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Piotr Nikiel

AGH – University of Science and Technology, Faculty of Metals Engineering and Industrial Computer Science, al. A. Mickiewicza 30 A, Krakow 30-059, Poland Corresponding author. E-mail: nikiel@agh.edu.pl

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AA2024/FLY ASH LIGHTWEIGHT COMPOSITES FABRICATED BY POWDER METALLURGY

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The paper presents the experimental results on the fabrication of lightweight aluminum alloy AA2024 matrix composites reinforced with fly ash microspheres using powder metallurgy (PM) via hot pressing. The primary aim was to achieve a composite with the highest possible matrix density while preserving undamaged microspheres. A mixture of powders consisting of AA2024 alloy powder and 10 wt.% fly ash was pressed at 550 °C and pressures of 5, 10, 20, and 30 MPa for two minutes. The obtained composites had densities in the range of 1.93-2.31 g/cm³. Microstructural analysis revealed uniform distribution of the fly ash microspheres in the aluminum alloy matrix. The hardness of the composites was comparable only between the samples pressed at 10, 20, and 30 MPa, falling within the range of 118 to 122 HV0.1. The sample pressed at 5 MPa exhibited significantly lower hardness of 98.8 HV0.1. The composites produced by pressing at 20 and 30 MPa were characterized by high yield strength and compression strength, ranging from 211 to 216 MPa and 300 to 354 MPa, respectively. The composites pressed applying lower pressures, especially those at 5 MPa, exhibited significantly lower values of plastic yield strength and compression strength, with values of 109 MPa and 115 MPa, respectively. Taking into account the density, the number of damaged microspheres, and the microstructure of the AA2024/fly ash composite, the optimal pressing range is between 10-20 MPa, but superior mechanical properties were achieved after pressing at 20 and 30 MPa.

Keywords: lightweight composites, AA2024 alloy, fly ash, powder metallurgy, hot pressing, microstructure, mechanical properties

INTRODUCTION

Composites based on lightweight metals such as aluminum and its alloys exhibit significant potential for applications in industries like automotive, aviation, and space. These materials offer a unique combination of properties that are often unattainable by conventional materials. The addition of ceramic particles to aluminum or aluminum alloys matrix increases their strength, stiffness and abrasion resistance, while maintaining low density. Composites reinforced with particles are manufactured by foundry or powder metallurgy methods, which can be followed by processing by means of mechanical working, e.g. forging [1-4], extrusion [5-7] or rolling [8, 9]. Examples of particles used to strengthen aluminum and aluminum alloy matrices are SiC, SiO₂, Al₂O₃, B4C, TiC, TiB₂, ZrO₂.

In recent years, composites with the addition of fly ash (ceramic microspheres) have been developed. Fly ash is an industrial waste by-product generated during the combustion of coal in thermal power plants. The main components of fly ash are SiO₂, Al₂O₃, Fe₂O₃, with a large number of trace elements. Fly ash having a density in the range 2.0-2.5 g/cm³ used in metal composites can improve various properties of matrix materials like stiffness, strength, wear resistance and can reduce the density. Fly ash cenospheres, which

are hollow fly ash particles having a density in the range 0.4-0.7 g/cm³, can be used to manufacture ultralight composite materials owing to their significantly low density [10].

Metal matrix composites based on aluminum and aluminum alloys matrix filled with fly ash have become attractive for various industries, including the automotive, marine, aviation, and aerospace sectors. The great interest in such composites is due, among others, to their low density, good compressive strength, excellent energy absorption capability, good ductility and cost-effectiveness [11-16].

Metal matrix composites with ceramic microspheres can be produced by casting methods like pressure assisted infiltration [11-15], stir casting [11-13, 16-24] and powder metallurgy methods [11-12, 24-26].

In the case of casting methods, there are problems with the wettability and floating or sinking of fly ash, which is associated its low density. Obtaining homogeneous distribution of microspheres in the matrix is still a challenge [17]. Researchers have applied various methods to improve the wettability of particles, including the addition of wettability agents, fluxes, preheating the ceramic particles, and coating the ceramic particles with Co, Cu or Ni [27-30].

Another issue arises from the chemical reactions occurring between aluminum and fly ash particles at temperatures above 700 °C. Silicon and iron are reduced by molten aluminum from their oxides in the fly ash. The solidification temperature of the aluminum melt is lower compared to pure aluminum as a consequence of enrichment in Si and Fe resulting from the reduction of Si and Fe from the SiO $_2$ and Fe $_2$ O $_3$ of the fly ash into the melt [31].

