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Hot Consolidation of Aluminium and Aluminium nano-MMC Powders by Equal Channel Angular Pressing

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In this work novel routes for consolidating aluminum powders via Equal Channel Angular Pressing (ECAP) are investigated. Furthermore, parameters were optimized for consolidating aluminium reinforced with nanoparticles (n-SiC) and nanofibers (Carbon nanotubes). Consolidating commercial purity aluminum powder with ECAP, approximately 60 % rise in hardness and strength were achieved compared to materials compacted by well established direct extrusion, at the same time losing about 2 times in ductility. Al-1 vol. % nano-SiC composites showed increase in hardness compared to composites consolidated by conventional methods.

Keywords: aluminium , powder metallurgy, metal matrix composites, Equal Channel Angular Pressing.

1. INTRODUCTION

Equal Channel Angular Pressing (ECAP) of metallic materials is an effective method for introducing high strains and microstructure refinement by simple shear. Therefore the strength of metals and alloys can be substantially increased. The ECAP processing has been extensively investigated for bulk aluminium alloys [1]. It is shown to be especially promising for age-hardenable alloys, whereas the strength properties of ECAP-treated non-age-hardenable alloys exhibit only minor improvements compared to well established cold rolling [2]. Quantitatively, the highest increase in strength is found for commercial purity aluminium. Ultimate tensile strength of 1050 aluminium can be increased by factor of about 5, ranging up to 192 MPa [3].

Recently, in addition to refinement of bulk materials, also compaction of metallic powders by ECAP has become into focus [4 – 7]. The metallic or composite powders are canned and pressed at elevated temperature through the die with backpressure (by inserted front stopper or by hydraulic valve) [5]. Therefore during one pressing cycle both the densification of the powders and the microstructure refinement takes place. This method allows consolidation of aluminium based nanocomposite powders, yielding high hardness and strength [8 – 9].

The mechanical properties of ECAP materials are mainly influenced by number of passes, processing temperature and the different routes (conventionally defined as A, B_c and C routes). The aim of the present study is to find the significance of these three variables on aluminum properties during consolidation by ECAP. The optimized processing parameters for increased ductility and strength of commercial purity aluminium will be transferred to compacting of Al-nanoSiC and Al-CNT (carbon nantube) composites.

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2. EXPERIMENTAL PROCEDURE

The atomized aluminium powders (grade AS011, ECKA GmbH) had a grain size of < 63 μm and purity of > 99.5 %. As the reinforcements SiC nanopowder (average BET grain size 32 nm) by plasma evaporation and condensation synthesis [10] and commercial grade multi-walled carbon nanotubes (Baytubes C 150 P, Bayer Materials Science AG) were used. The powders were mixed by attritor milling, as described in [11].

The loose powders were loaded in a copper capsule and heated in a resistance furnace with holding time of one hour in air atmosphere. The samples were transported to the non-heated ECAP die during 3 – 4 seconds and pressed at the rate of 5 mm/s through the angle of 90°. For all the samples front stopper as back-pressure was used. The pressure for compacting 16 mm × 16 mm square specimens was in the range of 300 MPa – 400 MPa.

For optimizing the ECAP processing parameters for increased mechanical properties, statistical experimental planning using 2-level factorial design was used. The analysis and optimization was performed with the support of software Design-Expert 7.0 by Stat-Ease Inc.

The range of pre-heating temperature was set from 150 °C up to 350 °C and number of passes from 2 up to 6. The angle of rotation was 0° (route A), 90° (route B) and 180° (route C). The full factorial design of experiments consisted of 9 runs (as shown in Table 1). Six repeated tests were performed at the center point (Run 1) for determining the reliability of the results.

The pressed samples were removed from the capsules and examined for mechanical performance (hardness, strength), density and appearance (material loss/length of the sample, quality/consistence of the surface). Tensile strength was measured on specimens with gauge diameter of 5 mm and gauge length of 25 mm.

