Advanced Composite Materials
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/tacm20

Synthesis of ZrO2 Particles Reinforced ZA25 Alloy Composites by Compocasting Process

Z. ácimovíc-Pavlovíc [a], K. T. Raíc [b], I. Bobíc [c] & B. Bobíc [d]

[a] Faculty of Technology and Metallurgy, University of Belgrade, Belgrade, Serbia
[b] Faculty of Technology and Metallurgy, University of Belgrade, Belgrade, Serbia;, Email: karlo@tmf.bg.ac.rs
[c] Faculty of Technology and Metallurgy, University of Belgrade, Belgrade, Serbia
[d] IHIS Research and Development Center, Belgrade, Serbia

Version of record first published: 02 Apr 2012.

To cite this article: Z. ácimovíc-Pavlovíc , K. T. Raíc , I. Bobíc & B. Bobíc (2011): Synthesis of ZrO2 Particles Reinforced ZA25 Alloy Composites by Compocasting Process, Advanced Composite Materials, 20:4, 375-384

To link to this article: http://dx.doi.org/10.1163/092430411X558512

VSP

Advanced Composite Materials 20 (2011) 375–384

ADVANCED COMPOSITE MATERIALS
brill.nl/acm

Synthesis of ZrO_2 Particles Reinforced ZA25 Alloy Composites by Compocasting Process

Z. Aćimović-Pavlović [a], K. T. Raić [a,*], I. Bobić [b] and B. Bobić [c]

[a] Faculty of Technology and Metallurgy, University of Belgrade, Belgrade, Serbia
[b] Vinca Institute of Nuclear Sciences, University of Belgrade, Belgrade, Serbia
[c] IHIS Research and Development Center, Belgrade, Serbia

Received 4 February 2010; accepted 3 January 2011

Abstract

Microstructures and compressive properties of Zn25Al3Cu/ZrO_2 particulate composites were studied. The composites were obtained by compocasting process through infiltration of 1 and 3 wt% ZrO_2 particles of different size into the semi-solid melt of the base alloy. The influence of reinforcing particles' size and quantity on microstructure and mechanical properties of the composites was examined. The composites have shown significant improvement of mechanical properties with respect to the base alloy. Increase in hardness and compressive yield strength of the composites was more expressed in the composites with coarse ZrO_2 particles.

Keywords

Metal matrix composites, ZA25 alloy, ZrO_2 strengthener, compocasting process, mechanical properties

1. Introduction

Metal matrix composites (MMCs) have several advantages over base alloys, which make them very useful as structural materials. The most obvious advantages are their high modulus, improved strength and enhanced strength-to-weight ratio. Among the discontinuously reinforced MMCs, particulate reinforced composites have attracted quite a lot of attention both in the industrial sector and the academic community.

There is an increasing need for knowledge concerning processing techniques and mechanical properties of particulate MMCs as their production methods become more sophisticated and commercial applications grow wider [1–4].

* To whom correspondence should be addressed. E-mail: karlo@tmf.bg.ac.rs
Edited by KSCM

 DOI:10.1163/092430411X558512

Zinc alloys with increased aluminum content (ZA alloys) started their development and wide application in the 1970s and mostly as alloys for casting. Besides excellent castability and high corrosion resistance, these alloys also have favorable mechanical and physical properties ratio, so they have often been used as substitute for other materials, such as grey iron, brass, bronze or some aluminum alloys, for different application scope. The most notable ZA alloys are designated as ZA12, ZA25 and ZA27 on the basis of their approximate aluminum content. These alloys are capable of replacing traditional bronze bearings, because of excellent bearing and wear resistance properties and low production cost. However, these alloys suffer from deterioration of mechanical properties at operating temperatures above 80°C [3]. It was shown that physical and some mechanical properties of MMCs with base ZA27 alloy reinforced with hard ceramic strengtheners [3–5] were better with respect to the matrix alloy at room temperature.

In view of these properties it was the aim of this work to obtain particulate composites with base ZA25 alloy using ZrO_2 particles as the reinforcement and to examine the influence of the reinforcing particles on microstructure and mechanical properties of the composite materials produced at room temperature. The composites were obtained by compocasting. This process is characterized by infiltration of reinforcing particles into the semi-sold melt of base alloys [4, 5]. The reinforcing material can be incorporated into metal matrix even though it is not wetted by the base alloy. The compocasting process has been used to produce ZA25 alloy based composites [6–9] with reinforcing ZrO_2 particles [9]. ZA25 alloy solidifies in a wide temperature range and is suitable for processing in the semi-solid state.

