Archives of Materials Science and Engineering

Volume 49
Issue 2
June 2011
Pages 97-102

International Scientific Journal published monthly by the World Academy of Materials and Manufacturing Engineering

Effect of rapid solidification on structure and mechanical properties of Al-6Mn-3Mg alloy

P. Lobry [a], L. Błażż [a,*], M. Sugamata [b], A. Kula [a]

[a] AGH-University of Science and Technology, Al. Mickiewicza 30, 30-059 Kraków, Poland
[b] Department of Mechanical Engineering, College of Industrial Technology, Nihon University, 1-2-1 Izumi-cho, Narashino-shi, Chiba 275, Japan
* Corresponding author: E-mail address: blaz@uci.agh.edu.pl

Received 20.03.2011; published in revised form 01.06.2011

ABSTRACT

Purpose: Experiments on rapidly solidified (RS) and industrially manufactured (IM) Al-6Mn-3Mg alloy were performed to test the effect of RS on the structure and mechanical properties of the material.

Design/methodology/approach: Annealing of as-extruded RS and IM samples was performed at 500°C in order to test the stability of structural components and related hardness of the materials. Mechanical properties of as-extruded RS and IM materials as well as the samples preliminarily annealed at 500°C / 6 h were tested by compression at 20°C-500°C. Structural investigations were performed using analytical transmission electron microscopy techniques.

Findings: The flow stress for RS-samples was found to be about 240 MPa higher at 293 K than that for IM material. However, the difference between the flow stress values observed for RS and IM samples was remarkably reduced at higher deformation temperatures. Annealing at 500°C was found to result in reduction of the RS-material hardness due to the recovery process and slightly marked coarsening of Al_6Mn particles. The particles observed in both as extruded and RS-samples annealed 500°C / 7 days were at least 10-times smaller than that for the industrial material.

Practical implications: Due to refined structure and the negligible particle coarsening at high annealing temperatures, the products made from RS Al-6Mn-3Mg alloy can be used at high service temperature applications.

Originality/value: Hardening of non-heat-treatable Al-6Mn-3Mg alloy is possible due to effective refining of Al_6Mn particles using the rapid solidification and adequate consolidation procedure of RS-powders. The highest flow stress was observed for RS samples tested at room temperature. However, increasing deformation temperature was found to result in reduced difference between the flow stress values received for RS and IM materials.

Keywords: Rapid solidification; Al-Mn-Mg alloy; Hot deformation; Formability; Al_6Mn particle fracture

Reference to this paper should be given in the following way:

P. Lobry, L. Błaż, M. Sugamata, A. Kula, Effect of rapid solidification on structure and mechanical properties of Al-6Mn-3Mg alloy, Archives of Materials Science and Engineering 49/2 (2011) 97-102.

PROPERTIES

1. Introduction

Commonly known methods for strengthening of aluminum alloys are generally based on the grain size refining and precipitation hardening. More advanced methods including mechanical alloying (MA) and powder metallurgy (PM) procedures are also reported to be very successful technique for effective refining of structural components and strengthening of alloys [1-4].

However, the methods are definitely more expensive than the rapid solidification (RS) of alloys, which consume less time at relatively low production costs. Rapid solidification provides effective grain refining accompanied with an oversaturation of solid solution and refining of structural constituents [5-8]. Moreover, the rapid solidification usually result in the development of transition structures of fine particles, which are formed at RS alloys Zn–Mg–Sc, Cu–Ga–Mg–Sc, Zn–Mg–Yb and Zn–Mg–Ti as well as aluminum alloys containing alloying elements such as Cu, Mg, Mn, Fe, Cr, Zn, Pd and other transition metals [9-12]. Quasicrystalline structure of particles in RS Al-Mn alloys was extensively studied since Shetchman et al. published in 1984 crystallographic details of icosahedral and decagonal structures [13]. It was found that the particles morphology and their crystallographic structure differ from typical constituent phases observed in aluminum alloys produced by means of industrial metallurgy methods (IM).

