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M. ZYGMUNT-KIPER*, L. BŁAŻ*, M. SUGAMATA**

EFFECT OF MAGNESIUM ADDITION AND RAPID SOLIDIFICATION PROCEDURE ON STRUCTURE AND MECHANICAL PROPERTIES OF Al-Co ALLOY

WPŁYW DODATKU MAGNEZU I PROCESU SZYBKIEJ KRYSTALIZACJI NA STRUKTURĘ I WŁASNOŚCI MECHANICZNE STOPU Al-Co

Tested Al-5Co and Al-5Mg-5Co materials were manufactured using a common ingot metallurgy (IM) and rapid solidification (RS) methods combined with mechanical consolidation of RS-powders and hot extrusion procedures. Mechanical properties of as-extruded IM and RS alloys were tested by compression at temperature range 293-773 K. Received true stress vs. true strain curves were typical for aluminum alloys that undergo dynamic recovery at high deformation temperature. It was found that the maximum flow stress value for Al-5Mg-5Co alloy was much higher than that for Al-5Co, both for IM and RS materials tested at low and intermediate deformation temperatures. The last effect results from the solid solution strengthening due to magnesium addition. However, the addition of 5% Mg results also in the reduction of melting temperature. Therefore, the flow stress for Al-5Mg-5Co alloy was relatively low at high deformation temperatures. Light microscopy observations revealed highly refined structure of RS materials. Analytical transmission electron microscopy analyses confirmed Al_9Co_2 particles development for all tested samples. Fine acicular particles in RS materials, ~1μm in size, were found to grow during annealing at 823K for 168h. As result, the hardness of RS materials was reduced. It was found that severe plastic deformation due to extrusion and additional compression did not result in the fracture of fine particles in RS materials. On the other hand, large particles observed in IM materials (~20μm) were not practically coarsened during annealing and related hardness of annealed samples remained practically unchanged. However, processing of IM materials was found to promote the fracture of coarse particles that is not acceptable at industrial processing technologies.
Keywords: Rapid solidification, high temperature deformation, Al-Co aluminum alloys

W artykule przedstawiono wyniki badań stopów Al-5Co i Al-5Co-5Mg, które zostały przygotowane metodą metalurgii konwencjonalnej (IM), oraz metodą szybkiej krystalizacji (RS) połączonej z mechaniczną konsolidacją szybko-krystalizowanych proszków i wyciskaniem na gorąco. Ocenę własności mechanicznych wyciskanych stopów IM oraz RS wykonano za pomocą prób ściskania w zakresie temperatury 293-773K. Przebieg krzywych σ_t-ε_t dla badanych materiałów jest typowy dla stopów aluminium ulegającym zdrowieniu dynamicznemu. Naprężenie maksymalne stopów Al-5Mg-5Co jest znacznie wyższe niż w stopach Al-5Co zarówno wykonanych metodą IM jak i RS. Wraz ze wzrostem temperatury ściskania maleje wpływ umocnienia roztworowego magnezu na własności badanych stopów. Podczas odkształcania w 623 K – 773 K naprężenie uplastyczniające dla stopu Al-5Co jest większe niż dla Al-5Co-5Mg. Wskazano, że przyczyną może być obniżanie się temperatury topnienia pod wpływem dodatku magnezu (zwiększenie temperatury homologicznej w próbach odkształcania). Obserwacje strukturalne materiałów po szybkiej krystalizacji wykonane z użyciem mikroskopii optycznej wykazały występowanie drobnoziarnistej struktury. Badania wykonane z użyciem transmisyjnej mikroskopii elektronowej potwierdziły występowanie we wszystkich badanych próbkach wydzieleń typu Al_9Co_2. Drobne wydzielenia w stopach RS o początkowej wielkości poniżej 1μm ulegają rozrostowi w czasie wyżarzania przez 168h w 823K, co powoduje zmniejszenie twardości szybko-krystalizowanych materiałów. Korzystną cechą tych materiałów jest m.in. ich zwiększona podatność na odkształcenie, która przejawia się brakiem pękania wydzieleń wskutek dużych odkształceń plastycznych wskutek wyciskania i późniejszego ściskania próbek. W materiałach IM, w których wielkość cząstek przekraczała 20μm, podczas wyżarzania nie obserwowano zauważalnego efektu rozrostu wydzieleni, co się wiąże z brakiem istotnych zmian twardości stopu podczas wyżarzania. Jednakże występowanie tak dużych cząstek po procesie IM jest nie do zaakceptowania w przemysłowych procesach przetwórstwa metali ze względu na pękanie wydzieleń podczas przeróbki, co na ogół prowadzi do makroskopowego pękania wyrobów.

1. Introduction

Refining of structural components by means of combined rapid solidification (RS) and mechanical alloying (MA) methods is often used for improving mechanical properties of metallic materials. It was reported that an application of MA and powder metallurgy (PM) methods can be applied with suc-

* AGH – UNIVERSITY OF SCIENCE AND TECHNOLOGY, 30-059 KRAKÓW, POLAND
** NIHON UNIVERSITY, TOKYO, JAPAN

cess to the manufacturing of high-strength light-metal-based composites and allows incorporating chemically reactive components such as metal oxides or silicides into the light aluminum/magnesium matrix [1-4]. Introduction of mentioned reinforcements into the melt by means of a common metallurgy method is usually unsuccessful because of the rapid chemical reaction between aluminum/magnesium matrix and the particle. On the other hand, MA/PM methods are very expensive and time consuming procedures. The last statement give a motivation for searching less expensive methods of the structure refining and related strengthening of light-metal based materials. Therefore, an application of combined RS and PM methods for strengthening of some aluminum alloys, which are usually manufactured using IM methods, seems to be a promising procedure for high-strength light-materials production [5-7]. RS methods, including melt spinning or spray deposition, result in a cooling rate of 10^4-10^6 K/s that usually result in an oversaturation of solid solution and the development of specific metastable structures such as quasicrystalline phases often reported for RS aluminum-transition-metal alloys [8-16]. Effective refining of some brittle structural components such as Si-particles in RS hypereutectoid Al-Si alloy was also found to increase the hot workability of the material [11]. Mentioned above structural features of RS alloys cannot be obtained if a common metallurgy method is applied. A week point of RS-powder's consolidation and PM-processing is related to the powder vacuum pressing at intermediate temperatures and hot extrusion procedures that affect the structure morphology and reduce related mechanical properties of the bulk material. Moreover, both the average size and distribution of particles in RS powder grains or fine RS flakes, highly depends on their size and related cooling rate [17, 18].

