

STRUCTURE AND PROPERTIES OF ALUMINUM ALLOYS WITH ADDITIONS OF TRANSITION METALS PRODUCED VIA COUPLED RAPID SOLIDIFICATION AND HOT EXTRUSION

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Abstract

Rapid solidification (RS) of Al - TM alloys (TM: Fe, Ni, Mn), combined with following mechanical consolidation of RS flakes was used to produce highly refined structure of the materials. RS flakes were manufactured using spray deposition of the molten alloy on the rotating water-cooled copper roll. The preliminary consolidation of the RS-flakes was performed at 320 °C by means of vacuum compression and 100-tonn press. As-compressed billets were then extruded at 400 °C using extrusion ratio $\lambda = 19$. Mechanical properties of as-extruded materials were examined at 20 °C - 550 °C by compression tests performed at constant true strain rate of $5 \cdot 10^{-3}$ (s⁻¹). It was found that the most effective strengthening of RS materials result from combined dispersion strengthening related to highly refined constituent particles and solid solution strengthening due to Mg addition. The effect of increased mechanical properties is pronounced at low and intermediate deformation temperatures, while at elevated temperatures deterioration of the properties is observed. Structural observations confirmed beneficial influence of the rapid solidification on an effective refining of intermetallic compounds, which is particularly visible for alloys with high concentration of transition metals.

Keywords: Aluminum, rapid solidification, hot extrusion, transition metals

1. INTRODUCTION

Conventional precipitation hardenable aluminum alloys are well known for their use as light-weight components in numerous engineering applications. Their usage, however, is strongly limited to the product service at low temperatures considerably lower than 300 °C, since higher service temperature affects their microstructure by its degradation, which in turn reduces mechanical properties. The development of rapid solidification (RS) processing has allowed refining structural components of aluminum alloys that provide superior properties of the product with respect to conventional materials tested at ambient and elevated temperatures [1-3]. The most promising materials in this regard are the aluminum based alloys with addition of transition metals (TM) such as: Fe, Ni, V, Mn, Cr etc. [4-5]. Superiority of rapid solidification (RS) over industrial metallurgy (IM) methods is associated with the formation of highly dispersed intermetallic particles, which guarantees effective strengthening of these alloys. Further potential advantages of rapid solidification emerge if combined with plastic consolidation (PC) by hot extrusion [6-7]. Due to high dispersion of precipitates, fracturing of relatively brittle phases does not occur during the material processing. Consequently, effective refinement of Al-matrix grains gives an extra strengthening effect to the overall strength of the material. Additionally, recent research results lead to the conclusion that the strength of the Al-TM alloys can be further increased by the addition of magnesium to aluminum matrix. It has been reported that the solid solution strengthening due to Mg addition effectively increases mechanical properties at low and intermediate temperatures [8-9]. In this paper the influence of rapid solidification on the microstructure and mechanical properties of Al-TM, (where TM = Mn, Fe, Ni) alloys was analyzed. Selected transition metals are known to form coarse intermetallic particles during traditional crystallization, however if rapid solidification and powder metallurgy techniques are applied advantageous morphology of intermediate and constitutive phases is expected to occurs. The research program was focused on the microstructural features of RS materials and their affect on the material properties of both RS-materials and samples produced by conventional ingot metallurgy (IM) method.

2. EXPERIMENT

Series of binary and ternary Al-TM (TM: Fe, Ni, Mn) alloys were prepared in order to determine the effect of rapid solidification on the structure and properties of the materials. The composition of tested materials has been carefully selected in order to show the effect of: (i) different type of alloying elements, (ii) different alloying concentration and (iii) magnesium addition. The chemical composition of tested materials is shown in **Table 1**.

