

M. KULCZYK*, J. SKIBA*, W. PACHLA*

MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AA5483 TREATED BY A COMBINATION OF ECAP AND HYDROSTATIC EXTRUSION

MIKROSTRUKTURA I WŁAŚCIWOŚCI MECHANICZNE STOPU AA5483 PO KOMBINACJI PROCESÓW ECAP ORAZ WYCISKANIA HYDROSTATYCZNEGO

Al-Mg alloys of the 5xxx series are strain hardenable and have moderately high strength, excellent corrosion resistance even in salt water, and very high toughness even at cryogenic temperatures to near absolute zero, which makes them attractive for a variety of applications, e.g. in systems exploited at temperatures as low as -270°C , and marine applications. The present study is concerned with the effect of a combination of 2 processes, which generate severe plastic deformation (SPD), equal channel angular pressing (ECAP) and hydrostatic extrusion (HE), on the microstructure and mechanical properties of an alloy that contain Al and Mg. The alloy was subjected to multi-pass ECAP followed by cumulative HE with a total true strain of 5.9. The microstructure of SPD samples was evaluated by transmission and scanning electron microscopy. The mechanical properties were determined by tensile tests and microhardness measurements. The combination of the two processes gave a uniform nanostructure with an average grain size of 70nm. The grain refinement taking place during the SPD processing resulted in the increase of the mechanical strength by 165% (YS) with respect to that of the material in the as-received state. The experiments have shown that the combination of HE and ECAP permits producing homogeneous nanocrystalline materials of large volumes.

Keywords: severe plastic deformation, hydrostatic extrusion, nanocrystalline structure, grain refinement, aluminum alloy

Stopy aluminiumu serii 5XXX (Al-Mg) umacniane odkształceniowo charakteryzują się relatywnie wysoką wytrzymałością, bardzo dobrą odpornością korozyjną szczególnie w wodzie morskiej i bardzo wysoką odpornością udarnościową nawet w temperaturach kriogenicznych. Własności te sprawiają, że te stopy są atrakcyjne dla wielu zastosowań gdzie wymagana jest praca w niskich temperaturach, nawet do -270°C oraz praca w środowisku morskim. W przeprowadzonych badaniach określono wpływ kombinacji dwóch procesów generujących duże odkształcenia plastyczne, przeciskania przez kanał kątowy (ECAP) oraz wyciskania hydrostatycznego (HE), na mikrostrukturę i własności mechaniczne stopu 5483. Zastosowano kombinację procesu kumulacyjnego wyciskania hydrostatycznego poprzedzonego procesem ECAP z łącznym odkształceniem rzeczywistym 5.9. Badania mikrostrukturalne zostały przeprowadzone z wykorzystaniem transmisyjnej mikroskopii elektronowej. Własności mechaniczne określono w statycznej próbie rozciągania oraz pomiarach mikrotwardości. Kombinacja obu procesów pozwoliła uzyskać materiał o jednorodnej nanostrukturze o średniej wielkości ziarna 70nm. Rozdrobnienie struktury spowodowało wzrost własności mechanicznych (granicy plastyczności) o około 165% w porównaniu do materiału przed odkształceniem plastycznym. Przeprowadzone eksperymenty wykazały że kombinacja procesów HE oraz ECAP pozwala na wytwarzanie jednorodnych nanomateriałów w dużych objętościach.

1. Introduction

Severe plastic deformation (SPD) is commonly applied to generate nanostructures in metals. SPD processes usually reduce the mean grain size to 100-500 nm (ultra-fine grained structures, UFG), and even below 100 nm (nanocrystalline structures, NC). They also allow achieving very promising combinations of high strength and good ductility [1, 2]. The main SPD techniques studied as applied to bulk metals include: high pressure torsion (HPT), equal-channel angular pressing (ECAP), multiple rolling, and cyclic extrusion-compression (CEC) [1, 3-6]. Recently, the hy-

drostatic extrusion (HE) method has been used for producing NC structures in such metals as, aluminum alloys, copper alloys, magnesium alloys, titanium, nickel and stainless steel [2, 7-12]. However, in the case of certain materials, e.g. pure copper, nickel, or the CuCrZr alloy, a combination of two SPD techniques, namely ECAP and HE, was necessary to obtain nano-/ultra-fine grained structures [10, 13]. The aim of the present study was to produce a nanostructure in the AA5483 alloy. According to the literature reports, a single SPD process is insufficient to obtain a nanostructure in the 5483 alloy [14]. The present study was concerned with the effect of a combination of two processes that induce severe plastic deformation

