JMEPEG (1998) 7:239-246

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Manufacturing and Recycling of A380Al/SiC$_{(p)}$ Composites

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(Submitted 30 June 1997; in revised form 24 November 1997)

The melting and degassing equipment and the process used in the production of particulate aluminum matrix composites is proposed. Also presented is the successful preparation of A380/80 μm SiC particulate aluminum alloy composites, where SiC particulate was evenly dispersed throughout an aluminum alloy matrix and a die cast chain wheel had very little porosity. In addition, recycling equipment for particulate aluminum matrix composites was designed. The old material and new material of these composites are mixed in ratios of 1:1, 2:1, and 3:1 by weight. After the first, second, and third recyclings, it was found that the amount of porosity and inclusion is much less for recycled composites. In addition, from composition analysis, it was found that the Si content increases with an increase in the amount of old materials and recycling times. From tensile strength tests, it was found that tensile strength and elongation are the same for new materials and composites with old material to new material weight ratios of 1:1 and 2:1 for the first to the third recyclings. However, tensile strength and elongation decrease for composites with an old material to new material weight ratio of 3:1 for the first to the third recyclings, in particular for material of the third recycling. If Si was added before the recycling process, though the formation of Al_3C_4 and Si crystal could be constrained, it would have no significant effect on the increase of elongation.

Keywords manufacturing, particulate aluminum matrix composites, recycling

1. Introduction

Aluminum alloy/SiC$_{(p)}$ composites can be prepared by using the vortex method under vacuum (Ref 1); powder metallurgy (Ref 2); compocasting (Ref 3); infiltration process (Ref 4); spray process (Ref 5); XD in-situ process, which involves heating various matrix and reinforcement precursor materials to high temperatures, such that a self-propagating exothermic reaction takes place (Ref 6); and Lanxide in-situ process (Ref 7). Two of these processes, the vortex method and compocasting, can be used for mass production of ingots and billets. These processes provide composites to downstream metal industries such as casting, forging, rolling, and extrusion to produce net-shaped products with a lower cast. The addition of silicon carbide particulate (SiC$_{(p)}$) to aluminum (Al) alloy can increase specific strength, specific stiffness, and wear resistance of the matrix. Thermal conductivity and the coefficient of thermal expansion can be modified according to the function of the products. The composites are used in aerospace and commercial applications such as brake rotor disks, bicycle frames, motorcycle sprockets, seamless automotive driveshaft tubes, chain wheels, golf club heads, and electrical equipment racks. Waste material is generated during the production of these products. For example, the manufacturing of motorcycle sprockets by die casting can generate many shot biscuits and waste materials in primary runners, branch runners, and overflow wells. Because the price of Al/SiC$_{(p)}$ composite is 2 to 3 times higher than that of substrate, if waste material were not recycled, the product cost would increase, and this material could not be applied in commercial industries. Thus, low cost recycling technology is a major bottleneck for this industry.

SiC particulates are thermodynamically unstable in a high temperature molten Al solution, and the following reaction can easily occur (Ref 8, 9):

$$3Al_{(1)} + 4SiC_{(s)} \rightarrow Al_3C_{4(s)} + 4Si_{(l)}$$

where Al_3C_4 is formed at the interface of SiC particulate and Al alloy, as SiC crystal is dissolved in molten Al solution. The formation thickness of $Al_3C_{4(s)}$ increases with an increase in reaction temperature and reaction time, and approaches saturation (Ref 10). The formation of Al_3C_4 may reduce the mechanical properties of SiC particulate and composites. Also, as the viscosity of the melt is increased and the fluidity of the molten, which composites solution, is decreased, it is disadvantageous to its die casting properties (Ref 11). To avoid formation of Al_3C_4, SiC particulate can be pretreated before being added to molten solution, by baking in a 700 to 1200 °C furnace for several hours. SiO_2 film is formed on the surface of the particles, which can enhance wetting between particles and molten solution, and formation of $Al_3C_{4(s)}$ is avoided (Ref 12). Avoiding overheating during the melting process (Ref 13) can also reduce formation of $Al_3C_{4(s)}$.

During the recycling process for waste materials of Al/SiC composites, besides reducing interface reaction, it is also necessary to carefully control porosity and inclusion of molten solution. Conventional salt fluxing cannot be used for the recycling of this material. $Cl_{2(g)}$ or nitrogen gas cannot be used for degassing because the wetting between SiC particles and the molten solution will be completely destroyed by this treatment. SiC particles may then float to the top of the molten solution, so agitation becomes impossible; thus SiC particles cannot combine with a molten Al solution. However, if impurity removal and degassing are not performed effectively, the mechanical properties of recycled composites can be greatly reduced. Therefore, this study first proposes the melting equip-

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ment and the process for producing Al/SiC$_{(p)}$ composites, and it uses the die casting method to produce bicycle parts. Also a low cost recycling method was developed for die casting waste material and for degassing using Ar, Cl_2, and N_2. Microstructure observation and property tests on recycled waste material were used to evaluate the processes. In addition, the distribution of SiC particles in Al alloy substrate and the influence on mechanical properties after recycling is discussed.

