

## The Influence of Casting Methods on Microstructure of Al-Mg-Sc-Zr Alloy

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**Two methods of fabrication of Al-Mg-Sc alloy are compared - conventional casting followed by cold-rolling and twin-roll casting. To the advantages of twin-roll casting belong the lower material and energy requirements during the production. However, twin-roll cast material exhibits different initial microstructure and routes established for conventionally cast materials cannot be applied. In twin-roll cast material the distribution of primary particles is more inhomogeneous - they form segregation near the center and edges of the sheet and their average size is smaller. On the contrary, the average sub-grain size is much higher in the twin-roll cast material.**

**Keywords:** Al-Mg, Twin-roll casting, Conventional casting, Central segregation, Particles distribution

### 1 Introduction

Al-Mg-based alloys are widely used in aerospace and ship-building industry due to their excellent weldability, reasonable corrosion resistance and possibility of superplastic forming [1-3]. Additional alloying with Sc and Zr offers a great potential for developing of new lightweight structural materials with exceptional mechanical properties thanks to presence of  $Al_3(Sc,Zr)$  precipitates which strengthen the alloy and increase the thermal stability [4-7].

One of the disadvantages of the Al-Mg type of alloys is that they may suffer from exfoliation corrosion [3,4]. Generally, exfoliation corrosion occurs when a highly directional microstructure is present in the material. Such is typical for sheets and strips produced by rolling of initial ingots. Therefore, new manufacturing processes minimizing the formation of a pancake structure with flat and elongated grains are under current research.

Twin-roll casting (TRC) is a typical example of manufacturing method, which enables casting of strips directly with the thickness requested for semi-finished product [8,9]. The most significant feature of this technology is a formation of rather equiaxed grains, in contrast to ones generated by a rolling procedure. However, the differences in the processing route compared to direct chill (DC) cast materials (omission of steps like homogenization, scalping, rolling and intermediate) leads to formation of a structure with appreciable different features [10,11]. Therefore, processing routes for TRC materials must be modified from those established for DC cast materials. Recently, the manufacturing and modification of high-strength Al-Mg-Si and Al-Mg-based alloys prepared by TRC are under intensive research [12,13]. However, there are only sparse information concerning the preparation and properties of TRC materials containing Zr and Sc [14,15].

In the present work two variants of Al-Mg-Sc-Zr alloy are compared regarding the microstructure and primary particles distribution.

