

From the values of σ_t for uniaxial tension and σ_c for the compression test, it results

$$\alpha = \frac{2}{\sqrt{3}} \frac{|\sigma_c| - \sigma_t}{|\sigma_c| + \sigma_t}, \quad \kappa = \frac{2}{\sqrt{3}} \frac{|\sigma_c| \sigma_t}{|\sigma_c| + \sigma_t} \quad (10)$$

For $\epsilon_0 = 0.1\%$, $\sigma_t = 16.0$ MPa and $|\sigma_c| = 23.8$ MPa. From these stresses, $\alpha = 0.113$ and $\kappa = 11.05$ MPa are obtained. The yield condition, resulting in an ellipse, is plotted in Fig. 10. The torsion results show slight deviations from the ellipse only.

Proposal for finite deformations under an electric field: In the preceding considerations, multiaxiality was determined for unpoled material and in the absence of any electric field. In order to allow considerations for the more general case of poled materials under an electric field, we propose the following rough estimation: Determine the uniaxial stress strain curves as done in Section 2.1 under the relevant poling and electric field conditions, use these data to compute the parameters α and κ of Equation 10, and construct the biaxial deformation diagram according to Fig. 10a. This may also be done for larger strains. A result is plotted in Fig. 10b for a plastic strain of $\epsilon_{pl} = 0.5\%$ and $E = 0$ and -0.5 kV/mm, with the full circles taken from Figs. 2 and 3.

In the present study, a commercial soft PZT is investigated in tests at varying electric fields. Strain measurements in pure tension and compression tests were carried out for the unpoled and the poled material. The deformation under biaxial loading is studied in torsion tests carried out on thin-walled tubes made of the unpoled PZT. These tests allow to determine the non-symmetric yield condition for the plastic deformations and to describe it with the Drucker-Prager criterion.

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- [1] H. Cao, A. G. Evans, *J. Am. Ceram. Soc.* **1993**, 76, 890.
- [2] A. Schäufele, K.-H. Härdtl *J. Am. Ceram. Soc.* **1996**, 79, 2637.
- [3] T. Fett, S. Müller D. Munz, G. Thun, *J. Mat. Sci. Letters* **1998**, 17, 261.
- [4] W. Chen, C. Lynch, *J. Eng. Mat. Techn.* **2001**, 123, 169.
- [5] T. Fett, D. Munz, G. Thun, Multiaxial deformation behavior of PZT from torsion tests, *J. Am. Ceram. Soc.*, submitted.
- [6] T. Fett, D. Munz, G. Thun, *J. Mater. Sci. Letters* **2000**, 19, 1921.
- [7] T. Fett, D. Munz, G. Thun, Stress-strain behaviour of a soft PZT ceramic under tensile and compression loading and a transverse electric field, *Ferroelectrics*, submitted.
- [8] T. Fett, D. Munz, *J. Test. Evaluat.* **2000**, 28, 27.
- [9] A. Nadai, *Theory of Flow and Fracture of Solids (Chapter 22)*, Vol.1, McGraw-Hill, New York, **1950**.
- [10] T. Fett, D. Munz, G. Thun, *J. Am. Ceram. Soc.* **1998**, 81, 269.
- [11] A. Kolleck, G.A. Schneider, F.A. Meschke, *Acta Mater.* **2000**, 48, 4099.
- [12] W. Prager, *An Introduction to Plasticity*, Addison-Wesley, Amsterdam, **1959**.

Effect of the Temperature of Molten Magnesium on the Thermal Explosion Synthesis Reaction of Al–Ti–C System for Fabricating TiC/Mg Composite**

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Considerable efforts have been devoted to the development of novel, lightweight engineering materials during the last decades.^[1] Metal matrix composites (MMCs) are a class of materials that seek to combine the high strength and stiffness of a ceramic with the damage tolerance and toughness provided by a metal matrix.^[2] Recently, there has been a great deal of interest in the development of particulate reinforced magnesium MMCs due their unique combination of properties such as a high specific modulus and high specific strength.^[3–7] The reinforcement based on TiC particulates is attractive for the magnesium alloy matrix because of its good wettability with magnesium.^[8] Importantly, novel processing techniques based on in-situ synthesis of magnesium MMCs have emerged in recent years.^[9–11] In-situ techniques involve exothermic chemical reactions among elements or between an element and a compound resulting in formation of a very fine and thermodynamically stable reinforcing ceramic phase within a metal matrix.^[9–12] This consequently provides thermodynamic compatibility at the metal–reinforcement interface.^[12–13] Since the reinforced surfaces are also likely to be free of contamination, a stronger matrix–reinforcement bond can be achieved.^[13–14]

