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## STUDIES OF ADVANCED TECHNOLOGIES USED IN THE MANUFACTURE OF PRODUCTS FROM ALUMINIUM ALLOYS

### BADANIA NAD ZAAWANSOWANYMI TECHNOLOGIAMI WYTWARZANIA WYROBÓW ZE STOPÓW AI

The test stands already put in operation and still under development, installed in the Light Metals Division Skawina of the Institute of Non-Ferrous Metals in Gliwice are described. The presentation includes 5MN horizontal direct-indirect extrusion press, 2,5MN vertical forging press, "MeltechConfex MC-260" device for continuous rotary extrusion process, "Melt-Spinning" plant for casting of thin strips, melting-casting plant for Mg alloy billets, heat treatment line, and new testing equipment, including Instron 5582 100 kN and Instron 600DX 600 kN testing machines with attachments for tests carried out at low and high temperatures, automatic hardness tester for HB, HV and HRC hardness measurements, portable X-ray diffractometer for measurement of internal stresses, optical emission spectrometer with channels for the analysis of aluminium and magnesium alloys, STEM 200kV transmission electron microscope, and apparatus for hydrogen content measurement in the solid state. Based on the experimental potential and apparatus available, present research opportunities were discussed and modern trends in the advanced technology of Al and Mg alloys fabrication were outlined.

*Keywords:* Al alloys, Mg alloys, heat treatment, mechanical properties, nanostructure, plastic deformation

Przedstawiono zainstalowane w Oddziale Metali Lekkich w Skawinie, Instytutu Metali Nieżelaznych w Gliwicach i będące w trakcie realizacji stanowiska doświadczalne (prasę poziomą współbieżno-przeciwbieżną o nacisku 5MN do wyciskania, prasę pionową kuzienną 2,5MN, urządzenie do ciągłego wyciskania na kole „MeltechConfex MC-260”, instalację do odlewania cienkich taśm „Melt-Spinning”, stanowisko topielno-odlewnicze wlewków ze stopów Mg, linię obróbki cieplnej) oraz nową aparaturę badawczą (maszyny wytrzymałościowe Instron 5582 100 kN oraz Instron 600DX 600 kN wraz z wyposażeniem do badań w obniżonych i podwyższonych temperaturach, automatyczny twardościomierz do pomiaru twardości metodą HB, HV, HRC, przenośny dyfraktometr rentgenowski do pomiaru naprężeń własnych, emisyjny spektrometr optyczny z kanałami do analizy stopów aluminium i stopów magnezu, transmisyjny mikroskop elektronowy STEM 200 kV, aparat do pomiaru zawartości wodoru w stanie stałym). W oparciu o potencjał doświadczalny i aparaturowy przedstawiono zarówno możliwości badawcze oraz nakreślono nowoczesne kierunki badań w obszarze zaawansowanych technologii stopów Al i Mg.

### 1. Introduction

The Light Metals Division Skawina of the Institute of Non-Ferrous Metals in Gliwice, Poland, has become the recipient of grants from the European Regional Development Fund under the following projects:

– No. POIG.02.02.00 -00-012/08-00 **"Retrofitting the Research Infrastructure of Małopolskie Centre for Innovative Technologies and Materials"**, The Operational Programme Innovative Economy 2007-2013, Priority 2. R & D Infrastructure, Measure 2.2. Support for development of research infrastructure of scientific entities,

– No POIG.02.01.00-12-062/09 **"Advanced Technology Incubator for Plastic Working of Light Alloys**

**based on Al and Mg"**, The Operational Programme Innovative

Economy 2007-2013, Priority 2. R & D Infrastructure, Measure 2.1. Development of high research potential centres,

- and of other measures afforded by the Ministry of Science and Higher Education for the purchase of pilot installations and test equipment for plastic working of Al and Mg alloys.

Currently, the Light Metals Division Skawina operates the following equipment:

– 5MN horizontal direct-indirect press with additional accessories which include: press handling equipment with puller, induction heater for zone heating of bil-

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- lets, stretch- straightening machine, installation for “on-line” quenching-type solution heat treatment,
- 2,5MN vertical forging press with accessories for physical modelling of the extrusion and die-stamping process,
- ”MeltechConfex MC-260” device for continuous rotary extrusion process, which allows using as a feed-stock both solid materials (rods, wires) and loose materials (RS powders and chips),
- PT-128 laboratory test stand allowing investigations of the process of the generation of electrolytic coatings on light metals, including anodic oxide coatings, conversion coatings (chromate and phosphate) and electrolytic coatings (also composite ones), in solutions of different chemical aggressiveness at both high and low temperatures,
- heat treatment line (furnaces for solution heat treatment and aging) with full control, monitoring and temperature recording system.

In 2011, test stands for melting and casting of Mg alloy billets will be installed, and a device for melt-spinning and casting of thin strips from Al and Mg alloys.

In parallel, under the funds acquired, the Light Metals Division Skawina was fitted with modern research equipment and control-measuring apparatus, including:

- Instron 5582 100 kN and Instron 600DX 600 kN testing machines with attachments for testing at high and low temperatures (in a chamber from  $-150^{\circ}\text{C}$  to  $350^{\circ}\text{C}$  and in a furnace up to  $1200^{\circ}\text{C}$ ) to enable cyclic (low cycle) and variable loading,
- Duramin-2500E versatile automatic hardness tester operating in a loading range of 1-250kG (2452N), enabling measurements by Vickers, Brinell, Rockwell, Superficial Rockwell and Knoop methods,
- portable X-ray diffractometer for measurement of internal stresses and retained austenite content (made by PROTO Canada) to enable non-destructive measurements,
- optical emission spectrometer for analysis of the chemical composition of aluminium and aluminium alloys, and magnesium and magnesium alloys. In addition, Spark-DAT option allows analysis of inclusions present in aluminium and magnesium alloys,
- equipment for the metallographic sample preparation, including LectroPol-5 electropolishing apparatus for electrolytic polishing and etching of metallographic specimens and TenuPol-5 apparatus for electrolytic thinning of specimens, both made by Struers,
- TABER<sup>®</sup> type device for testing the abrasion resistance and wear index of coatings,
- thermovision camera and optical pyrometer,
- digital recording camera operating at speeds of up to 600 000 frames/sec,

- apparatus for measurement of electrical conductivity and ultrasonic flaw detector,
- compact set of gravity poured dies to study the technological properties of liquid aluminium alloys (METAL HEALTH SYSTEM made by N – TEC); it enables testing metal tendency to the formation of shrinkage cavities (Tatur Test), testing the hot crack formation tendency, making standard tensile samples, fluidity test, and examination of coarse oxide inclusions in a ”K Type” cast sample,
- STEM 200 kV high-resolution transmission electron microscope,
- apparatus for hydrogen content measurement in the solid state.

