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ScienceDirect
Trans. Nonferrous Met. Soc. China 27(2017) 2548−2554
Transactions of Nonferrous Metals Society of China
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Microstructure and mechanical properties of Al−TiB_2/TiC in situ composites improved via hot rolling

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Received 27 June 2016; accepted 16 December 2016

Abstract: A kind of Al−TiB_2/TiC in situ composite with a homogenous microstructure was successfully prepared through in situ reaction of pure Ti and Al−B−C alloy with molten aluminum. In order to improve the distribution of the particles and mechanical properties of the composites, subsequent hot rolling with increasing reduction was carried out. The microstructure evolution of the composites was characterized using field emission scanning electron microscopy (FESEM) and the mechanical properties were studied through tensile tests and microhardness measurement. It is found that both the microstructure uniformity and mechanical properties of the composites are significantly improved with increasing rolling reduction. The ultimate tensile strength and microhardness of the composites with 90% rolling reduction reach 185.9 MPa and HV 59.8, respectively, 140% and 35% higher than those of as-cast ones. Furthermore, the strengthening mechanism of the composite was analyzed based on the fracture morphologies.
Key words: in-situ composites; TiB_2/TiC particles; rolling; mechanical property

1 Introduction

Aluminum-based metal matrix composites (AMMCs) reinforced by ceramic particles have received increasing attention due to their high specific strength, good wear resistance, excellent dimensional stability and superior damping capacity [1,2]. A combination of these properties is not available in a conventional material. The properties of AMMCs depend on the type, size, content, bonding and spatial distribution of ceramic particles [3]. The fabrication process affects the properties of AMMCs to a large extent. Generally, particle reinforced aluminum composites are produced using several conventional and specific methods, and liquid metallurgy route is widely preferred owing to its simplicity, low cost, near net shape and mass production [4]. The particles are either added externally or formed inside the molten metal in liquid metallurgy route. The latter method is named as in situ fabrication [5]. It is known that the particle reinforced composites synthesized by in situ processes have clearer interface between matrix and reinforcement particles, and better matrix−reinforcements interfacial thermodynamic stability compared with exogenously formed processes [6].

Fine grain sizes, uniform distribution, homogenous reinforcement and strong bonding of reinforcements with matrix will certainly improve the mechanical properties of composites. However, the major drawback of in situ composites is segregation and clustering of particles at the grain boundaries [7,8]. It is difficult to avoid segregation during formation and solidification process for the melt of the composites. In this regard, plastic deformation processes, for example, forging, extrusion, or rolling is usually used to improve the homogeneity of the particle distribution in the composites [9]. However, the microstructures and mechanical properties of AMMCs prepared through melt reaction-plastic deformation have yet to be investigated systematically. Therefore, an attempt is made to study the microstructure and mechanical properties of AMMCs prepared through melt reaction and subsequent hot rolling.

Foundation item: Project (51501092) supported by the National Natural Science Foundation of China; Project (30915011332) supported by the Fundamental Research Funds for the Central Universities, China; Project (2014M550292) supported by China Postdoctoral Science Foundation
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DOI: 10.1016/S1003-6326(17)60283-8

In this work, a kind of Al−TiB_2/TiC composite was prepared through in situ melt reaction of Al−Ti and Al−B−C alloys, and subsequent hot rolling was carried out to improve the microstructure and mechanical properties. TiB_2 and TiC ceramic particles are supposed to be the outstanding reinforcement in Al alloys for their good thermodynamic stability, high hardness, high melting point, high modulus, high corrosion resistance and low density [10−12]. The microstructure evolution and mechanical properties of the Al−TiB_2/TiC composite with different rolling reductions were investigated. The strengthening mechanism and fracture mode of the Al−TiB_2/TiC composites were also analyzed.

2 Experimental

In this study, an Al−8B−2C master alloy supplied by Shandong Al&Mg Melt Technology Co. Ltd., Ti sponge (99.8%) and commercial pure Al (99.7%) were used as raw materials. Ti was added into Al melts at the outset in a medium frequency furnace, then the Al−8B−2C alloy was added into Al−Ti melts when the melt temperature rose to abut 950 °C. After holding for 15 min, an Al−3.6TiB_2−1.4TiC (namely Al−5%TiB_2/TiC in the following text) in situ composite was prepared. Then, a series of plates with a length of 20 mm, width of 10 mm, and thickness of 10 mm were cut from the ingot of the composites for further rolling process.

