SUPERPLASTICITY OF MAGNESIUM ALLOYS

Greger M.[1], Kocich R .[1], Kander L .[2]
[1]VSB-Technical University of Ostrava, Faculty of Metallurgy and Materials Engineering, Department of materials forming, 17.listopadu 15, 708 33 Ostrava Poruba, Česká republika
miroslav.greger@vsb .cz
[2]VÍTKOVICE - Research institute, Pohraniční 693/31, 706 02 Ostrava - Vítkovice, Czech Republic

SUPERPLASTICITA HORČÍKOVÝCH SLITIN

Greger M.[1], Kocich R.[1], Kander L.[2]
[1]VŠB-Technická univerzita Ostrava, Fakulta metalurgie a materiálového inženýrství,katedra tváření materiálu, 17.listopadu 15, 708 33 Ostrava Poruba, Česká republika
miroslav.greger@vsb.cz
[2]VÍTKOVICE-Výzkum a vývoj, spol. s r.o., Pohraniční 693/31, 706 02 Ostrava - Vítkovice, Česká republika

Abstrakt

Příspěvek je zaměřen na problematiku tváření hořčíkových slitin na bázi Mg-Al-Zn s odstupňovaným obsahem Al. Tyto slitiny jsou známé svou obtížnou tvařitelností, která plyne z jejich krystalografického uspořádání. Avšak při využití vhodných metod tváření, jako například SPD procesů, lze dosáhnout i u těchto slitin velice dobrých výsledků při zaměření na jejich finální mechanické vlastnosti, a to nejen pevnostní, ale i plastické. V experimentu byly využity klasické technologie tváření a rovněž technologie SPD - ARB a ECAP. Bylo dokázáno že i takto obtížně tvařitelné materiály mohou dosahovat velmi vysokých plastických vlastností. Jak plyne ze závěrů tohoto příspěvku, po provedené plastické deformaci vykazovaly použité materiály ze slitin AZ61 a AZ91 superplastické chování, což potvrzují i dosažené výsledky kdy, tažnost do přetržení u slitiny AZ91 činila 418 %. Tento experiment prokázal rovněž velký vliv předchozí plastické deformace na konečné hodnoty mechanických vlastností. Jak bylo ověřeno daleko příznivější výsledky přináší válcování ve více krocích oproti válcování v jediném průchodu. Při využití technologie ECAP byl získán materiál o jehož velikost zrna činila $d \approx 0{,}7\mu m$. Taktéž zařazení nižší teploty při posledním průchodu skrze matrici způsobuje získání vyšších finálních vlastností. Naproti tomu technologie ARB umožnila získání materiálu o velikosti zrna v rozmezí $d \approx$ 1-10 µm. Nicméně druhá technologie s sebou přinesla vyšší pevnostní vlastnosti. Již 3 cykly byly dostačující ke snížení původní velikosti zrna pod hranici 10 µm.

Abstract

The paper is focused on issues of processing of non-ferrous metals in practice, namely on Mg-Al-Zn based Magnesium alloys with graded Al contents. It is well known that forming of these alloys is difficult since this feature is caused by their crystallographic arrangement. Nevertheless, application of appropriate methods of forming, such as e.g. some of SPD processes, makes it possible to achieve even in these alloys with comparatively very low formability very good results with orientation on their final mechanical properties, not only strength but also proven plastic properties. Methods ARB and ECAP were used in the described experiment. It was proved that hardly forming materials could achieve very high plastics properties. After making plastics deformation, the using materials of alloys AZ61 and AZ91 analysed superplastics behaviour, it was certified by obtaining results, when ductility to rupture of alloy AZ91 was 418 %, it is demonstrated at conclusion of the article. The experiment proved big influence of previous plastics deformation to ending values of mechanical properties. It was verified that better results are at rolling in more steps compared to rolling in one pass. It was obtained the material about grain size d ≈ 0,7μm during using the technology of ECAP. The low submission temperature at last pass through die, it causes obtaining higher final properties. Abreast of it the technology ARB enabled to get material of grain size in interval d ≈ 1-10 μm. The second technology brings higher strength properties. Only 3 cycles were sufficient to lower original grain size under limit 10 μm