Mentioned features of RS alloys provide the motivation for searching new methods, based on RS procedures, which allow the modification and the refining of structural components in some aluminum alloys such as Al-Co or Al-Mg-Co. The heat treatment, including ageing, is useless procedure for the particle strengthening of aluminum-cobalt alloys. Therefore, the basic item of the experimental work described below was focused on the effect of RS on the structure and particles morphology as well as mechanical properties of RS Al-Co alloys. Al-Mg-Co alloy was used for testing the effect of solid solution strengthening due to Mg-addition that is expected to overlap the precipitation hardening resulted from the refining of intermetallic grains in RS materials.

2. Experimental

Al-5Co and Al-5Mg-5Co alloys used in experiments were manufactured from high purity components by means of the industrial metallurgy method. Chemical composition of tested materials is shown in Table 1.

The rapid solidification and RS-powders consolidation was performed using adequate laboratory equipment. Melted alloys were atomized by means of the spray deposition on the rotating water-cooled copper cylinder [6, 16]. Pressured nitrogen gas was used to atomize the melt and protect the spray oxidation. As received RS-flakes, 1-4 mm in size and 0.1-0.3 mm in thickness, were compressed at 500MPa in thin-wall AA6061 cans and vacuum degassed at 623K using pressure of 1,33-13,3μPa. Following extrusion of both IM and RS materials was performed at 673 K using extrusion ratio λ =25. As received rods, 7 mm in diameter, were used for further experiments.

TABLE 1

Chemical composition of tested alloys

Denotation	Co (wt. %)	Mg (wt.%)	Al (wt.%)
Al-5Co	4.98	–	In balance
Al-5Co-5Mg	4.67	4.45	In balance

Mechanical properties of the material were tested by means of hardness and compression tests. Compression tests were performed at 293K-773K using samples of 6 mm in diameter and 8 mm in height. The samples were deformed $\varepsilon_t \cong 0.4$ at true strain rate of $\dot{\varepsilon}$ =5$\cdot$10^{-3} s^{-1} and water quenched within ~3 s after deformation finish.

As deformed samples were cut longitudinally and Vickers hardness tests were performed on mechanically polished flat surface of the sample. At least ten measurements were performed using SHIMADZU hardness tester and indenter load 0,9N (0.1 kG) to estimate the average hardness of the sample.

Preliminary structural analyses were performed on samples prepared by means of standard metallographic technique and light microscopy observations. Structural details were tested using analytical transmission electron microscope JEM2010 operating at accelerating voltage of 200 kV. The microscope was equipped with scanning transmission electron microscopy device (STEM) and X-ray energy disperse analysis system (EDS). Thin foils for TEM/STEM observations were grinded mechanically and finally ion thinned using GATAN PIPS-691 unit. Complementary observations were performed on scanning electron microscope HITACHI SU-70.

Annealing experiments at 823K were performed in order to evaluate the stability of RS material structure and hardness of the samples annealed within 7 days. The intermetallic particle size was measured from STEM and TEM pictures taken for initial RS materials and samples annealed at 823K/6h and 823K/7days. The number of particles at 50 nm intervals was normalized and percentage fraction of the particles vs. particle size was displayed in histograms.

3. Results

A set of true stress vs. true strain curves for RS Al-5Mg-5Co and RS Al-5Co are shown in Fig. 1. The shape of $\sigma_t - \varepsilon_t$ characteristics observed at high deformation temperatures is typical for aluminum alloys undergoing dynamic recovery [5, 19]. Hot compression tests were performed on as-extruded RS IM materials as well as on the samples annealed at 823K/6h. Both RS and IM as extruded or annealed samples did not fracture during compression within used strain range. True stress maximum vs. deformation temperature dependence for as-extruded materials and samples annealed at 823K/6h is shown in Fig. 2. It should be mentioned that the maximum flow stress at 293K-373K was measured at $\varepsilon_t \approx 0.4$ i.e. before a steady-state flow regime was reached.

