from the melt with significant supercooling (T_β^s temperature) β ($CdSn_4$ based secondary solid solution, darker areas in the figures) compound solidified (see Fig.2. and 3). The solidification of β phase will be completed at T_β^f. In both phases, a significant difference of concentration formed between the center and the edge of the phases because the diffusion in the solid phase during solidification is slow. The peritectic process practically was failed for the same reason. In the course of cooling the $CdSn_4$ compound first transformed to α phase (in equilibrium the 1.6 wt% Cd alloy at T_e eutectoid temperature contains 66% Sn and 34% $CdSn_4$ compound) and then the remained compound transformed to α+γ phases in an eutectoid process. The γ phase is a Cd based solid solution (Fig. 2) [16].

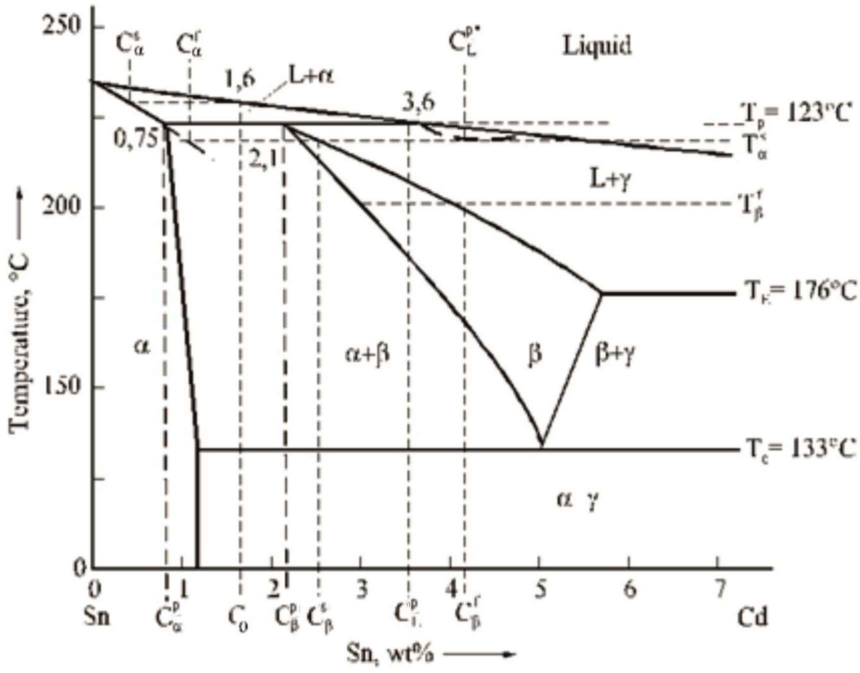

Fig. 2. Sn-Cd equilibrium phase diagram [16]

