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Solidification and interfacial structure of *in situ* Al-4.5Cu/TiB_2 composite

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In situ particle reinforced Al-4.5Cu/TiB_2 composite was fabricated with TiO_2, H_3BO_3, Na_3AlF_6 powders and Al-4.5Cu alloy by reaction in melt. The composite can be directly casted into moulds to make composite parts. TiB_2 particles distribute uniformly in the matrix. The average size of TiB_2 particles is 0.93 μm. At the atomic scale, TiB_2 is hexagonal, and exhibits hexagon or quadrilateral shape. The orientation relationships exist in the interfaces between TiB_2 particle and α-Al, and between the reinforced small Al_2Cu phase and α-Al in the composite. They are $[0\bar{3}32]_{TiB_2} // [110]_{\alpha-Al}$, $(01\bar{1}1)_{TiB_2} // (002)_{\alpha-Al}$ and $[\bar{1}\bar{3}2]_{\alpha-Al} // [123]_{Al_2Cu}$, $(211)_{\alpha-Al} // (111)_{Al_2Cu}$. TiB_2 particle is nucleation site for α-Al matrix growth in the composite. The interface between TiB_2 particles and the matrix is clean and well bonded. No reaction product has been found through HREM observation. This is beneficial to the strength of the composite. The as-cast Al-4.5Cu/TiB_2 composite exhibits mechanical excellent properties: the tensile strength is 416.7 MPa, the yield strength is 316.9 MPa, and the elongation is 3.3 pct.

1. Introduction

The microstructure of matrixes in the metal matrix composites (MMCs) is greatly influenced by reinforcements. The reinforcement can change solidification process of matrixes. The nucleation and growth of the metal matrix and the distribution of particles in the matrix just reflect this. For example, 10 vol. pct TiB_2 particles can make crystallites of Ti-51.5Al-1.4Mn alloy fine [1]. Because Ti alloy can grow on the TiB_2 particles. In principle, the investigation of the solidification microstructures of MMCs is very important.

From strengthening principle of the composite, a basic hypothesis is that the matrix combines with the reinforcement by some way. This decides on the strength of interface and the mechanism of load transmission [2, 3].

To achieve highest strength, it is necessary to have enough interfaces binding strength. Geng [4] had studied the interface of SiC_p/Al composite fabricated by powder metallurgy. He reported that there were no SiO_2, Al_2O_3 and Al_4C_3 on the interface between SiC particle and Al matrix. He found that aluminum had diffused into SiC particle, while there was no silicon or carbon in the matrix. He thought that the bond between SiC particle and Al was diffusion bonding. And he reported that the eight kinds of orientation relationships exist in the interface between SiC whisker and Al matrix. The literature on *in situ* particle reinforced composite is limited. Wang [5] had studied the interface of *in situ* TiC_p/Al composite. He found that the orientation relationship was $(111)_{Al} // (111)_{TiC}$, $[011]_{Al} // [011]_{TiC}$. In this work, we investigate the microstructure aspects and the interface of *in situ* Al-4.5Cu/TiB_2 composites fabricated from TiO_2-H_3BO_3-Na_3AlF_6-Al by reaction in melt. The objectives are to investigate the forming mechanism of solidification microstructure of *in situ* Al-4.5Cu/TiB_2 composite.

2. Preparation of specimen and analysis method

In situ Al-4.5Cu/TiB_2 composite was fabricated from TiO_2-H_3BO_3-Na_3AlF_6-Al system by reaction in melt [6]. The $10 \times 10 \times 10$ mm specimens were made by machining, and mechanically polished. In addition, the specimens were extracted using NaOH water solution to get reinforced powders. X-ray diffraction (XRD) analyzes on the mechanically polished specimens and the reinforced powders. The polished specimens were observed in an optical microscope or a scanning electron microscope (SEM) to investigate the solidification microstructure of the composite.