During the in-situ composite fabrication, however, the “inert” matrix acts as a diluent, which may make the propagation of the combustion wave unstable owing to the strong heat dissipation in the “inert” metal matrix.^[8,15] In this case, therefore, if the phases formed are different from the thermodynamically stable phase, they might remain as metastable phases in the product, leading to inhomogeneous microstructures. The mechanisms responsible for the in-situ formed TiC/Mg composites in the Al–Ti–C system are still not well

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understood. The purpose of the present study is to investigate the effect of the temperature of molten magnesium on the thermal explosion synthesis reaction of the Al-Ti-C system for fabricating a TiC/Mg composite.

The preforms in this study were made from commercial powders of 30 wt.-% aluminum (98.0 % purity), 56 wt.-% titanium (99.5 % purity), and 14 wt.-% graphite (99.9 % purity), all with an average particle size less than 44 μm . Titanium and carbon powders were at a ratio corresponding to that of stoichiometric TiC. After being sufficiently mixed, the blends were pressed into cylindrical preforms (20 mm diameter and 15 mm length) by using a stainless steel die with two plungers. The green preforms were pressed uniaxially at pressures ranging from 70–75 MPa to obtain densities of 75 % \pm 2 % theoretical density. According to our previous study,^[10] the preforms were preheated in a vacuum oven at 450 °C for 3 h to eliminate moisture. About 1 kg of commercial AZ91D magnesium alloy melts were prepared in a graphite crucible in an electric resistance furnace under a SF₆/Ar protective atmosphere at 700, 750, 800, 850, and 900 °C, respectively. The preforms with a preheat temperature of 450 °C were then added into the prepared molten magnesium. The starting point of the reaction can be determined by observing the evidence of a dazzling light that originates from the reacted preform. When the exothermic reaction was over, the magnesium melt was held at that temperature for 10 min. Stirring was carried out with a graphite stirrer for 20 min. to assist the dispersion of the generated TiC particulates into the molten magnesium. The composite melts were cast into a copper mold to produce ingots of Φ 55 \times 100 mm.

Microstructures and phases of the composites were investigated by using scanning electron microscopy (SEM) (Model JSM-5310, Japan) and XRD (Model D/Max 2500PC Rigaku, Japan).

According to,^[10] the exothermic reaction that occurs between molten magnesium and preform is a self-propagating high-temperature synthesis (SHS) reaction with a simultaneous combustion mode. In this process, the entire preform is heated by the heat of molten magnesium at a constant rate to the ignition temperature (T_i) to initiate the reaction. It can also be considered a thermal explosion synthesis (TES) mode reaction, since the exothermic reaction is initiated throughout the entire preform.^[16,17]

Figures 1 and 2 show the SEM microstructures and XRD patterns of TiC/AZ91D composites utilizing the TES reaction between molten magnesium and Al-Ti-C preforms with the temperature of molten magnesium of 700, 750, and 900 °C, respectively. When the temperature of molten magnesium is 700 °C in addition to fine TiC particulate, a little amount of blocky TiAl_x is also found in the composite. When the temperature is increased from 750 °C to 900 °C, only TiC is found in the composites.

Figure 3 shows the changes of incubation time for the TES reaction on the molten magnesium temperature. The duration of the incubation time for the TES reaction is taken from the