Rapid solidification is an effective method for the particles refining in aluminum alloys, which cannot be hardened due to conventional heat treatment and ageing procedures. It is commonly believed that both the solidification rate and chemical composition of the alloy substantially affect the particles morphology and their structure. However, following consolidation of RS powders by means of the hot compression and hot extrusion procedures may transform the particles structure and their distribution that finally affect the bulk material properties. Therefore, experiments on the rapid solidification of Al-Mg-Mn alloy, combined with mechanical consolidation of RS powders, were performed. Structure and mechanical properties of the bulk RS-material were analyzed with respect to the properties of IM material in order to test the effect of the production method on the structure and mechanical properties of received materials.

2. Material

Experiments were performed on aluminum alloy containing 5.71 wt% Mn and 2.82 wt% Mg named hereafter Al-6%Mn-3%Mg alloy. Tested materials were produced by means of a common metallurgical technology, i.e. industrial method (IM) and powder metallurgy method combined with rapid solidification of the material (RS). Hot extruded IM rods, 7 mm in diameter, were manufactured from as-cast material using cross-section reduction ratio of λ=19. Rapid solidification of the alloy was performed by means of a spray deposition of the melted alloy on water-cooled copper cylinder. High pressure nitrogen was used for the spray deposition to avoid the oxidation of the melt. Fine RS-flakes were compressed in a thin-wall AA6061 container of 40 mm in diameter, using 100-tonn press and following vacuum degassing at 400ºC. The RS-charge was extruded at the same conditions as used for IM material. The materials were produced in Nihon University laboratories in Tokyo – Japan, and delivered for further testing to the University of Science and Technology in Cracow – Poland.

Annealing tests at 500ºC were performed at 500ºC for 5min – 7 days on IM and RS samples. Hardness of the samples was measured by means of a micro-Vickers tester at an indenter load of 9.8 N. More than 10 measurements were performed to determine an average hardness of the sample.

Samples for compression tests, 10 mm in high and 6 mm in diameter, were machined from as-extruded rods. Compression tests at 20ºC-500ºC were performed using constant true strain rate $5 \cdot 10^{-3}$ s^{-1}. The tests were performed both for as-extruded samples and samples annealed at 500ºC / 6 h. Deformation finish at the compression was limited to the true strain of $\varepsilon_t \cong 0.4$ in order to avoid barreling of the sample. Flaky graphite was used to reduce the friction between the sample and anvils.

Samples for light microscopy and scanning microscopy observations were prepared using standard metallography methods and fine etching with 10% water solution of Tucker's reagent. Thin foils for TEM/STEM observations were prepared by means of mechanical grinding and final ion thinning on Gatan's PIPS-691 machine. Structure observations were performed using transmission electron microscope JEM 2010, equipped with the scanning transmission electron microscopy (STEM) device and the energy disperse X-ray analysis system (EDX) controlled by an INCA software.

3. Results and discussion

The structure of as-extruded industrial (IM) and rapid solidified (RS) Al-6Mn-3Mg samples observed by means of scanning electron microscopy is shown in Fig. 1a and Fig. 1b respectively. Relatively coarse particles observed in IM microstructure are typical for commonly observed structures of industrial Al-6Mn-3Mg products. The size of particles in IM material is varied within 10-100 μm. Large particles were fractured during the hot extrusion that resulted in the increase of the material porosity.

Rapid solidification results in efficient refining of the particles, which are 50-600 nm in size. Some variation of the particles morphology in the RS-material structure was observed on the cross-sectioned rod, as shown in Fig. 1b. The last effect is related to some differences of the solidification rate for particular RS-flakes that depend on the melt-drop size. Therefore, primary flakes can be relatively easily discriminated in a bulk RS-material structure. It is worth stressing that both SEM and following STEM/TEM observation did not reveal any noticeable fracturing of fine particles in RS material.

Typical structure of as extruded RS Al-6Mn-3Mg alloy, observed by means of STEM method, is shown in Fig. 2. Uniform distribution of fine Mn-rich particles in Al-Mg matrix is observed locally if the area that belongs to the single pre-existing flake. The material was well consolidated and the pores on the border between the flakes were not found.

Long-term annealing of RS material at 500ºC results in the particles coarsening. The particles size reached 1-3 μm after annealing at 500ºC / 7 days. As mentioned above, the particle size distribution is not uniform (see: Fig. 1b). Therefore, the parameters mentioned above were measured at the area containing the smallest particles observed in tested RS-samples. For comparison purposes, an initial structure of as-extruded RS material and the structure of the sample annealed at 500ºC / 7 days are shown in Fig. 3a and Fig. 3b respectively.