2. Experimental

2.1. Compocasting

ZA25/ZrO_2 composites were obtained by compocasting using ZrO_2 particles as strengtheners. The nominal composition (in wt%) of the base ZA25 alloy was as follows: 25 Al, 3 Cu and Zn remainder. The amount of infiltrated ZrO_2 particles was 1 and 3 wt%, while particles of two different sizes (approximately 10 and 100 μm) were used.

Size and quantity of the reinforcing particles as well as infiltration time and active mixing time in the compocasting process (for three series of experiments: A, B and C) are given in Table 1.

The compocasting was performed using the apparatus presented in Fig. 1(a), while hot-pressing of the obtained composites was done in a special pressing tool that is shown in Fig. 1(b).

The processing part of the apparatus consists of a laboratory electro-resistance furnace of 2 kW power and a mixer. The crucible (72 mm inner diameter) was made of Al_2O_3, and a thermocouple was placed within a vertical hole in the crucible wall.

Table 1.
Parameters of the compocasting process

Type of particles	ZrO_2		
Particles' size (μm)	10		100
Particles' quantity (wt%)	1 (A)	3 (B)	3 (C)
Infiltration time (min)	3	5	2
Active mixing time (min)	10		10

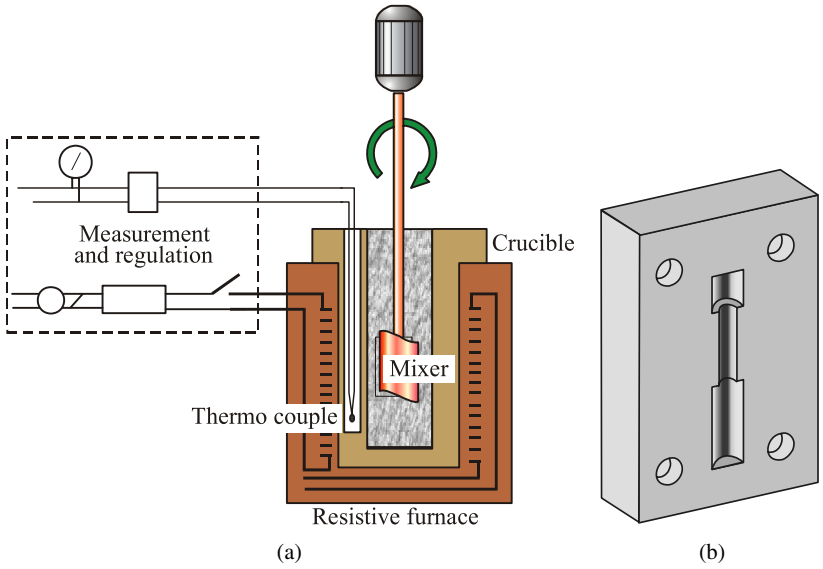


Figure 1. Schematic view: (a) apparatus for compocasting, (b) pressing tool. This figure is published in color in the online version.

A steel plate (100 × 38 × 2 mm^3) coated with aluminum-oxide was used as the active part of mixer (paddle stirrer).

ZA25 alloy was charged into the crucible of the electro-resistance furnace. Then the alloy was melted and overheated to 600°C to clean the slag from the melt surface. The temperature of the melt was then lowered to the operating temperature (468 ± 3°C). At this temperature the solid fraction in the alloy melt was approximately 30 wt%. After immersion of the paddle stirrer in the alloy, melt mixing was started and then infiltration of reinforcing particles into the semi-solid melt of the base alloy. Infiltration of the reinforcing particles was performed with mixing in approximately isothermal mode. When the mixing was completed, the composite material was poured into the steel mold preheated to 420°C. In order to reduce porosity of the composite castings, while preserving the morphology obtained at pouring, hot-pressing was done at 150 MPa operating pressure using the pressing tool illustrated in Fig. 1(b).