## 2. Examples of investigations

Jointly with other test apparatus, the above mentioned devices enable solving complex research problems in the field of advanced technology of the plastic forming and materials engineering of Al and Mg alloys. In the area of plastic forming, this refers, e.g. to the manufacture of products from non-conventional Al and Mg alloys by the extrusion and die forging of RS powders and ribbons. The aim of these technologies is to provide products of ultrafine grain and nanometric structure and above standard mechanical properties [1,2]. For example, these are the studies carried out on AlCuMgFeNi and AlZnMgCu alloys manufactured from consolidated powders and ribbons [3,4].

The materials were prepared by hot plastic consolidation, which means that the stock produced by rapid solidification (e.g. atomisation, spinning disc casting) was undergoing further integration in a hot extrusion process [5,6,7,8]. In this process, the temperature, pressure and deformation cause the consolidation of powders and ribbons, producing materials of the density close to theoretical values [9,10]. Hot plastic consolidation is used to produce materials, which manufactured by the methods of traditional metallurgy, either do not meet the imposed requirements or cannot be produced by other alternative techniques.

For studies, fractions of 50-150  $\mu\text{m}$  granulation and chemical composition as shown in Table 1 were selected.

TABLE 1  
Chemical composition AlCuMgFeNi and AlZnMgCu alloys

Alloy	Fe	Si	Cu	Zn	Ti	Mn	Mg	Ni	Cr	Zr
AlCuMgFeNi	1,53	0,24	2,39	0,02	0,01	0,005	1,53	1,63	–	0,57
AlZnMgCu	0,07	0,03	1,53	5,47	–	–	2,61	–	0,23	0,42

The structure finally obtained in the investigated alloys produced by hot consolidation of the powdered stock is that of ultrafine grains. The grain size (Figures 1 and 2) is by two orders of magnitude smaller than in the alloys manufactured by traditional methods (casting of ingots, extrusion), and therefore in research of this type it is very important to evaluate the mechanical properties of rods in as-extruded state and after heat treatment. The

mechanical properties obtained in static tensile test are given in Table 2.

At the next stage of investigations, the AlZnMgCu alloy stock after hot plastic consolidation was subjected to cold hydrostatic extrusion. The example of microstructure produced in the examined materials by hydrostatic extrusion is shown in Figure 3, while respective mechanical properties in as-extruded state and after heat treatment are given in Table 3.

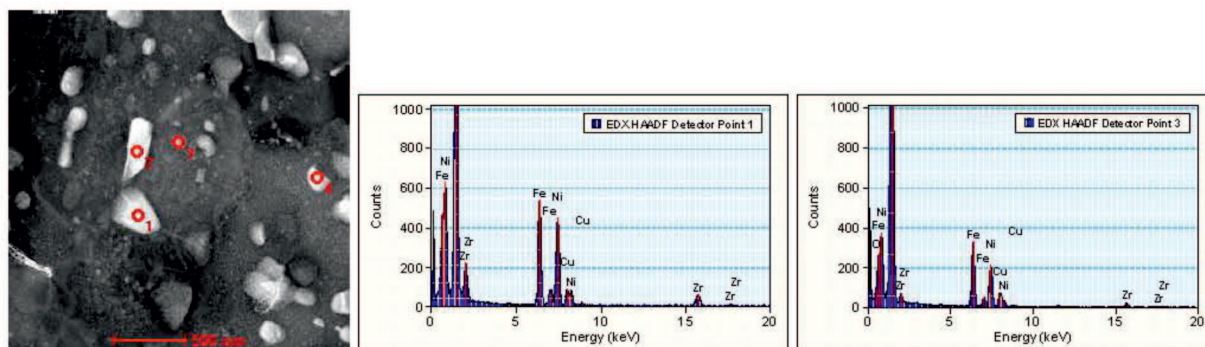


Fig. 1. Example of TEM microstructure in rods extruded from AlCuMgFeNi alloy powder (grain size 1-4 μm) and the results of local chemical analysis

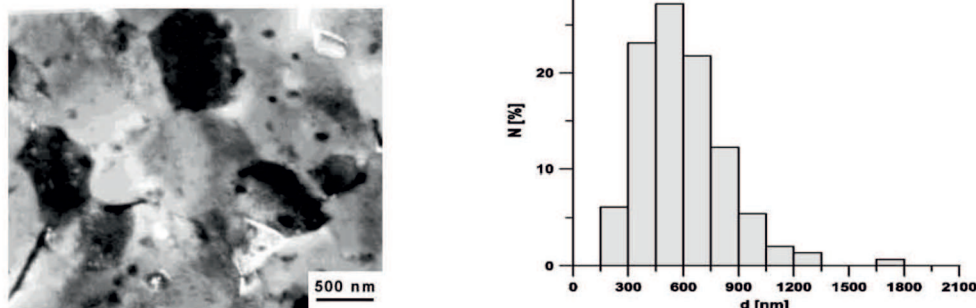


Fig. 2. Example of TEM microstructure in rods extruded from AlZnMgCu alloy powder and a histogram of grain size distribution (average grain size ~ 600nm)

TABLE 2  
 Mechanical properties from the tensile test and Brinell Hardness of samples in as-extruded state and after heat treatment [11]

Alloy	Temper	HB	Mechanical properties		
			R <sub>p0,2</sub> [MPa]	R <sub>m</sub> [MPa]	A <sub>5</sub> [%]
AlCuMgFeNi	T6	123	319	447	9,4
	T6*	125	372	471	8,3
AlZnMgCu	F	110	332	399	12,6
	T6	173	571	603	11,5
	T73	172	581	601	10,5

