

**Łukasz Wzorek, Marcel Wiewióra, Mateusz Wędrychowicz, Tomasz Skrzekut,
Piotr Noga, Jakub Wiewióra, Wojciech Sajdak, Maria Richert**

Effect of rapid solidification aluminum alloys with different Si contents on mechanical properties and microstructure

Wpływ szybkiej krystalizacji stopów aluminium
z różną zawartością Si
na własności mechaniczne oraz mikrostrukturę

Abstract

Rapid solidification is a relatively new and effective way of producing ultrafine-grained UFG aluminum alloys with enhanced mechanical properties. Due to a significant cooling rate close to nearly 10^6 K/s, it is possible to obtain a material with grain size far below 100 nm. In the present study, RS aluminum alloys with an Si content in a range of 5–10 wt% were produced during melt spinning. As a result, materials in the form of ribbons were produced. The as-received flakes were subjected to cold pressing into cylindrical billets with a diameter of 40 mm. Hot extrusion of pre-compacted material was subsequently performed at a temperature of 450°C with a press ram speed of 3 mm/s and extrusion ratio of $\lambda = 25$. In this work, the influence of brittle phases on the mechanical properties of as-extruded rods will be examined. Both tensile and microhardness tests were performed in order to determine the mechanical properties of the obtained profiles. It has been shown that brittle-phase refinement during melt spinning significantly influences the mechanical properties of the tested materials.

Keywords: solid bonding, rapid solidification, hot extrusion

Streszczenie

W wyniku szybkiej krystalizacji możliwe jest otrzymanie struktury drobnziarnistej stopów aluminium o podwyższonych własnościach mechanicznych. Znaczna szybkość chłodzenia na poziomie 10^6 K/s umożliwia otrzymanie wielkości ziarna na poziomie 100 nm. W prezentowanych w pracy

Łukasz Wzorek Ph.D. Eng., Marcel Wiewióra M.Sc. Eng., Mateusz Wędrychowicz M.Sc. Eng., Tomasz Skrzekut M.Sc. Eng., Piotr Noga M.Sc. Eng., Jakub Wiewióra M.Sc. Eng., Wojciech Sajdak M.Sc. Eng., Maria Richert D.Sc. Ph.D. Eng.: AGH University of Science and Technology, Faculty of Non Ferrous Metals, Krakow, Poland; marcelw@agh.edu.pl

badaniach szybkiej krystalizacji poddano stopy aluminium o zawartości 5 oraz 10% wagowych Si. Otrzymane taśmy poddano wstępnemu prasowaniu na zimno do form brykietów o średnicy 40 mm. Proces wyciskania przeprowadzono w temperaturze 450°C z prędkością tłoka równą 3 mm/s. Stopień przerobu wynosił $\lambda = 25$. W niniejszym opracowaniu badano wpływ kruchych faz na własności mechaniczne wyciskanych prętów. W tym celu wykonano próbę jednoosiowego rozciągania oraz pomiary mikrotwardości otrzymanych profili. Wykazano znaczny wpływ rozdrobnienia kruchych faz podczas szybkiej krystalizacji na własności mechaniczne badanych materiałów.

Słowa kluczowe: konsolidacja plastyczna, szybka krystalizacja, wyciskanie na gorąco

1. Introduction

Al-Si alloys are widely used in the modern automotive industry to produce engine blocks and their parts [1, 2], and this is mainly due to their good castability, high wear resistance, low thermal expansion coefficient, and preferable specific strength [3]. Conventional casting of Al-Si materials leads to the formation of polygonal, star-like, or oblong eutectic silicon phases whose size has a significant impact on mechanical properties [4]. Therefore, to improve these properties, additional refinement of silicon particles has to be applied, using chemical modifiers such as Sb and Sr or, alternatively, using mechanical methods such as ECAP [5], spray forming, or rapid solidification. The aim of this study is to present the effect of silicon particle refinement obtained by the rapid solidification (RS) technique on the mechanical properties of materials with different Si contents. Compared to alloys cast by conventional means, alloys produced by RS exhibit a higher Si refinement ratio, increased chemical homogeneity, enhanced supersaturation with alloying elements, and improved mechanical properties [6–9]. To make use of products from the melt spinning process (i.e., ribbons, possible in any industrial application), their plastic consolidation has to be performed. In this process, the ribbons pre-compacted into the form of small billets are extruded at an elevated temperature [5, 10]. The experimental results presented in this study have proven that a high cooling rate during RS has a significant effect on both the structure and mechanical properties of the examined materials.

