DEVELOPMENT OF STRUCTURE AND MECHANICAL PROPERTIES IN AZ91 ALLOY USING BY ARB PROCESS

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VÝVOJ STRUKTURY A MECHANICKÝCH VLASTNOSTÍ U HOŘČÍKOVÉ SLITINY AZ 91 PŘI POUŽITÍ ARB PROCESU

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Abstrakt

ARB proces je jeden z procesů založen na využití velké plastické deformace, který může plynout v dosažení vysoce pevných materiálů s ultra jemnozrnou strukturou. Změny mikrostruktury a mechanických vlastností pásků z hořčíkové slitiny AZ91, vyrobených metodou kumulovaného válcování při teplotě 360°C byly cílem tohoto experimentu. Mikrostruktura válcovaných materiálů ukazuje na vznik subzrn uvnitř původních zrn, které vykazují stopy deformace, jež je protáhla ve směru válcování.V takto vyválcovaných materiálech se místně vyskytovaly oblasti s velikostí zrna kolem 1 μm, přičemž tímto procesem vzrostla pevnost až na 392 MPa. Pevnost slitiny AZ91 se zvýšila z původních 168 MPa (bez deformace a po tepelném zpracování) na 334 MPa po 4 cyklech, s rostoucím počtem průchodů však už nebyl nárůst pevnosti tak výrazný. Provedený experiment ukázal, že po 5 cyklech klesla střední velikost zrna z původních 120 μm až na 3,6 μm. Typická dvojčata, která vznikala při plastické deformaci v případě kování a konvenčním podélném válcování, se při použití ARB technologie nevyskytovala, což je pravděpodobně způsobeno velikostí deformace, která byla rozhodujícím činitelem již při prvním průchodu kde převýšila hodnotu 60%, naproti tomu při kování či válcování to nebylo více než 30%.

Abstract

Accumulative rolling bonding (ARB) is a kind of severe plastic deformation process which can produce high strength metals with ultra fine (sub-micron) grained microstructure. Microstructure and mechanical properties of AZ91 alloy sheets manufactured by accumulative roll bonding (ARB) at temperature 360°C were investigated. Microstructure of rolled materials indicates formation of sub-grains inside the original grains, which show traces of deformation, which elongated them in direction of rolling. In the AZ91 sheet, the grain size was locally reduced down to 1 μm by the ARB process and the strength increased up to 392 MPa, The strength of the AZ 91 alloy increased from 168 MPa (without deformation and after heat treatment) to 334 MPa after the 4 cycles, but during subsequent ARB processing it rose only very slightly. Experimentally was proved that the mean grain size after 5 cycles decreased from initial 120 μm to 3,6 μm. Typical twins, which were formed at plastic deformation in case of forging and conventional longitudinal rolling did not occur when ARB technology was used, which is probably caused by amount of deformation, which was the decisive factor already at the first pass, where it exceeded the value of 60%, while on the other hand at forging or rolling this amount of deformation was not higher than 30%.

Key words: Plastic deformation, grain size, magnesium alloy, mechanical properties.

1. Principles of technology

Accumulative roll-bonding (ARB) is the only process based on use of severe plastic deformation (SPD process), which uses deformation by rolling. It was discovered in 1998. Its principle is shown in Fig. 1. Rolling is the most advantageous process of metal processing for continuous production of heavy plates, thin sheets and bars. Total reduction of material is, however, substantially limited due to the fact that transverse dimension s of material shrink with increasing pass reduction. In case of the verified ARB process thin sheet was rolled by applying 50% deformation and it was afterwards divided to two sheets, which were stacked up on each other in such a way that original thickness was obtained, and then the sheet was rolled again. In order to ensure good bonding surface of material was degreased prior to stacking and cleaned by emery paper. Rolling was performed at high temperatures, below the temperature of material re-crystallisation. Due to the fact that it is possible to repeat these procedures without limitations, it is possible to achieve by the ARB process extremely high plastic deformations of material. At the beginning number of sheets entering the ARB process for processing by *n*-cycles is 2, resulting number is then 2^n. For example in case of application of 10 cycles initial number of sheets is 1024 and average thickness of initial sheet is less than 3 mm. Due to the fact that deformation intensity according to Mieses at 50% deformation is 0.80, overall deformation intensity after *n* cycles is 0.8 *n.* Table 1 gives changes in geometry of samples in the course of the ARB process.