The as-cast composite plate was preheated at 300 °C for 30 min in a resistance furnace and then rolled on the laboratory rolling mill with a certain reduction in thickness about 1mm. This process was repeated and then a series of samples with different rolling reductions of about 20%, 40%, 80% and 90% were obtained, respectively. During each rolling pass, the samples were heated at 300 °C for 1 min.

The microstructures of the composites were characterized using a field emission scanning electron microscope (FESEM, Quanta 250F) equipped with Oxford energy dispersive X-ray spectrometer(EDS) and X-ray diffraction (XRD，Bruker D8). The tensile tests were conducted at ambient temperature on the Walter +bai LFM 20 kN universal test machine at an initial strain rate of 5.6×10^{-4} s^{-1}. The tensile test specimens were machined from the processed sheets oriented along the rolling direction according to the ASTM: E8M standard. Thus, the tensile direction of the specimen was parallel to RD of the sheets. Vickers hardness experiment was carried out on the surface of RD−TD plane for the multilayered composite using a tester (HMV-G 21DT) at the load of 0.49 N and holding for 15 s. For each specimen, at least seven randomly selected points were tested to obtain a mean value with a standard deviation error.

3 Results and discussion

3.1 Microstructure evolution of Al−5%TiB_2/TiC in situ composites

The XRD pattern of the prepared Al−5%TiB_2/TiC composites is shown in Fig. 1(a). According to the XRD pattern, it can be found that TiB_2 and TiC reinforcement phases were successfully formed in the Al matrix in the present condition. Figure 1(b) shows the microstructure of the composites and lots of reinforced particles can be located in the matrix. It is noticed that most of the particles distributed along the grain boundaries and the agglomerated particles connected with each other formed long particle clusters. As being illustrated in the magnified micrograph (Fig. 1(c)), it can be confirmed

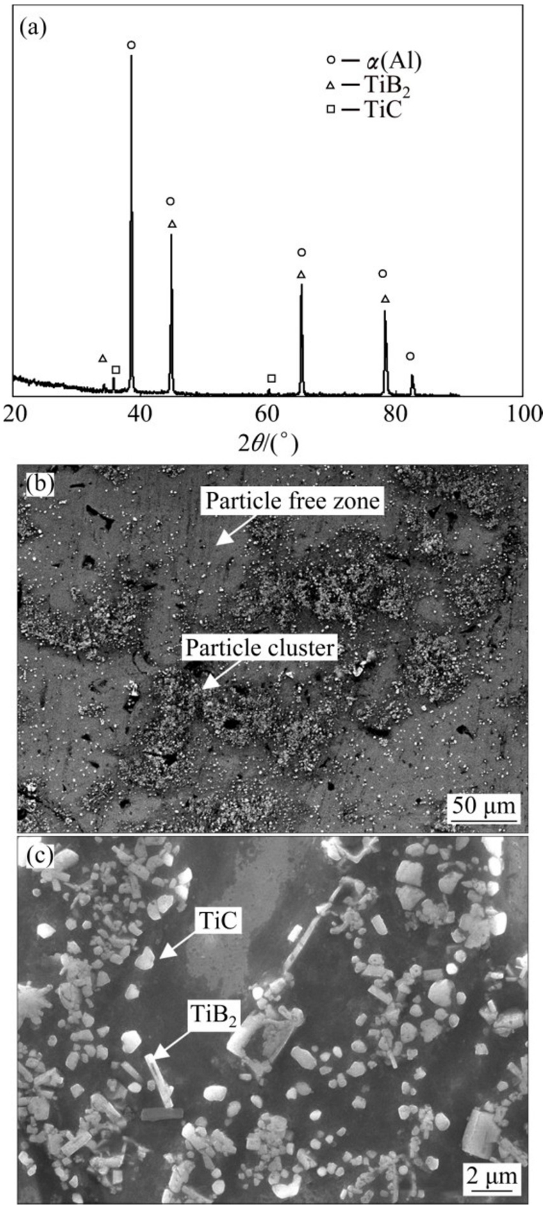

Fig. 1 XRD pattern (a) and FESEM micrographs at low (b) and high (c) magnification of Al−TiB_2/TiC in situ composites

that the white particles are TiC and the gray particles are TiB_2 [13]. The size of TiB_2 and TiC particles is in the range of 0.5−2 μm and the particles easily form big clusters due to their large surface to volume ratio and their attractive Van der Waals interactions [14].