Keywords: superplasticity, ARB, rolling, structure and properties

1. Introduction

Ratio of exploitation of magnesium based materials very rapidly increases at present. This is given not by its service properties, but also by its very low mass and also certain possibility of its use as replacement of Al based materials. Production of final products made of Mg alloys is, however, accompanied by many factors, which must be mastered for its successful implementation into practice [1]. These issues comprise among others the problems related to forming of these alloys, i.e. the problems ensuing for their crystallographic substance, such as small number of slip planes or occurrence of inter-metallic phases, which deteriorate formability [2,3]. Partial contribution to solution of these problems, apart from metallurgical modifications, consists also in unconventional methods of forming based on SPD processes, which can be a certain variant of elimination of some of existing drawbacks of classical forming processes [4,5].

2. Experiment

Forming of Mg-Al-Zn alloys mentioned above (namely AZ91 after T4) was realised by conventional way, i.e. by rolling, Fig. 1. There were, nevertheless, used tow different ways of rolling in order to enable determination of differences of different approach at deformations as such. These rolled products were in the next stage subjected to the technology of Equal Channel Angular Pressing (ECAP) [6]. Materials processed in this manner were subjected to a hot tensile test for determination of the obtained mechanical values.
Another SPD method that was used was the ARB technology, which was applied on alloys AZ91+T4, AZ61+T4. Table 1 gives their chemical composition.

Table 1 Chemical composition of used alloys

Alloy	Chemical composition %									
	Al	Zn	Mn	Si	Cu	Fe	Sn	Ni	Pb	Ce
AZ91-A	8.95	0.76	0.21	0.041	0.003	0.008	0.01	0.003	0.059	0.01
AZ61-B	5.92	0.49	0.15	0.037	0.003	0.007	0.01	0.003	0.034	0.01

2.1 Conventional rolling and ECAP

Materials made of the alloy AZ 91 (Fig.2) and AZ 91+T4 (Fig. 3), which were first rolled by:

a) single pass

b) 3 passes with intermediate heating to rolling temperature, Fig. 4.

and then pressed, were subjected to hot tensile test in order to determinate a possibility of super-plastic behaviour. Equal channel angular pressing was made in two stages. The first stage consisted of 4 passes at the temperature 250°C. It was followed by the second stage consisting of 1 pass at the temperature 180°C (Fig. 5). The samples were similarly as in the previous cases re-heated to the chosen forming temperature in a muffle furnace with connected inert atmosphere Ar_2 [7]. After obtaining of the required temperature and a 5-minute dwell at this temperature the material was charged into thermally insulated matrix with resistance heating, the temperature of which was identical to that of the chosen forming temperature.

