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MICROSTRUCTURE AND PLASTICITY OF HOT DEFORMED 5083 ALUMINUM ALLOY PRODUCED BY RAPID SOLIDIFICATION AND HOT EXTRUSION

BADANIA MIKROSTRUKTURY I PLASTYCZNOŚCI ODKSZTAŁCANEGO NA GORĄCO SZYBKO-KRYSTALIZOWANEGO I WYCISKANEGO STOPU ALUMINIUM 5083

The objective of the present study is to analyze the mechanical properties and thermal stability for rapidly solidified and extruded 5083 aluminum alloy (RS). Compression tests were performed in order to estimate flow stress and strain rate sensitivity relation for 5083 alloy in the temperature range of 20°C to 450°C. For the comparison purposes, conventionally cast and extruded industrial material (IM) was studied as well. Deformation tests performed at room temperature conditions show that rapidly solidified material exhibits about 40% higher yield stress (YS=320 MPa) than conventionally cast material (YS=180 MPa), while the deformation at 450°C results in significant decrease of flow stress parameters for RS material (YS=20 MPa) in comparison to IM material (YS=40 MPa). Strain rate sensitivity parameter determined for high temperature conditions indicates superplasticity behavior of RS material. Structural observations show that under conditions of high-temperature deformation there are no operating recrystallization mechanisms. In general, grain size below 1μm and size of reinforcing phases below 50 nm is preserved within the used deformation temperature range.
Keywords: rapid solidification, Al-Mg alloy, thermal stability, superplasticity

Celem niniejszej pracy było zbadanie własności mechanicznych oraz stabilności termicznej szybkokrystalizowanego a następnie wyciskanego stopu aluminium 5083. Testy ściskania przeprowadzone w zakresie temperatur od 20°C do 450°C przy dwóch różnych prędkościach odkształcenia pozwoliły na wyznaczenie charakterystyk naprężenia płynięcia materiału oraz jego czułości na prędkość odkształcenia w funkcji temperatury. Wyniki odniesiono do wyciskanego w identycznych warunkach, konwencjonalnie odlewanego materiału (IM – industrial material) o tym samym składzie chemicznym. Uzyskane dane wskazują, że szybkokrystalizowany materiał posiada w temperaturze pokojowej ok. 40% wyższą granicę plastyczności wynoszącą 320 MPa w stosunku do materiału odlewanego – 180 MPa. Natomiast w najwyższej temperaturze deformacji naprężenie uplastyczniające stopu RS jest dwukrotnie niższe niż dla materiału IM (40 MPa dla materiału odlewanego w porównaniu z 20 MPa dla materiału szybko krystalizowanego). Wyznaczony w najwyższej temperaturze parametr czułości na prędkość odkształcenia dla stopu RS (Rapid Solidification) osiąga wartości zbliżone do 0,3 co wskazuje na nadplastyczność tego materiału. Przeprowadzone badania elektrono-mikroskopowe wskazują na brak aktywnych mechanizmów odbudowy struktury. Zarówno wielkość ziarna w materiale RS będąca na poziomie poniżej 1μm jak i wielkość faz umacniających nie przekraczających 50 nm nie ulegała zmianie w całym zakresie stosowanych temperatur.

1. Introduction

Aluminium alloys development is currently one of the most important subjects in the automotive industry. It has been recognized that commonly known Al-Zn-Mg, Al-Cu-Mg alloys, which are widely used for production of highly loaded components in automotive industry are able to achieve relatively high levels of tensile strength through various regimes of thermo-mechanical treatment. However, those alloys suffer from insufficient corrosion resistance and plasticity. On the contrary, Al-Mg alloys have excellent corrosion resistance, good ductility but only moderate strength parameters. In the last decade, significant efforts have been made to improve mechanical properties of Al-Mg alloys through altering chemical composition [1, 2] or application of new processing routes [3-6]. Among them substantial attention is paid to the novel techniques of the microstructure refining, which according to the Hall-Petch rule leads to increase of the mechanical properties. Fine structural components can be obtained on the way of the severe plastic deformation (SPD) [7], thermo-mechanical

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processing (TMP) or the rapid solidification (RS) [8] of the material. It has been known that application of different preparation routes results in formation of different grain size and intermetallic phase distributions. Both of these structural elements strongly influence material properties such as strength at room temperature, thermal stability and high temperature formability [9, 10]. It is readily recognized that in the range of ultra fined grained (UFG) sized materials improvement of strength parameters is observed in comparison to conventional alloys. Furthermore, superplastic behavior during high temperature deformation can be expected [11-13]. Grain refinement results in shift of superplastic deformation regime towards lower temperatures and higher strain rates [14].

