

FABRICATION PROCESS AND MECHANICAL PROPERTY OF METAL MATRIX COMPOSITE WITH NANO-SIZE SiC PARTICLE PRODUCED BY VORTEX METHOD

Akio KAWABE, Toshiro KOBAYASHI

Shizuoka industrial research institute, 2078 Makigaya, Shizuoka, Japan

Toyohashi University of Technology, 1-1 Hibarigaoka, Tempaku-cho, Toyohashi, Japan

ABSTRACT The effects of various pre-treatments on the spatial distribution pattern of particles and the mechanical properties of 0.3 μm SiCp / Al composites are investigated through vortex method. The optimum conditions to obtain the uniform condition of such minute particles are clarified. Mechanical properties and age-hardening behaviors are investigated on these materials. The influence of Ca addition on the dispersion of particles and age-hardenability is also investigated.

Keywords : MMCp, vortex method, nano-size SiC particle, agglomeration, Ca addition

1. INTRODUCTION

The fabrication of metal matrix composite (MMCp) by vortex process is the general method due to the advantage of its convenience⁽¹⁾⁽²⁾. However in the case of fine particles the surface property of the particle appears dominantly, especially in nano-size particles the agglomeration occurs and the fabrication of MMCp is reduced to difficulties.

It is analyzed that the strength of the MMCp is dominated by the ascent of the dislocation density⁽³⁾. Further, as the dispersion strengthening mechanism can be anticipated, dispersions of more fine particles into the matrix are required.

In this study, the influences on various primary factors for solving an agglomeration problem are examined using SiC particles of 0.3 μm (the minimum diameter in the market) into 6061 Al alloy by the vortex method. And mechanical properties and age-hardening behavior were tested on this MMCp.

2. EXPERIMENTAL PROCEDURE

2.1. Materials

6061 aluminum alloy were prepared for this experimental materials and dissolved in a graphite crucible. 10 vol% SiC / Al composites were fabricated under various fabrication conditions by the vortex method equipment, as indicated in Table 1. Stirring condition in the liquid phase is $993\text{K} \times 1.8\text{ks}$, and in semi-solid phase is $918 \sim 923\text{K} \times 3.6\text{ks}$. After that, the melt was casted in the metal mold of $\phi 65\text{mm} \times 200\text{mm}$, and pressed by 300kN . Casting temperature was 1023K , pre-heat temperature of metal mold is 723K . The materials were observed by the optical microscope and SEM to investigate the dispersion of particles.

As a consequence of experiments in fabrication process, optimum fabricating condition of MMCp is adopted and different materials were produced according to table 2. These materials were extruded in the temperature of 673K , and $\phi 10\text{mm}$ rod was obtained under the condition of extrusion ratio 36. The rod materials were tested for the purpose of the measurements of the age-hardening and the tensile strength property.

2.2. Pre-treatment of SiC particles

Pre-treatment procedure of SiC particles consists of cleaning by using the ultrasonic cleaner and steeping in the HF solution. The ultrasonic washing was performed for 300s in 1 vol% nitric acid solution with SiC particles. After that, the head of the nitric acid solution was abandoned and the remaining solution containing SiC particles was added in the HF solution in 86.4ks. The concentration of this HF solution was approximately 10 mass%. After being washed with water, SiC slurry was evaporated to dryness in the atmosphere of 393K . The obtained SiC particles were crushed by the mortar, and used to the disagglomeration process.

