

PREPARATION OF Al BASED COMPOSITE REINFORCED WITH FINE DROSS PARTICLES

PRIDOBIVANJE KOMPOZITOV NA OSNOVI ALUMINIJA, DISKONTINUIRANO OJAČANIH Z DELCI ALUMINIJEVE ŽLINDRE

VARUŽAN M. KEVORKIJAN¹, B. ŠUŠTARŠIČ², M. TORKAR²,
G. CHIARMETTA³

¹Independent researcher, Lackova 139, 2341 Limbuš, Slovenia

²IMT, Lepi pot 11, 1000 Ljubljana, Slovenia

³Stampal, S.p.A., Via Albenga 78, 10 090 Cascine Vica, Italy

Prejem rokopisa - received: 1998-11-16; sprejem za objavo - accepted for publication: 1998-12-07

In the casting-house aluminum dross represents a permanent source of waste generation, especially on the end of secondary dross processing, when a fine, low metal content dust, consisting mostly of Al_2O_3 phase, is usually generated by tumbling. In the present work, this finely powdered by-product was employed as a low cost reinforcement in discontinuously reinforced aluminum matrix composites. By using the fine (less than $50\ \mu m$) fraction of non-metallic particles generated during aluminum dross treatment and part of the in-house recovered aluminum alloy (A 380), low cost aluminum dross metal matrix composites (AIDMMCs) were routinely produced by the conventional squeeze casting technique and the pressure infiltration. The tensile strength and hardness properties of up to 20 vol.% fine dross particles in AIDMMC were examined. Special attention was paid to the intrinsic economy of the overall process in order to develop a real low cost metal matrix composite suitable for applications in the automotive industry. It was also demonstrated that AIDMMCs can be easily separated into their two constituents parts, the matrix alloy and the dross particles, by simply re-melting and dross treatment. The ability of AIDMMCs to be recycled in a cost effective way can additionally initiate large tonnage applications for these materials in the automotive sector. In this way, the procedure applied can lead to the production of MMCs at a lower cost per kg than the price of (even recycled) aluminum alloys, as a result of the extremely low price of the filler. The initial results also suggest that the fine fraction of aluminum dross particles is a promising filler for light and wear resistant automotive parts.

Key words: metal-matrix composites (MMCs), particle reinforced composites, aluminum dross particles, squeeze casting, pressure infiltration, tensile properties, wear properties

V delu je opisana uporaba ciklonske frakcije delcev aluminijeve žlindre, sestavljenih pretežno iz Al_2O_3 (s povprečno velikostjo delcev pod $50\ \mu m$) kot ojačitvene faze za pripravo diskontinuirano ojačanih kompozitov na osnovi regenerirane aluminijeve zlitine A-380. Regenerirano zlitino smo pridobili s stiskanjem posnemkov iz sekundarnega litja v Altek-ovi stiskalni napravi. Vzorce kompozita smo izdelali s tlačnim vlivanjem in s tlačno infiltracijo prahu žlindre v tovarni Stampal S.p.A., kjer omenjeni tehnologiji uporabljajo v redni proizvodnji avtomobilskih delov. V nadaljevanju smo, poleg običajnih metalografskih raziskav, določili trdnost in trdoto vzorcev kompozita, ojačanih z 20 vol% delcev žlindre. Posebno pozornost smo namenili ekonomski evalvaciji predlagane tehnologije ojačitve regeneriranih aluminijevih zlitin. Ekonomska analiza je pokazala, da so regenerirane aluminijeve zlitine, ojačane z delci žlindre, cenejše od aluminijevih zlitin, kar velja tudi za avtomobilске komponente, pripravljene s tehnologijo tlačne infiltracije. Poleg tega smo ugotovili, da uporaba finih delcev aluminijeve žlindre omogoča zelo enostavno recikliranje obrabljenih izdelkov s ponovnim pretaljevanjem in iztiskanjem Al taline iz nekovinskega ostanka na Altek-ovi stiskalni napravi. Zaradi svoje ugodne cene in možnosti učinkovitega recikliranja je ojačitev regeneriranih aluminijevih zlitin s ciklonsko frakcijo delcev žlindre vsekakor zanimiva za pripravo zelo lahkih, mehansko manj obremenjenih avtomobilskih delov, ki so v prvi vrsti izpostavljeni abraziji.

