Separation of PRMMC into Matrix Alloy and Reinforcements by Nozzle Filtering Method

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Abstract

The SiC_P/Al-4mass%Cu alloy composites fabricated by a low pressure infiltration process (LPI process) were remelted and separated by nozzle filtering method. In the separation process, the PRMMC specimen was placed in a container with a small nozzle at the bottom. The molten PRMMC was forced to flow out through the nozzle by applying a certain pressure of Ar gas. Most of the molten matrix alloy flowed out through the nozzle and the remainder in the container consisted of SiC particles and a part of the matrix alloy. The particle volume fraction of the remainder was higher than that of the original PRMMC and the remainder would work as a filter to separate SiC particles from the matrix alloy melt. When nozzle tip angle was ranged from 60 to 120 degree, about 80% of matrix alloy in the PRMMC was separated and

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few SiC particles were observed in the separated matrix alloy. The surface of recovered SiC particles became slightly roughened due to the reaction with the molten matrix during the separation process. However, this is not expected to affect their reuse.

Keywords: Metal matrix composite, Recycling, Separation technique, Filtering

1. Introduction

In recent years, the application of MMCs which have some excellent physical and mechanical properties, has been extended to various fields, especially to automotive parts to improve fuel efficiency and performance of the automobiles. With increasing the applications of MMCs into the automobile parts, the amount of waste MMCs is expected to increase in near future. In addition, as the global social trend, it is required to build a recycling-based society to save natural resources. In Japan, for example, it has been aimed to improve the recycling rate of waste automobile parts (about 75% now) up to 95% by 2015. To improve the recycling rate of waste automobile parts, it is necessary to develop and improve the recycle techniques because various kinds of materials are used such as metals, plastics and ceramics. There are two methods for recycling of waste MMCs, that is, one is the remelting for reuse and the other is the separation into reinforcement and matrix alloy. The separation is more desirable for the recycler because the separated matrix alloys and reinforcements will be easy to be reused and, especially, the reinforcements are generally expensive. Some physical and mechanical separation processes have been proposed (Nishida, 2001). However, the efficiency of these separation techniques is not satisfactory. In our earlier works, the present authors have found that most of matrix alloy was separated from PRMMCs in the experiments of fused spinning deposition process (FSD process) (Oishi et al., 2004; Oishi et al., 2004, Ohishi et al, 2004; Mizumoto et al., 2007) and have termed this separation method “nozzle filtering method”. In this work, to establish the nozzle filtering method as the recycling process for MMCs, the optimum separation conditions and the effect of the separation process on the separated matrix alloy and reinforcements were investigated.

2. Experimental Procedure

In this work, PRMMC specimens for the separation experiments were fabricated by LPI process (Mizumoto et al, 2002; Mizumoto et al., 2004; Mizumoto et al., 2004; Mizumoto et al., 2005). The Al-4mass% Cu alloys were used as matrix and SiC particles (particle size: 75μm) were used as reinforcement particles. The volume fraction of the reinforcement particles in the PRMMC specimens was 20vol%. Then the PRMMC specimens were separated by nozzle filtering method. **Figure 1** shows the schematic illustration of the apparatus for the nozzle filtering method. The PRMMC specimen about 10g was set in the container with a small nozzle at the bottom. In order to investigate the effect of reactivity of the container material with the molten matrix alloy on the separation process, the containers were made of silica which was reactive to the molten aluminum alloy, or KC carbon ceramic which was unreactive. Mizumoto et al. (2007) have reported an optimum nozzle size for nozzle filtering method and the nozzle size was 0.75mm in diameter. The PRMMC specimen was remelted at 1273K by H.F induction heating when silica tube was used as the container material. When KC carbon ceramic was used, the PRMMC specimen was remelted by electric furnace. Then the molten PRMMC specimen was forced to flow out through the nozzle by applying a certain pressure of Ar gas on the molten PRMMC specimen. The microstructures of the matrix alloy flowed out through the nozzle and the remainder in the container were observed by optical microscope. The chemical composition of the separated matrix alloy was examined using SEM-EDX. To recover the SiC particles from the remainders, the remainders were disintegrated by pickling in hydrochloric acid ($HCl:H_2O=1:3$). The surface morphology of the recovered SiC particles was observed using SEM.