2. Experimental Process

2.1 Manufacturing of A380Al/SiC$_{(p)}$ Composites

Aluminum A380 is remelted at 700 °C in the furnace shown in Fig. 1, where a flux is added and a protective atmosphere blanketed by nitrogen or argon gas is applied in the furnace in a partially closed system. The feeding container and agitator means are mounted in or above the furnace shown in Fig. 1. The

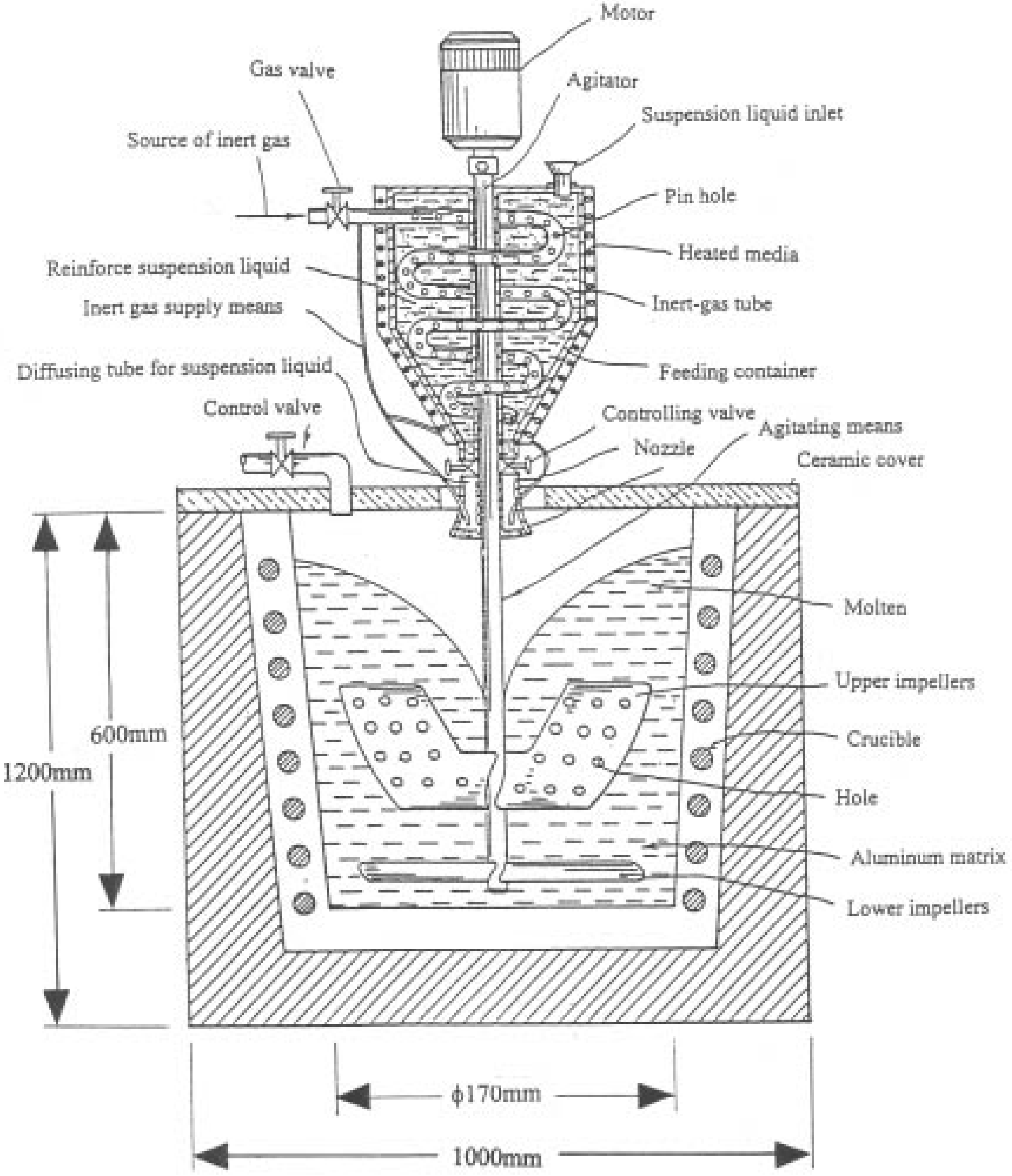


Fig. 1 A380Al/SiC$_{(p)}$ composites melting equipment diagram

driving motor drives the impellers of the agitator of 400 revolutions per minute (rpm).

A suspension liquid (with 10 wt% reinforcing material) is prepared by adding reinforcing material of ceramic particulate silicon carbide (99.9%) of β (beta) phase (average particle size 80 μm irregular shape) to distilled water in the presence of a 0.05 wt% dispersing agent, produced by R.T. Vanderbuilt Co., tradename Darven C, containing aluminum polymethacylate as a major ingredient. The suspension liquid is oscillated by ultrasonic waves for 30 min and is poured in the feeding container. By using the gas control valve to adjust the flow rate of nitrogen into the container and opening the suspension liquid control valve, the suspension liquid, suspended with the particulate, is then quantitatively sprayed through the nozzle onto the molten solution of the aluminum alloy. The rotation speed of the agitator is reduced to 80 rpm after finishing the spraying of the suspension liquid into the molten alloy. The rotation continues for 10 min. The motor is switched off, and the feeding container with the agitator is removed.

