Residual stresses of the as-cast Mg-xCa alloys with hot sprues by neutron diffraction

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Abstract. Residual stresses in Mg-xCa (0.5 wt. % and 1.0 wt. %) cast alloys were investigated by neutron diffraction using a mould with two preheating temperatures of 250 °C and 450 °C. Results show that the increase of Ca content decreases the residual stress in the hot sprue region. These results are quite in agreement with those obtained by the measurement of hot tearing susceptibility, which shows the increment in Ca content improves the castability of Mg-Ca alloys.

Introduction

Hot tearing or hot cracking is often a major casting defect and have significant impact on the quality of casting products. For the subsequent processing or service, those components free from casting defects and with a very low residual stress are preferred. All thermal contraction associated with decreasing temperature and the change in materials state exerts thermal stress during casting. Thermal stress involves different consequences such as distortion, crack, hot tear, and residual stress [1]. Hot tearing is a complex solidification phenomenon which is still not fully understood, though various mechanisms have been proposed [2].

Research on magnesium alloys is getting more attractive in recent years due to their potential in 3C (consumer, computer, communication) and automotive industries [1]. Most previous studies on hot tearing susceptibility were carried out on alloys series like Mg-Al [2, 3], Mg-Zn [4] and Mg-RE (Rare Earths) [5-7]. Different to aluminum alloys and steels, magnesium alloys have low elastic constants. Therefore, a very small residual stress can cause the distortion of casting components. Previous investigations on hot tearing of Mg-Al and Mg-Zn alloys have shown that casting defects such as hot cracks could occur at the hot spot due to accumulation of thermal stresses during solidification.

The alloying element Ca plays a quite important role in adjusting the mechanical properties of structural or medical magnesium alloys [3, 8-10]. The addition of Ca in AZ alloys improves the creep resistance due to the formation of thermal stable intermetallic Al_2Ca [10]. Owing to that both Mg and Ca have an excellent biocompatibility, Mg-Ca alloys are being considered as the potential degradable biomaterials [11]. The present investigation quantifies the distribution of residual stress in castings of Mg-Ca alloys using neutron diffraction from the point of view of their castability. Neutron diffraction for stress analysis is preferred because of its high penetration depth and flexible gauge volume choices for coarse grained as-cast ingot. Furthermore, it is also possible to perform an *in-situ* strain study during the solidification process with high flux reactor source. The obtained thermal stress distribution is used to evaluate the formation of hot tearing in Mg-Ca alloys in combination with the experimental results.

Experimental

The ingots for the neutron diffraction measurement were prepared using the setup of hot tearing evaluation system which was discussed in detail elsewhere [12]. A cylindrical mild steel crucible coated with hexagonal BN was used for melting in an electrical resistance furnace. High purity Ar + 0.2 % SF_6 protective gas mixture was used for melt protection. Pure magnesium (99.9 wt. %.) and

calcium (99.6 wt. %) were used as starting materials. Calcium was added to the melt at 700 °C. The melt was stirred for 5 min. manually. Then the melt was held at the pouring temperature for about 5 min before casting. The pouring temperature was set at 100 °C above the liquids temperature. The melt was poured into a mould coated with a thin layer of BN, which was preheated to the temperature of 250 °C and 450 °C. The macroscopic picture of a typical casting ingot is shown in Fig. 1 (a). The end of the casting was fixed to a certain position to simulate the constraint shrinkage, as marked with arrow. For comparison, a sample was cast without constraint shrinkage. The upper head (marked with red dashed box and white arrow in Fig.1 (a)) was later machined off, and the rest (both the sprue and rod, also marked in Fig. 1 (a)) was used for the diffraction. All the samples used for stress measurements were in the same size. In this study, two Ca contents (0.5 wt. % and 1.0 wt. %) were selected. The stress-free reference sample was machined from the head as a disk of Ø2×2 mm^2 from each Mg-Ca alloy. The microstructures of transversal cross-section of the rod near the junction area were checked by Optical microscopy (OM). The samples were cold mounted, ground with SiC sandpaper until 2500, polished with water free OPS and 1 μm diamond suspension, etched with picric acid. OM was performed with a LEICA DMI5000 M microscope. The grain size was measured by linear intercept method.

