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Determination of Heat Treatment Parameters of Gypsum Molds in the Al/CF Composite Manufacturing Process

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Abstract

This paper presents a novel method for producing aluminium matrix composites reinforced with short carbon fibers. The composite casting process was preceded by studies on the determination of heat treatment parameters of gypsum molds. Taking into account the decomposition temperature of carbon fibers of 396˚C in an oxygen atmosphere, tests were carried out to verify the possibility of casting in a mold annealed at 380˚C. For this purpose, the moisture content of the gypsum mass was annealed at 730˚C and 380˚C was compared. The effect of a lower annealing temperature on the roughness of the aluminium matrix was also investigated, and a composite casting was made by saturating the CF preform with an aluminium alloy. The obtained castings were subjected to metallographic analysis. As a result of the conducted research, it was found that it is possible to obtain an Al/CF composite without the use of additional protective coatings and high saturation pressures, which increase production costs.

Keywords: Carbon fibre, Al/CF composite, Manufacturing of Al/CF composite

1. Introduction

Low density, high thermal conductivity, stiffness, dimensional stability, and strength are the characteristics of Al/CF composites, thanks to which they have found wide application in the automotive and aviation industries [1]. Moreover, carbon fiber improves the tribological properties of composites [2]. Potential applications of aluminium matrix composites also include the electronics and energy industries [2,3,4]. For this type of application, due to the requirements for heat dissipation in electronic devices, high thermal conductivity and a low coefficient of thermal expansion (CTE), which carbon fiber is characterized by, are essential. Research is ongoing on the application of Al/CF composites as energy detectors in the Large Hadron Collider at CERN [4].

In mechanical engineering, one of the fundamental and most important properties of materials is mechanical strength. The strength of Al/CF composites depends on the composite manufacturing method. Most often, due to the poor wettability of the Al-CF system, metallic coatings are used. For an Al/CF composite made by the squeeze melt infiltration technique with a Cu coating, the tensile strength was 140 MPa, while for Al/CF with a Ni coating, it was 80 MPa [5]. In [3] the author obtained a tensile strength of 135±4 MPa for an Al/CF composite with a Ni coating during a pressureless infiltration process. For comparison, the strength of pure aluminium, not subjected to heat treatment, is approximately 60-70 MPa [6]. From a practical point of view, the use of both coatings and high pressure values in the squeeze casting method (even in the range of 70-100 MPa) [7,8] is costly, hence an attempt was made to produce an Al/CF composite without the use of protective coatings and high pressures.

For the production of aluminium matrix composite castings, a precision casting method, the lost-wax method, is often used [9,10]. In the case of composites with infiltrated porous reinforcement, the reinforcement is impregnated with wax or paraffin to prevent the gypsum mass from entering the pores of the reinforcement. A crucial step in this method is the annealing of the gypsum mold, during which the wax (or paraffin) melts, opening the pores of the reinforcement, and the mold gains strength and organic compounds are removed from it. Manufacturers recommend that the annealing process be carried out incrementally to a temperature of at least 700˚C. In the available literature and in information provided by manufacturers, it is not specified why such an annealing temperature is recommended. In works [9,10] the authors annealed molds at a temperature of 1150˚C. There are also known results of strength tests of gypsum molds annealed at a temperature of 700˚C [11,12,13]. Unfortunately, carbon fibers in an oxygen atmosphere degrade at temperatures above 396˚C, which creates problems during the production of Al/CF composites [14]. Therefore, the thermal decomposition of casting gypsum, whose main component is calcium sulfate dihydrate, also known as anhydrite ($CaSO_4 \cdot 2H_2O$), was analyzed.

Anhydrite occurs in three varieties, as (Fig. 1):

– soluble α-anhydrite (α-$CaSO_4$III) formed in a vacuum during the dewatering process of the α-hemihydrate at 100 °C or in the range 110-129 °C at normal atmospheric pressure;
– soluble β-anhydrite (β-$CaSO_4$III) obtained by dehydrating the β-hemihydrate in a vacuum at 100 °C or by treating the dihydrate at 200 °C in low humidity air;
– insoluble anhydrite ($CaSO_4$II) occurs naturally in sedimentary rocks [13].