The composite thus obtained containing 10 wt% 80 μm silicon carbide is maintained at 700 °C for degassing. The degassing equipment is shown in Fig. 2. The degassing pipe is inserted into the furnace to immerse all perforations on the degassing pipe into the molten solution for two min. Cold water is directed into the heat exchanger, and the exhaust fan or vacuum pump is started in order to reach a pressure of 1.33 N/m^2 to suck gases from the melt. The degassing pipe is slowly moved in the molten solution (without stirring) to suck gasses for about 30 min to complete the degassing operation.

The degassing pipe is then removed from the furnace. The molten aluminum alloy reinforced with silicon carbide is then cast into an ingot, which is then placed in a homogenizing or air furnace at 180 °C for 24 h for homogenization. The homogenizing furnace is then cooled to room temperature. Metallurgic specimens were then cut from composites and prepared by using 100 to 1200 grid carbimet paper and polishing with 0.05 μm Al_2O_3 particulate suspension liquid. A Nikon optical microscope (OM) was used to observe the distribution of SiC particulate in matrix and the distribution and condition of porosity.

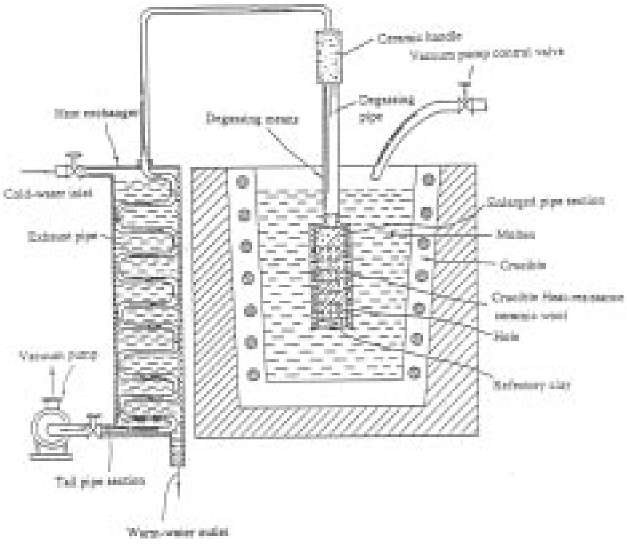


Fig. 2 A380Al/SiC$_{(p)}$ composites degassing equipment dia-

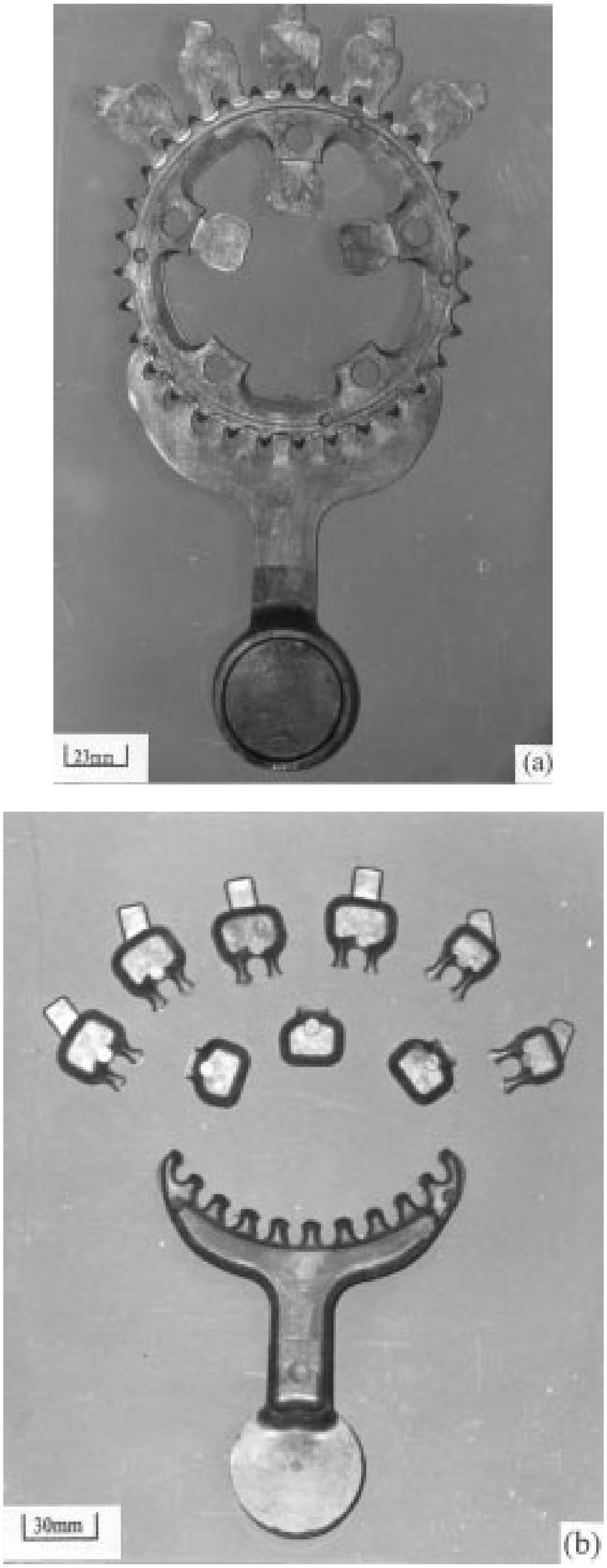
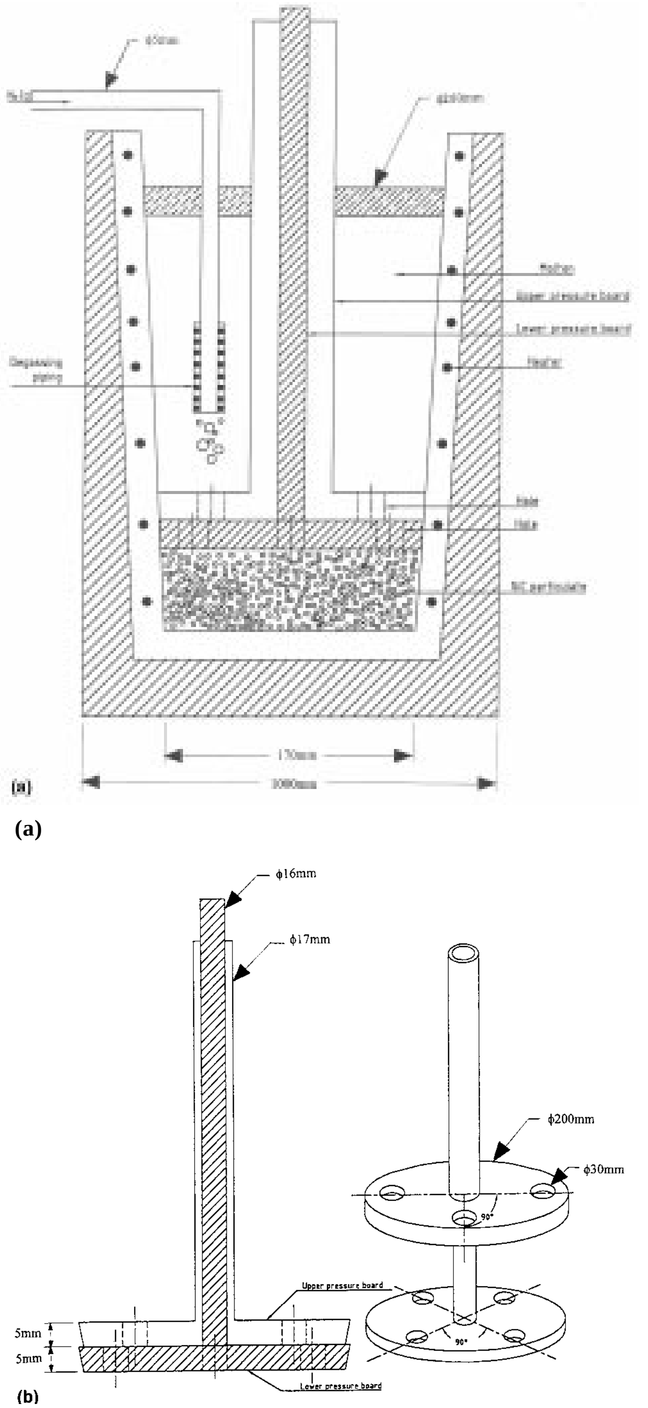