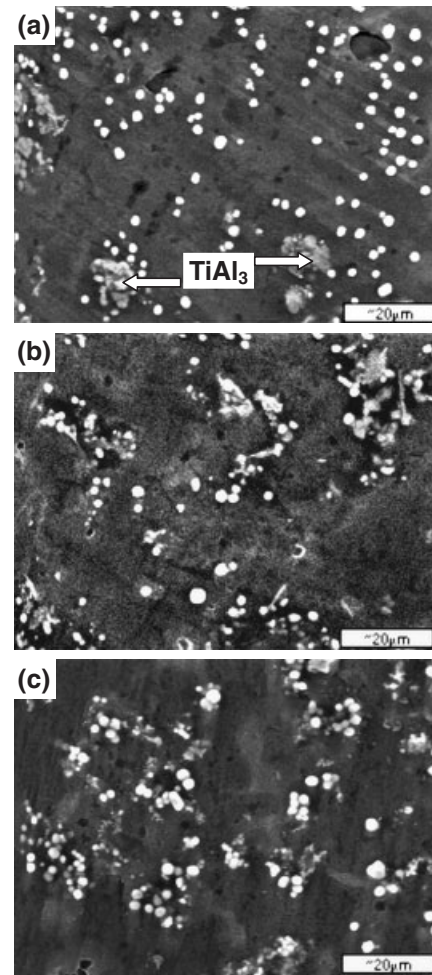


Fig. 1. SEM microstructures of TiC/AZ91D composites utilizing the TES reaction between molten magnesium and Al-Ti-C preforms with a temperature of molten magnesium of a) 700 °C, b) 750 °C, and c) 900 °C.

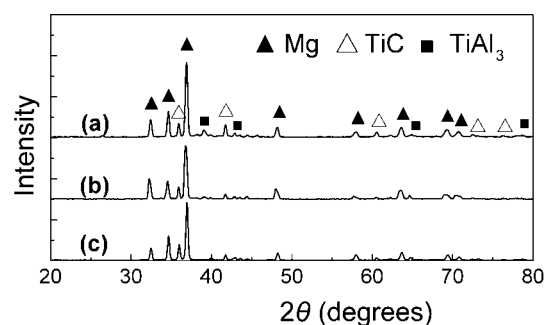


Fig. 2. XRD patterns of TiC/AZ91D composites utilizing the TES reaction between molten magnesium and Al-Ti-C preforms with a temperature of molten magnesium of a) 700 °C, b) 750 °C, and c) 900 °C.

point of putting the preform in the molten magnesium to the starting point of exothermic reaction. In this study, the incubation time is measured by the second counter. The temperature profile of a preform undergoing the TES reaction in molten magnesium should be determined by the heat released from the compound formation as well as heat transfer from or to the reaction environment (molten magnesium). The degree of

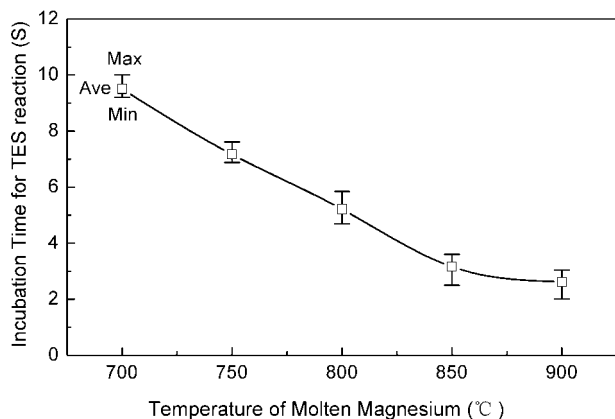


Fig. 3. The changes of incubation time for the TES reaction on the molten magnesium temperature.

precombustion reaction, therefore, should have a very close relationship with the temperature profile. In this study, the precombustion reaction is the reaction that occurs during the duration of the incubation time for the TES reaction process.

The heating rate to ignition temperature of the preform increases as the temperature of molten magnesium is increased. Lee et al.^[16] have studied the effects of heating rate on the temperature profile of the TES mode reaction in a furnace under an argon protective atmosphere. Based on the experiment, they concluded that the volume fraction of the reactants consumed decreases as the heating rate is increased during the precombustion duration in Al-Ti system. According to our previous study,^[10] the addition of aluminum provides an easier route for the TiC formation in the Al-Ti-C preform. During the TES process, the reactions between Al and Ti to form TiAl_x (TiAl, TiAl₃) compounds occur initially, which further react with C to form a more thermodynamically stable TiC.^[10] It is believed that the Al-Ti reaction is initiated by the heat of molten magnesium; however, further TiAl_x-C reaction is ignited by the heat release of the Al-Ti reaction. Therefore, the Al-Ti reaction is a predominant reaction during the precombustion duration in the incubation time for the TES reaction. Philpot et al.^[18] noted that the phases formed during the precombustion reaction are not always the same as the intended (thermodynamically stable) phase, because reaction kinetics determines which phase will form during the reaction. When the temperature of the molten magnesium is 700 °C, the heating rate to the preform is relatively lower. In this case, a significant fraction of reactants is consumed during the precombustion duration, forming TiAl_x compounds, which might remain as metastable phases in the product. The TiAl_x phases were found in the composite, as shown in Figures 1a and 2a, confirming that the existence of the precombustion reaction did occur between Al and Ti in the Al-Ti-C preform and molten magnesium.