Neutron diffractometer STRESS-SPEC at FRM II (Heinz Maier-Leibnitz Zentrum, Garching, Germany) was used to measure the residual strain [13]. The measurements were performed at room temperature. A wavelength of 0.147 nm produced by Ge (311) monochromator was selected to measure the peak position variation of (21.1) planes of Mg. The investigated gauge volume 3×3×2 mm^3 was controlled using a primary slit of 3×3 mm^2 and a radial collimator with FWHM (full width of half maximum)= 2 mm. Fig. 1 (b) shows the setup for strain scanning. Three dimensional stress measurements are also illustrated. Due to relatively large grains of the cast ingot, the sample was oscillated around Ω angle with amplitude of ± 5°. An 8 mm^3 cubic sample cut from the ingot was used as the stress-free reference.

Triaxial strain scanning (radial, hoop, and axial) was carried out with a step size of 10 mm through the central line of sprue and rod. The lattice strain (ε_{hkl}) for the (21.1) reflection at a constant wavelength was calculated by,

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{hkl}^0}{d_{hkl}^0} = \frac{\sin \theta_{hkl}^0}{\sin \theta_{hkl}} - 1 \tag{1}$$

where d_{hkl} is the lattice spacing of (21.1) planes in the residual stressed materials and θ_{hkl} the corresponding diffraction angle, d_{hkl}^0 is the lattice spacing of *hkl* planes in the stress-free materials and θ_{hkl}^0 the corresponding diffraction angle. With Hook's law the residual stress was then calculated using Young's modulus of 44 GPa and Poisson's ratio of 0.293.
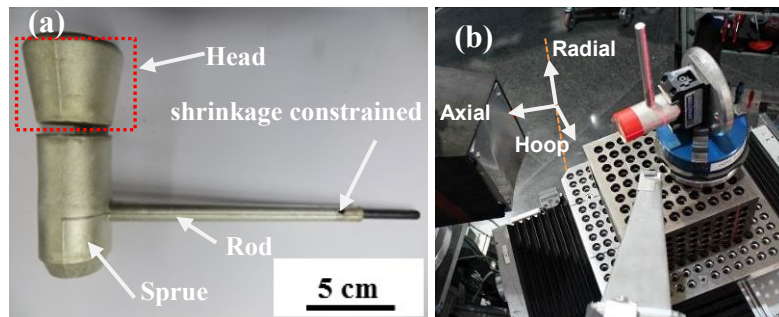

Fig. 1 (a) Macroscopic picture of a typical casing; (b), View of the instrument setup at STRESS-SPEC for the axial direction strain scanning of the Mg- 1.0 wt. % Ca sample.

Results and discussions

The microstructures (perpendicular to the hoop direction) of Mg - 0.5 wt. % Ca and Mg - 1.0 wt. % Ca alloys cast at 450 °C are shown in Fig. 2 (a) and (b), with an average grain size of 370 ± 115 μm and 185 ± 59 μm, respectively.

Distribution of the triaxial residual stresses (radial, hoop, and axial) along the central line for Mg-1.0 wt. % Ca alloy with a mold temperature of 250 ℃ is plotted in Fig. 3(a). The sample was cast without constraint shrinkage. The position of junction area is marked by the red dashed line. Both the radial and hoop stresses show similar variations from left to right of rod end. There is barely any stress from the junction to the end of the rod. The casting ingot for the residual stress measurement is shown in Fig. 3(b), as can be seen, the sample is freely contracted during solidification. No evidence of crack was found at the junction area. For normal constrained shrinkage casting, it is totally broken at the junction area due to the high stress concentration (Fig. 3(c)).
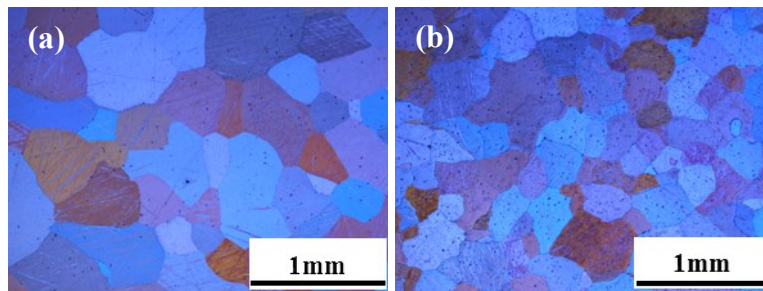