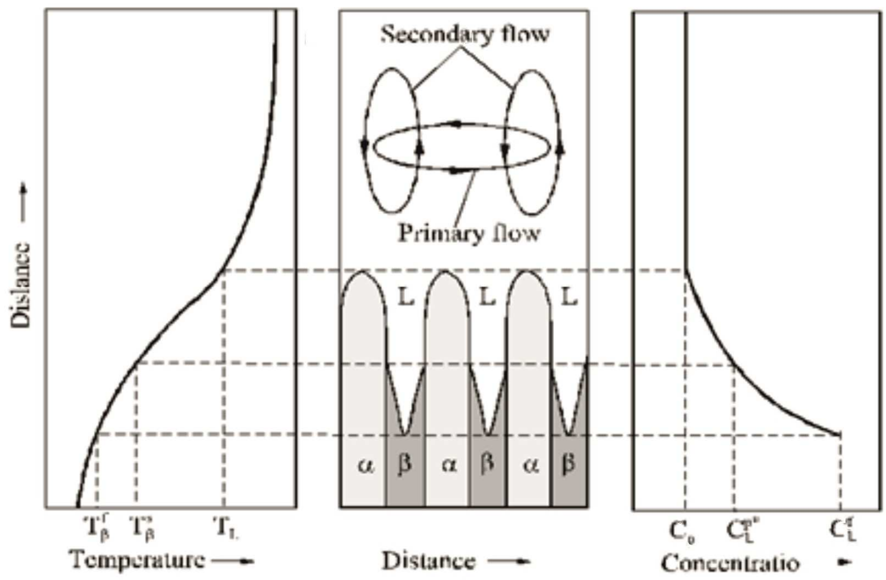

Fig. 3 shows the sketch of developing of microstructure, the temperature and the concentration distribution in the melt during solidification. The concentration of the melt at the base of the cells (at the end of solidification) is much higher (C_L^f) than before the cells, where – if assumed there is no melt flow – the concentration is the same as that of the alloy C_0.

Fig. 3. Temperature distribution, microstructure, concentration distribution in the melt during solidification

Fig. 4a, 4b and 4c show different magnification of the columnar oriented microstructure. Fig. 4c shows in higher magnification that the black particles are Cd phase ones. Cadmium also precipitated in the primary solidified, supersaturated Sn cells along the low-angle grain boundaries, revealing the latter.

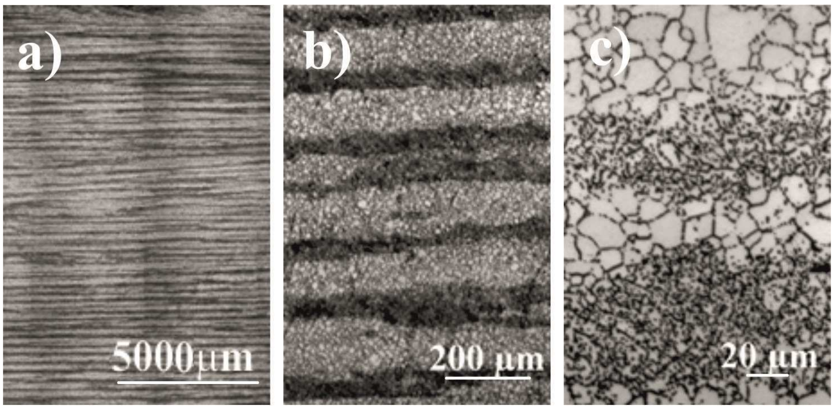

Fig. 4. Columnar cell structure in the non-stirred part of the specimen in different magnification (Light microscope)

Fig. 5.a illustrates the concentrations distribution measured in the direction perpendicular to the cells. The Cd concentration in the Sn cells (light areas) is nearly the nominal value, while between the cells it is 5-7 wt%. The latter value also indicates that the peritectic process did not occur; the solidification process was finished with the formation of the $CdSn_4$ phase. In Fig 5.b the idealized concentration distribution is shown which is very similar to the measured one. (Note that the measurement was carried out without standard, so the measured values are for reference only.)

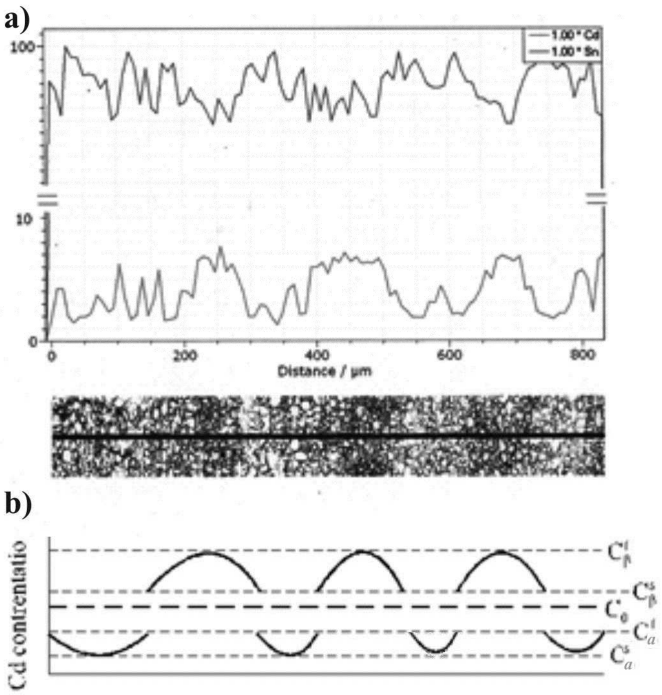

Fig. 5. Measured (a) and idealized (b) concentration distribution in the cells within the non-stirred part of the specimen