In order to determine the optimal mixing time, preliminary mixing of the composite mixture with 3 wt% ZrO_2 particles (10 μm particle size) was done in the first phase of the experiment. The active mixing time (time of mixing after termination of particle infiltration) was 10, 15 and 45 min, respectively. At certain intervals, a small specimen of the composite material was taken. It was established (by microscopic observation) that satisfactory distribution of reinforcing particles was achieved after 10 min while prolonged mixing time did not result in any improvement of the particle distribution. As a result of that finding, the active mixing time of ZrO_2 particles in all three series of experiments (A, B and C) was set at 10 min (Table 1). The reinforcing particles of different size and quantity were infiltrated into the semi-solid melt of the base alloy (Table 1). Distribution of ZrO_2 particles and their influence on the mechanical properties of the composites obtained were studied.

The change of stirrer rotation speed with time in all three series of experiments is shown in Fig. 2.

Line AB represents continuous increase in mixing rate from the initial (50 rpm) up to the operating rate (500 rpm). Line BC represents the value of mixing rate during thixoforming of ZA25 alloy semi-solid melt. The goal of thixoforming was to achieve the structure transformation of the base alloy. After thixoforming, the mixing rate was reduced to 250 rpm and infiltration of reinforcing ZrO_2 particles was performed at a constant mixing rate (line DE). When infiltration of the reinforcing particles was completed, the mixing rate was increased again to 500 rpm (line EF) and held constant at that value (line FG) during the next 10 min (active mixing time of the composite's semi-solid melt). After that, mixing was stopped (point G) and the melt of obtained composite was left to cool spontaneously up to pouring (point I). Line HI represents the time from the mixing termination up to the pouring out moment.

Structural and mechanical characteristics of the obtained composite materials were studied [9].

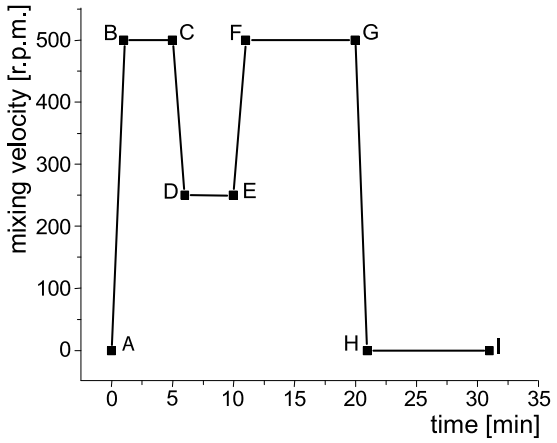


Figure 2. Mixing rate in dependence on time.

2.2. Microstructural Investigations

The samples of the composites were investigated in the as-cast state as well as after hot-pressing. After usual metallographic preparation, the microstructure was examined by a Zeiss Axiovert optical microscope (OM).

2.3. Compression Tests and Hardness

Mechanical properties of ZA25/ZrO_2 composites were tested on appropriate specimens. The specimens were obtained from the composite samples that were subjected to hot-pressing. The samples were machine cut and specimens for compression test [10] were obtained (6 mm in diameter and 8 mm in height, $h/d < 2$). Specimens of the same size were also machine cut from the base ZA25 alloy that had been obtained by standard melting and casting procedure in a laboratory electro-resistance furnace. Compression tests were performed using an Instron 1185 machine for mechanical testing at a compression speed of 1 mm/min. A detailed description of the testing procedure was given in [9]. Mechanical tests were performed in accordance with instructions given in [10].

The room temperature hardness tests were conducted using a hardness tester (Karl Frank GMBH). Five hardness readings were taken for each specimen at different locations.

3. Results and Discussion

3.1. Microstructure

Microstructure of the composite (A series) is shown in Fig. 3. Reinforcing particles are distributed between primary particles of the base alloy. The primary particles are of elliptic shape that indicates a change in their morphology during the compocasting process.

Microstructure of the composite (B series) is shown in Fig. 4. Different areas of the sample are presented (a, b, c) according to the scheme given in [9]. An even

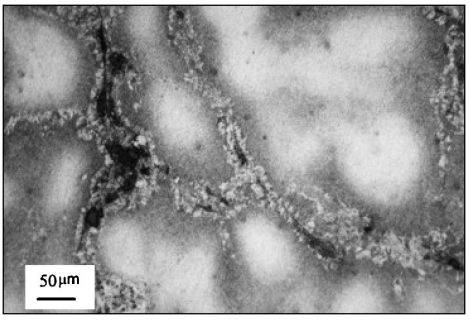


Figure 3. Microstructure of ZA25/ZrO_2 composite after hot-pressing. OM, etched. Sample A: 1 wt% ZrO_2, 10 μm particle size.