In the case of powder metallurgy (PM), the processing temperature during the production of metal matrix composites is significantly lower compared to foundry methods, resulting in less interaction between the matrix and the reinforcement. One of the advantages of PM products is also the possibility to achieve a uniform distribution of particles in the matrix [26].

PM methods are versatile because of the wide variety of particle volume fractions that can be incorporated into composites, and the fact that reactive metals can be used as matrices, which are not amenable to liquid-state processing [14]. The basic PM method that can be used to produce a metal/fly ash composite is a combination of pressing and sintering.

The major limitation of utilizing powder metallurgy for manufacturing composites with the addition of fly ash is the high pressures applied during the pressing of the powder mixture. The same problem occurs in foundry methods, albeit to a lesser extent, during processes like stir casting or infiltration. Figure 1 shows the microstructure of an Al/fly ash composite produced by hot pressing a mixture of Al powder and 10 wt.% fly ash under the pressure of 60 MPa (Fig. 1a) and by hot extrusion of the compact (Fig. 1b).

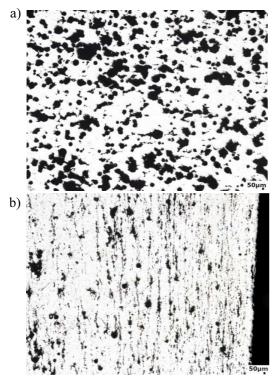


Fig. 1. Microstructure of Al/fly ash composite manufactured by: a) hot pressing and b) hot extrusion of compact

A significant part of the hollow particles was fractured in both processes, especially during hot extrusion where the fly ash particles were also fragmented and created bands along the extrusion direction.

The primary aim of this study is to determine the optimal hot pressing parameters for the production of a lightweight composite consisting of AA2024 alloy and fly ash, ensuring a high matrix density while preserving the integrity of the hollow fly ash particles.

MATERIALS AND METHODS

Attempts to create a lightweight composite using powder metallurgy were conducted by hot pressing a powder mixture of AA2024 alloy powder and 10 wt.% fly ash. The choice of aluminum alloy 2024 as the base material was driven by its superior characteristics, such as good wear resistance, good fatigue strength, average machinability, and considerable corrosion resistance. Additionally, it has a low solidus temperature, slightly above 500 °C. The chemical compositions of the alloy powder and fly ash are presented in Tables 1 and 2, respectively.

TABLE 1. Chemical composition of AA2024 powder

Chemical composition of AA2024 powder [wt.%]								
Si	Fe	Cu	Mn	Mg	Ni	Zn	Ti	Al
0.42	0.36	4.01	0.78	1.53	0.04	0.07	0.06	Bal.

TABLE 2. Chemical composition of fly ash

Chemical composition of fly ash [wt.%]						
Al_2O_3	Fe ₂ O ₃	SiO ₂	K ₂ O	CaO	MgO	TiO ₂
30-38	1-3	50-60	0.1-2	1-4	0.2-2	0.5-3

Hot pressing was carried out under isothermal conditions by means of a hydraulic press. Before processing, the dies were coated with boron nitride grease using a spray. In the first stage, tests of hot pressing the AA2024 alloy powder were conducted. Pressing was performed at 530 and 550 °C under a low pressure of 5, 10, 20 MPa for 2 minutes. The weight of the samples was approximately 75 g, and their diameter was 48 mm.

The compacting parameters of the aluminum alloy powder and fly ash were selected based on the results of pressing the alloy powder. A mix of powders, AA2024 and 10 wt.% (~30 vol.%) fly ash, was prepared by blending for two hours in a double-conical mixer. Hot pressing was carried out at a temperature of 550 °C and pressures of 5, 10, 20, and 30 MPa for 2 minutes using the same dies as pressing the pure AA2024 powder. The liquid phase content in the AA2024 alloy powder was approximately 5% [32].

Metallographic specimens were machined from central regions of the compacts and prepared using standard metallographic techniques. Microscopic observa-

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tions of etched and unetched metallographic specimens were conducted with a Leica DM4000M light microscope. The specimens were etched using Keller's reagent an aqueous solution containing 1.5 mL HCl, 2.5 mL HNO_3 , and 1 mL HF.

Density measurements of the green compacts were performed by means of the geometrical method. The hardness of the specimens was determined employing a DURAMIN-40 M1 hardness tester with a load of 0.98 N applied for 10 seconds. Both the metallographic observations and the hardness test were conducted on longitudinal sections.

Compression tests were carried out utilizing a Zwick Z250. The cubic specimens cut from the compacts had the dimensions $10 \times 10 \times -15$ mm. The test was performed at the strain rate of 0.1 s^{-1} , with the compression direction being consistent with the direction of hot pressing of the powders.

