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ACCUMULATIVE ROLL BONDING OF CONTINUOUSLY CAST AL SHEETS

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ABSTRACT

Accumulative Roll Bonding (ARB) is a promising method for industrial applications since it enables the production of large amounts of ultra-fine grained (UFG) materials. The process involves multiple repetition of sheet surface pre-processing, stacking, rolling, and cutting. The rolling bonds the sheets and after 6 to 8 cycles, UFG materials with high strength and good ductility are produced. Fine second phase dispersion and small grain size are intrinsic features of continuously cast sheets making them a good starting materials for ARB. The paper presents first experience with the ARB processing of AA8006 continuously cast sheet and high purity aluminium. The effect of rolling temperature on the quality of roll bonding, grain refinement, and hardness was studied. After two cycles at 200°C, mean grain size of 0.4 to 0.8 μm is achieved in AA8006 sheets, but areas with extremely fine grains of 0.1 to 0.3 μm in diameter are also found. Hardness increases significantly after the first two cycles and it rises a little during subsequent cycles. ARB processing of AA8006 sheet at 350°C results in better bonding but in smaller increase in hardness due to recrystallization and grain coarsening occurring during heating to 350°C. High purity Al sheets were ARB processed at room temperature and compared with AA8006 ARB sheets. Trends of hardness increase similar to those in AA8006 are observed in high purity Al. Positron lifetime measurements reveal a substantial increase of dislocation density after the first cycle and moderate increase with further ARB cycles. The high fraction of positrons trapped at dislocations evidence the substantial grain refinement occurring upon ARB processing of pure Al and is also confirmed by TEM.

1. INTRODUCTION

Bulk ultra-fine grained (UFG) materials with improved strength can be prepared either by Equal Channel Angular Pressing (ECAP) or Accumulative Roll Bonding (ARB). ARB is a promising method for industrial applications since it enables the production of large amounts of UFG material. The process involves multiple repetition of sheet surface pre-processing, stacking, rolling, and cutting. The rolling bonds the sheets and after 6 to 8 cycles, UFG materials with high strength and relatively high ductility are produced (Tsuji, Kamikawa, Kim, and Minamino 2004). ARB has been successfully used to prepare UFG sheets from several ingot cast aluminium alloys (Tsuji et al. 2004). Aluminium alloys issued from continuous twin-roll cast (TRC) strips exhibit fine second phase dispersion and very small grains (Slámová 2001), thus they are expected to be good starting materials for ARB processing and have thermally stable UFG structures. The paper presents first experience with ARB processing of TRC AA8006 sheets and high purity aluminium as reference material. The effect of temperature on the quality of bonding, grain refinement, and hardness has been studied.

2. EXPERIMENTAL

Twin-roll cast, homogenised AA8006 (Table 1) and cold rolled sheets of 2.5 mm thickness, and a hot rolled AA1199 (Al99.99) 9.0 mm plate, supplied by AL INVEST Břidličná, were used in the experiments. AA1199 alloy was cold rolled to thickness of 2.0 mm. Fully annealed materials were prepared by annealing for 0.5 h at 400°C (AA8006) and 350°C (AA1199). ARB processing consisted in the repetition of the following steps: i) surface treatment (degreasing in tetrachlorethylene and wire-brushing with stainless steel 0.3 mm wire brush; ii) stacking of two pieces of 300 x 50 x 2.5 mm; iii) pieces joining by Al wires; iv) heating to the appropriate temperature, 200°C and 350°C, respectively, in an electrical furnace; v) bonding by rolling to 50% reduction without lubricant. Roll diameter of 340 mm and peripheral speed 0.7 m×min^{-1} were applied in all cases. In order to prevent the propagation of edge cracks, specimen edges were trimmed and smoothed down.

Table 1. Chemical composition of AA8006 alloy (wt.%).