### 2 Experimental

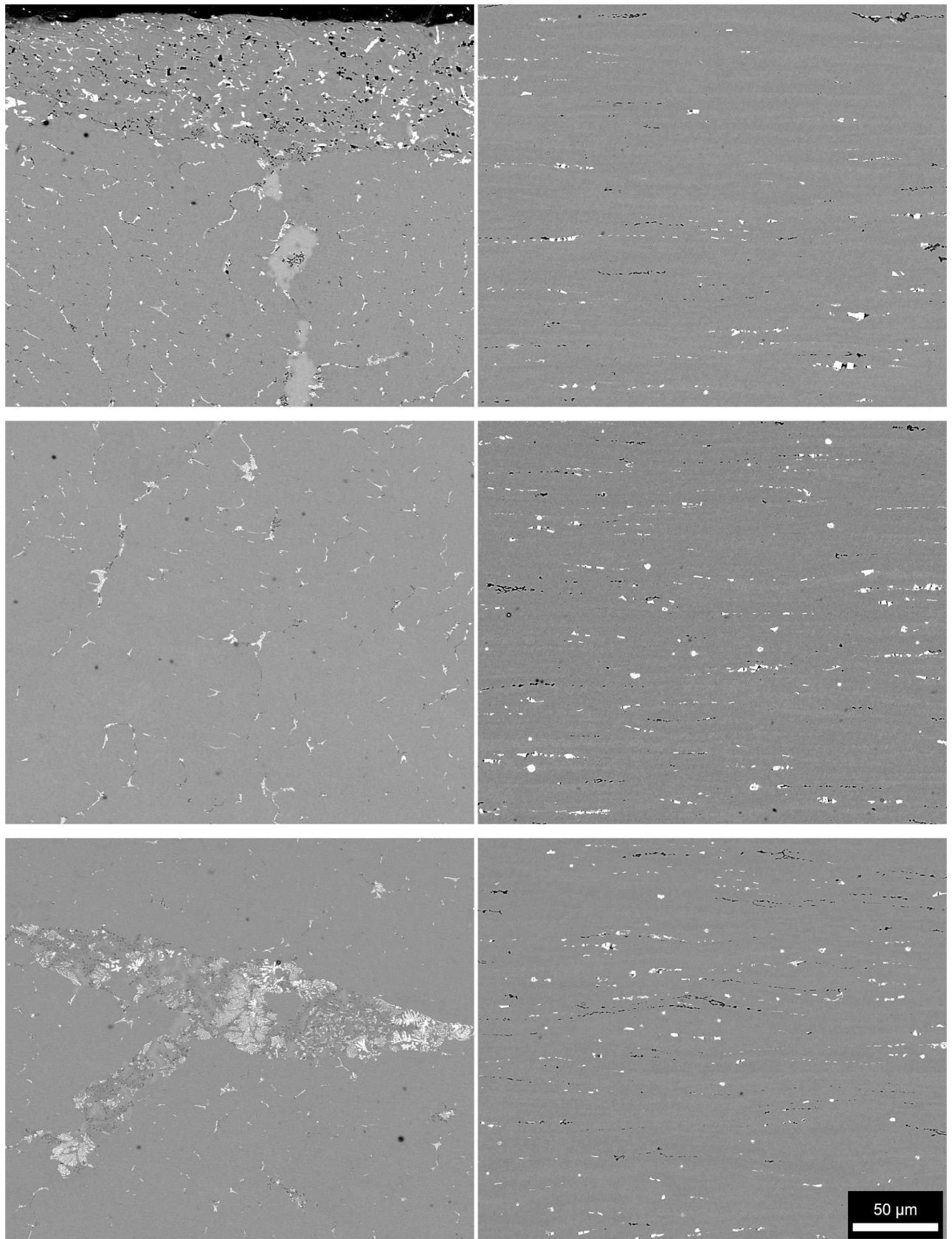
Aluminium alloy with composition 3.24 wt.% Mg, 0.19 wt.% Sc, 0.14 wt.% Zr, 0.16 wt.% Mn, 0.11 wt.% Si and 0.21 wt.% Fe was cast in two variants: twin-roll casting to 5 mm and conventional casting with subsequent cold-rolling to thickness 5 mm. The microstructure was observed by scanning electron microscope (SEM) FEI Quanta 200 equipped with energy dispersive spectroscopy (EDS) detector EDAX for chemical analysis and by transmission electron microscope (TEM) JEOL 2000FX. Specimens for TEM were electropolished in 30%  $HNO_3$  solution in methanol at -15 °C.

### 3 Results and discussion

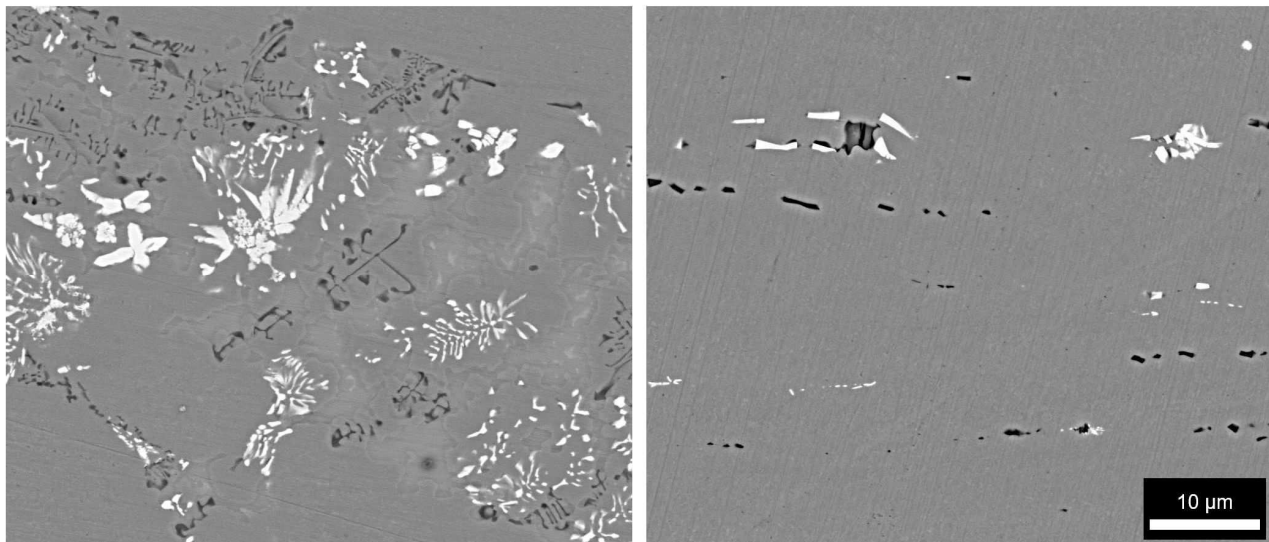
Both twin-roll cast and conventionally cast materials contain high density of primary particles (Figure 1 and 2). The notable difference lies mainly in their spatial distribution within the strip thickness and in their size.