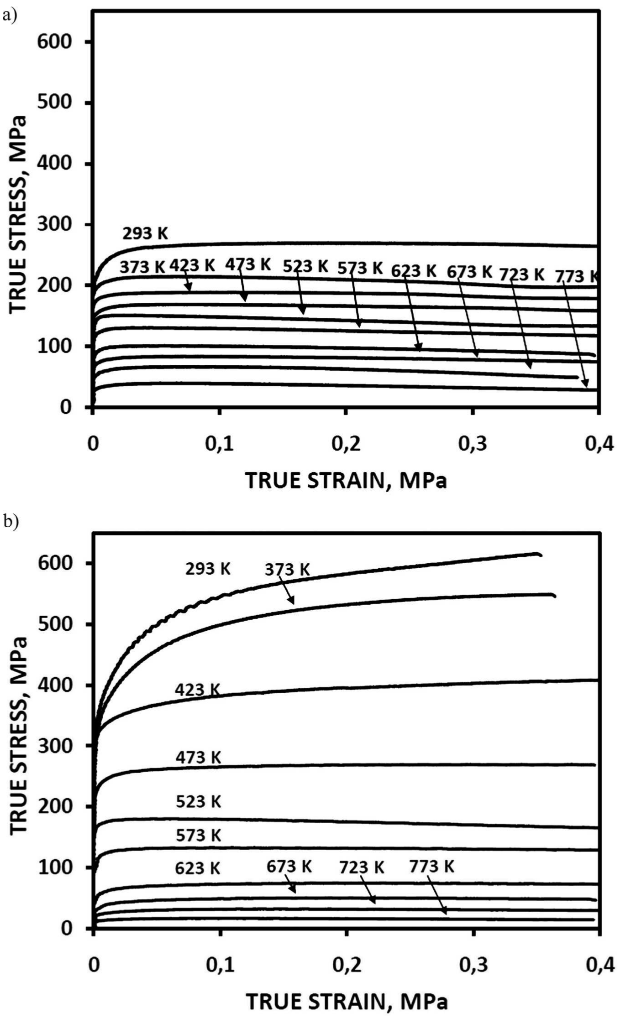


Fig. 1. True stress - true strain curves received by the compression test performed at deformation temperatures marked in the figure: (a) – RS Al-5Co; (b) – RS Al-5Mg-5Co

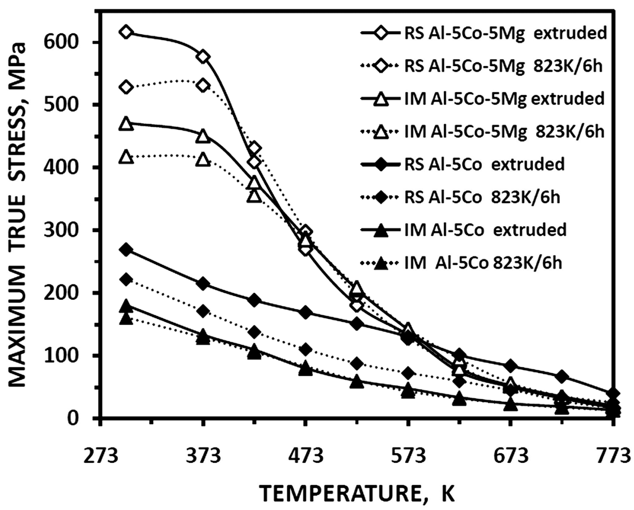


Fig. 2. Effect of deformation temperature on the maximum flow stress for IM Al-5Co, RS Al-5Co, IM Al-5Co-5Mg and RS Al-5Co-5Mg as extruded alloys and samples annealed at 823K/6h. The materials and heat treatment conditions are listed in the legend

Hardness of samples deformed by compression is shown in Fig. 3. It is worth mentioning that the solid solution hardening due to the magnesium addition in Al-5Mg-5Co alloys, result in more effective hardening than the grain refining caused by RS procedure both for Al-5Mg-5Co and Al-5%Co alloy (Fig. 3a). Mentioned effect is particularly evident for samples preliminarily annealed at 823K/6h (Fig. 3b).

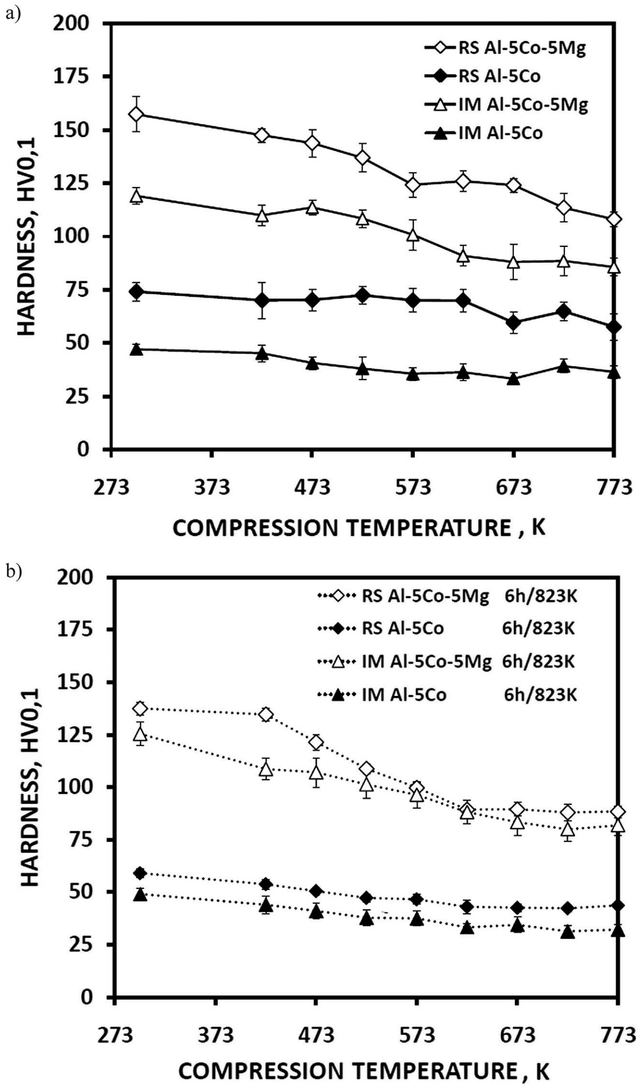


Fig. 3. Hardness of samples deformed by compression ($\varepsilon_t \approx 0.4$) vs. deformation temperature: (a) as-extruded RS and IM materials; (b) RS and IM samples preliminarily annealed at 823K/6h. Tested materials are listed in a legend

The effect of annealing at 823K on the material structure and related mechanical properties was tested. RS and IM alloys were annealed within 7 days and the sample hardness vs. annealing time relation was analyzed with respect to the intermetallic particles size distribution. Hardness of rapidly solidified alloys was reduced from 137HV to 87 HV and 70HV to 39HV for Al-5Co and Al-5Co-5Mg alloys, respectively. Hardness of IM materials was practically stable and some fluctuations can be ascribed to the experimental error rather than the effect of annealing.