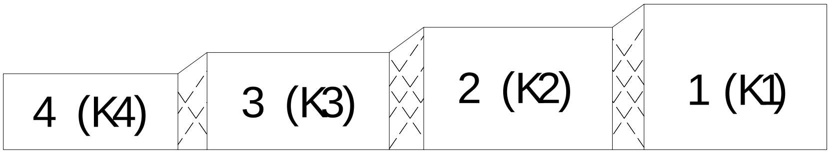

Fig.1 Shape of samples prior to rolling

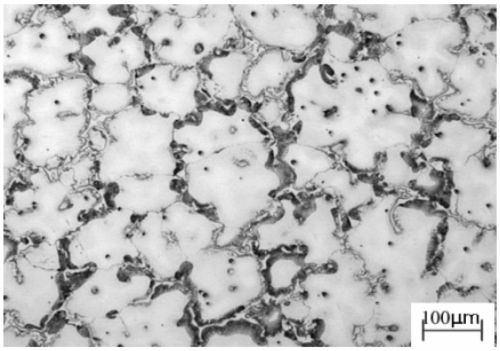

Fig.2 AZ91 alloy without T4

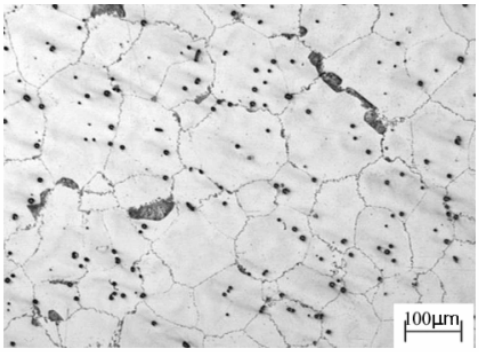

Fig.3 AZ91 alloy after T4

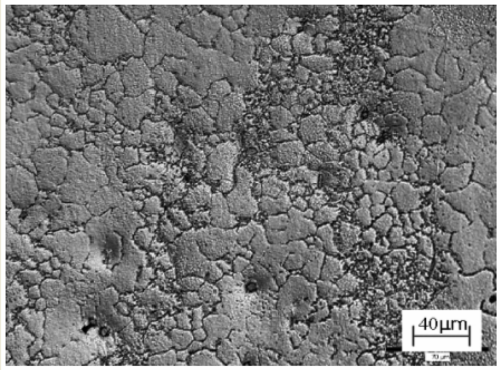

Fig.4 AZ91 + T4 after 3rd pass rolling

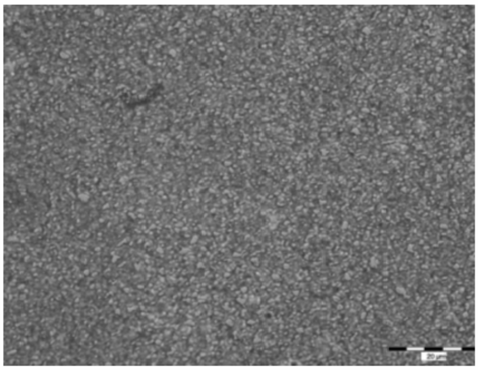

Fig.5 AZ91 + T4 after rolling and ECAE

2.2 Hot Tensile test

Temperature used at the tensile test was 250 °C and strain rate was $\dot{e} = 2x10^{-4}$. The samples obtained after processing by ECAP technology were adjusted to the required shape and then subjected to the tensile test (Fig. 6), during which the set temperature was controlled by PID regulator, which used a thermo-couple situated directly on the tested sample. Material rolled first by single pass (I, II, III) and then pressed, achieved elongation of approx. 200 %, while materials first rolled by several passes and then pressed, achieved elongation of up to 413 % [8]. Before the tensile test microstructures of both groups did not differ significantly from each other.

Table 2 Values of strength and elongation of AZ 91 alloy +T4 after ECAP

Marking of sample	AZ 91 + T4	
	Elongation [%]	UTS [MPa]
I	294	15
II	286	19
III	-	-
K1	418	28
K2	384	32
K4	358	58.7

Table 2 gives obtained values of elongation in individual samples after hot tensile test, where there are apparent the differences mentioned above between various methods of rolling applied prior to application of the ECAP technology, which has important influence on final plastic properties of obtained materials. An increase of plasticity with growing applied deformation can be observed at rolling by both methods, i.e. at rolling by single pass and rolling by several passes. In the latter case the obtained ductility was higher, which was probably caused by more homogenous structure obtained byre-crystallisation processes, which at this type of rolling could have developed more than at single-pass rolling.