Fig. 3 (a) A380Al/SiC$_{(p)}$ composites bicycle chain wheel die casting article. (b) Shot biscuit, runner, overflow well of bicycle chain wheel die casting article

2.2 Recycling of A380Al/SiC$_{(p)}$ Composites

Bicycle chain wheel, as shown in Fig. 3(a), was prepared according to the die casting parameters as shown in Table 1 for A380Al/SiC$_{(p)}$ composites. The waste composite material was recovered from shot biscuits, runners, and overflow wells shown in Fig. 3(b). The SiC particulate density (3120 kg/cm^3) is greater than that of Al alloy molten solution (2780 kg/m^3). After the composite molten solution is held for a period of time, SiC particulate settle at the bottom of the crucible to form a layer with high concentration SiC particulate. In addition, a set of upper and lower press plates are shown in Fig. 4. The press plates are made up of graphite with four holes on each plate. The holes of the upper and lower plates are crossed at 90°, so that eight holes can be sealed when the upper and lower plates contact each other. At the beginning of recycling, the lower plate is preheated and lowered slowly. Aluminum alloy molten solution flows through four holes without disturbing SiC particulate settled at the bottom of the crucible until the lower plate touches the high concentration SiC particulate accumulation layer. The upper plate, which has been preheated, is lowered until it contacts the lower plate. The holes on the upper plate and lower plate cross each other, so eight holes are sealed. The material on the top of the upper plate is Al alloy molten solution, which will not disturb the SiC particulate accumulation layer under the lower plate. Then a conventional degassing occurs on the top of the upper plate. The upper and lower plates are removed from the molten solution after it has been cleaned, and the inclusion and oxidation film on the molten solution surface are removed. The agitation system is then introduced to a suitable position in molten solution, and graphite impellers are rotated to get convection flow of the SiC particulate accumulation layer and the molten solution to mix them homogeneously. During the agitation process, Ar gas is used to protect the upper molten solution.