Element	Mn	Fe	Si	Cu	Mg	Zn	Ti	Al
Content	0.40	1.51	0.16	0.006	0.003	0.012	0.014	Balance

The initial materials and the deformed microstructures were examined using polarised light microscopy (LM) and transmission electron microscopy (TEM). Standard TEM foils 3 mm in diameter were prepared by electrolytic twin-jet polishing (-30°C, 30 V) using 6% solution of perchloric acid in methanol. TEM observations were carried out at 200 kV. Vickers hardness HV10 measurements were used for evaluating the strength of ARB processed materials. Positron lifetime (PL) measurements (Hautojarvi and Corbel 1995) were used to estimate dislocation density and arrangement in ARB processed AA1199 samples. A fast-fast PL spectrometer (Bečvář, Čížek, Lešták, Novotný, Procházka and Šebesta 2000) with timing resolution of 160 ps (FWHM ^{22}Na) at coincidence count rate of 120 s^{-1} was employed.

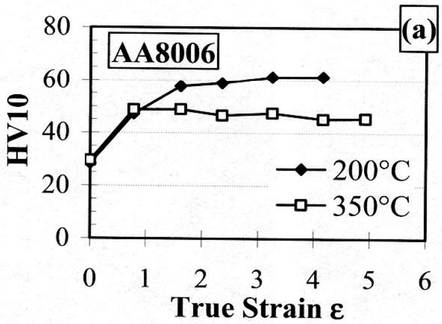
3. RESULTS AND DISCUSSION

The AA8006 sheets used for ARB processing were recrystallized with grain size of < 20 μm in rolling direction (RD) and 15 μm in normal direction (ND) (Table 2). The grain size of the AA1199 initial material was more than twice larger (Table 2) as compared to TRC AA8006 sheets. In all cases, grains slightly elongated in RD were observed.

Table 2. Grain size of initial and ARB processed AA8006 and AA1199 sheets (in μm).

	initial condition (LM)		ARB processed (TEM)		
Alloy - ARB Temperature	RD	ND	1 cycle	3 cycles	5 cycles
AA8006 - 200°C	15	12	1.0	0.6	0.5
AA8006 - 350°C	19	14	1.3	1.3	1.3
AA1199 - 20°C	46	38	2.0	1.0	-

After initial failures to achieve good roll bonding, five successful ARB cycles were performed at 200°C with AA8006 alloy. The results of those experiments are reported in (Karlík, Homola and Slámová 2004) and are compared with the results of ARB processing at 350°C in this paper. ARB at 200°C results in hardness increase from 28 to 60 kg/mm^2 (after two cycles). HV10 rises a little during subsequent cycles (Fig. 1a). Better roll bonding is obtained when AA 8006 sheets are processed at 350°C, but smaller increase in hardness is achieved. HV10 increases from 30 to 50 kg/mm^2 and no increase is observed after the second cycle (Fig. 1a). The hardness of high purity Al increases more than 2.5 times as result of two ARB cycles at 20°C and does not further increase during subsequent cycles (Fig. 1b). It is significantly lower as compared to the TRC AA8006 specimens ARB processed at 200°C.



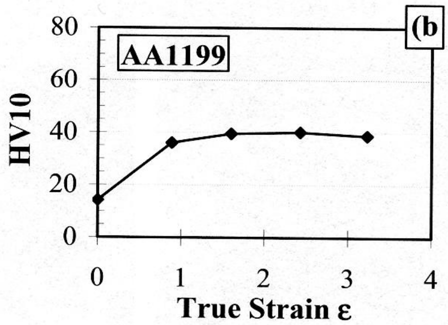


Fig. 1. Variation of sheet hardness with true strain introduced by ARB in AA 8006 sheets processed at 200 and 350°C (a) and in AA 1199 processed at 20°C (b).

