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Effect of the Temperature of Accumulative Roll Bonding on the Microstructure and Properties of Twin-Roll Cast AA8006 Alloy

P. Homola[1,2,a], M. Slámová[1,b], M. Karlík[2,c], J. Čížek[3,d] and I. Procházka[3,e]

[1] VÚK Panenské Břežany, s.r.o., 250 70 Odolena Voda, Czech Republic

[2] FNSPE, Czech Technical University, Trojanova 13, 12000 Praha 2, Czech Republic

[3] Faculty of Maths and Physics, Charles University, Ke Karlovu 3, 12116 Praha 2, Czech Republic

[a]vuk@volny.cz, [b]slamova.vuk@volny.cz, [c]karlik@kmat.fjfi.cvut.cz, [d]jcizek@mbox.troja.mff.cuni.cz, [e]ivanp@mbox.troja.mff.cuni.cz

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Abstract. Accumulative Roll Bonding (ARB) does not require any special equipment and enables the production of large amounts of ultra-fine grained (UFG) materials. Grain refinement is thermally stable in materials with finely dispersed particles such as twin-roll cast (TRC) aluminium alloy sheets, favourable materials for manufacturing UFG sheets. The results of a study of the effect of ARB temperature on bonding quality, structure and properties of TRC AA8006 sheet are presented. Examinations by light and transmission electron microscopy, positron annihilation spectroscopy (PAS), hardness and tensile tests were used in the study. After two cycles at 200°C, mean grain size of 0.4 - 0.8 μm is achieved, but areas with extremely fine grains of 0.1 to 0.3 μm in diameter are also observed. Hardness increases significantly after two cycles and it rises a little in subsequent cycles. Processing at higher temperatures (up to 350°C) results in better bonding but it produces smaller increase in hardness. Significant increase of dislocation density is observed by PAS after the first cycle at 250°C but it does not continue during subsequent cycles. Partial recrystallization occurs in samples processed at temperatures above 250°C explaining the smaller increase in hardness. Softening level depends on both ARB temperature and number of cycles. The thermal stability of refined structures produced by ARB at 250°C is better than these formed at higher temperatures.

Introduction

Accumulative Roll Bonding (ARB) is a new promising process of grain refinement by severe plastic deformation. ARB does not require any special equipment and enables the production of large amounts of ultra-fine grained (UFG) materials. These features favour the application of ARB in industrial practice. ARB was successfully applied in the manufacturing UFG sheets from several aluminium alloys and pure Al produced from ingot cast materials [1, 2]. However, it is well known that the efficiency of grain refinement, strain hardening and thermal stability can be improved using materials with fine particles and small grain size. Such microstructures are intrinsic for continuously twin-roll cast (TRC) aluminium alloy sheets. The first results about ARB processed continuously cast AA8006 alloy were reported in [3]. An investigation aimed at studying the effect of ARB processing temperature on the quality of bonding, microstructure and properties of a commercial TRC AA8006 sheet was undertaken later. This paper presents results obtained by light (LM) and transmission electron microscopy (TEM), positron annihilation spectroscopy (PAS), and hardness and tensile tests on samples processed in the temperature range from 200 to 350°C.

Experimental

Commercial TRC AA8006 sheet (Table 1) of 2.0 mm in thickness was used as input material. Fully recrystallized sheets were prepared by annealing for 0.5 h at 450°C. ARB processing consisted in the repetition of 5 steps: i) degreasing in 4-chlorethylene and wire-brushing with stainless steel 0.3 mm

wire brush; ii) stacking of pieces of 300×50×2 mm^3; iii) joining by Al wires; iv) heating to 200, 250, 300 and 350°C, respectively; v) rolling with 50% reduction without lubricant. The temperature increase in the samples prepared for ARB, after they were put in preheated furnace, was calibrated in time. That way, the input rolling temperature was always exactly determined. Roll diameter of 340 mm and peripheral speed of 0.7 $m \cdot min^{-1}$ were applied. In order to prevent propagation of edge cracks, specimen edges were trimmed and smoothed down after each ARB cycle.

Chemical composition of AA8006 alloy [wt.%].