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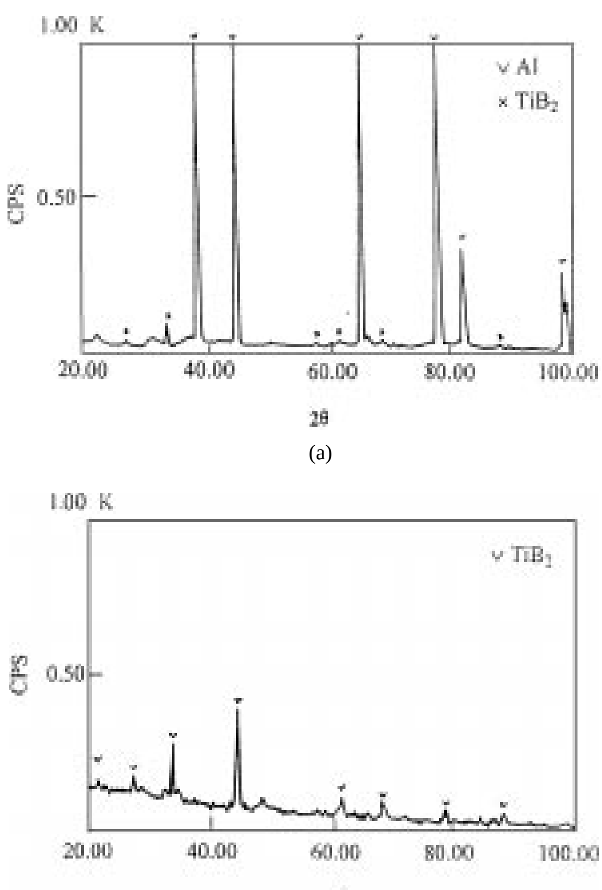
Electric spark working made the thin foils of 0.5 mm thickness. They were then reduced to 30 μm thickness mechanically. And they were dug to 15 μm thickness by Gatan Dimple apparatus. The very thin foils were further reduced to make TEM or HREM specimens at little angle by ion milling technique. The microstructure and interfacial structure in the composite were studied by TEM and HREM techniques.

3. Solidification microstructure

Fig. 1 is XRD patterns of the composite and powders extracted from the composite. The presence TiB_2 peaks indicate that TiB_2 is formed in the composite. TiO_2 and H_3BO_3 in the raw materials have been either reduced or skimmed off before pouring. The powder extracted from the composite is TiB_2, as evidenced by the "v" peaks in Fig. 1b. There is no Al_2Cu peak in the two XRD patterns, because Al_2Cu phase is very fine or dissolved by NaOH extracting solution. Al_2Cu phase has not been identified by XRD.

From above analyses, *in situ* TiB_2 particles were formed from TiO_2-H_3BO_3-Na_3AlF_6-Al system, and as-cast *in situ* Al-4.5Cu/TiB_2 composite can be fabricated by reaction in melt.

The solidification microstructure of Al-4.5Cu/10vol.%TiB_2 composite is shown in Fig. 2. TiB_2 particles distribute in the matrix uniformly. They are nearly spherical shapes. The mean size of TiB_2 particles is 0.93 μm, the most probable size is 0.5 μm. The fine TiB_2 particles are beneficial to the strength and ductility of the composite.



(a)

(b)

Figure 1 X-ray diffraction patterns (a) the composite (b) the extracted powders.

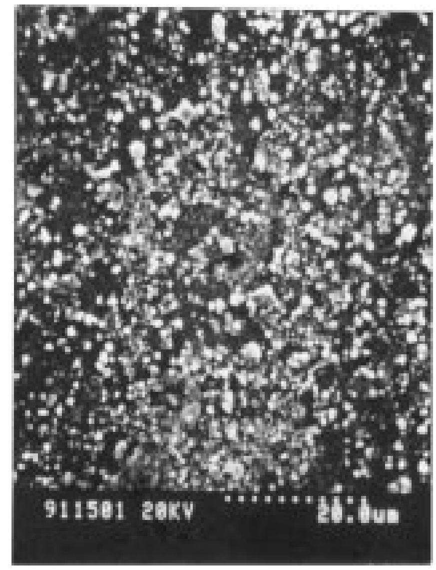


Figure 2 The solidification microstructure of Al-4.5Cu/TiB_2 composite.