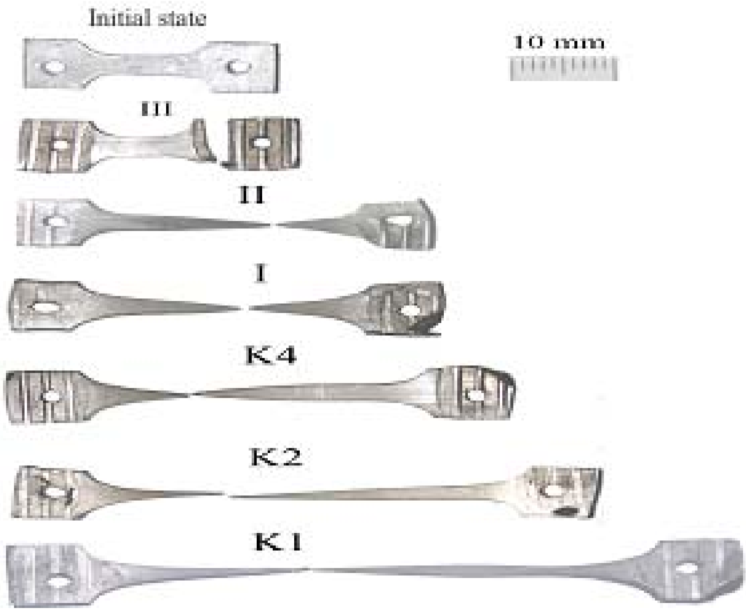

Fig.6 Samples of AZ91+T4 alloy after ECAP and hot tensile test

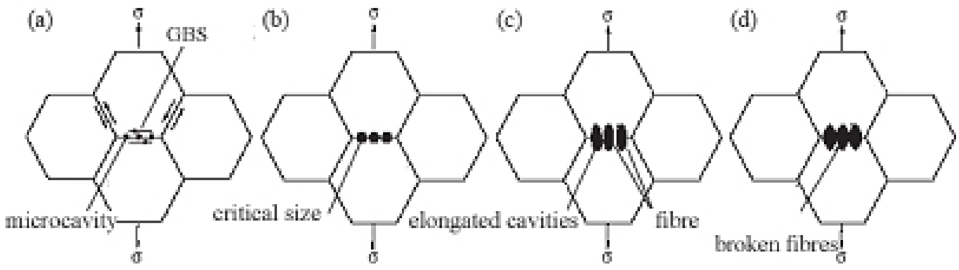

Fig.7 Mechanism of rupture at super-plastic forming

Due to the fact that slip along grain boundaries is under such conditions of deformation in these alloys one of the main deformation mechanisms, it seems that micro-cavities were formed at the grain boundaries, which gradually grew into "O-shaped" cavities [9-11]. These cavities afterwards protruded during plastic deformation as places of concentration of strain, and since they affected these original micro-cavities, as it can be seen in Fig. 7, there occurred elongation of edges of these cavities in the form of fibres, which broke after exceeding their strength. Resulting rupture that occurred at plastic deformation was caused by joining of individual cavities after breaking of elongated fibres.

Sample taken from the alloy AZ 91 elongated at the temperature of 250°C under constantly applied strain around the value of 15 MPa to rupture, as it is demonstrated in Fig. 8.

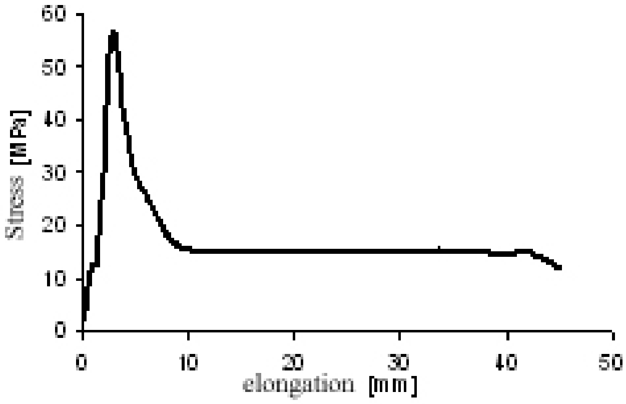

Fig.8 Diagram of ECAPed AZ91 alloy after hot tensile test

2.3 ARB

For experimental verification of the ARB process there were produced two strips from the alloys AZ91+T4, AZ61+T4, which served as initial material. Initial dimensions of each strip were the following: thickness 4 mm, width 50 mm and length 200 mm. Experiment was made at the temperature of 380°C. The heat distortion temperature for this technology was chosen also with respect to the results of previous experiment, at which gradual samples were rolled. The samples were rolled at the first pass by deformation of 62.5% in direction of height [12]. In all other passes by 50% height deformation. Strain rate varied in the interval from 16.83 to 17.78 s^{-1}.