4. Structure observations

Characteristic structure of as extruded IM Al-5Co alloy and the sample annealed at 823K/168h (7 days) are shown in Fig. 5. It was often observed that some coarse particles were broken due to the high strain induced by the extrusion with λ = 25 and following deformation by the compression. Because of the low resolution at optical microscopy observations any noticeable coarsening of fine particles was observed. In contrary to IM materials, very fine particles were observed for both RS Al-5Co and Al-5Mg-5Co alloys (Fig. 6). TEM observations lead to the conclusion that no cracks or voids are formed at both as extruded and annealed RS materials.

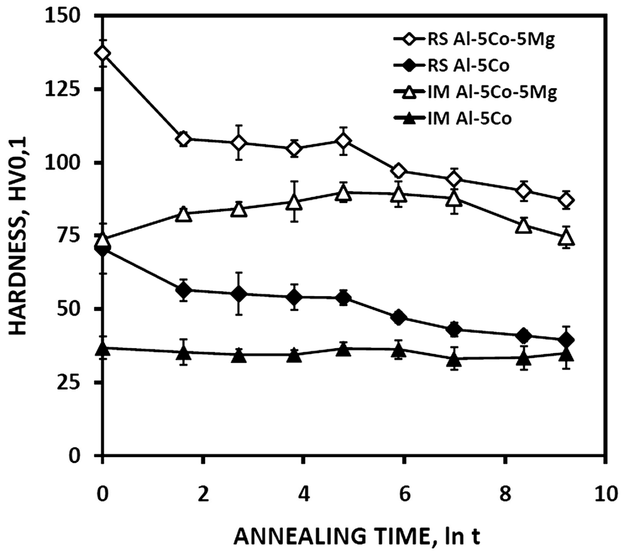
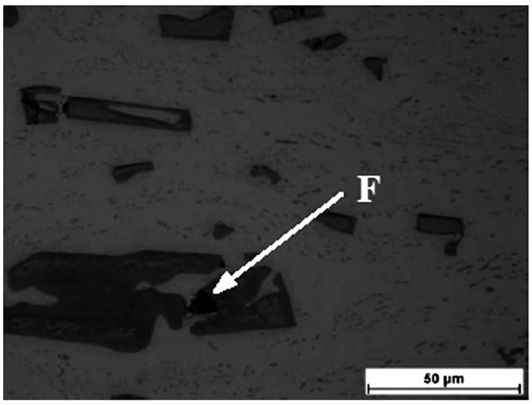


Fig. 4. Hardness vs. annealing time curves received for IM and RS Al-5Co and Al-5Co-5Mg alloys annealed at 823 K

a)


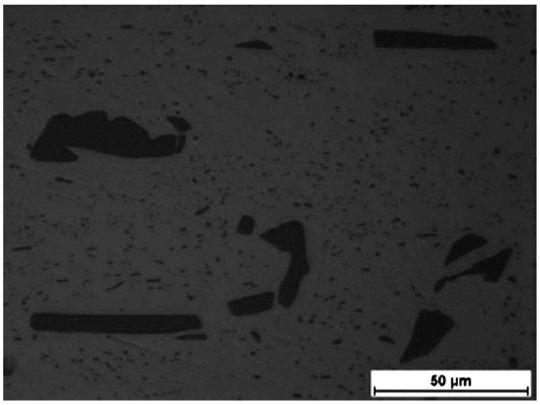
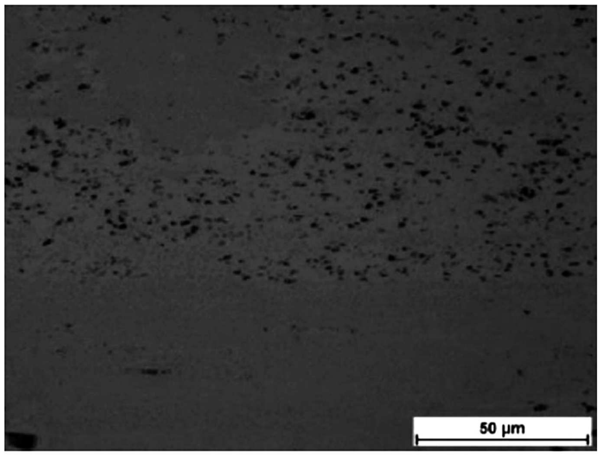
b)


Fig. 5. Structure of I/M Al-5Co alloy observed on longitudinal section using light microscopy: (a) – as extruded material; (b) sample annealed at 823 K/168 h (F – fracture)

a)


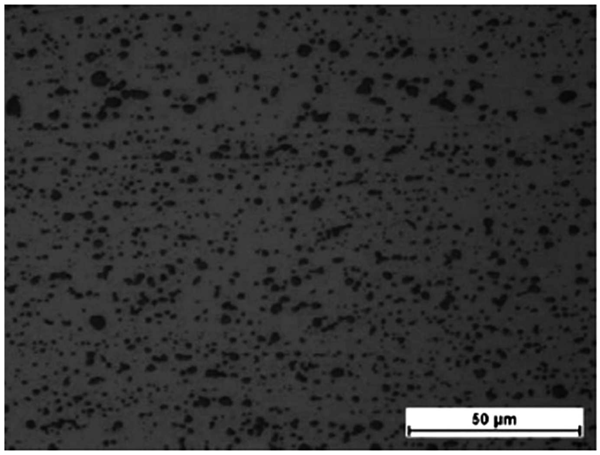
b)


Fig. 6. Structure of RS Al-5Co alloy: (a) as extruded material; (b) sample annealed at 823 K/168 h (optical microscopy, longitudinal section)

Detailed analysis of the material structure was performed by means of analytical transition electron microscopy methods. Selected diffraction pattern analysis of precipitates revealed the development of Al_9Co_2 phase both for IM and RS Al-5Co samples (Fig. 7). In contrary to RS alloy, fine cracks at coarse particles were often observed at IM materials as marked (F) in the Fig. 7a. Chemical composition of particles, tested by EDS, confirmed characteristic stoichiometry of elements for Al_9Co_2 phase.