(a)

(b)

Fig. 4 (a) A380Al/SiC$_{(p)}$ composites waste material recycle equipment diagram. (b) Upper and lower plate diagram

The waste material recycling process is used to implement first, second, and third recyclings of mixed material with new material and waste material at 1:1, 1:2, and 1:3 weight ratios, where new material means 380Al/10wt% SiC$_{(p)}$ composite that is melted the first time without any die casting operation. Waste material is obtained from die casting shown in Fig. 3(b). The third recycling means the waste material has been through the die casting operation three times and then mixed with new material, where SiC particulates are 99.7% of β-phase with an average particle size of 80 μm irregular shape. During recycling, new material is melted in a 700 °C furnace, inclusion and gas are removed from the molten solution, and a suitable amount of waste material is then added. An alkaline cleaning water soluble sequestering agent (Cillolin-GD200; Schafer, Chemische Fabrik, GmbH) is used in the die casting operation before adding waste material. It is dried at 150 °C. The waste material has to be immersed into the new material molten solution completely to avoid high oxidation before melting. In addition, 2 l/min dry Ar gas is introduced at the top of the molten solution for protection during the melting process. After the

Table 1 Die casting parameter for A380Al/SiC$_{(p)}$ composites bicycle chain wheel

Die casting machine	350 tons
Cavity	1
Mold temperature	200 ± 3 °C
Thickness of gate	1.7 mm
Velocity of gate	42.15 m/s
Diameter of plunger	57.15 mm
Velocity of plunger	2.6 m/s
Filler time	0.056 s
Al melting temperature	680 ± 5 °C

waste material is melted, it is held for 34 min to ensure SiC particulates settle at the bottom of the crucible. To determine settling time, H_2O and SiC particulates are mixed at the same volume of the above composites in a transparent vessel, and 10 min is added for settling to be complete and the agitation to be stopped. The assumptions of this simulation experiment include the temperature being 700 °C and the viscosity of the Al alloy molten solution being the same as H_2O.

The graphite upper and lower plates are preheated to 200 °C. The lower plate is pressed slowly down to the molten solution. When the lower plate touches the SiC particulate accumulation layer, the upper plate is pressed slowly down to the molten solution. The inclusion is removed from the surface of the molten solution, and part of the ALCU-AL SM8 inclusion removal agent (ALCU-AL SM8, Schafer, Chemische Fabrik, GmbH, D-53762 Hennef/Sieg) is added without strong agitation. Next, spare ALCU-AL SM8 is slowly added and pressed into the molten solution. The amount of ALCU-AL SM8 is 0.3 wt% of the Al alloy molten solution. After the inclusion is removed, Ar gas is introduced through a stainless steel degassing pipe with many ϕ1 mm holes, as shown in Fig. 2. Then the degassing operation is implemented. The degassing pipe is freely moved in the Al alloy molten solution to enhance the degassing effect. Plasma coated ZrO_2 is used to protect the outer surface of the stainless steel pipe. The composite molten solution is held 15 min after degassing. The upper and lower plates are removed from the molten solution and the oxidation film, and inclusions are removed from the molten solution surface. The graphite agitation shaft and impeller are put into the molten solution to agitate it under the protection of Ar gas. The rotation speed is 80 rpm to ensure that the SiC particulate settled at the bottom of the crucible is suspended evenly in molten solution. This composites molten solution is then poured into a permanent mold (preheated to 350 ± 10 °C) of tensile specimen. New material to waste material is mixed to 1:1, 1:2, and 1:3 weight ratios, and a fluorescent spectrophotometer is used to analyze the composition of the first, second, and third recycling material. Carbimet paper, 100 to 1200 grid, is used to polish all specimens; then they are fine polished with 0.3 μm Al_2O_3 particle suspended solution. A Nikon optical microscope (OM) is used to observe the distribution of SiC particulate, porosity, and inclusion throughout the matrix.

The recycling material is fabricated into tensile specimens, and the dimensions are shown in Fig. 5. T5 heat treatment is applied, the heat treatment involves heating the material to 220 ± 3 °C, holding it for 5 h, and then cooling it to room temperature. Three tensile specimens are put into an universal tensile machine (Shimadzu Ag-1000A) to do a tensile test under 5 mm/min crosshead speed and room temperature to get tensile properties. Data from all three are taken to determine an average value for each tensile property.

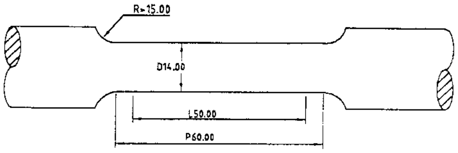


Fig. 5 Specimen used for tensile tests. All dimensions are in mm.

A scanning electron microscope (SEM) is used to observe the morphology of the tensile fracture surface. Before the observation, the specimen is put in a supersonic acetone to clean the surface, and then it is goldplated for 1.5 min after drying.

3. Results and Discussion

3.1 *The Distribution of SiC Particulate in Al Alloy Matrix*

Figure 6(a) shows the distribution of SiC particulate in Al alloy matrix for a gravity casting tensile specimen. The micrograph in Fig. 6(a) shows that SiC particulate can be evenly dispersed through 380Al alloy matrix by using the melting

