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Electrical conductivity and mechanical properties of the solid state recycled EN AW 6082 alloy

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Abstract: In the last few years there is a demand for the new technologies development in order to increase scrap reuse potential and CO_2 emission savings. This paper deals with novel aluminum recycling process using severe plastic deformation (SPD). Aluminum was recycled from machined chips without any remelting (solid state recycling-SSR) in order to reduce environmental pollution and to increase material yield during the process. Machined chips were cleaned, compacted and hot extruded. After hot extrusion semi-finished product (aluminum bar) was processed with equal channel angular pressing (ECAP) in order to improve mechanical properties and electrical conductivity. ECAP process temperature and the number of recycled samples pass through tool where varied. After SSR process electrical conductivity and mechanical properties of recycled samples were evaluated. The efficiency of the SSR process was confirmed and quality recycled samples were produced.

Izvorni znanstveni rad
Sažetak: Posljednjih godina postoji potreba za razvijanje novih tehnologija s ciljem povećanja potencijala ponovne upotrebe otpada i smanjenja emisije CO_2. Ovaj rad se bavi inovativnom tehnologijom recikliranja aluminija koristeći veliku plastičnu deformaciju (engl. "severe plastic deformation"-SPD). Aluminij je recikliran iz odvojene čestice bez pretaljivanja (recikliranje u čvrstom stanju, engl. solid state recycling- SSR) s ciljem smanjenja zagađenja okoliša i povećanjem iskorištenja materijala tokom procesa. Odvojene čestice su očišćene, sabijene i istisnute u toplom stanju. Nakon istiskivanja u toplom stanju, polu-proizvod (aluminijska šipka) je obrađen s kutnim kanalnim istiskivanjem (engl. "equal channel angular pressing"-ECAP) s ciljem poboljšanja mehaničkih svojstva i električne provodljivosti. Temperatura ECAP procesa i broj prolaza recikliranih uzoraka kroz alat su varirani. Nakon SSR procesa električna provodljivost i mehanička svojstva recikliranih uzoraka su određena. Učinkovitost SSR procesa je potvrđena i dobiveni su kvalitetni reciklirani uzorci.

1. Introduction

In the last few years there is a demand for development new technologies in order to increase scrap reuse potential and emission savings [1]. Aluminum is second most recycled metal and it is of the main importance for further development in aluminum recycling technology. In this paper aluminum recycling without remelting was used, so called solid state recycling (SSR). The main difference compared with conventional recycling is in energy and material savings. High metal reactivity and material losses are responsible for lower material yield in conventional recycling process [1-2]. Usually, machining chips are recycled by SSR process.
Nevertheless, sheet, foils or wire could also be recycled. The most used method for SSR is hot extrusion, but lately severe plastic deformation processes are utilized in order to improve recycled samples mechanical properties. Some of the used processe are: incremental equal channel angular pressing (iECAP) integrated into hot extrusion [3], high pressure torsion (HPT) [4], friction stir extrusion (FSE)[5].
It has been shown by various authors that if all necessary conditions are fulfilled quality recycled samples can be obtained via solid state recycling route. These conditions are the combination of high temperature, plastic deformation and pressure [6]. Other authors have developed the mathematical model to describe influence on metal material solid bonding and according to the model high temperature, increase in normal contact stress, shear stress and strain lead to better material bonding [7].
In this paper, solid state recycling was performed by the combination of direct hot extrusion (DE) and following ECAP process. Mechanical and physical properties (electrical conductivity) are compared after DE and also after combination DE+ECAP. However, ECAP was

performed at various process parameters in order to evaluate process influence on SSR samples quality. Usually, researchers have investigated mechanical properties of SSR samples, microstructure and density. [8]. According to literature review, electrical conductivity of SSR samples was not investigated. The Al-Mg-Si alloys (6xxx) have been widely used as conductors due to the good combination of strength and electrical properties compared with other Al alloys [9]. Selected metal for this research is relatively new alloy EN AW 6082 due to excellent behavior during machining process.