Typical structure of as extruded materials and samples annealed at 823K/6h and 823K/168h, revealed by STEM, are shown in Fig. 8 and Fig. 9 for RS Al-5Co and RS Al-5Co-5Mg, respectively. Estimation of particles coarsening during the annealing was difficult as the morphology of particles had varied at given observation area due to the annealing effect as well as because of varied particle morphology at preliminary structure of RS-flakes. STEM and TEM pictures were used for the particle size assessment and received results were shown on histograms presented in Fig. 10. The prevailing number of particle size at RS materials was within dimensions of 100 nm – 200 nm.

The annealing at 823K/6 h was found to result in particles coarsening that resulted in dominating particle size of 150-500 nm in receiving. Prolonged annealing up to 168h resulted in remarkable reduction of fine particle numbers that seems to be accompanied by the coarsening of some particles even up to 0.5 μm – 1.2 μm in size. However, the reason of so large particle coarsening effect at RS Al-5Co as shown in Fig. 10a, is not clear.

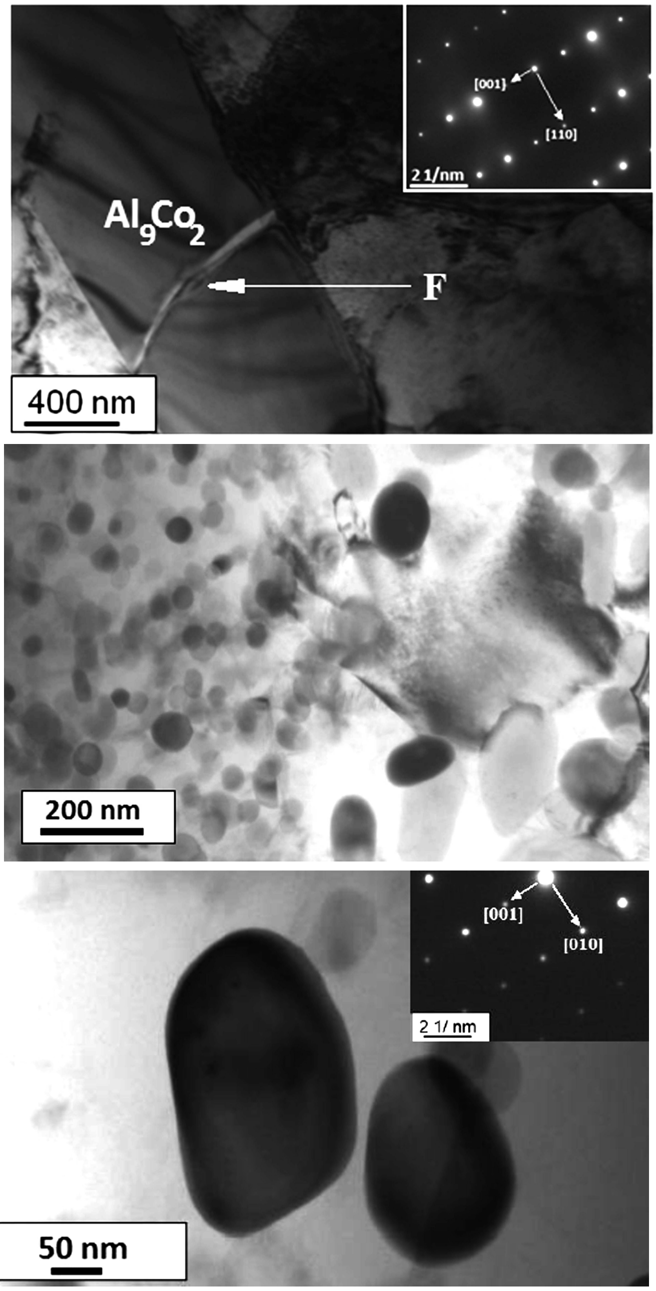


Fig. 7. Structure of as-extruded Al-5Co rod observed by means of TEM: (a) coarse Al_9Co_2 fractured particle (see: F) revealed in IM material. Adequate SAD pattern is shown in TEM picture; (b) RS material containing fine particles; (c) enlarged detail of RS material structure and incorporated SAD pattern taken from fine Al_9Co_2 particle