Detailed analysis of the material structure was performed by means of analytical transmission electron microscopy (TEM/EDX). For example, the chemical analysis of Mn-rich particle shown in Fig. 4 revealed Al:Mn:Mg atomic ratio to be 87.3 : 12.1 : 0.6 that is close to the stoichiometry of Al_6Mn phase i.e. 85.7 : 14.3 : 0. Some excess of both aluminium and magnesium at EDX analysis can be ascribed to the additional radiation of the neighbouring Al(Mg) matrix. Selected area diffraction pattern analysis (SAD) of the particle "A" (Fig. 4) confirmed typical structure of Al_6Mn phase, which is characterized by the following orthorhombic lattice parameters: a=0.75518 nm, b=0.64978 nm, c=0.88703 nm, space group *Cmcm* [8, 9].

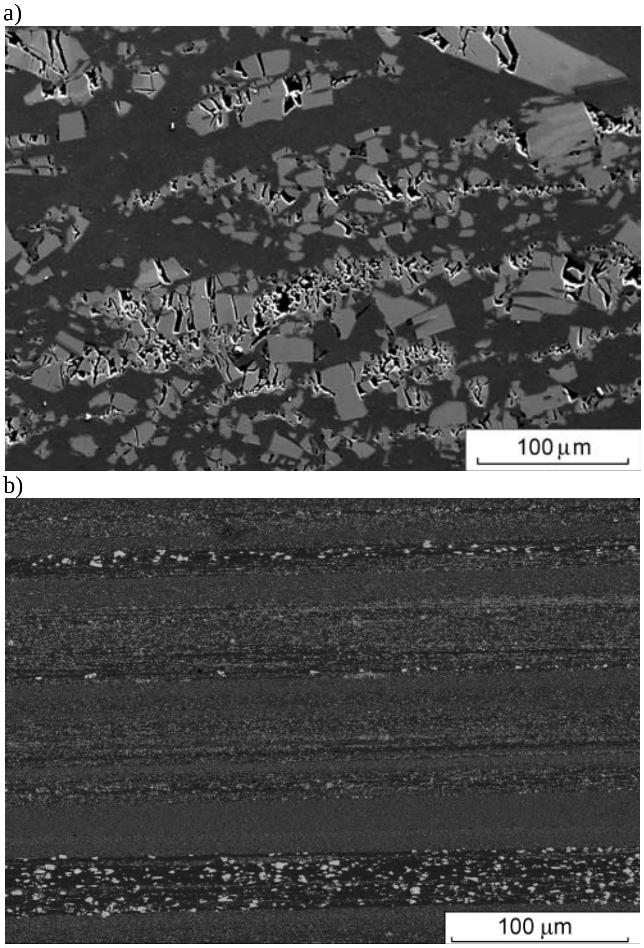
a)
b)


Fig. 1. Structure of as-extruded Al-6Mn-3Mg alloy: a) IM material; b) RS material (longitudinal cross-section; SEM)

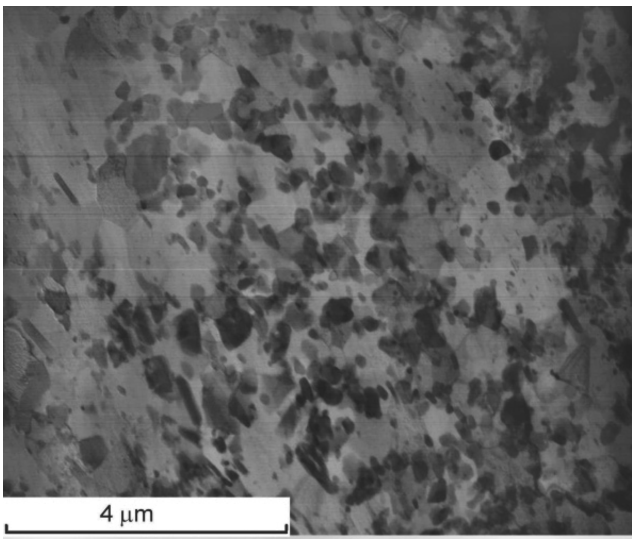


Fig. 2. Structure of as-extruded RS Al-6Mn-3Mg alloy (STEM)