Light microscopy indicates that AA8006 samples ARB processed at 200°C exhibit deformed grain structure typical for heavily cold rolled aluminium sheets. TEM examinations after the first ARB cycle reveal subgrains of size from 0.5 to 1.5 μm (Fig. 2a). During subsequent cycles, low-angle boundaries convert to high-angle boundaries (Figs. 2b,c) appearing in a typical fringe contrast (Fig. 2b). The size of the majority of grains is in the range from 0.4 to 0.8 μm, the largest grains are of 1.2 μm in diameter. Dislocation density of in subgrains or grains remains almost unchanged throughout all cycles of

ARB processing and is indicative for a recovered substructure. In the samples with 2nd and 5th cycles, 150 μm wide areas with much finer grains (0.1 to 0.3 μm in diameter) are observed (Fig. 2b). The mechanism of formation of UFG areas is still unclear. XEDS analysis does not show any difference in matrix composition in these areas, neither fine-dispersed particles that could pin grain boundaries are observed.

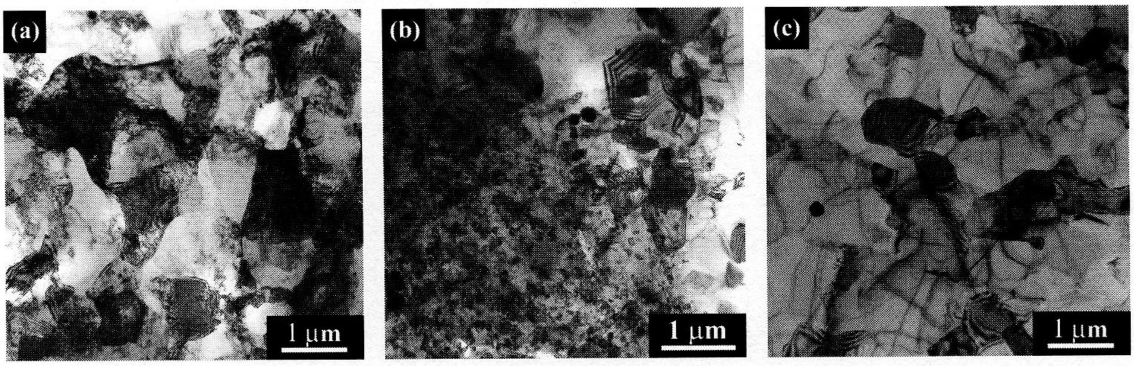


Fig. 2. TEM micrographs of TRC AA8006 sheets ARB processed at 200°C: 1 cycle (a), 2 cycles (b), and 5 cycles (c). View in the plane perpendicular to ND.

In contrast to AA8006 specimens ARB processed at 200°C, coarser grains form in specimens processed at 350°C. Post-processing examinations by light microscopy (LM) show grains elongated in RD, but after the 2nd cycle the grain size in ND is much larger than that expected after several ARB cycles, which would introduce significant plastic deformation. TEM examinations (Table 2, Fig. 3) confirm that the average grain size refines in the 1st ARB cycle down to 1.3 μm and it remains almost unchanged by further ARB processing. However, areas of grain size as small as 500 nm are also locally observed. Both LM and TEM indicate that recrystallization and grain coarsening occur during heating to 350°C. Therefore, the recrystallization causes the low sheet hardening achieved by the ARB processing at 350°C.