Fig. 2 Optical microstructures of alloys at the mould temperature of 450 °C, (a) Mg-0.5 wt. % Ca, (b) Mg-1.0 wt. % Ca.
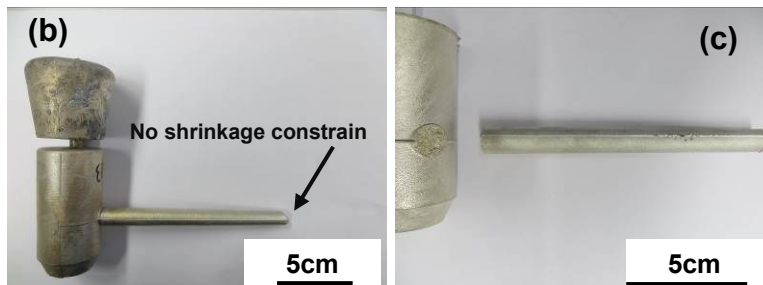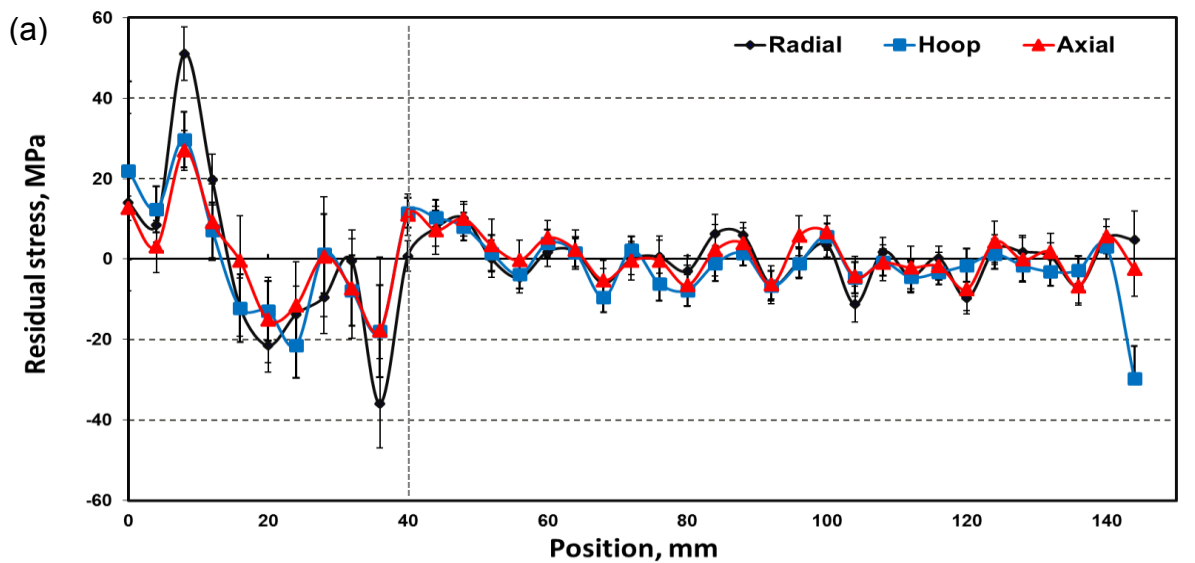

Fig. 3 (a) Triaxial residual stress distribution of the Mg-1.0 wt. % Ca casting at the mould temperature of 250 ℃; macroscopic pictures of casings (b) free shrinkage (for stress measurement), (c) constrained shrinkage.

During the last stage of solidification, the thermal contraction of dendrites is hindered by the previously solidified dendrites, which results in a tensile stress. This tensile stress caused by non-uniform thermal contraction of the coherent dendrites might generate gaps and cavities. If the

liquid can flow freely, the gaps and cavities will be filled up with the residual liquid. These microscopic defects will be "healed", and thus hot cracks will not appear [14]. In case the liquid cannot flow freely in the constrained shrinkage casting, the stresses will pile up at the weak area (e.g., the junction area for this kind of mould) and finally result in hot crack. The freely contracted Mg-1.0 wt. % Ca cast at a mould temperature of 250 °C shows no obvious stress concentration, which is in good agreement with its hot tearing susceptibility.