\* solution heat treatment in salt medium

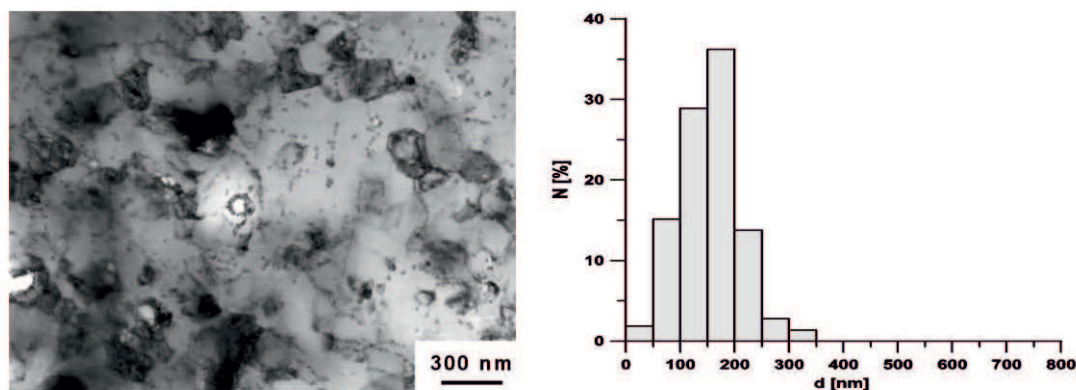


Fig. 3. Example of TEM microstructure in rods (cross-section) hydrostatically extruded from AlZnMgCu alloy (average grain size – 160nm) and a histogram of grain size distribution

TABLE 3  
Mechanical properties obtained in the tensile test and Brinell Hardness of samples hydrostatically extruded from the AlZnMgCu alloy stock after heat treatment [11]

Alloy	Temper	HB	Mechanical properties		
			$R_{p0.2}$ [MPa]	$R_m$ [MPa]	$A_5$ [%]
AlZnMgCu	F	130	381	460	7
	T6	178	621	670	5
	T9	–	686	710	2,4

The results of mechanical tests carried out on an AlZnMgCu alloy manufactured by hot plastic consolidation of the powdered stock indicate great possibilities for producing ultrafine-grain and nanometric structures.

An important area of the research covers all problems related with the technology of quasi-isothermal direct and indirect extrusion of Al and Mg alloys. The concept of quasi-isothermal extrusion involves the basic parameters of the direct and indirect extrusion process of individual products, i.e. billet temperature, temperature gradient along the billet length, ram speed range in function of the extrusion ratio  $\lambda$ , and extrusion speed in function of temperature stability along the length of the extruded product [12,13]. The fixed parameters of the quasi-isothermal extrusion process should result in achievement of temperature comprised along the entire product length within the range of values adequate for the subsequent “water wave” solution heat treatment of product and obtaining after the aging process to conditions T1 and T5 the mechanical properties in conformity with relevant standards [14].

Maintaining on press equipment the temperature constant and comprised in a range of the solution heat treatment values, depending on alloy type, on the critical cooling rate until the required degree of solutioning is

reached, and on the size and weight of products, can be achieved by several methods, including:

- changing ram speed and adjusting it to the extrusion force,
- **zone heating of billet (ram speed constant),**
- **ram speed control depending on temperature of the extruded product measured with a 3T optical pyrometer.**

In the present study, the method of zone heating of billets was applied and, in the second place, the ram speed control depending on product temperature.

In the first variant, the main objective of the study was to determine the parameters of the gradient heating of billets on indirect press of maximum 28 MN force and of the quasi-isothermal extrusion of rods and sections, cooled in a “water wave” device [12].

The basic press unit responsible for quasi-isothermal extrusion was modern induction heater with heating zones, enabling taper heating of billets up to a length of 1270 mm with maximum gradient of 60°C. With temperature distribution repeatable along the entire length of billet it was possible to precisely determine the extrusion parameters, such as the ram speed, extrusion speed, and temperature of the starting and end part of the extruded

product. Determination of these parameters required a number of extrusion tests to be conducted.

Investigations were carried out on  $\phi$  245x1200 mm billets made from the EN AW-6082 and EN AW-7075 alloys [13]. Temperature distribution was examined with *INFRAMETRICS 760B* thermovision system provided with special computer-operated programme.

The results of static thermovision investigations were presented in the form of computer-plotted thermograms. Figure 4 shows examples of thermograms obtained for billets heated to the following temperatures:

A – start 400°C, end 460°C, B – start 500°C, end 530°C. Figure 5 shows thermograms plotted for the starting part, middle part and end part of the rod extruded from billet heated according to the pattern shown in Figure 4B. The temperature changes along the length of the extruded rod plotted in function of time (and time only as the extrusion speed was known and kept constant) are also shown in Figure 5. On thermograms, the temperature values relating to the places selected on the surfaces of the examined rods and billets were plotted.

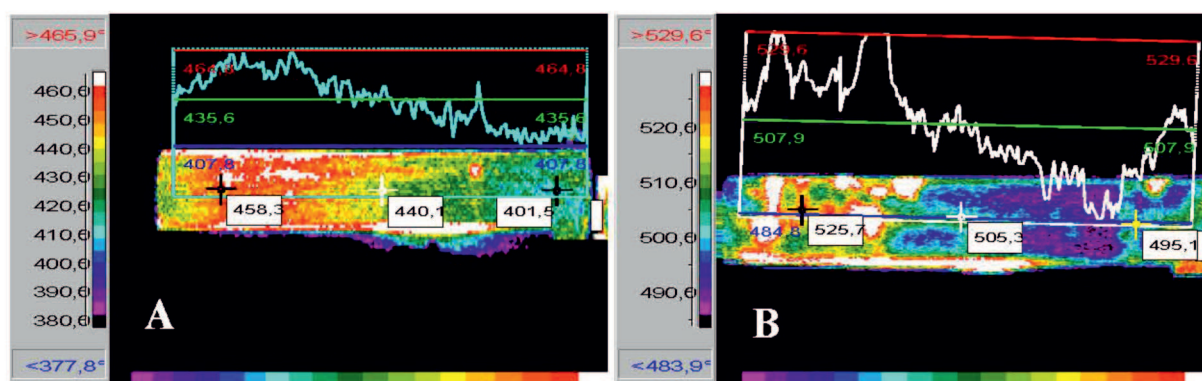


Fig. 4. Temperature distribution on billet surface – A: beginning of billet 400°C, end of billet 460°C, B: beginning of billet 500°C, end of billet 530°C

