

MICROSTRUCTURE AND PROPERTY OF Al/TiC COMPOSITES PREPARED BY REACTION SYNTHESIS^①

Zhang Erlin, Zeng Xiaochun, Zeng Songyan, Li Qingchun

School of Material Science and Engineering,
Harbin Institute of Technology, Harbin 150001

ABSTRACT A novel *in-situ* process—Reaction Synthesis has been used to fabricate Al/TiC composites, the phase constitute, microstructure and mechanical property of the Al/TiC composite have been investigated by the use of XRD and SEM. It has been shown that the reaction synthesized TiC particulate is submicron in size (about $0.1 \sim 0.8 \mu\text{m}$), spherical in shape and smooth in surface. In macrostructure the distribution of the TiC particulate in matrix is homogeneous, but in microstructure there is micro-segregation of the TiC particulate at the intergranular. The cast structure become fine because of the existence of the TiC particulate. In addition, with the increasing of weight percentage of TiC particulate in alloy, the yield strength and ultimate tensile strength of the composite increase significantly, e. g. at 15%TiC, 117% and 103% increase respectively, in the same time the elongation of the composite is still more than 4.5%. In the end, the analysis has been done by SEM on the fracture morphology of the composite. The results show that the fracture is ductility, and mainly because of the cluster of TiC particulate to intergranular and the synthesis of bar morphology Al_3Ti .

Key words composites microstructure mechanical property particulate reaction synthesis

1 INTRODUCTION

Metal matrix composites (MMCs) have attracted increasing attention because of their superior mechanical properties such as high specific strength, high specific stiffness and elevated-temperature stability and lower cost in recent years. Particularly in aerospace and automotive industries, they are predicted to have extensive application prospects. During conventional fabricating processes such as mechanical mixing, it is difficult for composite to arrive at the expectant mechanical properties^[1-3], because the added particulates are large in size and have oxides on the surface, the presence of such oxide will inhibit interface binding between ceramic phase and matrix. More recently, many researches have been done on a new approach—*in situ* process, including SHS (Self-propagating High-temperature Synthesis)^[4], XDTM^[5] and

VLS^[6]. Because the dispersoid ceramic particulate is *in-situ* in matrix, the interface between ceramic and matrix is free from oxides, and the interfacial contact strength is high, in addition the particulate is of submicron and the distribution is homogeneous, the composite are of better mechanical properties.

In this paper, a novel *in-situ* method—Reaction Synthesis has been reported. The microstructure and mechanical properties of the composites prepared by reaction synthesis have been investigated.

2 EXPERIMENTAL

High purity (99.7%) titanium powder, 99.6% aluminum powder, 99.3% carbon powder and pure aluminum ingot (99.9%) were used in this experiment. The three start powders were 325 mesh size, corresponding to less than $44 \mu\text{m}$ size. The process of reaction

① Supported by the Aerospace Fund; Received May 8, 1995, accepted Sep. 18, 1995

synthesis to prepare metal matrix composites (MMCs) is shown in Fig. 1. The three pow-

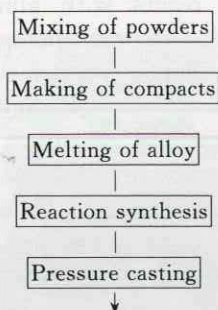


Fig. 1 Process flow diagram of reaction synthesis of prepare composites

ders were mixed with catalyst and bonding, dry in ball mill for 24 h according to a definite molar ratio. The mixed powders were uniaxially pressed into green compacts having a diameter of 40 mm and an approximate height of 10 mm, corresponding to a green density of approximate 50%~60% of theoretical. The powder compact was placed into the molten metal immediately. The processing time and temperature will depend on the molar ratio of mixed powders; processing time can range from 2 min to 20 min; processing temperature is selected between 800~1 000 °C depending upon the molar ratio of mixed powder, the matrix chemistry as well as the melt quantity and weight fraction (or volume fraction) desired. The reaction was carried out at a constant temperature for an appropriate length of time to ensure complete conversion of titanium and carbon in the powder compact to titanium carbide. After completion of the reaction, the melt was agitated and then cast by pressure. Fig. 2 shows a schematic diagram of the reaction synthesis for product of metal matrix composites.