The anhydrous product $CaSO_4$II can be most easily obtained by heating α-anhydrite III at a temperature of 220°C (a) or by calcining β-anhydrite III at a temperature of 350°C (b) (Fig. 1). There is also a third method: dehydration of dihydrate anhydrite in an evaporator at 42°C.

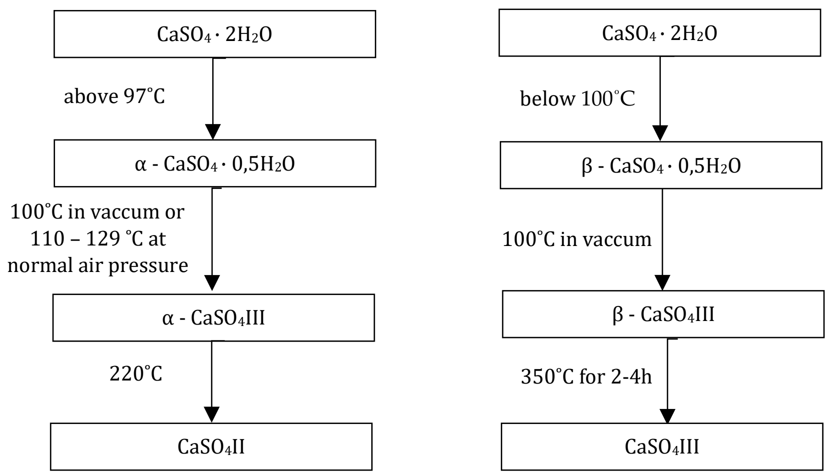


Fig. 1. Diagram of transformations taking place in calcium sulfate: a) in an atmosphere of saturated water vapor or in water, b) in vacuum or in unsaturated air [Based on 15]

According to Paduchowicz et al., water loss from the gypsum mass occurs at a temperature of 300°C [15,16]. In 2003, Luk and Darvell mentioned the possibility of annealing gypsum molds at temperatures below 450°C for the production of dental castings [17]. Based on these premises, the gypsum mass was annealed at temperature of 380°C, which did not cause degradation of the carbon fiber. The aim of the research was to verify whether an annealing temperature lower than that recommended by manufacturers would allow safe filling of the mold with liquid metal. The following sections of the article present the results of tests on the moisture content of casting gypsum annealed at temperatures of 380˚C and 730˚C. Two roughness parameters (Ra, Rz) of aluminium castings made in molds annealed at both temperatures were also compared. Confirmation of the assumed hypotheses and preliminary research was the production of an Al/CF composite casting.

2. Materials and methods

2.1. Moisture content of gypsum mass

"R&R Argentum" jewelry casting gypsum mass (JCGM) was used for the tests. The first step was to determine the moisture content of the gypsum powder. For this purpose, three Petri dishes were prepared, each containing 100±0.01 g of gypsum powder. The samples were dried in a laboratory oven at 100˚C until their masses stabilized.

Mass measurements were taken every hour. A 50±0.01 g gypsum sample was also prepared and dried in a laboratory moisture analyzer at 110˚C. The dried casting gypsum was transferred to a desiccator and, after reaching ambient temperature, it was taken to prepare the gypsum mass. The gypsum mass was prepared according to the following proportions: 100 g of gypsum and 40 g of water. The exact masses of the components used are shown in Table 1.

Table 1.
Masses of components used to prepare gypsum mass [g]

Lp.	R&R Argentum gypsum	Water
1.	100,09	40,07
2.	99,98	39,59
3.	100,32	39,93
4.	100,76	40,4

Both components were mixed and left to set the gypsum mass, then transferred to crucibles and weighed again. The crucibles were transferred to a Nabertherm N150 chamber furnace. The prepared samples are shown in Figure 2. Annealing was carried out at temperatures of 380°C and 730°C, with sample mass measurements taken according to the graph shown below (Fig. 3).