Production of Al-Mg-Si wires usually include thermo-mechanical processing which consist of solution treatment, water quenching and cold drawing into wires. Following this procedure second-phase precipitates Mg_2Si are formed in the grain interior. Such microstructure show enhanced precipitation hardening and electrical conductivity slightly increment. The reason for such material behavior is explained as a result of partial purification of the Al matrix from Mg and Si solute atoms since the solute atoms dissolved in the matrix and Guinier-Preston (GP) zones are responsible for the determinal effect on electrical conductivity [9-10]. Usually, well-known Matthiessen's rule [10] is used to describe electrical resistivity (ρ) dependence on several microstructural features:

$$\rho=\rho_0+\Delta\rho_S+\Delta\rho_P+\Delta\rho_V+\Delta\rho_D+\Delta\rho_B \quad (1)$$

ρ_0 describe resistivity of pure solvent metal and $\Delta\rho$ stand for the increase in electrical resistivity due to atoms in solid solution (S), precipitates (P), vacancies (V), dislocation (D) and grain boundaries (B). Electrical resistivity is the inverse value of the electrical conductivity. Good combination of mechanical and physical properties has always been a great challenge for electric conductors production. Lately, , the number of attempts increases in order to produce electrically conductive materials with high strength by severe plastic deformation approach without any electrical conductivity decrement [9-13].

With this approach, SPD is used to refine microstructure to ultra-fine grain structure (UFG), while SPD at elevated temperature cause decomposition of solid solution and formation of nano-sized second phase precipitates via dynamic aging. UFG materials with second phase precipitates can have high mechanical properties, but also enhanced electrical conductivity due to the very low content of solute atoms and absence of GP zones in Al matrix. These nano-sized precipitate have been identified as β'- Mg_2Si, and they are formed during SPD processing of Al-Mg-Si aluminum alloy at elevated temperatures [11-13].

The main aim of this paper is to show the possibility to produce material with good mechanical properties from small metal waste and with good electrical conductivity by solid state recycling process utilizing severe plastic deformation.

2. Experimental procedure

In order to investigate the possibility of EN AW 6082 alloy recycling via SSR recycling route, direct hot extrusion (DE) in combination with ECAP process was utilized. Chemical composition of selected aluminum alloy EN AW 6082 : 0,7-1,3 % Si, 0-0,5% Fe, 0-0,1% Cu, 0,4-0,1 % Mn, 0,6-1,2 % Mg, 0-0,25% Cr, 0-0,2% Zn, 0-0,1% Ti, Other 0-0,1%.

Metal waste in form of machined chips from milling and turning are taken from the real industrial manufacturing process. There were few steps to perform SSR route:

- Cleaning of contaminated machined chips by the ultrasonic bath at temperature of 60°C in a time of 20 min utilizing universal detergent, Fig. 1b.
- Briquetting of machined chips into 38 mm diameter and 70 mm high briquettes with 300 kN force, Fig. 1a.
- Briquettes preheating in a time of 30 min on extrusion temperature and direct hot extrusion at 400 °C and 450°C with 7.11 extrusion ratio.
- ECAP processing of the extruded bars with 15 mm diameter at three temperatures: 20°C, 160°C and 300°C. At 20°C and 160°C 1 and 3 passes were applied, while at 300 °C only one pass was applied.

a b

Figure 1. a) Chips cleaning utilizing ultrasonic bath b) Machined chips briquetting phase

Figure 1. a) Čišćenje odvojene čestice ultrazvučnom kupkom b) Faza briketiranja odvojenih čestica

Force measurement during briquetting phase was achieved utilizing HBM load cell C6A 1MN. After SSR route (DE+ECAP) mechanical properties and electrical conductivity of all obtained samples were evaluated. Properties of recycled samples obtained only with DE are also evaluated and compared with referent values of extruded bar from manufacturer in O (annealed), T4 (solution heat treated and naturally aged) and T6 (solution heat treated and artificially aged) temper conditions. Systematized overview for all 7 solid state recycling processing routes is given in Tab. 1, as well as mechanical and physical properties of recycled samples.

