FORMATION AND DISPERSION BEHAVIOR OF TiC PARTICLES IN LIQUID ALUMINUM THROUGH THE DECOMPOSING REACTION OF UNSTABLE CARBIDES.

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ABSTRACT

A novel technique has been developed for fabricating in situ formed TiC_p/Al composites. In this process, fairly stable TiC particles are in situ synthesized in liquid aluminum by the interfacial reaction between an Al-Ti melt and, from the view point of thermodynamics, a comparatively unstable carbide such as SiC or Al_4C_3. It is possible in the present process to generate TiC particles of nearly 1 μ m in diameter, even though Al_4C_3 less than 40 μ m was utilized as raw material. The carbon to titanium atomic ratio of the resulting TiC was quantitatively estimated by EPMA. On the other hand, the dispersion behavior of TiC particles in the matrix depended on the size of the starting SiC material: decomposing finer SiC produced more uniform dispersion of TiC particles accordingly. The structure of in situ composites were also affected by the fabrication temperature. Hence, it has been found that the most suitable condition for fabrication should be applied depending on the size of the raw material, even if the same kinds of carbide were used.

Key words: in situ production, TiC_p/Al composite, SiC, Al_4C_3
interfacial reaction, thermodynamical stability

1. INTRODUCTION

They say the fabrication process of metal matrix composites by the melt stirring method is superior to other in the cost of fabrication, but it has several problems which prevent the appearance of stable properties of composites: such that the wettability between melt and reinforcement being not good enough or the brittle and weak reaction layer being formed at the interface between the melt and the reinforcement. Lots of studies such as improving wettability by alloy elements or selecting thermodynamically stable carbides have been carried out to approach these problems.

In recent years, a new technique for fabricating composites which utilizes the reaction between materials has been developed. P.Sahoo et al.(1) reported stable TiC_p/Al composites fabricated by bubbling carbonaceous gas into an Al-Ti melt and kept excellent mechanical properties of these composites even under high temperature. However, using carbonaceous gas as the carbon source seems to cause some practical problems: necessity of additional apparatuses to keep clean working environment,inference of generated hydrogen gas and difficulty to control the volume fraction of the reinforcement. In this study, In situ formed TiC_p/Al composites are fabricated through the reaction between an Al-Ti melt and unstable carbide to solve above problems. This process is basically conducted by the melt stirring method. The effects of process parameters such as melt temperature, stirring time, sorts and size of raw carbide were investigated to establish fundamental knowledge concerned about this novel process.

2.EXPERIMENTAL PROCEDURE

An Al-Ti alloy of total 80 g was melted in an MgO crucible under an argon atmosphere by an induction furnace. Two kinds of carbides whose thermodynamical stability is comparatively low were prepared to add into the liquid Al-Ti alloy whose concentration of titanium was previously adjusted, and incorporated by the melt stirring method. At first, Al_4C_3 of less than 40 μm in diameter was used to observe the change of the microstructure with stirring time when the melt temperature was kept at 1573K. On the other hand, SiC was used to investigate the effects of its particle size (0.6, 3, 14 μm in average diameter) and the melt temperature (1473 ~ 1573K) respectively on the formation and dispersion behavior of TiC particles. In this experiment, the volume fraction of TiC particles involved in every composite was controlled at 5.5%. After the in situ reaction, the melt containing reinforcements was cast and solidified in a permanent mold. Hot extrusion was utilized to form it into a rod shape. The tensile properties of the composite was examined using a rod type specimen having 15 mm in length of parallel part and 4mm in diameter, and a usual tensile testing machine at a constant rate of displacement. Cross sections of the material as cast and extruded were also observed by an optical microscope and SEM. Moreover the quantitative and qualitative analyses of various kinds of element were carried out by WDX and EDX.

3.RESULTS AND DISCUSSION

3.1 Synthesis of TiC by decomposing Al_4C_3

Fig.1 shows the EPMA analysis of a specimen made at 1573K. It is clear from the back scattered electron images as shown in Fig.1 (C) that about 1 μ m in diameter of white particulate precipitates seems to be TiC particles, because of (C) titanium and (d) carbon images almost completely overlapped, moreover (b) aluminum one vanished at the position of these particles. The quantitative analysis by WDX clarified that the in situ formed TiC had the composition of $TiC_{0.93}$, which belonged to composition range between $TiC_{0.5\sim 0.95}$.[2] The microstructural changes in composites with increasing stirring time were shown in Fig.2. At first, (a) shows the typical structure of an Al-Ti alloy matrix. As shown in Fig.2 (b), lots of Al_4C_3 which was not consumed were observed and surplus titanium was precipitated as intermetallic compound Al_3Ti after five minutes stirring time. Ten minutes later (c), almost all of Al_4C_3 was consumed and the formation of fine TiC particles was found with progressing in situ reaction and subsequently, the amount of Al_3Ti decreased. Fifteen minutes later (d), the supplied Al_4C_3 was almost completely decomposed. It shows that the reaction nearly achieved equilibrium state because the structure 30 minutes later (e) had hardly changed comparing with (d). The overall reaction in this process is expressed as follows:

$$Al_4C_3 + 3Ti \rightarrow 3TiC + 4Al$$

Therefore, the degree of in situ reaction in a certain composites could be estimated by investigating the amount of titanium included in the matrix as Al_3Ti or solid solution. Then the change of the concentration of surplus titanium was measured by EDX, and these results vs. stirring time are shown in Fig.3. The broken line in this figure stands for the concentration of surplus titanium after the reaction calculated under this experimental conditions. The concentration of titanium in matrix decreased with increasing stirring time because of the consumption of titanium by the formation of TiC. However the content of titanium is asymptotic towards the broken line, which shows good agreement with experimental results, so Al_4C_3 seems to be perfectly decomposed after more 15 minutes stirring time. Hence, the above results demonstrate that Al_4C_3 free composites are successfully fabricated in this process.

3.2 Synthesis of TiC by decomposing SiC

In a case when SiC particles were utilized to fabricated in situ TiC_p/Al composites, the formation of about 1 μm in diameter TiC particles was also confirmed. Generally speaking, the finer the size of SiC particles is, the more difficult the incorporation of SiC particles into melt becomes.[3] However, it is possible to incorporate 0.6 μm in diameter fine SiC particles into melt under high melt temperature such as 1473K. It was predicted that the interfacial reaction rate would be changed with solid-liquid interfacial area in the case of using carbide particles as carbon source into melt. The structure of composites made by adding each size of SiC particles are compared in Fig.4. It is obvious from (a) that about 1 μm or less TiC particles are almost uniformly dispersed in the composite made by adding 0.6 μm SiC. Almost the same distribution can be also produced when adding 3 μm SiC (b). However, as shown in (c), in the case of 14 μm SiC the distribution behavior of TiC particles is similar to that of the composite made from Al_4C_3, remaining lots of SiC particles and surplus titanium forming Al_3Ti in the matrix of the composites, resulting in a low degree of the in situ reaction. Hence, the particle size of supplied carbide affects not only the rate of in situ reaction but also the dispersion behavior of in situ formed TiC particles. Moreover, Fig.5 shows the effect of melt temperature on the microstructure of composites fabricated from 3 μm SiC. TiC particles formed at the melt temperature of (a)1473K or (b)1573K are comparably the same in size. But, at (C)1673K, a part of formed particles is observed to grow more than 5 μm in diameter. This may be due to the fact that the supplying carbon into the melt was increased because the decomposing reaction of SiC was promoted with increasing melt temperature. Hence, the suitable fabrication condition of in situ formed TiC_p/Al composites would be changed depending on each size of raw carbide even if the same kinds of carbide were used.

4.CONCLUSIONS

(1) In situ formed TiC_p/Al composites are successfully fabricated by the interfacial reaction between an Al-Ti melt and Al_4C_3 or SiC.

(2) The composition of formed TiC is estimated at $TiC_{0.93}$.

(3) The size of resulting TiC particles is about 1 μ m in diameter in spite of using an order of magnitude larger starting carbide.

(4) The finer carbide was used, the more uniform dispersion of in situ formed TiC particles appeared.

(5) The higher temperature of fabrication was, the larger the size of formed TiC particles became.

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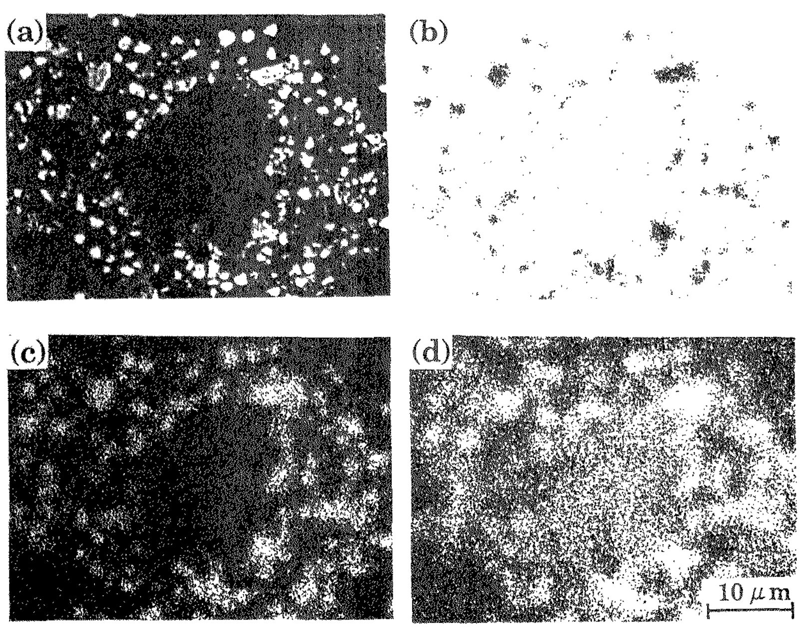

Fig.1 EPMA of in situ formed TiC/Al composite by adding Al_4C_3 at 1573K.
(a) back scattered electron image and X-ray images of (b)Al, (c)Ti and (d) C.