4. Microstructure and interfacial structure

To improve the properties, it is necessary to investigate the morphology of *in situ* TiB_2 particle and the interfacial structure between TiB_2 particle and the matrix. In Al-4.5Cu/TiB_2 composite, TiB_2 particle is hexagonal and tends to facet. Most of TiB_2 particles exist in hexagon or quadrilateral (Fig. 3). The larger surfaces of TiB_2 particle are low energy and compact planes. When TiB_2 particle is precipitated from the Al-4.5Cu melt, it develops into single crystal by the growth of the compact planes preferentially. When Al-4.5Cu/TiB_2 composite solidifies, TiB_2 particles can provide a lot of nucleation sites for Al-4.5Cu matrix growth. This makes the crystallite of the composite fine.

Fig. 4 is TEM image of Al-4.5Cu matrix. There is very fine Al_2Cu phase precipitated from the matrix. The interface between Al_2Cu and α-Al exhibits the following orientation relationship:

$$[\overline{13}2_{\alpha-\mathrm{Al}}] \,//\, [123]_{\mathrm{Al_2Cu}}$$
$$(211)_{\alpha-\mathrm{Al}} \,//\, (111)_{\mathrm{Al_2Cu}}$$

Al_2Cu and α-Al is bond well. So the precipitation of Al_2Cu can strengthen Al-4.5Cu matrix.

Fig. 5 is TEM diffraction of two phases in Al-4.5Cu/TiB_2 composite. The orientation relationship is:

$$[0\bar{3}32]_{\mathrm{TiB_2}} \,//\, [110]_{\alpha-\mathrm{Al}}$$
$$(01\bar{1}1)_{\mathrm{TiB_2}} \,//\, (002)_{\alpha-\mathrm{Al}}$$

Because there is an orientation relationship at the interface between TiB_2 particle and α-Al, the interface can be a coherent one. Bramfitt [7] calculated the misfit of two-dimensional lattice, using the follow formula:

$$\delta_{(hkl)_n}^{(hkl)_s} = \sum_{i=1}^{3} \left[\left| d[uvw]_s^i \cdot \cos\theta - d[uvw]_n^i \right| \Big/ d[uvw]_n^i \Big/ 3 \right] \cdot 100\% \qquad (1)$$

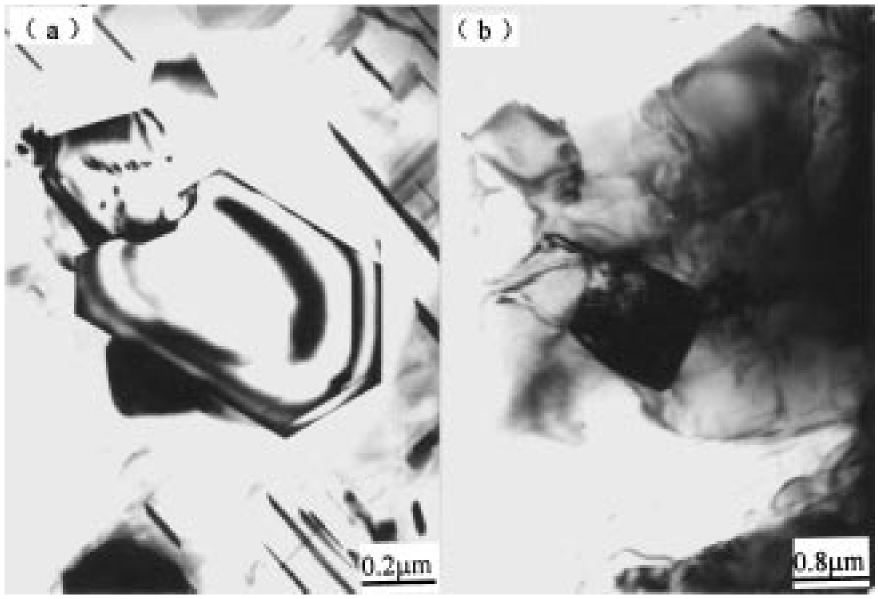


Figure 3 Morphology of TiB_2 particle by TEM (a) hexagon (b) quadrilateral.