Depending on the increase in the temperature of molten magnesium, the incubation time for the TES reaction is decreased exponentially because the increase in the molten magnesium temperature reduces the time taken to reach the ignition conditions such as the ignition temperature. Todes

reported that the adiabatic induction period of TES (t_{ad}) at T_i was described as follows.^[12,19-20]

$$t_{ad} = \frac{RT_i^2}{E} \frac{c_p}{Qk_0} \exp(E/RT_i) \quad (1)$$

where E is the activation energy, $J\ mol^{-1}$; c_p is the thermal capacity, $J\ K\ mol^{-1}$; Q is the heat of reaction, $J\ mol^{-1}$; R is the gas constant, $8.314\ J\ mol^{-1}\ K^{-1}$; T_i is the ignition temperature, K ; and k_0 is the pre-exponential factor of the homogeneous reaction, s^{-1} (It should be noted that the $c\rho$ in^[12,19] may be a typographical error, and they actually mean c_p in Equation 1).

From Equation 1, it follows that the adiabatic induction period of TES (t_{ad}) has an inversely proportional relationship with the reaction heat (Q). Furthermore, the reaction heat increases with the increase in the volume fraction of the reactants, which are a source of exothermic heat. According to,^[16] the volume fraction of the reactants consumed decreases as the heating rate is increased during the precombustion duration, and another result of this is that the reaction heat increases as the temperature of molten magnesium is increased during the combustion duration. Consequently, the adiabatic induction period of the TES (t_{ad}), which is the same as the incubation time for the TES reaction, has an inversely proportional relationship with the volume fraction of the reactants. As a result, the incubation time decreases as the temperature of molten magnesium is increased. This relationship is in accordance with the result of this experiment, as shown in Figure 3.

Based on the experimental result, higher temperature of molten magnesium results in higher conversion of the reactants in the Al-Ti-C preform to the thermodynamically stable TiC particulate during the TES reaction process. This phenomenon should be due to the formation of a lesser amount of precombustion products and lower incubation time.

The duration of the incubation time for the TES reaction of the Al-Ti-C preform decreases as the temperature of molten magnesium is increased. Higher temperature of molten magnesium results in higher conversion of the reactants to the thermodynamically stable TiC particulate during the TES reaction process.

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- [1] J. C. Li, Z. K. Zhao, Q. Jiang, *Adv. Eng. Mater.* **2003**, *5*, 119.
- [2] T. M. T. Godfrey, P. S. Goodwin, C. M. Ward-Close, *Adv. Eng. Mater.* **2000**, *2*, 85.
- [3] F. Chmelik, F. Moll, J. Kiehn, P. Lukac, B. L. Mordike, K.-U. Kainer, *Adv. Eng. Mater.* **2000**, *2*, 600.
- [4] A. LUO, *Metall. Mater. Trans.* **1995**, *A26*, 2445.
- [5] M. Svoboda, M. Pahutova, K. Kucharova, V. Sklenicka, T. G. Langdon, *Mater. Sci. Eng.* **2002**, *A324*, 151.
- [6] S. Jayalakshmi, S. V. Kailas, S. Seshan, *Compos.* **2002**, *A33*, 1135.
- [7] S. F. Hassan, M. Gupta, *J. Alloy. Compd.* **2002**, *345*, 246.
- [8] Q. C. Jiang, X. L. Li, H. Y. Wang, *Scripta. Mater.* **2003**, *48*, 713.

