

J. BOGUCKA\*

## INFLUENCE OF TEMPERATURE OF ACCUMULATIVE ROLL BONDING ON THE MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AA5251 ALUMINUM ALLOY

### WPLYW TEMPERATURY AKUMULACYJNEGO WALCOWANIA PAKIETOWEGO NA MIKROSTRUKTURĘ I WŁASNOŚCI MECHANICZNE STOPU AA5251

The influence of bonding temperature on microstructure and mechanical properties of AA5251 alloy sheets have been analyzed in the paper. The alloy was deformed with the method of accumulative roll bonding (ARB) in various temperature conditions i.e. at ambient temperature up to 5<sup>th</sup> cycle ( $\varepsilon = 4.0$ ) and using pre-heating of sheet packs at 200°C and 300°C up to 10 cycles ( $\varepsilon = 8.0$ ). The deformed material was subjected to structural observations using TEM, measurements of crystallographic texture with the technique of X-ray diffraction and tensile tests.

It was established that the temperature of roll-bonding had a significant effect on the structure evolution and the observed changes of mechanical properties. High refinement of microstructure and optimum mechanical properties were obtained for the material processed at lower temperatures, i.e. at ambient temperature and pre-heating at 200°C. Recovery structure processes occurring during deformation were observed in the alloy bonded with pre-heating at 300°C and therefore mechanical properties were lower than for the alloy bonded at lower temperatures.

*Keywords:* accumulative roll bonding, microstructure, texture, mechanical properties, AA5251 aluminum alloy

W pracy analizowano wpływ temperatury spajania blach ze stopu AA5251 na zmiany zachodzące w mikrostrukturze i własnościach mechanicznych. Stop odkształcono metodą akumulacyjnego walcowania pakietowego w zróżnicowanych warunkach temperaturowych, tj. w temperaturze otoczenia do 5-przepustu ( $\varepsilon = 4.0$ ) oraz przy zastosowaniu wstępnego nagrzewania pakietów w 200°C i 300°C do 10-cykli ( $\varepsilon = 8.0$ ). Odkształcone próbki poddano obserwacjom strukturalnym z wykorzystaniem TEM, przeprowadzono pomiary tekstury krystalograficznej wykonanej metodą dyfrakcji promieniowania rentgenowskiego, a na podstawie próby rozciągania określono własności mechaniczne.

Zaobserwowano, że istotny wpływ na rozwój mikrostruktury oraz obserwowane zmiany własności mechanicznych ma temperatura prowadzonego procesu spajania. W przypadku niższych temperatur, tj. dla materiału przetwarzanego w temperaturze otoczenia oraz w 200°C, wraz ze zwiększaniem stopnia odkształcenia obserwowano silne rozdrobnienie mikrostruktury oraz uzyskano najlepszą kombinację własności mechanicznych. Prowadzenie procesu spajania przy wstępnym nagrzewaniu blach w temperaturze 300°C wskazywało na zachodzenie w trakcie odkształcania procesów odnowy struktury, co przekładało się na niższe w porównaniu do temperatury 200°C własności wytrzymałościowe.

## 1. Introduction

The intensive studies of nano and ultrafine grained (UFG) materials have been carried out for the last years. The increase of interest has come from the fact that the nano and ultrafine grain materials reveal relatively high mechanical properties. An increased strength, greater hardness and higher ductility in such materials have been reported. Also a lower temperature of superplastic flow occurring in these materials permits to reduce the formation temperature of products of complicated shape. Severe plastic deformation (SPD) methods, like equal channel angular pressing (ECAP) method [1-7], accumulative roll bonding (ARB) [8-11], high pressure torsion (HPT) [12,13] and cyclic extrusion compression (CEC) [14-15] have been frequently applied to produce the UFG materials. The

accumulative roll bonding (ARB) method consists in repeated rolling with the 50% reduction of two metal sheets stacked in a pack. These cyclic processes enable inducing very high deformations in order to obtain ultrafine crystalline structure.