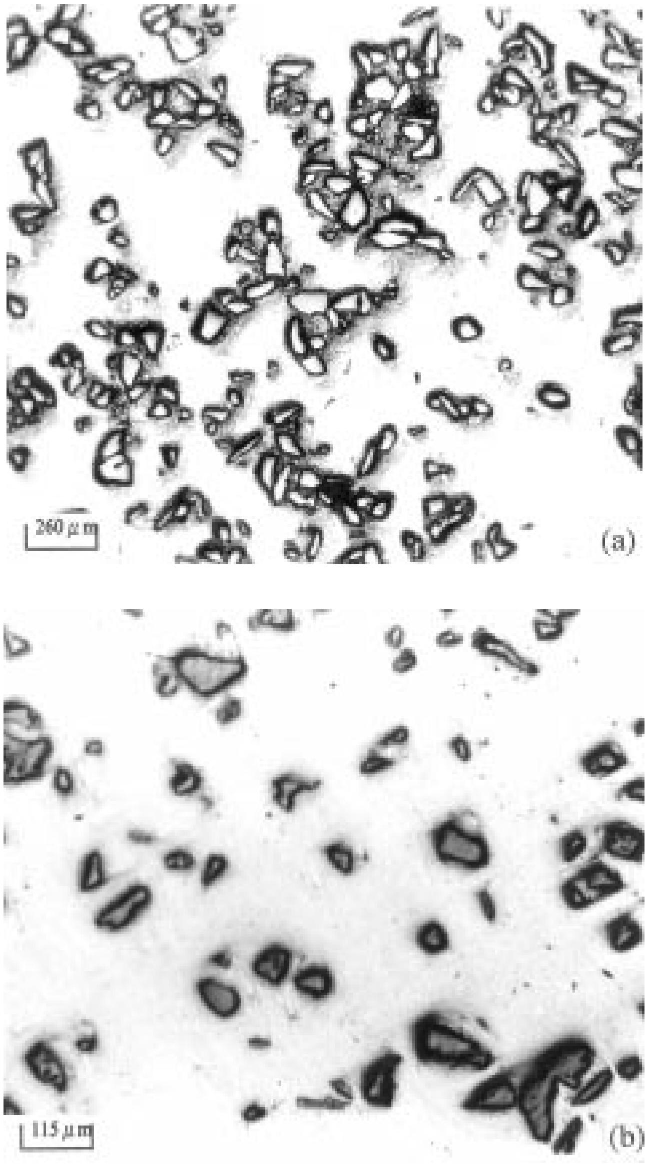


Fig. 6 Optical micrograph for the distribution of SiC particulate in A380Al alloy matrix (a) gravity casting and (b) die casting

equipment and the process proposed in this study. This dispersion occurs because when the homogeneous SiC particulate suspension liquid is to be dispersed or sprayed onto the surface of high temperature Al alloy solution, the distilled water in the suspension liquid will be splashed to the surface of the molten Al alloy due to the very high temperature difference between the high temperature Al alloy solution and the suspension liquid. This promotes the dispersion of this strengthening phase. The strengthening phase forms an evenly dispersed strengthening film on the surface of Al alloy molten solution due to the speedy evaporation of distilled water. This dispersed strengthening phase film can mix evenly with the Al alloy molten solution by the convection force produced by mixing. After casting solidification, the strengthening phase can be dispersed evenly through the Al alloy matrix.

In addition, very little of porosity is found in the matrix when the degassing equipment and the process proposed in this study are used. The degassing theory states that the porous degassing hollow pipe is inserted into the molten solution to form a closed space, and gas is then sucked by a vacuum pump. A low pressure area is produced in the closed space. When the moving degassing pipe meets gases or bubbles in the molten solution, these high pressure bubbles are sucked into the degassing pipe and are removed by the pressure difference. This produces a good degassing effect. It is found that SiC particulate can be evenly dispersed through an Al alloy matrix to get particulate aluminum alloy matrix composites with less porosity by using the melting and degassing equipment described. In addition, the microscopic structure of die castings shown in Fig. 6(b) shows the distribution of SiC particulate to be greater in the matrix than in the gravity tensile castings. Because the cooling rate of die casting is quicker than that of gravity casting and the growth of dendrite is inhibited in time, SiC particulate is entrapped and evenly distributed.

3.2 The Properties of Recycled A380Al/SiC$_{(p)}$ Composites

The waste material used comes from A380Al/SiC$_{(p)}$ composites bicycle chain wheel die casting shot biscuits, runners, and overflow wells where overflow well waste material contains a high amount of porosity. There is a thicker oxidation film, sequestering agent, and inclusion. If this oxidation film, sequestering agent, and inclusion cannot be removed effectively during the recycling process, then the recycling die casting will contain a high amount of porosity and inclusion as shown in the micrograph of Fig. 7. The recycling equipment

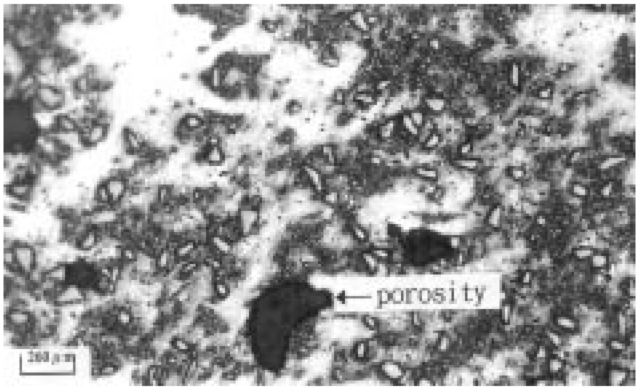


Fig. 7 Optical micrograph for waste recycle of A380Al/SiC$_{(p)}$ composites bicycle chain wheel