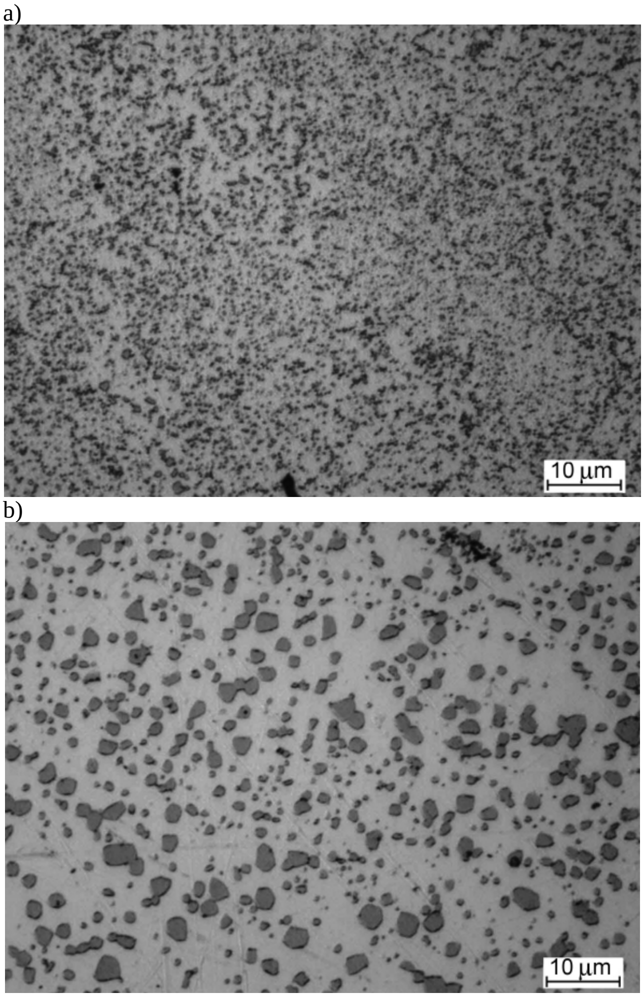
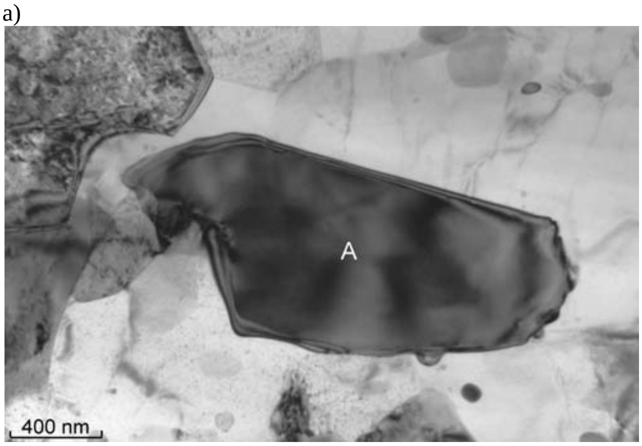
a)
b)


Fig. 3. Effect of annealing on the structure of RS Al-6Mn-3Mg alloy: a) as extruded material; b) sample annealed at 500°C / 7 days (light microscopy)

Similar SAD pattern analyses were performed for a number of fine particles in RS material, but any quasicrystalline structures were found. The development of quasicrystals having icosahedral and decagonal structures was reported for aluminium alloys containing some transition metals including manganese additions [8, 12]. However, the development of quasicrystalline structures of Mn-rich particles in RS Al-6Mn-3Mg material was not confirmed. It seems reasonable to conclude that the hot compression of RS-flakes and the following extrusion at 400ºC resulted in the transformation of transition phases into the stable Al_6Mn-type structure. Unfortunately, as-solidified flakes were not tested by TEM. Therefore, the development of expected metastable structures in Al-6Mn-3Mg alloy was not confirmed for the unprocessed RS-material.