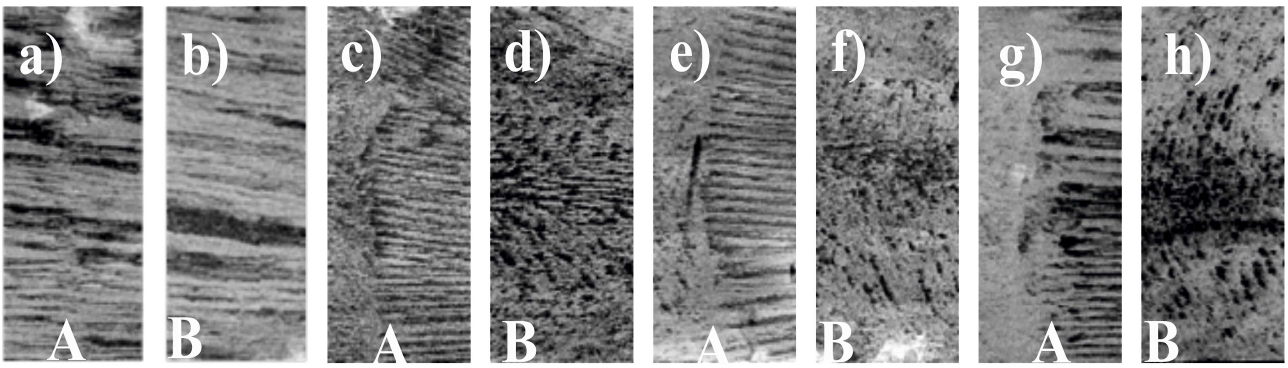

Fig. 6. The effect of stirring on the microstructure
a: 3 mT, A; b: 3 mT, B; c: 10 mT, A; d: 10 mT, B;
e: 20 mT, A; f: 20 mT, B; g: 60 mT, A; h: 60 mT, B
where: A: stirred/non-stirred boundary, B: stirred part

Fig. 6a, c, e, g, show transitional zone between the stirred and non-stirred parts, while Fig. 6b, d, f, h illustrate the structure of the stirred parts at 3, 10, 20 and 60 mT. During stirring at 3 mT no separation of the structure occurs. During stirring at 10, 20 and 60 mT the columnar structure is broken, an equiaxial structure is formed in the stirred parts and a “Christmas tree” microstructure observed as in other experiments is also was formed [10]. Fig. 7 shows the “Christmas tree” type structure formed in the stirred part in lower (a) and higher (b) magnification (20 mT). The lighter areas in the figures are the primarily crystallized Sn solid solution, the broken branches of the “Christmas tree” (darker spots) are $CdSn_4$ compound formed at the end of solidification and eutectoidally transformed during cooling.

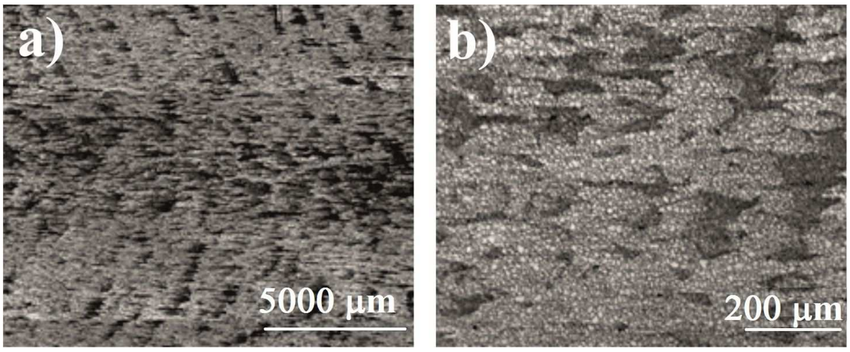

Fig. 7. “Christmas tree” type of microstructure formed in the stirred part of the specimen (20 mT) (Light microscope)