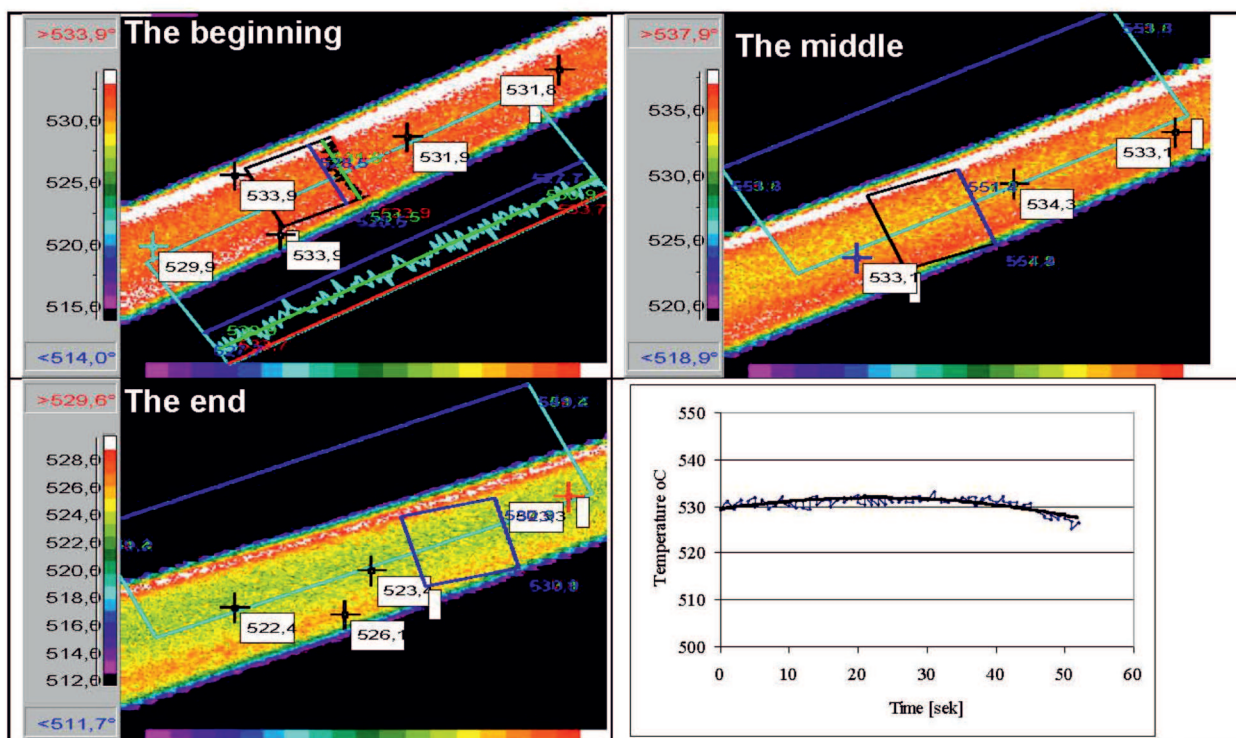


Fig. 5. Temperature distribution on surface of the extruded bar, and changes of temperature distribution during the extrusion of bar

The thermovision measurements of temperature gradient in a billet subjected to zone-heating in induction heater (start – 400°C, end – 460°C, and start – 500°C, end – 530°C) revealed the distribution of temperature zones (fields) along the billet length. In the case of aluminium alloys, measurements of this type are not easy because aluminium is characterised by very low degree of emissivity, which is a serious obstacle in obtaining clean and clear images. The places on the billet, which are either dirty or oxidised more strongly have different values of the emissivity, and hence result the white spots that appear in the thermovision image (extending far beyond the adopted scale of temperature). The next stage of the investigations was temperature determination along the entire length of the extruded rod before it entered the "water wave." For example, along the entire length of a rod made from the EN AW-6082 alloy, this temperature was comprised in a range of 532°C – 527°C, thus indicating the quasi-isothermal character of an indirect extrusion process and properly selected billet temperature gradient and extrusion speed.

The indirect extrusion tests carried out on EN AW-6082 and EN AW-7075 alloys have proved the possibility of carrying out the solution heat treatment of products made from these alloys in a "water wave" type device, yielding very high mechanical properties and satisfactory structure.

The EN AW-7075 alloy was used for extrusion of  $\phi 60$ mm rods ( $\lambda = 17.6$ ), while from EN AW-6082 alloy, rods of  $\phi 70$ mm were extruded ( $\lambda = 12.5$ ). The process parameters are given in Table 4, while Table 5 compares the mechanical properties of extruded rods subjected to either "on-line" solution heat treatment (to T5 condition) on press equipment, or to traditional in-furnace treatment (to T6 condition), followed by artificial aging.

The measurements of temperature taken along the entire length of the extruded rod confirmed the possibility of creating the conditions adequate for quasi-isothermal extrusion on an indirect press. By application of the conical billet heating technique, the possibility of "water wave" solution heat treatment of rods made from the EN AW-6082 and EN AW-7075 alloys was confirmed along with the possibility of obtaining high mechanical properties after aging.

Parameters of billet preheating for the indirect extrusion process

TABLE 4

Alloy	Temperature parameters					
	EN AW-7075	Container temp. – 380°C			initial heating – 350°C	
Billet temperature						
section I		section II	section III	section IV	section V	section VI
400°C		405°C	410°C	415°C	420°C	420°C
EN AW-6082	Container temp.– 450°C			initial heating – 490°C		
	Billet temperature					
	section I	section II	section III	section IV	section V	section VI
	500°C	500°C	510°C	510°C	520°C	530°C

Mechanical properties of rods extruded from 7075 and 6082 alloys

TABLE 5

Alloy / rod	Temper	HB	Mechanical properties		
			R <sub>p0.2</sub> [MPa]	R <sub>m</sub> [MPa]	A <sub>5</sub> [%]
7075 $\phi 60$ mm	T5	158	575	651	11,8
	T6	165	578	653	9,3
6082 $\phi 70$ mm	T5	119	372	401	17,5
	T6	124	332	355	13,3

Using the method of ram speed control in function of the temperature of the extruded product, measured with a 3T optical pyrometer, the technological tests were conducted at OFAJ in Nagpur, India, to verify in practical application the concept of quasi-isothermal extrusion of selected Al alloy items on a 65MN direct-indirect press made by ZAMET – BUDOWA MASZYN SPÓŁKA AKCYJNA in Tarnowskie Góry.