Ključne besede: kompoziti s kovinsko osnovo, diskontinuirano ojačani z delci aluminijeve žlindre, tlačno vlivanje z vtiskanjem, tlačna infiltracija, preskušanje natezne trdnosti, preskus abrazijske obrabe

1 INTRODUCTION

The high cost of current MMCs compared to aluminum alloys has inhibited production on a large industrial scale, for example in the automotive industry. In attempts to overcome this limitation, several R&D programs¹⁻³ were focused on the refinement of aluminum-based MMCs using low cost industrial waste by-products as the reinforcement particulate. Reinforcing an aluminum alloy with particles of a second phase can improve the physical, mechanical and tribological properties of the material, or it may result in material savings at little detriment to the properties desired. This could reduce the cost and the weight of energy intensive metals for potential applications in engineering components for a new

generation of vehicles. However, the future commercialization of these materials in automotive industry will be also influenced by the ability to recycle the scrap and the final components when its useful life is over.

On the other hand, discovering a complete, environmental-friendly and economical consumption of powdered by-products generated during aluminum melting operations and dross processing represents a crucial need of advanced aluminum smelters^{4,5}.

Recently fly ashes from coal combustion have been successfully combined with aluminum alloys using the foundry process to produce a class of MMCs called Ashalloys^{6,8}. It was demonstrated by Rohatgi et al.^{1,3,6} that Ashalloys offer the advantages of reducing the disposal volumes of the electric utility industry, providing a

high value-added use of fly ash, and at the same time introduced a class of new materials with improved properties at reduced cost.

Stir casting and other liquid metal routes⁹ were used to synthesize Ashalloy composites. However, both the lightness and non-wetting nature of fly ash particles tend to cause rejection and floatation of fly ash in an aluminum melt. Because of this, in some novel attempts the pressure infiltration technique was also applied in order to produce composites with higher volume fractions of non-metallic filler which have improved microstructural and mechanical properties.

Fly ashes and powdered dross particles are mixtures of different oxides. In both cases, the main constituents are SiO_2 and Al_2O_3 . However, fly ashes typically contain 60 wt.% of SiO_2 , 26 wt.% Al_2O_3 and a very low iron oxide content. In contrast, fine powdered dross particles collected in a cyclone usually consist of 80 wt.% of Al_2O_3 and only 1-2 wt.% of SiO_2 . The residue is composed of salts (aluminum nitrides and carbides may also be present, as well as metals oxides derived from the molten alloy).

Chemically pure alumina particles are well established as reinforcement in a broad portfolio of commercially available MMCs. Based on this, one can speculate that the introduction of fine dross particles, consisting of more than 80 wt.% alumina, into a Al matrix should result in a substantial improvement of hardness and wear resistance of the composite material, especially in comparison with Ashalloys reinforced with low alumina content fly ash particles.

The main objective of this work was to investigate the possible benefits and the advantages of using of a new reinforcing phase consisting of fine dross particles in aluminum-based MMCs. Special attention was paid to the intrinsic economy of the overall process in order to develop a real low cost metal matrix composite suitable for the application in the automotive industry. It was also recognized that AlDMMCs can be easily separated into its two constituents parts, the matrix alloy and the dross particles by convenient re-melting and dross treatment¹⁰. One can expect, that the ability of AlDMMCs to be recycled in a cost effective way could additionally initiate the large tonnage applications for these materials in the automotive segment.

By combining the fine fraction of dross particles as a reinforcement and in-house recovered aluminum alloy as the matrix, low cost MMCs with valuable properties were prepared using conventional squeeze casting or pressure infiltration techniques. In this way, it was demonstrated that the applied procedure could lead to production of MMCs at a lower cost per kg than the price of (even recycled) aluminum alloys, as a result of the extremely low price of the filler.

2 EXPERIMENTAL PROCEDURE

2.1 Materials Used for Experiments

Recycled A 356 alloy was used for preparation of composites. The cyclone fraction of dross particles was collected by the firm IMPOL, d.d. in Slovenska Bistrica, Slovenia. The chemical analysis of powdered specimens indicated that dross particles contain ≈ 80 wt.% of Al_2O_3 , ≈ 1.5 wt.% of SiO_2 , ≈ 1.5 wt.% of metallic aluminum, ≈ 10 wt.% of salts and ≈ 7 wt.% of AlN. The results of SEM, TEM and X-ray diffraction analysis of dross particles indicated that the particles are angular and predominantly consist of beta alumina (≈ 40 -60 wt.%) and spinel (20-30 wt.%). The average particle size (d_{50}) of the cyclone fraction was 67 μm .