A critical point for ultra fined grained structure is strong susceptibility to activation of recrystallization processes. It is known that thermal stability of the material can be enhanced by the addition of element such as Zr, Sc, Cr, Mn which forms highly dispersed intermetallic compounds that provides thermal stabilization by the grain boundary pinning during hot deformation [10, 15, 16].

The aim of this study was to examine the influence of the rapid solidification process on the structure refinement of the 5083 alloy with correlation to the mechanical properties and thermal stability at high temperatures.

2. Experimental

The experiments were performed on rapidly solidified and extruded (RS) 5083 aluminum alloy with standard composition given in Table 1.

TABLE 1
Chemical composition of AA5083 alloy (wt.%

Mg	Mn	Si	Zn	Fe	Cr	Ti	Al
4.00-4.90	0.40-0.10	max. 0.40	max. 0.25	max. 0.40	max. 0.25	max. 0.15	balance

In this study, the melt spinning process was applied in order to achieve high cooling rates. Obtained material in the form of ribbons with the thickness in the range of 50 to 100μm was cold compacted to form of billets, 8 cm height and 4 cm in diameter, degassed and extruded at 400°C with the cross section reduction ratio of λ =25. Finally, rods 8 mm in diameter were obtained. It was noticed that surface of as extruded material is characterized by good quality, free from typical extrusion defects. For comparison purposes, the conventionally cast and hot extruded 5083 aluminum alloy was prepared as well (IM – industrial material). Material was cast to 40 mm in diameter steel crucible and homogenized at 530°C for 12hrs. As-prepared billet was hot extruded at the same processing parameters as in the case of RS material.

Mechanical investigations at room and elevated temperatures were conducted by means of the tensile and compression test performed on samples machined from as extruded rods. Servo-hydraulic MTS880 machine capable of high strain rates tests was used in the experiment. Samples for compression, 8 mm in height and 6 mm in diameter, were tested at two different strain rates 10^{-2} s^{-1} and $1s^{-1}$ within temperature range of 20°C to 450°C. The high strain rate tests were performed during experiments in order to test material behavior at conditions close to the industrial ones. Three zone split furnace with temperature stabilization better than a 2°C was used during tests. Samples before compression were held at given temperature for 10 minutes in order to equalize the temperature.

Tensile tests were performed at room temperature and initial strain rate of 10^{-2} s^{-1}, bone shaped specimens with the gauge length of 20 mm and 5 mm in diameter were used. Mechanical parameters for both compression and tension tests were determined by using at least three samples measurements. In order to test thermal stability of the material Vickers hardness measurements were performed for the isothermally annealed and hot deformed samples.

Structural observations were performed for as extruded and deformed samples. Specimens were cut along plane parallel to the extrusion direction and subjected to preparation for structure observations. For surface preparation mechanical polishing followed by gentle ion thinning were used. Final sample etching step has proven unsuccessful for the RS material due to submicron structure of the alloy, thus grains were revealed by means of the contrast channeling technique with BSE detector. Additionally, grain size, dislocations and phases distribution were analyzed with transmission electron microscopy. TEM specimens were cut from material mechanically thinned and prepared by twin jet electrochemical polishing. For both thin foil and bulk samples HITACHI SU-70 scanning electron microscope equipped with transmitted electron detector (TE) and back scattered electrons (BSE) detector was used.