The phase analysis was conducted in a Rikagu D/max-RB X-Ray diffractometer. The micrography analysis of final product was observed by HITACHI S-570 scanning electron microscope (SEM). The macrostructure was observed by an Olympus optical microscope. The piece-shape tensile specimens were machined to the gauge width of 6.3 mm and

gauge height of 2.0 mm and gauge length of 20 mm. All samples were tested at room temperature in an Instron testing machine. The strain rate was selected at $3.3 \times 10^{-3} \text{ s}^{-1}$. The data is an average of five testing results.

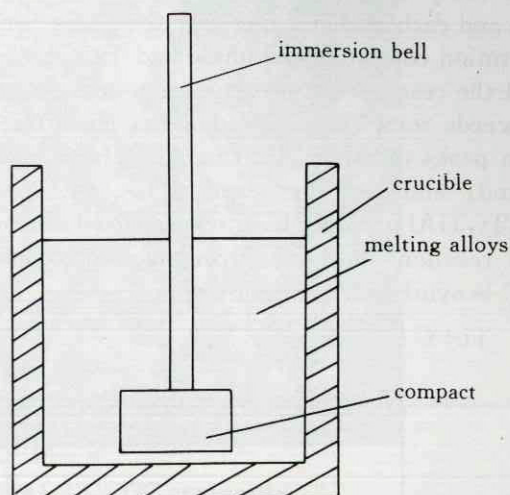


Fig. 2 A schematic diagram of the reaction synthesis for product of MMCs

3 RESULTS AND DISCUSSION

3. 1 Phase Constitution and Microstructure

Fig. 3 shows the XRD pattern of the mixed powder ball milled for 24 h. It is shown clearly that there are diffraction peaks of aluminum, titanium and carbon, no diffraction

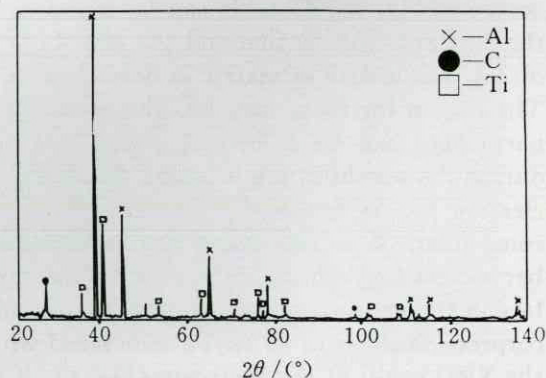


Fig. 3 The XRD pattern of the powders milled for 24 h

peaks of other phases such as TiC, Al_3Ti , indicating no chemical reaction has occurred.

The XRD pattern of the reaction synthesised composite with 15% TiC is shown in Fig. 4. There are only diffraction peaks of Al and TiC phases, no evidence of residual titanium and carbon, displaying that the phase constitution consists of Al phase and TiC phase, and the reaction between titanium and carbon proceeds completely. In addition, no diffraction peaks of Al_3Ti , TiAl or Al_4C_3 have been found, showing the reaction to synthesise Al_3Ti , TiAl or Al_4C_3 have not occurred during the reaction synthesis processing, and only TiC is synthesized as follows:

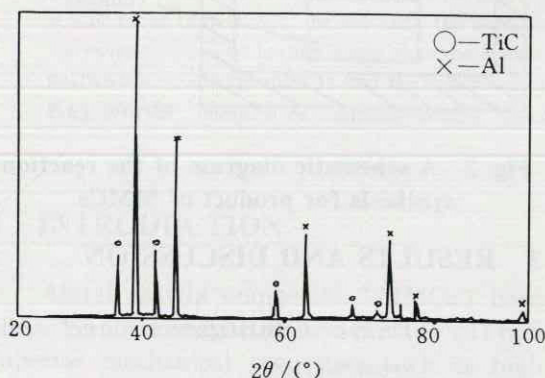
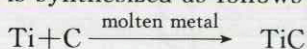