Fig. 4 shows the distribution of triaxial residual stress along the central line of Mg-1.0 wt. % Ca at a mould temperature of 450 °C. The sample was cast under constrained shrinkage. All the radial, hoop and axial stresses exhibit similar trend from the junction area to the end of the rod. From left surface to the junction the residual stress exhibits first as tensile then as compressive and then again as tensile type. The maximum compression stresses appear around at position 20 mm, which are 50 ± 9 MPa, 28 ± 7 MPa and 48 ± 7 MPa for redial, hoop and axial, respectively. Then the stresses increase and change to tensile stresses, reach the maximum at position 36 mm (4 mm away from the junction area), which are 53 ± 5 MPa, 20 ± 6 MPa and 35 ± 7 MPa for redial, hoop and axial, respectively. Among the three kinds of residual stresses, the radial stress showed the highest value.
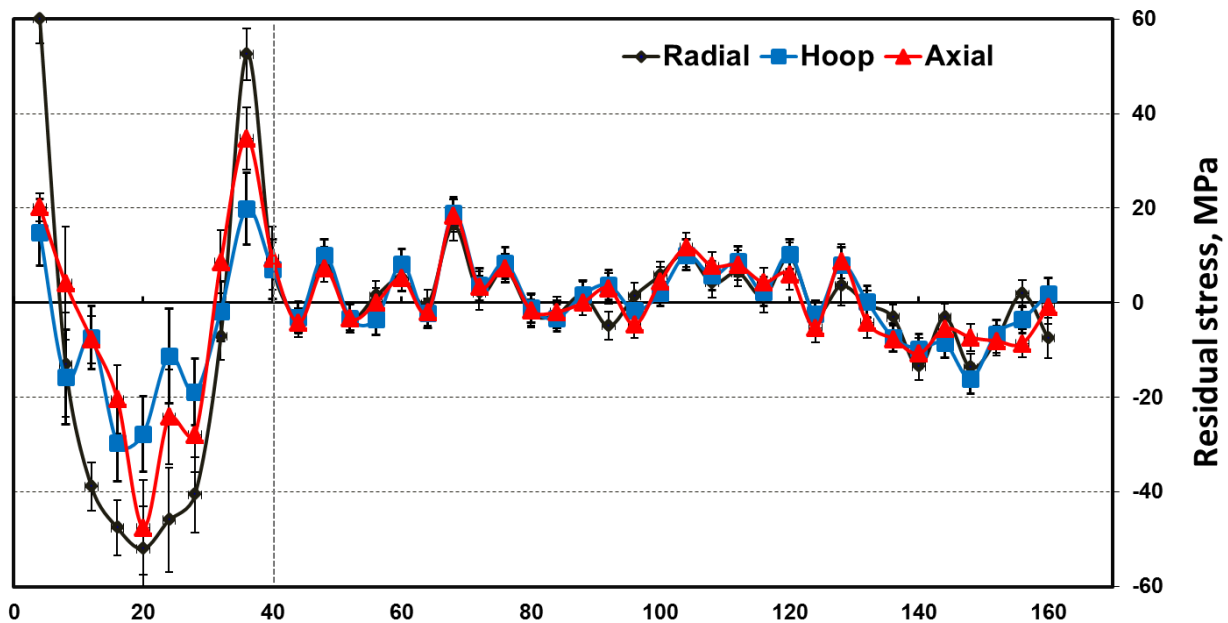

Fig. 4 Triaxial residual stress distribution of the Mg-1.0 wt. % Ca sample at the temperature of 450 ℃.

As illustrated above, the tensile residual stress may finally result in hot crack. The tensile residual stress detected in Mg-1.0 wt. % Ca at 450 ℃ indicates that, there is a high risk to produce the hot cracks at the junction area. Comparing the Mg-1.0 wt. % Ca samples cast at different mould temperatures, the sample cast at 250 ℃ is completely broken at the junction. While the casting at 450 ℃ shows no visible crack at the junction area, it still exists high tensile stress. Consequently, the sample with a lower mould temperature of 250 ℃ shows a higher hot tearing tendency. The sample cast with a lower mould temperature has a higher thermal gradient which can cause a higher internal thermal stresses. As a result, the initiation and propagation of hot crack are promoted [15].