The mechanisms of SPD, that give the structure refinement, reveal a partial similarity to the changes observed in the conventional methods, in which the phenomenon of deformation is accompanied by recrystallization and /or phase transformations. It is assumed that the essential role in the SPD techniques is played by a shear mechanism, which is responsible for the fragmentation of elongated subgrains as well as the increase of disorientation angle between them resulting in the structure refinement [16]. Additionally, the size of the finally obtained grain is affected by technological parameters of the deformation process, i.e. character, degree, rate

\* INSTITUTE OF METALLURGY AND MATERIALS SCIENCE PAS, 25 REYMONTA STR., 30-059 KRAKÓW, POLAND

and temperature of deformation as well as the stacking fault energy and phase morphology of material [17].

There are numerous papers in the literature referring to the analysis of microstructure and texture and their influence on mechanical properties of different metals and alloys deformed by the ARB method [8-11,18-21]. In the case of Al alloys the best mechanical properties which were coupled with a strong refinement of structure and significant contribution of high angle grains have usually been reported for 4 up to 6 cycles of ARB rolling, [9,18-21]. However, there are many contributions on aluminum alloys like AA1xxx, AA6xxx and AA8xxx [18-24] deformed using the ARB technique and few on AA5xxx series with elevated magnesium content [25, 26].

The aim of the present paper has been the analysis of the influence of deformation temperature on the microstructure and texture correlated with the mechanical properties of sheets of AA5251 commercial Al alloy containing precipitates of second phase subjected to accumulative roll bonding.

## 2. Material and experimental

The experimental investigations were performed on the commercial AA5251 Al alloy, whose composition is presented in Table 1. The sheets were recrystallized at 400°C for 60 min before the ARB process obtaining the grain size of 70÷80 μm. The packets for experiments consisted of two sheets of dimensions: thickness 1 mm, width 30 mm and length 250 mm. The scheme of the ARB method is demonstrated in Fig. 1. The parameters of the process, i.e. temperature, duration time and number of deformation cycles are contained in Table. 2. The roll-bonding process was carried out using a laboratory rolling mill of duo-type, with the roll mill diameter of 150 mm and the circumferential speed of 4.33 m/min.

TABLE 1  
Chemical composition (wt. %) of AA5251 alloy

Mg	Mn	Fe	Si	Zn	Cu	Ti	Cr	Al
1.81	0.50	0.42	0.24	0.09	0.03	0.03	0.03	Bal.

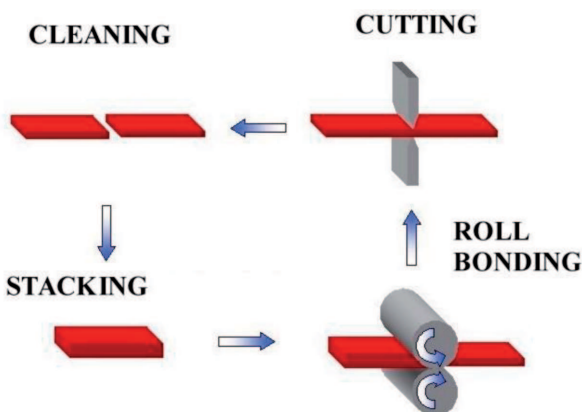


Fig. 1. Scheme of ARB processing

The evolution of microstructure was studied using a transmission electron microscope (TEM) Philips CM20 at the acceleration voltage of 200 kV. The samples were taken from the

parallel section of the deformed sheets (i.e. section RD-ND, rolling direction – normal direction, respectively).

TABLE 2  
Parameters of the ARB process

Material	Temperature [°C]	Time [min.]	Cycle number	Logarithmic strains
AA5251	room	-	1÷5	0.8÷4.0
	200	4	1÷10	0.8÷8.0
	300	10		

The mechanical properties were obtained in the static tensile test carried out at ambient temperature using an Instron 3382 testing machine on samples cut out parallel to the rolling direction.

## 3. Results and discussion

The accumulative roll bonding of the AA5251 alloy was carried out at ambient temperature at first, but the sheets strengthened quickly and what resulted in their cracking after 5 cycles along almost the whole length of the packet (Fig. 2). That is why, the sheets were subjected to pre-heating at 200°C and 300°C. The TEM observations revealed differences of the sample morphology in dependence on conditions of the ARB process.