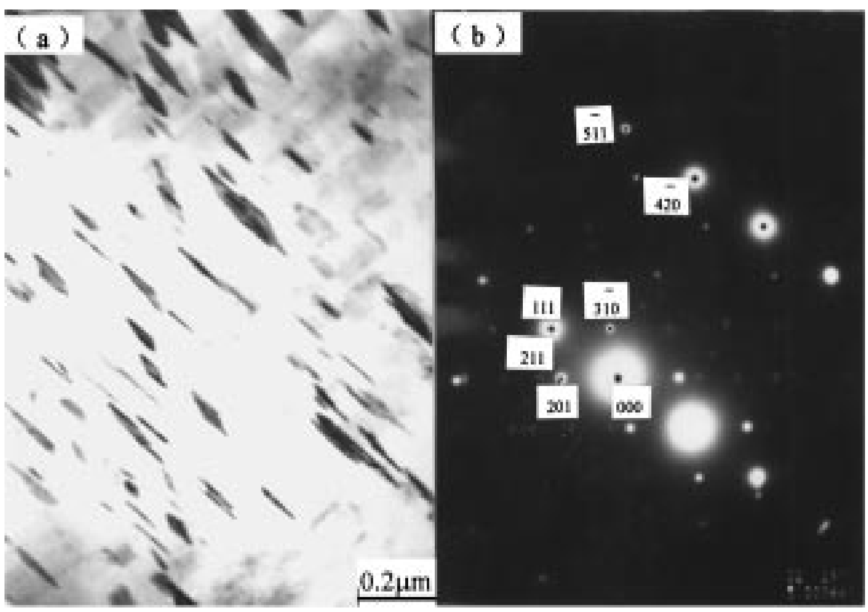


Figure 4 TEM diffraction of Al-4.5Cu matrix (a) morphology of Al_2Cu (b) diffraction pattern.

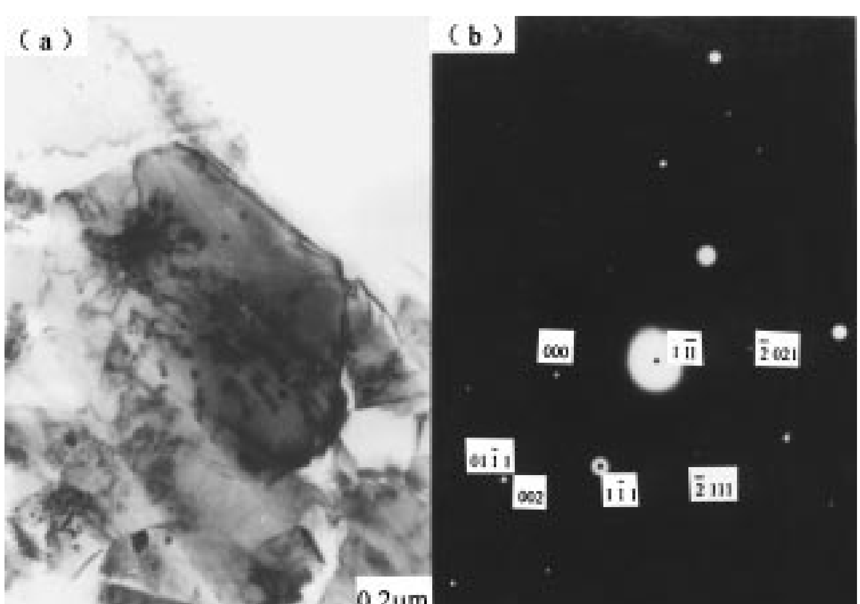


Figure 5 TEM diffraction of Al-4.5Cu/TiB_2 composite (a) morphology of TiB_2 particle (b) diffraction pattern.

TABLE I The matching parameter of $(01\bar{1}1)_{TiB_2}$ and $(002)_{\alpha\text{-Al}}$

$[uvw]_{\alpha-Al}$	[011]	[100]	[010]
$[uvw]_{TiB_2}$	[011]	[101]	[110]
$[uvw]_{\alpha-Al}$	4.05	4.05	4.05
$[uvw]_{TiB_2}$	2.037	2.037	1.515
θ	0	0	0
δ(%)	1.217	1.217	1.217