Fig. 2. Ceramic samples prepared for roasting

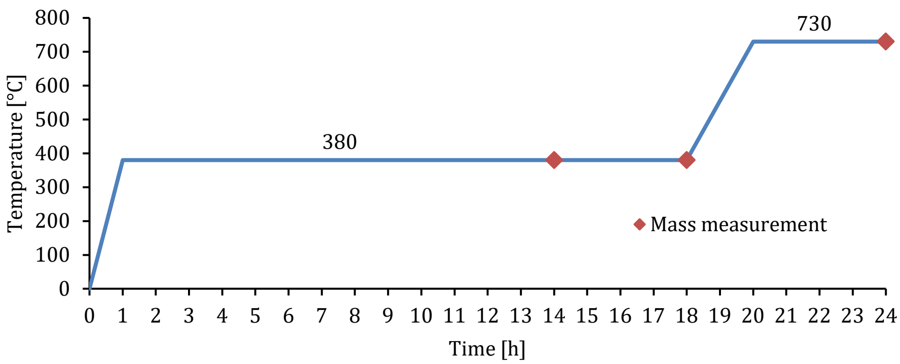


Fig. 3 Annealing cycle with marked points for mass measurement

The moisture content of the samples was calculated using formula 1.

$$W = \frac{a - b_T}{a} \cdot 100\% \quad (1)$$

Where:
a - sample mass before annealing [g]
b_T - sample mass after annealing at temperature T [g]
W – moisture content [%]

2.2. Roughness

The next stage of the research was to produce castings from AlSi11 aluminium alloy in the form of plates with dimensions: 17 mm x 4 mm x 30 mm (width x thickness x height). The alloy used in the tests constituted the matrix of the produced Al/CF composite. The castings were made using the lost-wax method. Wax plates were made in a "mirror" surface mold, from which casting models in the shape of cuboids with a thickness of 4 mm were cut. The models were placed on a gating system as shown in Figure 4.

Four such assemblies were made, placed in a perforated sleeve, and filled with gypsum mass. Two molds were annealed incrementally to a temperature of 380°C, and the remaining two incrementally to a temperature of 730°C. The annealing cycles are shown in Figure 5.

a) b)

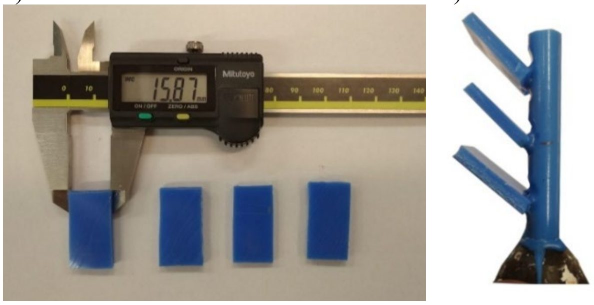
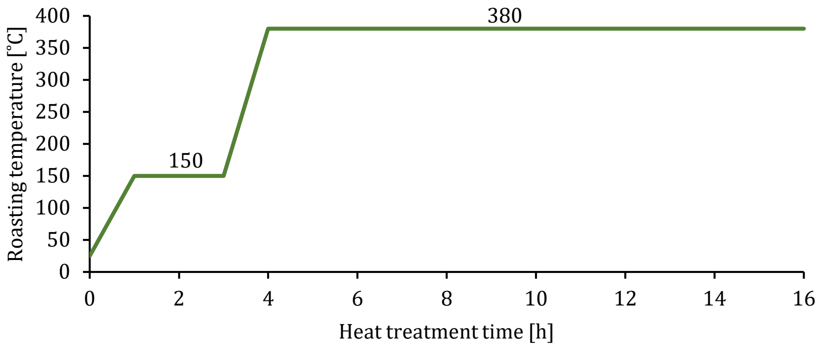


Fig. 4. Wax model: a) single tiles, b) with the filling system

a)



b)