Macrosegregation

Two types of flow occur in the melt due to rotating magnetic field. The melt flows around the sample axis (primary flow) and parallel to its axis (secondary flow) (Fig. 3). The primary flow rate is by an order of magnitude higher than that of the secondary flow [15]. Due to the primary flow the higher concentration part of melt flows in the direction of the probe axis, where it is enriched, developing the trunk and branches of the “Christmas tree”. Fig. 8 shows the distribution of Cd concentration as function of radius at section 1, 2 and 3 in Fig. 1 (B = 60 mT). The concentration is significantly higher in the vicinity of the probe axis and at its edge than around the half-radius at section 2 and 3. This phenomenon has not been observed in similar experiments; in the case of Al alloys the lowest concentration was at the edge of the specimen [10]. The explanation is not yet known. Section 1 is close to the section when stirring was turned on; the difference here is much smaller than at sections 2 and 3 being farther from the section of stirring turn on.

Due to the effect of secondary flow, cadmium rich melt flows upwards along the axis, being replaced by the melt poorer in cadmium. As a result, moving from the section of stirring turn on the mean concentration and the difference between the center and half-radius concentration was increasing continuously. Increased magnetic induction results increased stirring intensity; in the current location (Figure 9, section 2 in different samples) a higher mean concentration is measured.

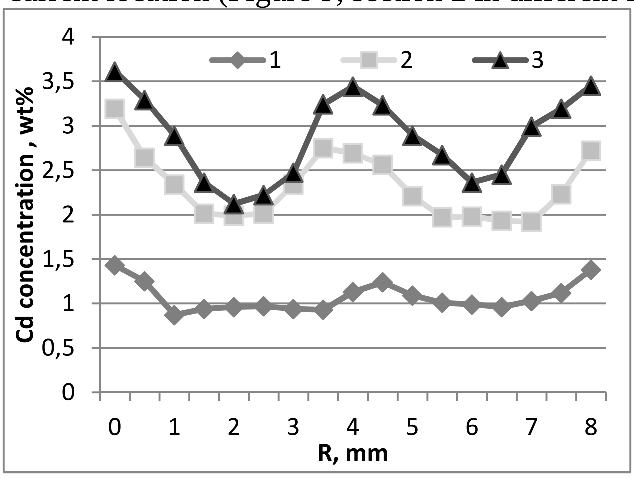

Fig. 8. Distribution of Cd concentration as function of radius from points 1, 2, 3 in Fig. 1. (B=60 mT)

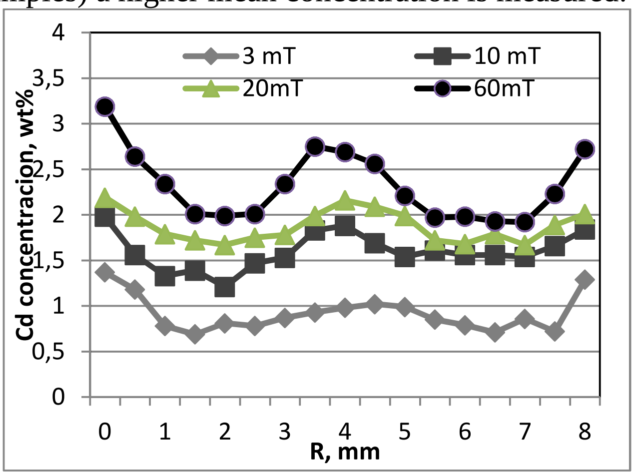

Fig. 9. Distribution of Cd concentration as function of radius from point 2 in Fig. 1 at different B values

Conclusion

Solidified a Sn-1.6 wt% Cd alloy with 0.02 mm/s motion velocity, 6 K/mm temperature gradient, a columnar (cellular) microstructure is formed. Stirring the melt with RMF at a magnetic induction of 3 mT the columnar structure is retained, while at 10 mT or higher values an equiaxial microstructure is solidified. A so-called "Christmas tree" microstructure is formed. Due to the effect of primary melt flow the Cd concentration in the center of the specimen and at its edge is significantly higher than at the half-radius. As a result of secondary flow, moving from the section of stirring turn on the mean concentration and the difference between the center and half-radius concentration increased continuously.