Table 1

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 and deformed microstructures were examined after anodising in Barker reagent under polarised light. LM observations were carried out in long transverse plane (TD-plane). Grain size l_L was measured by chord intercept method in rolling (RD) and normal (ND) directions. The grain size of the initial AA8006 sheets was <20 μm in RD and <15 μm in ND (Table 2). TEM foils of 3 mm in diameter were prepared by electrolytic twin-jet polishing (-30°C, 30 V) using 6% solution of $HClO_4$ in methanol and observed at 200 kV. Vickers hardness measurements performed on sheet surface were used for fast evaluation of work hardening in ARB materials. Positron lifetime (PL) measurements [5] were used to estimate dislocation density and arrangement in both input and ARB samples. A fast-fast PL spectrometer [6] with timing resolution of 160 ps (FWHM ^{22}Na) at coincidence count rate of 120 s^{-1} was employed. Tensile tests at initial strain rate of 8.3×10^{-4} s^{-1} were carried out on samples of 20 mm in gauge length cut in TD. The thermal stability of ARB processed samples was evaluated by isochronal 1.8 ks annealing at temperatures from 200 to 450°C, hardness measurements and microstructure examinations.

Results and Discussion

The results of ARB processing at 200°C and 350°C are reported in [3, 4] and are compared with the results of ARB at 250 and 300°C in this paper. Fig. 1a shows hardness evolution as result of ARB processing at all checked temperatures. It can be seen that ARB at 200°C results in hardness increase from 28 to 60 (after two cycles) and rises a little during subsequent cycles.

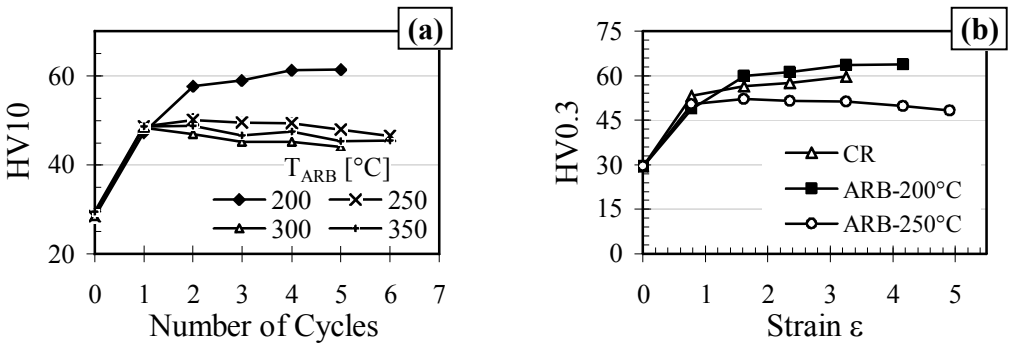

Fig. 1 Variation of hardness with number of ARB cycles in samples processed at 200 to 350°C (a); Hardness evolution in cold rolled and ARB processed samples (b).

Better roll bonding is obtained when the sheets are processed at temperatures > 200°C but smaller increase in hardness is achieved. At 250°C, HV10 increases from 30 to 50 during the first cycle. No increase is observed after the second cycle (Fig. 1a). At higher temperatures the hardness increases only in the first cycle and decreases in subsequent cycles. Fig. 1b compares hardness evolution in samples conventionally cold rolled (CR) to equivalent strains ε corresponding to strains induced by 1

to 4 ARB cycles. It can be seen that the work hardening by CR at small strains (ε=0.8) is similar to this produced by ARB but at higher stains the hardening by ARB at 200°C is larger.

Fig. 2 shows the variation of ultimate strength and elongation with increasing number of ARB cycles at temperatures from 250°C to 350°C. As in the case of hardness, the strength of samples processed at 250°C is higher than this of samples processed at higher temperatures and accordingly, the elongation is lower. Strength increase is observed in all cases in the first ARB cycle, for processing at 250°C R_m increase continues at moderate rate up to the 3rd cycle, whereas at higher temperatures a decrease is observed. The tensile elongation drops in the first cycle for all processing temperatures and keeps almost constant with further ARB cycles increase.