3. Results and Discussion

3.1. Behavior of reinforcement particles during separation process

Figure 2 shows the microstructures of 20vol% SiC particle reinforced Al-Cu alloy composite before separation experiment and the remainder in the container after separation experiments. The volume fraction of SiC particles in the remainder seems to be higher in comparison with that in the PRMMC specimen before separation. The area fraction of SiC particles, assumed to be equal to the volume fraction in this work, on the different locations in the remainder was measured by image analysis. The relationship between the particle volume fraction and the distance from the bottom of the container is shown in **Fig. 3**. The particle volume fraction in the remainder was higher than that in the PRMMC specimen (about 20%) and increased gradually towards the nozzle. This indicates that SiC particles move downwards with a flow of the molten matrix alloy and are aggregated near the bottom of the container, resulting in the formation of a loose skeleton structure. Wang et. al. (2003) have reported that the reinforcement particles in PRMMC (5 vol% Al_2O_3/Zn-Al alloy composite) increased the viscosity of molten PRMMC significantly. In the present work, the particle volume fraction in the remainder became higher with approaching the nozzle, resulting in a drastic increase of the viscosity of the molten PRMMC at the bottom part. This increase of the viscosity would make the skeleton structure remain near the nozzle. In addition, the skeleton structure would work as a filter to separate SiC particles from the matrix alloy melt.

3.2. Effect of nozzle shape on the separation process

It is expected that the nozzle shape will influence the movement of the reinforcement particles, that is, the flow of the molten PRMMC, during separation process. The separation experiments using the container with various nozzle shapes were carried out. The effect of applied pressure on nozzle filtering method has been reported by Mizumoto et al. (2007) and the applied pressure in this work was 0.25 MPa. Schematic illustrations of nozzle shapes are shown in **Fig. 4**. Nozzle tip angles were varied from 30 to 180 degree. To evaluate the effect of nozzle shape on the separation process quantitatively, the recovery of the matrix alloy was introduced. The recovery was defined by the ratio of the weight of separated matrix alloy to the weight of matrix alloy in PRMMC before separation. **Figure 5** shows the relationship between the recovery and the nozzle tip angle. The highest recovery, 90%, was obtained when the nozzle tip angle was 30 degree (nozzle E). When the nozzle tip angle ranged from 60 to 120 degree (nozzle B-D), the recovery was almost constant, about 80%. The lowest recovery was obtained when the nozzle tip angle was 180 degree (nozzle A). The particle area fractions in the separated matrix alloy measured by image analysis are plotted in **Fig. 6**. It is noteworthy that when the nozzle tip angle was 180 degree, many SiC particles were observed in the separated matrix alloy. This result suggests that the skeleton structure, which works as a filter, would not be formed without the slope of the nozzle part. When nozzle tip angle ranged from 60 to 120 degree, lower SiC particle volume fractions were observed. On the other hand, when the nozzle tip angle was 30 degree, the highest SiC particle volume fraction was obtained. It is considered that the skeleton structure would be formed so slowly due to a smaller slope in the nozzle that the SiC particles would flow out together with the melt through the nozzle before the formation of skeleton structure. It is, therefore, found that the slope of the nozzle part is important for the formation of the skeleton structure and the optimum nozzle tip angle ranges from 60 to 120 degree.

3.3. Effect of the separation process on the separated matrix alloy and the reinforcement particles

In order to investigate the effect of container material on the separated matrix alloy, separation experiment was carried out using the silica container and KC carbon ceramic container. When the silica container was used, the separated matrix alloy contained 8 wt% silicon. This silicon content is considered to be derived from the reaction between silica container and molten matrix alloy during separation process. On the other hand, when the KC carbon ceramic container was used, no change was observed in the chemical composition of the separated matrix alloy. In addition, the KC carbon ceramic container was able to be machined easily and to be used repeatedly. Thus it is suggested that the KC carbon ceramic will be promising as container material in nozzle filtering method.