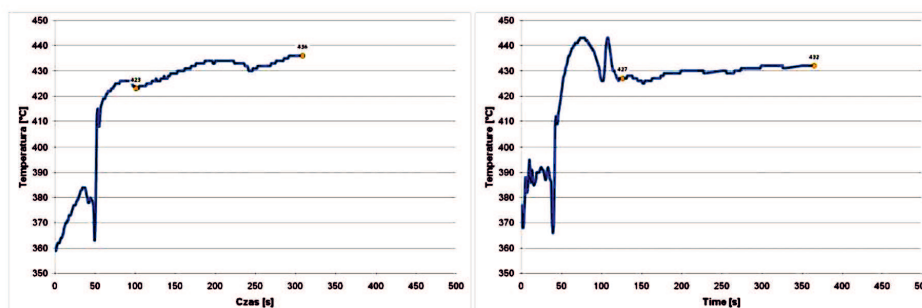
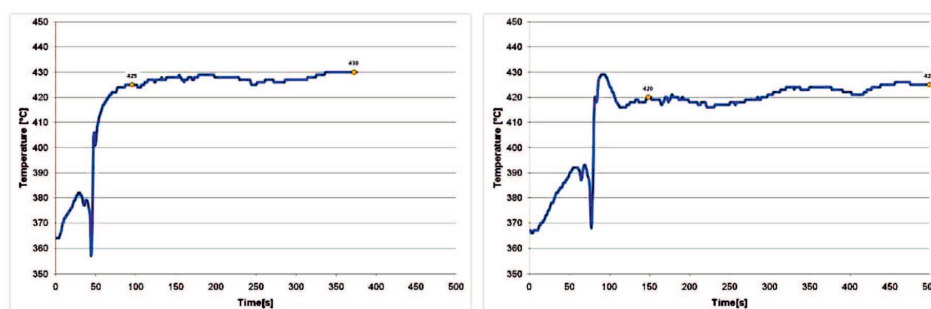
Trials of quasi-isothermal extrusion were carried out on AA7075 alloy, extruding rods of 160mm diameter. Table 6 contains detailed parameters of the extrusion process, while Figures 6 and 7 show temperature distributions along the length of the extruded rod. As recommended, the difference in temperature between the rod starting part and end part not larger than 5°C was sought.

The research programme included the extrusion of the evenly heated billets (certain differences in temperature between the billet starting part and end part were due to the heater operating regime and to different time lapse during which the billet remained in the heater). For control of the extrusion process, a computer-operated programme, developed specially for this purpose and installed in the press, was successfully used, enabling the determination of "K" factor. This is the factor that shows the ram speed increase or decrease, which means change in the extrusion speed and hence change in the amount of heat generated during the extrusion process, the latter one, in turn, affecting the temperature of the extruded product. The "K" factor greater than unity means the increasing ram speed; the value smaller than unity means the speed reduced in properly selected zones of the billet (stages of extrusion process).

TABLE 6

Metallic radii of rare earth metals and magnesium [12]

Billet No.	Billet temp. [°C]	Container temp. [°C]	Ram speed [mm/s]	Extrusion speed [m/min]	„K” Factor
1	406-426	420	2	0,8	K=1
2	407-439	420	1,5	0,8	K=0,9
3	446-447	420	2	0,6	K=0,85
4	392-424	420	2	0,8	K=0,8

Fig. 6. Temperature distribution for  $\phi 160$  mm rod, billet No. 1 (left) and 2 (right)Fig. 7. Temperature distribution for  $\phi 160$  mm rod, billet No. 3 (left) and 4 (right)

First, the temperature must get stabilised – the temperature of the starting part of the rod is always lower. Sometimes the operator has to put the rod in line with the press axis, including places where measurements are taken with the pyrometer, as it often happens that the rod somewhat bent “escapes” the measurement area, resulting in frequent temperature fluctuations at the beginning of the process.

In constant-speed extrusion (K factor = 1), differences of up to 13°C (Fig. 6, billet No. 1) occurred; in quasi-isothermal extrusion, for K = 0.9 and reduced ram speed, and for K = 0.85 and 0.8 (ram speed identical as in the case of extrusion with K = 1 but reduced in respective zones of the billet), the required difference in temperature of max. 5°C (the difference between the starting and end part of the extruded rod) was obtained. Using special programme for the quasi-isothermal extrusion process control, allowing for changes in the ram speed during extrusion, it was possible to stabilise the temperature along the entire length of the extruded rod.

In the area of materials engineering, studies of both utilitarian and cognitive character are carried out to better know the properties of materials based on Al and Mg alloys with ultrafine grain structure, obtained by deformation during the ECAE extrusion process. An example of such studies is the research conducted on AlCu4SiMg alloy [15-18]. The effect of total strain on structure and properties was examined by light microscopy, transmission electron microscopy and hardness measurements (Table 7). The structure during deformation was chang-

ing from the band type with high density of dislocations to nearly-equiaxed nanograins with high-angle boundaries (Fig. 8). The results indicate that nearly-equiaxed grains were formed by complex mechanism of mutually crossing microbands and the processes of static and dynamic structure recovery. The interoperational annealing resulted in softening of the structure and created favourable conditions for the formation of equiaxed nanograins. The average grain size after seven ECAE passes was 183nm, and more than 20% of grains had a size below 100 nm. Moreover, areas of equiaxed grains were present, too. Hardness became stabilised after the second pass, and its value was nearly two times higher than in the starting material.

Similar studies were conducted on the MgAl6Zn0,6 magnesium alloy within the range of true strain values  $\varepsilon=1.15 - 4.6$  at an elevated temperature of 300°C [19,20].