Applicability of RS aluminium alloys to the manufacture of products designed for a high temperature service is considerably limited if the coarsening of structural components occurs and causes the deterioration of mechanical properties. Therefore, experiments on annealing of RS alloys at high temperature range are necessary to provide essential information on the stability of the material structure and the related stability of mechanical properties.

a)



b)

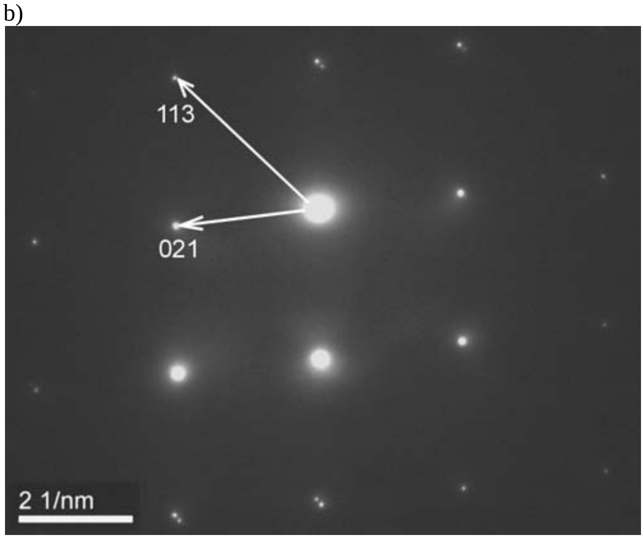


Fig. 4. Microstructure of as extruded RS Al-6Mn-3Mg alloy: a) TEM picture; b) diffraction pattern received from the particle "A", which correspond to the Al_6Mn-type structure (see: Tab. 1)

Table 1.
Calculated and measured values for interplanar distances $d_{(hkl)}$ and related angle between {hkl} planes marked in the SAD pattern displayed in Fig. 4b

Interplanar distances for $d_{(hkl)}$	Calculated for Al_6M	Measured from SAD pattern	Difference %
$d_{(021)}$	0.3474 nm	0.34 nm	2
$d_{(113)}$	0.2535 nm	0.25 nm	1.3
Angle between (021) and (113) planes, deg.:	49.9	49.0	1.7

The effect of annealing at 500ºC on the RS and IM material hardness is shown in Fig. 5. The highest hardness value was observed for as-extruded RS material. The hardness was reduced from 157 HV to 93 HV for as-extruded material and the sample annealed at 500ºC / 7 days, respectively. The hardness of as-extruded RS material was higher ~75% than that for IM material. Reduction of the hardness due to the annealing was relatively low for the IM material as the coarsening of large particles was practically negligible in comparison to the noticeable coarsening of highly refined particles in RS material. After prolonged annealing of IM material, the hardness value was reduced from 90 HV to 78 HV for as-extruded and annealed samples, respectively.

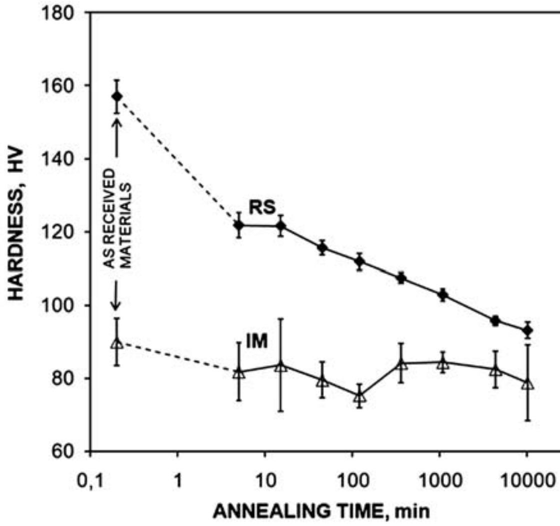


Fig. 5. Effect of annealing at 500^{o}C on the hardness of RS and IM Al-6Mn-3Mg alloy

Mechanical properties of Al-6Mn-3Mg alloy were tested by means of compression tests at 20ºC-500ºC performed at constant true strain rate. True stress vs. true strain curves received for as-extruded RS and IM materials are shown in Fig. 6a and Fig. 6b, respectively. Compression tests were also performed for RS and IM samples annealed at 500ºC / 6 h to test the annealing effect on the high-temperature flow stress characteristics. It is a common believe that an intense dynamic recovery at high enough deformation temperatures is responsible for the steady-state flow regime observed on σ_t-ε_t curves. Strain hardening at 20ºC-150ºC is not followed by the steady-state flow within the limited strain range used in experiment.