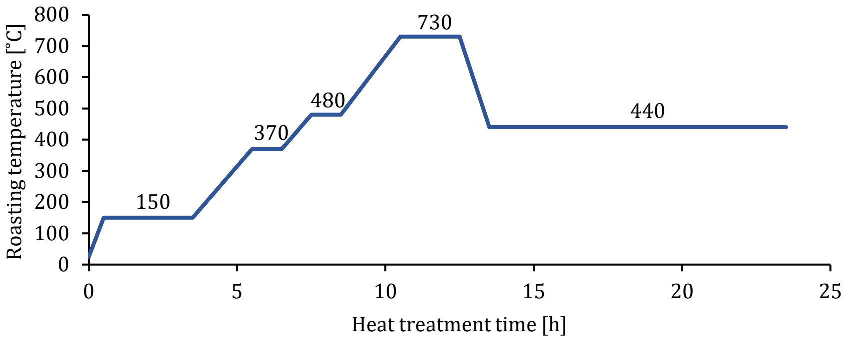


Fig. 5. Gypsum mold annealing curves at temperatures: a) 380˚C, b) 730˚C

The molds were placed in a vacuum chamber. A cut-out from the AlSi11 aluminium alloy ingot was melted in a crucible at a temperature of 700°C and refined. After achieving a pressure of 800 Pa in the chamber, the molds were poured with liquid metal. The temperatures of the poured molds were 243°C, 142°C (for molds annealed at 380°C) and 225°C and 185°C (for molds annealed at 730°C), respectively. The mold was placed in the vacuum chamber with a temperature meter is shown in Figure 6.

Fig. 6. Gypsum mold before the casting process

Fig. 7. AlSi11 cast

After solidification, the casting was removed from the mold, cleaned of gypsum mass residues, the gating system was cut off, and the surface roughness was tested. Figure 7 shows one of the castings after removal from the mold.

2.3. Roughness testing

The surface roughness assessment was performed using a MarSurf PS 10 MAHR roughness tester. The measurements were made using the profilometric method over a length of 4.8 mm. The measurement concerned the Ra and Rz parameters.

2.4. Composite casting

The next stage of the research was to produce AlSi11/CF composite castings in the shape of plates with dimensions of 76 mm x 17 mm x 4 mm. The reinforcement was prepared using needled carbon felts from M-Carbo, made of short compressed carbon fibers with a fiber diameter ranging from 7 µm to 10 µm and a length of 50 µm to 80 µm. Three reinforcement preforms were impregnated with paraffin. The paraffin filled the pores present in the reinforcement structure and prevented the gypsum mass from penetrating between the carbon fibers. The preforms were placed on a gating system as shown in Figure 8. The prepared assembly was placed in a metal tube with holes and filled with casting gypsum mass. The mold was annealed according to the cycle shown in Figure 5a. The reinforcement was infiltrated with the AlSi11 casting alloy. The infiltration conditions, including physico-chemical phenomena, will be described in a subsequent publication. The obtained composite was used to prepare a metallographic specimen.

Fig. 8. CF preform on the gating system

3. Results and discussion

3.1. Moisture content of the ceramic mass

Table 2 shows the results of the moisture content test of gypsum powder dried at 100°C. The average moisture content of the powder was 1.46%.

Table 2.
Measurements and moisture content of "R&R Argentum" gypsum powder

Lp.	Mass of the vessel [g]	Mass of gypsum [g]	Total weigth [g]	Mass [g] measured over time					%W
				1 h	2 h	3 h	4 h	5 h	
1	61.2	99.98	161.18	160.27	159.77	159.66	159.65	159.64	1.508
2	51.48	99.98	151.46	150.5	150.11	149.98	149.96	149.95	1.480
3	53.79	99.98	153.77	152.92	152.51	152.37	152.35	152.34	1.400
Average value									1.463

For comparison, the result obtained using the laboratory moisture analyzer was 1.52%. The drying time was 1h 17min 44s. The reaction of gypsum setting with water is an exoenergetic reaction, during which some of the water evaporates, hence the mass after setting (a) was taken as the total sample mass. The percentage water content was calculated according to formula (1). The results obtained are shown in Table 3.