Table 1. Mechanical and physical properties of solid state recycled samples

Tablica 1. Mehanička i fizička svojstva reckliranih uzorka u čvrstom stanju

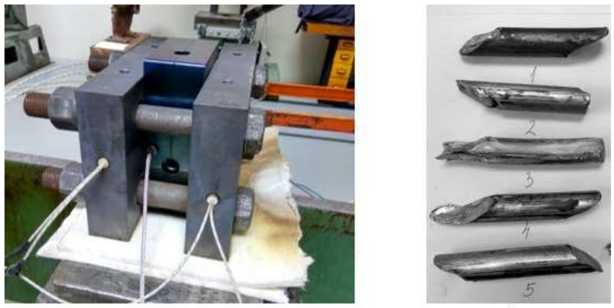
<table>
<tr><td colspan="8">Properties of solid state recycled samples obtained utilizing DE+ECAP route</td></tr>
<tr><td>Sample</td><td>ECAP temperature [°C]</td><td>ECAP pass</td><td>DE temperature [°C]</td><td>σ_{UTS} [MPa]</td><td>$\sigma_{0,2\%}$ [MPa]</td><td>δ [%]</td><td>IACS [%]</td></tr>
<tr><td>1</td><td>300</td><td>1</td><td>450</td><td>139,7</td><td>73</td><td>31,3</td><td>47,48</td></tr>
<tr><td>2</td><td>160</td><td>1</td><td>400</td><td>174,7</td><td>127</td><td>23,8</td><td>46,05</td></tr>
<tr><td>3</td><td>160</td><td>3</td><td>450</td><td>174,6</td><td>144</td><td>18,3</td><td>46,54</td></tr>
<tr><td>4</td><td>20</td><td>1</td><td>450</td><td>249,4</td><td>236</td><td>8,2</td><td>43,80</td></tr>
<tr><td>5</td><td>20</td><td>3</td><td>400</td><td>265,9</td><td>227</td><td>10</td><td>44,01</td></tr>
<tr><td colspan="8">Properties of solid state recycled samples obtained utilizing DE route</td></tr>
<tr><td>Sample</td><td>DE temperature [°C]</td><td>σ_{UTS} [MPa]</td><td>$\sigma_{0,2\%}$ [MPa]</td><td colspan="2">δ [%]</td><td colspan="2">IACS [%]</td></tr>
<tr><td>6</td><td>400</td><td>150</td><td>91</td><td colspan="2">24,3</td><td colspan="2">43,05</td></tr>
<tr><td>7</td><td>450</td><td>160</td><td>101</td><td colspan="2">24,6</td><td colspan="2">44,5</td></tr>
<tr><td colspan="8">Conventionally produced EN AW 6082 extruded bar properties</td></tr>
<tr><td></td><td>State</td><td>σ_{UTS} [MPa]</td><td>$\sigma_{0,2\%}$ [MPa]</td><td colspan="2">δ [%]</td><td colspan="2">IACS [%]</td></tr>
<tr><td></td><td>O</td><td>≤160</td><td>≤110</td><td colspan="2">≥14</td><td colspan="2">/</td></tr>
<tr><td></td><td>T4</td><td>≥205</td><td>≥110</td><td colspan="2">≥14</td><td colspan="2">42</td></tr>
<tr><td></td><td>T6</td><td>≥295</td><td>≥250</td><td colspan="2">≥8</td><td colspan="2">44</td></tr>
</table>

In Tab. 1 σ_{UTS} stands for ultimate tensile strength (UTS), $\sigma_{0,2\%}$ stands for yield strength, δ stands for elongation and % IACS is the unit for electrical conductivity. Fig. 2a shows utilized ECAP tool and Fig. 2b shows obtained recycled samples after the process. The main goal of the ECAP process is to introduce notable plastic strain into the material and to refine microstructure. Tool geometry in ECAP process is mainly defined by two angles, inner die angle ϕ and outer die angle ψ. Iwahashi et al. [14] established simple analytical approach according to which plastic shear strain γ in the shear zone determined by the outer corner angle is function of the die angles ϕ and ψ:

$$\gamma = 2\cot\left(\frac{\phi}{2}+\frac{\psi}{2}\right)+\psi\,\mathrm{cosec}\left(\frac{\phi}{2}+\frac{\psi}{2}\right) \qquad (2)$$