3. Results

3.1. Structure and mechanical properties of extruded materials

The longitudinal section of as extruded RS 5083 alloy with its cast IM 5083 counterpart observed by means of scanning electron microscopy is shown in Fig. 1a and Fig. 1b, respectively. It is clearly seen that structure of both materials is characterized by equiaxed grains which indicates that dynamical recrystallisation processes was active during hot extrusion. Structure of the IM material is typical for cast and extruded materials with relatively large grain size (15μm in size) and coarse primary intermetallics, seen as white phases on the Fig. 1b and Fig. 2. Structural elements identified on the Fig. 2 were found to be complex intermetallic compounds that consist of aluminum, manganese, iron and chromium elements. Primary intermetallics are formed during casting procedure and are stable at subsequent homogenisation and deformation procedures.

Structure of RS materials is highly refined in comparison to IM material. Grains are equiaxied with uniform size distribution and average size of 700 nm. Furthermore, significant improvement in reduction of primary compounds size is evidenced for RS alloy. The growth of intermetallic particles was suppressed, thus formation of very fine (up to 50 nm in size), round shaped phases was observed. Particles were uniformly distributed over the material structure, both inside grains and along grain boundaries. Based on the chemical analysis, intermetallic particles were identified as Mn, Fe and Cr rich phases (Fig. 3). No voids in RS material were detected which confirms that full material densification during plastic consolidation was achieved.

Typical strain-stress curves for as extruded RS and IM 5083 alloys are shown in Fig. 4. Serrated flow behavior of both IM and RS materials is characteristic of Al-Mg alloys system. Calculated values of yield strength (YS), ultimate tensile strength (UTS) and total elongation (EL) are presented in Table 2. Properties of similar materials processed by different production routes are also shown for comparison purposes. It is clearly seen, that 5083 alloy processed by RS route exhibit about 40% higher YS and 20% UTS than IM material, however in-

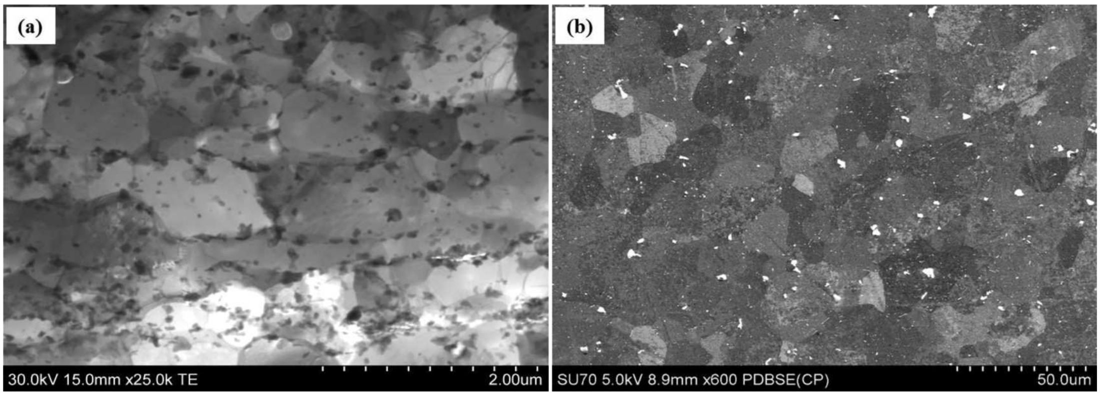

Fig. 1. SEM images of the 5083 alloy prepared by different processing routes: a) structure of RS material obtained by means of TE detector, b) structure of IM material obtained by BSE detector