Maximum flow stress vs. deformation temperature is shown in Fig. 7. The flow stress is reduced with temperature because of the temperature effect and intensified dynamic recovery process. The maximum flow stress for RS material was reduced from 626 MPa to 18 MPa at 20ºC and 500ºC, respectively. It is worth stressing that the flow stress at 20ºC-150ºC was measured at $\varepsilon_t \cong 0.4$ i.e. before the steady state flow was reached. Therefore, the flow stress value was measured at $\varepsilon_t \cong 0.002$ for selected samples and the data for $\sigma_{0.002}$ were shown in Fig. 7 to display the effect of strain hardening during deformation at low temperatures. Relatively high flow stress values for both RS and IM materials were observed with respect to the results received for technical purity aluminium [14]. However, the difference between the flow stress for tested IM and RS samples becomes unexpectedly low at high deformation temperature range (250ºC-550ºC). The last effect suggests that the refinement of Al_6Mn particles for the RS-material result in very limited particles strengthening of the material processed at high deformation temperatures.

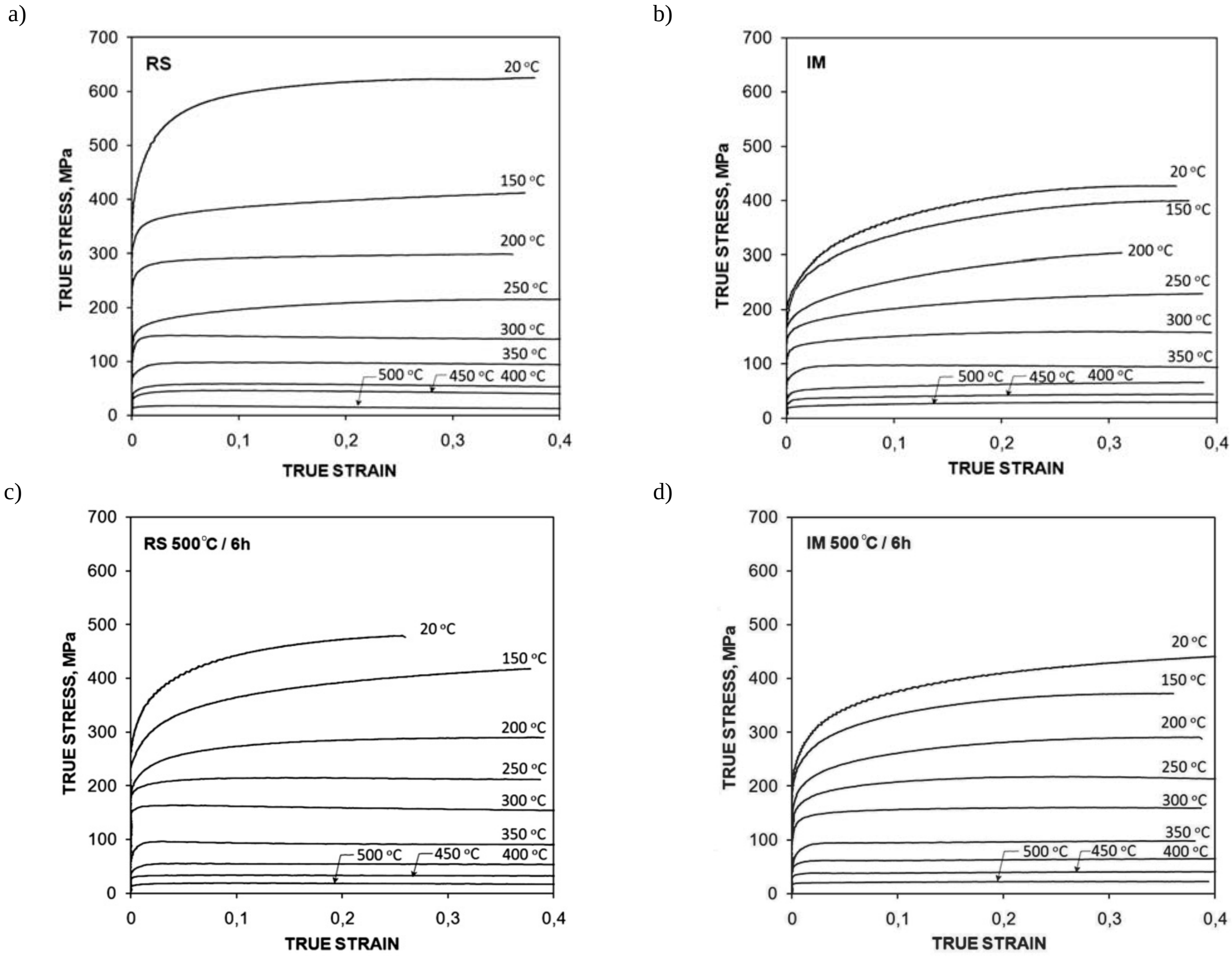


Fig. 6. Flow stress curves received for Al-6Mn-3Mg alloy: a) as-extruded RS material; b) as-extruded IM material; c) RS samples annealed at 500^{o}C / 6 h; d) IM samples annealed at 500^{o}C / 6 h

The hardness of hot deformed samples, measured at room temperature, is shown in Fig. 8. The results are more consistent with structural features of the hot deformed samples. Fine particles produced during rapid solidification of the alloy result in definitely higher hardness of RS material than that for IM or annealed RS and IM samples. The hardness of RS material was reduced from 180 HV to 122 HV for samples deformed at 20ºC and 500ºC, respectively. Adequate reference data for IM material were 115 HV and 94 HV for 20ºC and 500ºC respectively. Reduction