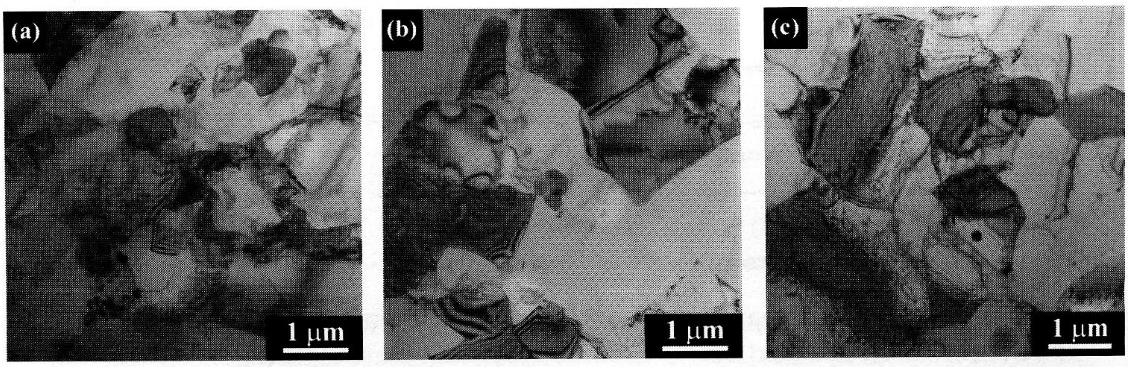


Fig. 3. TEM micrographs of TRC AA8006 sheets ARB processed at 350°C: 1 cycle (a), 3 cycles (b) and 5 cycles (c). View in the plane perpendicular to ND.

ARB processed AA1199 samples were first studied by PL measurements. Their PL spectra could be fitted by two exponential components with lifetimes τ_1 and τ_2. The shorter component τ_1 is the contribution of free positrons, while τ_2 is a contribution of positrons trapped at defects. Fig. 4a shows the evolution of τ_1 and τ_2 with increasing ARB cycles. It can be seen that τ_1 decreases as result of ARB processing, whereas τ_2 is almost unchanged. According to previous results of PL measurements in aluminium (Čížek, Procházka, Kmječ and Vostrý 2000), it can be assumed that $\tau_2 \approx 243$ ps is the lifetime of positrons trapped at dislocations introduced by ARB processing. Fig. 4b shows that the relative intensity I_2 of the dislocation component drastically increases after the first ARB cycle but changes only moderately during subsequent ARB cycles. This is indicative for the substantial increase of dislocation density during the first ARB cycle, while further cycles do not cause any significant change. The application of the two-state trapping model (Hautojarvi et al., 1995) to the data reveals that dislocations in ARB processed samples are distributed non-homogeneously, i.e. they are arranged in cell or subgrain boundaries. This hypotesis was verified by TEM examinations.

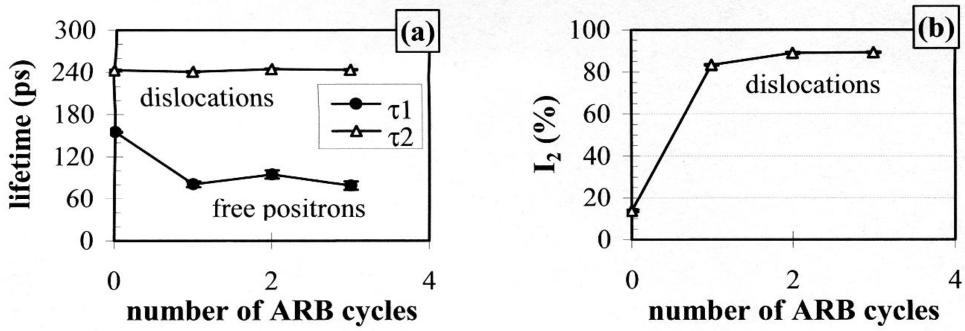


Fig. 4. Effect of ARB processing of AA1199 sheet at 20°C on positron lifetime (a) and intensity of the dislocation component of positron lifetime (b).

Light and TEM microscopy indicates that initial AA1199 sheet is fully recrystallized with grain size of 45 μm (Table 2) and very low dislocation density of 10^{12} m^{-2} (Fig. 5a).