Figure 7 shows SEM images of as received and recovered SiC particles. Compared with SiC particles as received, the surface of recovered SiC particles seems to be slightly rough and would be covered with aluminum carbide (Al_4C_3). It has been reported that Al-Cu alloy melt reacts with SiC particles, resulting in a degradation of the surface of SiC particles (Mizumoto et al., 2004). Tham et al. (2001) have reported that a strong interface bonding results from the tendency for the Al_4C_3 reaction layer to form a semi-coherent interface with an aluminum matrix. Thus the recovered SiC particles are expected to be reused and exhibit a strong bonding with matrix in Al-based PRMMC.

4. Conclusions

1. The matrix alloy was separated from PRMMC specimens by nozzle filtering method. It is considered that a skeleton structure would be formed near the nozzle part as a result of an aggregation of reinforcement particles, which acts as a filter to separate reinforcement particles from PRMMC specimens.

2. The slope of the nozzle part enhances the aggregation of reinforcement particles in the flow of the molten PRMMC and be an important factor for the formation of the skeleton structure near the nozzle. It is suggested that the optimum nozzle tip angle ranges from 60 to 120 degree.

3. The separated matrix alloy contained 8 wt% silicon when a silica container was used. The silicon is considered to result from the reaction between molten Al-Cu alloy and silica tube during separation process. On the other hand, no change in the chemical composition of the separated matrix alloy was observed when KC carbon ceramic container was used. Thus KC carbon ceramic will be promising as a container material in nozzle filtering method.

4. The surface of SiC particles recovered from the remainder in the container was slightly degraded due to the reaction between molten matrix alloy and SiC particles during separation process. However, the degree of degradation did not appear enough to compromise the feasibility of particle recycling.

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References

Nishida, Y., 2001. Introduction to Metal Matrix Composites, Corona Publishing Corp., Tokyo, pp.154-162.

Ohishi, K., Morimoto, T., Mizumoto, M., Kagawa, A., 2004. Shape Control of Cylindrical and Rectangular Shape Solid Parts Fabricated by Fused Spinning Deposition Method. J. JFS. 76, 582-587.

Ohishi, K., Adachi, S., Mizumoto, M., Kagawa, A., 2004. Shape Control of Cylindrical and Rectangular Shape Solid Parts Fabricated by Fused Spinning Deposition Method. J. JFS. 76, 915-920.

Mizumoto, M., Ohgai, T., Kagawa, A., 2007. Novel Separation Technique of Particle Reinforced Metal Matrix Composites by Fused Deposition Method. Materials Science

Forum. 539-543, 1028-1032.

Mizumoto, M., Murano, T., Kagawa, A., 2002. Microstructure Control of Particle Reinforced Metal Matrix Composites Fabricated by Low-Pressure Infiltration Process. Materials Transactions, JIM. 43, 2629-2634.

Mizumoto, M., Tajima, Y., Kagawa, A., 2004. Thermal Expansion Behavior of SiC_P/Aluminum Alloy Composites Fabricated by a Low-Pressure Infiltration Process. Materials Transactions, JIM. 45, 1769-1773.

Mizumoto, M., Kaneko, Y., Kagawa, A., 2004. Microstructure Control of Fiber Reinforced Metal Matrix Composites Fabricated by Low Pressure Infiltration Process. J. Japan Inst. Metals. 68, 1047-1052.

Mizumoto, M., Ezaki, H., Kagawa, A., 2005. Fiber Orientation Control and Evaluation of Characteristics of Short Fiber Reinforced Metal Matrix Composite Fabricated by Low-pressure Infiltration Process. Material Science Forum. 475-479, 2559-2562.

Wang, J., Guo, Q., Nishio, M., 2003. The apparent viscosity of fine particle reinforced composite melt. J. Mater. Process. Technol. 136, 60-63.