As received particles displayed a bulk density of 1.42 g/cm^3 , a tap density of about 1.49 g/cm^3 , and a density measured by means of a pycnometer of 2.28 g/cm^3 .

2.2 Experimental Procedure of Squeeze Casting

For fabrication of composite specimens by squeeze casting, a three piece die was designed and fabricated from 1040 steel. The die cavity (1.8 cm internal diameter), the bottom and the top punch were case hardened to a maximum hardness of 55 Rc to a depth of 2.5 mm. The squeeze casting procedure was as follows. Dross particles were packed dry without binders in a mold made of a rigid but porous ceramic board (Zircar Products, Inc.). When the packing was completed the mold was closed with a thin porous ceramic cover. The assembly was then heated to $\approx 750^\circ\text{C}$ and subsequently placed in the die cavity of a squeeze caster, which had been preheated to 400°C . The alloy melt, degassed and superheated by 100 - 150°C , was immediately poured into the die cavity containing the preform assembly and pressurized to 170 MPa, maintaining this pressure until the metal had completely solidified.

The casting containing the composite was then extracted from the die and allowed to cool in air.

2.3 Experimental Procedure of Pressure Infiltration

Pressure infiltration experiments were performed with small lab scale infiltration equipment. The main part of this unit was a water cooled pressure chamber in which a graphite crucible for molten aluminum alloy was surrounded by two semi-cylindrical heating elements. The dross particles were packed in a quartz tube of 6 mm ID and 200 mm length. After packing, the tube was immersed 2 cm in the melt which was kept in a graphite crucible.

An argon gas pressure was then applied at a constant rate (0.02 MPa/s) up to 0.7 MPa. At the end of infiltration, the argon gas in the pressure chamber was vented out, the lid of the pressure chamber removed, and the

tube containing the specimen removed from the holder and allowed to cool in air.

Once the infiltration specimen was cool, the quartz tube was broken carefully to obtain the composite specimen.

2.4 Experimental Procedure of Recycling of AlDMMCs

Recycling of AlDMMCs were investigated in an apparatus consisted of a mullite crucible placed inside a resistance heated vertical muffle furnace having a bottom pouring arrangement. The bottom hole of the crucible (12 mm Φ) was plugged with a graphite stopper. The mixing assembly consisted of a DC variable speed motor, the spindle and the dispersion impeller having blades angled from about 15° to about 45° from a line perpendicular to the shaft. The use of a specially designed impeller having angled blades provided a shearing and wiping action which effectively caused the separation of dross particles from the molten aluminum. The impeller was machined from stainless steel and then coated with Armeco 552 ceramic adhesive.

The crucible was provided with a protective cover and an inert gas bubbling arrangement.

After complete re-melting of composite specimens, combined by a vigorous mixing and treating of the melt by argon gas fluxes, the hot dross particles were carefully skimmed into two-piece cast alloy steel skimbox set and pressed immediately in special press unit (Altek International Inc.) in order to achieve the maximum recovery of aluminum alloy. The pressed dross is then discharged into storage hopper or bin for further recycling at the dross processor.

2.5 Testing Techniques

Volume percentage of dross particles in the matrix: The volume percentage of dross particles in the squeeze cast and gas pressure infiltrated composites was measured on polished samples by a LECO 2001 Image Analyzer. Three pictures were quantitatively analyzed from one sample.

Hardness and Microhardness: A selected number of squeeze cast and gas pressure infiltrated samples were tested for Brinell hardness and microhardness. The sample surface was polished to 600 grit. The Brinell hardness of specimens was measured using a 5 mm diameter hardened steel ball indenter. A load of 250 kg was applied for 30 s. Vickers microhardness measurements (20g load) were taken from the aluminum matrix.

Tensile Testing: Tensile specimens of gauge length of 10 mm and cross section of 6x2 mm were machined from thin plates. Tensile tests were conducted using an Instron testing machine and a 10 mm extensometer.

Wear Testing: Preliminary dry sliding wear testing of squeeze cast and of some gas pressure infiltrated specimens against an SAE 52100 steel counterface was carried out using a pin-on-disc wear testing machine with a

sliding speed of 1.0 m.s⁻¹ and an applied contact load of 60 N. Wear rates were calculated using the relationship:

Wear Rate = Volume of Wear (mm³)/Sliding Distance (m)

To start the test, the sample was lowered by the operator onto the already rotating disc; contact was maintained for 2000 seconds.