Vickers hardness at 10 N was taken from the center area of the sample cross section. Archimedes principle was used for the density measurements. As a reference hot extrusion was used for compacting Al powder. The powder

Table 1. Process variables of the ECAP consolidation of aluminium powders and obtained response values of compacts

Run	Temp. °C	No. passes	Route	Sample integrity	Sample length, mm	UTS,** MPa	Elongation, %	Density, %	Hardness, HV_{10}
1*	250	4	B	5.2(+/–0.8)	60(+/–1)	148(+/–30)	6.4(+/–3.8)	99.4(+/–0.4)	49.4(+/–1.7)
2	350	6	A	4	57	146	12.8	99.0	41.4
3	150	2	C	5	55	75	0.2	99.4	50.6
4	150	6	A	1	38	188	9.1	99.0	53.9
5	350	2	C	7	62	54	0.4	99.3	44.5
6	200	2	A	7	51	159	11.3	99.3	49.5
7	350	2	A	7	56	147	12.7	99.6	46.1
8	150	6	C	2	34	82	0.1	99.6	52.1
9	350	6	C	3	46	138	9.5	99.3	41.6

*Test results from average of 6 samples.

**Ultimate Tensile Strength.

was loaded in an extrusion die with 14 : 1 of extrusion ratio and heated in a resistance furnace in air at 500 °C. The die was transported to the hydraulic press and extrusion was carried out at the rate of 1 mm/s.

3. RESULTS AND DISCUSSION

The compacted samples were evaluated for sample integrity and length after processing, tensile strength, elongation to fracture, density and hardness (see Table 1).

3.1. Structural properties

The specimen integrity after processing was evaluated qualitatively for fractures, capsule removal and tip degradations.

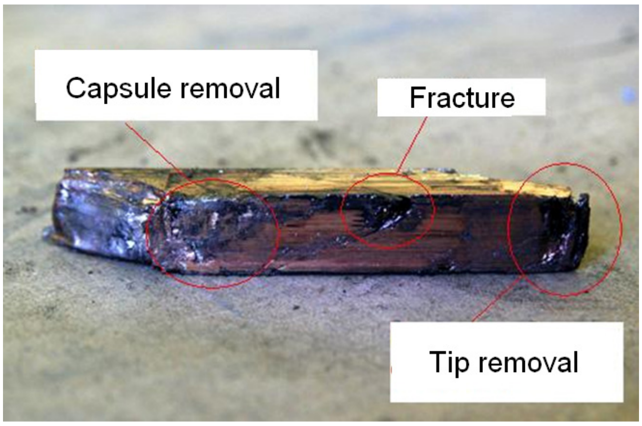
Fig. 1 shows two specimens, one with grade 7 (run 5 – Fig. 1, a) and another with grade 3 (run 9 – Fig. 1, b).

It is know that the extent of backpressure is critical for the defects occuring during the ECAP processing [12]. Materials with increased hardness are prone to cracking and defective structure. Therefore higher temperatures or higher back-pressures are needed for decreased defects. In this work the extent of back-pressure could not be adjusted, as front stoppers were used. The amount of defects were not affected by the ECAP route, but different structure of the defects was observed at different routes. The route B was distinguished by the fractures at the corner of the sample, whereas tip removal was characteristic for route A. The length of the sample is also decreased with the decrease in preheating temperature. At lower temperature the material has higher brittleness and therefore the tips are prone to cracking. The density of the samples is always above 99 % and not significantly affected by the processing temperature.

3.2. Mechanical properties

The voids and fractures in the samples resulted in highly fluctuating strength and elongation results. This is also characterized by the repeated tests from run 1 in Table 1, where large error values were obtained. Nevertheless, the highest values that were obtained in this work (ultimate tensile strength of 188 MPa at 9.1 % of elongation for run 4) is outstanding regarding the results published in the literature [3]. Nevertheless, it must be emphasized that the integrity of the sample in this case (run 4 in Table 1) was the smallest, having large amount of deep cracks and capsule degradation.

a



b

Fig. 1. Examples of compacted specimens. a – from run 5, 350 °C, 2 passes, route C (length of the specimen 62 mm); b – from run 9, 350 °C, 6 passes, route C (length of the specimen 46 mm)

For comparison tensile testing samples were prepared by powder extrusion at 500 °C. Compared to hot extrusion the ECAP can yield ultimate tensile strength increase at around 60 % with the decrease in elongation from 25.2 % down to 9.1 % (see Fig. 2).