Table 1 Nominal and analyzed composition of tested alloys

Nominal composition (mass %)	Analyzed composition (mass %)	Density (Mg/m ³)
Al-2Mn	Al-2.02Mn	2.72
Al-4Fe	Al-4.34Fe	2.78
Al-4Fe-4Ni	Al-4.29Fe-4.06Ni	2.89
Al-4Fe-4Ni-5Mg	Al-3.94Fe-3.95Ni-4.59Mg	2.80

The alloy ingots were manufactured by means of common metallurgy method using pure Al and pure alloying components. RS procedure was carried out utilizing argon gas atomizing of the melt and subsequent quenching of the spray on a water-cooled rotating cooper roll. The preliminary consolidation of as received RS-flakes was performed using vacuum compression under a pressure 100 MPa to produce cylindrical billets of 45 mm in diameter. The billets were then hot extruded at 400 °C with a cross-section reduction of $\lambda = 19$. Finally, extruded rods of 7 mm in diameter were received. For comparison purposes material counterparts prepared by conventional techniques were studied as well. Samples with dimensions 8 mm in length and 6 mm in diameter were machined from the as-extruded rods and used for compression tests. Compression tests, within the temperature range of 25 °C - 550 °C, were carried out at constant true strain rate $5 \cdot 10^{-3}$ (s⁻¹) using modified Instron testing machine. Flaked graphite was used to reduce the friction between the sample and anvils during compression testing. The sample was water-quenched within 1 - 3 s after completion of the deformation at $\epsilon_t \approx 0.4$. Hitachi SU-70 scanning electron microscopy (SEM) equipped with SE, BSE and TE detectors was used for microstructure observations. Samples for SEM observations were prepared using standard metallography methods, i.e.: mechanical grinding and polishing with diamond suspensions. Thin foils for SEM-TE (Transmitted Electrons) observations were finally polished by means of electrochemical method and Struers TENUPOL polishing system.

3. RESULTS AND DISCUSSION

Preliminary microstructural investigations of rapidly solidified Al-TM alloys revealed similar features of their microstructure. Commonly known attribute of RS procedure is related to effective suppression of the intermetallic grains growth in comparison to coarse grained industrial materials. However, it was found that the effect of particles size reduction is more pronounced for highly concentrated alloys (e.g. Al-4Fe-4Ni) than that for materials with low TM content (e.g. Al-2Mn). Therefore, for the purpose of this paper, experimental results related to significantly varied microstructural features will be presented. Typical microstructure of as extruded Al-2Mn and Al-4Fe-4Ni alloys, observed at low magnifications are shown in **Fig. 1**. One can conclude that the microstructure of both Al-2Mn IM (**Fig. 1a**) and Al-2Mn RS (**Fig. 1b**) alloy is very similar, wherein evidently larger intermetallic particles are observed in industrial material (**Fig. 1a**).