Table 3.
Weights of samples annealed at temperatures of 380˚C and 730˚C and humidity of the gypsum mass

Lp.	Mass of the crucible [g]	a [g]	380˚C, 13h		380˚C, 17h		380˚C, 17h and 730˚C, 4h	
			b_{380} [g]	W_1 [%]	b_{380} [g]	W_2 [%]	b_{730} [g]	W_3 [%]
1.	311.41	138.78	99.18	0.34	99.18	0.34	99.11	0.29
2.	304.39	138.51	99.36	0.32	99.34	0.30	99.27	0.25
3.	303.49	138.85	99.40	0.35	99.40	0.35	99.32	0.29
4.	298.41	139.91	99.95	0.31	99.95	0.31	99.89	0.27
Medium				0.33		0.33		0.28

The average water content in ceramics annealed for 13 hours at 380°C was 0.33% and 0.32% for ceramics annealed for 4 hours longer. For comparison, after annealing at 730°C, the moisture content of the ceramics was slightly lower at 0.27%. Such small, practically negligible values were the basis for concluding that annealing gypsum molds at 380°C allows for effective water removal and thus for safe pouring of the mold with liquid metal.

3.2. Roughness

Figure 9 shows the average of the measurements of the Ra and Rz parameter measurements obtained for AlSi11 alloy plates annealed according to two cycles (Fig. 5).

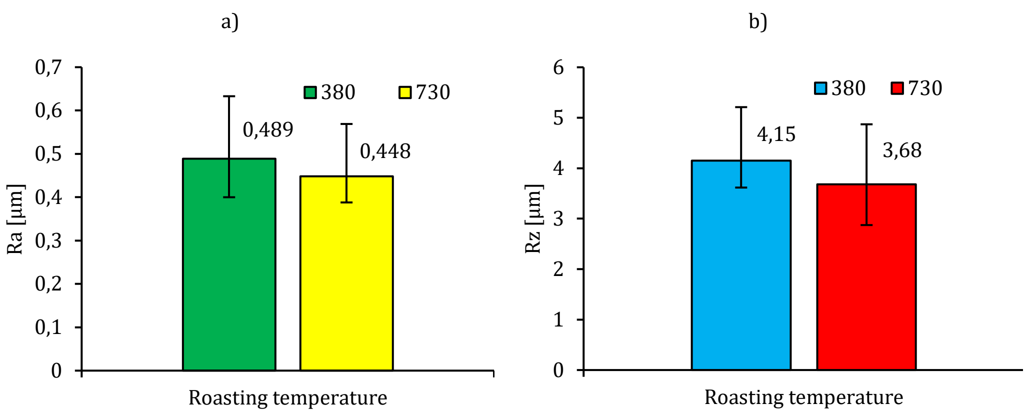


Fig. 9. Parameter a) Ra, b) Rz depending on the roasting temperature

Both roughness parameters have lower values for samples annealed at 730°C. Nevertheless, the differences between the results are marginal. For Ra, the difference between samples annealed at 380°C and 730°C was almost 0.04 µm, or 8.4%. For Rz, it was 0.47 µm, or 11.3%. It was found that a lower annealing temperature of 380°C does not significantly affect the surface quality of the castings.

3.3. Composite castings

The obtained composites (Fig. 10) were subjected to metallographic analysis. Images taken with a Nikon Eclipse MA200 optical microscope are shown in Fig. 11. The carbon fiber did not degrade. It is clearly visible against the matrix background. The mat, which served as reinforcement, was characterized by a disordered structure of carbon fibers, hence the visible uneven distribution of reinforcement in the matrix. It can be observed that the metal filled the spaces between the carbon fibers, with a size of approximately 6.23 µm.

Fig. 10, Al/CF composite

(a)

(b)