of the materials hardening with increasing deformation temperature result from intensified dynamic recovery rather than the particle coarsening. Transmission electron microscopy observations did not reveal any noticeable coarsening of particles due to the hot deformation of the samples.

4. Conclusions

Rapid solidification combined with mechanical vacuum consolidation and hot extrusion of RS-flakes result in refined structure of Al-6Mn-3Mg alloy and effective reduction of the material porosity caused by the cracking of Al_6Mn particles. In contrary to RS material, an intense cracking of large particles was observed for IM material extruded at 400ºC.

High mechanical properties of RS alloy are ascribed to effective refinement of Al_6Mn particles. The annealing of as extruded RS material at 500ºC/6h was found to result in reduction of mechanical properties mostly because of the coarsening of Al_6Mn particles.

Both the flow stress at compression tests and the hardness of as deformed samples for RS material were higher than that for an industrial material (IM) if measured at room temperature. However, increased deformation temperature was found to result in reduced difference between the flow stress values for RS and IM samples.

Acknowledgements

Financial support under grant No 11.11.180.447 is kindly acknowledged. The work was presented as an oral presentation on Science Seminar: Integrated studies of fundamentals of metals plastic deformation, *PLASTMET*, 30.11.-3.12.2010, Łańcut, Poland.

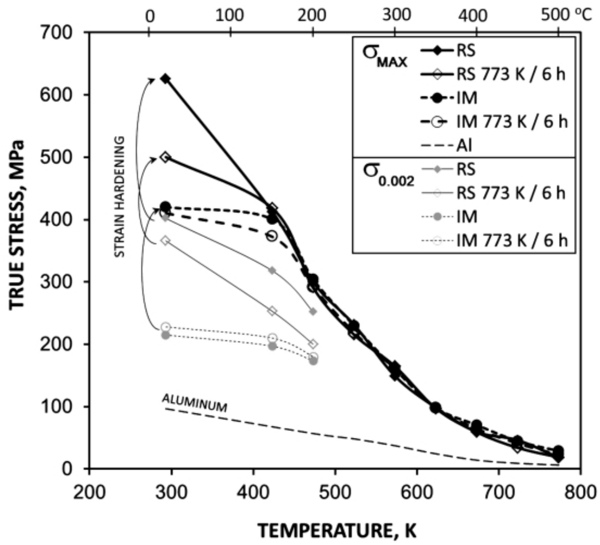


Fig. 7. Effect of deformation temperature on the flow stress maximum, σ_{MAX} (bold lines) and selected data of $\sigma_{0.002}$ (grey lines) are shown for RS and IM Al-6Mn-3Mg alloy. Results received for as extruded materials (solid lines) and samples annealed at 500ºC / 6 h (dotted lines) are shown. Adequate data for aluminum of technical purity are also displayed for comparison purposes [14]