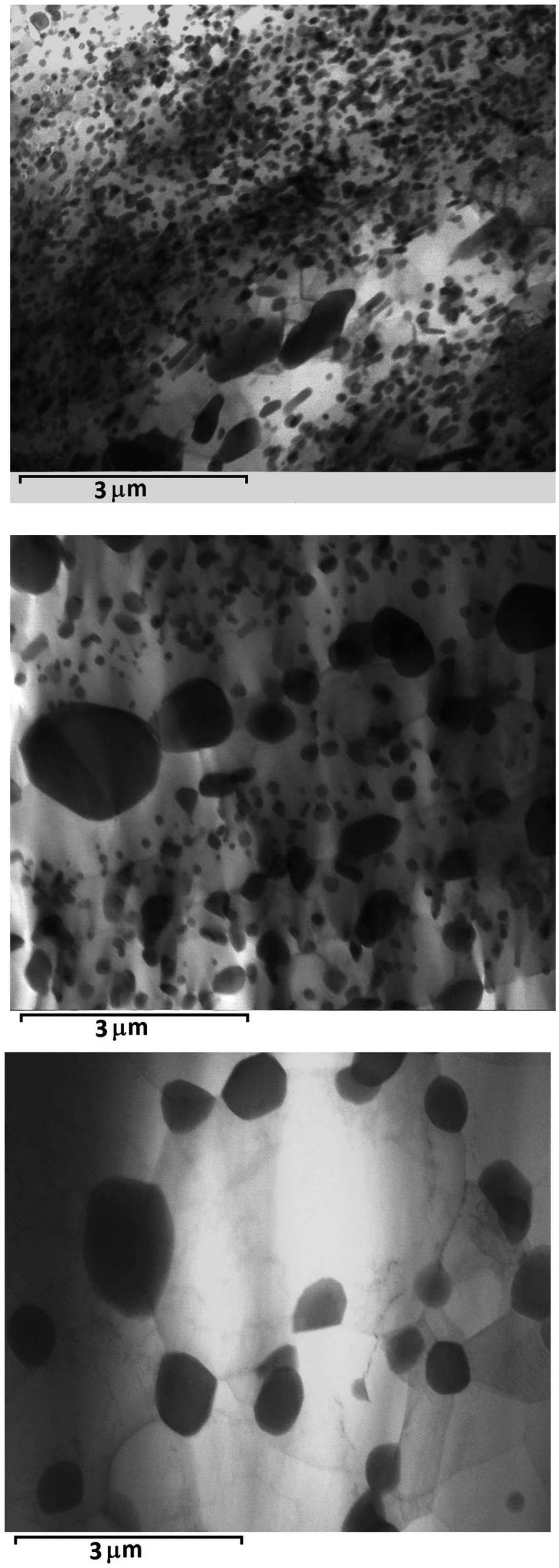


Fig. 8. STEM Structure RS Al-5Co alloy (a) as-extruded (b) annealed 6h/823K (c)annealed 7d/823K

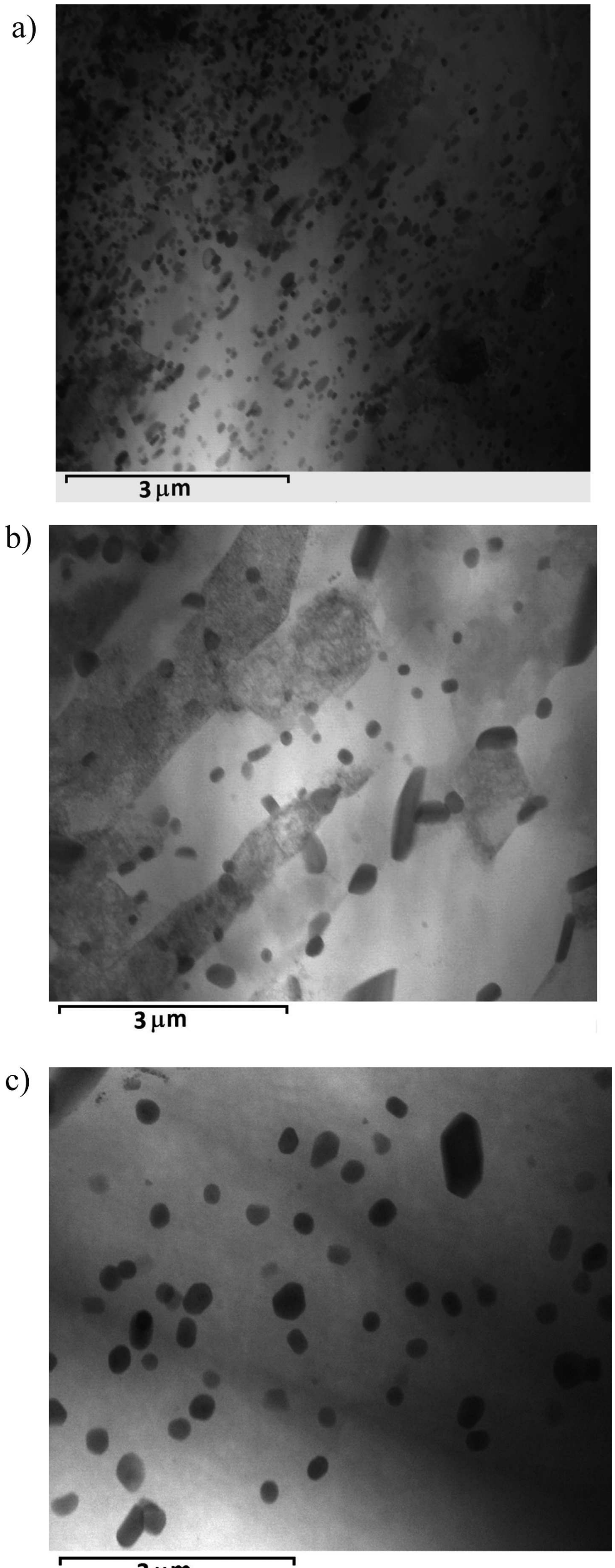


Fig. 9. STEM Structure of RS Al-5Co-5Mg alloy (a) as-extruded material (b) sample annealed at 823K/6h (c) sample annealed at 823K/168h