- [9] Q. C. Jiang, H. Y. Wang, J. G. Wang, Q. F. Guan, C. L. Xu, *Mater. Lett.* **2003**, *57*, 2580.
- [10] H. Y. Wang, Q. C. Jiang, X. L. Li, J. G. Wang, *Scripta Mater.* **2003**, *48*, 1349.
- [11] M. A. Matin, L. Lu, M. Gupta, *Scripta Mater.* **2001**, *45*, 479.
- [12] I. H. Song, D. K. Kim, Y. D. Hahn, H. D. Kim, *Scripta Mater.* **2003**, *48*, 413.
- [13] B. S. S. Daniel, V. S. R. Murthy, G. S. Murty, *J. Mater. Process. Technol.* **1997**, *68*, 132.
- [14] S. C. Tjong, Z. Y. Ma, *Mater. Sci. Eng.* **2000**, *R29*, 49.
- [15] I. Gotman, M. J. Koczak, E. Shtessel, *Mater. Sci. Eng.* **1994**, *A187*, 189.
- [16] S. H. Lee, J. H. Lee, Y. H. Lee, D. H. Shin, *Mater. Sci. Eng.* **2000**, *A281*, 275.
- [17] J. J. Moore, H. J. Feng, *Prog. Mater. Sci.* **1995**, *39*, 243.
- [18] K. A. Philpot, Z. A. Munir, J. B. Holt, *J. Mater. Sci.* **1990**, *22*, 159.
- [19] A. G. Merzhanov, A. E. Averson, *Combust Flame* **1971**, *16*, 89.
- [20] O. M. Todes, *Zh. Fiz. Khim.* **1933**, *4*, 71.

composites (MMCs) are advanced materials that have been one of the hot fields of materials research.^[1,2]

MMCs, especially aluminum- and titanium-based materials, have a high potential for advanced structural applications in which high specific strength and modulus, as well as good elevated-temperature resistance, are important. Particulate-reinforced MMCs are of special interest because of their ease of fabrication, low cost, and more isotropic properties. The properties of MMCs are controlled by the size and volume fraction of the reinforcement phase as well as by the nature of the matrix–reinforcement interface: an optimum set of mechanical properties tends to be obtained when fine and thermally stable ceramic particulates are dispersed in a metal matrix.

Traditionally, MMCs have been produced by processing techniques such as powder metallurgy,^[1,2] preform infiltration,^[3] spray deposition, mechanical alloying, and various casting technologies, such as squeeze casting, rheocasting, and comocasting.^[4–8] Such methods are based on the addition of the particulate reinforcement to the matrix material in molten or powder form. The scale of the reinforcing phase is limited by the starting-particle size, which is typically of the order of microns to tens of microns, and rarely less than 1 μm .

In traditional MMC processing it is often necessary to improve the wetting between the molten metal and the particulate (which is typically poor) in order to obtain a good bond between the matrix and reinforcement. This problem can be resolved by adding a strongly reactive alloying element such as Mg or Li, or by coating the particulate. Mechanical stirring or pressure infiltration can also be helpful. Other difficulties, such as uneven distribution of reinforcements, interfacial reactions between the matrix and reinforcements, and control of volume fraction are often encountered during the fabrication of MMCs. Such factors can adversely affect the properties of the product.

In recent years, new processing techniques based on in situ production of MMCs have emerged. In situ techniques involve a chemical reaction resulting in the formation of a very fine and thermodynamically stable reinforcing ceramic phase within a metal matrix. As a result, this provides thermodynamic compatibility at the matrix–reinforcement interface. The reinforcement surfaces are also likely to be free of contamination and, therefore, a stronger matrix–dispersion bond can be achieved. Some of these technologies include DIMOXTM, XD, PRIMEMTM, reactive gas infiltration, high-temperature self-propagating synthesis (SHS), liquid–solid or solid–gas–liquid reactions,^[9–14] and plasma in situ MMCs.^[15]

Thus in situ composites are a new topic of interest in MMC research. They have some advantages over traditional MMCs, such as excellent interfacial bonding and easy production technology. In situ hybrid composites may contain many kinds of reinforcing phases and possess excellent properties, so they are another new research interest.

Fabrication, Microstructure, and Mechanical Properties of Ti/Al Composite**

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The demand for advanced materials in modern industry and technologies is increasing, because pure metals and simple alloys can not meet the needs of society. Metal matrix

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