2.3. Disagglomeration

Table 1 Fabrication chart and results

No.	Pre-treatment		Jet mill	Ca (mass%)	Mixture		Bulk density ($\times 10^{-3}\text{kg/m}^3$)	Result
	ultrasonic	HF			L	L + α		
A	—	—	—	—	●	●	(0.73)	×
B	—	—	—	2	●	●	(0.73)	×
C	●	●	●	—	●	●	0.7	×
D	●	●	●	—	●	●	0.42	×
E	●	●	●	—	●	●	0.24	△
F	●	●	●	2	●	●	0.23	○
G	—	—	●	2	●	●	0.19	×
H	●	●	●	2	●	—	0.23	×
I	●	●	●	0.5	●	●	0.25	△

Table 2 Chemical composition for this study

Sample No.	Ca (mass %)	SiC (vol %)
J	0	0
K	0.5	0
L	0.5	5
M	0.5	10

The jet-mill apparatus was employed to disagglomerate the SiC powder obtained through the pre-treatment procedure. SiC powder was adjusted to less than $420\ \mu\text{m}$ by passing through the sieve, and provided to the jet-mill apparatus in a rate of $1 \times 10^{-4}\ \text{kg}$ per second. The apparent bulk density of the obtained SiC powder was determined by measuring the weight and the volume of it.

2.4. Tensile test

Age -hardening curve of each material was measured experimentally. Solution heat treatment and aging temperature were $803\text{K} \times 3.6\text{ks}$, and 453K . The hardness variation was measured using Rockwell hardness tester. From the result of the aging test, peak-aging heat treatment was adopted to all specimens. Tensile strength values were obtained in all samples with and without T6 treatment.

3. RESULTS OF EXPERIMENT

3.1. Structure test

Figure1 indicates the microstructure of samples from A to I. No agglomeration was regarded in F which is the sample containing Ca 2 mass%, and SiC particles were pre-treated and disagglomerated. The formation like a some agglomeration was regarded in E (no Ca) and I (Ca 0.5 mass%) by an optical microscope observation. However, it was judged that it isn't the agglomeration by the SEM observation. The agglomerations from several $10\ \mu\text{m}$ to several $100\ \mu\text{m}$ were observed with other samples.

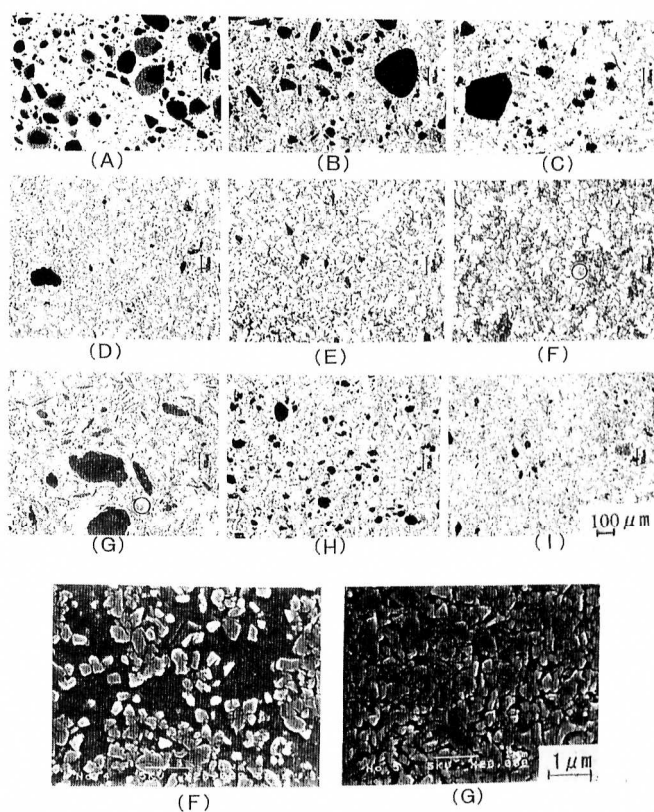


Fig.1 Microstructures of materials

3.2. Influence of pre-treatment on dispersion

Many of black materials rose up on the surface of the water after being washed by using the ultrasonic cleaner. From the result of EPMA analyzation, large amounts of carbon were detected.

To separating the mixture and removing the dirties on the surface of particles, washing by using ultrasonic cleaner is thought to be necessary.