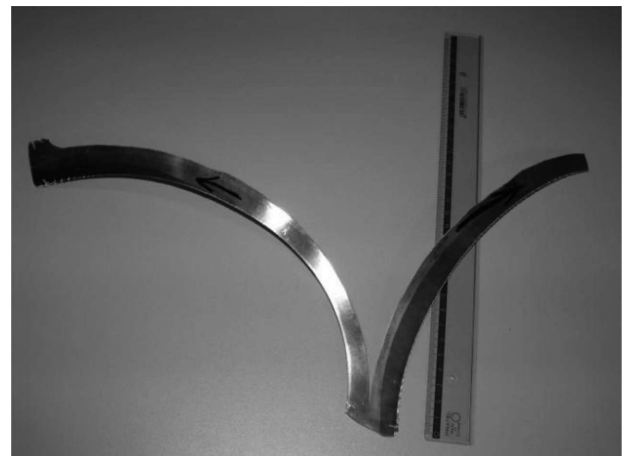


Fig. 2. Typical defect of sheet in the form of longitudinal crack in AA5251 alloy bonded with the ARB technique at ambient temperature after 6 cycles

Fig. 3 shows the evolution of microstructure of pre-heated alloy at 200°C after 2-, 6- and 9-fold rolling. The material after two ARB cycles revealed a significant elongation of grains in the rolling direction with partially preserved primary grain boundaries and a dislocation cell substructure, which formed inside the primary grains (Fig. 3a). The increase of the deformation degree caused a strong refinement of microstructure and a complete loss of the primary boundaries. The areas of strongly elongated grains, whose thickness did not exceed 200 nm and the areas of localized plastic deformation in the form of shear bands were observed in the material after 6 cycles (Fig. 3b). A further increase of deformation did not affect sub-

stantially the microstructure refinement, (Fig. 3c), although a slight increase of the shear band volume was visible.

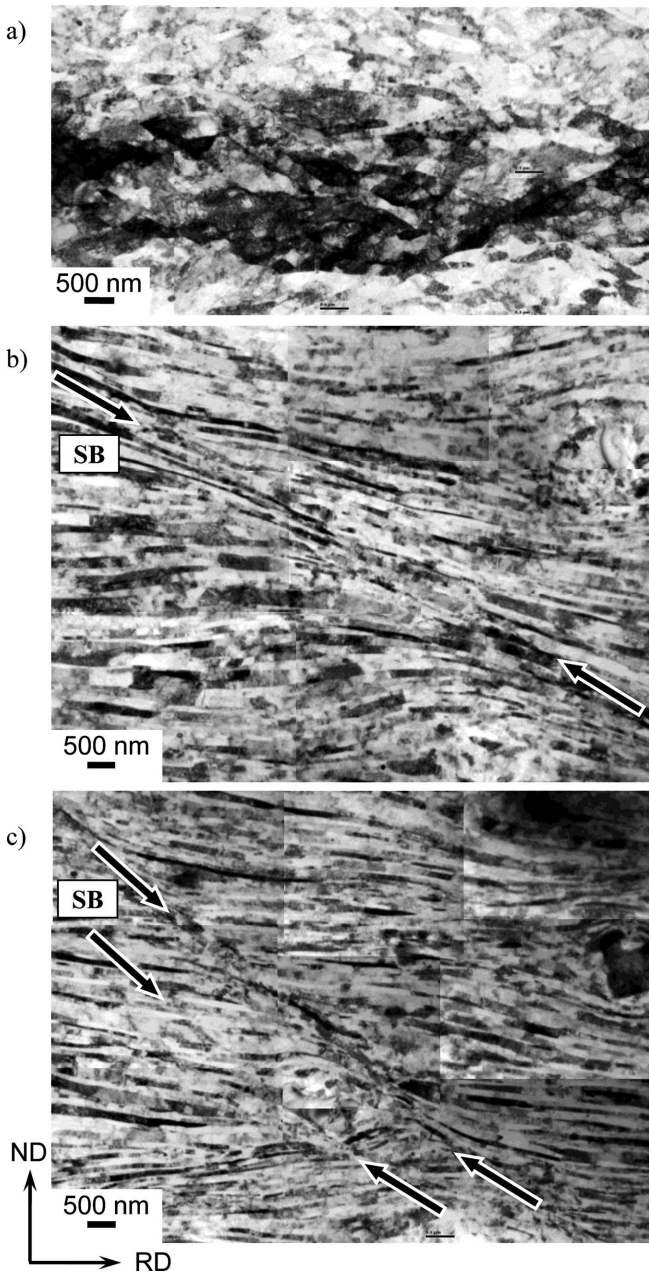


Fig. 3. TEM microstructure of the alloy pre-heated at 200°C deformed by: (a) 2 cycles, (b) 6 cycles, (c) 9 cycles. Section ND-RD

The alloy that was heated at 300°C before bonding had a different structure. The microstructure was locally elongated in the rolling direction and consisted of grains of 500 nm in size, in which new subgrains formed (Fig. 4a). The microstructure of the alloy after 6 cycles of deformation had the thickness of individual grains above 500 nm (Fig. 4b). The areas of strongly elongated grains in which the recovery processes (recrystallization) took place resulting in the appearance of equiaxial subgrains of low dislocation density were locally observed.