In order to improve the non-uniform distribution of TiC and TiB_2 reinforcement particles in the Al matrix, the composites were processed by rolling deformation after being preheated. Figures 2(a)−(d) show the microstructures (in RD−TD planes) of the Al−5%TiB_2/TiC composites with 20%, 40%, 80%, 90% rolling reduction, respectively. It can be seen that the distribution of the reinforcement particles was improved with increasing rolling reduction. After 90% rolling reduction, the particle free zone in the matrix became smaller and the original densely agglomerated distributed loosely.

Figure 3 demonstrates the SEM micrographs of Al−5%TiB_2/TiC composites with different rolling reductions at a higher magnification. It can be seen that a large particle cluster is actually composed of multiple smaller ones due to their attractive van der Waals interactions to reduce the total surface energy in the melt. Thus, when the rolling reduction is smaller than 40%, the distribution of agglomerated particles is not improved obviously (Figs. 3(a) and (b)). While after rolling for 80% reduction, it can be seen in Fig. 3(c) that some cracks initiated in the large close packed particle clusters. With increasing the deformation (Fig. 3(d)), the large particle agglomeration cracked and was divided into several submicro ones. And then, the average particle size of TiC and TiB_2 was reduced. Meanwhile, the number of small particles with submicro size in the matrix increased due to the separation of particle agglomeration. Therefore, the particle distribution in the matrix was significantly improved and became more homogenous after rolling deformation.

Grain structures of the composites are shown by the polarized light optical micrograph after electroetching the polished specimens. It can be seen that the as-cast Al−TiB_2/TiC composites have an average grain size of 30.6 μm, as shown in Fig. 4(a). However, after rolling for 90% reduction, the grains of Al matrix are elongated and lots of grains are significantly refined due to the fragment of the elongated grains during the rolling process, as shown in Fig. 4(b).

3.2 Mechanical properties of in situ Al−5%TiB_2/TiC composites

Engineering stress−strain curves of the in situ Al−TiB_2/TiC composites with different rolling reductions (in RD−ND planes) are shown in Fig. 5(a). The ultimate tensile strength (UTS) and elongation (EL) of the composites with different rolling reductions are shown in Figs. 5(b) and (c). Compared with the as-cast one, the as-rolled Al−TiB_2/TiC composites showed higher strength and lower ductility. It is found that the elastic modulus of the composite also increases with