* POLISH ACADEMY OF SCIENCES, INSTITUTE OF HIGH PRESSURE PHYSICS (UNIPRESS), 29/37 SOKOŁOWSKA STR., 01-142 WARSZAWA, POLAND

Chemical composition of the 5483 aluminum alloy (wt%)

Mn	Mg	Si	Fe	Cu	Cr	Zn	Ti	Zr
0.55-0.1	4.5-5.0	max 0.2	max 0.15	max 0.05	max 0.2	max 0.2	max 0.05	max 0.2

(SPD), ECAP followed by HE, on the microstructure and mechanical properties of the Al-Mg alloy. The Al-Mg alloys of the 5xxx series are strain hardenable, and have a moderately high strength, excellent corrosion resistance even in salt water, and very high toughness even at cryogenic temperatures (to near absolute zero). These advantages make them attractive for a variety of applications, such as e.g. in systems exploited at temperatures as low as -270°C and marine applications.

2. Experimental procedure

The chemical composition of the 5483 aluminum alloy used in the experiments is given in Table 1. AA5483 samples in the as-received state were subjected to SPD processing using the two deformation paths:

1. Hydrostatic extrusion (HE) conducted at room temperature to a total cumulative true strain of 4.65,
2. 2 passes of equal channel angular pressing (ECAP, C route) followed by hydrostatic extrusion with a total true strain of 5.9.

In both cases the final diameter of the product was 5 mm. After the HE, the samples were water cooled at the die exit. The microstructures, before and after the deformation, were analyzed by transmission electron microscopy (TEM). The grain size was determined by measuring the equivalent diameter (d_2) defined as the diameter of the circle with a surface area equal to that of a given grain. The mechanical properties were evaluated by microhardness measurements under a load of 200g, and by static tensile tests conducted at room temperature at a strain rate of 10^{-4}s^{-1} . The microhardness (HV0.2) of the material was measured at transverse cross-sections of the rods.

3. Results and discussion

The material in the as-received state had a coarse grained microstructure with the average equivalent grain diameter of $10\mu\text{m}$, Fig. 1a. Both the SPD processes (HE and ECAP+HE) resulted in a significant refinement of the microstructure, but the two SPD microstructures differed substantially in their character. In the HE processed sample, the microstructure contained dislocation cells (with low misorientation angles between them) and highly heterogeneous regions, which is typical of materials subjected to conventional deformation, Fig. 1b, whereas the ECAP/HE sample had a structure with well developed nano-grains and pronounced grain boundaries with high misorientation angles, Fig. 1c. The grain/cells diameters were 250 nm after HE and 70 nm after ECAP+HE. The process combining the two SPD processes, ECAP and HE, applied to the AA5483 alloy, increased the deformation degree and changed the deformation paths, yielding a homogenous

nano-structure with equiaxial grains. The grain size distribution was typical of homogeneous fine nano-structures, Fig. 2. In our earlier studies [10, 13], a similar effect was observed in nickel, copper and CuCrZr alloy.

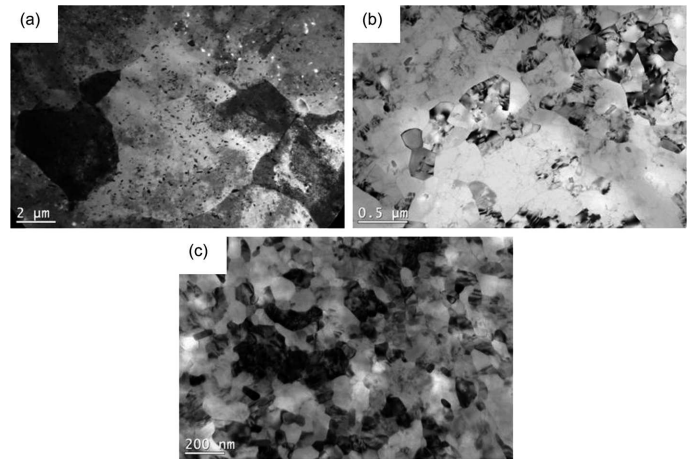


Fig. 1. Microstructure of the 5483 aluminum alloy: (a) in the as-received state, (b) after HE, and (c) after the combination of ECAP + HE