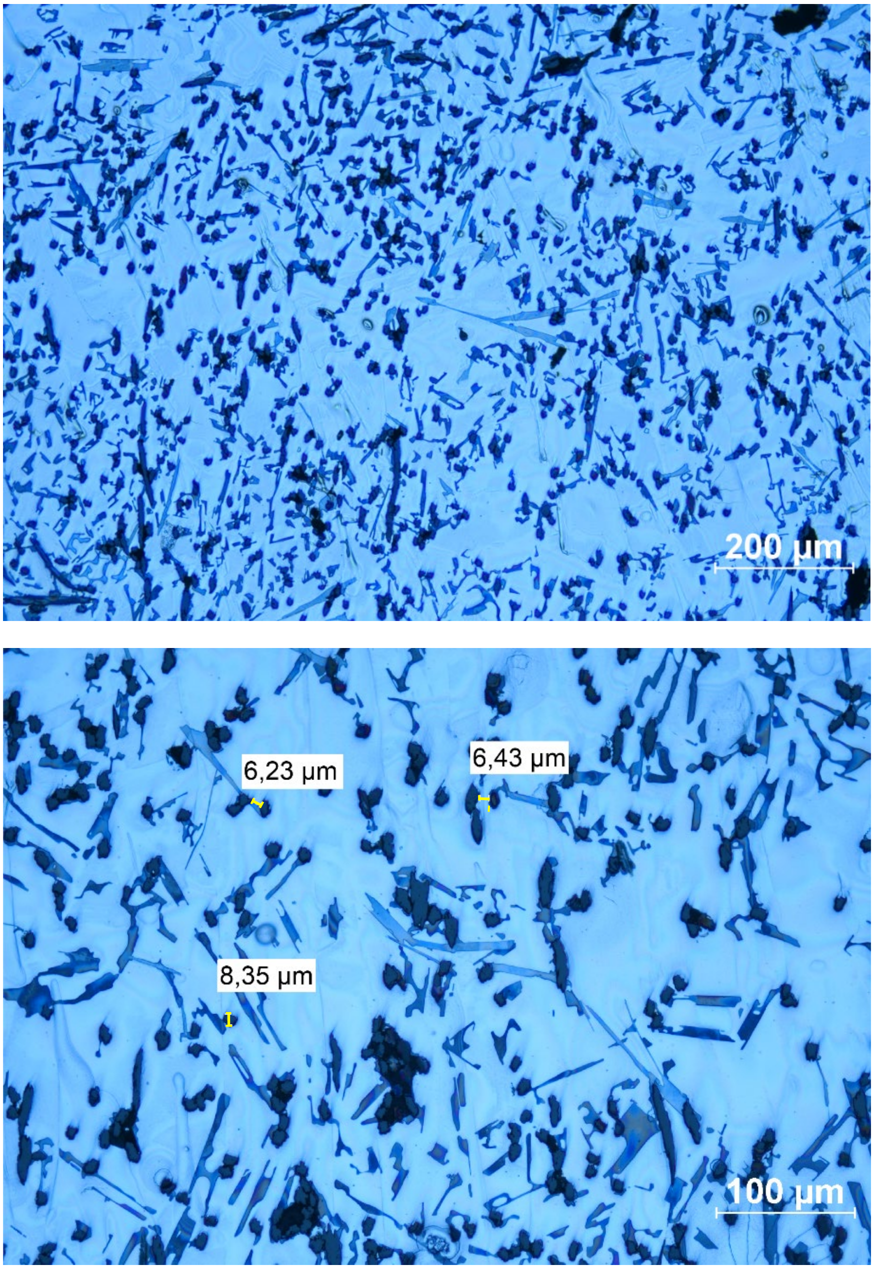


Fig. 11. Microstructure of AlSi/CF composite: (a) general view, (b) smallest distances between fibers

4. Conclusion

The research carried out in this paper allows for the following conclusions:

- It was shown that after 13 hours of annealing the mold at 380°C, the final moisture content of the ceramic was 0.33% and thus did not significantly differ from the moisture content after annealing at 730°C, which was 0.28%.
- Annealing the ceramic gypsum mold at 380°C for 13 hours allows for its safe filling with liquid metal.
- The research has shown that annealing the ceramic mold at a temperature lower than the decomposition temperature of carbon fibers does not significantly affect the surface quality of the casting. The obtained roughness parameters (Ra<0.5 μm, Rz≈4 μm) are comparable to the parameters obtained after precise surface grinding.
- Annealing the ceramic mold at 380°C is an effective method of preparing the mold for the production of metal composites

reinforced with carbon fiber. Most importantly, it eliminates the need for additional protection of the fibers against thermal degradation.

Declarations

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Competing Interests Financial interests: Authors declare they have no financial interests.

Author Contributions All authors contributed to the study conception and design. Material preparation, data collection, and analysis were performed by Paulina Szymańska, Michał Szymański and Paweł Szymański. The first draft of the manuscript was written by Paulina Szymańska. All authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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