ECAP tool in this work have inner die corner angle value 90°, and outer die angle 12° defined with 3 mm radius. The diameter of the ECAP tool channel is 15.1 mm. The lubrication used on room temperature and 160 °C was graphite grease, while on 300°C lubrication based on Mo_2S was utilized. Temperature measurement was taken with K-type thermocouple probe and regulation was achieved using HOTSET cartridge heaters and PID controller. Every sample was preheated 10 min inside ECAP tool and 10 min after channel pass. ECAP was performed by pressing one sample on the other in order to achieve semi-continuous process.

Mechanical testing was performed according to the ASTM E8 standard for metal material tensile testing. The used device was "Instron 8801" 5 kN universal machine, Fig. 4a. Tensile specimens were prepared with gauge original length 11 mm and diameter 2.5 mm.

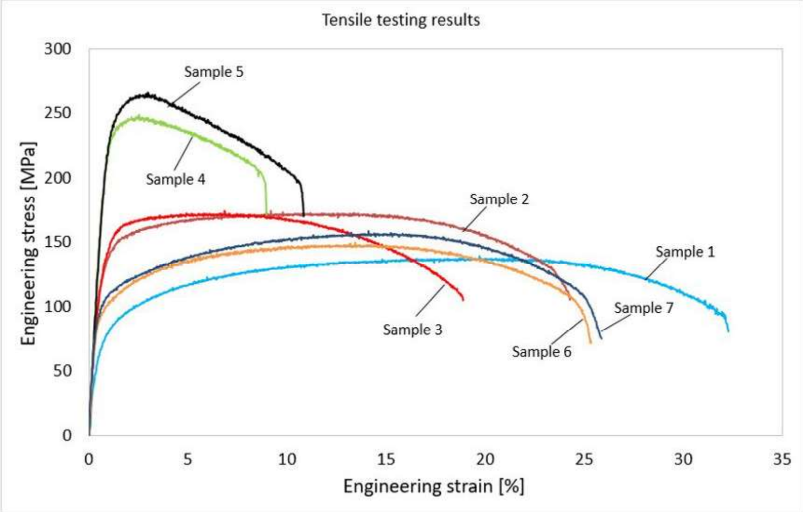


a b

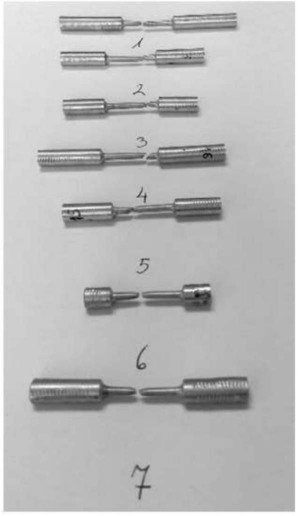
Figure 2. a) ECAP tool b) Recycled samples (DE+ECAP),

Slika 2. a) ECAP alat, b) Reciklirani uzorci (DE+ECAP)

Samples were machined from the central part of the recycled samples in order to obtain homogeneous microstructure. Tensile testing was performed at room temperature and initial strain rate was $1.8\cdot10^{-3}$ s^{-1}. Fig. 3a shows engineering stress vs. engineering strain diagrams for all 7 solid state recycled samples. Tensile samples after testing are presented in Fig. 3b. The electrical conductivity of the alloy was measured at room temperature using the eddy current method on Olympus NORTEC 600C device according to the ASTM E 1004 standards, Fig. 4b. The electrical conductivity was expressed as IACS (%) unit, which stands for International Annealed Cooper Standard. At least 20 electrical conductivity measurements were taken for each sample and the arithmetic value was taken as the final value.



a



b

Figure 3. a) Engineering stress vs. engineering strain b) Samples after tensile testing
Slika 3. a) Dijagram naprezanje u odnosu na relativno produljenje b) Uzorci nakon vlačnog testiranja

a

b

Figure 4. a) Universal tensile testing machine ''Instron 8801'' b) Eddy current measuring device
Slika 4. a) Univerzalna vlačna kidalica ''Instron 8801'' b) Uređaj za mjerenje vrtložnim strujama

3. Results and analysis

After experiments, obtained results for the ultimate tensile strength are in the range from 139,7 MPa to 265,9 MPa, elongation is in the range from 8,2 % to 31,3% and yield strength is in the range 73 MPa to 227 MPa. These properties are achieved without any additional heat treatment, such as artificial aging.