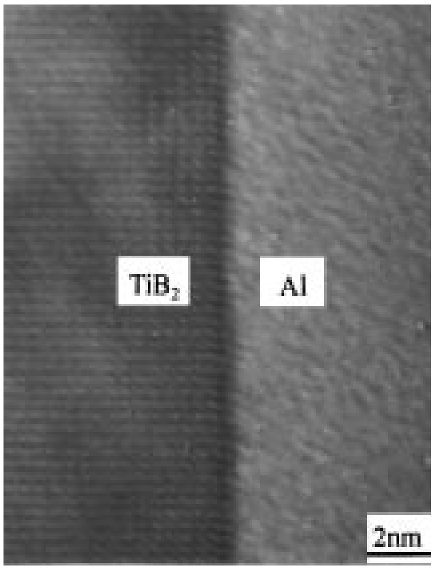


Figure 6 Interface between TiB_2 particle and Al-4.5Cu matrix (HREM).

where $(hkl)_s$—the low index plane of the base; $(hkl)_n$—the low index plane of the nucleation solid; $[uvw]_s$—the low index orientation of $(hkl)_s$; $[uvw]_n$—the low index orientation of $(hkl)_n$; $d[uvw]_s$, $d[uvw]_n$—the atom distance along the orientation $[uvw]$; θ—the angle between $d[uvw]_s$ and $d[uvw]_n$.

The parameters of TiB_2 particle and α-Al are substituted into formula 1,we get the results of the misfit, as shown in Table I. The lattice misfit between $(01\bar{1}1)_{TiB_2}$ and $(002)_{\alpha\text{-Al}}$ at $[0\bar{3}32]_{TiB_2}$ // $[110]_{\alpha-Al}$ is 1.217 pct. It is less than 15 pct. So the interface between TiB_2 particle and α-Al is semi-coherent relationship. α-Al can grow on the face of TiB_2. That is, TiB_2 particle is heterogeneous core of α-Al growth. The crystalline of the composite is substantially made fine.

The interface image is shown in Fig. 6. TiB_2 particle binds with α-Al well. There is no brittle compound between them. So *in situ* TiB_2 can improve the strength and ductility of Al-4.5Cu/TiB_2 composite. Table II is the comparison of mechanical properties of Al-4.5Cu/TiB_2 and others composites. It can be seen that the properties of Al-4.5Cu/TiB_2 composite are superior to those of extruded 6061/Al_2O_3 [8] and 2024/Al_2O_3 [9], and much better than those of as-cast A356/SiC [10] composites.

5. Conclusion

1. Al-4.5Cu/TiB_2 composite can be fabricated from TiO_2-H_3BO_3-Na_3AlF_6-Al system by reaction in melt. The composite can be directly cast into parts. The fabricating price is very low.

2. TiB_2 particles distribute in the matrix uniformly. Their mean size is about 0.93 μm in the solidification microstructure of the composite.

3. TiB_2 particle exhibits hexagon or quadrilateral shape at the atomic scale. The orientation relationship in the interface between TiB_2 particle and α-Al is:

$$[0\bar{3}32]_{TiB_2} // [110]_{\alpha-Al}$$
$$(01\bar{1}1)_{TiB_2} // (022)_{\alpha-Al}$$

4. The lattice misfit between TiB_2 and α-Al is 1.217 pct. The interface between TiB_2 particle and α-Al is semi-coherent. TiB_2 particle can be nucleation site for α-Al growth.

5. Al-4.5Cu/TiB_2 composites exhibit superior strength and ductility.

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TABLE II Properties comparison of composites

Materials	σ_b (MPa)	$\tau_{0.2}$ (MPa)	δ (%)	State	References
Al-4.5Cu/5vol.%TiB_2	358	258.3	1.89	as-cast	this work
Al-4.5Cu/7vol.%TiB_2	387.5	283.3	2.8	as-cast	this work
Al-4.5Cu/10vol.%TiB_2	416.7	316.9	3.3	as-cast	this work
A356/10vol.%SiC	303	283	0.6	as-cast	8
A356/15vol.%SiC	331	324	0.3	as-cast	8
A356/20vol.%SiC	352	331	0.4	as-cast	8
A356/10vol.%SiC	223	—	4.5	as-cast	
2024/20wt.%Al_2O_3	207	—	0.3	as-cast	9
6061/10vol.%Al_2O_3	338	297	7.6	s-extruded	8
6061/20vol.%Al_2O_3	379	359	0.1	s-extruded	8
2024/5wt.%Al_2O_3	345	249	8.2	s-extruded	10