The effects of HF treatment on the surface of SiC were investigated by ESCA analysis. The results were shown in Fig.2. After HF treatment, the amount of SiO₂ on the surface of SiC was recognized to be reduced. In the case of a small amount of SiO₂ on the surface of SiC, SiC particles trend not to agglomerate. Fig.3 indicates this phenomenon by measuring the degree of agglomeration of SiC by using particle distribution measurement equipment. Three kinds of

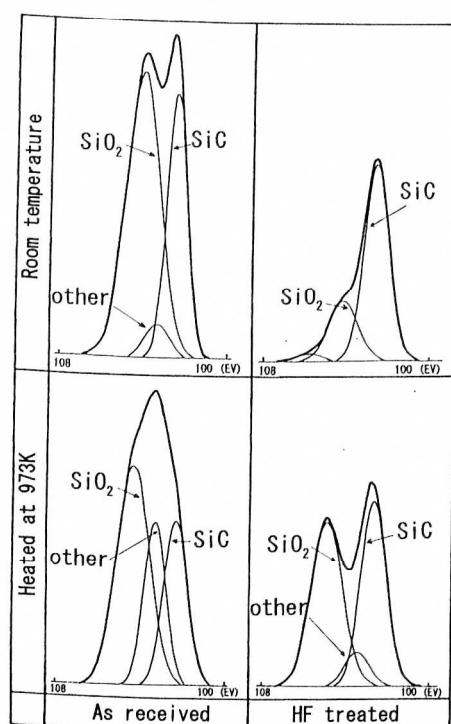


Fig. 2 Surface analysis results of SiC particle

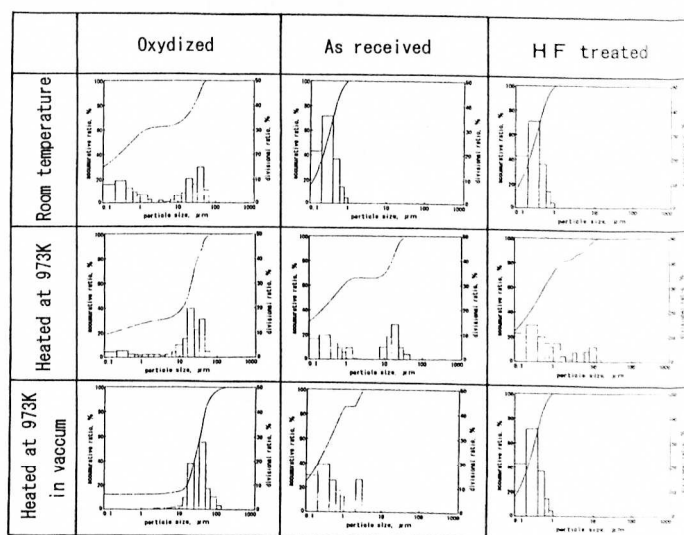


Fig.3. Particle distribution measurement results of 0.3 μm SiC in variously pre-treated conditions

powder were used in this experiment. ① : Large amount of SiO₂ by conducting the heating in atmosphere, ② : As-received, ③ : Small amount of SiO₂ by conducting the HF treatment. Consequently, it was found that not only the less amount of SiO₂ on the surface of SiC (③) but also less amount of oxygen in the atmosphere is necessary to avoid the agglomeration.

3.3. Influence of disagglomeration treatment

Generally, though fine particles have the tendency to agglomerate easily, the agglomerating force becomes weaker in proportion to the void ratio of the powder. Consequently, it can be

thought that if the void ratio of the powder becomes large the particles can be easily dispersed. Therefore three kinds of samples with various void ratios were prepared by jet-mill equipment. Bulk density after disagglomerating is shown in table 1.

From the results of microstructure of C~E, it was found that the number and the size of the agglomeration were decreased in proportion to the reduction of bulk density. However, the low bulk density is not sufficient condition. In the case of low bulk density for G, a lot of large agglomeration can be observed, because of no pre-treatment.