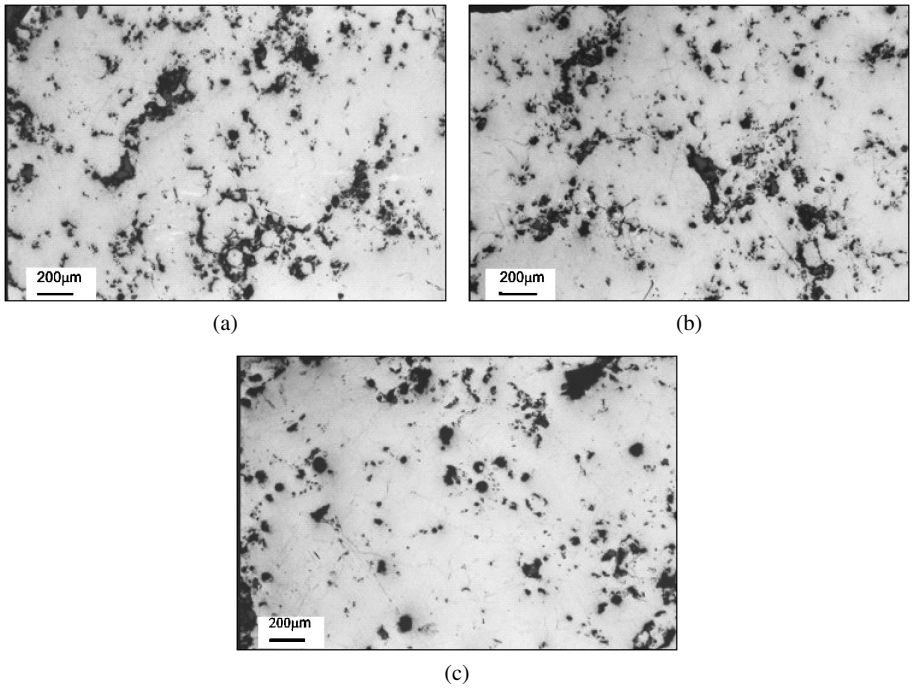
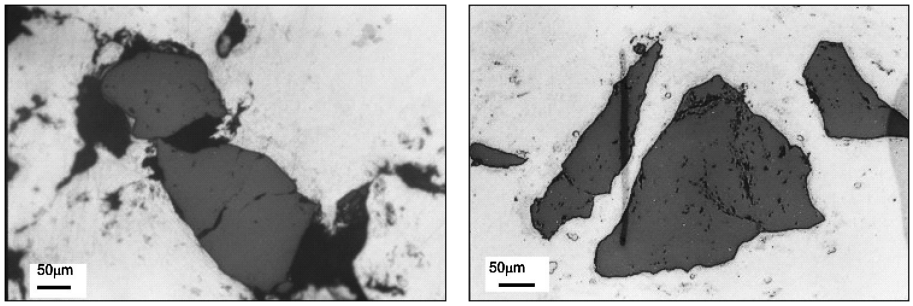


Figure 4. Microstructure of ZA25/ZrO_2 composite as-cast. OM, polished. Sample B: 3 wt% ZrO_2, 10 μm particle size. (a) Central part, (b) middle part, (c) outermost part.

distribution of reinforcing particles on the composite surface can be seen. A considerable tendency towards agglomeration of ZrO_2 particles was not noticed although some small clusters of B type [11] were observed. Besides, reinforcing particles do not produce chemical bonds with base ZA25 alloy. The agglomeration of ZrO_2 particles was noted in the composites when prolonged mixing time was applied [9].

The influence of hot-pressing on the structure of composites, and therefore on the mechanical properties, is demonstrated in Fig. 5. A pure mechanical bond was formed between reinforcing particles and the base alloy that is without any chemical reaction on the boundary surface.

A few cavities around ZrO_2 particles can be seen in Fig. 5(a). Due to a weak mechanical bond between the particles and the base alloy some particles have been seen to fall out from the as-cast sample of the composite during metallographic preparation. The observed cavities around reinforcing particles are potential places for appearance of micro-cracks. By hot-pressing in a hydraulic press, the sample with microstructure presented in Fig. 5(b) was obtained. Reinforcing particles and the base alloy are tightly bonded and the cavities around ZrO_2 particles were not observed. The mechanical tests performed confirmed a significant improvement in the composite's mechanical properties when the composites were subjected to hot-pressing.