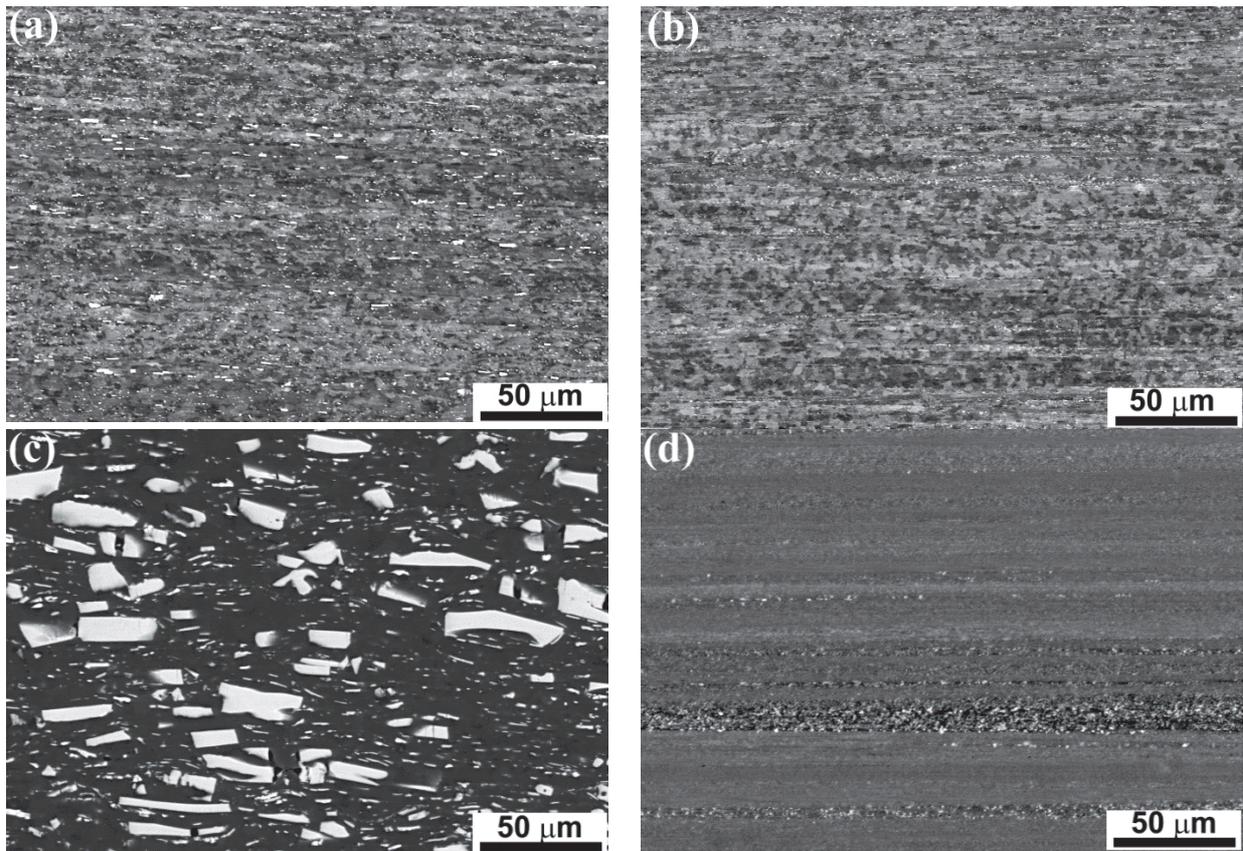


Fig. 1 SEM microstructure of (a, b) Al-2Mn, (c, d) Al-4Fe-4Ni alloys produced by (a, c) conventional IM method and (b, d) rapid solidification technique

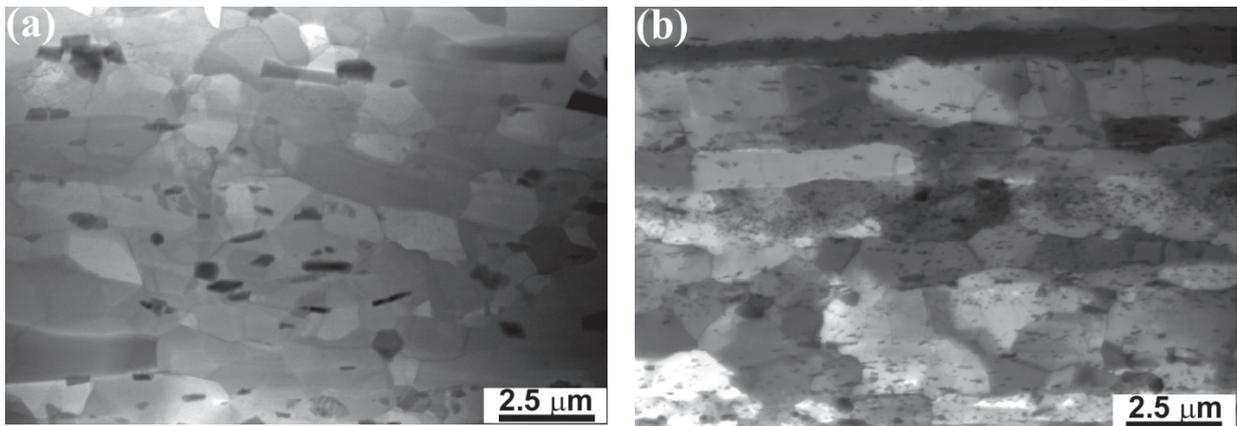


Fig. 2 STEM microstructure of as extruded (a) IM Al-2Mn and (b) RS Al-2Mn alloys

On the other hand, significant improvement in the reduction of primary intermetallic compound size for Al-4Fe-4Ni alloys is observed, as shown in **Fig. 1c** and **Fig. 1d**. Some locally observed differences in the particles' morphology observed in **Fig. 1d** correspond to the different solidification rate of a liquid-metal which are varied in their size; i.e. the coarser drop is formed the slower solidification rate and coarser particles in individual flake are grown. It is worth to note that the grain size of both industrial and rapidly solidified Al-2Mn alloys is very similar as evidenced in **Fig. 1a, b** and **Fig. 2**. It is known that Mn addition is often used in metallurgy practice to reduce the grain coarsening in commercial aluminum alloys. As so, the conclusion on similar grain size for IM and RS Al-2Mn alloys seems to be acceptable. Opposite effect was observed for alloys with Fe and Ni

additions; relatively coarse-grained microstructure has been usually observed for IM materials, while RS alloys exhibit enhanced effect of the grain size refinement [10].