3.4. Influence of Ca addition on dispersion

It is well known that Ca addition to the Al melt improves the wettability between the Al melt and SiC particles⁽⁴⁾. From the results of C~E, it is found that well disagglomerated SiC particles after being pre-treated tended not to agglomerate. Based upon this fact, 2 mass% Ca was added to the melt. No agglomeration was observed. F shows the no agglomeration structure.

3.5. Tensile strength test

Figure 4 indicates the age-hardening curve of No.J~No.M. Peak-age of 6061 Al alloy (No.J) was attained approximately at 3.6 ks. On the contrary, it was shown that the tendency of age-hardening proceeded at the time of 18 ks for the sample of K with Ca 0.5 mass%. However this

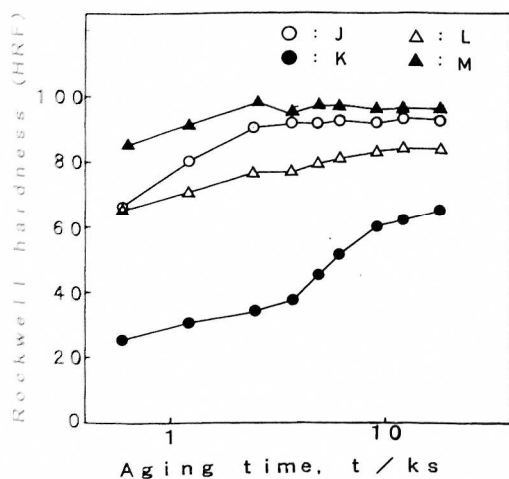


Fig.4 Age-hardening curves measured at 453K

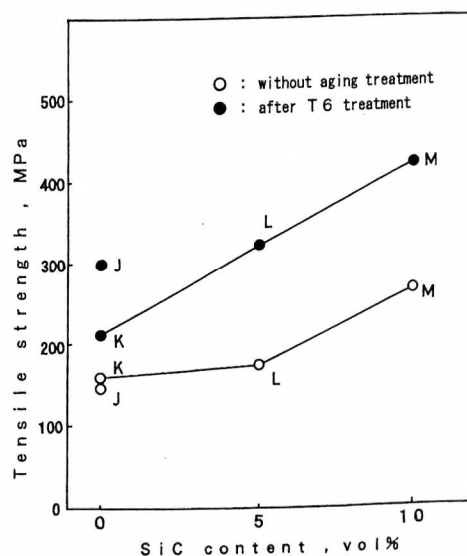


Fig.5 Effect of $0.3 \mu\text{m}$ SiC particle volume fraction on the tensile strength

value of the hardness is lower than 6061 Al alloy. On the other hand peak-age of MMCp (No.M) with SiC 10 vol% was attained approximately at 2.4 ks. Peak-age time of MMCp with SiC 5 vol% (No.L) was middle between No.K and No.M corresponding to the SiC content.

The relation between the tensile strength and the SiC volume content in MMCp is shown in Fig.5 For the sample of M, the increase of the tensile strength by involving SiC particle was approximately 100 MPa without T6 treatment. This corresponds to 1.7 times of 6061 Al alloy without T6 treatment. On the other hand, the increase of the tensile strength of MMCp (Ca 0.5 mass%) was approximately 150 MPa (L,M) by conducting T6 treatment. This increase is nearly the same value as 6061 Al alloy with T6 treatment (170 MPa). In general, it is well known that age-hardening does not occur in the case of Al alloy with Ca content, therefore the increase of the tensile strength does not yield⁽⁵⁾. However, it has been found that for the MMCp the tensile strength ascends independently of Ca content with T6 treatment.

4. CONCLUSION

- (1) It was found that MMCp with no agglomeration can be fabricated by conducting the following procedure; pre-treatment, disagglomeration, Ca addition, and stirring in the semi-solid range.
- (2) The tensile strength of 420 MPa was obtained after T6 treatment for the MMCp with SiC 10 vol%.
- (3) Sufficient age-hardening can be obtained in spite of Ca addition in the MMCp fabricated in this study.

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