Table 1 General regularities of ARB process

No. of cycles	1	2	3	4	5	6	7	8	9	10	11	*n*
No. of layers	2	4	8	16	32	64	128	256	512	1024	2048	2^n
No. of joining boundaries	1	3	7	15	31	63	127	255	511	1023	2074	2^n-1
Layer distance [μm]	500	250	125	62,5	31,2	15,6	7,8	3,9	1,9	0,96	0,48	$1000/2^n$
Total strain [%]	50	75	87,5	93,8	96,9	98,4	99,2	99,6	99,8	99,9	99,99	$1-(1/2^n)\cdot 100$
Corresponding strain	0,8	1,6	2,4	3,2	4,0	4,8	5,6	6,4	7,2	8,0	8,8	$(2/\sqrt{3}\cdot \ln 2)\cdot n=0,8n$

Table 2 summarises results of experiments on selected materials after the ARB process, assuming that two sheets with thickness of 3 mm are stacked on each other and then rolled with application of 50% deformation per one cycle.

Surfacing is unavoidable for obtaining of good bonding. During one pass of the ARB process there exists critical deformation (pass reduction), below which it is difficult to obtain sufficient for bonding. This degree of critical deformation depends on material and temperature of forming. It is usually necessary to have the relative pass reduction of at least 35 %; so far the ARB process is comparable with conventional rolling. What concerns requirements to rolling equipment, the ARB process does not require apart from stand of rolling mill with sufficient capacity for realisation of single-pass reduction any special equipment. Material fracture present serious problem of the ARB process. Due to the fact that big amount of total plastic deformation is accumulated in material and rolling is not a hydrostatic process, there sometimes occur cracks on edges of sheets, particularly at greater number of realised cycles. In case of highly ductile materials, such as e.g. pure aluminium, it is possible to obtain sheets with dimensions 3 mm (thickness) x 50 mm (width) x 300 mm (length) with ultra fine grain (UFG) without forming of cracks also in laboratory conditions.

Table 2 Resulting properties of selected materials after ARB process

Material	ARB process	Microstructure	Grain size [μm]	Strenght [MPa]
4N-Al	7cycles at Tr	„pancakes"-ultrafine grain	0,67	125
100-Al(99%Al)	8 cycles at Tr	„pancakes"-ultrafine grain	0,21	310
5052-Al(Al-2,4 Mg)	4 cycles at Tr	Ultrafine lamellas	0,26	388
5083-(Al - 4,5 Mg - 0,57 Mn)	7 cycles at 100°C	Ultrafine lamellas	0,08	530
6061-Al(Al- 1,1 Mg - 0,63 Si)	8 cycles at Tr	Ultrafine lamellas	0,10	357
7075-Al(Al-5,6Zn-2,6Mg-1,7Cu)	5 cycles at 250°C	„pancakes"-ultrafine grain	0,30	376