Tham, L.M., Gupta, M., Cheng, L., 2001. Effect of Limited Matrix–Reinforcement Interfacial Reaction on Enhancing the Mechanical Properties of Aluminum–Silicon Carbide Ccomposites. Acta mater. 49, 3243-3253.

Figure captions

Fig. 1 Schematic illustration of the apparatus for nozzle filtering method.

Fig. 2 Microstructures of (a) 20vol% SiC_P/Al-Cu alloy composites before separation experiment and (b) the remainder in the container after separation experiment.

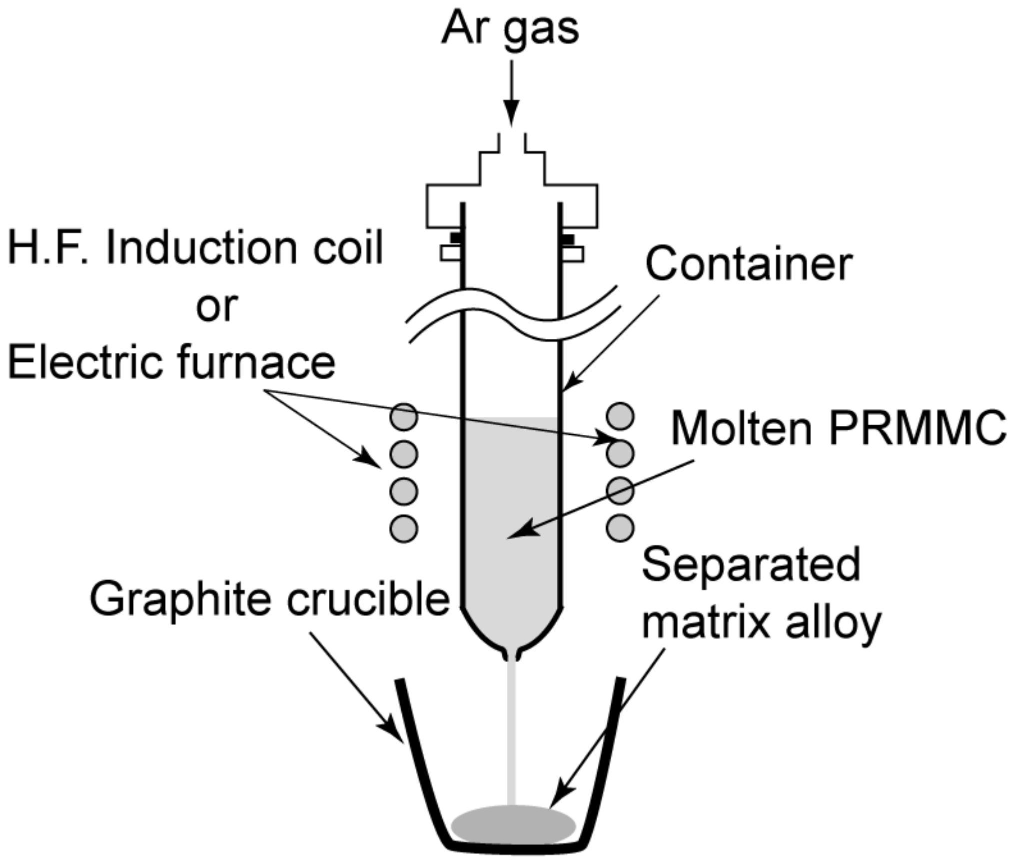
Fig. 3 Relationship between the particle volume fraction in the remainder and the distance from the bottom of the container.

Fig. 4 Schematic illustrations of nozzle shapes. Nozzle tip angles were varied from 30 to 180 degree.

Fig. 5 Relationship between recovery and nozzle tip angle.

Fig. 6 Relationship between particle volume fraction in the separated matrix alloy and nozzle tip angle.

Fig. 7 SEM images of SiC particles (a) as received and (b) after separation experiment.