The principal factor for increase in material hardness after ECAP was the preheating temperature (Fig. 4, a). Although there is also certain negative effect from number of passes and angle of rotation, these effects lay in the error of replicate measurements (Fig. 4, a). Figure 4, b, represents the predicted hardness values for varying preheating temperatures and number of cycles at route A.

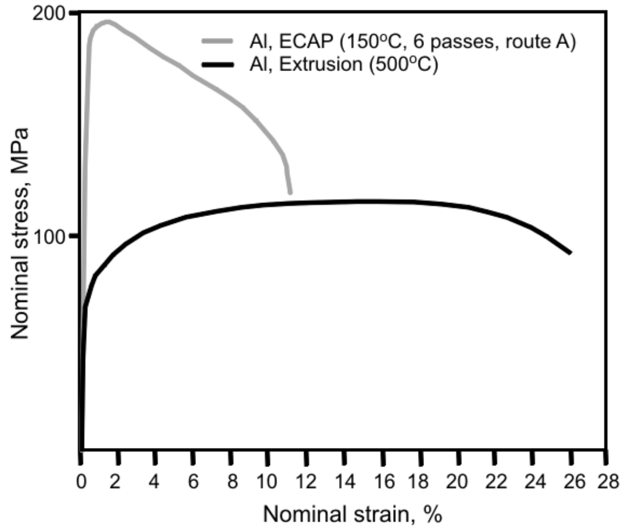


Fig. 2. Tensile strength of ECAP consolidated (run 4) and hot extruded aluminium

Obviously the temperature is the principal factor for hardness difference. The reason for negative effect of number of cycles is not known.

3.3. Optimisation of processing parameters for compacting aluminium based nanocomposites

Due to the large number of failed specimens for tensile testing the optimization of processing parameters could not be performed according to the responses of ultimate tensile strength (UTS) and elongation to fracture. It is well known that there is a constant relationship between strength and hardness of the metallic materials. For aluminium alloys UTS ~ 3HV is obtained [13]. In the present work the UTS values for which the elongation was greater than 1 % were compared with the Vickers hardnesses. The ratio of UTS:HV for the studied materials was 3.39 (+/–0.16) which is consistent with the results for ECAP processed aluminium [14]. The hardness and UTS of the materials are in good agreement (Fig. 3, A) and for further strength optimization hardness response was used.