RESULTS AND DISCUSSION

Effect of pressure on density

Hot compacted specimens of AA2024 alloy powder applying pressures of 5, 10, 20 MPa and at 530, 550 °C are shown in Figure 2.



Fig. 2. Hot-pressed AA2024 specimens at 530 and 550 °C and 5, 10, 20 MPa pressure

The density of the majority of the samples exceeds 2.7 g/cm³, comparable to that of the solid material (Table 3). However, in one case, the sample pressed at 530 °C and the pressure of 5 MPa had a density of 2.6 g/cm³. Despite achieving high density, the samples pressed at lower pressures (5 and 10 MPa) at 530 °C showed signs of crumbling at the edges, as illustrated in Figure 2.

Based on the density and quality of the compacts manufactured by powder metallurgy, the parameters for pressing a mixture of AA2024 powder and fly ash were selected. The mixture was pressed only at a higher temperature of 550°C and at pressures of 5, 10, 20, and 30 MPa applied for 2 minutes. As a result, compacts with a diameter of 48 mm and a height ranging from 14 to 16.7 mm were obtained. The samples are shown in Figure 3.

TABLE 3. Density of AA 2024 compacts

Sample	Pressing temperature [°C]	Pressure [MPa]	Density of samples [g/cm³]
1		5	2.606
2	530	10	2.740
3		20	2.758
4		5	2.739
5	550	10	2.746
6		20	2.770



Fig. 3. Hot-pressed AA2024/fly ash specimens at 550 $^{\circ}$ C and 5, 10, 20, 30 MPa pressure

The samples produced from the powder mixture exhibited good quality on their surfaces, except for the one pressed at the lowest pressure of 5 MPa, where signs of crumbling at the edges were observed. The density of the compacts is notably affected by the pressure. Figure 4 illustrates the relationship between the true density and pressure during hot pressing. As the pressure increases, so does the density of the compacts. The specimen compacted at the pressure of 5 MPa exhibited the lowest density 1.93 g/cm³, while the one compacted at 30 MPa yielded the highest density at 2.31 g/cm³.

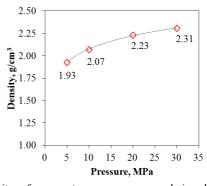


Fig. 4. Density of compacts versus pressure during hot pressing Al2024/fly ash at 550 $^{\circ}\mathrm{C}$

Effect of pressure on macro and microstructure

The microstructure of the hot-pressed AA2024 powder is shown in Figure 5 and is characterized by very fine grains. Precipitates are mainly localized along the grain boundaries. There was no significant influence of the pressing parameters on the microstructure, except in the case of the sample pressed at the temperature of 530 °C, where significant porosity was observed.

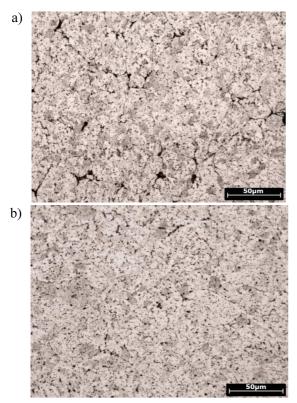


Fig. 5. Microstructure of compacts from AA2024 powder hot-pressed with following parameters: a) temperature 530 °C, pressure 5 MPa, b) temperature 550 °C, pressure 10 MPa

The surface of the cut composite AA2024/fly ash specimens after grinding and polishing is presented in Figure 6, while the macrostructure observed under a light microscope is shown in Figures 7-10a. The microstructure is shown in Figures 7-10b-c.

The distribution of the fly ash microspheres is homogeneous in the matrix of the AA2024 alloy. In the case of the sample pressed at the lowest pressure of

5 MPa and the temperature of 530°C, one can observe the boundaries of the powder particles and a significant number of pores (Fig. 7). The pores are located between the powder particles and around the microspheres. The samples pressed utilizing higher pressures of 10-30 MPa exhibit significantly lower porosity, and the boundaries of the powder particles are less visible (Figs. 8-10). In the specimen pressed at the highest pressure of 30 MPa, a significant portion is composed of microspheres that fractured due to the applied force (Fig. 10).