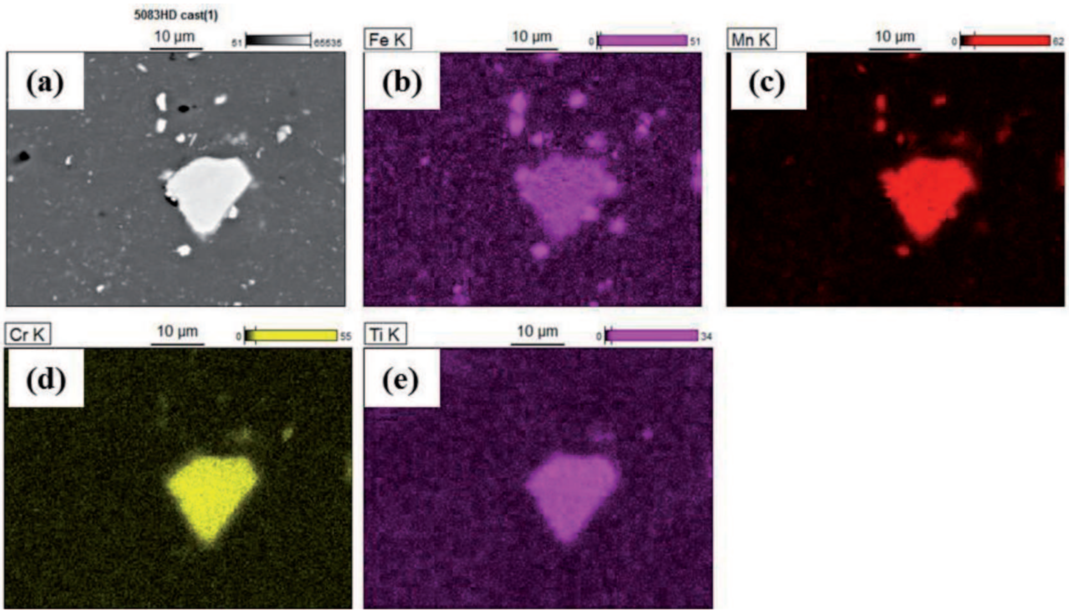

Fig. 2. SEM image of IM material (a) with related chemical mapping results for: (b) iron (Fe), (c) manganese (Mn), (d) chromium (Cr) and (e) titanium (Ti) elements

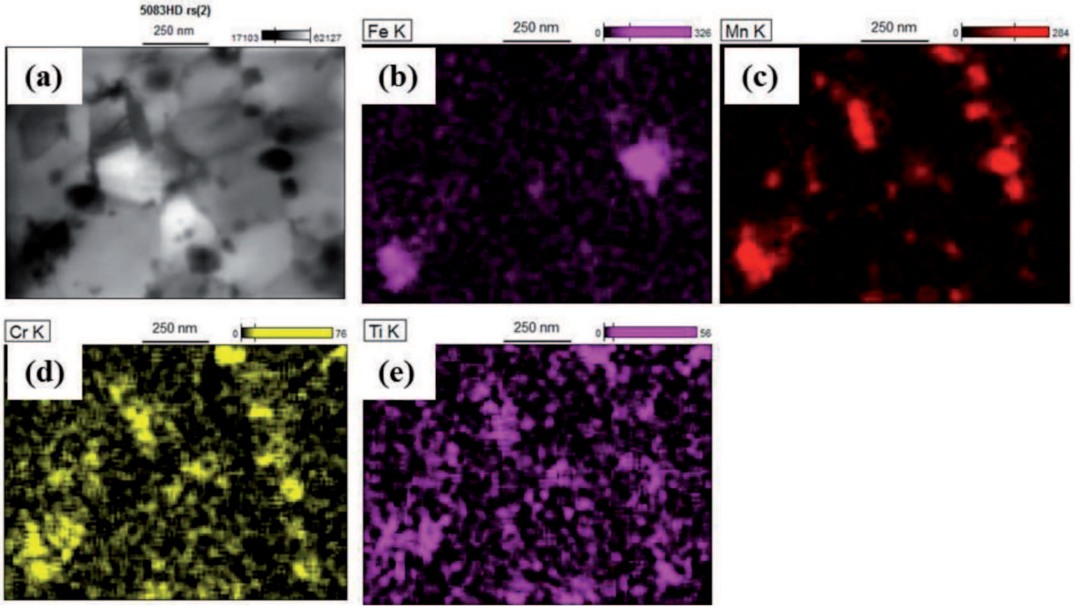

Fig. 3. STEM image of RS material (a) with related chemical mapping results for: (b) iron (Fe), (c) manganese (Mn), (d) chromium (Cr) and (e) titanium (Ti) elements