Acknowledgments

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References

[1] W.A. Tiller, K.A. Jackson, J.W. Rutter and B. Chalmers: The redistribution of solute atoms during the solidification of metals, Acta Metallurgica, Vol. 1. July (1953) 428-437

[2] M. Hainke, J. Friedrich, G. Müller: Numerical study on directional solidification of AlSi alloys with rotating magnetic fields under microgravity conditions, Journal of Materials Science, 39, (2004) 2011-2015

[3] S. Steinbach, L. Ratke: The effect of rotating magnetic fields on the microstructure of directionally solidified Al–Si–Mg alloys, Materials Science and Engineering A 413–414 (2005) 200–204

[4] F.D. Bai, M.H. Sha, T.J. Li, L.H. Lu: Influence of rotating magnetic field on the microstructure and phase content of Ni–Al alloy, Journal of Alloys and Compounds 509 (2011) 4835-4838.

[5] Z. Chen, X.L. Wen, C.L. Chen: Fluid flow and microstructure formation in a rotating magnetic field during the directional solidification process, Journal of Alloys and Compounds 491 (2010) 395-401.

[6] J.J. Guo, X.Z. Li, Y.Q. Su, S.P. Wu, H.Z. Fu, Formation mechanism of band structure and phase selection during directional solidification of peritectic alloys, Acta Metallurgica Sinica. 41 (2005) 599-604.

[7] H. Yasuda, N. Notake, K. Tokieda, I. Ohnaka: Periodic structure during unidirectional solidification for peritectic Cd-Sn alloys, Journal of Crystal Growth 210 (2000) 637-645

[8] R. Trivedi, J. S. Park: Dynamics of microstructure formation in the two-phase region of peritectic systems, Journal of Crystal Growth 235 (2002) 572–588

[9] A. Noeppel, A. Ciobanas, X.D. Wang et al: Influence of forced/natural convection on segregation during the directional solidification of Al-based binary alloys, Metallurgical and Materials Transactions B 41(1), 2010, pp. 193-208

[10] A. Rónaföldi, J. Kovács, A. Roósz: Investigation and visualisation of melt flow under rotating magnetic field, Transactions of the Indian Institute of Metals (2-3) pp. 213-218. (2007)

[11] B. Fragoso, H. Santos: Effect of a rotating magnetic field at the microstructure of an A354, Journal of Materials Research and Rechnology, 2 (2) (2013) 100-109

[12] L. Wang, Jun Shen, Zhao Shang, Hengzhi Fu: Preparation of gradient material in Sn–Cd peritectic alloy using rotating magnetic field, Journal of Crystal Growth 375 (2013) 32–38

[13] L. Wang, Jun Shen, Ling Qin, Zhourong Feng, Lingshui Wang, Hengzhi Fu: The effect of the flow driven by a travelling magnetic field on solidification structure of Sn–Cd peritectic alloys, Journal of Crystal Growth 356 (2012) 26–32

[14] L. Wang, J. Shen, Z. Feng, H. Fu: Effect of rotating magnetic field on microstructure formation of directionally solidified Sn-1.6 Cd peritectic alloy, Applied Physics A, 113 (2013) 177-183

[15] lSi7Mg0.6 alloy during directional solidification, Materials Science and Engineering A, 413–414 (2005) 236–242

[16] W.J. Boettinger: The structure of directionally solidified two-phase Sn-Cd peritectic alloy, Metallurgical Transactions 5, (1974) 2023-2031