2. Experimental procedure

Binary Al-Si (5, 10 wt%) alloys were prepared by adding pure Si to the AA1050 aluminum melt obtained at 850°C by induction heating. The chemical composition of the AA1050 alloy is given in Table 1. Afterwards, the obtained materials were subjected to the process of melt spinning performed with the use of a copper wheel (14.8 cm in diameter), which rotated at a speed of 2800 rev/min. The melting of the aluminum alloy was carried out at a temperature of 850°C in a graphite chamber in an argon atmosphere. The obtained

ribbons (Fig. 1) were pre-compacted at room temperature under a pressure of 240 MPa into a form of round (40 mm diameter, 10 mm thick) briquettes with a total weight of 25 g. As a reference material, an AA1050 (CM 1050) solid alloy with the same addition of Si (CM 5%Si, CM 10%Si) and alloy were cast in a steel die. After the pre-compacting process, the billets were preheated at 450°C for 20 minutes in a press container. This operation provided uniform temperature distribution inside the material. The billet weighing 200 g was extruded at a temperature of 450°C with a ram speed of 3 mm/s and extrusion ratio of 25. As a result of this operation, a profile with a round cross-section of 8 mm diameter was obtained. Tensile test samples with a gauge length of 25 mm and a diameter of 5 mm were machined from the extruded profiles. Testing of the mechanical properties was performed on a Zwick/RoellZ050 machine at room temperature and with a constant strain rate of $8 \cdot 10^{-3} \text{ s}^{-1}$. In order to evaluate the size of the Si particles after extrusion, microstructure examinations were performed under a Hitachi SU-70 SEM. Moreover, microhardness tests were made using a Shimadzu HMV-G hardness tester and a load of 0.9807 N (HV 0.1). Samples for microstructural examinations were prepared by grinding and polishing the cross-sections of the extruded materials.

Table 1. Chemical composition of AA1050

Element	Al	Cu	Fe	Mg	Mn	Si	Ti	V	Zn	Balance
weight %	99.5	0–0.05	0–0.4	0–0.05	0–0.05	0–0.25	0–0.03	0–0.05	0–0.05	0.03

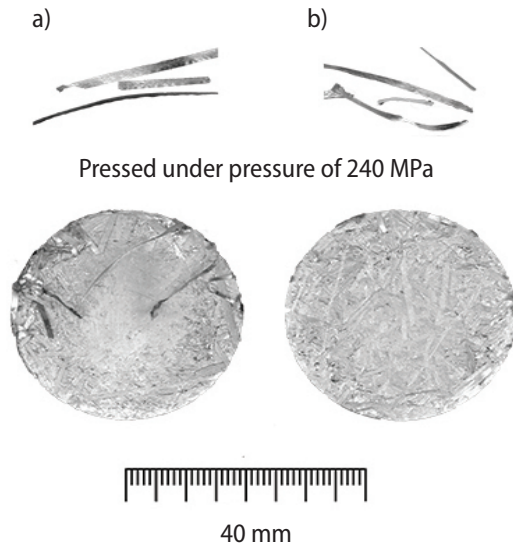


Fig. 1. Images of ribbons with Si content: a) RS 5%Si; b) RS 10% Si – before and after pre-compaction

3. Surface of as-extruded rods

Figure 2 shows the surface quality of as-extruded rods with different silicon content obtained from rapid solidification ribbons (RS) and cast material (CM). As can be observed, all rods show good overall surface quality with no visible defects such as cracks or pores. Smooth and glossy surfaces were observed along the entire length of the profiles regardless of Si content. Still, it has to be pointed out that good surface quality in each profile does not necessarily mean the sound bonding of chips inside the material. To determine the latter parameter, additional microstructure observations and tensile tests were performed.

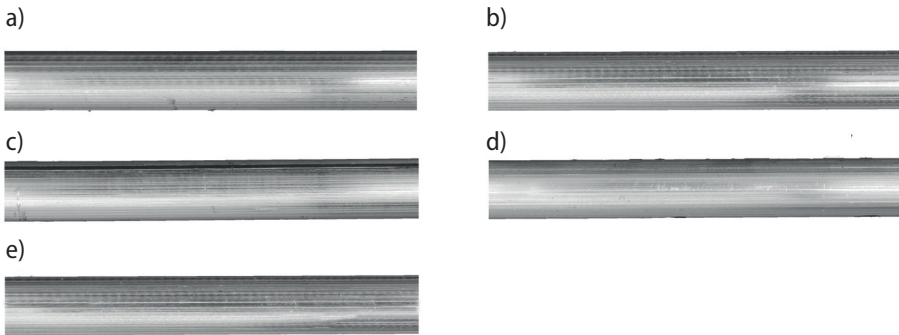


Fig. 2. Surface quality of as-extruded rods: a) RS 5%Si; b) CM 5%Si; c) RS 10%Si; d) CM 10 % Si; e) CM 1050