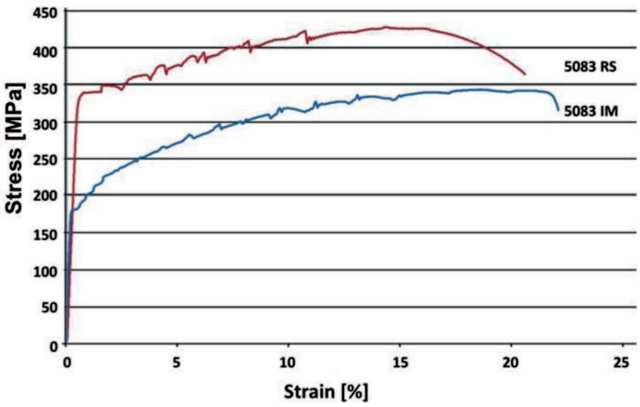

Fig. 4. Tensile stress – strain curves for 5083 IM and RS materials obtained at room temperature and initial strain rate of 10^{-2} s^{-1}

TABLE 2
Mechanical properties of Al-Mg alloys prepared by various processing routes

Material	YS [MPa]	UTS [MPa]	El. [%]	Ref.
5083 RS	320	430	20,3	This work
5083 IM	180	335	22.1	This work
Cryomilling+ extrusion	334	462	8,4	[4]
ECAP	375	427	8,0	[5]
TUC (Torsion under compression)	355	385	15	[6]

crease of mechanical properties is not connected with the loss of the plasticity – both materials revealed the same total elongation. Rapidly solidified and extruded 5083 material has properties similar to those obtained by other non-conventional production methods such as severe plastic deformation and thermo-mechanical processing (Table 2).

3.2. Mechanical properties at elevated temperatures

Mechanical properties of as – extruded RS and IM materials were examined at 20°C – 450°C by compression tests performed at two different strain rates of 10^{-2} s^{-1} to 1 s^{-1}. Typical flow stress curves for RS material are shown in Fig. 5. It is clearly seen that maximum flow stress during deformation at elevated temperatures is achieved at early stage of compression and remains unchanged or slightly decreased during subsequent deformation. The same flow stresses behavior was observed for both IM and RS materials at all tested deformation strain rates.

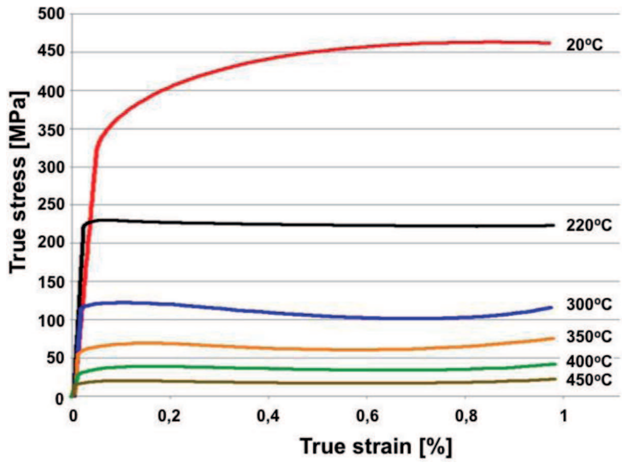

Fig. 5. True stress – true strain curves for compression test of RS material at various temperatures and strain rate of 10^{-2} s^{-1}

The effect of deformation temperature on the maximum flow stress is shown in Fig. 6 and Fig. 7 for RS and IM material, respectively. Strain rate sensitivity parameters m, calculated from two order of magnitude change of deformation rate, is also included on the figures. It is easy to recognize that the higher compression temperature the more pronounced difference between flow stresses of both materials is observed. For example, at the deformation conditions of 450°C and strain rate of 10^{-2} s^{-1}, flow stress for the IM and RS materials are 48 MPa and 19 MPa (Fig. 6), respectively. Simultaneously, strain rate sensitivity parameter at given temperature condition is 50% higher for RS material (m=0.3) in comparison to IM alloy (m=0.2).