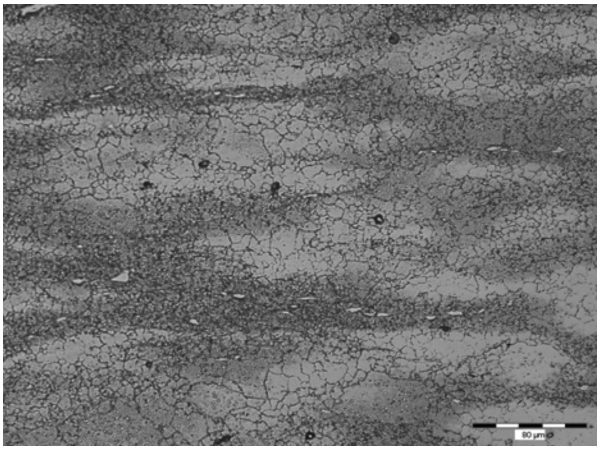

Fig.9 Final microstructure of AZ91 after 5th pass at ARB process

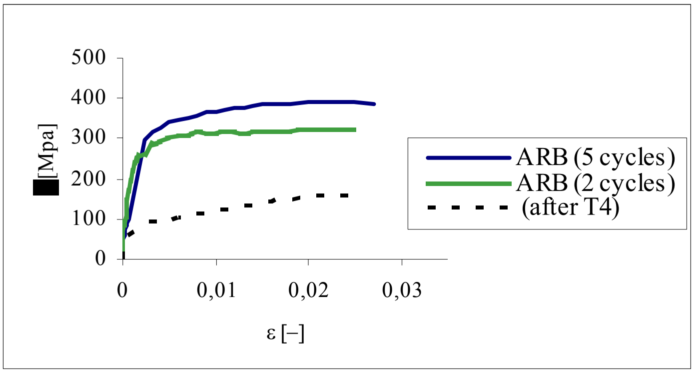

Fig.10 Mechanical properties of AZ91 at the temperature 360°C

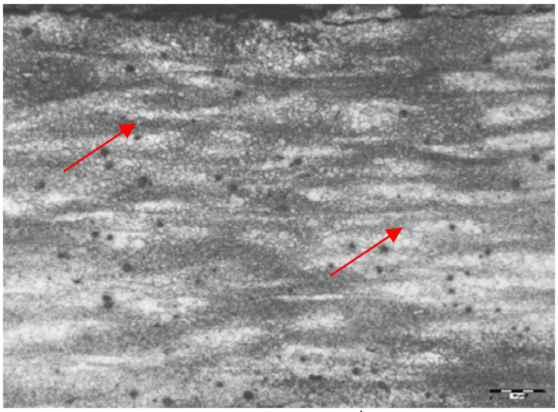

Fig.11 Final microstructure of AZ61 after 3rd pass at ARB process

High efficiency of this process is demonstrated also in the Fig. 10, which shows growth of strength of the alloy AZ91 in dependence on number of realised cycles in relation to the original non-deformed state. The values of strength increased more than 2.5 times after five accomplished cycles.

At several places marked by an arrow there are visible traces of original boundaries of individual rolled layers, which have mostly disappeared. This was observed both for the alloy AZ91 and the alloy AZ61. Number of visible places of original boundaries decreased with increasing number of accomplished cycles.

As it is demonstrated by the enclosed photos, there can be seen evident traces of crystallisation (Figs. 9, 11), which refined the structure already after 3 cycles almost 20x, if we take into consideration the original structure with average size of 120 μm (Fig. 3). Micro-structure of rolled materials indicates formation of new grains inside the original grains, elongated in direction of rolling. Central parts of the rolled product are represented by fine-grain structure more than surface parts. The original boundaries disappeared at many places and new grains began to form at their place. The figures 12 and 13 demonstrate that interposed deformation at the ARB process sufficed already after the 3rd cycle for decreasing of the grain size from the original size down under 10 μm in both types of alloys.