4. Microstructure observations

Figure 3 shows the microstructure of an extruded profile cross section. Rapid cooling of the molten metal lead to the significant refinement of Si particles, much higher than in the traditional process [6, 11]. Additionally, the cast materials featured a notable amount of porosity detected on the profile cross-sections (Fig. 3a, c). Compared to the reference materials, the microstructure of as-extruded aluminum rods after rapid solidification (designated as RS 5%Si and RS 10%Si) was characterized by a high ratio of silicon particle refinement and the presence of intermetallic phases (Fig. 3c, d). They had spherical and regular morphologies without cracks and pores. Some researchers suggest that the arrested growth of silicon particles is the effect of limited diffusion caused by rapid solidification [4]. The high cooling rate also increases the refinement ratio of the intermetallic phases [12]. Cast materials show the presence of plate particles of the intermetallic phases (Fig. 3a, c). In RS materials, these phases are much finer and have a spherical or rounded shape with a diameter of ~ 300 nm (Fig. 3b, d). Using an EDS (Energy Dispersive Spectroscopy) detector and literature data [12, 13], these phases were identified as an

Al-Si-Fe compound (Fig. 4). The average diameter of the intermetallic phases in the CM 5%Si and CM 10%Si materials where the cooling rate was approx. 10^1 – 10^2 K/s was equal to 1.88 μm and 1.98 μm , respectively. In contrast, in the rapidly solidified alloys cooled at a rate of approx. 10^5 – 10^6 K/s, the average diameter was 250 nm for the RS 5%Si alloy and 280 nm for the RS 10%Si alloy.

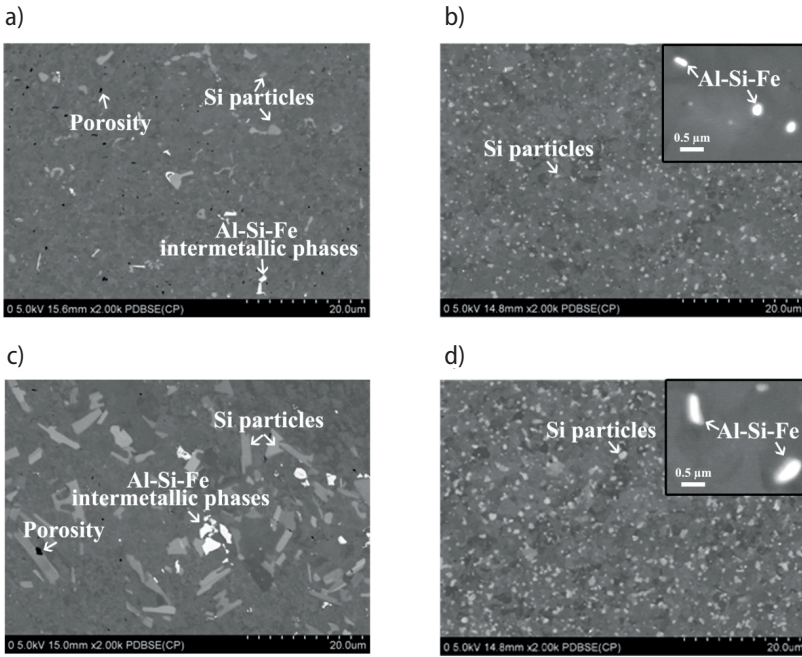


Fig. 3. Microstructure of as-extruded rods- cross section: a) CM 5%Si; b) RS 5%Si; c) CM 10%Si; d) RS 10%Si