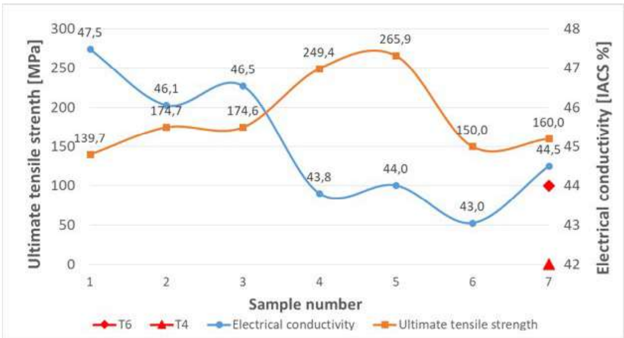
Specimens that were recycled only by hot extrusion have similar UTS (sample 6 UTS=150 MPa and sample 7 UTS= 160 MPa) as EN AW 6082 aluminum alloy in annealed condition (O), Tab. 1. However for both mentioned recycled samples elongation seems to be twice of the minimum elongation for annealed EN AW 6082, Tab. 1. However electrical conductivity (EC) of the recycled samples is 43,05% IACS (sample 6) and 44,5% IACS (sample 7) which is closer to EN AW 6082 in T6 temper (44% IACS).

According to the results, electrical conductivity can be increased by ECAP procedure at elevated temperature (300 °C) and for sample 1 EC is 47.48% IACS. However, UTS and YS are low, 139.7 MPa and 73 MPa, respectively, while elongation is, 31.3%.

In order to increase recycled samples quality and mechanical properties, some of the samples are processed with ECAP tool at room temperature. For samples 4 and 5 UTS is 249.4 MPa and 265.9 MPa, respectively. These values are much higher than EN AW 6082 in T4 temper and little lower than mentioned alloy in T6 temper, Tab 1. Sample 4 and 5 have EC 43.80% IACS and 44.01% IACS, respectively. YS for sample 4 and 5 is 236 MPa and 227 MPa, respectively. This means an increase for 114% in YS for sample 4 and 106% for sample 5 compared with EN AW 6082 in T4 temper. Sample 5 also have 25% higher elongation than EN AW 6082 in T6 temper. Sample 2 and 3 after hot extrusion are processed with ECAP tool at 160 °C and UTS is 174.7

MPa and 174.6 MPa, respectively. The electrical conductivity of sample 2 and 3 is 46.05% IACS and 46.54% IACA, respectively. This means enhanced EC compared with EN AW 6082 in T6 and T4 temper, Fig. 5c. Despite lower UTS than EN AW 6082 in T4 and T6 temper, elongations are much higher while for samples 2 and 3 they are 23.8% and 18.3%, respectively. YS compared with minimum listed value EN AW 6082 in T4 temper is increased for 15% and 30% for samples 2 and 3, respectively.

Fig. 5a show values of electrical conductivity compared with UTS, Fig. 5b shows values of EC compared with YS and Fig. 5c shows values of EC compared with elongation for 7 SSR samples. .