The effect of true strain on structure was investigated by light microscopy, transmission electron microscopy and EBSD orientation mapping test (Fig. 9).

It was found that deformation by ECAE has led to fragmentation of the  $\gamma$  phase and dissolution or coagulation of the eutectic precipitates. The presence of high-density dislocations and forest dislocations was observed. Inside the deformation bands, dislocation cells of nanometric size were formed, and their number was increasing proportionally to the strain rate.

A significant increase of hardness was stated in the material deformed by ECAE with true strain  $\varepsilon = 4.6$ .

AlCu4SiMg alloy properties after deformation by ECAE and heat treatment

TABLE 7

Stage of treatment $\varepsilon$ / annealing	Subgrain size [nm]	HB	$R_{p0.2}$ [MPa]	$\Delta\sigma_{\Delta\varepsilon}$ , [MPa]
0	~ 20000	57.4	179	592
1.15	–	79.2	–	–
2.3	204	87.3	284	383
300°C/10min	320	64.2	200	378
3.45	–	81.7	–	–
4.6	203	84.4	316	231
250°C/10min	308	69.1	251	275
5.75	–	81.5	–	–
6.9	163	82.2	303	189
200°C/10min	228	79.2	307	187
8.05	183	81.4	–	–



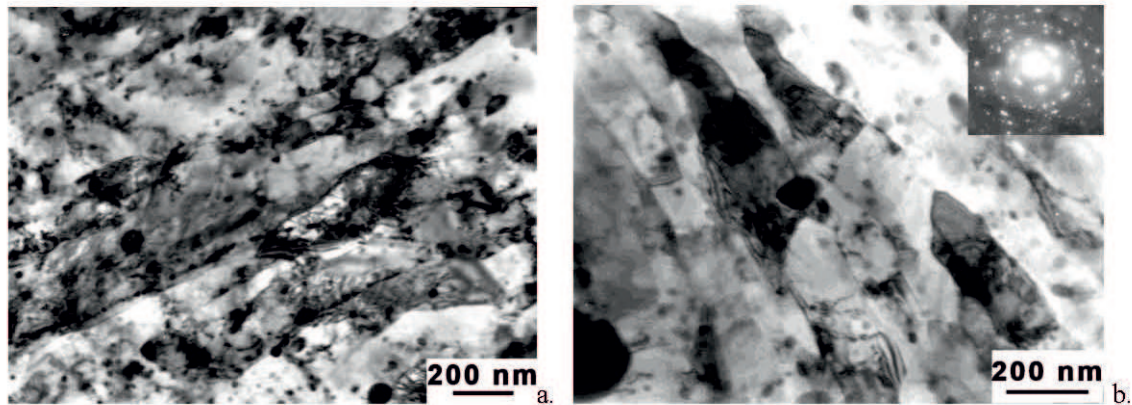


Fig. 8. Sample structure after deformation with true strain  $\varepsilon = 2.3$  (a) and  $\varepsilon = 8.05$  (b)

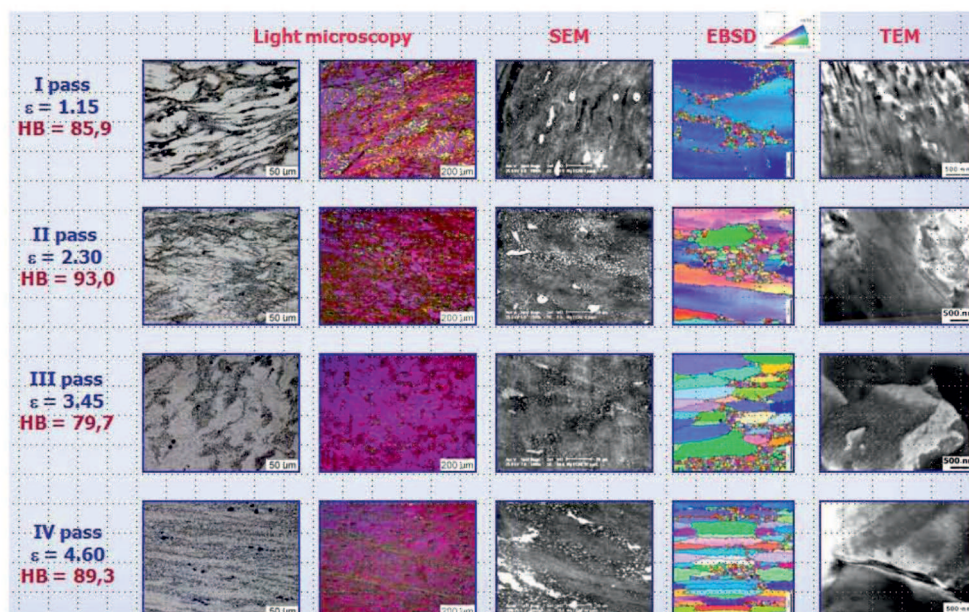


Fig. 9. Collective results of structure examinations and Brinell hardness measurements

Another example of studies is characterisation of structure and properties of new materials based on light alloys. The research was carried out on an AlSi alloy with additions of Sc [21-23]. The possibility to use a cast alloy from the 4xxx series, i.e. a hypoeutectic silumin, to produce cold rolled sheets for deep drawing was reported. The effect of alloying elements like Mg and Sc on the mechanical and deep drawing properties was investigated, along with the corrosion resistance of AlSi7 alloy sheet metal after various types of heat treatment in the temperature range of 160-250°C.

The mechanical properties of test sheets were increasing after cold rolling, starting with the alloy without any alloying elements, passing next to alloy with scandium and to alloy with magnesium, and reaching the highest level in alloy with Mg and Sc, i.e.  $R_m = 220-250$  MPa and  $R_{p0.2} = 194-218$  MPa. The coefficient of anisotropy for S3 and S4 sheet metal was approx-

imately 6%. The sheets with Mg suffered cracks in a cupping test (Fig. 10).

It was found that the properties of AlSi7 alloy sheet metal with additives of Sc and Mg are similar to the properties of sheet metal made from the 5xxx series alloys, and so the former one can be used by the automotive industry for deep drawn parts.