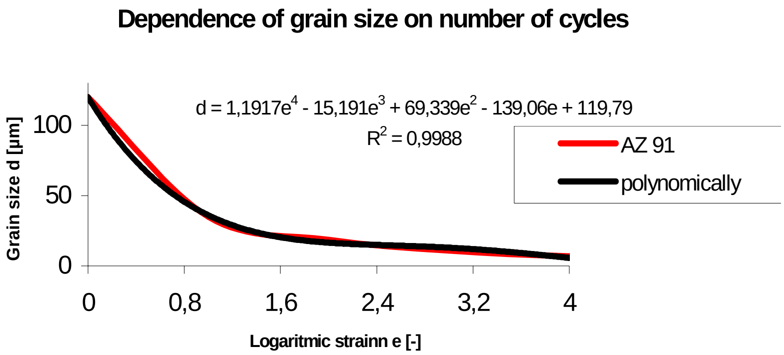

Fig.12 Grain size on logarithmic strain dependence of AZ91 alloy at ARB process

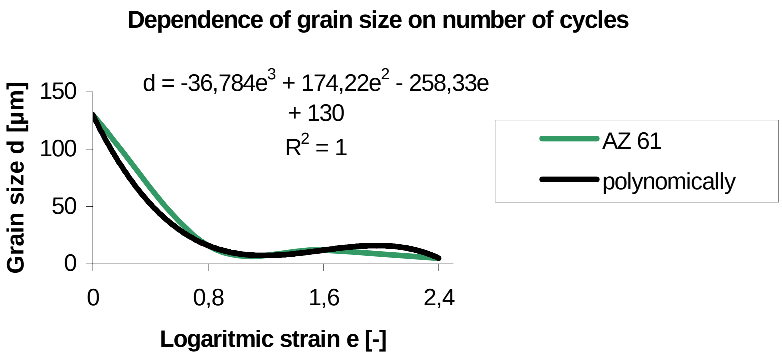

Fig.13 Grain size on logarithmic strain dependence of AZ61 alloy at the ARB process

Comparison of obtained strength in individual types of alloys after application of various forming technologies is shown in Fig. 14. It is evident, that the best method for obtaining the highest values of strength is the ARB process, however, this is achieved at the expense of plastic properties. Contrary to that the ECAP technology is an optimum compromise.

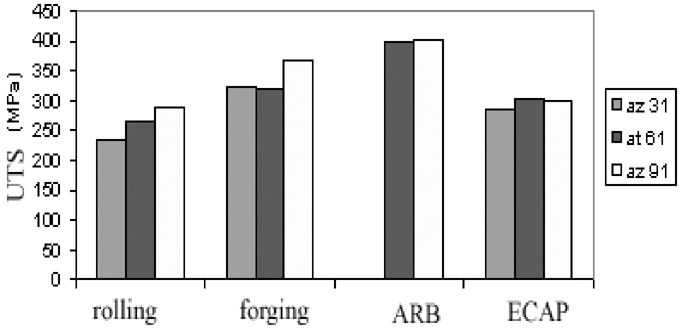

Fig.14 UTS of Mg alloys at different technologies

3. Conclusion

It is evident from micro-structures and mechanical tests that at high temperatures big elongation and lower strength are achieved after ECAP in comparison with conventional methods of forming, which is caused probably by the following factors:

1) There occurred disintegration of original precipitates to small particles, which facilitated movement of dislocations (e.g. by transversal slip), resulting in recovery of micro-structure.
2) Comparatively small grain size, which enables slip deformation mechanism at the grain boundaries.

It means that during plastic deformation realised by the ECAP technology there occurred disintegration of staminate precipitates. There is also obvious occurrence of precipitates in the form of formations, the size of which exceeded 10 μm, but only in materials that were rolled by single pass. In materials rolled by several passes the distribution of precipitates is comparatively homogenous, with decreasing magnitude of deformation there is visible a growing proportion of longer staminate formations, which did not disintegrate into these smaller particles, which indicates also influence of magnitude of previous deformation at rolling.

It was therefore proved that the used ARB technology is a perspective tool for obtaining of highly fine-grain structures in Mg-Al Alloys. It contributes at the same time to homogenisation of micro-structure and to substantial limitation of negative consequences of dendritic segregation on mechanical properties of these alloys.

Acknowledgements

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