Fig. 4 The XRD pattern of Al/15%TiC composite

Fig. 5 shows the cast structure of the composite (15%TiC). It can be found that the cast structure is fine and the distribution of TiC particulate in matrix is homogeneous. The reason for these may be: the *in-situ* TiC particulate can be a heterogeneous nucleus during the solidification process. thus the nucleation rate is raised and the cast grain become finer. It is also found that there exist bar morphology phase with crack on the surface in the matrix, determined by electron microprobe analysis to be Al_3Ti . Combined with the XRD result of the composite (Fig. 4), it is suggested that the content of synthesised Al_3Ti is very little. The reason for synthesis

of Al_3Ti may due to: during the synthesis process, the carbon combusts, and titanium is excessive and reacts with aluminum to be Al_3Ti . Analyzing the morphology of Al_3Ti , it is shown that there exist defects on the surface of Al_3Ti such as cracks and pits, so it is possible for crack to initiate at the defect of Al_3Ti during tensioning process and result in fracture. In addition, Al_3Ti with bar morphology has separating function to the matrix during tensioning process. It is suggested that it is necessary to reduce or eliminate the synthesis of bar morphology Al_3Ti during the preparing of MMCs.

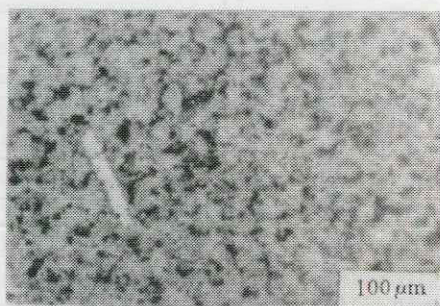


Fig. 5 The cast structure of Al/15%TiC composite

Fig. 6(a) shows the microstructure of the composite (15%TiC). The small bright grain due to TiC particulate, the reaction synthesized TiC is found to be a mixture of mostly spherical grain with some of that are rather rectangular and block in shape, with smooth and clear surface, about $0.1 \sim 0.8 \mu\text{m}$ in size. It is also found that there exists micro-segregation of TiC particulate at intergranular (Fig. 6(b)), owing to: (1) the wettability of TiC ceramic by molten metal is poor. It has been reported that the contact angle between molten aluminum and TiC ceramic is about 118° under 1000°C temperature and Ar_2 atmosphere, so TiC particulates are rejected by solidifying front during solidification process, resulting in segregation to specific location, such as the intercellular/interdendritic regions; (2) the *in-situ* TiC particulate is of submicron and of high surface energy, so it is

difficult to well distribute in the matrix metal.

Fig. 7 is a HREM morphology of the in-

terface between the TiC particulate and Al matrix. It is shown that the interface is clear