3 RESULTS AND DISCUSSION

Figure 1 shows the solidification microstructure in A-356-22.8 vol.% dross particles composite. It can be observed that the distribution of reinforcement in the composite is not uniform. Individual dross particles as well as clusters formed by small size dross particles are found in the interdendritic regions, which can influence the strength of composite, causing the composites to have lower hardness and lower strength. On the other hand, it appears the interfacial contact between aluminum alloy matrix and dross particles to be good.

The results for the volume percentage of dross particles in the prepared specimens are given in **Table 1**. As evident, both techniques are effective in the preparation of high dross particle content composites with more than 20 vol.% of filler phase.

The Brinell hardness and Vickers micro hardness of non-reinforced A 356 alloy and selected reinforced species of composite prepared by squeeze casting and gas pressure infiltration are compared in **Table 2**. It was found that the micro hardness of composites reinforced with dross particles in the matrix aluminum area is higher than in non-reinforced A356 alloy. This is probably due to an increase in the volume of precipitated phases, the formation of some intermetallics or a high dislocation density.

The results of a preliminary investigation of the tensile properties of squeeze cast and gas pressure infiltrated specimens are collected in **Table 3**.

As evident, both grades of composite reinforced with fine dross particles show very good strength. However, in future work a very detailed inspection of the tensile properties of a larger number of specimens with more uniform microstructure will be necessary in order to confirm these findings. Moreover, a very detailed analysis of the solid, semi-solid, and liquid formability in a larger series of specimens will be also required before the possible competitiveness of these materials in the automotive industry can be properly assessed.

The preliminary dry sliding wear rate of the squeeze cast and the gas pressure infiltrated parts against an SAE 52100 steel counterface is shown in **Figure 2**. The wear resistance of dross containing castings was also compared with the resistance of non-reinforced A356 alloy. As expected, the addition of dross particles to the aluminum alloy significantly increased its wear resistance. Based on this, both grades of composite material are suitable candidates for development of tribocomponents

for the automotive industry. However, a very detailed analysis of different wear characteristics including sliding wear behavior, steel counterface wear, friction and bearing performances should be performed in future work.

The wear tests indicate that the composites may also be considered as replacements for cast iron in large volume production for brakes and driveshafts. In that case, dross particles should be previously covered by a carbon layer (produced by pyrolysis of phenolic resin) and finally nickel coated.

Table 1: Volume percentage of dross particles in squeeze cast and gas pressure infiltrated specimens of composite

Tabela 1: Volumnski delež delcev žlindre v vzorcih kompozita pridobljenih s tlačnim vlivanjem z vtiskanjem in tlačno infiltracijo

Test No.	Dross particles, vol. %	Al matrix, vol. %
SQUEEZE CAST SAMPLES		
1	23.1	76.9
2	24.6	75.4
3	22.8	77.2
Average	23.5	76.5
GAS PRESSURE INFILTRATED SAMPLES		
1	21.8	78.2
2	20.3	79.7
3	29.8	71.2
Average	20.6	79.4

Table 2: Average* hardness and micro hardness of reinforced and non-reinforced species

Tabela 2: Povprečna* trdota in povprečna mikrotreda ojačane in neojačane matrice

Specie	Brinell Hardness BHN	Vickers micro hardness, HK (in the matrix aluminum area)
Non-reinforced A356 alloy	60	73
Squeeze casting Grade	108	83
Gas pressure infiltrated grade	102	80

*Average values obtained from 5 different measurements

Table 3: Tensile properties of squeeze cast and gas pressure infiltrated composite specimens

Tabela 3: Natezna trdota pripravljenih vzorcev kompozita

Material	YS (MPa)	Ultimate Tensile Stress (MPa)	Elongation (%)
Non-reinforced A356 alloy (T6)	205	280	6
Squeeze cast grade	164	228	3.2
Gas pressure infiltrated grade	148	202	2.9

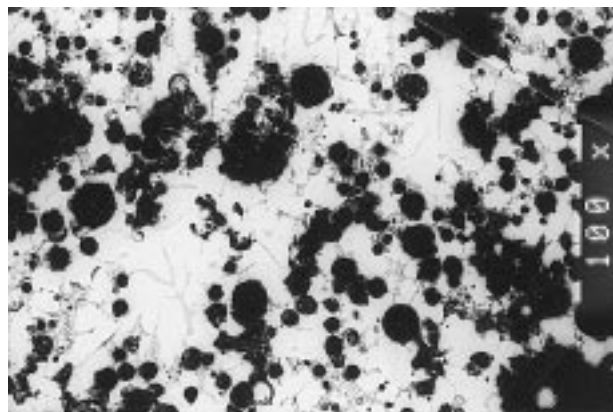


Figure 1: Photomicrograph of A356- 22.8 vol.% dross particles

Slika 1: Mikrostruktura kompozitnega materiala z 22.8 vol.% delcev žlindre

4 ECONOMICAL EVALUATIONS

The cost of a composite material can be estimated by the following formula:

$$(\$ / \text{kg})_{\text{composite}} = X_1 (\$ / \text{kg})_{\text{matrix alloy}} + X_2 (\$ / \text{kg})_{\text{reinforcement}} + (\$ / \text{kg})_{\text{production}}$$

where X_1 is the weight fraction of matrix alloy and X_2 is the weight fraction of the reinforcement in the composite.

The cost of matrix alloy is ≈ 1.4 $\$/\text{kg}$ (this is the average commercial value of pressed drained aluminum), the cost of the cyclone fraction of dross particles is ≈ 0.06 $\$/\text{kg}$ and the production cost is the cost of gas pressure infiltration or squeeze casting which is typically

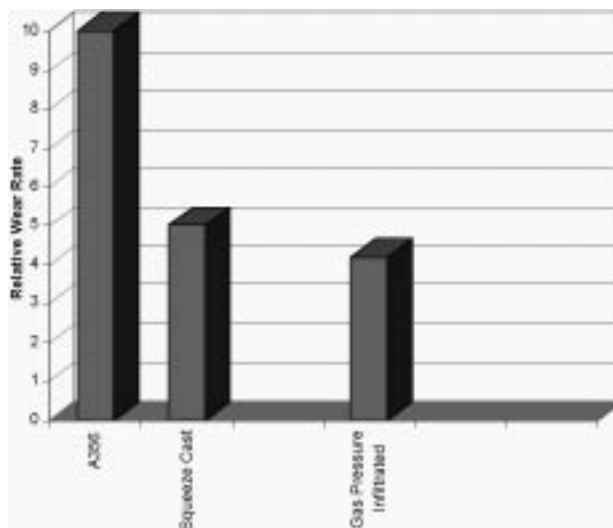


Figure 2: The comparison of relative wear rates for non-reinforced A356 alloy, squeeze cast and gas pressure infiltrated test specimens of composite material

Slika 2: Primerjava relativne obstojnosti proti abrazijski obrabi neojačane matrice in kompozitnega materiala pridobljenega s tlačnim vlivanjem z vtiskanjem ter tlačno infiltracijo

about 8.5 -12 \$/kg for an annual production volume up to 10 millions parts,¹¹.

Taking into account that $X_1 \approx X_2 = 0.5$, one can estimate that the price of 1 kg of composite material (taking into account the cost of composite constituents) is about 50% lower than the cost of non-reinforced aluminum alloy. At the same processing conditions, the cost of finished AlMMC components will be slightly lower than the cost of the same squeeze cast components made by non-reinforced aluminum alloy. Note that in the case of squeeze cast components the production cost is almost the same for non-reinforced and reinforced aluminum alloy. Further promotion of this concept must be on a value-added basis, i.e., on the improvement of the cost-to-quality ratio. On the other hand, there must be a market demand for improved properties before consumers will agree to pay for them. The automotive market-the largest potential market for MMCs-is traditionally interested in low cost articles with improved performance, and this is the significant promise for the fabrication of cast products from aluminum-dross reinforced composites. Of course, if the final consumers consider price alone, there will be little incentive for developing innovative solutions.

5 CONCLUSION

The cyclone fraction of dross particles, which is a common waste by-product in aluminum smelters, is a promising reinforcing phase for the cost effective environmentally safe discontinuously reinforced aluminum based composites.

Squeeze casting and gas pressure infiltration of packed dross powder, both of which are widely accepted processes within industry, were applied to prepare composite specimens with more than 20 vol.% of non-metallic filler.

Preliminary inspection of hardness, tensile properties and wear behavior of selected species showed that the new composite is an attractive material for tribocomponents and may be applicable for some less critical and cost effective engineered components with possible widespread application in transportation industry.

The decrease of the price of composite-caused by replacing expensive aluminum alloy by an almost cost-free ceramic filler rich in alumina - makes this material very competitive. However, a further very detailed evaluation of the ratio between cost and the improvements achieved in commercial properties of the composite should be studied before this material can be considered for industrial application.

ACKNOWLEDGEMENTS

The authors thank the Slovene Ministry of Science and Technology and IMPOL d.d. from Slovenska Bistrica for the support of this investigation.

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