Concerning the conventionally cast material, the particles are aligned along the rolling direction in lines. Such distribution was created by the rolling step applied to the material after casting in order to reach the sheet thickness of 5 mm. The original grains elongated and with them also the particles copying the grain boundaries. The distribution is relatively homogeneous within the sheet thickness.

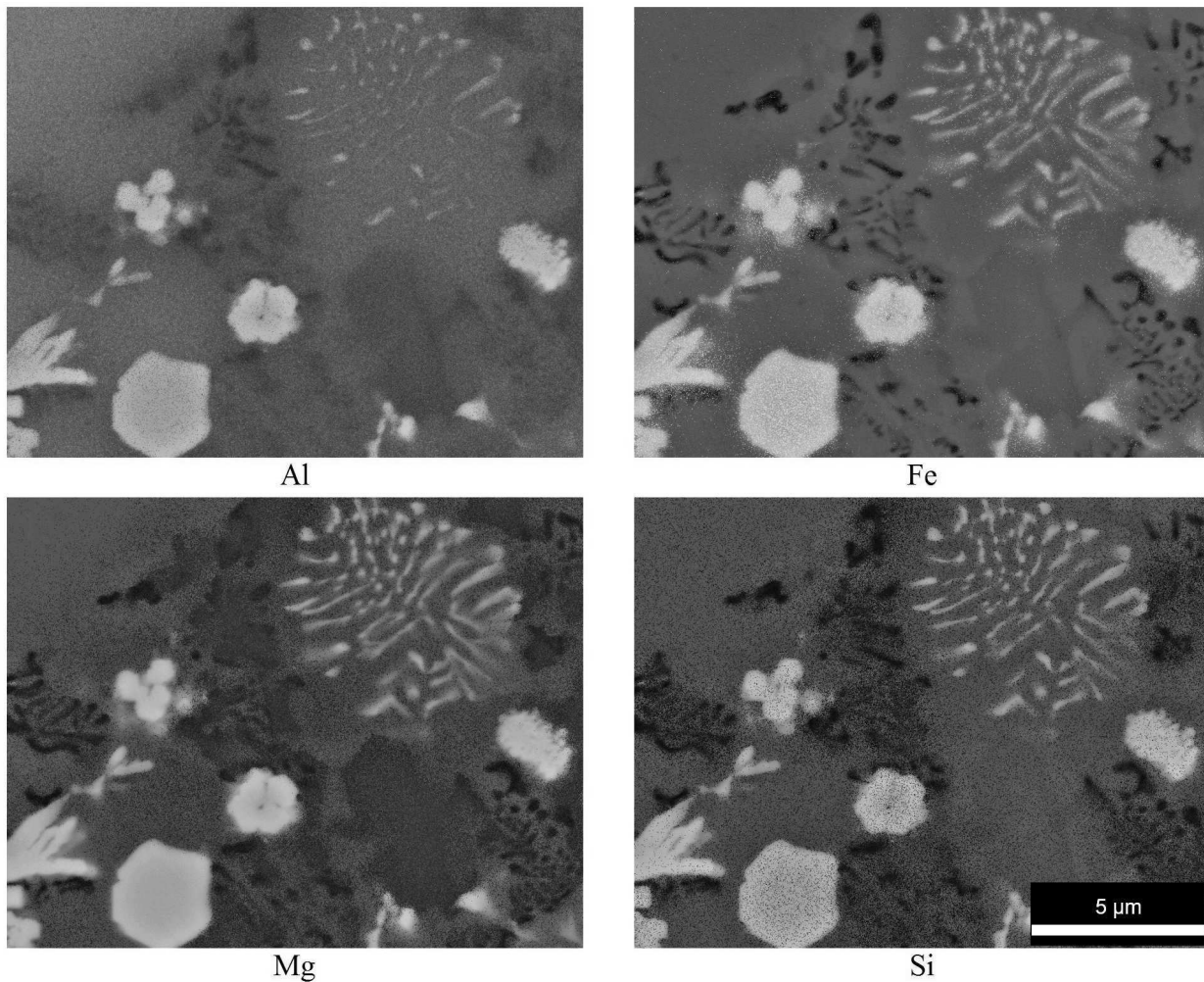
The situation is completely different in the twin-roll cast material which is studied directly in the as-cast state. The dominant features of the particle distribution are macrosegregations in the central part and also near the strip surface. The central segregations were already described for several types of twin-roll cast aluminium alloys, see e.g. [16]. They form during solidification of the strip when the unsolidified part of the material is enriched in the solutes and during the final stage of solidification eutectic solute-rich channels form. Their size, distribution and their presence itself can be influenced by the casting parameters [13,17]. The central segregation is characterized by huge agglomeration of primary particles with dendritic structure and length in order of 500  $\mu m$  in the rolling direction, 100  $\mu m$  in the transversal direction and height 100  $\mu m$  in the normal direction.