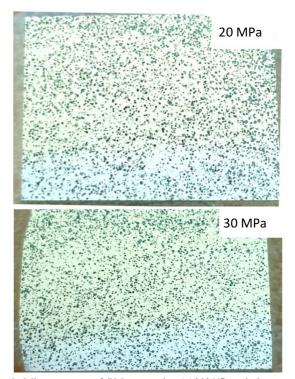


Fig. 6. Microstructure of PM composite AA2024/fly ash hot-pressed under pressures of 20 and 30 MPa at 550 °C

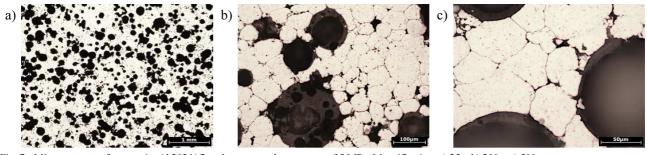


Fig.~7.~Microstructure~of~composite~Al~2024/~fly~ash~compacted~at~pressure~of~5~MPa.~Magnification:~a)~25x,~b)~200x,~c)~500x

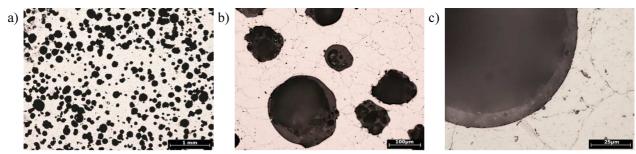


Fig. 8. Microstructure of composite Al 2024/ fly ash compacted at pressure 10 MPa. Magnification: a) 25x, b) 200x, c) 1000x

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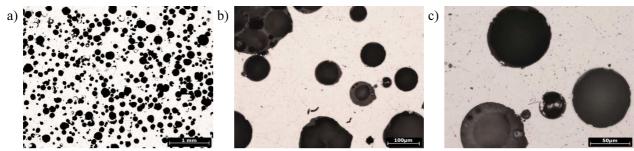


Fig. 9. Microstructure of composite Al 2024/ fly ash compacted at pressure 20 MPa. Magnification: a) 25x, b) 200x, c) 500x

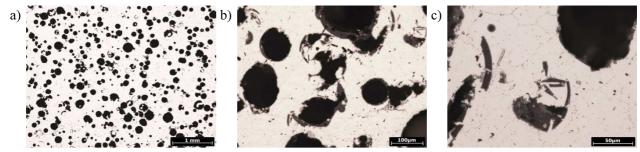
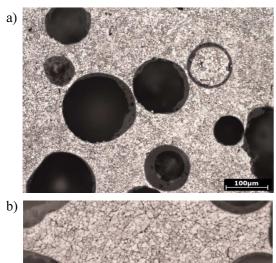


Fig. 10. Microstructure of composite Al 2024/ fly ash compacted at pressure 30 MPa. Magnification: a) 25x, b) 200x, c) 500x

The etched microstructure is shown in Figure 11, characterized by a very fine equiaxed grain structure with a size below $10 \mu m$.



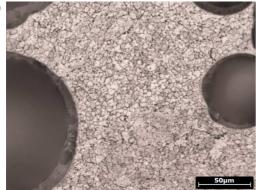


Fig. 11. Etched microstructure of composite Al 2024/fly ash compacted at pressure 20 MPa. Magnification: a) 200x, b) 500x

Considering the porosity and the presence of fractured microspheres, the most favorable option is pressing at a pressure in the range of 10-20 MPa, and the microstructure of the produced powder composite is comparable to that of cast composites.

Effect of pressure on hardness

The Vickers hardness results are presented in Tables 4 and 5. Table 4 shows the results for the compacts made from AA2024 powder, and Table 5 presents the results for the compacts made from AA2024/fly ash.

TABLE 4. Average hardness of AA2024 powder compacts

No.	Processing parameters	Average hardness HV0.1
1	T = 530 °C p = 5 MPa	96.34 ± 8.26
2	T = 530 °C p = 10 MPa	102.87 ± 2.03
3	T = 530 °C p = 20 MPa	97.69 ± 1.26
4	T = 550 °C p = 5 MPa	111.37 ± 1.77
5	T = 550 °C p = 10 MPa	106.14 ± 1.65
6	T = 550 °C $p = 20$ MPa	106.81 ± 1.53

The hardness of the specimens manufactured from the alloy powder primarily depends on the processing temperature, whereas the pressing pressure does not significantly influence the hardness. Following pressing at 530 °C, the hardness ranges from 96.3 to 102.9 HV0.1, and after pressing at 550 °C, the hardness falls within the range of 106.1 to 111.4 HV0.1.

In the case of the AA2024/fly ash composite, the hardness is comparable only for the samples pressed at 10, 20, and 30 MPa, falling within the range of 117.9 to 122.0 HV0.1. The sample pressed by applying the pressure of 5 MPa exhibits significantly lower hardness at 98.8 HV0.1, attributed to its considerable porosity. The hardness of the AA2024/fly ash specimens is more

than 10 HV0.1 higher than that of the specimens manufactured from powder AA2024, regardless of the pressing pressure, except for the samples pressed under the pressure of 5 MPa, where the hardness is comparable.