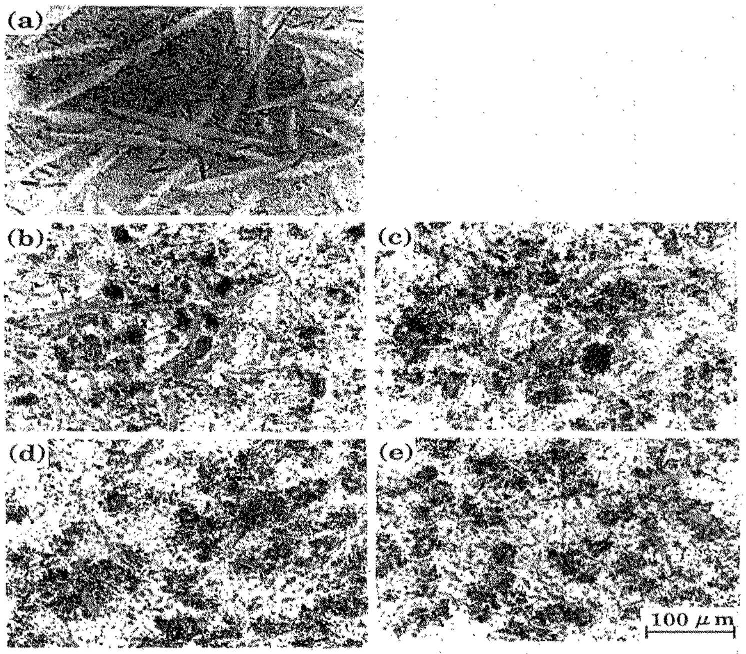

Fig.2 The effects of stirring time on the microstructure of in situ TiC/Al.
(a) before stirring. (b) 5min, (c) 10min, (d) 15min and (e) 30min later.

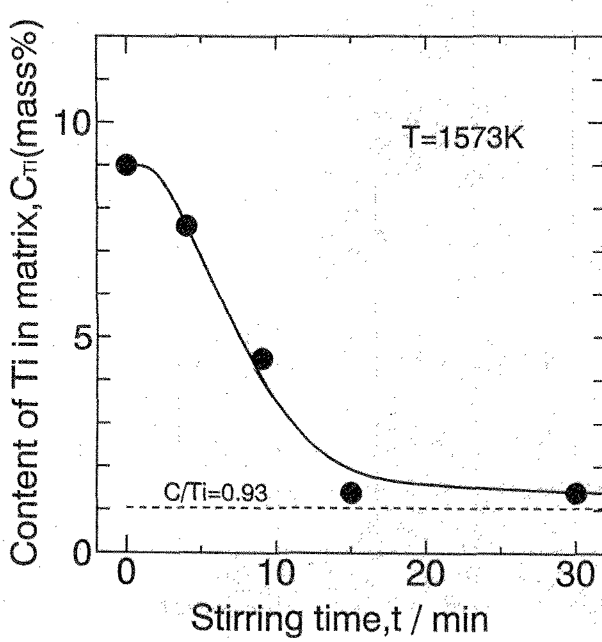

Fig.3 The Variation of the content of titanium in the matrix
with stirring time for adding Al_4C_3 system at 1573K.

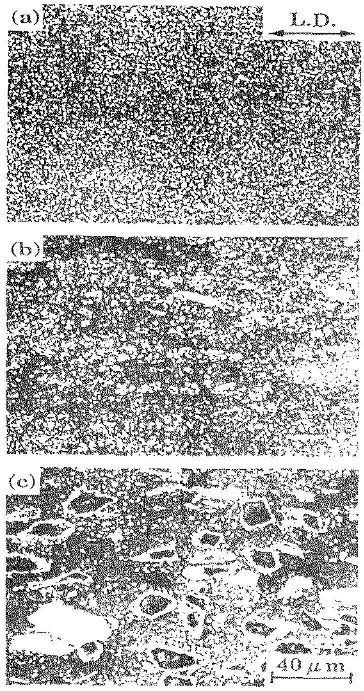

Fig.4 Scanning electron micrographs of in situ TiC composite made at 1473K by decomposing (a) 0.6 μm,(b)3 μm and (d) 14 μm SiC particles.

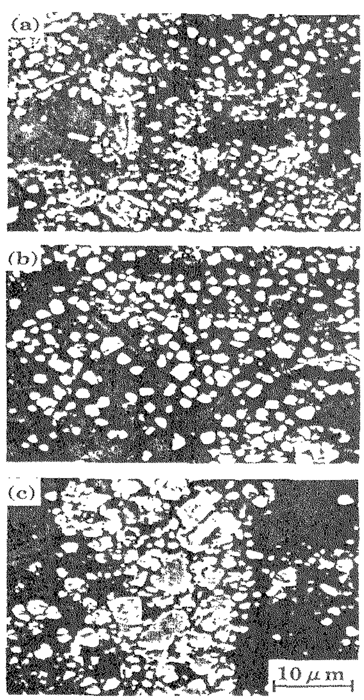

Fig.5 Scanning electron micrographs showing the size distribution of TiC particles formed by decomposing 3 μm SiC at (a) 1473K, (b) 1573K and (c) 1673K.