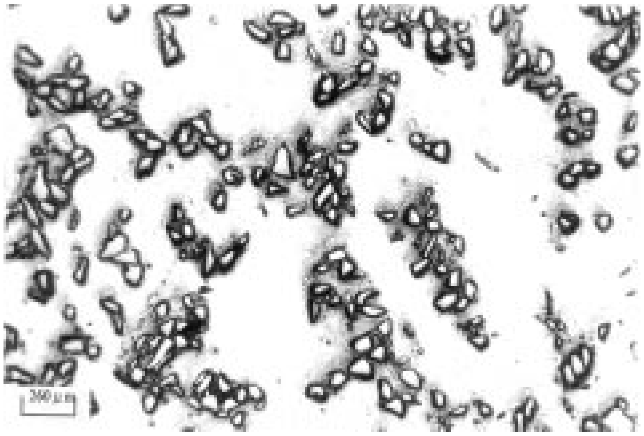


Fig. 9 Micrograph for A380Al/SiC$_{(p)}$ composites. Old material to new material of 3:1 weight ratio after third recycling with addition of Al-Si alloy

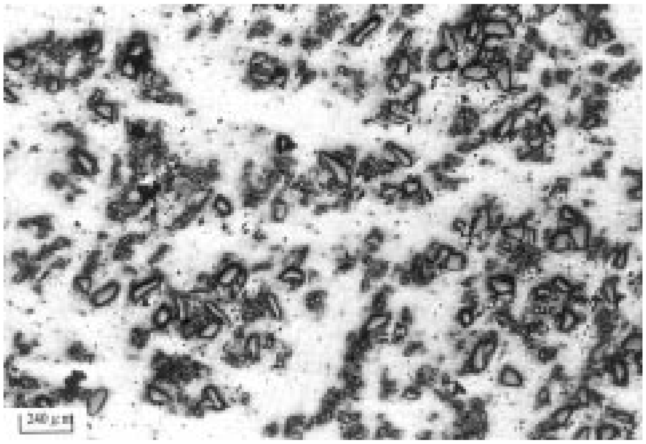


Fig. 8 Micrograph for A380Al/SiC$_{(p)}$ composites. Old material to new material of 3:1 weight ratio after third recycling

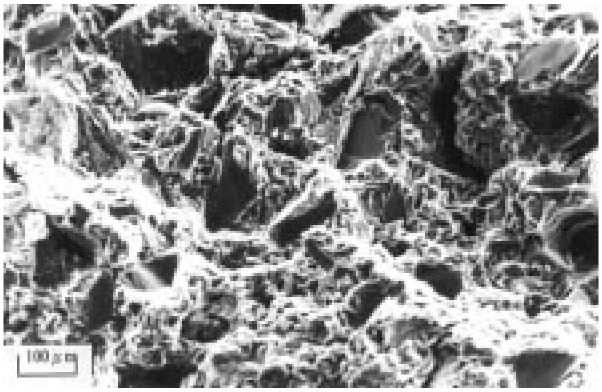


Fig. 10 Tensile fracture surface of A380Al/SiC$_{(p)}$ composites. Old material to new material of 3:1 weight ratio after third recycling

and process proposed are used to mix old material and new material at 1:1, 2:1, and 3:1 weight ratios, and the composition after first, second, and third recycling is shown in Tables 2(a) to (c). It is shown in the tables that the Si content increases significantly with increase in recycling times, while the Mg content decreases a little, and the other ingredients have changed very little. Increase of Si is more significant with a 3:1 weight ratio and third recycling material. In the meantime, it is found that the Si content of recycling material is greater than that of the original material. Figure 8 shows the microscopic structure of a 3:1 weight ratio and third recycling material. It contains less porosity and inclusion than those of Fig. 7. Therefore, use of the degassing equipment designed in this study can effectively decrease porosity and inclusion of recycling material. During the recycling process, the reaction of SiC particulate and Al molten solution occurred even when the temperature is controlled at 700 °C. In addition, Si crystal created near SiC particulate is shown in Fig. 8. This is due to the SiC particulate being thermodynamically unstable in a high temperature Al molten solution, where an Al_3C_4 interface layer and SiC crystal is easily formed, and supersaturated Si crystal is formed near the SiC particulate. The increase of SiC crystal content is also shown in Table 2. If Al-Si alloy is added to the third recycling process to increase Si content to 9.0 wt%, then the reaction is inhibited to reduce the formation of Si crystal, as shown in Fig. 9. This is because Si is added to reverse the reaction and reduce the formation of Al_3C_4 (Ref. 13).

The tensile properties of $380Al/SiC_{(p)}$ composites with old and new material at 1:1, 2:1, and 3:1 weight ratios after first, second, and third recycling are shown in Tables 3(a) to (c). Tables 3(a) and (b) show that the mechanical properties of old and new material weight ratios 1:1 and 2:1, after first, second, and third recycling are the same as those of new material. Table 3(c) shows that the tensile strength of old and new material weight ratio 3:1 after first, second, and third recycling is less than that of new material, while the elongation is much less, and worst of all, for third recycling material. The amount of Al_3C_4 interface reaction layer and Si crystal is increased with recycling times and an increase in the amount of old material. Also, these products will have reduced mechanical properties (Ref 11). If Al-Si alloy is added to old and new material weight ratio 3:1 after the third recycling material to increase Si content to 9.0 wt%, then its yield strength and tensile strength are increased to the same level as those of first and second recycling material.