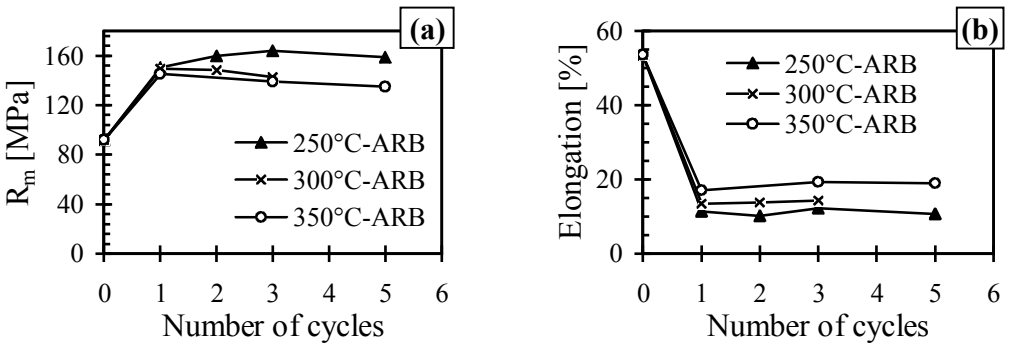

Fig. 2 Variation of ultimate strength R_m (a) and elongation (b) with number of ARB cycles of samples processed at 250 to 350°C.

Light microscopy and TEM examinations indicated that samples ARB processed at 200°C exhibit deformed grain structure typical for heavily cold rolled aluminium sheets [3]. The grain size (in ND-plane) after the first ARB cycle varies from 0.5 to 1.5 μm. During subsequent processing, the size of the majority of grains is in the range from 0.4 to 0.8 μm (Table 2). In [4], the microstructure of samples ARB rolled at 200°C and 350°C was compared. Coarser grains form during processing at temperatures above 200°C (Table 2). It was found out that recrystallization and grain coarsening occurs during heating before ARB and by this the grain size keeps constant with increasing number of cycles. Moreover, due to pre-rolling recrystallization, low hardening is achieved by ARB processing at 350°C (Fig. 1a and Fig. 2a).

Grain size of initial and ARB processed samples [in μm]. Table 2

ARB Temperature	l_L (initial, LM)		$d_{subgrain}$ (ARB processed, TEM)					
	RD	ND	1 cycle	2 cycle	3cycle	4 cycle	5 cycle	6 cycle
200°C	15	12	1.0	1.0	0.6	0.6	0.5	-
250°C	15	12	1.2	-	1.0	-	-	1.2
350°C	19	14	1.3	-	1.3	1.3	1.3	1.3

TEM micrographs of samples ARB processed at 250°C are in Fig. 3. The upper and lower figures show the view from ND and TD, respectively. The view from ND shows nearly equiaxed grains during the whole processing. After the 1st cycle, both grains with diffuse boundaries and tangles of dislocations in their interiors and grains without or with very low dislocation density in deformation cell interiors are observed (Fig. 3a). Heating to 250°C and additionally during rolling probably causes the observed recovery. Grains of mean size of 1.2 μm form in the 1 cycle (Table 2). Grain morphology as observed from ND is almost unchanged after subsequent cycles. However, the grain size decreased to 1 μm and sharp and clean cell boundaries form (Fig. 3b). Besides the 1 μm grains, fine grains of 500 nm in diameter were occasionally found in some regions. At higher strains ($\varepsilon \geq 3.2$), predominantly grains with well-defined boundaries form the microstructure and the average

grain size increases to 1.2 μm (Fig. 3c). The observed recovery and coarsening of the grain structure correspond to the evolution of hardness and strength of the material (Fig. 1a, Fig. 2a).

