



IN SITU FRACTURE OBSERVATION OF A TiC/Ti MMC PRODUCED BY COMBUSTION SYNTHESIS

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Introduction

Combustion synthesis involving exothermic chemical reactions has been widely utilized to fabricate various in situ metal matrix composites (MMC) during the past decade[1-5]. This process eliminates the interface incompatibility between matrix and reinforcement by creating more thermodynamically stable reinforcements based on their nucleation and growth from parent matrix phase. Thus composites via combustion synthesis demonstrate high specific strength and modulus, as well as excellent oxidation and creep resistance[6-8].

Recently, an in situ SEM observation has been performed to investigate the fracture behavior in a variety of MMCs prepared by conventional processes[9-12]. However few papers about the in situ SEM observation on fracture behavior have been reported in the combustion-synthesized MMC. In the present study, a combustion-synthesized TiC/Ti MMC was fabricated for examining the fracture behavior using in situ SEM observation. The purpose of this paper is twofold : (1) the influence of reinforcement characterization on fracture behavior, including particle size, aspect ratio and interfacial chemistry and (2) the comparison of similarity/disparity of fracture processes between combustion-synthesized and conventional MMCs.

Experimental

The material used in this study was a 10 vol%TiC particulate-reinforced-Ti MMC which was fabricated by vacuum melting and combustion synthesis reaction. After stoichiometric amount of Ti and graphite powders were fully blended, they were compacted into pellets. These pellets along with Ti sponge were melted using non-consumable vacuum arc remelting (VAR), and then cast into a graphite mold (20 mm in diameter). Subsequently, the TiC/Ti MMC was hot-swaged into a rod of 9 mm diameter. Phase identification was conducted by a Siemens D5000 x-ray diffractometer. A LECO 2000 image analyzer was used to characterize TiC particulate. In addition, the pure Ti specimens were prepared via the same process as mentioned above for the comparison of mechanical properties between

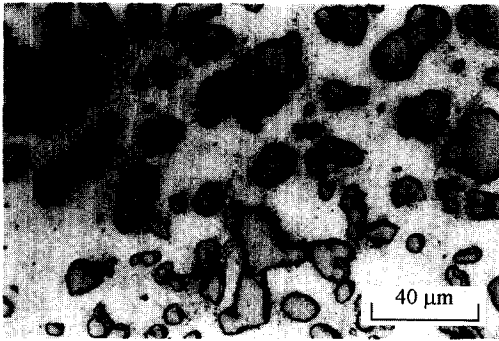


Figure 1. Micrograph of TiCp/Ti composite after hot-swaging.

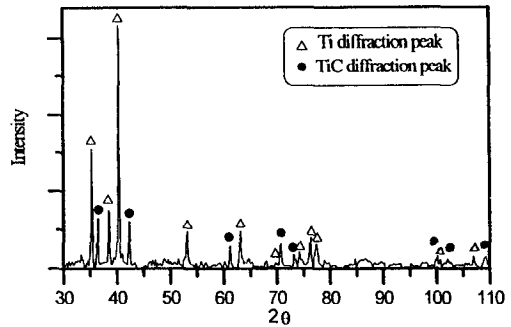


Figure 2. X-ray pattern of TiC/Ti composite system.

Ti MMC and pure Ti. Tensile samples of 4 mm gauge diameter and 20 mm length were machined from hot-swaged rods and then tested at room temperature using a MTS machine. In situ fracture studies were performed with an electronically controlled loading device mounted in a Philips 515 SEM at a rate of 0.01 mm/min. The specimen for in situ tension has a cross section of 1.5mm x 1mm in gage[11].

Results

The cast structure of 10vol%TiC/Ti produced by combustion synthesis consists of a typically dendrite-shaped TiC in α -Ti matrix. After hot-swaging, the dendritic TiC was broken into fine particles, and the particles were distributed uniformly in matrix as illustrated in Fig. 1. The x-ray analysis of hot-swaged in situ MMC is shown in Fig. 2, indicating that only the TiC phase is present in Ti matrix. The quantitative characterization of TiC particulate conducted by a LECO 2000 Image Analyzer reveals that TiC has an average particle size of 9.64 μm , with aspect ratio being 1.49. The distribution of TiC particle size is mainly ranged from 3 ~ 25 μm as shown in Fig. 3. The tensile properties of both 10vol%TiC/Ti and pure Ti are listed in Table 1. According to the results of Table 1, the strength of in situ TiC/Ti MMC is improved markedly as the result of the presence of TiC particulate.