The {111} pole figures obtained with the method of x-ray diffraction for sample after 2<sup>nd</sup>, 6<sup>th</sup> and 9<sup>th</sup> cycle, which was pre-heating at 200°C are shown in Fig. 5. The {100} <011> shear orientation prevailed in the texture of the alloy at low deformation degrees. The orientation underwent a

transformation directed towards two complementary positions of C{112} <111> orientation with the increase of the cycle number. Such a behavior is typical for most of fcc metals, for which the {100} <011> orientation is strongly unstable in the planar state of deformation and which undergoes a very fast decomposition through the rotation towards two complementary positions of the C orientation.

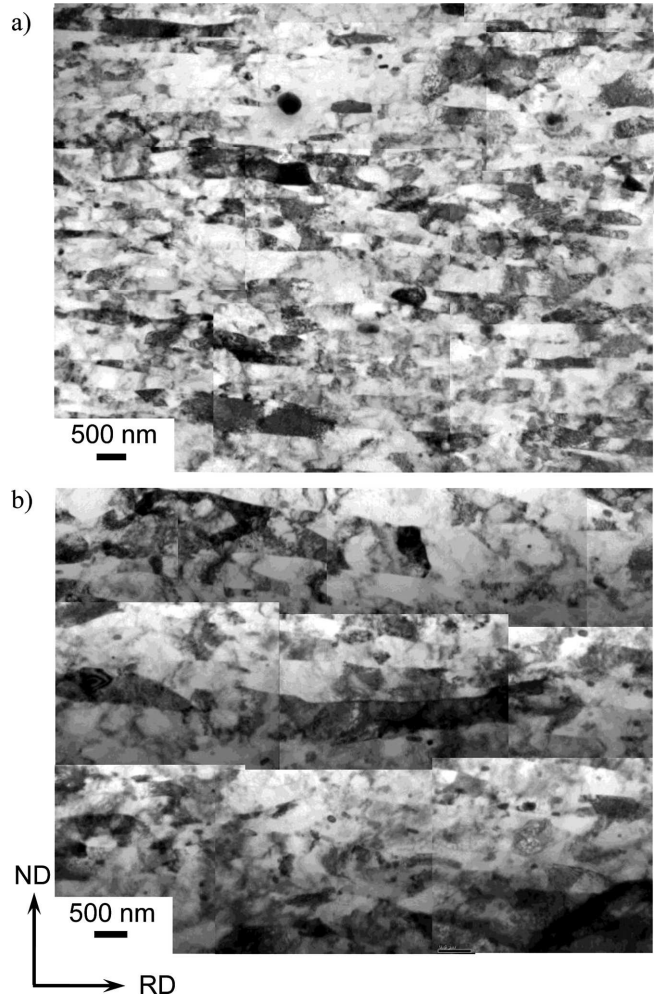


Fig. 4. TEM microstructure for the alloy pre-heated at 300°C deformed by: (a) 2 cycles, (b) 6 cycles. Section ND-RD

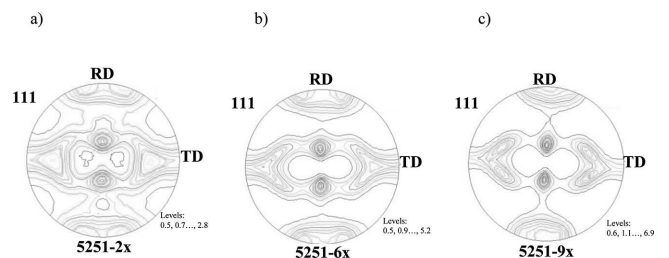


Fig. 5. Pole figures {111} obtained with the x-ray diffraction method: a) after 2 cycles, b) after 6 cycles and c) after 9 ARB cycles for the alloy preheated at 200°C