Elongation is still lower. Though the addition of Al-Si alloy can inhibit an Al and SiC reaction, to avoid the formation of Al_3C_4 and Si crystal and keep the fluidity at the same level, the addition of Al-Si alloy will increase the Si content to reduce the ductility of Al alloy matrix. The fracture surface morphology shown in Fig. 10 shows that brittle fracture occurs. By using the waste material recycling equipment and the proposed process, one can recycle $A380Al/SiC_{(p)}$ composites bicycle chain wheel waste material for first and second recycling to get suitable strength and ductility.

4. Conclusions

This study proposes the melting and degassing equipment for particulate aluminum alloy matrix composites, and

Table 2(a) Composition analysis for recycled $A380Al/SiC_{(p)}$ composites
Old material to new material of weight ratio 1:1

	Alloying element content wt%							
Material	**Si**	**Fe**	**Cu**	**Mn**	**Mg**	**Zn**	**All other elements**	**Al**
New	8.63	0.68	0.1	0.05	0.57	0.05	0.2 max	Remainder
Recycle No. 1	8.64	0.70	0.1	0.05	0.57	0.05	0.2 max	Remainder
Recycle No. 2	8.64	0.71	0.1	0.04	0.56	0.05	0.2 max	Remainder
Recycle No. 3	8.67	0.70	0.1	0.05	0.56	0.05	0.2 max	Remainder

Table 2(b) Composition analysis for recycled $A380Al/SiC_{(p)}$ composites
Old material to new material of weight ratio 2:1

	Alloying element content wt%							
Material	**Si**	**Fe**	**Cu**	**Mn**	**Mg**	**Zn**	**All other elements**	**Al**
New	8.63	0.68	0.1	0.05	0.57	0.05	0.2 max	Remainder
Recycle No. 1	8.65	0.69	0.1	0.05	0.56	0.05	0.2 max	Remainder
Recycle No. 2	8.68	0.70	0.1	0.05	0.54	0.05	0.2 max	Remainder
Recycle No. 3	8.70	0.71	0.1	0.04	0.52	0.05	0.2 max	Remainder

Table 2(c) Composition analysis for recycled $A380Al/SiC_{(p)}$ composites
Old material to new material of weight ratio 3:1

	Alloying element content wt%							
Material	**Si**	**Fe**	**Cu**	**Mn**	**Mg**	**Zn**	**All other elements**	**Al**
New	8.63	0.68	0.1	0.05	0.57	0.05	0.2 max	Remainder
Recycle No. 1	8.68	0.70	0.1	0.05	0.53	0.05	0.2 max	Remainder
Recycle No. 2	8.70	0.73	0.1	0.04	0.52	0.05	0.2 max	Remainder
Recycle No. 3	8.90	0.73	0.1	0.05	0.50	0.05	0.2 max	Remainder

Table 3(a) Tensile properties for recycled A380/SiC$_{(p)}$ composites

Old material to new material of weight ratio 1:1

	Tensile properties		
Material system	**Yield stress, 1 kgf/mm^2**	**Ultimate tensile stress, kgf/mm^2**	**Elongation, %**
New	14.42	26.57	3.0
Recycle No. 1	14.57	26.43	2.7
Recycle No. 2	14.45	26.50	3.0
Recycle No. 3	14.21	25.47	3.0

Table 3(b) Tensile properties for recycled A380/SiC$_{(p)}$ composites

Old material to new material of weight ratio 2:1

	Tensile properties		
Material system	**Yield stress, 1 kgf/mm^2**	**Ultimate tensile stress, kgf/mm^2**	**Elongation, %**
New	14.42	26.57	3.0
Recycle No. 1	14.31	26.52	2.8
Recycle No. 2	13.79	25.86	2.6
Recycle No. 3	14.28	26.31	2.6

Table 3(c) Tensile properties for recycled A380/SiC$_{(p)}$ composites

Old material to new material of weight ratio 3:1

	Tensile properties		
Material system	**Yield stress, 1 kgf/mm^2**	**Ultimate tensile stress, kgf/mm^2**	**Elongation, %**
New	14.42	26.57	3.0
Recycle No. 1	12.17	23.42	1.9
Recycle No. 2	12.76	24.13	1.9
Recycle No. 3	11.83	21.76	1.4
Recycle No. 3 with addition of Al-Si Alloy	12.44	23.78	1.3

A380Al/SiC particulate composites have been prepared, where 80 μm SiC particulates were evenly dispersed throughout the aluminum alloy matrix and the die casting article had very low porosity. In addition, this composite is die cast into bicycle chain wheel. Die casting old material and new material is recycled in weight ratios of 1:1, 2:1, and 3:1 with first, second, and third recycling. Very little porosity and inclusion was found in recycling composites. The conclusions for properties are as follows:

- In recycling material, Si content increases with recycling times and amount of old material.
- For old material to new material with weight ratios of 1:1 and 2:1, after first to third recycling, the tensile strength and elongation are the same as those of new material.
- For old material to new material with a weight ratio of 3:1, after first to third recycling, the tensile strength is slightly reduced, while elongation is significantly reduced compared to new material, especially for the third recycling.
- During the recycling process, though Si content is increased to 9 wt% to decrease the formation of Al_3C_4 and Si crystal, it does not increase elongation.

Acknowledgment

This work was supported by the National Science Council, Taiwan, Republic of China.

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