TABLE 5. Average hardness of AA2024/fly ash powder compacts

No.	Processing parameters	Average hardness HV0.1	Hardness measurement indentation
1	T = 550 °C $p = 5 MPa$	98.79 ± 7.33	
2	$T = 550 ^{\circ}\text{C},$ p = 10 MPa	117.89 ± 4.62	
3	T = 550 °C $p = 20 MPa$	118.79 ± 2.88	
4	$T = 550 ^{\circ}\text{C}$ p = 30 MPa	121.99 ± 3.17	

Effect of pressure on compression strength

Representative engineering stress-engineering strain curves are presented in Figure 12. The average values of yield stress (YS), compression strength (CS), specific yield stress (YS/ ρ), and specific compression strength (CS/ ρ) are listed in Table 6.

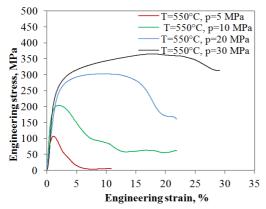


Fig. 12. Engineering stress-engineering strain curves obtained from compression tests of AA2024/fly ash composite manufactured by pressing mixed powders at 5, 10, 15, 20 MPa at 550 °C

TABLE 6. Mechanical strength properties of AA2024/fly ash composite determined in compression test

No.	Processing parameters	YS [MPa]	CS [MPa]	$\begin{array}{c} YS/\rho \\ \left[\frac{MPa}{\frac{g}{cm^3}}\right] \end{array}$	$\frac{\text{CS/p}}{\left[\frac{\text{MPa}}{\frac{g}{\text{cm}^3}}\right]}$
1	$T = 550 ^{\circ}\text{C}$ p = 5 MPa	109 ± 12.7	115 ± 11.3	56.5	59.7
2	$T = 550 ^{\circ}\text{C}$ p = 10 MPa	174 ± 1.6	198 ± 7.6	83.9	95.6
3	$T = 550 ^{\circ}\text{C}$ p = 20 MPa	216 ± 10.3	300 ± 3.1	96.8	134.6
4	$T = 550 ^{\circ}\text{C}$ p = 30 MPa	211 ± 8.5	354 ± 15.6	91.5	153.5

The yield stress, and compression strength of the AA2024/fly ash composite strongly depend on the hot pressing parameters. Pressing a mixture of powders with at the pressure of 5 MPa led to obtaining a low density of the AA2024 matrix, which results in its low yield stress and compressive strength, respectively 109 and 115 MPa. The highest strength properties of the Al2024/fly ash composite were obtained after pressing at 20 and 30 MPa. The yield stress is comparable in these cases and reaches values of 216 and 211 MPa after pressing at 20 and 30 MPa, respectively. The compression strength of the AA2024/fly ash composite is the highest after processing at 30 MPa, reaching a value of 354 MPa, which is over 50 MPa higher than the composite pressed at 20 MPa. Similar relationships occur when referring the yield stress and compression strength to the true density of the composites. The specimens pressed at 20 and 30 MPa also exhibited better plasticity, with plastic strain at the maximum stress of 8% and 16% for the samples pressed at 20 and 30 MPa, respectively.

CONCLUSIONS

An alternative to producing syntactic foam by casting methods can be powder metallurgy methods like hot pressing of powders. Hot pressing with the liquid phase of the mixture of Al 2024/fly ash powder requires using low pressures to obtain a high density of matrix and with undamaged microsphere particles.

The following conclusions were drawn from the study:

- Lightweight composites were manufactured by hot pressing AA2024 and fly ash powders, with a density in the range of 1.93-2.31 g/cm³.
- The manufactured composites exhibit uniform distribution of the fly ash microspheres in the aluminum alloy matrix.
- The density of the AA2024/fly ash composites is related to the pressure during hot pressing. The pressure of 5 MPa is too low to attain the appropriate matrix density, while the pressure of 30 MPa leads

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to the destruction of a significant number of microspheres.

- The yield stress, compression strength, and plastic deformation are significantly greater for the composites manufactured by hot pressing at higher pressures of 20 and 30 MPa.
- Considering the density, microstructure, hardness, yield stress, and compression strength, the optimal pressure range of hot pressing a mixture of AA2024 powder and 10 wt.% fly ash at 550 °C is between 20-30 MPa.

Based on the obtained research results, the planned further studies will aim to manufacture an AA2024/fly ash composite with a significantly lower density by gradually increasing the fly ash content.

Acknowledgement

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