(a) (b)

Figure 5. Microstructure of ZA25/ZrO_2 composite. OM, polished. Sample C: 3 wt% ZrO_2, 100 μm particle size. (a) As-cast, (b) after hot-pressing.

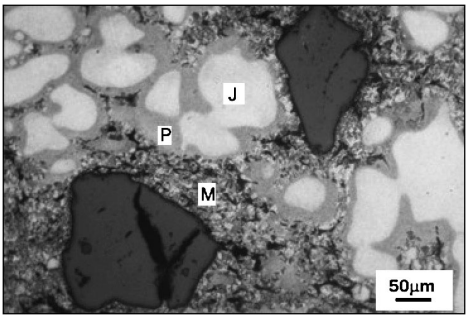


Figure 6. Microstructure of ZA25/ZrO_2 composite after hot-pressing. OM, etched. Sample C: 3 wt% ZrO_2, 100 μm particle size.

An etched microstructure of the composite (C series) is shown in Fig. 6. It can be seen that primary particles of the base alloy consists of dendrite core J (α-phase rich in aluminum) and dendrite periphery P ($\alpha + \eta$ phase mixture). The interdendritic space M is filled with η phase (a mixture of η crystals rich in zinc and detached dendrite branches). Reinforcing particles are located in the interdendrite phase and statistically distributed between the primary particles.

It can be seen that primary particles of the base alloy have been significantly modified during compocasting. They became elliptic in shape which means that primary dendrite structure was transformed. Due to the short mixing time the increase in size of primary particles was not large. This could possibly be explained by secondary crystallization of the primary particles on the reinforcing particles. When the reinforcing particles are brought into semi-solid melt of the base alloy a thin metal layer is formed upon these particles. This layer represents a kind of boundary surface that contributes to the structure refinement. The other explanation could be that broken dendrite branches when moving through the semi-solid melt make the growth of primary particles more difficult.

Table 2.
Mechanical properties of ZA25/ZrO_2 composites at room temperature

Material	Quantity of ZrO_2 particles (wt%)	Size of ZrO_2 particles (μm)	Compressive yield strength $Rp_{0.2}$ (MPa)	Hardness (HV)
ZA25 as-cast	0	–	420	115
ZA25/ZrO_2 composites (after hot-pressing)	1	10	444	125
	3	10	444	133
	3	100	592	175

3.2. Mechanical Properties

The results of testing mechanical properties of obtained composites (all three series of experiments: A, B, C) are given in Table 2. These results can be compared with the results for the base ZA25 alloy that are presented in the same table.

According to the results given in Table 2, the regularity in hardness values as a function of the quantity and size of reinforcing particles cannot be observed. However, a clear conclusion can be drawn that hardness of the composites is higher than hardness of the base ZA25 alloy. This increase in composite hardness was expected considering the high hardness of ZrO_2 particles. The second explanation for higher hardness of the composites is in the fact that hard reinforcing particles act like obstacles during movement of dislocations and make the plastic flow of material more difficult. It can be expected that this increase in composite hardness is to be maintained at higher operating temperatures. Having in mind the importance of these properties for practical application of the composites, this phenomenon should have been studied additionally.

Llorca and Elices [12] have studied the deformation and failure mechanism of composites. They observed that the composites failed by the progressive fracture of the reinforcing particles present in the microstructure and the final fracture of the composites results from the crack propagation through the matrix between the particles. On the other hand, mechanical properties of the cast ZA25 alloy/zircon composites are considerably influenced by the presence of zircon particles [5].

It can be seen in Table 2 that compressive yield strength of the composites (all three series of experiments) is higher with respect to compressive yield strength of the base alloy. This increase in compressive yield strength of the composites can be explained by the influence of numerous factors: bond strength between the base alloy and reinforcing particles, distribution of reinforcing particles in the base alloy, mechanical properties of the base alloy and added reinforcing particles, etc. The results have also shown that particle size influenced the compressive yield strength of the composites. With an increase in particle size, values of compressive yield strength increase as well. This could be explained considering that hard and brittle reinforcing particles impede the motion of dislocations through the soft matrix alloy and make the plastic flow of material more difficult. This increase in compres-

sive yield strength of obtained composites could be the motivation for their wider application in future.