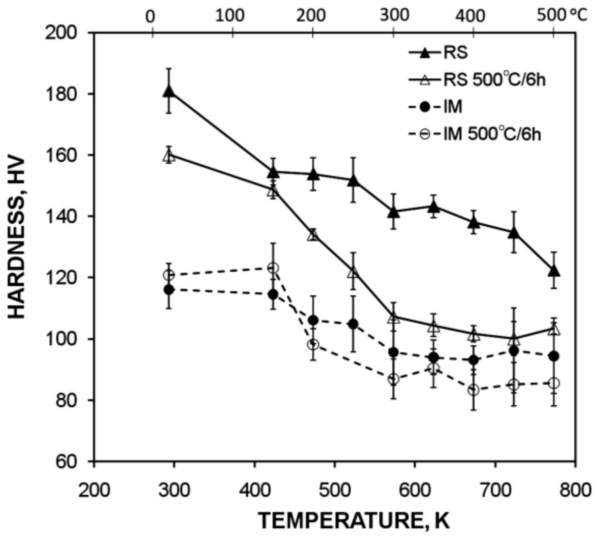


Fig. 8. Effect of deformation temperature on the hardness of as-deformed RS Al-6Mn-3Mg material and RS samples preliminarily annealed at 500ºC / 6 h (bold line). Results received for as extruded IM material and IM samples preliminarily annealed at 500ºC / 6 h are also shown (dotted line)

References

[1] G.E. Totten, D.S. Scott MacKenzie (Eds.), Physical Metallurgy and Processes in Handbook of Aluminum, vol. 1, Marcel Dekker Inc., New York, Basel, 2003.

[2] K. Seimiya, M. Sugamata, L. Blaz, J. Kaneko, Structures and properties of P/M materials of Al-Mg-oxide (GeO_2, SnO_2, PbO) systems processed by mechanical alloying, Journal of Japan Society of Powder and Powder Metallurgy 53 (2006) 899-908.

[3] J. Kaneko, M. Sugamata, L. Blaz, R. Kamei, Aluminum Based Materials Containing Low Melting and High Melting Metals Produced by Mechanical Alloying with Addition of Metal Oxides, Materials Science Forum 396-402 (2002) 161-166.

[4] L. Błaż, Mechanically alloyed aluminium - metal oxide composites, Ores and nonferrous metals 1 (2005) 24-32 (in Polish).

[5] L. Blaz, M. Sugamata, G. Wloch, J. Sobota, A. Kula, Structure and consolidation of rapidly solidified Meso 10 alloy flakes, Journal of Alloys and Compounds 506 (2010) 179-187.

[6] L. Blaz, M. Sugamata, A. Kula, G. Wloch, J. Sobota, Mechanical consolidation of rapidly solidified Meso 20 alloy - structure and mechanical properties, Journal of Alloys and Compounds (in print).

[7] J. Chang, I. Moon, C. Choi, Evolution of Microstructure and tensile strength of rapidly solidified Al-4.7%Zn-2.5%Mg-0.2%Zr-X %Mn Alloys, Metallurgical and Materials Transaction A 29 (1998) 1873-1882.

[8] B. Grushko, T. Ya. Velikanova, Structural Studies of materials. Formation of Quasicristals and Related Structures in Systems of Aluminum with Transiton Metals. I. Binary Systems Formed by Aluminum with 3d Metals, Powder Metallurgy and Metal Ceramics 43 (2004) 72-86.

[9] A. Kontio, P. Coppens, New Study of the Structure of $MnAl_6$, Acta Crystallographica B 37 (1981) 433-435.

[10] T. Ishimasa, Y. Kaneko, H. Kaneko, New group of stable icosahedral quasicrystals: structural properties and formation conditions, Journal of Non-Crystalline Solids 334-335 (2004) 1-7.

[11] R. Divakar, D. Sundararaman, V.S. Raghunathan, Al-Cu-Fe quasicrystalas: Stability and microstructure, Progress in Crystal Growth and Characterisation 34 (1997) 263-269.

[12] R. Popescu, A. Jianu, M. Manciu, R. Nicula, R. Manaila, Chemical trends of icosahedral order in Al-Cu-TM quasicrystals, Journal of Alloys and Compounds 221 (1995) 240-247.

[13] D. Shetchman, I. Blach, D. Gratias, J.W. Cahn, Metallic phase with long-range orientational order and no translational symmetry, Physical Review Letters 53 (1984) 1951-1954.

[14] unpublished data.