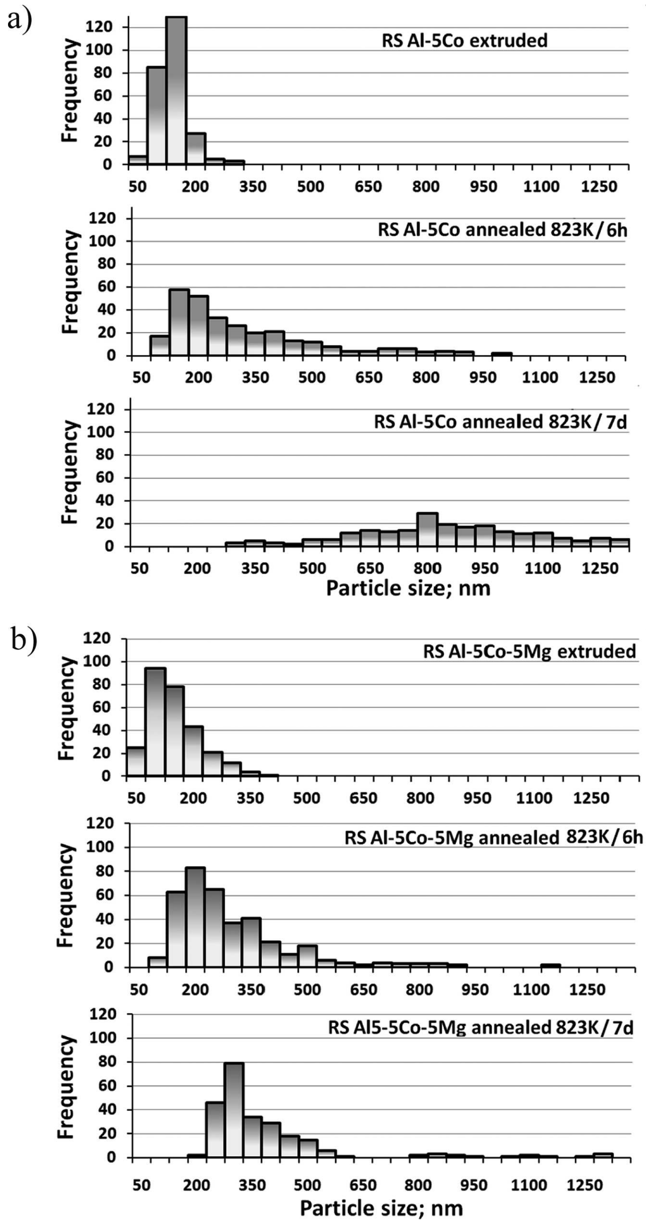
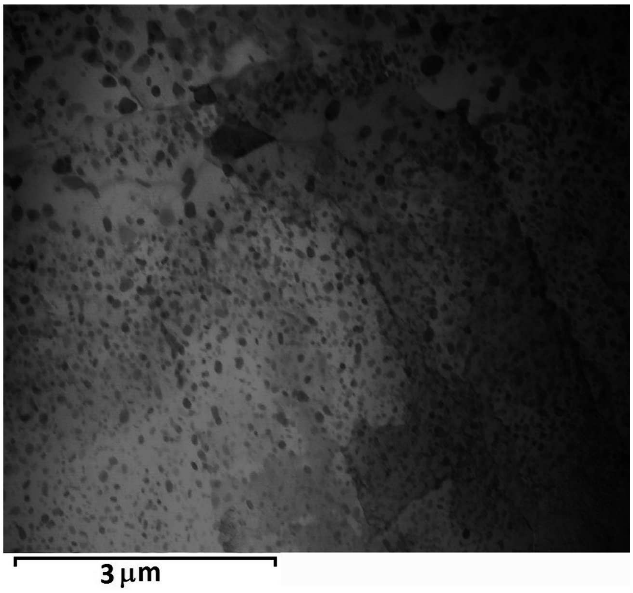


Fig. 10. Distribution of Al_9Co_2 particles in as-extruded and annealed RS alloys (a) Al-5Co; (b) Al-5Co-5Mg. Heat treatment conditions (823K/6h, 823K/168h) are marked in the figure

TEM/STEM structure observations were also performed for selected samples deformed by compression. Structure of samples deformed $\varepsilon_t \cong 0.4$ at 823K is shown in Fig. 11a and Fig. 11b for RS Al5-5Co and RS Al-5Co-5Mg alloy, respectively. It is worth stressing that particles were observed at wide area available at the thin foil and it was concluded that the particles size was not noticeably affected by the high temperature deformation. Some differences in the particle morphology are related to preliminary RS material structure rather than the deformation process.

a)



b)

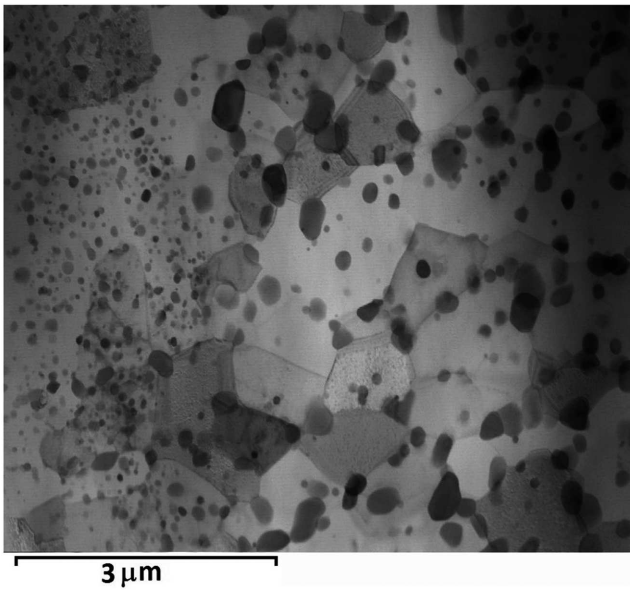


Fig. 11. STEM structure of RS alloys deformed $\varepsilon_t \cong 0{,}4$ at 773K by compression: (a) Al-5Co (b) Al-5Co-5Mg

5. Discussion

Processing of IM Al-5Co and IM Al-5Co-5Mg alloys can raise some difficulties because of the coarse Al_9Co_2 particles fracturing. Micro-cavities growth at some interphase boundaries as well as the fracture of coarse particles is often observed at hot extruded materials (Fig. 5, Fig.7a). It is commonly known that the crack works as a stress concentrator during deformation of the material that induces the material fracturing during processing and reduces mechanical properties of the product. Therefore, development of voids and micro-cracks is usually not accepted both at processing technologies and at commercial use of products.