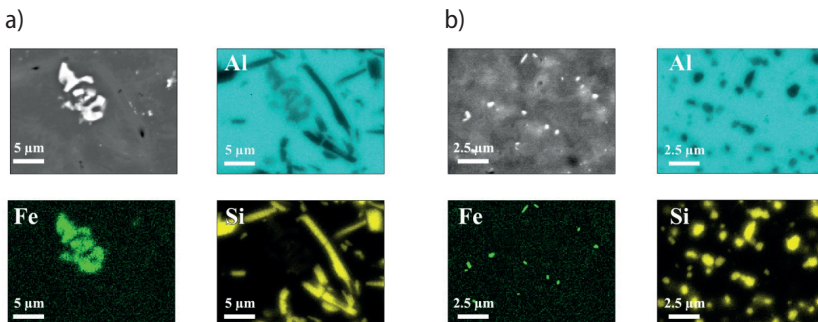


Fig. 4. The results of EDS analysis a) CM 10%Si; b) RS 10%Si

5. Mechanical properties

Two separate plots are shown in Figure 5 (Engineering stress-strain curves) and Figure 6 (Microhardness of all tested materials). From these plots, it follows that mechanical properties such as YS, UTS, and microhardness increase with a higher Si content, but at the cost of decreasing plasticity (Tab. 2). Elongation of the CM 10%Si alloy reached only 11%. When compared to the reference material designated as CM 1050, the significant decrease of plasticity can be attributed to the high content of Si and presence of intermetallic particles (Fig. 3c). After conventional casting, the particles of primary and eutectic silicon and of the intermetallic Al-Si-Fe phases were characterized by an irregular and oblong needle-like morphology (Fig. 3a, c) [4]. This shape had a negative influence on the mechanical properties of the extruded rods. Based on the results of a uniaxial tensile test and microhardness measurements, it can be concluded that changes in the distribution of phases and their morphology resulting from RS are directly reflected in the increase of mechanical properties [9, 15, 16]. The results presented in Figure 5 suggest that alloys made by rapid solidification preserve their high strength and plasticity. Changes in Vickers microhardness depending on the solidification mode are shown in Figure 6. It was observed that microhardness values of the CM 5%Si alloy cast by the traditional process and of the rapidly solidified RS 5%Si alloy were at similar levels. A microhardness increase of ~30% was observed in the RS 10% Si alloy. The comparison was made with an alloy of the same chemical composition but having undergone the conventional solidification process (CM 10% Si).

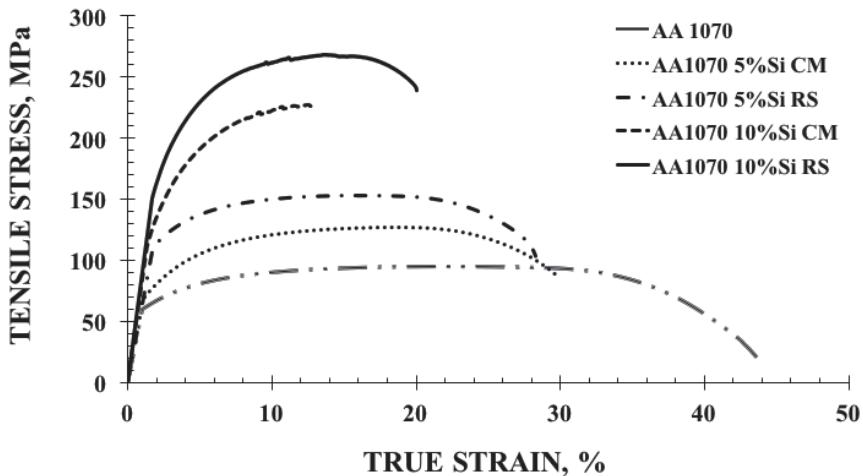


Fig. 5. Engineering stress-strain curves

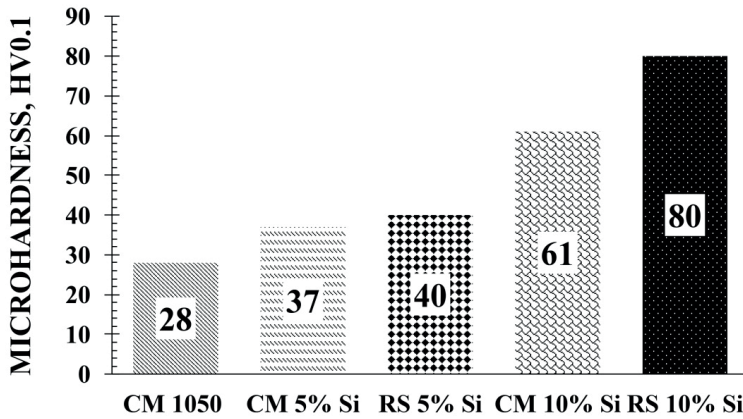


Fig. 6. Microhardness for all tested materials

Table 2. Mechanical properties of obtained rods

	YS [MPa]	UTS [MPa]	ELONG. [%]	HV 0.1
CM 1050	60	95	44	28
CM 5% Si	80	130	30	37
RS 5% Si	120	155	28	40
CM 10% Si	130	230	11	61
RS 10% Si	170	270	19	80

6. Conclusions

1. High Si content in the extruded material raises the YS, UTS, and microhardness with a simultaneous drop of plasticity when compared to materials with the same Si content obtained by conventional technology.
2. Silicon particles after RS are characterized by a regular, spherical shape without eutectic crystals or large intermetallic particles.
3. The rapidly solidified RS 10%Si alloy has levels of microhardness, YS, and UTS nearly 3 times higher as compared to the reference material (which is the CM AA1050 alloy obtained by conventional technology).
4. Rapidly solidified alloys after plastic consolidation are free of any visible porosity.
5. The rapid solidification of materials containing more than 5% Si preserves their high mechanical and plastic properties when compared to materials extruded from solid billets.
6. In materials obtained by the process of rapid solidification and hot-extrusion, the intermetallic phases of Al-Si-Fe had an average diameter of ~300 nm.