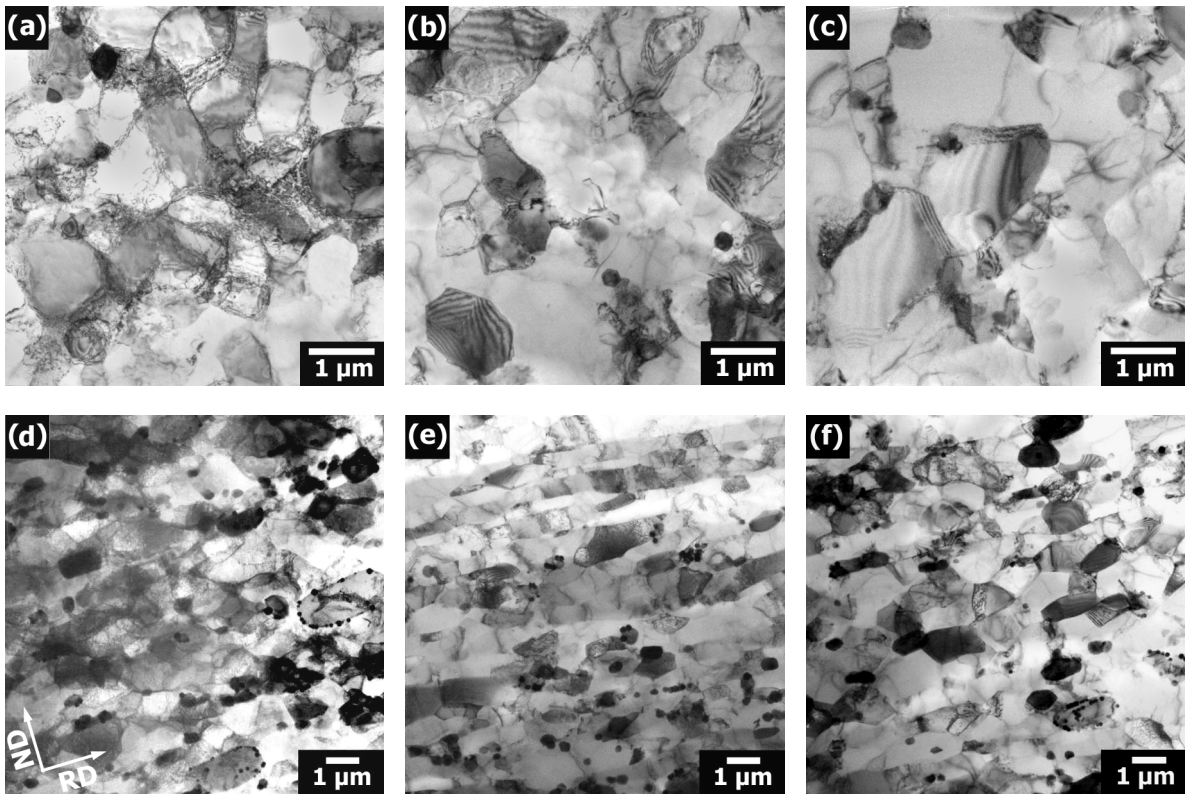

Fig. 3 TEM micrographs of sheets ARB processed at 250°C. View from ND: (a) 1 cycle, (b) 3 cycles and (c) 6 cycles. View from TD: (d) 1 cycle, (e) 3 cycles and (f) 6 cycles.

TEM observations from TD reveal a lamellar boundary (LB) structure, especially after the third cycle (Fig. 3e). The mean LB spacing after 3 cycles is of 550 nm. LB structure is typical for heavily deformed materials. It is well established [2] that heavy cold rolling and ARB processing result in grain subdivision by low-angle boundaries and that they convert to high-angle grain boundaries with increasing strain. Bands with much finer grains (80 to 250 nm in ND) were found in the sample processed by 6 cycles. Fig. 4 shows a general view of such band, the selective area diffraction pattern and corresponding band area. The thickness of these fine-grained bands varies from 200 nm to 3.5 μm. The occurrence of such UFG regions was reported in [3] for the material processed at 200°C. Grains of 0.1 to 0.3 μm in diameter (view from ND) were observed. In agreement with [2], it is assumed that bands of extremely refined grains are situated at the interface of bonded pieces and form due to the intensive friction and shear deformation involved in surface brushing. Thus, it can be concluded that ARB sheets are by their nature composite materials consisting of layers of nano-sized and sub- micrometer grains. The thickness of nano-sized layers is determined by the conditions of surface brushing preceding the bonding in each ARB cycle, i.e. by the load applied on sheet surface.

The positron lifetime spectra of ARB samples could be fitted by two exponential components with lifetimes of τ_1, τ_2 and relative intensities I_1, I_2 ($I_1 + I_2 = 100\%$). The shorter lifetime τ_1 corresponds to free positrons, the longer one τ_2 is a contribution of positrons trapped at defects. The development of lifetimes and I_2 with increasing ARB cycles is in Fig. 5a and Fig. 5b, respectively. In the first cycle τ_2 increases, whereas subsequently it becomes constant and lies close to 243 ps, which corresponds to dislocations in Al [7]. It indicates that positrons are trapped at dislocations introduced by ARB processing.