A systematic study of the crack initiation and progressive fracture spread in this type of MMC under tension via an in situ SEM observation is illustrated in Fig. 4 and 5. There are various stages during the fracture process. When external loading exceeded the yield strength of Ti (393 MPa), plastic deformation occurred in matrix, causing the TiC protrusion beyond Ti matrix (Fig. 4(a)), as compared with undeformed structure (Fig. 1). As the loads were further increased (500 MPa), some small parti-

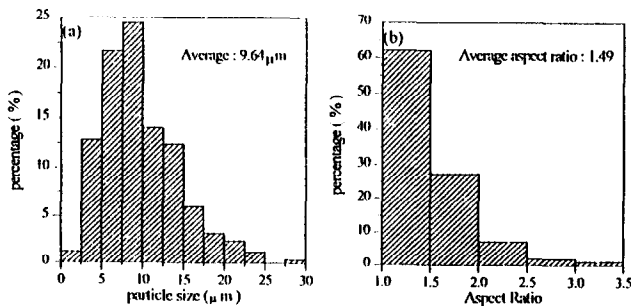


Figure 3. Quantitative analysis of (a) Particle size and (b) aspect ratio histograms of TiC particle in Ti.

TABLE I
Room Temperature Tensile Properties of 10vol%TiCp/Ti Composite and Pure Ti

sample \ properties	Y.S. (MPa)	U.T.S (MPa)	Elongation (%)
Ti	393	467	20.7
10vol%TiCp/Ti	651	697	3.7

cles ($< 10 \mu\text{m}$) with high aspect ratio started to crack (Fig. 4(a)). It is apparent that these cracks were vertical to the direction of tensile load. And then, more and more these particles were continually cracked with increasing loading. As it can be seen in Fig. 4(b), the crack-tips ahead of fractured particles were blunted and the crack propagation was retarded. Fig. 4(c) also shows two fractured particles with blunt tip at the crack front where these two crack-tips were not coalesced at the stress level of 550 MPa, although they were very close. In other word, no significant macro-fracture was found at this stage.

When stresses exceeded around 556 MPa, the large particles were cracked gradually with particle cracking. At this stage, the crack direction is also perpendicular to the loading direction. As the stresses reached a critical value, crack coalescence in large particles took place obviously and rapidly, resulting