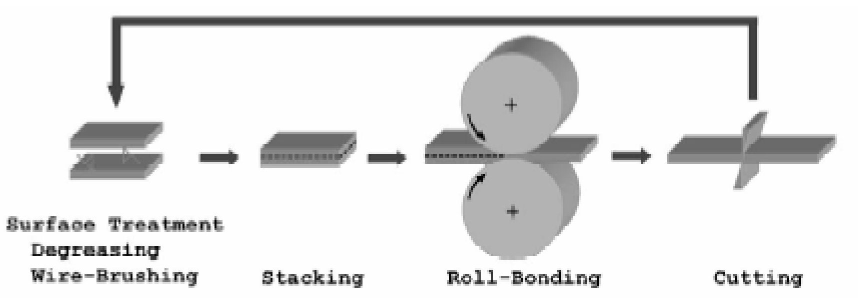

Fig.1 Schematic illustration showing the principle of the ARB process

1.1 Materials processed by ARB process

Materials processed by several cycles of the ARB process have fully ultra-fine grain (UFG) structure, containing mostly ultra-fine grain, size of which is less than 1 μm. Boundaries between grains are distinct, but irregular, and number of dislocations inside grains seems to be small. Results of these observations are similar as in case of severely deformed materials, obtained by different processes of severe plastic deformation (SPD processes). The most characteristic feature of materials with ultra fine grain processed by the ARB process is their directional structure (elongated grains of the type „pancakes"). Grains are elongated in direction of rolling. These microstructures resemble lamellar structures observed in severely deformed materials. It is confirmed unequivocally that, elongated UFG structures are not formed by sub-grains, but by grains with high-angle boundaries. High density of these boundaries of grains in materials processed by the ARB process was confirmed also in macro-areas by SEM/EBSD analysis (scanning electron microscopy / energy-dispersive spectrometry). Mechanism of formation of UFG structures during SPD processes is still being discussed. Recent research works indicate, however, that formation of UFG does not occur by conventional discontinuous re-crystallisation, but by continuous re-crystallisation (or by re-crystallisation „in-situ"), characterised by further division of ultra fine grains, and by migration of grain boundaries at short distance. Materials processed by the ARB process demonstrating elongated UFG structures have very high strength. Grain sizes and corresponding ultimate tensile strengths of various UFG materials produced by the ARB process are given in table 2. In most cases the average thickness of grain of elongated UFG structures UFG (type „pancake“) or ultra-fine lamellar structures varies in the range from 100 to 200 nm. High pure materials have tendency to show bigger size of grains. Similar materials processed by the ARB technology at lower temperatures show smaller grains. UFG materials have ultimate tensile strength twice up to four times higher than initial materials with conventional grain size. On the other hand materials processed by the ARB process have limited ductility (as expressed by tensile test parameter) due to early plastic instability.

1.2 Deformation in materials processed by the ARB process

It follows from the above text that materials with UFG microstructure produced by the ARB process have many common features with severely deformed materials with lamellar structure rolled in conventional manner. However, contrary to conventional rolling the shearing strain in UFG materials does not only concentrate to sub-surface layers, but it penetrates in a complex manner through the sheet thickness in the course of several cycles of the ARB process (Fig. 1). Due to the fact that shearing strain does not lead to change of material thickness, the consistent area of shearing strain should „add-up“ to the sheet deformation intensity. In other words, intensive deformation applied in n cycles of the ARB process without lubrication is much higher than 0.8 n, which was calculated just from reduction of thickness. Substantially bigger cumulated deformations must be one of the causes of more rapid development of UFG structures in materials subjected to processing by the ARB process. It is also necessary to take into account influence of changed path penetration of deformation. In materials processed by the ARB process there occur rather complex combination of plane strain and shearing strain, in dependence on position of the concrete place in material along its thickness and on number of cycles. It means that deformation path in the given zone of material in each cycle changes substantially between shearing strain and compressive plane strain. As a result of this the central part of sheet shows comparatively weak texture, even after big pass reduction at rolling. Although the role played by deformation path at grain refinement is still being discussed, the existing results of research of the ARB process indicate, that change of deformation path influences formation of UFG, in other words, it influences more rapid division of ultra-fine grains.

Possible explanation of the fact, that the ARB process is more advantageous for grain refinement than conventional rolling, lies therefore in „excess“ shearing strain. Roll-bonding at the ARB process is usually performed without lubrication. It is known, that at rolling without lubrication, or under conditions of minor lubrication, large part of excess shearing strain is realised in sub-surface zones of sheet. This causes also quite different texture than in central zones. Previous observations of structural development [1] during ARB demonstrated formation of lamellar structure at high amounts of deformation, which indicates similar development as at conventional rolling, where also the distance between the grains decreases continuously with growing deformation, however, in materials after the ARB process the distances were always smaller than after conventional rolling, although levels of deformation were equivalent.

2. Experimental

For experimental verification of the ARB process there were produced two strips from the alloy AZ91+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 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. In all other passes by 50% height deformation. Strain rate varied in the interval from 16.83 to 17.78 s^{-1}.