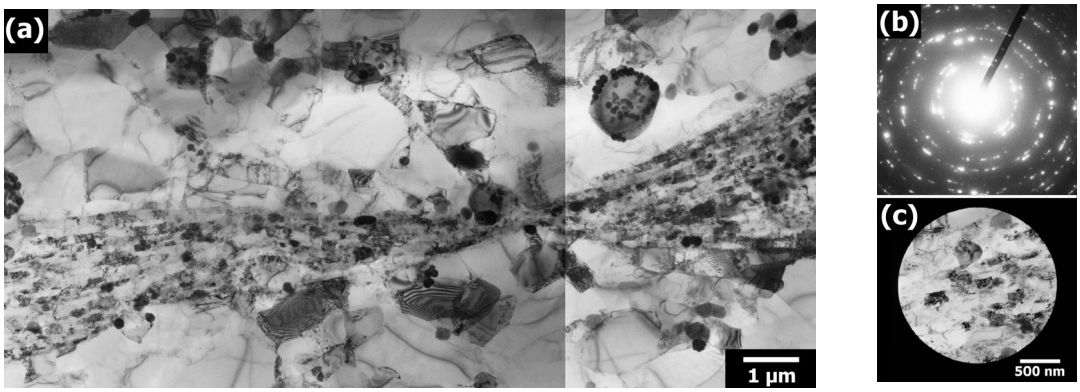

Fig. 4 Band of very fine grains in sample after 6 ARB cycles: (a) general view; (b) selective area (SA) diffraction pattern; and (c) corresponding size of the SA aperture. View from TD.

The increase of dislocation density is reflected by a significant increase of I_2 as compared to the initial material. In the same time, τ_1 decreases according to the two-state trapping model [5]. The next ARB cycles do not lead to further dislocation density increase and even a moderate I_2 decrease occurs. Thus, there is a rapid increase of dislocation density in the 1st cycle, while further cycles do not cause any significant change. Similar changes were observed in ARB deformed 4N Al [8]. Fig. 5b shows that $I_2 \approx 60$ % for the initial material, i.e. there is a large fraction of trapped positrons in the non-deformed alloy, contrarily to high purity Al, cf. [8]. Moreover, τ_2 for the initial material (Fig. 5a) is lower than that corresponding to dislocations in Al. It indicates that the defects in the initial material are not the same as these introduced by ARB (i.e. dislocations). A definite conclusion about the nature of defects in the initial material can not be made at present. Nevertheless, one can expect positron trapping in second-phase particles and/or at particle-matrix interfaces. Preliminary investigations performed by coincidence Doppler broadening support such a picture. The application of the two-state trapping model [5] to the data reveals that dislocations in ARB samples are distributed non-homogeneously, i.e. they are arranged in cell or subgrain boundaries. This hypotesis was verified by TEM examinations (Figs. 3 and 4).

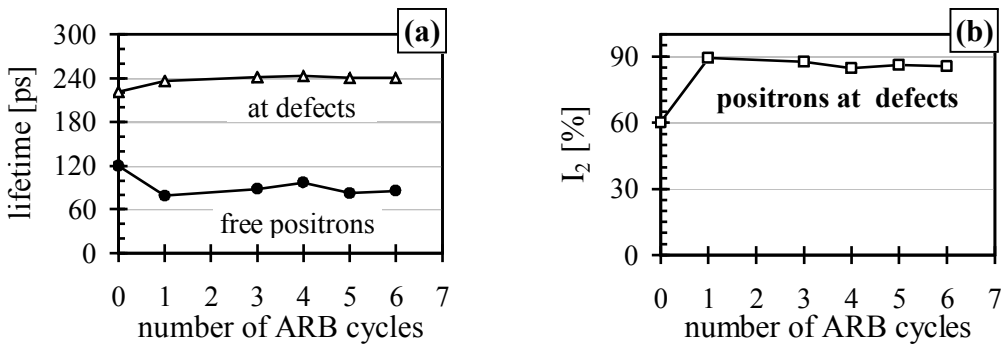

Fig. 5 Variation of positron lifetimes (a) τ_1 (free) and τ_2 (trapped) and intensity of the dislocation component of positron lifetime (b) with increasing number of ARB cycles at 250°C.

