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COMPOSITES THEORY AND PRACTICE

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THE EFFECT OF SINTERING TEMPERATURE, SINTERING TIME AND REINFORCEMENT PARTICLE SIZE ON PROPERTIES OF Al-Al_2O_3 COMPOSITES

The main aim of this paper was to investigate the effect of sintering temperature, sintering time and reinforcement particle size on the properties of Al-Al_2O_3 composites. Three sintering temperatures were applied: 500, 550 and 600°C for 30, 60 and 90 minutes. Experiments were performed using specimens containing 0, 5 and 10% of alumina. The average particle sizes of alumina were 2, 10 and 20 μm. The investigated properties included relative density, hardness and compressive strength. Microstructural observations showed that Al-Al_2O_3 composites could be successfully formed for all of the studied combinations of sintering temperature, sintering time, alumina particle sizes and amount of reinforcement. The relative density of all the composites increased when the particle size of alumina was reduced. The highest relative density was obtained at 600°C. Higher hardness and compressive strength were observed in samples containing finer Al_2O_3 particles. The variations in hardness and compressive strength of Al-Al_2O_3 composites were dependent on the sintering temperature and time. Increasing sintering time at 500 and 550°C led to a gradual increase in hardness and compressive strength. Similar results were obtained as well after sintering at 600°C when the sintering time was increased up to 60 minutes. A prolonged sintering time up to 90 min had a contrary effect on the hardness and compressive strength of the composites.

Keywords: metal-matrix composite, aluminium, Al_2O_3 particles, powder metallurgy

WPŁYW TEMPERATURY SPIEKANIA, CZASU SPIEKANIA ORAZ WIELKOŚCI CZĄSTEK ZBROJĄCYCH NA WŁASNOŚCI KOMPOZYTÓW Al-Al_2O_3

Głównym celem badań było przeanalizowanie wpływu temperatury spiekania, czasu spiekania oraz wielkości cząstek zbrojących na własności kompozytów Al-Al_2O_3. Zastosowano trzy temperatury spiekania: 500, 550 i 600°C oraz trzy czasy: 30, 60 oraz 90 minut. Eksperymenty prowadzono na próbkach wykonanych ze spiekanego proszku aluminium oraz na próbkach zawierających dodatkowo 5 i 10% tlenku glinu. W badaniach używano proszku Al_2O_3 o trzech wielkościach cząstek: 2, 10 oraz 20 μm. Badano wpływ parametrów wytwarzania kompozytów na ich gęstość względną, twardość oraz wytrzymałość na ściskanie. Obserwacje mikroskopowe wykazały, że kompozyty Al-Al_2O_3 mogą być skutecznie wytwarzane przy zastosowaniu wszystkich użytych kombinacji temperatury spiekania, czasu spiekania oraz zastosowanych ilości oraz wielkości cząstek zbrojących. Gęstość względna kompozytów wzrastała wraz ze zmniejszaniem się wielkości zastosowanych cząstek Al_2O_3. Najwyższą względną gęstość otrzymano dla kompozytów spiekanych w temperaturze 600°C. Wyższa twardość oraz wyższa wytrzymałość na ściskanie była uzyskiwana w próbkach zawierających mniejsze cząstki tlenku glinu. Zmiany twardości i wytrzymałości na ściskanie były uzależnione od czasu oraz temperatury spiekania. Wzrost czasu spiekania w temperaturach 500 oraz 550°C prowadził do stopniowego wzrostu twardości oraz wytrzymałości na ściskanie. Podobne rezultaty otrzymano także po spiekaniu w temperaturze 600°C, gdy czas spiekania był krótszy niż 60 minut. Jednak wydłużenie czasu spiekania do 90 minut spowodowało spadek twardości oraz wytrzymałości na ściskanie.

Słowa kluczowe: kompozyt z osnową metaliczną, aluminium, cząstki Al_2O_3, metalurgia proszków