References

- [1] Ye H.: An Overview of the Development of Al-Si-Alloy Based Material for Engine Applications. *Journal of Materials Engineering and Performance*, 12, 3 (2003), 288–297
- [2] Srivastava V.C., Mandal R.K., Ojha S.N., Venkateswarlu K.: Microstructural modifications induced during spray deposition of Al-Si-Fe alloys and their mechanical properties. *Materials Science and Engineering A*, 471, 1–2, (2007), 38–49
- [3] Todeschini P., Champier G., Samuel F.H.: Production of Al-(12-25) wt % Si alloys by rapid solidification: melt-spinning versus centrifugal atomization. *Journal of Materials Science*, 27 (1992), 3539–3551
- [4] Xu C.L., Wang H.Y., Qiu F., Yang Y.F., Jiang Q.C.: Cooling rate and microstructure of rapidly solidified Al-20 wt% Si alloy. *Materials Science Engineering A*, 417, 1–2 (2006), 275–280
- [5] Misiolek W.Z., Haase M., Khalifa N.B., Tekkaya A.E., Kleiner M.: High quality extrudates from aluminum chips by new billet compaction and deformation routes. *CIRP Annals – Manufacturing Technology*, 61, 1 (2012), 239–242
- [6] Van Rooyen M., Colijn P.F., de Keijser T.H., Mittemeijer E.J.: Morphology and mechanical properties of melt-spun and conventionally cast aluminium, AlMg and AlSi alloys before and after hot extrusion. *Journal of Materials Science*, 21 (1986), 2373–2384
- [7] Barekar N.S., Hari Babu N., Dhindaw B.K., Fan Z.: Effect of intensive shearing on morphology of primary silicon and properties of hypereutectic Al-Si alloy. *Materials Science and Technology*, 26, 8 (2010), 975–980
- [8] Sheng S.D., Chen D., Chen Z.H.: Effects of Si addition on microstructure and mechanical properties of RS/PM (rapid solidification and powder metallurgy) AZ91 alloy. *Journal of Alloys and Compounds*, 470, 1–2 (2009), 17–20
- [9] Tawfik N.L.: Mechanical properties of rapidly solidified ribbons of some Al-Si based alloys. *Journal of Materials Science*, 32, 11 (1997), 2997–3000
- [10] Haase M., Ben Khalifa N., Tekkaya A.E., Misiolek W.Z.: Improving mechanical properties of chip-based aluminum extrudates by integrated extrusion and equal channel angular pressing (iECAP). *Materials Science and Engineering A*, 539 (2012), 194–204
- [11] Nikanorov S.P., Volkov M.P., Gurin V.N., Burenkov Y.A., Derkachenko L.I., Kardashev B.K., Regel L.L., Wilcox W.R.: Structural and mechanical properties of Al-Si alloys obtained by fast cooling of a levitated melt. *Materials Science and Engineering A*, 390, 1–2 (2005), 63–69
- [12] Zhang Y., Liu Y., Han Y., Wei C., Gao Z.: The role of cooling rate in the microstructure of Al-Fe-Si alloy with high Fe and Si contents. *Journal of Alloys and Compounds*, 473, 1–2 (2009), 442–445
- [13] Dutta B., Rettenmayr M.: Effect of cooling rate on the solidification behaviour of Al-Fe-Si alloys. *Materials Science and Engineering A*, 283 (2000), 218–224
- [14] Griger A., Stefaniay V.: Equilibrium and non-equilibrium intermetallic phases in Al-Fe and Al-Fe-Si alloys. *Journal of Materials Science*, 31, 24 (1996), 6645–6652
- [15] Gutierrez-Urrutia I., Munoz-Morris M.A., Morris D.G.: Contribution of microstructural parameters to strengthening in an ultrafine-grained Al-7% Si alloy processed by severe deformation. *Acta Materialia*, 55, 4 (2007), 1319–1330
- [16] Youan-Youan L., Da-Tong Z., Leo N.T., Wei-Wen Z.: Rapidly solidified hypereutectic Al-Si alloys prepared by powder hot extrusion. *Transactions of Nonferrous Metals Society of China*, 12 (2002), 878–881