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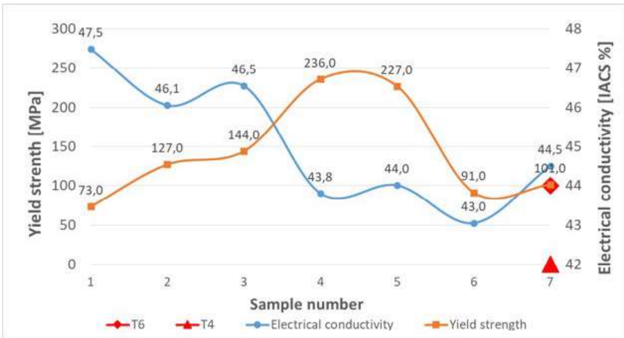

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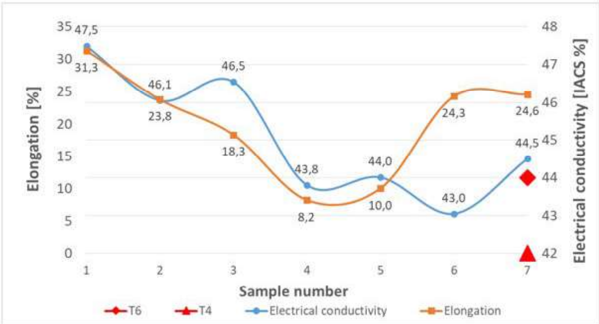

c

Figure 5. a) Ultimate tensile strength and electrical conductivity for 7 SSR samples b) Yield strength and electrical conductivity for 7 SSR samples c) Elongation and. electrical conductivity for 7 SSR samples

Slika 5. a) Vlačna čvrstoća i električna provodljivost 7 uzoraka recikliranih u čvrstom stanju b) Granica razvlačenja i električna provodljivost 7 uzoraka recikliranih u čvrstom stanju c) Elongacija i električna provodljivost 7 uzoraka recikliranih u čvrstom stanju

According to Fig. 5a and Fig. 5b it can be clearly indicated that with increase in ultimate tensile and yield strength electrical conductivity decrease. On the other hand with elongation decrement, electrical conductivity decrease, except for samples recycled only with DE, Fig. 5c. However, samples processes only with DE have lower YS and UTS, without any EC increment, Fig. 5a and 5b.

The attention should be at sample 2 and 3, which have considerably higher UTS and YS and enhanced electrical conductivity compared with EN AW 6082 in T4 and T6 temper. Electrical conductivity and mechanical properties are increased compared with samples 6 and 7. This indicates that with severe plastic approach quality SSR samples can be obtained and with enhanced electrical conductivity. Furthermore, additional improvement in UTS and YS are showed for samples 4 and 5, without any EC properties degradation.

Finally, due to a large number of influential parameters and their interaction further optimization of the SSR process should be performed in order to achieve even better combination of mechanical and physical properties.

4. Conclusion

According to this investigation, solid state recycling of EN AW 6082 alloy can be successfully and effectively performed by the combination of DE and ECAP process. Obtained recycled samples have an excellent combination of mechanical and physical properties without any remelting phase, which means low energy consumption. Manipulation of recycled samples properties can be easily achieved by changing process parameters. Following conclusion can be drawn:

- Improvement of mechanical properties and physical properties of SSR samples produced only with DE can be achieved by additional SPD utilizing ECAP process.

- Samples processed with DE+ECAP at room temperature have enhanced UTS and YS, without any heat treatment, while EC remains high. Sample 5 has 10% lower UTS and 9% lower YS compared with EN AW 6082 in T6 conditions, while elongation is 25% higher and electrical conductivity is equal. However, only 3 pass through ECAP were performed. Increase in pass number should result with higher mechanical properties.
- Samples processed with DE+ECAP at 160°C have an excellent combination of mechanical properties and EC. Sample 3 has higher YS, elongation and EC than minimum listed values of EN AW 6082 aluminum alloy at T4 temper for 30%, 30.7% and 10.8%, respectively. However, UTS is 15% lower.
- Samples processed with DE and ECAP at 300 °C have 47.48% IACS. However, UTS and YS are 139.7 MPa and 73 MPa, respectively, while elongation is very high, 31.3%.

Finally, further investigation directions for solid state recycling should be based on severe plastic deformation approach with DE and ECAP at the temperature range between 100° and 200°C in order to optimize dynamic recrystallization influence on physical and mechanical properties. Except electrical conductivity, it is very important to determine recycled samples density, metallography and microstructure. Investigation of additional heat treatment on mechanical properties and especially physical properties should also be included.

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