**Fig. 1** Comparison of the particles distribution in the twin-roll cast (left) and conventionally cast (right) materials. The regions near the edge of the strip (top), in the bulk of the material (center) and in the central part of the sheet (bottom) are depicted. Viewed in the transversal direction.



**Fig. 2** Detailed SEM image of the two main types of particles present in the twin-roll cast (left) and conventionally cast (right) materials.



**Fig. 3** EDS maps for chemical analysis of particles present in the studied material – Al, Fe, Mg and Si. The matrix contains mainly aluminium, dark particles are  $Mg_2Si$ , bright particles are rich in iron and some areas within the segregation are rich in magnesium.

The segregations near the surfaces are characteristic with higher particle density than the bulk of the material, where the particles are distributed evenly at the grain

boundaries. The thickness of the enriched strip near the surface varies from 100 to 300  $\mu m$ . Several larger segregates under the surface region were also observed.

Two main types of particles were observed in both types of materials. Their chemical composition was determined by energy dispersive spectroscopy in SEM. The white particles are rich in iron; the black particles are  $\text{Mg}_2\text{Si}$  phase. An example of the elements distribution map (Al, Mg, Si and Fe) is given in Figure 3. These two types of particles were observed in both the twin-roll cast and conventionally cast materials. In addition, in the TRC material areas within the macrosegregation contained regions rich in Mg (without Si) – compare the red areas in Figure 3.

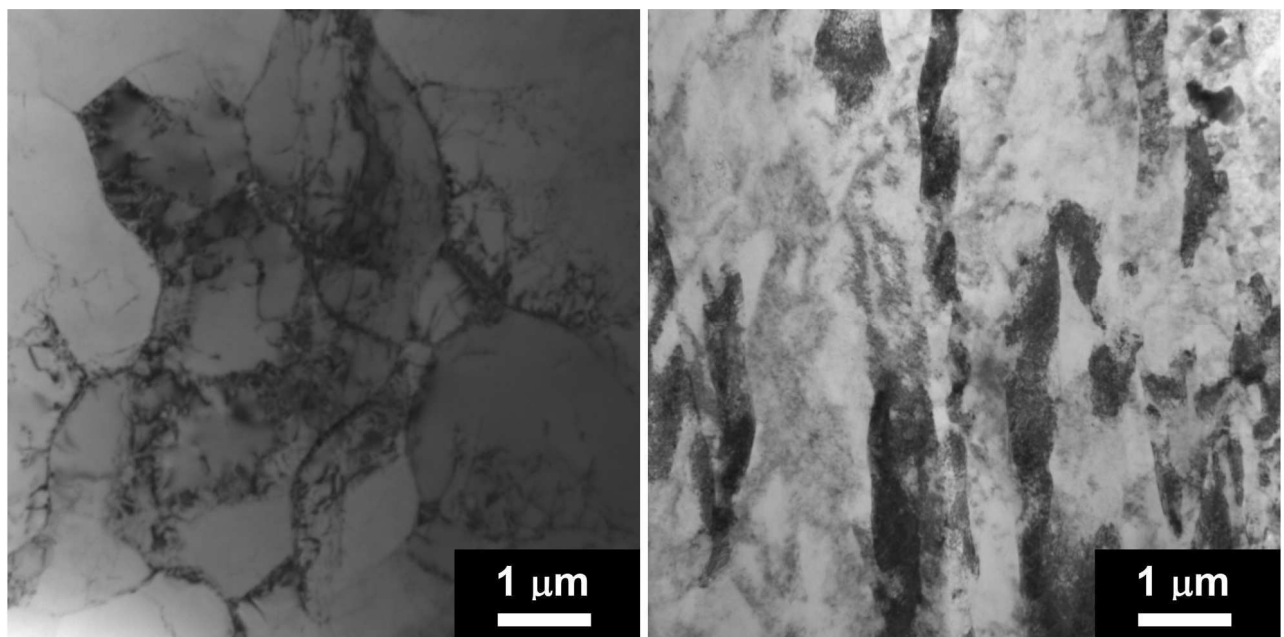
The relative size of both types of the primary particles in both materials can be compared in Figure 2. The detailed analysis of the particle size and shape distribution within the whole thickness of the strip revealed that both the particle size and area fraction is higher in the conventionally cast material. The high cooling rate during twin-roll casting may be responsible for the lower particle density as during the short time of solidification lower amount of solutes forms primary particles and the rest remains in the solid solution, causing the solid solution supersaturation which is typical for various twin-roll cast aluminium alloys [18].