4. Conclusion

Synthesis of ZA25/ZrO_2 particulate composites was performed by compocasting process. Structural and mechanical characteristics of the composite materials obtained were investigated. By metallographic studies it was revealed that infiltration of reinforcing ZrO_2 particles into matrix ZA25 alloy was completed successfully. The particles do not form chemical bonds with base alloy. An even distribution of reinforcing particles has been achieved although some small clusters of ZrO_2 particles were observed. This effect was more expressed in the composites with small particles (10 μm). During the compocasting process, primary particles of the base alloy became elliptic in shape which means that the dendritic structure was transformed into a non-dendritic one.

Obtained ZA25/ZrO_2 composites have shown significant improvement of mechanical properties with respect to the base alloy. Observed increase in hardness and compressive yield strength of composites was more expressed when using large ZrO_2 particles (100 μm). Higher bond strength between reinforcing particles and the base alloy was attained by hot-pressing of the composites that also caused increase in mechanical properties.

In view of the above it can be concluded that ZA25/ZrO_2 particulate composites with predicted properties could be obtained by optimizing the technological parameters of the compocasting process. Structural and mechanical properties of the composites indicate good prospects for a compocasting process to be used in producing composite materials, but also point out the need for further investigations with the goal of the process development and wider application in practice.

Acknowledgement

This work was supported by the Ministry of Science and Technology Development of Republic of Serbia.

References

1. M. D. Huda, M. S. J. Hashmi and M. A. El-Baradie, MMCs: materials, manufacturing and mechanical properties, in: *Key Engineering: Materials*, G. M. Newaz, H. Neber-Aeschbacher and F. H. Wohlbier (Eds), pp. 37–64. Trans Tech Publications, Aedemansdorf, Switzerland (1995).
2. R. J. Barnhurst, Guidelines for designing zinc alloy bearings — a technical manual, *SAE Trans* **97**, 164–170 (1988).
3. H. X. Zhu and S. K. Liu, Mechanical properties of squeeze cast zinc alloy matrix composites containing alpha-alumina fibers, *Composites* **24**, 437–442 (1993).
4. I. Bobic, M. T. Jovanovic and N. Ilic, Microstructure and strength of ZA-27-based composites reinforced with Al_2O_3 particles, *Mater. Letts* **57**, 1683–1688 (2003).

5. S. C. Sharma, B. M. Girish, D. R. Somashekar, R. Kamath and B. M. Satish, Mechanical properties and fractography of zircon-particle-reinforced ZA-27 alloy composite, *Compos. Sci. Technol.* **59**, 1805–1812 (1999).
6. I. Bobić, Z. Miskovic, M. T. Jovanovic, B. Lukic, S. V. Markovic, Z. Acimovic, D. N. Krstic, The effects of mold preheating and heat tretment on the microstructure and tensile properties of ZnAl25Cu3 as-cast alloy, *J. Metallurgy* **3**, 193 (1997).
7. A. Vencl, I. Bobic and Z. Miskovic, Effect of thixocasting and heat tretment on the tribological properties of hypereutectic Al-Si-alloy, *Wear* **101016**, 5–11 (2007).
8. I. Bobić, Development of metal forming process in semi-solid state (rheocasting and compocasting process) and process controlling effects on quality of ZnAl25Cu3 alloy products, *PhD Thesis*, Faculty of Technology and Metallurgy, Univesity of Belgrade (in Serbian) (2002).
9. S. Kastelec Macura, The effects of mixing parameters on the quality of ZnAl27Cu3 based alloy composite obtained through compocasting, *MSc Thesis*, Faculty of Tecnology and Metallurgy, University of Belgrade (in Serbian) (2007).
10. H. A. Kuhn, Uniaxial compression testing, in: *ASM Handbook, Vol. 8: Mechanical Testing and Evaluation*, S. D. Henry and W. Scott Jr. (Eds), p. 339. ASM International Materials Park, OH (2000).
11. T. C. Tszeng, The effects of particle clustering on the mechanical behavior of particle reinforced composites, *Composites Part B* **29**, 299–308 (1998).
12. J. Llorca and M. Elices, Fracture resistance of fiber reinforced ceramic matrix composites, *Acta Metall. Mater.* **38**, 2485–2492 (1990).