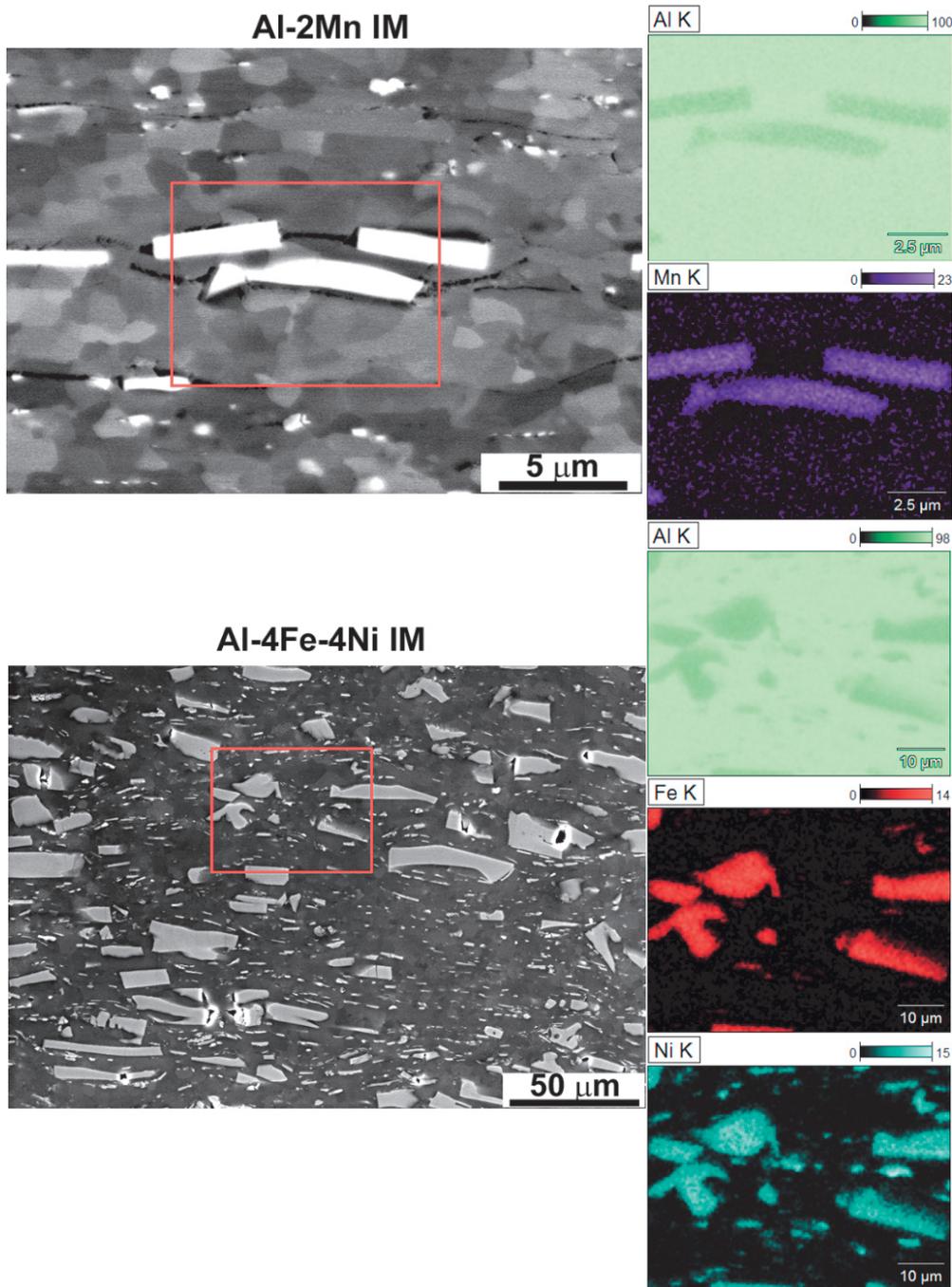


Fig. 3 Element map distribution for as extruded Al-2Mn IM and Al-4Fe-4Ni IM alloys

Preliminary identification of particles composition in tested alloys was performed by means of SEM/EDS analysis. Distribution of elements shown in **Fig. 3** lead to the conclusion that the intermetallic phases observed in the microstructure of Al-2Mn consist of aluminum and manganese, while particles observed in the microstructure of Al-4Fe-4Ni were found to be ternary compounds, which consist of aluminum, iron and nickel.