INTRODUCTION

Aluminium metal matrix composites (Al MMCs) are a group of new, advanced materials which have found various applications in aerospace, automotive and military/defence industries. This is due to their low density, high strength, good wear and corrosion resistance as well as low coefficient of thermal expansion [1-4]. Mostly all Al MMCs include SiC, B_4C or Al_2O_3 particles. Al-SiC and Al-B_4C systems are reactive systems, as they produce Al_4SiC_4, Al_3BC, AlB_2 or Al_4C_3 compounds at the interface of the particles and metal [5]. The presence of these compounds, especially Al_4C_3, is detrimental to the composites properties. Compared to SiC and B_4C particles, alumina has shown better thermal stability at high temperatures. Furthermore, no intermediate phases are expected at the interface of Al-Al_2O_3 and the only reaction is the dissolving of Al_2O_3 into aluminium. The processing techniques for Al MMCs can be classified into: powder metallurgy (PM), liquid state processing and semisolid processing. Casting products can be made by dispersing ceramic particles into molten aluminium with ultrasonic vibration or by blowing reducible oxides of high melting point metals into an aluminium melt. On the other hand, powder metallurgy is the most common production technique for MMCs. The main advantage of PM compared to casting is better control of the distribution of the reinforcement in PM compacts. Solid state diffusion, dependent on time and sintering temperature, plays a major role in PM in the formation and growth of interparticle bonding [6]. The particle size and amount of reinforcement have a great effect on the mechanical properties of Al MMCs [7-10]. A classic example is sintered aluminium powder (SAP), produced using very fine aluminium powders (0.1÷1μm) strengthened by up to 14% Al_2O_3 [11-13]. SAP has properties quite different from those of materials fabricated by conventional techniques. The oxide that forms immediately on the surface of aluminium is not reduced back to metal during sintering and the resulting powder product contains a substantial amount of oxide. This oxide prevents grain growth and movement of dislocations at the boundaries or through them and produces high strength (up to 400÷500 MPa at 14% Al_2O_3), high creep resistance and insensitivity to high-temperature exposure. The modulus of elasticity increases with the oxide content to reach values of 77÷80 GPa at 12÷14% Al_2O_3, declining with temperature as does the strength. The damping capacity of SAP is about 20 times higher than that of aluminium [12]. Neutron or ion irradiation hardens the material therefore it can be used in nuclear reactors [11]. The material properties depend on the amount of naturally formed oxide. Heating powder to increase the thickness of the oxide film does not increase strength and only reduces ductility. The oxide formed on the powder is up to 10 nm thick, amorphous and contains absorbed water. The absorbed water reacts with the metal to form an additional oxide and release hydrogen that may produce porosity at the grain boundaries as well as/in addition to cracking or blistering. Vacuum treatment or high-temperature sintering before complete compacting reduces the hydrogen content and eliminates most if not all cracking. Small additions of aluminium fluoride also reduce the effect of hydrogen. The main drawback of these SAP composites is their high production cost. In the present work, the Al-Al_2O_3 composites were formed using aluminium and alumina powders because it is the most economical production technique. Since the used Al and Al_2O_3 powders were much coarser than those used in SAP, the produced composites had been supposed to have lesser mechanical properties than dispersion strengthened composites. On the other hand, the proper addition of reinforcements to aluminium composites can have a positive effect on the mechanical properties, such as hardness, compressive strength and wear resistance. The main aims of the present work were to investigate the effect of sintering temperature, sintering time, alumina particle size and the amount of reinforcement on the mechanical properties of Al-Al_2O_3 composites made by mean of the conventional PM technique.

EXPERIMENTAL PROCEDURE

In the experiment, an aluminium powder (99.99% Al, 0.003% Cu, 0.003% Ti, 0.002% Si and 0.002% Zn) with an average particle size of 40 μm was used as a matrix. Alumina (97.4% α-Al_2O_3, 1.3% TiO_2, 1.1% CaO, 0.2% Fe_2O_3) with an average particle size of 2, 10 and 20 μm was used as a reinforcement. Proper proportions of the powders (containing 0, 5 or 10% Al_2O_3) with ethanol as a process control agent were placed in a planetary ball mill for 60 min. at 120 rpm, where the ball to powder ratio was 8:1. After drying, the composite powders were uniaxial pressed (under pressure of 100 MPa) to produce samples. The green compacts were sintered in argon at 500, 550 and 600°C for 30, 60 and 90 min. After fabrication, the samples were cut, mounted in a cold setting resin, mechanically ground with a grade 1000 abrasive paper and finally chemically polished in an aqueous solution of 100 ml H_2O, 80 ml H_3PO_4 and 4 ml HNO_3 (temp. 85°C, time 4 min.). Microstructural observations were performed using a scanning electron microscope, JMS 5400 equipped with EDX spectroscopy and an optical microscope, NEOPHOT 2. For the study of the structure using an optical microscope, the samples were etched with a solution of 5 pct HF. The hardness measurements were performed on a Brinell scale with a ball diameter of 2.5 mm and a load of 294 N. Samples 10 mm x 10 mm x 10 mm, made from fabricated composites, were subjected to compression tests on an AMSLER screw machine at a strain rate of 0.5 mm per minute.

RESULTS AND DISCUSSION

Al-Al_2O_3 composites were successfully formed for all the studied combinations of sintering temperature, sintering time, alumina particle sizes and amount of reinforcement. Figure 1 shows examples of the microstructures of the fabricated composites.