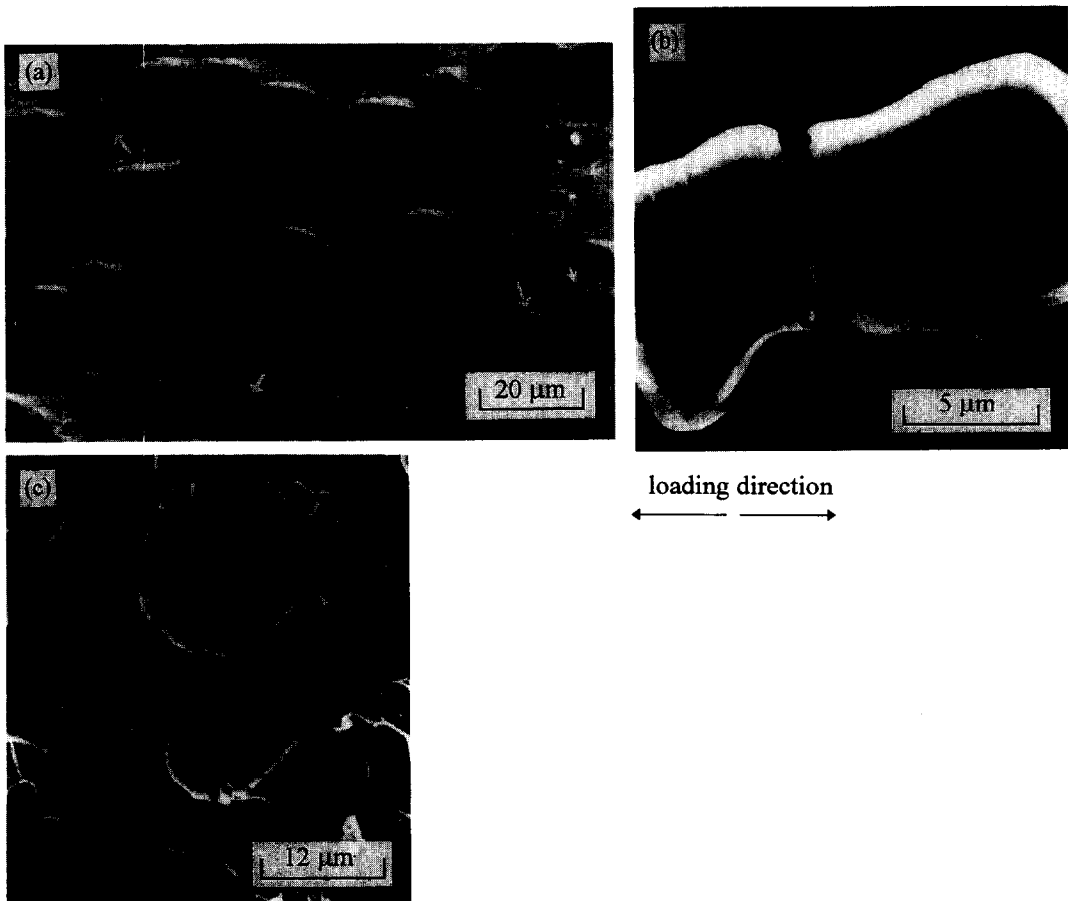


Figure 4. (a) Fracture of small particles with high aspect ratio (arrow indicated) at a load of 458MPa (b) SEM micrograph of blunting phenomenon of a cracked particle and (c) two particles nearby.

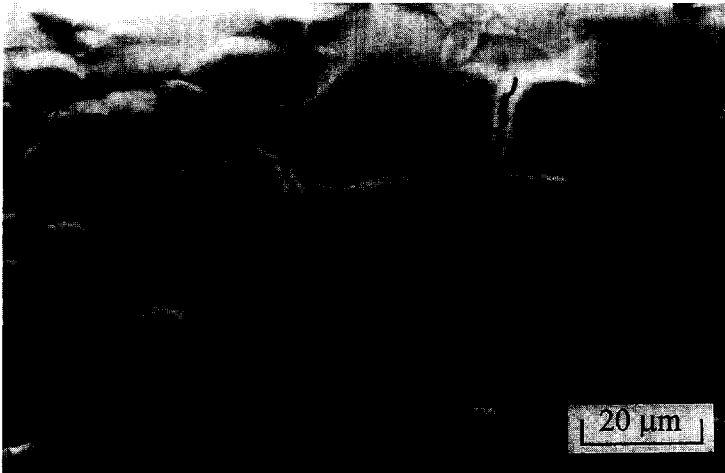


Figure 5. Cracking of larger particles at a higher stress level of 556MPa.

in the fracture of whole specimen. On the basis of this in situ observation, various stages of fracture process in this composite can be characterized, namely plastic deformation in matrix, cracking in small particles, cracking in large particles and coalescence of large cracks.

As it can be seen in Fig. 6, the fracture mode of TiC particulates belongs to a typical cleavage, rather than a pull-out of debonding, showing that TiC/Ti interfaces have a good cohesion. In order to investigate the particle size effect on fracture behavior, a quantitatively fractographic analysis was conducted from a fractured sample by counting the cracked and un-cracked TiC particles near the fracture surface. In all, more than 400 TiC particles with sizes of three main groups ($< 10 \mu\text{m}$, $10\text{-}20 \mu\text{m}$ and $> 20 \mu\text{m}$) were taken and counted. Fig. 7 shows that the fraction of particles with sizes smaller than $10 \mu\text{m}$, 10 to $20 \mu\text{m}$ and larger than $20 \mu\text{m}$ are 52%, 29% and 19%, respectively. That is to say, the most TiC particles near the fracture-end were smaller than $10 \mu\text{m}$. This result is fully consistent

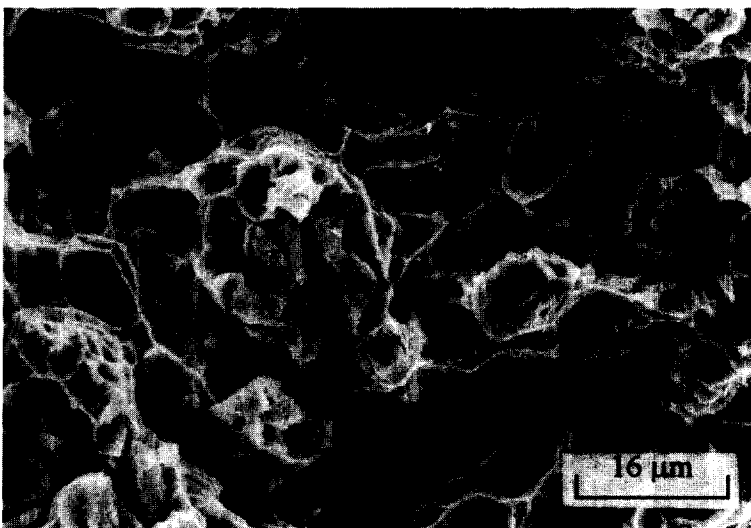


Figure 6. Fractography of 10TiCp/Ti composite after tensile test.