The triaxial residual stress distribution of Mg-0.5 wt. % Ca at mould temperature of 450 ℃ is shown in Fig. 5. This sample was also cast under constrained shrinkage. For comparison, all the radial, hoop and axial stresses are analyzed along the central line. Due to relatively coarse grains in the ingot the calculated stress has a large error bar. However, no obvious residual stress is evidenced along the detected line for all the three directions. This might be due to the fact that a small crack can be found at the junction area, as show in Fig. 6(a) and (b), pointed by the arrow. As the hot crack initiates and propagates, the piled up stress releases simultaneously. As a result, the residual stress is

not obvious, which is in agreement with the experimental results. Nevertheless, Mg-0.5 wt. % Ca still exhibits a higher hot tearing susceptibility than Mg-1.0 wt. % Ca. A higher content of Ca in Mg-Ca alloys provides more eutectic phases at the final stage of solidification, which is beneficial for "healing" of the hot crack, in other words, for suppressing the hot tearing susceptibility. However, this need more detailed investigations, which are still in work.
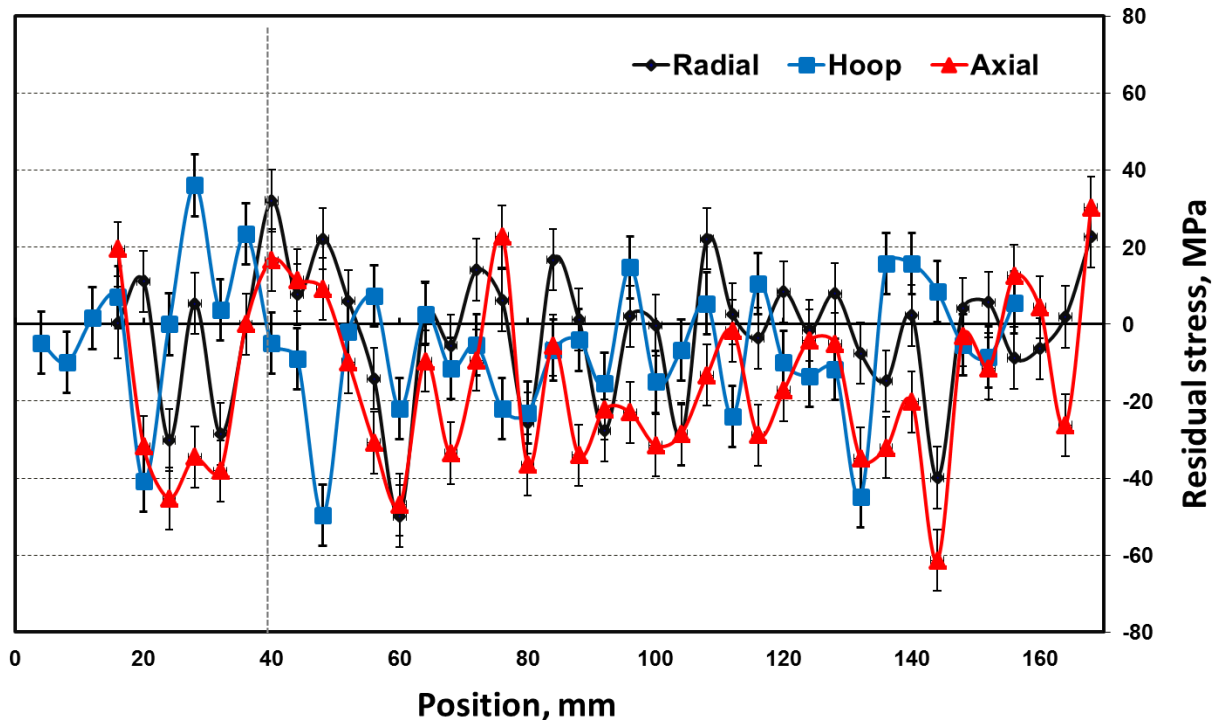

Fig. 5 Triaxial residual stress distribution of the Mg-0.5 wt. % Ca sample at the mould temperature of 450 °C.
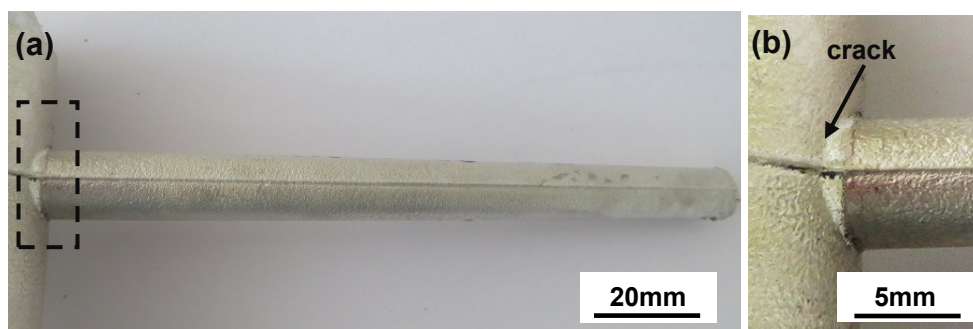

Fig. 6 (a) Macroscopic picture of Mg-0.5 wt. % Ca sample at a mould temperature of 450 °C, (b) close up of the junction area.