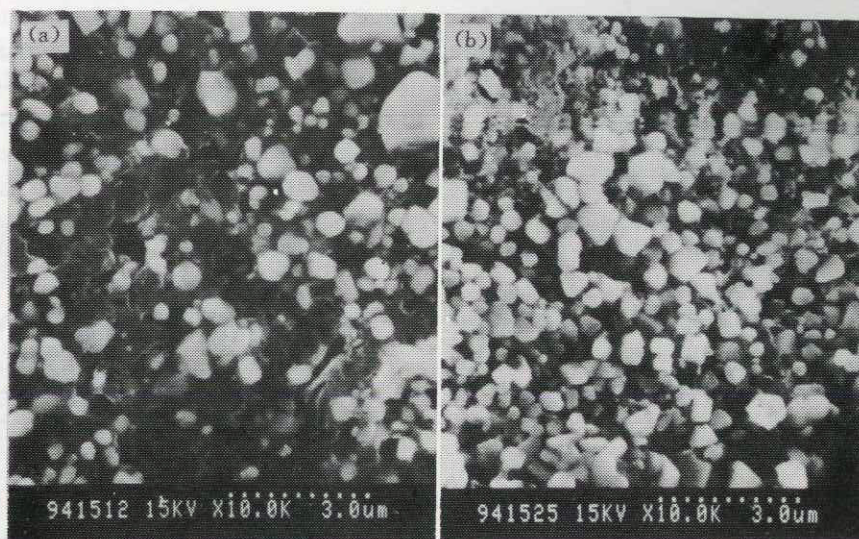


Fig. 6 The microstructure of Al/15%TiC composite prepared by reaction synthesis

(a)—inner grain; (b)—inter grain

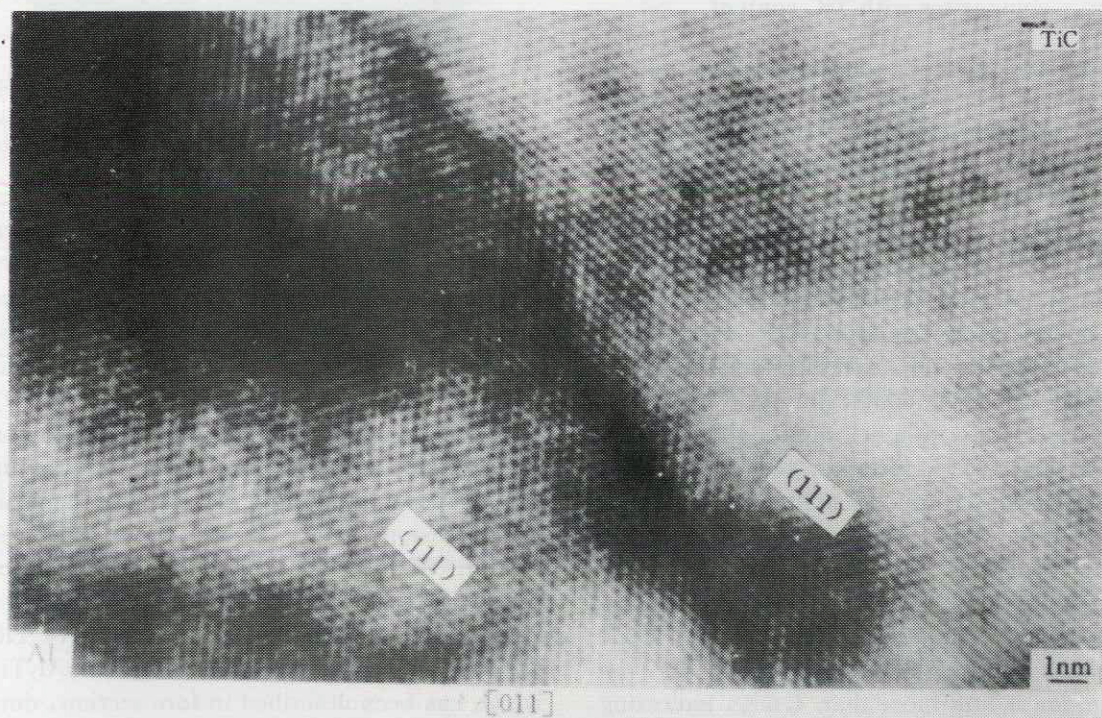


Fig. 7 HREM image of TiC-Al interface in the Al/TiC composite in direction of $[011]$ of TiC particulate

and no product has been found, indicating that no chemical reaction has happened in the interface between the Al matrix and ceramic particulate.