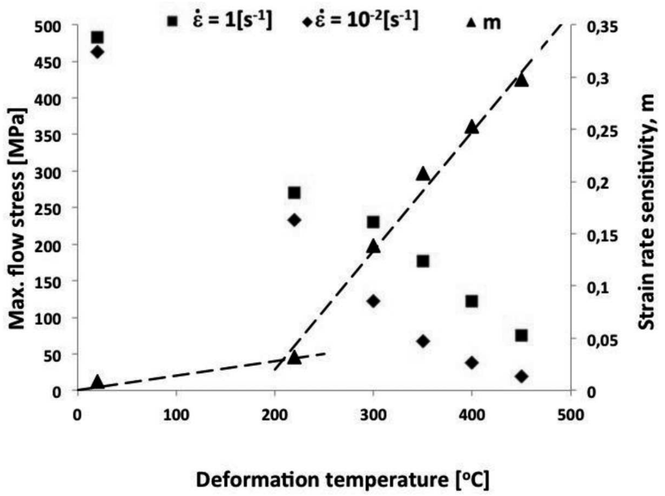

Fig. 6. The effect of deformation temperature on the maximum flow stress and strain rate sensitivity parameter of RS 5083 material. Compression test was performed at constant strain rate of 10^{-2} s^{-1} and 1 s^{-1}

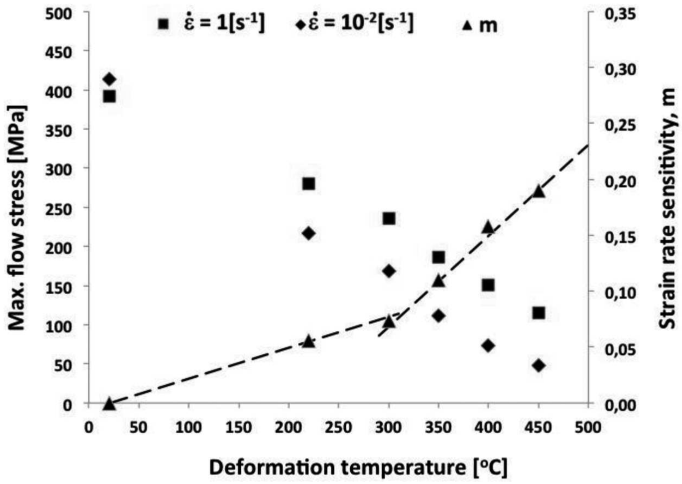

Fig. 7. The effect of deformation temperature on the maximum flow stress and strain rate sensitivity parameter of IM5083 material. Compression test was performed at constant strain rate of 10^{-2} s^{-1} and 1 s^{-1}

TABLE 3

Results of hardness measurements for isothermally annealed and hot deformed 5083 alloy

Isothermal annealing		5083 RS		5083 IM	
		$HV_{0.5}$	Std. dev.	$HV_{0.5}$	Std. dev.
As extruded		111,0	2,2	81,1	4,1
450°C	0,5h	110,3	1,7	80,6	1,6
	1h	108,5	3,3	79,7	2,9
Compression test					
	strain rate				
Deformation at 400°C	10^{-2} s^{-1}	101,7	5,1	82,4	2,5
	1 s^{-1}	107,6	1,1	82,9	3,3
Deformation at 450°C	10^{-2} s^{-1}	98,5	2,5	80,5	3,3
	1 s^{-1}	103,9	3,0	83,9	2,7

3.3. Thermal stability

Preserving high mechanical properties during hot deformation processes is the basic challenge for RS materials. It is know that higher processing temperatures requires lower forces, which is more favorable from technological point of view, however more pronounced recovery processes in the materials structure are observed. In order to determine thermal stability of tested material, isothermal annealing process at 450°C was performed followed by hardness measurements. Effect of deformation temperature on the hardness of as – deformed samples was studied as well. Received results are shown in Table 3. Samples submitted to isothermal annealing shows excellent stability i.e. after 1 h of heat treatment hardness decrease is negligible. Hardness of hot deformed samples slightly decrease and is more pronounced for samples deformed at lower strain rate and higher deformation temperature. Hardness of IM samples is not affected neither by temperature condition nor deformation rate.