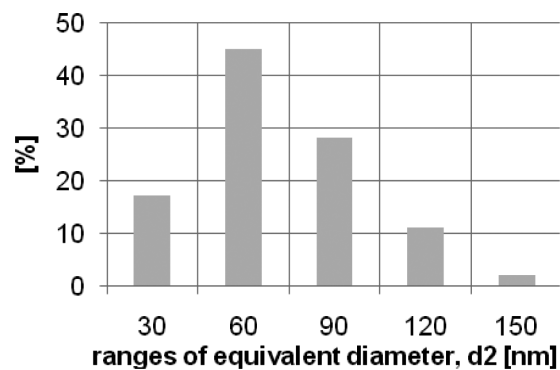


Fig. 2. Grain size distribution in the 5483 aluminum alloy treated by the combination of ECAP + HE with total (cumulative) true strain of 5.9

The results of the tensile test and microhardness measurements are given in Table 2. The sample in the as-received state has a low strength ($Y_S=220\text{MPa}$) with 15% elongation which can be attributed to its coarse grained microstructure. The strong grain refinement obtained in our experiments resulted in an increase of mechanical strength. The yield strength increased by 110% after HE and even by 165% after ECAP+HE, whereas the ductility remained moderate (after ECAP+HE it was 6.5%). This advantageous combination of the exceptionally high strength and reasonable ductility could be achieved thanks to the very small grain size (70 nm) which imparts grain boundary strengthening according to the well known Hall-Petch relationship, and to the presence of nanotwins in-

side the small grains, which are known to increase the ductility of NC materials [15]. The highest microhardness was achieved after the combined ECAP + HE processing, namely 182HV0.2, which was twice as high as that in the as-received state and higher than that obtained after each of these SPD processes applied independently. Figure 3 compares the mechanical properties of the 5483 alloy treated by the ECAP+HE combination obtained in our experiments with those obtained by various other methods reported in the literature [14]. The proposed technique which is a combination of these two SPD methods that use different deformation paths permits improving the mechanical properties of the material with respect to those achievable by the individual methods applied independently. This achievement can be attributed to the homogeneous nanostructure of the material obtained thanks to the proposed method.

TABLE 2
Mechanical properties of 5483 aluminum alloy obtained by various deformation path

Processes	UTS [MPa]	YS [MPa]	Elongation [%]	Microhardness HV0.2
as "received"	380	220	15	0
HE	504	458	10	135
ECAP	–	–	–	155
ECAP+HE	630	586	6.5	187

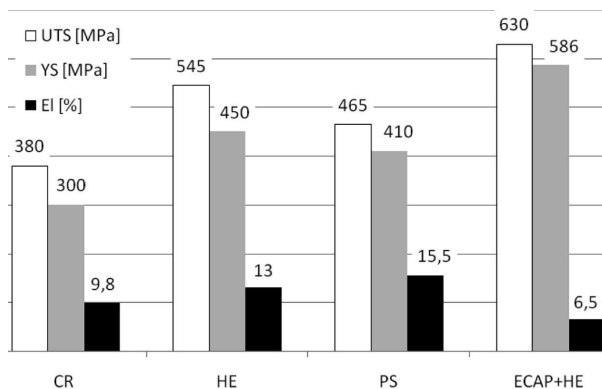


Fig. 3. Comparison between the properties of the 5483 aluminum alloy treated by various methods (CR – cold rolling, HE – hydrostatic extrusion, PS – plastic consolidation) [14] and combined ECAP+HE (our recent work)

4. Conclusions

It has been shown that the mechanical properties of the AA 5483 alloy can be significantly improved by refining its grains to the nanometric scale. To obtain a nanostructure (with the average grain diameter below 100 nm) with well developed high angle grain boundaries, a combination of ECAP and HE processing is required. The grain refinement taking place during the combined SPD processing results in the increase of the mechanical strength (YS) by 165% with respect to that in the as-received state. The mechanical strength achieved using the proposed combined technique is also better than that of

the material treated by the individual SPD methods separately. Our experiments have shown that the combination of ECAP with HE can be considered to be an effective method of the fabrication of unique engineering materials with excellent mechanical properties.