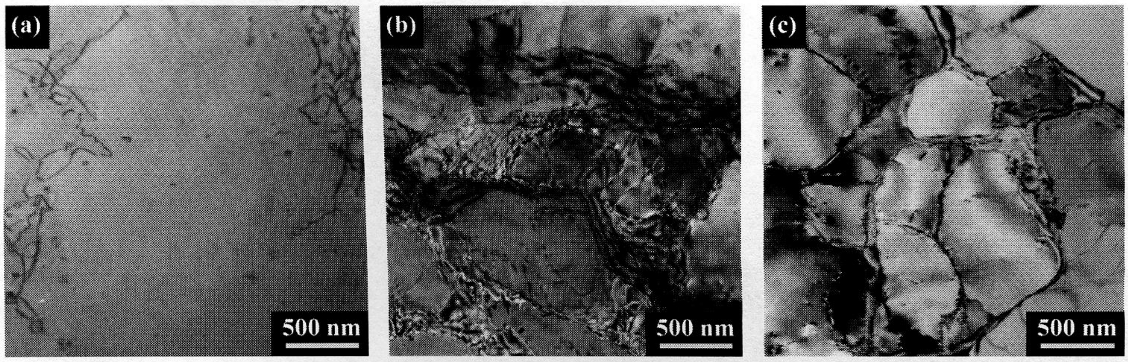


Fig. 5. TEM micrographs of AA1199 sheet ARB processed at 20°C: initial material (a), 1 cycle (b), 3 cycles (c). View in the plane perpendicular to ND.

In accordance with PL measurements, TEM shows well-defined deformation cells and subgrains of mean size 1-2 μm (Fig. 5b) formed during the first ARB cycle and low dislocation density in subgrain interiors (3.10^{13} m^{-2}). However, subgrain formation due to dynamic dislocation recovery is not complete. Fig. 5b shows low angle boundaries in which dislocations can still be well distinguished. Subgrain boundaries become more perfect with increasing number of ARB cycles (Fig. 5c) and the density of dislocations in subgrain interior, 7.10^{12} m^{-2}, is much lower as compared to the first cycle. After 3

ARB cycles, formation of small recrystallization nuclei is also observed. The subgrain size after 3 ARB cycles of AA1199 specimens is of 1 μm, therefore much larger than in the TRC AA8006 specimen processed at 200°C.

4. CONCLUSIONS

Accumulative roll bonding at 200°C and 350°C was applied in order to prepare ultra-fine grained materials from continuously cast AA8006 aluminium alloy and at 20°C, respectively, from direct chill cast pure aluminium (AA1199). Roll bonding of AA8006 specimens at 350°C was more successful than at 200°C. However, ARB processing at 350°C does not result in significant grain refinement and strength increase as does ARB processing at 200°C. Recrystallization and grain coarsening during heating to 350°C cause small grain refinement and strengthening observed at this temperature. The grain refinement of continuously cast AA8006 sheets processed at a temperature, sufficiently low to prevent recrystallization during heating, is more significant. Grains of size as small as 100 nanometres are produced in part of the material subjected to 5 cycles of ARB processing. 6 cycles of 350°C ARB processing of AA8006 sheets produces grains of mean size of 1.3 μm, but areas of grain size as small as 500 nm are also locally observed. ARB processing of high purity aluminium at 20°C introduces more than two-fold increase in hardness due to grain refinement from 45 μm to 1 μm grain size after 3 ARB cycles. PL measurements allow easily estimate dislocation density and arrangement and are in good agreement with TEM observations.

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REFERENCES

Bečvář, F., Čížek, J., Lešták, L., Novotný, I., Procházka, I., and Šebesta, F. (2000). Nucl. Instr. Meth. A. 443, 557-577.

Čížek, J., Procházka, I., Kmječ,T., and Vostrý, P. (2000). phys. stat. sol. (a) 180, 439-458.

Hautojärvi, P., and Corbel C. (1995). In: Positron Solid State Physics, Eds. A. Dupasquier and A.P. Mills (IOS Press, Varena) 491-532.

Karlík, M., Homola, P., and Slámová, M. (2004). Accumulative roll-bonding: First experience with a twin-roll cast AA8006 alloy. Accepted for publication in J. of Alloys and Comp.

Slámová, M. (2001). Annealing Response of AA8006 and AA8011 Thin Strips - Effect of Pre-Treatment and Strain Level. Aluminium 77, 10, 801-808.

Tsuji, N., Kamikawa, N, Kim,H.W., and Minamino, Y. (2004). Fabrication of Bulk Nanostructured Materials by ARB (Accumulative Roll Bonding) Process. (TMS Symp., Charlotte, USA) Submitted.