Summary

Neutron diffraction was successfully introduced to measure the residual stress in Mg-Ca hot tearing ingots with relatively coarse grains. The obtained results are in good agreement with the hot tearing tendency results. Hot crack will not appear in freely contracted castings during solidification. High mould temperature will decrease the hot tearing tendency as well as the higher content of Ca for Mg-Ca alloys.

References

[1] K.U. Kainer, Magnesium Alloys and Technology, Wiley-VCH GmbH, Weinheim, Germany 2003.

[2] Z.S. Zhen, N. Hort, Y.D. Huang, O. Utke, N. Petri, K.U. Kainer, Hot tearing behaviour of binary Mg-1Al alloy using a contraction force measuring method, Inter.J. of Cast Metals Res. 22 (2009) 331-334.

[3] Y.S. Wang, B.D. Sun, Q.D. Wang, Y.P. Zhu, W.J. Ding, An understanding of the hot tearing mechanism in AZ91 magnesium alloy, Mater. Lett. 53 (2002) 35-39.

[4] L. Zhou, Y.D. Huang, P.L. Mao, K.U. Kainer, Z. Liu, N. Hort, Influence of composition on hot tearing in binary Mg–Zn alloys, Inter. J. of Cast Metals Res. 24 (2011) 170-176.

[5] Z. Wang, Y. Huang, A. Srinivasan, Z. Liu, F. Beckmann, K. Kainer, N. Hort, Experimental and numerical analysis of hot tearing susceptibility for Mg–Y alloys, J. of Mater. Sci. 49 (2014) 353-362.

[6] Z. Wang, Y. Huang, A. Srinivasan, Z. Liu, F. Beckmann, K.U. Kainer, N. Hort, Hot tearing susceptibility of binary Mg–Y alloy castings, Mater. & Des. 47 (2013) 90-100.

[7] Z. Wang, Y.D. Huang, A. Srinivasan, Z. Liu, K.U. Kainer and N. Hort, Influences of Y Additions on the Hot Tearing Susceptibility of Mg-1.5wt.%Zn Alloys, Mater. Sci. Forum. 765 (2013) 306-310.

[8] Q.D. Wang, W.D. Chen, X.Q. Zeng, Y.Z. Lu, W.J. Ding, Y.P. Zhu, X.P. Xu and M. Mabuchi, Effects of Ca addition on the microstructure and mechanical properties of AZ91magnesium alloy, J. of Mater. Sci. 36 (2001) 3035-3040.

[9] W.-C. Kim, J.-G. Kim, J.-Y. Lee and H.-K. Seok, Influence of Ca on the corrosion properties of magnesium for biomaterials , Mater. Lett. 62 (2008) 4146-4148.

[10] A.A. Luo, M.P. Balogh and B.R. Powell, Creep and microstructure of magnesium-aluminum-calcium based alloys, Metall Mater Trans A. 33 (2002) 567-574.

[11] Y. Wan, G. Xiong, H. Luo, F. He, Y. Huang and X. Zhou, Preparation and characterization of a new biomedical magnesium-calcium alloy, Mater. & Des. 29 (2008) 2034-2037.

[12] Z.S. Zhen, N. Hort, Y.D. Huang, N. Petri, O. Utke and K.U. Kainer, Quantitative Determination on Hot Tearing in Mg-Al Binary Alloys, Light Metals Technology 2009. 618-619 (2009) 533-540.

[13] M. Hofmann, J. Rebelo-Kornmeier, U. Garbe, R. C. Wimpory, J. Repper, G. A. Seidl, H. G.Brokmeier and R. Schneider, Stress Spec: Advanced Materials Science at the FRM II, Neutron News, 18 (2007)27-30.

[14] L. Katgerman and D.G. Eskin, In search of the prediction of hot cracking in aluminium alloys, Springer Berlin Heidelberg, Germany. (2008) 11-26.

[15] S. Lin, A study of hot tearing in wrought aluminium alloys, PhD thesis, University of Québec at Chicoutimi, Canada. 1999.