The effects of sintering temperature, sintering time, alumina particle size and amount of reinforcement on the relative density, hardness and compressive strength of Al-Al_2O_3 composites are depicted in Tables 1-3. The theoretical densities of aluminium, Al+5% Al_2O_3, Al+10% Al_2O_3 and alumina are 2.7, 2.744, 2.789 and 3.97 g/cm^3, respectively [14]. In order to reduce errors, the data shown below are the average of five experimental values.

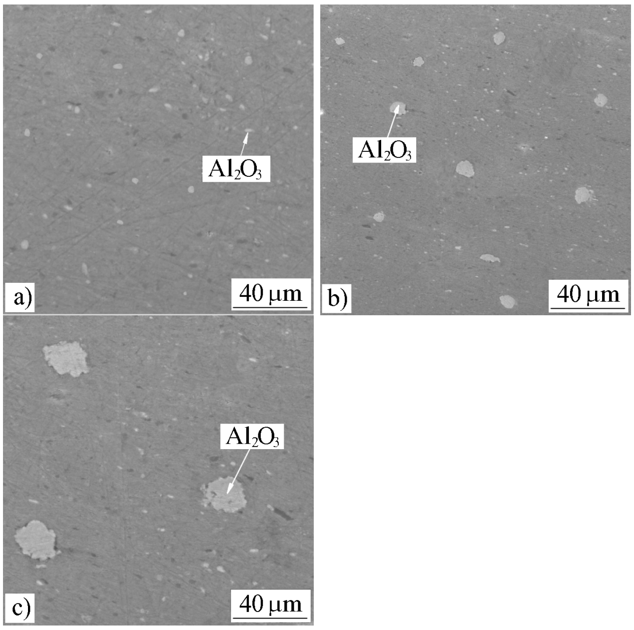

Fig. 1. Microstructures of composites containing 5% Al_2O_3 with particle sizes of 2 μm (a), 10 μm (b) and 20 μm (c) sintered at 600°C for 60 min

Rys. 1. Mikrostruktury kompozytów zawierających 5% Al_2O_3 o wielkościach cząstek 2 μm (a), 10 μm (b) i 20 μm (c) spiekanych w temperaturze 600°C przez 60 minut

TABLE 1. **Effect of sintering time, alumina particle size and amount of Al_2O_3 on relative density, hardness and compressive strength of composites sintered at 500°C**

TABELA 1. **Wpływ czasu spiekania, wielkości cząstek i ilości Al_2O_3 na gęstość względną, twardość oraz wytrzymałość na ściskanie kompozytów spiekanych w temperaturze 500°C**

Temp. [°C]	Time [min]	Alumina particle size [μm]	Amount of Al_2O_3 [%]	Relative density [%]	Brinell hardness [HB]	Compressive strength [MPa]
500	30	pure Al	-	98.67	15.1	81
		2	5	98.07	48.4	212
			10	97.52	59.7	241
		10	5	97.11	26.4	186
			10	96.56	44.6	198
		20	5	96.42	23.9	175
			10	96.02	41.3	202
	60	pure Al	17,2	98.81	17.2	94
		2	5	98.25	51.3	243
			10	97.67	65.4	275
		10	5	97.24	43.8	218
			10	96.61	58.7	242
		20	5	96.50	40.3	192
			10	96.25	50.2	224
	90	pure Al	25.9	98.96	25.9	99
		2	5	98.36	54.9	248
			10	97.96	70.4	281
		10	5	97.38	45.7	218
			10	96.83	61.2	232
		20	5	96.67	42.2	201
			10	96.48	50.6	222

TABLE 2. **Effect of sintering time, alumina particle size and amount of Al_2O_3 on relative density, hardness and compressive strength of composites sintered at 550°C**

TABELA 2. **Wpływ czasu spiekania, wielkości cząstek i ilości Al_2O_3 na gęstość względną, twardość oraz wytrzymałość na ściskanie kompozytów spiekanych w temperaturze 550°C**

Temp [°C]	Time [min]	Alumina particle size [μm]	Amount of Al_2O_3 [%]	Relative density [%]	Brinell hardness [HB]	Compressive strength [MPa]
550	30	pure Al	-	98.93	15.9	83
		2	5	98.38	53.7	246
			10	97.85	68.2	275
		10	5	97.47	28.8	212
			10	96.91	49.2	236
		20	5	96.74	24.7	205
			10	96.42	46.3	231
	60	pure Al	-	99.18	25.9	98
		2	5	98.55	58.2	275
			10	97.96	72.4	307
		10	5	97.51	48.7	241
			10	96.94	64.1	263
		20	5	96.84	44.5	218
			10	96.57	51.8	251
	90	pure Al	-	99.28	38.1	102
		2	5	98.59	59.2	277
			10	98.27	75.3	312
		10	5	97.73	50.1	245
			10	97.15	65.8	266
		20	5	96.95	46.2	222
			10	96.88	54.6	254