Numerous studies conducted at IMN-OML Skawina include advanced structure examinations using electron microscopy, combined with chemical analysis in microregions and EBSD orientation mapping analysis. The scanning microscopy combined with EBSD has created possibilities for direct phase analysis. The chemical analysis in microregions cannot clearly identify the intermetallic phases – the qualitative chemical composition can be the same, while the crystallographic structure will be different – e.g.  $Mg_2Si$  phases or iron phases.

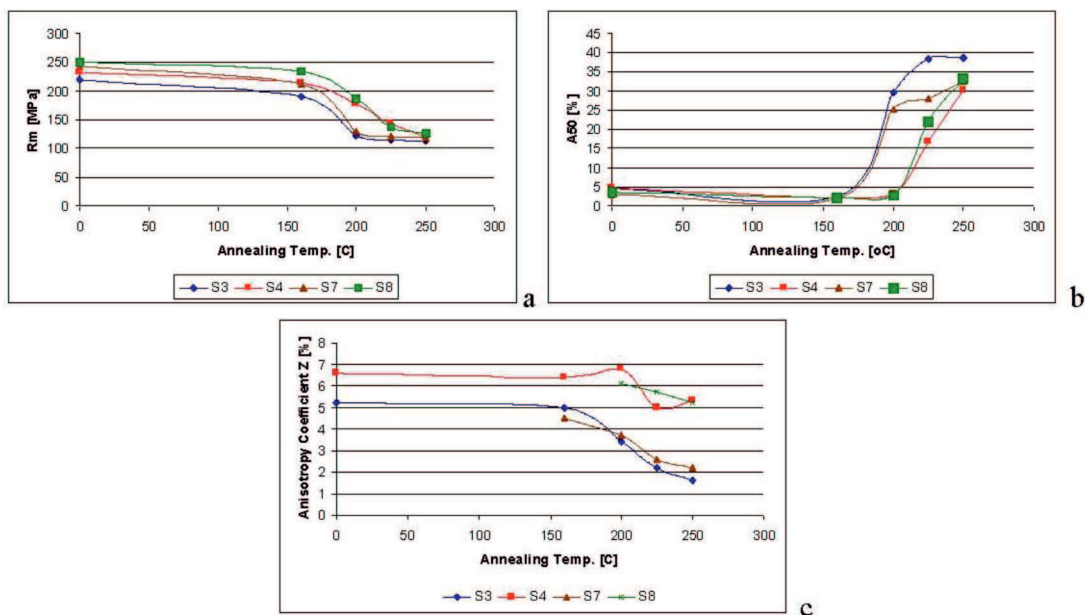


Fig. 10. The mechanical properties of tested alloys: tensile strength  $R_m$  [MPa] (a), elongation  $A_{50}$  [%] (b), the coefficient of anisotropy Z [%] earing test results (c)

TABLE 8

The morphology of precipitates in 2014 alloy

	Cooling rate 20 K/sec	Cooling rate 0,5 K/sec
	<b>1.1</b>	<b>1.4</b>
<b>Mg-0,2% Si-0,5% Mg/Si=0,4</b>		
	<b>2.1</b>	<b>2.4</b>
<b>Mg-0,8% Si-0,5% Mg/Si=1,5</b>		
	<b>3.1</b>	<b>3.4</b>
<b>Mg-0,2% Si-1% Mg/Si=0,2</b>		
	<b>4.1</b>	<b>4.4</b>
<b>Mg-0,8% Si-1% Mg/Si=0,8</b>		

In industrial aluminium alloys of complex chemical composition, even in those included in one and the same family, totally different phases, depending on the content of individual alloying elements, can occur. In alloys from the 2xxx and 6xxx series, the content of these phases and their chemical composition as well as morphology depend on both the Mg/Si ratio and the cooling rate [24,25].

The results of investigations carried out on 2014 alloy are compiled in Table 8.

Consequently, for 2014 alloy the following has been stated:

- Regardless of chemical composition, iron phases had the form of "Chinese script" and were enriched in copper at a level of about 8-10 wt.% and in magnesium at a level of about 0.5 wt.%. Depending on cooling rate, the Fe + Mn/Si ratio assumed different values and was approximately 1.5 - 3 for the cooling rate of 20 K/sec and approximately 4.5 - 5 for the cooling rate of 0.5 K/sec. The Fe + Mn/Si ratio also depended on Si content in the examined material. Regardless of chemical composition, in terms of crystallography, the iron phase was described as an  $Al_{93,38}Cu_{6,02}Fe_{24}Si_{16,27}$  cubic phase (Th) [m3] with lattice parameter  $a = 12.643 \text{ \AA}$ .
- The AlCu copper phases often contained an addition of Mg (about 0.2 wt.%) and Si (about 0.2-0.3 wt.%) These phases were identified as tetragonal phases (D4h) [4mmm] with lattice parameters  $a = 6.06 \text{ \AA}$ ,  $c = 4.87 \text{ \AA}$ . In samples where the Mg/Si ratio was 0.2 and 0.4, the AlCu phases assumed the form of blocks with Cu content varying in their composition. In samples with the Mg/Si ratio equal to 0.8 and 1.5, copper phases occurred mostly in the form of complex eutectic phases containing  $Mg_2Si$  (Oh cubic phase) [m3m] with lattice parameter  $a = 6.39 \text{ \AA}$ .

### 3. Summary

The above examples of studies conducted in the area of the, so called, advanced technologies of the fabrication of Al- and Mg-based alloys using equipment and apparatus operating under the investment project "Advanced Technology Incubator for Plastic Working of Light Alloys based on Al and Mg" create positive conditions for a qualitative breakthrough in research of the new Al and Mg alloy technologies. The potential that the Light Metals Division Skawina currently has at its disposal should be reasonably used as a proposal for cooperation with scientific entities of the Polish Academy of

Sciences, Universities and Institutes. Some possibilities have also been created to extend further this cooperation under the EU Framework Programmes.