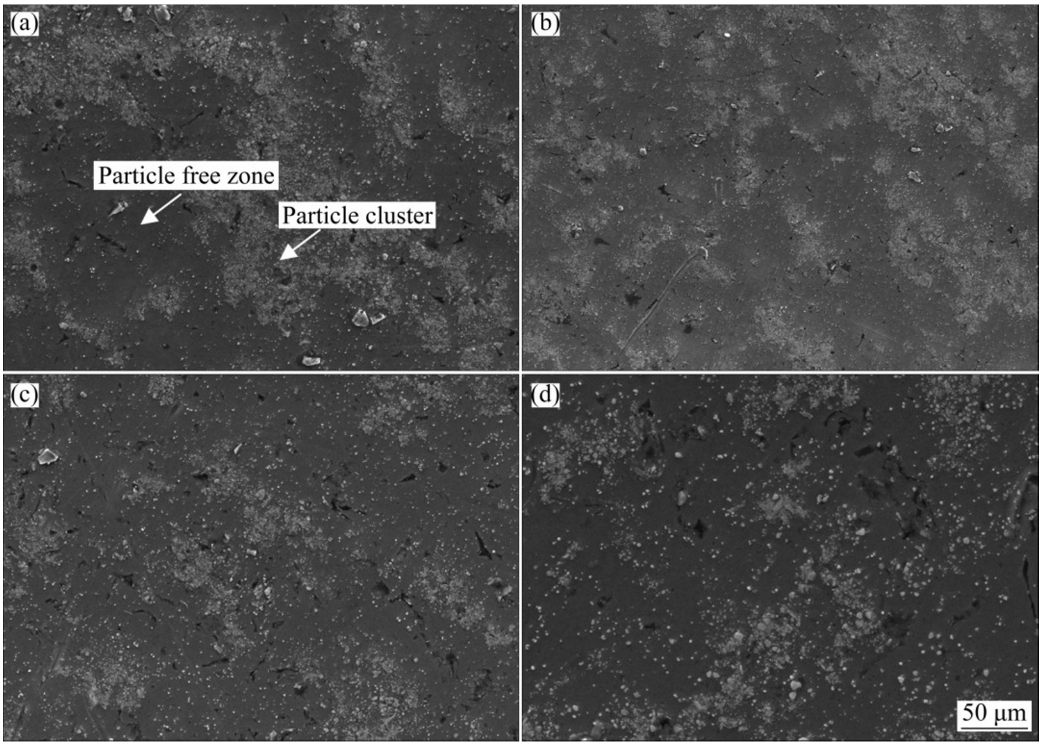

Fig. 2 Microstructures of Al−TiB_2/TiC in situ composites with different rolling reductions (in RD−TD plane): (a) 20%; (b) 40%; (c) 80%; (d) 90%

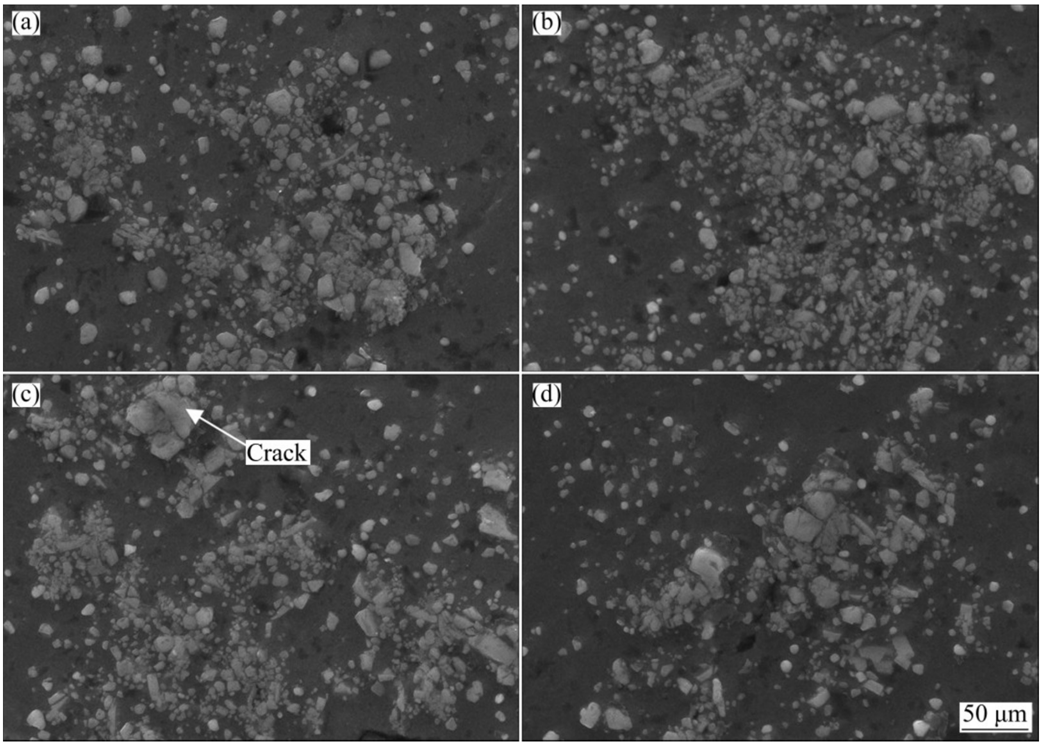

Fig. 3 Effect of rolling reduction on particle distribution of composites in RD−TD plane: (a) 20%; (b) 40%; (c) 80%; (d) 90%

