

In situ processing of dense Al_2O_3 –Ti aluminide interpenetrating phase composites

D. Horvitz^a, I. Gotman^{a,*}, E.Y. Gutmanas^a, N. Claussen^b

^aDepartment of Materials Engineering, Technion, Haifa 32000, Israel

^bAdvanced Ceramics Group, Technische Universität Hamburg-Harburg, 21071 Hamburg, Germany

Received 11 January 2001; received in revised form 26 June 2001; accepted 9 July 2001

Abstract

Self-propagating high-temperature synthesis (SHS) of compacted blends of nano-size TiO_2 and micron-size Al powders was used to fabricate in situ alumina–TiAl/Ti₃Al interpenetrating phase composites. Combustion wave propagation and pressureless or pressure-assisted thermal explosion (TE) modes have been investigated, and samples' temperature in the course of combustion synthesis has been accurately monitored. The ignition of self-sustained reaction in the system studied occurred at temperatures well above the melting point of Al, and temperature evolution in the samples was affected by interfacial barriers and by heat transfer to the surrounding ambience. The application of a moderate pressure (~ 50 MPa) during thermal explosion at 950 °C yielded near fully dense (up to 98% TD) Al_2O_3 –TiAl/Ti₃Al composites with fine micron size interpenetrating ceramic and intermetallic networks. The aluminide component had a very fine submicron $\gamma + \alpha_2$ lamellar microstructure. The TE procedure performed under uni-axial pressure between press rams (reactive forging) seems especially attractive due to the extremely short processing cycle (several minutes) and near-net-shape capability. This approach is compared with reactive hot pressing (RHP) where only partial conversion of reagents into products was observed after one hour exposure at 950 °C. © 2002 Elsevier Science Ltd. All rights reserved.

Keywords: Al_2O_3 ; Composites; Microstructure-final; Porosity; Self-propagating high-temperature synthesis (SHS); TiAl

1. Introduction

Since first proposed almost a decade ago,¹ metal- and intermetallic-oxide interpenetrating phase composites (IPC) have been drawing considerable attention as potential materials for demanding structural applications. Due to the unique morphology of IPC, i.e. a continuous matrix network penetrated by a continuous reinforcement network, the attractive features of each constituent may be utilized to their best, giving rise to truly remarkable properties of the composite as a whole.

Composites with interpenetrating networks are difficult (if not impossible) to fabricate using traditional PM routes developed for the fabrication of composites with continuous matrices discontinuously reinforced by fibers, whiskers or particles. The most common way of producing IPCs is, therefore, by infiltrating liquid metal into a porous ceramic preform.² This method, however,

is essentially restricted to the manufacturing of Al-containing composites whose high temperature application is limited due to the low melting temperature of Al. An approach that may allow us to synthesize a significantly wider range of interpenetrating phase composites is in situ processing. Reactively formed matrices and reinforcements of in situ composites are completely different from the starting reagents, they are thermodynamically compatible and are often arranged as interpenetrating networks.³

The present work deals with in situ processing of Al_2O_3 /TiAl ceramic/intermetallic interpenetrating phase composite. The advantage of intermetallic containing Al_2O_3 composites with interpenetrating networks is the fact that the Al_2O_3 matrix still dominates the thermo-mechanical behavior, i.e. wear resistance, environmental stability, high temperature strength, etc., while the intermetallic phase improves toughness by crack bridging. TiAl has been chosen as an intermetallic reinforcement due to the favorable combination of low density, high melting temperature and excellent oxidation resistance. Recently, such alumina–aluminide alloys (3A) have

* Corresponding author. Tel.: +972-4-829-2112; fax: +972-4-832-1978.

E-mail address: gotman@tx.technion.ac.il (I. Gotman).