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### REFERENCES

- [1] J. Król, J. Senderski, M. Dumańska, M. Lech-Grega, T. Stuczyński, Z. Zamkotowicz, Wpływ dodatku cyrkonu na strukturę wysokowytrzymałych stopów aluminium, Aluminium 2000, Krynica 8-10 marzec (2000).
- [2] J. Dutkiewicz, J. Senderski, Z. Zamkotowicz, A. Kłyszewski, W. Maziarz, Zagadnienie rozdrobnienia ziarna w stopie AlCuAgZr do odkształcania nadplastycznego, Aluminium 2000, Krynica 8-10 marzec (2000).
- [3] B. Płonka, J. Senderski, M. Lech-Grega, S. Boczkal, S. Wierzbiński, Structure and mechanical properties of AlCuAg alloy after hot plastic consolidation with powders and ribbons, 5<sup>th</sup> International Conference of PhD Students, University of Miskolc, Hungary, 14-20 VIII (2005) 173-178.
- [4] B. Płonka, B. Augustyn, P. Kurtyka, W. Szymański, S. Wierzbiński, Konsolidacja plastyczna na gorąco w procesie wyciskania stopów Al wytwarzanych metodami szybkiej krystalizacji, Materiały konferencyjne XXXII Szkoła Inżynierii Materiałowej Kraków-Krynica, 441-446 (2004).
- [5] J. Senderski, T. Stuczyński, B. Płonka, Badania nad procesem wyciskania prętów ze stopów AlSi26Ni8 i AlSi26Ni6Fe2, wytwarzanych metodą metalurgii proszków, Materiały konferencyjne XXX Szkoła Inżynierii Materiałowej Kraków-Ustroń, 547-552, (2002).
- [6] B. Płonka, Wpływ warunków wytwarzania na właściwości użytkowe materiałów proszkowych konsolidowanych w procesie wyciskania WMN, AGH Kraków, (2006) (praca doktorska).
- [7] T. Stuczyński, J. Senderski, B. Płonka, Manufacturing of AlSiNi(Fe) powders and consolidation of semi-products. International Conference – Aluminium in Transport 2003, Tomaszowice, 11-25 (2003).
- [8] J. Senderski, S. Boczkal, H. Dybiec, S. Kivivuo, G. Krallics, M. Lech-Grega, B. Płonka, T. Stuczyński, Plastic Forming of Aluminium Alloys Powders; Foundation of Materials Design; Research Signpost 321-354 (2006).

- [9] ASM Specialty Handbook. Aluminum and Aluminum Alloys. ASM International, 649-690 (1993).
- [10] K. Laue, H. Stenger, Extrusion. American Society for Metals, USA, (1981).
- [11] European Standard EN 515: Aluminium and aluminium alloys – Wrought products – Temper designations, (1993).
- [12] J. Senderski, L. Pierewicz, B. Płonka, Technologia, urządzenia i sterowanie procesem przesyłania wyrobów wyciskanych na wybiegu prasy ze stopów Al serii 6XXX i 2XXX, Rudy i Metale Nieżelazne, R47, nr 6, 284-288 (2002).
- [13] T. Stuczynski, Projekt celowy 6 ZR7 2005 C/06611 Opracowanie i wdrożenie technologii wytwarzania wlewków ze stopów serii 2xxx, 5xxx, 6xxx i 7xxx stanowiących wsad dla procesu wyciskania na nowouruchomionej przeciwbieżnej prasie 2800 T w Grupie Kęty S.A., (materiały niepublikowane).
- [14] C. Kramer, Intensive cooling of light metal alloy extrusion by air and water. Aluminum Extrusion Technology Seminar, 397-407 (2000).
- [15] S. Boczkal, M. Lech-Grega, J. Senderski, B. Płonka, Microstructure and mechanical properties of AlCu4SiMg Alloy severely deformed by ECAE method; Inżynieria Materiałowa **5**, 489-494 (2008).
- [16] S. Boczkal, M. Lech-Grega, J. Senderski, B. Płonka, Structure evolution under severe plastic deformation of 2014 aluminium alloy. XII International Conference on Electron Microscopy of Solids Kazimierz Dolny 5-9 June, (2005).
- [17] S. Boczkal, M. Lech-Grega, W. Szymański, Characterization of the grains in 2014 aluminium alloy after Equal Channel Angular Extrusion (ECAE) process, Image Analysis & Stereology, (2006).
- [18] M. Lech-Grega, S. Boczkal, J. Senderski, B. Płonka, Processing and characterization AlCu aluminium alloys obtained by ECAE; Solid State Phenomena **114**, 165-170 (2006).
- [19] M. Lech-Grega, S. Boczkal, B. Płonka, Ewolucja struktury stopu MgAl6Zn0,6 odkształcanego metodą ECAE w podwyższonych temperaturach; Aluminium (2007), Kliczków.
- [20] M. Lech-Grega, S. Boczkal, B. Płonka, Structure evolution of MgAl6Zn0,6 after ECAE pressing at evaluated temperature. Euromat, Norymberga Niemcy, (2007).
- [21] M. Lech-Grega, A. Kłyszewski, M. Opyrchal, W. Szymański, J. Żelechowski, Charakteryzacja blach ze stopów Al-Si7 z dodatkiem Sc i Mg przeznaczonych do głębokiego tłoczenia; Polska Metalurgia w latach 2006-2010; wyd. Naukowe „AKAPIT”, Kraków; ISBN 978-83-60958-64-3, 701-709, (2010).
- [22] M. Lech-Grega, A. Kłyszewski, M. Opyrchal, W. Szymański, J. Żelechowski, Characterization of the AlSi7 with Sc and Mg Sheets Use to Deep Drawing; Proc. of the 11<sup>th</sup> International Conference on Aluminium Alloys, 22-26 Sept. 2008 Aachen Germany; 2402-2408 (2008).
- [23] M. Lech-Grega, A. Kłyszewski, T. Stuczynski, J. Żelechowski, W. Szymański, The 4xxx Aluminium Alloy Sheets for Use in Deep Drawing; TMS 2005 134<sup>th</sup> Annual Meeting and exhibition (The Minerals, Metals & Materials Society), (2005).
- [24] M. Lech-Grega, The influence of Mg/Si Ratio on Phase Compositions in 2014 Aluminium Alloys after Casting and Heat Treatment Proceedings of The 12<sup>th</sup> International Conference on Aluminium Alloys, Yokohama, Japan, (2010).
- [25] M. Lech-Grega, S. Boczkal, Iron Phases in Model Al-Mg-Si-Cu Alloys; Materials Science Forum **674**, 135-140 (2011).