The changes of mechanical properties as a function of temperature and deformation degree for the AA5251 alloy in comparison with the literature data for the AA6016 alloy based on work [18] are illustrated in Fig. 6. The AA6016 alloy was preheated at 230°C and roll-bonded up to 8<sup>th</sup> cycle. The



changes of yield point and tensile strength of the sheets bonded at ambient temperature and preheated at 200°C correspond up to 5th cycle and they tend to grow (Fig. 6 a-b). The highest tensile strength ( $R_m$ ) was obtained after 7 cycles. The sheets preheated at 300°C showed the parameters at constant level of about 195 MPa and 280 MPa of  $R_{0,2}$  and  $R_m$ , respectively. A visible influence of bonding temperature was observed for the case of elongation (Fig. 6c). A decreasing tendency was noted for the samples bonded at ambient temperature, while the sheets preheated at 200°C had the constant elongation more or less 5%. The material preheated at 300°C revealed the highest elongations in the whole range of deformation which, however, fell together with the increase of number of cycles. The results of mechanical properties obtained for the AA5251 alloy correlated quite well with the data of [18], in which the dependences of mechanical properties on the ARB cycle number of the AA6016 sample had an increasing tendency. The curves showed in Fig. 6 are similar to these for the AA5251 alloy preheated at 200°C.

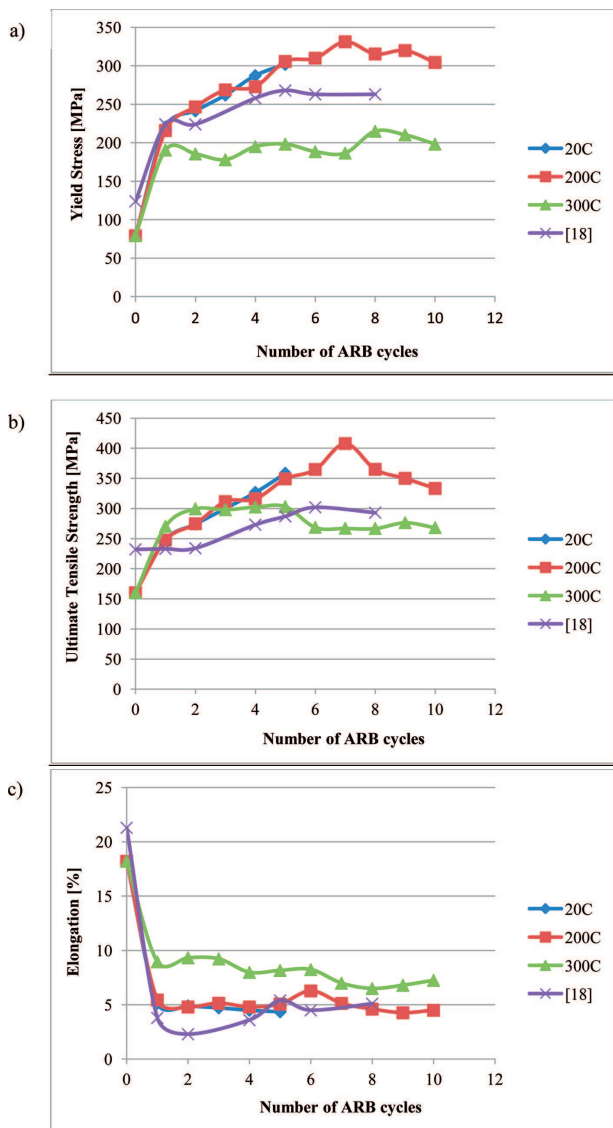


Fig. 6. Influence of preheating temperature and number of ARB cycles on: a) yield point, b) tensile strength and c) elongation of the AA5251 alloy