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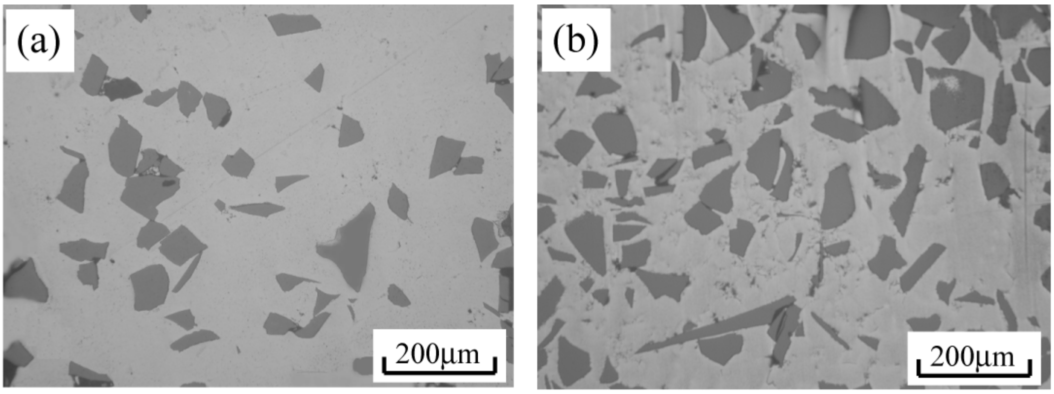

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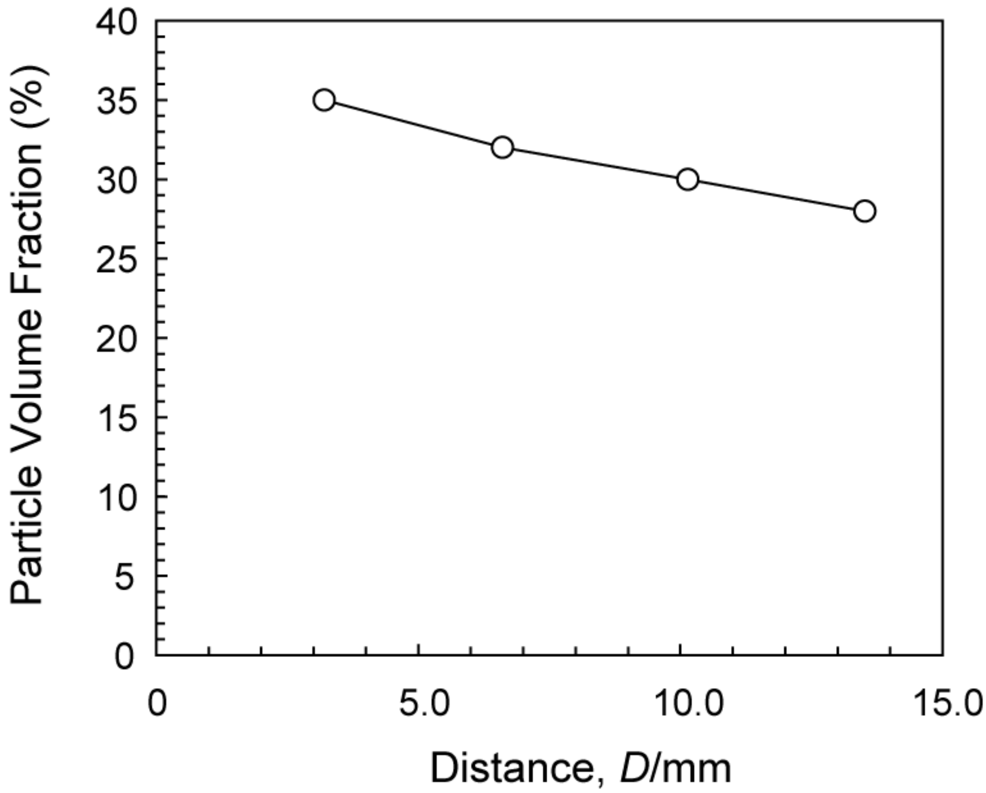