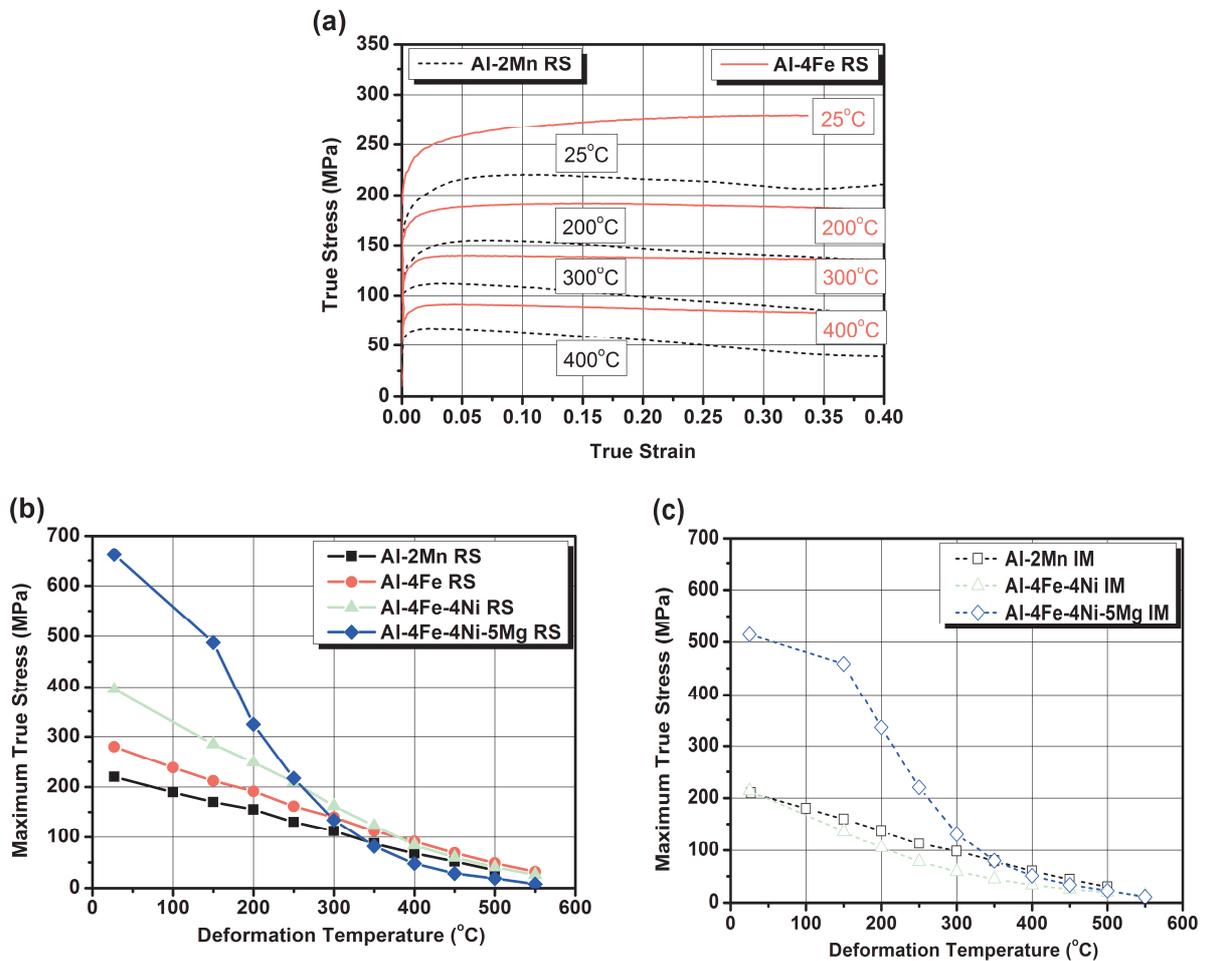


Fig. 4 (a) Examples of true stress-true strain curves for rapidly solidified Al-2Mn and Al-4Fe samples. Deformation temperature is marked on the figure. Effect of deformation temperature on the maximum true stress for: (a) rapidly solidified Al-TM (TM: Mn, Ni, Fe) alloys and (b) its ingot metallurgy counterparts