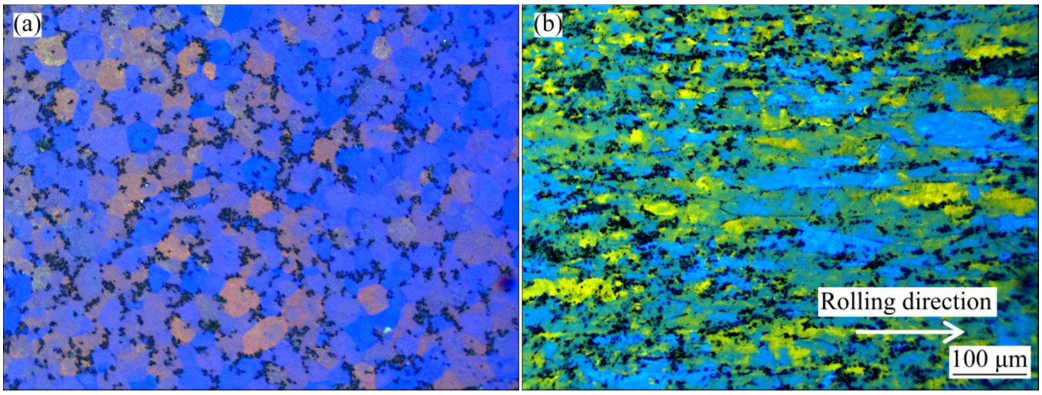

Fig. 4 Polarized light optical micrograph of Al−TiB_2/TiC in situ composites: (a) As-cast; (b) After rolling with 90% reduction

increasing rolling reduction. It is supposed that uniform distribution of reinforcements coupled with good matrix−reinforcement interfacial integrity resulted in an increase in internal stress between reinforcement and matrix, and finally led to the enhancement of elastic modulus [15]. The UTS of the composites increased with increasing rolling reduction and it increased from 77.2 to 185.9 MPa after 90% rolling reduction, 140% higher than that of the as-cast one. However, the ductility had a dramatic decrease after rolling deformation. It is worthy to note that the elongation of the composites increased slightly after 80% rolling reduction and reached 8.9% after 90% rolling reduction.

It is known that strain hardening or dislocation strengthening plays the main role in the strength increase in the rolling deformation. Plastic deformation introduces the fast dislocation propagation in the matrix and around the reinforcement particles, which improves the strength of composites and results in the ductility decrease. With increasing rolling reduction and multiplication of dislocations, the grains of aluminum matrix tend to be refined and then grain refinement dominates the strength variation. On the other hand, microstructure evolution (Figs. 2 and 3) indicates that with increasing rolling reduction, the distribution of the reinforcement particles in the aluminum matrix changes to be more uniform after the second cycle and the sizes of most particles are under 1.0 μm, which are suitable reinforcement for Orowan strengthening [16]. Thus, both the strength and elongation increase because the uniformity of reinforcement particles has a major effect on the strength and ductility of metal matrix composites [17,18].

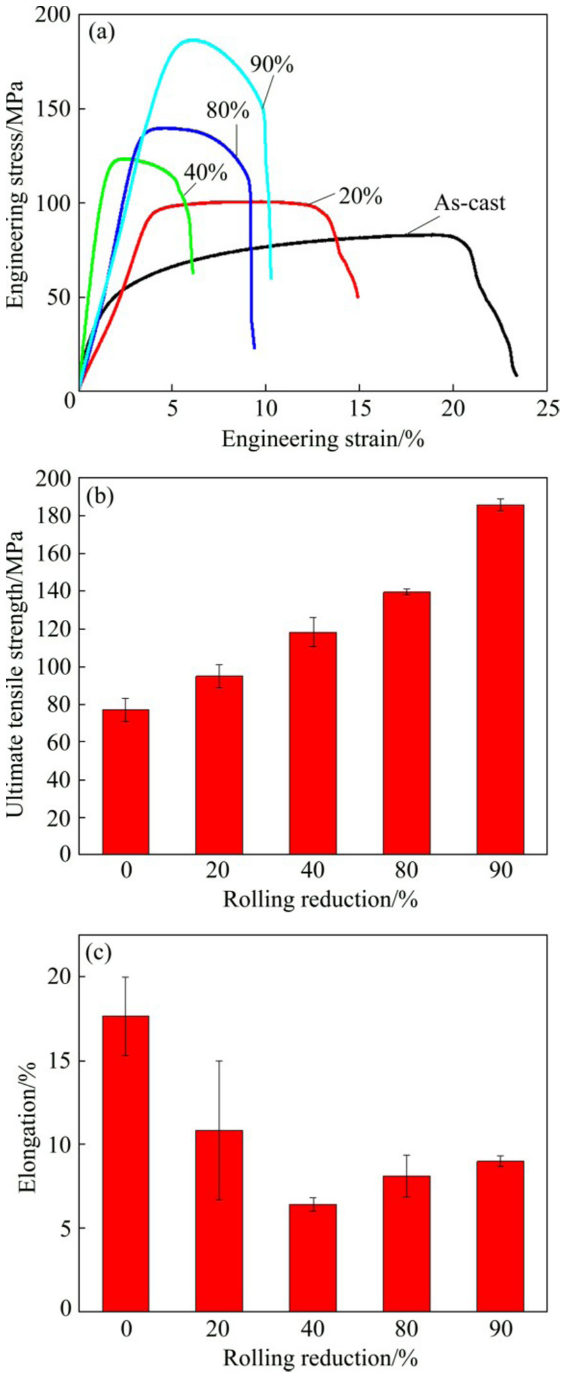

Fig. 5 Tensile properties of Al−TiB_2/TiC composites with different rolling reductions: (a) Engineering stress−strain curves; (b) Variation of UTS vs reduction; (c) Variation of elongation vs reduction