Fig. 6 shows hardness changes due to annealing. Small softening occurs at 200°C but hardness drops more rapidly above this temperature. Up to 300°C, HV decrease rate is smaller in samples ARB processed at 350°C and it depends on the induced strain. Up to 300°C, the sample processed at 250°C by 6 cycles softens more rapidly as compared to this with 2 cycles. The samples processed at 300°C and 350°C with 2 and 6 cycles behave in the same manner. LM examinations revealed that at temperatures < 350°C, only partial recrystallization occurs in annealed samples. The recrystallized fraction depends on both temperature and ARB cycles. At temperatures ≥ 350°C, the samples are fully recrystallized. Non-uniform grain size with coarser grains at bonded interface is observed in

samples processed by 2 ARB cycles. However, much more uniform grains form at annealing of the samples with 6 cycles. The finest and most uniform grains form in the samples ARB processed by 6 cycles at 250°C and the coarsest but uniform grain are in ARB samples with 6 cycles at 350°C.

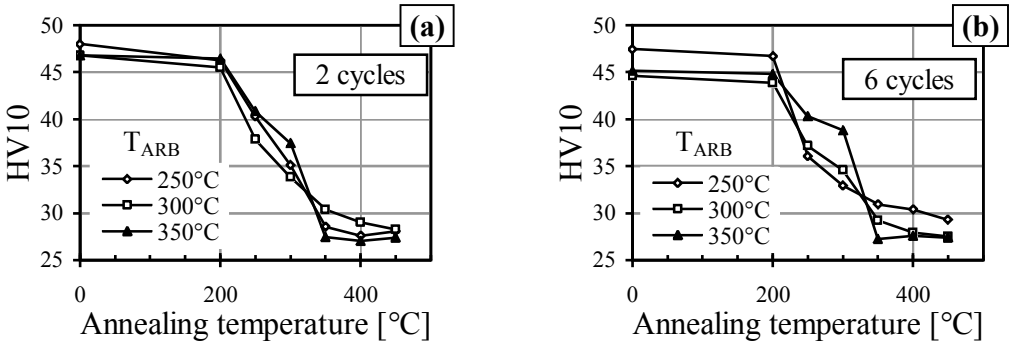

Fig. 6 Hardness variation of samples ARB processed at temperatures between 250°C and 350°C and then annealed for 1.8 ks at temperatures from 200°C to 450°C: a) 2 cycles; b) 6 cycles.

Summary

An investigation aimed at studying the effect of ARB temperature on the quality of bonding, microstructure and properties of TRC AA8006 sheet was carried out. It is demonstrated that materials with grains refined to submicrometer size (down to 80 nm in ND) can be prepared by ARB processing at 200°C or 250°C. Hardness and dislocation density increase significantly during 1-2 cycles and rise a little during subsequent cycles. Processing at higher temperatures results in better bonding but it produces smaller increase in hardness and thermally less stable refined grains.

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References

[1] N. Tsuji, N. Kamikawa, H.W. Kim and Y. Minamino: Ultrafine Grained Materials III (TMS, OH, 2004), p. 219.

[2] N. Tsuji, X. Huang and H. Nakashima: Evolution of Deformation Microstructure in 3D (Riso National Laboratory, Denmark 2004), p. 147.

[3] M. Karlík, P. Homola and M. Slámová: J. of Alloys and Compounds 378 (2004), p. 322.

[4] M. Slámová, P. Homola, M. Karlík, J. Čížek, I. Procházka and M. Cieslar: Evolution of Deformation Microstructure in 3D (Riso National Laboratory, Denmark 2004), p. 521.

[5] P. Hautojärvi and C. Corbel: Positron Solid State Physics, Eds. A. Dupasquier and A.P. Mills (IOS Press, Varena 1995), p. 491.

[6] F. Bečvář, J. Čížek et al.: Nucl. Instr. Meth. A. 443 (2000), p. 557.

[7] J. Čížek, I. Procházka, T. Kmječ and P. Vostrý: phys. stat. sol. (a) 180 (2000), p. 439.

[8] M. Slámová, P. Homola, P. Sláma, J. Čížek, I. Procházka and M. Cieslar: these proceedings.