3. Results and discussions

After exiting from rolls there were visible cracks formed particularly on the sample edges and ends, but they did not penetrate more deeply than 10 mm from the edge to the centre. It was discovered at measurement of sample temperature with use of infra-red thermometer IR 2C 300 directly in the rolling gap, that temperature fall caused by heat removal through rolls was partly eliminated by generation of deformation heat. A samples was taken from the rolled material after each pass. It served for determination of changes in microstructure in thus formed material. The enclosed photos document visible traces of re-crystallisation, which refined structure already after 3 cycles almost 20 times in comparison with the initial structure with average grain size of 120 μm (Fig. 3). Microstructure of rolled materials (Fig. 4-8) indicates formation of sub-grains inside the original grains, which show traces of deformation, which elongated them in direction of rolling. Central zones of the rolled product are represented by ultra-fine grain structure from greater part than sub-surface zones, and original boundaries became at many places extinct and new grains began to be formed instead of them. Typical twins, which were formed at plastic deformation in case of forging and conventional longitudinal rolling did not occur when ARB technology was used, which is probably caused by amount of deformation, which was the decisive factor already at the first pass, where it exceeded the value of 60%, while on the other hand at forging or rolling this amount of deformation was not higher than 30%. This fact therefore shows, that plastic deformation occurred particularly by mechanism of grain slippage. Structure became more homogenised with the growing amount of deformation (number of passes) – recovered grains were formed in remaining parts of material, and grains size continued to shrink. It also followed from the results, that β phase ($Mg_{17}Al_{12}$) started to precipitate at increasing number of places with increasing number of passes. Structure contained after the last (fifth) pass the zones of grains with size around d = 1μm. Fig. 10 show dependence of grain size on number of passes.

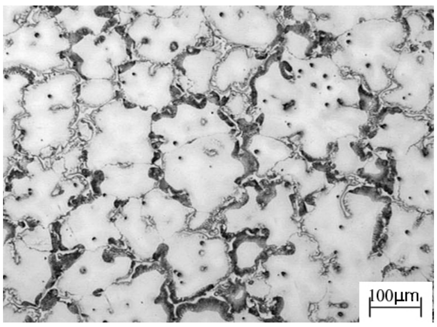

Fig.2 AZ91 (as cast state,) undeformed

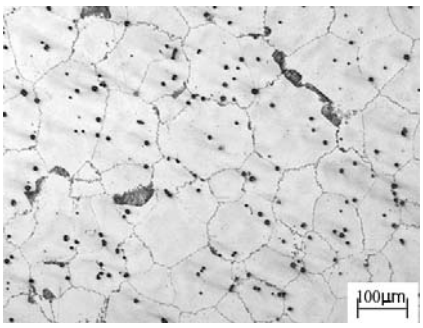

Fig.3 AZ91 (after T4) undeformed

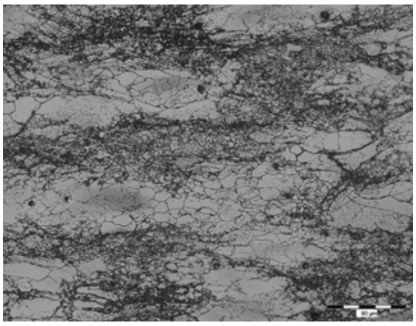

Fig.4 AZ91 after ARB (1pass)

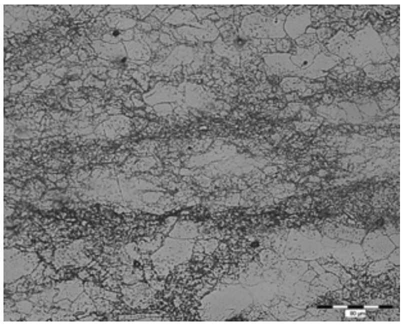

Fig.5 AZ91 after ARB (2 pass)

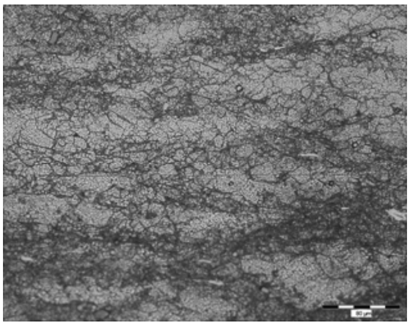

Fig . 6 AZ91after ARB (3 pass)