The microhardness of the as-rolled Al−TiB_2/TiC composites was also measured on the RD−TD planes, as shown in Fig. 6. It illustrated that the microhardness of the composites increased with increasing rolling reduction. After 90% reduction, the hardness became HV 59.8, 35% higher than that of the as-cast one of HV 44.2. It is considered that hard TiC and TiB_2 particles impeded the motion of the soft Al matrix during rolling, and then increased the dislocation density in the Al matrix, thereby resulting in the hardness improvement [19].

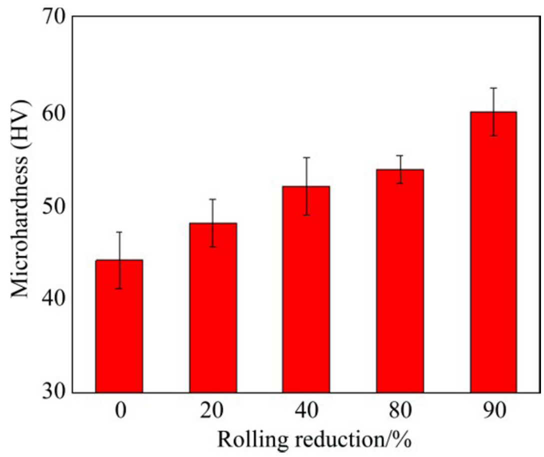

Fig. 6 Variation of microhardness of Al−TiB_2/TiC composites vs rolling reduction

Figure 7 illustrates the fracture surfaces of the Al−TiB_2/TiC composites after tensile tests to investigate the failure mechanisms. It shows that the as-cast composite exhibits a typical ductile facture with deep equiaxed dimples and the reinforcement particles are distributed at the bottom of dimples (Fig. 7(a)). It is known that ductile fracture occurred by the nucleation of microvoids, followed by their growth and coalescence [20]. The presence of particles at the bottom of some dimples can be considered as an evidence for the nucleation of voids from particle−matrix interfaces. After 40% rolling reduction, the sizes of dimples on the fracture surface are much smaller (Fig. 7(c)). However, the dimple sizes decreased slightly with further increasing rolling reduction. It is found that the presence of the particles can change the morphology of dimples in a manner that the dimples nucleated at the particle sites are deeper and larger than that nucleated in other regions. As shown in Fig. 2, it can be found that a microstructure with more uniform distribution of particles was obtained after 80% rolling reduction. Therefore, the shallow and small sized dimples can be found on the fracture surfaces of the composites with rolling reduction (Figs. 7(d) and 7(e)).

4 Conclusions

1) Al−5%TiB_2/TiC in situ composite with a homogenous microstructure was successfully prepared using the melt reaction method combined with hot rolling processing. The TiB_2 and TiC particles, ranging from 0.5 to 2 μm in size, agglomerated along the grain boundary in the as-cast composites.