4. Discussion

In order to test the influence of rapid solidification on 5083 alloy two different processing routes were applied. Rapidly solidified (RS) material in form of flakes as well as conventionally cast and homogenized material were extruded at the same conditions. Effective improvement of mechanical properties due to RS procedure is clearly seen. Yield stress parameter determined for tested alloys was 330MPa and 180MPa for RS and IM materials, respectively. It was found that high strength of as-extruded RS material was accompanied by relatively high plasticity calculated above 20%. Contributions of the several strengthening mechanism for Al-4.5%Mg alloys are discussed in detail in paper [17]. It has been shown that among all possible strengthening options, grain refinement is a major strengthening mechanism observed in UFG Al-Mg materials.

It is well recognized that superplastic behavior can greatly enhance formability of the alloys. Typically in the 5xxx alloy series strain rate sensitivity parameter m can be as high as 0.5 with possible tensile test elongations up to 1000% [14]. Such values are usually obtained for the high temperature range of 500°C to 550°C and strain rates equal or below 10^{-2} s^{-1}.

Based on the literature data, majority of Al-Mg superplastic experiments are conducted on TMP materials with the grain size in range of 3μm to 15μm [11-14]. Structural stability during such condition is major problem and it is amplified in the regime of submicron grain size. In this paper it is shown that there is no significant change of properties during isothermal annealing at 450°C of RS material. Literature data indicate that TMP or SPD technologies can greatly refine grain size, however sufficient refinement of primary intermetallic compounds cannot be achieved by those techniques. Contrary, effective dispersion and uniform distribution of nano-scale intermetallic particles is possible by means of rapid solidification technique as shown in Fig. 1a. It is known that fine scale intermetallic phases can act as the pining sites preventing grain boundary migration at high temperatures which have beneficial effect on the thermal stability of the alloy. However, hot deformation experiments conducted at the same temperature showed 10% loss of alloy hardness. It is clear that combine effects of temperature and deformation leads to the structural changes towards material equilibrium conditions i.e. grains or intermetallic phases coarsening. Consequently, in order to avoid undesirable structural changes of UFG 5083 alloy temperatures above 450°C should be avoided. Experimental results of thermal stability are consistent with those obtained for 5083 alloys stabilized by the Zr and Sc additions [10, 15, 16]. It was found that distribution of nano-scale Al_3(Sc,Zr) phases formed during thermo-mechanical treatment of the alloy is very similar to the distribution Mn and Cr rich phases that are formed during rapid solidification and hot extrusion.

Performed experiments showed higher strain rate sensitivity for the RS material (m=0.3) in comparison to the IM alloy (m=0.2). It is clearly seen that the grain size has a strong influence on strain rate sensitivity parameter (Fig. 6, 7). Slight variation of slope inclination at about 300°C can be observed for (Fig. 7) IM material with medium grain size of 15μm, whereas for RS material with 700 nm grain size a significant change around 200°C can be noticed (Fig. 6). Above those temperatures there is a substantial increase of "m" parameter (especially in RS case) that is connected to activation of alternative deformation mechanism involving processes at grain boundary regions.

5. Conclusions

Rapid solidification was found to be effective technique for refining structural components in 5083 alloy. Highly refined intermetallic particles as well as grain size were found to be beneficial to the improvement of mechanical properties. Furthermore, increase of mechanical properties had no negative influence on the material plasticity.

Ultra fine grained structure revealed opposite effect on the material strength at elevated temperatures. Activation of the alternative deformation mechanism connected to the grain boundary sliding resulted in significant decrease of flow stress. Strain rate sensitivity experiments indicate on superplasticity behavior of RS material, particularly during high deformation rates (10^{-2} s^{-1} to 1 s^{-1}) and high deformation temperatures (450°C).

Rapidly solidified and extruded 5083 alloy revealed high resistant to the recovery processes. This effect was attributed to the presence of very fine (below 50nm in size), highly dispersed intermetallic compounds which effectively suppresses grain boundary mobility at elevated temperatures.

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