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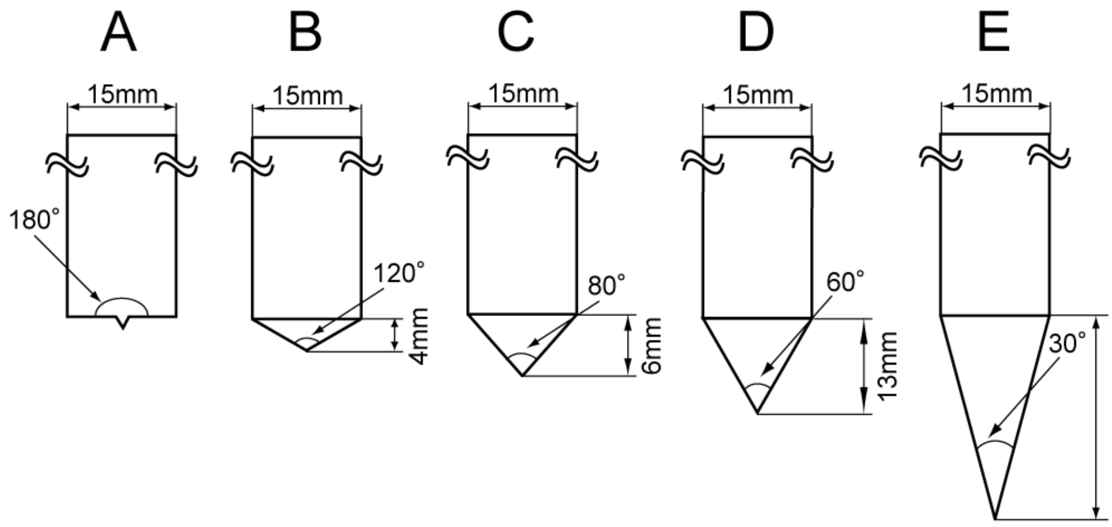

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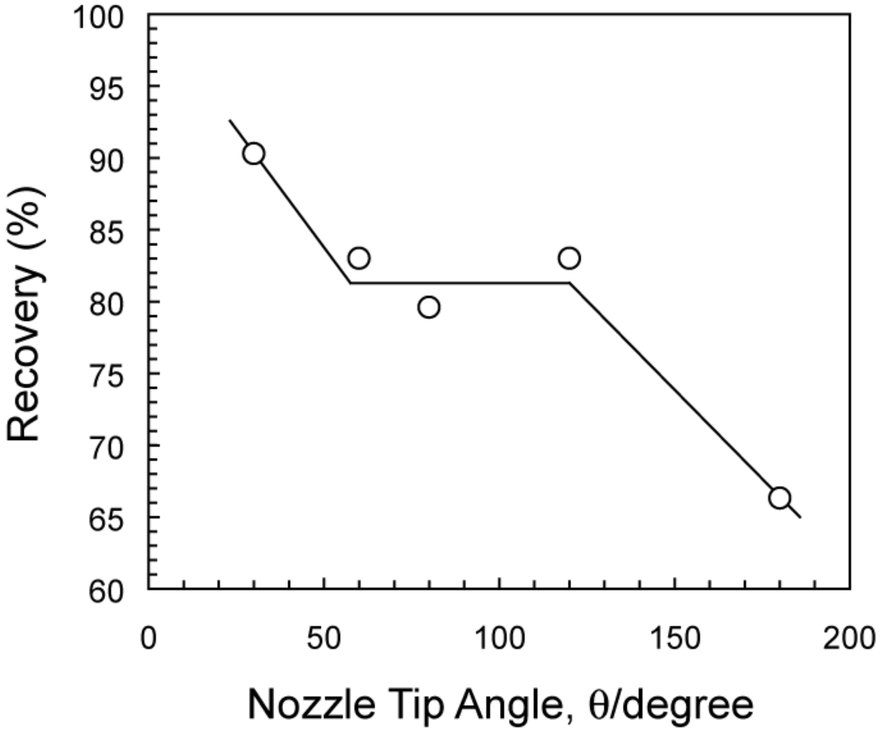