The ultracrystalline structure produced in the AA5251 alloy due to intensive deformation at appropriately selected conditions of ARB resulted in high mechanical properties. The increase of properties observed in the alloy subjected to preheating at 200°C was an effect of two overlapping processes. The dislocation density grew together with the increase of deformation bringing about the generation and movement of dislocation tangles and their localization at grain boundaries [17]. It led to the formation of a cell dislocation structure, which due to the occurrence of phenomenon of subgrain coalescence and climbing of dislocations transformed into subgrains of increasing disorientation angle [27]. Another factor, which affected the refinement process and the increase of disorientation angle, was the localization of plastic deformation in the form of the shear bands observed. It was the reason that the structure of elongated subgrains of about 100-200 nm was obtained after the 6 cycles (Fig. 3b). Despite a relatively high temperature of the preliminary heating (200°C), the recovery processes did not take place, which might be due to the presence of very small precipitates of size below 1  $\mu\text{m}$ . The ARB process carried out on the samples preheated at 300°C resulted in the formation of material of different microstructure and lower mechanical properties. That was caused by the recrystallization processes of the matrix together with the grain growth (Fig. 4). The subgrains elongated in the direction of rolling as well as the areas of low density of dislocations showed that the processes were of cyclic character. It caused the stabilization of strength properties together with the decrease of plastic ones due to the systematic increase of grain size.

The curves of mechanical properties in dependence on number of cycles and the structure observation of the AA5251 alloy are similar to the literature data on Al alloys e.g. [18,19,22,28]. The increase of deformation degree in the ARB process brought about the improvement of mechanical properties up to a limit number of cycles, which was 6-7 cycles for the analyzed alloy, while the subgrain width varied from 100÷500 nm. The temperature of the ARB process had a fundamental importance for the development of microstructure and properties. When too high, it led to the intensification of recovery processes very often accompanied by the grain growth [29], when too caused fast strengthening of metal resulting in cracking and problems with bonding of the rolled sheets [8]. That was also true for the examined AA5251 alloy. The rolling at ambient temperature led to strengthening and cracking of sheets after 5 cycles of rolling (Fig. 2), whereas after preheating at 300°C the increase of cycle number did not give as a consequence the growth of mechanical properties. The visible decrease of  $R_m$  value after 7 cycles of ARB (preheated at 200°C) was most probably due to the enlarged number of bonded sheets in which the distance between big precipitates of the second phase decreased (Fig. 7).

## REFERENCES

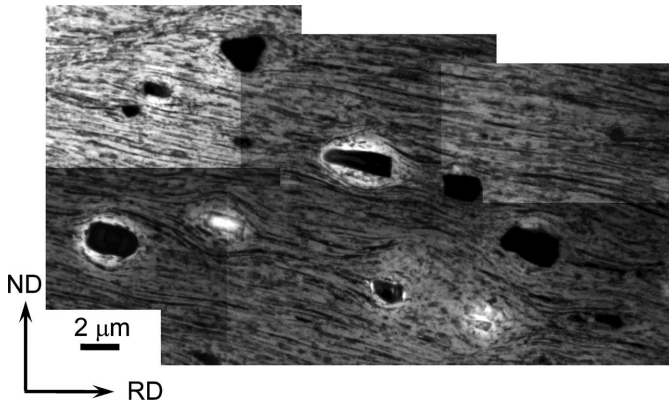


Fig. 7. TEM microstructure of the AA5251 alloy pre-heated at temperature 200°C deformed with 9 cycles. Section ND-RD

#### 4. Conclusions

The results obtained for the AA5251 alloy bonded through cyclic rolling (ARB) indicated the possibility to obtain the material with the UFG structure and high mechanical properties provided the appropriate parameters of the process were preserved. The examinations revealed a strong influence of temperature of processing on microstructure and mechanical properties of the analyzed alloy.

The ARB technique enabled obtaining the material with the ultracrystalline structure. The mechanisms which determined the structure evolution and its impact on the changes of mechanical properties strongly depended on temperature and the deformation degree i.e. the number of ARB cycles. The refinement of microstructure occurred as a result of fragmentation of primary grains in the range of lower temperatures, while a higher temperature of preliminary annealing intensified the processes of recovery giving as a consequence the increase of grain and a decrease of mechanical properties.

The best combination of mechanical properties was obtained for the material preheated at 200°C and processed 6 times. It was when a strongly deformed morphology with the banded arrangement of grains of about 100 nm wide was observed.

The increase of cycle number above the specific limit does not cause the further increase of mechanical properties. It is most probably connected with the fact, that the higher deformation degree above the limit induced the further decrease of thickness of individual layers of sheet which contained more and more rolling defects as well as large precipitates of the second phase.

In the range of high deformation degrees the structure recovery occurred locally in the areas of critical density of dislocations connected with the simultaneous accumulation of strain in the adjacent regions. The cyclic character of these processes determined the course of changes of mechanical properties leading to their stabilization.