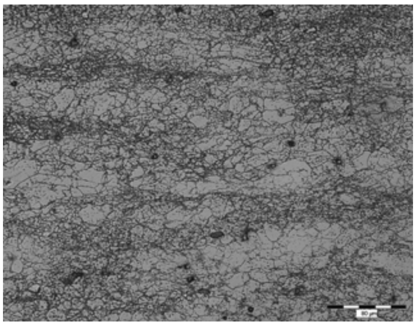

Fig . 7 AZ91after ARB (4 pass)

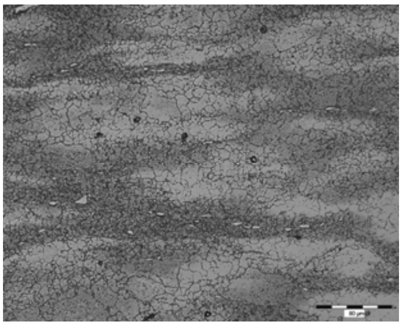

Fig.8 AZ91 after ARB (5 pass)

Fig.9 AZ91 po ARB (5 cycles)

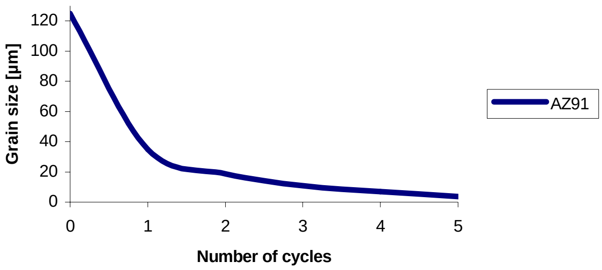

Fig.10 Change of grain size on number of passes

Full rolled materials were moreover evaluated by tensile test, at which influence of grain size on resulting mechanical properties was manifested itself. Strength of thus rolled materials achieved after 2 passes the value of 323 MPa, and the fifth pass it was 392 MPa. This increase was not, however, as high as compared with the state before deformation (Fig. 3).

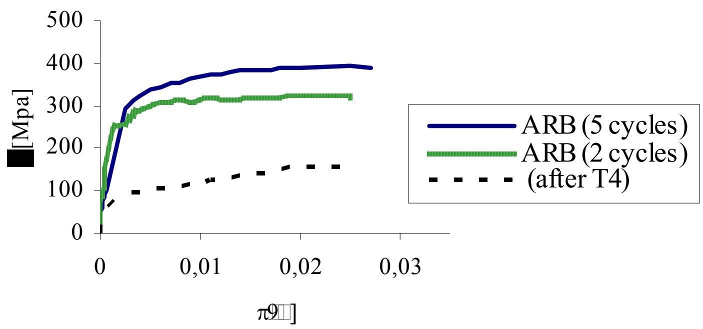

Fig.11 Mechanical properties of AZ91 at temperature 360°C

4. Conclusion

It is therefore obvious that this method appears to be highly efficient for obtaining distinctly better final mechanical properties, which reflect substantially reduced grain size in comparison with the original state (after T4). Evaluation of average grain size after each pass (cycle) confirmed the fact, that grain size in resulting structure decreases with increasing number of cycles. Already after the first cycle, in which the pass reduction was 62.5% there new grains at many places of matrix were formed, as documented in Fig. 4, and with increasing deformation the share of re-crystallised matrix increased and grain size continued to decrease down to final 3,6 μm.

Acknowledgements

This work was carried out under project number MSM 6198910013, funded by the Ministry of Education, Youth and Sports of the Czech Republic.

Literature

[1] Greger M., Kocich R.: Zpráva o řešení projektu za rok 2005, (IMPULZ FI-IM/033)
[2] Lee S.H., Saito Y., Tsuji N., Utsunomiya H., Sakai T.: Role of shear strain in ultragrain refinement by accumulative roll-bonding (ARB) process, Scripta Materialia 46, 2002, p. 283.
[3] Perez-Prado M.T., del Valle J.A., Ruano O.A.: Achieving high strength in commercial Mg cast alloys through large strain rolling, Materials Letters 59, 2005, p. 3231.
[4] Lee S.H., Sakai T., Saito Y., Utsunomiya H.: : Microstructures and mechanical properties of 6061 aluminum alloy processed by accumulative roll-bonding, Materials Science and Engineering A325 (2002) p. 228–235.