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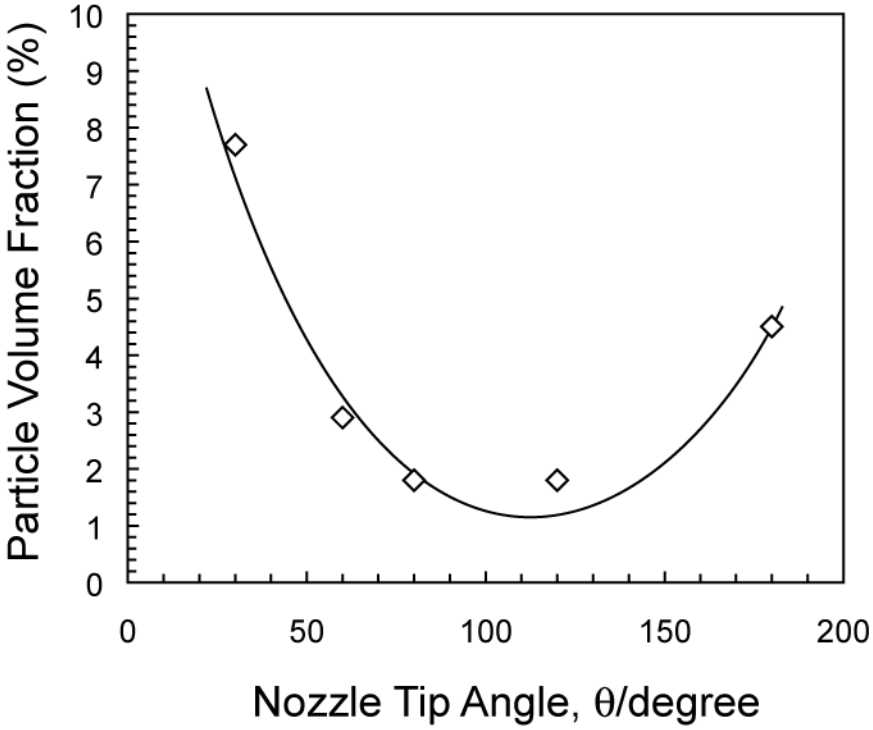
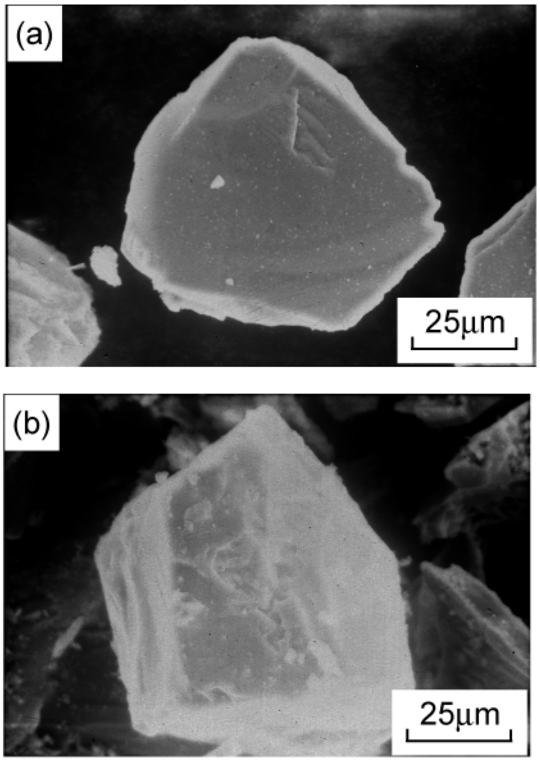


Fig. 6 Relationship between particle volume fraction in the separated matrix alloy and nozzle tip angle.



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