- [1] R.Z. Valiev, T.G. Langdon, *Progress in Materials Science* **51**, 881 (2006).
- [2] V.M. Segal, *Mater. Sci. Eng.* **A197**, 157 (1995).
- [3] Z. Horita, M. Furukawa, M. Nemoto, A.J. Barnes, T.G. Langdon, *Acta Mater.* **48**, 3633 (2000).
- [4] J. Kuśnierz, W. Baliga, J. Bogucka, *Applied Crystallography*, ed. H. Morawiec, D. Stróż, World Sci. Publ. Co., 181, London 2004.
- [5] R.B. Figueiredo, T.G. Langdon, *Mater. Sci. Eng. A* **501**, 105 (2009).
- [6] K.R. Cardoso, D.N. Travessa, W.J. Botta, A.M. Jorge Jr., *Mater. Sci. Eng. A* **528**, 5804 (2011).
- [7] H. Paul, T. Baudin, F. Brisset, *Arch. Metall. Mater.* **56**, 245 (2011).
- [8] Y. Saito, H. Utsunomiya, N. Tsuji, T. Sakai, *Acta Mater.* **47**, 579 (1999).
- [9] N. Tsuji, Y. Ito, Y. Saito, Y. Minamino, *Scr. Mater.* **47**, 893 (2002).
- [10] D. Terada, M. Inoue, H. Kitahara, N. Tsuji, *Materials Transactions* **49**, 41 (2008).
- [11] J. Kuśnierz, J. Bogucka, M.-H. Mathon, T. Baudin, *Arch. Metall. Mater.* **53**, 179 (2008).
- [12] Z. Horita, T.G. Langdon, *Mater. Sci. Eng.* **A410-411**, 422 (2005).
- [13] R.Z. Valiev, A.V. Sergueeva, A.K. Mukherjee, *Scr. Mater.* **49**, 669 (2003).
- [14] M. Richert, J. Richert, *Inżynieria Materiałowa* **2**, 73 (2001).
- [15] M. Richert, B. Leszczyńska, *Arch. Metall. Mater.* **53**, 721 (2008).
- [16] H. Paul, *Tekturowe uwarunkowania procesu rekrytalizacji pierwotnej w metalach o sieci Al*, IMIM PAN, Kraków 2002.
- [17] M. Lewandowska, *Kształtowanie mikrostruktury i właściwości stopów aluminium metodą wyciskania hydrostatycznego*, *Prace Naukowe Inżynieria Materiałowa z.* 19, 2006.
- [18] I. Topic, H.W. Höppel, M. Göken, *J. Mater. Sci.* **43**, 7320 (2008).
- [19] H.W. Kim, S.B. Kang, N. Tsuji, Y. Minamino, *Acta Mater.* **53**, 1737 (2005).
- [20] J. Bogucka, H. Paul, T. Baudin, M.-H. Mathon, *Inżynieria Materiałowa* **5**, 1347 (2010).
- [21] J. Kuśnierz, J. Bogucka, *Mater. Sci. Forum* **495-497**, 797 (2005).
- [22] Z.P. Xing, S.P. Kang, H.W. Kim, *Scr. Mater.* **45**, 597 (2001).
- [23] K.T. Park, H.J. Kwon, W.J. Kim, Y.S. Kim, *Mater. Sci. Eng.* **A316**, 145 (2001).
- [24] J. Bogucka, H. Paul, M. Bieda, T. Baudin, *Solid State Phenom.* **186**, 112 (2012).
- [25] S. Roy, S.D. Singh, S. Suwas, S. Kumar, K. Chattopadhyay, *Mater. Sci. Eng.* **A528**, 8469 (2011).
- [26] M.R. Toroghinejad, F. Ashrafizadeh, R. Jammaati, *Mater. Sci. Eng.* **A561**, 145 (2013).
- [27] J. Adamczyk, *Odszałcenie plastyczne, umocnienie i pękanie*, Wydawnictwo Politechniki Śląskiej, Gliwice 2002.
- [28] S.H. Lee, Y. Saito, T. Sakai, H. Utsunomiya, *Mater. Sci. Eng.* **A325**, 228 (2002).
- [29] M. Slamova, P. Homola, M. Karlik, *Mater. Sci. Eng.* **A462**, 106 (2007).