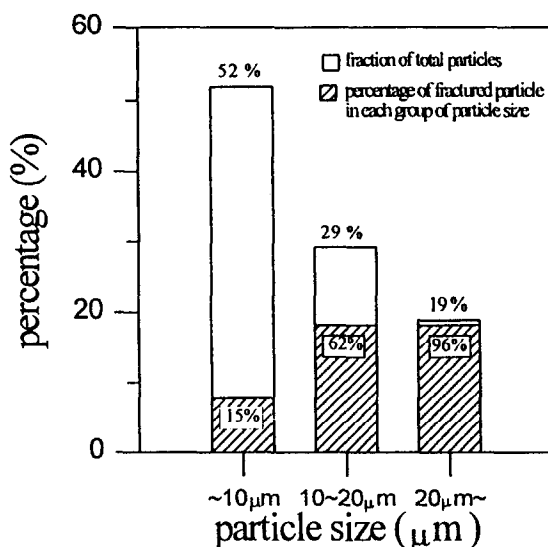


Figure 7. Dependence between particle size and fracture behavior by counting fractured particles statically nearby the fracture surface.

with the previous data obtained from image analyzer. From the statistic investigation only 15% fine particles ($< 10\ \mu\text{m}$) were broken after fracture. However, nearly total large particles ($> 20\ \mu\text{m}$), approximate 96%, were found to be broken. Obviously, the fracture behavior of Ti MMC is greatly affected by the particle size of reinforcement.

Discussion

The results apparently show that particle cracking is the main reason for the tensile fracture of the in situ TiC/Ti composite at room temperature, even though few cracks exist near the interfaces. From the fractographic observation, the cleavage fracture of TiC particles also supports this result. On this basis, the interfacial strength in this type of composite is strong enough to withstand the stress concentration loaded at the interfaces and to transfer the stress from Ti matrix to TiC particulate. Therefore the composite is reinforced by TiC particles leading to the increase of strength as shown in Table 1. This result is quite different with $\text{Al}_2\text{O}_3/\text{Al-Si}$ systems [12], in which the decohesion of reinforcement/matrix interface is the main mechanism for the fracture behavior and impair the mechanical properties of composites at room temperature.

With respect to the influence of particle size on fracture processes, a study conducted by Mummery and Derby [13] in the SiC/Al composite showed that there is a transition in fracture mechanism from decohesion to particle cracking on increasing particle size. In addition, crack initiation is found to occur at a lower stress for the large particle than smaller one. Similarly, an investigation by Weng et al. [11] in SiC/6061Al composites concluded that crack initiation inside the large particle leads to fracture. Meanwhile, debonding at interface layer is subject to occur in the smaller particle system. In our present system, various stages of fracture process were observed by using in-situ SEM. It is apparent that there is no transition in fracture mechanism from decohesion to particle cracking as the result of particle size change. Similar to the previous studies, the fracture mechanism is attributed to the coalescence of cracks of large particles, although some cracks inside the small particles with high aspect ratio is first observed primarily at the lower stress level. According to the present study, in situ MMCs possess an excellent interfacial characteristic for transferring stress from matrix to reinforcements and

are able to accommodate the concentrated stress at the crack tip by a blunting mode which retards the coalescence of cracks effectively. On these bases, controlling the particle size and aspect ratio in this kind of MMCs seems to be important for rendering their mechanical behaviour, particularly in improving the ductility and toughness.

Regarding the deformation mechanism and interfacial characteristic in MMCs, Lu and Sass [14] have explored the deformation processes in the vicinity of Pt-NiO interfaces tested by the periodic crack method. They suggested that dislocations propagate from the interface and react to form immobile dislocations, acting to nucleate microcracks. However, some factors associated with interfacial chemistry and reinforcement characterization should be taken into account since they greatly influence the fracture mechanism. For TiC/Ti systems, a quantitative analysis of interfacial chemistry using electron-energy-loss-spectroscopy was carried out by Gu et al.[15]. They concluded that a dissolution-type reaction between Ti and TiC occurred, causing a continuous carbon-deficient zone and rendering good mechanical properties. In the present study of the TiC/Ti MMC via casting route, a continuous carbon-deficient zone is expected to exist in accordance with the thermodynamics and the equilibrium C-Ti phase diagram. Hence the interfacial structure and chemistry change of in situ composites need to be characterized to account for the facts that the interfaces are strong enough to resist fracture. These are still questions to be answered and should be the subject of further study.

Conclusions

According to the present results, it can be concluded that the tensile fracture process at room temperature in a TiC/Ti MMC produced by combustion synthesis has various stages, including plastic deformation in matrix, cracking in small particles, cracking in large particle and coalescence of large cracks. Statistically, the particle size and aspect ratio play a crucial role in controlling and determining the fracture behavior. With regards to the fracture mechanism in this type of MMC, studies on the interfacial chemistry and the related structure should be made to confirm this aspect.

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