Hot compression tests on as extruded materials were performed at constant strain rate of $5 \cdot 10^{-3} \text{ s}^{-1}$ within temperature range of 20 - 550 °C. Representative stress-strain curves, which are typical in their shape for all tested materials are shown in **Fig. 4a**. True stress-true strain characteristics observed at high deformation temperatures is typical for aluminum alloys undergoing dynamic recovery [11]. Maximum flow stress value depends on deformation temperature as shown in **Figs. 4a, 4b**. Combination of rapid solidification with hot extrusion procedures results in increased flow stress value with respect to IM materials (**Figs. 4a, 4b**). The effect of enhanced mechanical properties is more pronounced for the materials with relatively high concentrations of transition metals, i.e. Al-4Fe, Al-4Fe-4Ni alloys. One may expect further improvement of mechanical properties if addition of Mn content increases. Addition of Mg was found to intensify the strengthening effect of tested material, particularly at low and intermediate temperatures (**Figs. 4a, 4b**). This result suggests that rapid solidification combined with solid solution hardening due to Mg leads to significant improvement of mechanical properties.

4. CONCLUSIONS

- Rapid solidification combined with hot extrusion was found to be very effective method for refining structural components, particularly for the alloys containing high concentrations of transition metals.

- The most effective strengthening of Al-TM alloys occurs if the microstructure refinement is combined with solid solution hardening attained due to Mg addition.
- Addition of Mn efficiently reduces the grain size in both IM and RS Al-2Mn alloys.

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REFERENCES

- [1] CHOWDHURY A.J.S., SHEPPARD T. Characteristics of an Al-7Fe-2Mo alloy prepared from RS powders. Key Engineering Materials, Vol. 38-39, 1990, pp. 263-276.
- [2] SKINNER D.J., OKAZAKI K. High strength Al-Fe-V at elevated temperatures produced by rapid quenching from melt. Scripta Metallurgica, Vol. 18, 1984, pp. 905-909.
- [3] HARIPRASAD S, SASTRY M.L., JERINA K.L. Deformation behavior of rapidly solidified fine grained Al-8.5%Fe-1.2%V-1.7%Si alloy. Acta Materialia, Vol. 44, 1996, pp. 383-389.
- [4] SUGAMATA M., KANEKO J., KIMURA N. Structure and properties of rapidly solidified P/M samples of Al-Mn-Cr alloys. Materials Science Forum, Vol. 416-418, 2003, pp. 359-368.
- [5] OKAZAKI K., SKINNER D.J. Al-Fe-Zr alloys for high temperature applications produced by rapid quenching from the melt. Scripta Metallurgica, Vol. 18, 1984, pp. 911-916.
- [6] TOKARSKI T. Thermo-mechanical processing of rapidly solidified 5083 aluminum alloy - structure and mechanical properties. Archives of Metallurgy and Materials, Vol. 60, 2015, pp. 177-180.
- [7] TOKARSKI T. The effect of plastic consolidation parameters on the microstructure and mechanical properties of various aluminum powders. Materials Science Forum, Vol. 674, 2011, pp. 141-146.
- [8] KULA A., BLAZ L., SUGAMATA M. Microstructure and mechanical properties of rapidly solidified Al-Fe-Ni-Mg alloys. Materials Science Forum, Vol. 674, 2011, pp. 165-170.
- [9] KULA A., BLAZ L., KUSPER P., SUGAMATA M., Mechanical and structural characterization of rapidly solidified Al-Fe-Mg alloys. Solid State Phenomena, Vol. 231, 2015, pp. 11-18.
- [10] KULA A., BLAZ L., SUGAMATA M. An analysis of the microstructure and mechanical properties of rapidly solidified Al-1Fe-1Ni-5Mg alloy. Key Engineering Materials, Vol. 641, 2015, pp. 3-9.
- [11] MCQUEEN H., SPIGARELLI S., KASSNER M.E., EVANGELISTA E. Hot deformation and processing of aluminum alloys, 2011, CRC Press, Taylor & Francis Group, LLC, New York.