In the conventionally cast material there the average

size of the Fe-rich particles is  $1.5\ \mu\text{m}$  and of  $\text{Mg}_2\text{Si}$   $1\ \mu\text{m}$ , the area fraction is 0.75 % and 0.35 %, respectively. In the twin-roll cast material the distribution is not homogeneous. The overall particle size is 1.2 and  $0.7\ \mu\text{m}$  for the Fe-rich and  $\text{Mg}_2\text{Si}$  particles, respectively, and the area fraction 1.2 % and 0.15 %. The area fraction in the central part and near the surfaces reaches 2.5 % and 1 % for the Fe-rich and  $\text{Mg}_2\text{Si}$  particles, respectively.

As lower amount of Mg formed  $\text{Mg}_2\text{Si}$  primary particles in the TRC material, the areas rich in Mg formed – within the central segregation, see Figure 3.

Detailed images from TEM (Figure 4) reveal other differences in the materials caused by different casting procedures. The subgrain size is much higher in the TRC material and also the dislocation density is much lower compared to the conventionally cast and rolled material – such huge change in the dislocation density was caused mainly by the rolling step in the material processing. Moreover, the elongation of subgrains is also apparent in the conventionally cast material. This observation confirmed the previous results [19] from light optical microscopy which revealed the shape of the whole grains – elongated in the conventionally cast materials and rather equiaxed in the twin-roll cast material.



**Fig. 4** Detailed observation of the microstructure (mainly subgrains) by TEM of the twin-roll cast (left) and conventionally cast (right) material.

#### 4 Conclusion

Twin-roll cast material, compared to the conventionally cast one after rolling to the comparable thickness, has less homogeneous distribution of primary phases and suffers from macrosegregation, both near the strip surface and in the center of the strip. Two types of primary particles were observed in both materials – iron rich phase and  $\text{Mg}_2\text{Si}$ . Moreover, Mg-rich regions appeared within the segregations in the twin-roll cast material. The average particle size is smaller in the twin-roll cast material.

The subgrains are smaller and elongated with higher

density of dislocations in the conventionally cast material.

#### Acknowledgement

*The work was supported by Czech Science Foundation project 16-16218S.*

#### References

- [1] FRIEDMAN, P. A., GHOSH A. K. (1996). Control of superplastic deformation rate during uniaxial tensile tests. *Metallurgical and Materials Transaction A*, 1996, vol. 27A, pp. 3030-3042.