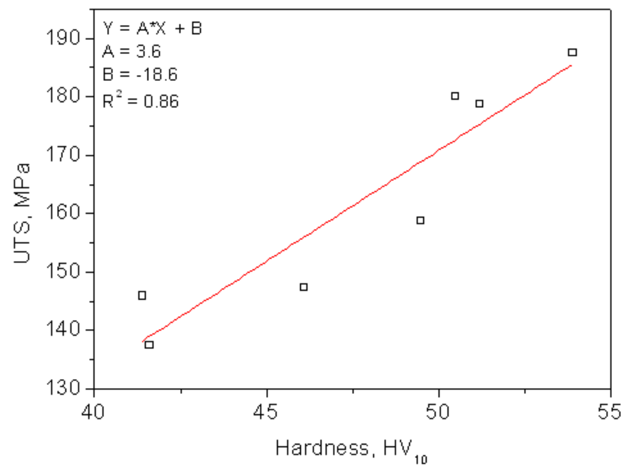
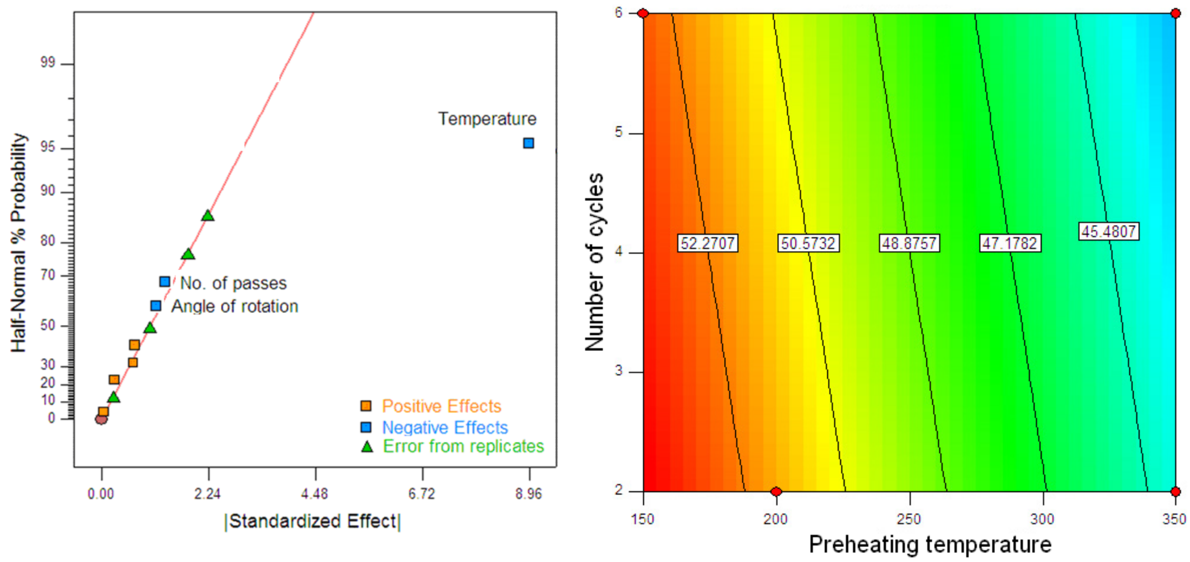


Fig. 3. Hardness versus ultimate tensile strength for samples with elongation at least 1 %

For the optimization of the processing parameters (temperature, number of passes, route) the significance of response values were determined in a scale of five for maximized result. The significance of each parameter is outlined in Table 2. The significance is inserted to the model to calculate the gradients in the experimental space.

The resulting optimized experimental conditions lay at 2 passes at the temperature of 274 °C through route A. The model response values are yielding hardness of 47.9 HV10



a b

Fig. 4. Half-Normal plot of hardness response for all the input parameters (a) and predicted aluminium hardness HV_{10} (b) according to the preheating temperature and number of cycles at route A. Measured hardness values indicated with red dots in (b)

and 168 MPa of UTS for pure aluminium. These input parameters were selected for compacting pre-mixed Al-1 vol.% nanoSiC and Al-6 vol.% CNT composites.

Table 2. Significance of response values for optimized ECAP technology

Response	Significance
Sample integrity	***
Sample length	**
Density	****
Hardness	*****

Due to the lack of the back pressure there was number of cracks in the samples and the nanocomposites were brittle. Therefore the strength properties of the composites could not be measured. Hardness of the composites was significantly increased, being 108 (+/–5) MPa for Al-6 vol.%CNT and 180 (+/–12) MPa for Al-1 vol.% nanoSiC. The hardness of consolidated Al-1 vol.% n-SiC is higher than achieved previously by hot pressing or extrusion [11]. This can be due to the further matrix grain refinement during ECAP compaction.

4. CONCLUSIONS

Processing parameters of preheating temperature, number of cycles and route for Equal Channel Angular Pressing were studied in this work. It was shown that the principal influence on the mechanical response of the materials and integrity of the samples is from the preheating temperature. The hardness and strength of the compacts decrease and integrity and length of the samples increase with the preheating temperature. The consolidation of the samples was finalized already after two cycles, disregarding the preheating temperature.

The aluminium reinforced with carbon nanotubes or nano-SiC were consolidated with the optimized processing parameters. Disregarding the large number of fractures in the composites, the hardness could be increased significantly.

Acknowledgments

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