Fortunately, no cracks were observed in tested fine-grained RS alloys. Rapid solidification of Al-5Co and Al-5Co-5Mg alloys combined with vacuum pressing and hot powder consolidation procedure, including extrusion at 673K, result in receiving of well-consolidated bulk material. Porosity of powder-made materials is negligible. In contrary to IM materials, very fine particles does not fracture in hot deformed RS Al-5Co and Al-5Mg-5Co alloys and any voids are formed at interphase boundaries (Fig. 6, Fig 7bc, Fig. 8a, Fig. 9a). No cracks or voids were found for both as extruded and annealed samples.

Structural observations revealed some differences in local particles morphology in tested RS alloys (Fig. 6, Fig. 7, Fig. 8). Local differences in the particle size can be ascribed to varied structure of initial flakes that were consolidated into the bulk material. Particle size at each flake depended on the rapidly solidified drop size and related cooling rate. Smaller drops were solidified at higher cooling rate and resulted structure was finer than that for other large flakes.

Structural observations and mechanical test lead to the conclusion that the magnesium addition result in solid solution hardening of the aluminum matrix and does not change the structure of Al_9Co_2 particles. Efficient strengthening of both IM and RS materials due to magnesium addition was observed for samples deformed at 293K-523K (Fig. 1 and Fig. 2). At higher deformation temperatures, magnesium hardening effect was reduced. The flow stress value for RS Al-5Co alloy deformed at 623K-773K was higher than that for other materials. It is worth stressing that the increase of the magnesium content results in *solidus* temperature reduction from 933 K to ~850 K for aluminum and Al-4.45%Mg alloy, respectively. Therefore, mentioned formerly effect of the flow stress reduction for Al-5Co-5Mg samples deformed at high deformation temperatures can be ascribed to the reduction of homologous temperature with respect to Al-5Co alloy.

Prolonged annealing of RS materials at 823K was found to result in some coarsening of particles (Fig. 10). However, in spite of 7-days annealing (168h), the particles still remained much smaller dimensions than that for IM alloys. The annealing at 823K/168h resulted in evidently reduced counts of very fine particles, which was accompanied by the coarsening of some particles even up to 0.5 μm – 1.2 μm. The reason of particularly intensive coarsening of the particles at RS Al-5Co (Fig. 10a) is not clear. In spite of the particles coarsening, the effect might probably result from varied distribution of particles in the material structure (Fig. 6) and highly localized structure observations related to the high magnification used at TEM and STEM methods. As result, the histogram displayed for RS Al-5Co annealed at 623K/168h may represent the area of particularly coarse-grained initial RS flake. On the other hand, the coarsening of particles might also result from enhanced diffusion of Co in Al-matrix containing Mg addition. However, the effect of magnesium on the diffusion coefficient value was not found in the literature [20].

Both the particles coarsening and recovery processes are responsible for the softening of RS Al-5Co and RS Al-5Co-5Mg alloys during annealing at 823K (Fig. 4). Hardness of IM materials was relatively stable if neglect some variations of the results raised probably from the experimental error. Both RS Al-5Co and RS Al-5Co-5Mg materials were found to remain noticeably higher hardness value than the reference materials i.e. manufactured by means of IM method. However, the solid solution hardening due to magnesium addition is evidently higher than the effect of the particle refining due to RS procedure. The solid solution hardening due to magnesium addition was more efficient than that induced by RS procedure both for as extruded material and the samples preliminarily annealed at 823K/6h or 823K/168h.

Similar conclusions on the particles coarsening can indirectly be deduced from the compression test results and hardness measurements performed for as-deformed samples (Fig.3). As the dynamic recovery is intensified with deformation temperature, the hardness value for hot deformed samples is reduced with temperature. However, observed strengthening due to RS procedure is lower than the effect of solution strengthening induced by the magnesium addition. Mentioned effect is particularly evident for samples preliminarily annealed at 623K/6h (Fig. 3b).

6. Conclusions

- Heavy deformation of IM Al-5Co and IM Al-5Co-5M alloys due to the hot extrusion (673 K, λ =25) was found to result in the coarse particles fracturing and the development of fine voids at some interphase boundaries. These disadvantageous material defects were not found in fine-grained materials if RS/PM procedures were used. Applied RS/PM procedures create very fine-grained material structure that is favorable for the processing and manufacture of well-consolidated bulk materials having high mechanical properties.
- Transmission electron microscopy analysis confirmed Al_9Co_2–type structure of the particles observed for both industrial and rapidly solidified Al-5Co alloy. Magnesium addition did not change the particles structure at IM and RS Al-5Co-5Mg materials.
- Highly refined particles, which are formed due to RS procedure, result in the hardening of both Al-5Co and Al-5Co-5Mg alloys. Relatively higher strengthening of IM and RS Al-5Co-5Mg materials was ascribed to the solid solution hardening of aluminum-magnesium matrix.
- Hot compression tests performed 293K-523K revealed high strengthening effect due to magnesium addition that was reduced with increasing deformation temperature. It was found that the flow stress value for samples deformed at 623K-773K was higher for RS Al-5Co than that for other tested materials.
- Combined rapid solidification and solid solution hardening due to the magnesium addition result in the most effective strengthening of tested aluminum-cobalt alloys.
- Mechanical properties of RS materials are evidently higher than these for IM alloys and retain the predomination of their properties independently of annealing and hot deformation conditions.

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