The effect of sintering temperature and sintering time on the relative density of Al-Al_2O_3 composites is obvious, because diffusion strongly depends on temperature and time in accordance with the Arrhenius equation [15]. Therefore, higher relative densities are achieved at higher sintering temperatures and with longer time. On the other hand, porosity is related to density. With the addition of alumina particles to the aluminium matrix, the relative density decreases independently of the sintering temperature and sintering time. The level of porosity is the lowest for pure aluminium. An addition of Al_2O_3 and increasing the particle size of alumina as well as the amount of reinforcement increase porosity. This can be explained by the lower compressibility of alumina compared to aluminium. Therefore, lower densities are obtained for composites containing 5% of alumina and even lower for those ones containing 10% of alumina. Similar results were received by Ahmad et al. [16]. It is well known that in particulate metal-ceramic composites containing the same amount of reinforcement with different sizes of particles, the distance between particles increases with an increase in the particle size [1, 4]. A strengthening phase acts as a barrier against the movement of the grain boundaries at high temperature during the sintering process. Therefore, the larger the particles of alumina used, the larger the grains in sintered composites are expected [10, 17]. Microstructural investigations proved the above statement. The reinforcement higher particle size and longer sintering time led to a higher grain size in the observed microstructures.

TABLE 3. **Effect of sintering time, alumina particle size and amount of Al_2O_3 on relative density, hardness and compressive strength of composites sintered at 600°C**

TABELA 3. **Wpływ czasu spiekania, wielkości cząstek i ilości Al_2O_3 na gęstość względną, twardość oraz wytrzymałość na ściskanie kompozytów spiekanych w temperaturze 600°C**

Temp. [°C]	Time [min]	Alumina particle size [μm]	Amount of Al_2O_3 [%]	Relative density [%]	Brinell hardness [HB]	Compressive strength [MPa]
600	30	pure Al	-	99.29	20.1	89
		2	5	98.64	58.8	265
			10	98.16	73.1	297
		10	5	97.71	30.1	230
			10	97.22	52.4	254
		20	5	96.97	28.9	212
			10	96.73	49.3	247
	60	pure Al	-	99.45	40.6	111
		2	5	98.84	61.7	294
			10	98.29	77.8	328
		10	5	97.80	52.4	258
			10	97.25	68.2	282
		20	5	97.17	48.4	235
			10	96.89	56.1	268
	90	pure Al	-	99.61	31.3	94
		2	5	98.89	58.3	253
			10	98.55	68.6	285
		10	5	98.07	45.6	212
			10	97.42	60.1	231
		20	5	97.22	35.6	201
			10	97.05	52.9	228

Generally, higher hardness and compressive strength were observed in samples containing finer Al_2O_3 particles. The same phenomenon was also noticed by other researchers [1, 18, 19] and it has been attributed to a greater interfacial area between the matrix and strengthening phase. On the other hand, large alumina particles have a tendency to fracture at compaction pressure, which leads to a higher porosity and subsequently to lesser hardness and compressive strength. It was also observed that both sintering temperature and sintering time had a strong impact on the mechanical properties. Increasing the sintering time at 500 and 550°C led to a gradual increase in hardness and compressive strength. Similar results were obtained as well after sintering at 600°C when the sintering time increased from 30 to 60 minutes. Opposite results were received when the sintering time increased from 60 to 90 minutes. The hardness and compressive strength of all the investigated samples was reduced after 90 min of sintering at 600°C, independent of the amount of alumina. The reduction can be explained according to the Hall-Petch theory [20]. At the sintering temperature of 600°C and sintering time of 90 minutes, considerable grain growth occurred which led to lower hardness and strength. The highest properties, hardness and compressive strength, were obtained for composites sintered at 600°C for 60 minutes.

CONCLUSIONS

The Al-Al_2O_3 composites were formed by powder metallurgy. The following conclusions can be drawn:

1. Al-Al_2O_3 composites can be successfully formed using all of the studied combinations of sintering temperature (500, 550 and 600°C), sintering time (30, 60 and 90 min), alumina particle size (2, 10 and 20 μm) and amount of reinforcement (5 and 10%).
2. The relative density of the composites was higher when the particle size of alumina was reduced.
3. The highest relative density of 98.89% was observed in specimens containing 5% Al_2O_3 with a particle size of 2 μm sintered at 600°C.
4. A larger alumina particle size and longer sintering time led to a higher grain size in the observed microstructures.
5. Higher hardness and compressive strength were observed in samples containing finer Al_2O_3 particles.
6. The highest hardness and compressive strength were obtained in specimens containing 10% of alumina with a particle size of 2 μm sintered at 600°C for 60 min, 78 HB and 328 MPa, respectively. A further increase in sintering time to 90 min. resulted in a reduction in hardness and compressive strength to 69 HB and 285 MPa.

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