- [2] YUZBEKOVA, D., MOGUCHEVA, A., KAIBYSHEV, R. (2016). Superplasticity of ultra-fine-grained Al-Mg-Sc-Zr alloy. *Materials Science and Engineering A*, 2016, vol. 675, pp. 228-242.
- [3] M. LIAO, N. C. BELLINGER, J. P. KOMOROWSKI (2013). Modeling the effects of prior exfoliation corrosion on fatigue life of aircraft wing skins, *International Journal of Fatigue*, 2013, vol. 25, pp. 1059-1067.
- [4] PENG, Y., LI, S., DENG, Y., ZHOU, H., XU, G., YIN, G. (2016). Synergetic effects of Sc and Zr microalloying and heat treatment on mechanical properties and exfoliation corrosion behavior of Al-Mg-Mn alloys. *Materials Science and Engineering A*, 2016, vol. 666, pp. 61-71.
- [5] FALLAH, V., LLOYD, D., J., GALLERNEAULT, M. (2017). Processing and characterization of continuous-cast AlMgSc(Zr) sheets for improved strength. *Materials Science and Engineering A*, 2017, vol. 698, pp. 88-97.
- [6] VLACH, M., ČÍŽEK, J., SMOLA, B., MELIKHOVA, O., VLČEK, M., KODETOVÁ, V., KUDRNOVÁ, H., HRUŠKA, P. (2017). Heat treatment and age hardening of Al-Si-Mg-Mn commercial alloy with addition of Sc and Zr. *Materials Characterization*, 2017, vol. 129, pp. 1-8.
- [7] VLACH, M., STULIKOVA, I., SMOLA, B., PIESOVA, J., CISAROVA, J., DANIS, S., PLASEK, J., GEMMA, R., TANPRAYOON, D., NEUBERT, V. (2012). Effect of cold rolling on precipitation processes in Al-Mn-Sc-Zr alloy, *Materials Science and Engineering: A*, 2012, vol. 548, pp. 27-32.
- [8] YUN, M., LOKYER, S., HUNT, J.S. (2000). Twin roll casting of aluminium alloys. *Materials Science and Engineering A*, 2000, vol. 280, pp. 116-123.
- [9] POKOVÁ, M., CIESLAR, M., LACAZE, J. (2012). TEM Investigation of Precipitation in Al-Mn Alloys with Addition of Zr. *Manufacturing Technology*, 2012, vol. 13, pp. 212-217.
- [10] LIU, W. C., ZHAI, T., MORRIS, J. G. (2004). Texture evolution of continuous cast and direct chill cast AA 3003 aluminum alloys during cold rolling, *Scripta Materialia*, 2004, vol. 51, pp. 83-88.
- [11] SLÁMOVÁ, M., KARLÍK, M., ROBAUT, F., SLÁMA, P., VÉRON, M. (2002). Differences in microstructure and texture of Al-Mg sheets produced by twin-roll continuous casting and by direct-chill casting, *Materials Characterization*, 2002, vol. 49, pp. 231-240.
- [12] SUN, K.M., LI, L., CHEN, S.D., XU, G.M., CHEN, G., MISRA, R.D.K., ZHANG, G. (2017). A new approach to control centerline macrosegregation in Al-Mg-Si alloys during twin roll continuous casting, *Materials Letters*, 2017, vol. 190, pp. 205-208.
- [13] WANG, Z., LI H., MIAO, F., FANG, B., SONG, R., ZHENG, Z. (2014). Improving the strength and ductility of Al-Mg-Si-Cu alloys by a novel thermo-mechanical treatment, *Materials Science and Engineering: A*, 2014, vol. 607, pp 313-317.
- [14] KŘIVSKÁ, B., ŠLAPÁKOVÁ, M., GRYDIN, O., Cieslar, M. (2017). The Microstructure Evolution of Al-Mg-Sc-Zr Alloy after Deformation by Equal Channel Angular Pressing. *Manufacturing Technology*, 2017, vol. 17, pp. 738-741.
- [15] CIESLAR, M., BAJER, J., ZIMINA, M., GRYDIN, O. (2016). Microstructure of Twin-roll Cast Al-Mg-Sc-Zr Alloy. *Manufacturing Technology*, 2016, vol. 16, pp. 1255-1259.
- [16] ŠLAPÁKOVÁ POKOVÁ, M., ZIMINA, M., ZAUNSCHIRM, S., KASTNER, J., BAJER, J., CIESLAR, M. (2016). 3D analysis of macrosegregation in twin-roll cast AA3003 alloy. *Materials Characterization*, 2016, vol. 118, pp. 44-49.
- [17] BIROL, Y. (2009). Analysis of macro segregation in twin-roll cast aluminium strips via solidification curves, *Journal of Alloys and Compounds*, 2009, vol. 486, pp. 168-172.
- [18] POKOVÁ, M., CIESLAR, M., SLÁMOVÁ, M. (2009). The influence of dispersoids on recrystallization of aluminium alloys. *International Journal of Materials Research*, 2009, vol. 100, pp. 391-394.
- [19] CIESLAR, M., BAJER, J., ŠLAPÁKOVÁ, M., KŘIVSKÁ, B., ZIMINA, M., GRYDIN, O. (2017). Microstructure of Twin-Roll Cast Al-Mg-Sc-Zr Alloy. *Materials Today: Proceedings*, 2017, in press.