Acknowledgements

This work was carried out within a NANOMET Project financed under the European Funds for Regional Development (Contract No. POIG.01.03.01-00-015/08).

REFERENCES

- [1] R. Valiev, Nanostructuring of metals by severe plastic deformation for advanced properties, *Nature* **3**, 511-516 (2004).
- [2] M. Kulczyk, J. Skiba, S. Przybysz, W. Pachla, P. Bazarnik, M. Lewandowska, High strength silicon bronze (C65500) obtained by hydrostatic extrusion, *Archives of Metallurgy and Materials* **57**, 859-862 (2012).
- [3] V.M. Segal, Materials processing by simple shear, *Mater. Sci. Eng. A* **197**, 157-164 (1995).
- [4] R. Valiev, R.K. Islamgaliev, I.V. Alexandrov, Bulk nanostructured materials from severe plastic deformation, *Progress in Materials Science* **45**, 103-189 (2000).
- [5] A.P. Zhilyaev, B.K. Kim, J.A. Szpunar, M.D. Baro, T.G. Langdon, The microstructural characteristics of ultrafine-grained nickel, *Materials Science and Engineering A* **391**, 377-389 (2005).
- [6] M. Richert, J. Richert, B. Leszczynska-Madej, A. Hotlos, M. Maslanka, W. Pachla, J. Skiba, AgSnBi powder consolidated by composite mode of deformation, *Journal of Achievements in Materials and Manufacturing Engineering* **39**, 161-167 (2010).
- [7] W. Pachla, M. Kulczyk, A. Mazur, M. Sus-Ryszkowska, UFG and nanocrystalline microstructures produced by hydrostatic extrusion of multifilament wires, *International Journal of Materials Research* **100**, 984-990 (2009).
- [8] B. Zysk, M. Kulczyk, M. Lewandowska, K.J. Kurzydowski, Effect of heat treatment and hydrostatic extrusion on mechanical properties of a Cu-CrZr alloy, *Archives of Metallurgy and Materials* **55**, 143-149 (2010).
- [9] W. Pachla, A. Mazur, J. Skiba, M. Kulczyk, S. Przybysz, Wrought magnesium alloys ZM21, ZW3 and WE43 processed by hydrostatic extrusion with back pressure, *Archives of Metallurgy and Materials* **57**, 485-493 (2012).
- [10] W. Pachla, M. Kulczyk, M. Sus-Ryszkowska, A. Mazur, K.J. Kurzydowski, Nanocrystalline titanium produced by hydrostatic extrusion, *Journal of Materials Processing Technology* **205**, 173-182 (2008).
- [11] M. Kulczyk, W. Pachla, A. Mazur, M. Sus-Ryszkowska, N. Krasilnikov, K.J. Kurzydowski, Producing bulk nanocrystalline materials by combined hydrostatic extrusion and equal channel angular pressing, *Materials Science Poland* **25**, 4, 991-999 (2007).
- [12] P. Czarkowski, A.T. Krawczynska, R. Slesinski, T. Brynk, J. Budniak, M. Lewandowska, K.J. Kurzydowski, Low temperature mechanical properties of 316L type stainless steel after hydrostatic extrusion, *Fusion Engineering and Design* **86**, 2517-2521 (2011).

- [13] M. Kulczyk, B. Zysk, M. Lewandowska, K.J. Kurzydłowski, Grain refinement in CuCrZr by SPD processing, *Physica Status Solidi A* **207**, 1136-1138 (2010).
- [14] P. Bazarzik, M. Lewandowska, M. Andrzejczuk, K.J. Kurzydłowski, The strength and thermal stability of Al-5Mg alloys nano-engineered using methods of metal forming, *Materials Science and Engineering A* **556**, 134-139 (2012).
- [15] K. Lu, Novel properties of nanostructured metals, *Materials Science Forum* **475-479**, 21-30 (2005).

Received: 20 April 2013.