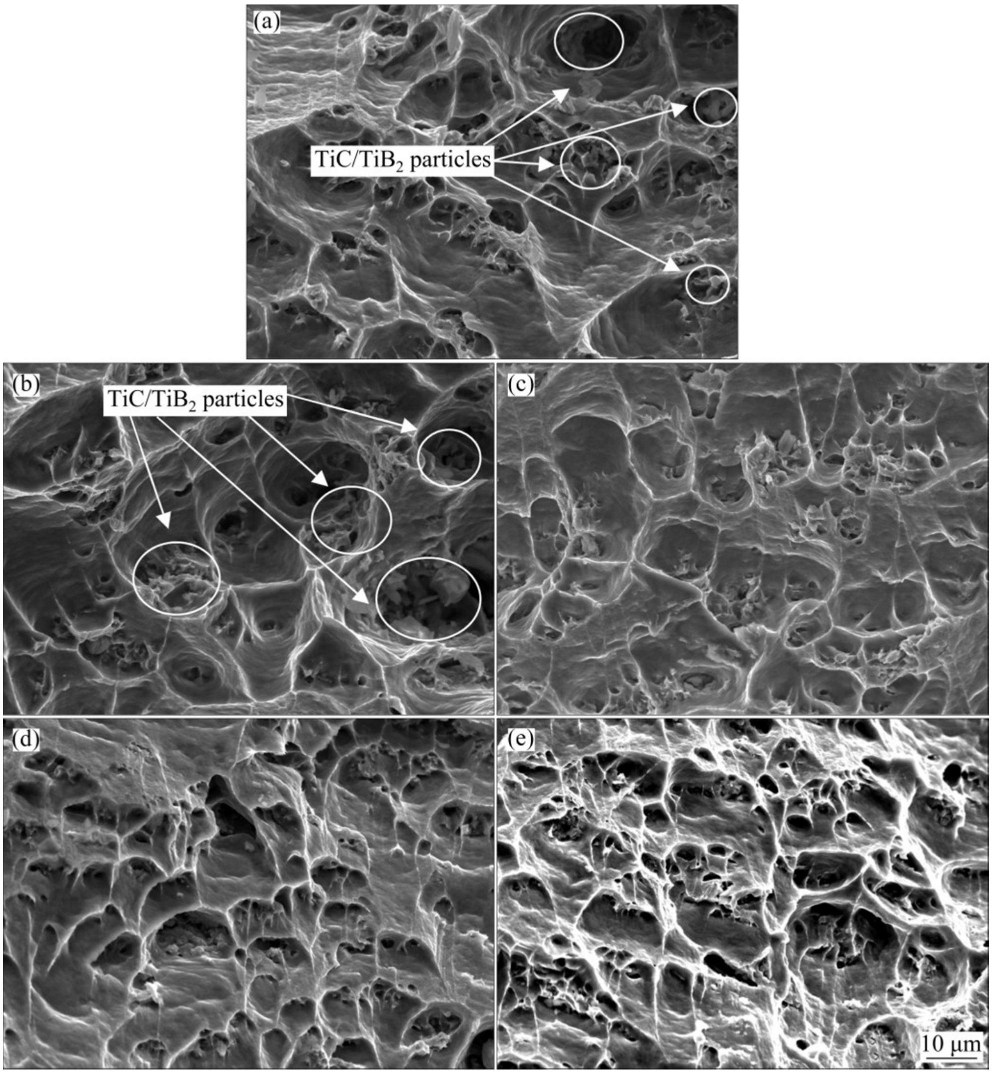

Fig. 7 Fractographs of Al−TiB_2/TiC composites in as-cast state (a), and after rolling with 20% (b), 40% (c), 80% (d) and 90% (e) reduction

2) The large particle agglomerations were effectively reduced after rolling process. The distribution of TiB_2 and TiC particles became much more uniform with increasing rolling reduction and a homogenous microstructure was obtained after 90% rolling reduction.

3) The mechanical properties of the Al−5%TiB_2/TiC in situ composites were significantly improved via the rolling process. The UTS and microhardness of the composites increased to 185.9 MPa and HV 59.8 after 90% rolling reduction, respectively, 140% and 35% higher than those of the as-cast ones. But the ductility dramatically decreased from 17.6% to 8.9%.

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高温轧制 Al−TiB_2/TiC 原位复合材料的显微组织及力学性能

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摘 要：在铝熔体中利用纯 Ti 与 Al−B−C 合金的原位反应成功制备出一种原位自生颗粒增强的 Al−TiB_2/TiC 复合材料，随后采用高温轧制改善其增强颗粒在基体中的分布与复合材料的力学性能。利用场发射扫描电镜表征复合材料在轧制变形过程中的显微组织演变过程，并通过拉伸和显微硬度试验测定复合材料的力学性能变化。结果表明：随着轧制压下量的增加，复合材料的显微组织得到明显改善，颗粒分布更加均匀；当压下量达到 90%时，复合材料的抗拉强度提高到 185.9 MPa，显微硬度达到 HV 59.8，与铸态样品相比分别提高了 140%和 35%。另外，基于拉伸样品的断口形貌分析了复合材料的强化机理。

关键词：原位复合材料；TiB_2/TiC 颗粒；轧制；力学性能

(Edited by Xiang-qun LI)