3.2 Mechanical Property

Fig. 8 shows the room temperature yield

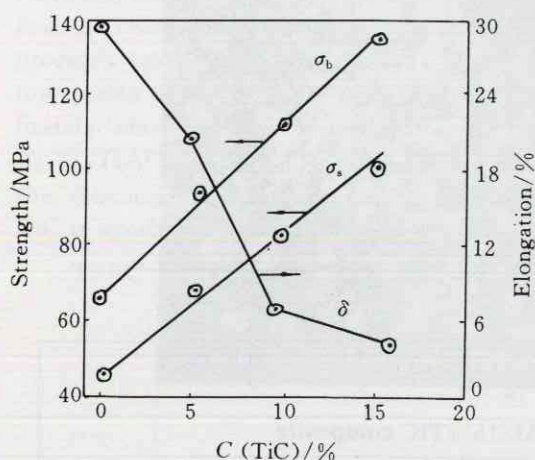


Fig. 8 The mechanical property of the composites with TiC content

strength and tensile strength and elongation of the composite as a function of weight percentage of TiC in the alloy. Strength at 0%TiC represents the property of Al matrix which is prepared by the same process without TiC particulate. It can be found that the yield strength of the composite appears to increase as a linear function of the weight percentage of TiC in the alloy, e. g. a 117% increase at 15% TiC. However, the difference between the matrix and the composite alloy is significant, even at low weight fraction of TiC, e. g. at 5% TiC. The ultimate tensile strength (UTS) of composite indicated a linear dependence on the weight fraction of TiC. When the content of TiC is 15%, the UTS of the composite is about 134 MPa, a 103% increase. With the increasing of weight fraction of TiC, the elongation decrease, and at 15% TiC, the elongation is still more than 4.5%, indicating that the composite has a definite ductility. After analysis, the reason for the strength enhancement may be: (1) the synthesis of fine

TiC particulate with smooth surface and homogeneous distribution of TiC particulate in matrix have better dispersion strengthened function on the alloy; (2) the fine cast structure of alloy because of the existence of TiC particulate (which has been detailed in frontal section) increase the strength of the alloy.

Fig.9 shows the microstructure of the

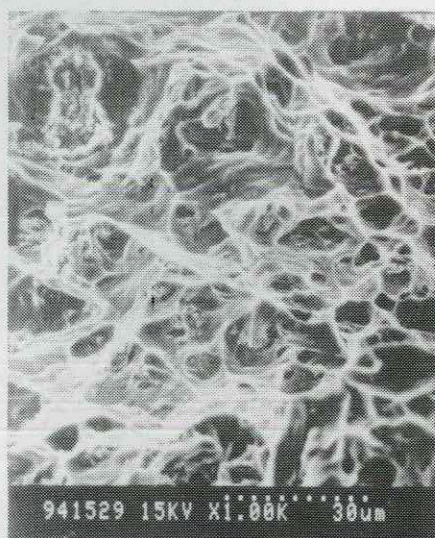


Fig. 9 A SEM microstructure of the fracture morphology of the composites at 15% TiC

fracture surface of the composite. It is shown that there are many dimples in the fracture surface, indicating that the composite belongs to ductility. It can be found that there exist many particulates in the dimple. Previous work^[7-9] on the fracture behavior of a variety of metal matrix composites (dispersion strengthened) contends that crack initiation in tensile specimens is preferred at large particulate/inclusion and regions where particles cluster. With the analysis of microstructure of the composites, it can be concluded that the crack initiates at the intergranular where TiC particles segregate and at the long bar Al_3Ti , which has been described in fore section, during the tensile process, the crack growths and results in the fracture of the composites. It is suggested that the reduction or elimination of

these coarse Al_3Ti and the segregation of TiC particulate at the intergranular will improve the strength and ductility over that currently observed.

The mechanical properties of several composites have been listed in Table 1. It can be found that being a method to prepare MMCs, the reaction synthesis is a viable and competitive method for the production of metal matrix composite.

Table 1 The mechanical properties of several composites

alloy	volume fraction/%	method	condition	σ_b /MPa
Al/TiC	15*	RS	as-cast	134
Al/TiB ₂	15	VS	extruded	349
Al/SiC	20	PM	extruded	115
Al/TiB ₂	15	XD TM	extruded	140.9

alloy	σ_s /MPa	E /GPa	δ /%	refence
Al/TiC	100	—	4.8	this paper
Al/TiB ₂	304	102	3.3	[10]
Al/SiC	75—83	—	—	[11]
Al/TiB ₂	107.3	—	4	[5]

Notes: *—weight fraction;

RS—reaction synthesis;

VS—vacuum sintering;

PM—powder metallurgy;

XDTM—exothermic dispersion

4 CONCLUSIONS

(1) Al/TiC composite has been prepared

by the use of reaction synthesis.

(2) TiC dispersion prepared by reaction synthesis is of submicron (about 0.1~0.8 μm) and homogeneously distribute in the matrix in macrostructure, and segregate at the intergranular in microstructure.

(3) Compared with matrix, the yield strength of the composite increased by 117% and the ultimate tensile strength increased by 103%, at the same time the elongation of the composite is still as high as 4.5%.

(4) The reduction or elimination of the segregation of TiC particulate and synthesis of bar morphology Al_3Ti will improve the strength and ductility of the composites.

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(Edited by Zhu Zhongguo)