been manufactured by reaction sintering of attrition milled powder blends consisting of cheap raw materials like metal oxides (e.g. TiO_2 , Fe_2O_3 , Nb_2O_5 , etc.) and aluminum,^{4,5} or by reactive infiltration of Al into porous $\text{Al}_2\text{O}_3/\text{TiO}_2$ preforms.⁶ For the $\text{Al}_2\text{O}_3/\text{TiAl}$ composite, both approaches are based on the exothermic displacement reaction $3\text{TiO}_2 + 7\text{Al} \rightarrow 2\text{Al}_2\text{O}_3 + 3\text{TiAl}$, however in neither one the heat of reaction is utilized. On the other hand, the reaction is sufficiently exothermic to become self-sustained so that the system is amenable for self-propagating high-temperature synthesis (SHS).^{7,8} SHS is an energetically efficient means of in situ materials processing, its main limitation being the high porosity of the synthesized products. To overcome this limitation, several processing approaches combining SHS with the application of an external pressure have been proposed.^{9–13} The present authors were successful in fabricating a wide range of dense ceramic matrix composites (TiC/TiB_2 , TiN/TiB_2 , $\text{TiC}/\text{Ti}_5\text{Si}_3$, etc.) via SHS in its thermal explosion mode performed either in a constrained die, or under uniaxial pressure between the press rams (reactive forging).^{14,15} The manufacturing of $\text{Al}_2\text{O}_3/\text{Al}_x\text{Ti}_y$ products from TiO_2 –Al powder blends was reported only in passing. In the present paper, the results of pressure-assisted thermal explosion processing of dense $\text{Al}_2\text{O}_3/\text{TiAl}$ interpenetrating phase composites are reported and discussed in more detail. For comparison, the fabrication of similar composites via a related solid state in situ processing route—reactive hot pressing—is also discussed.

2. Experimental procedure

Homogeneous powder blends containing 30 mol% TiO_2 (anatase, $\leq 0.1 \mu\text{m}$) and 70 mol% Al ($< 5 \mu\text{m}$) were prepared by high energy attrition milling for 2 h. A low 4:1 balls-to-powder ratio was used for attrition milling in order to avoid mechanical alloying of powders. 70% dense rectangular ($16 \times 16 \times 20 \text{ mm}$) and cylindrical ($18 \text{ mm dia} \times 20 \text{ mm height}$) compacts were prepared from the blend by cold pressing. The microstructure of a green 3TiO_2 –7Al compact is shown in Fig. 1(a), and it can be described as Al particles ~ 0.5 – $5 \mu\text{m}$ in size surrounded by a continuous TiO_2 matrix.

Following compaction, the samples were either placed into a furnace preheated to 700 – 850°C or, alternatively, between the preheated (800 – 1000°C) rams of an Instron testing machine (Fig. 2). The temperature of the samples between the press rams was measured by two W-3Re/W-25Re thermocouples ($d = 0.2 \text{ mm}$) placed into drill holes reaching the sample center: one in the middle of the sample and the other at a distance of 2.5 mm from its upper edge (see Fig. 2). A computer card (National, Ltd.) provided accurate temperature monitoring ($\pm 2.5^\circ\text{C}$) with a time resolution of 0.001 s .

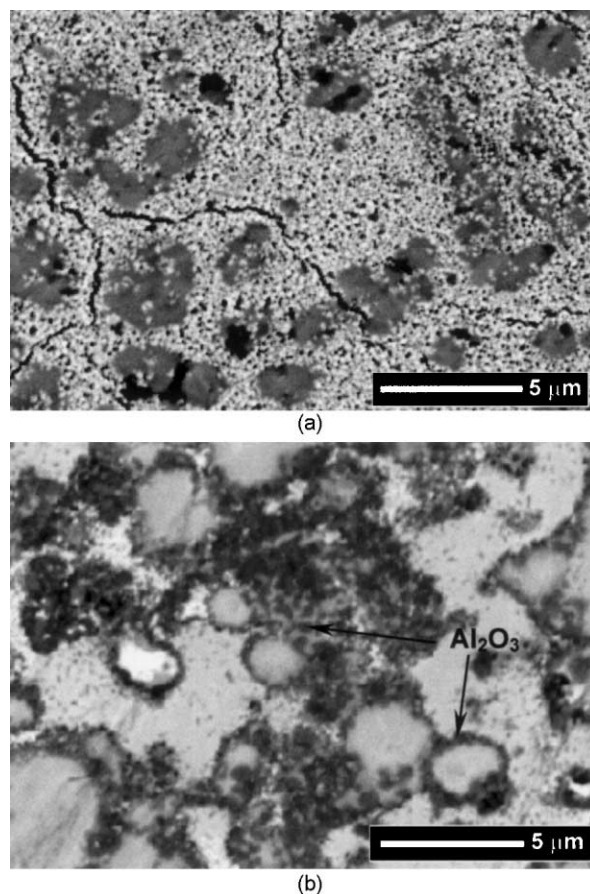


Fig. 1. A representative SEM image in backscattered electrons of a 3TiO_2 –7Al blend: (a) as compacted; (b) after reactive hot pressing at 950°C , 1 h.

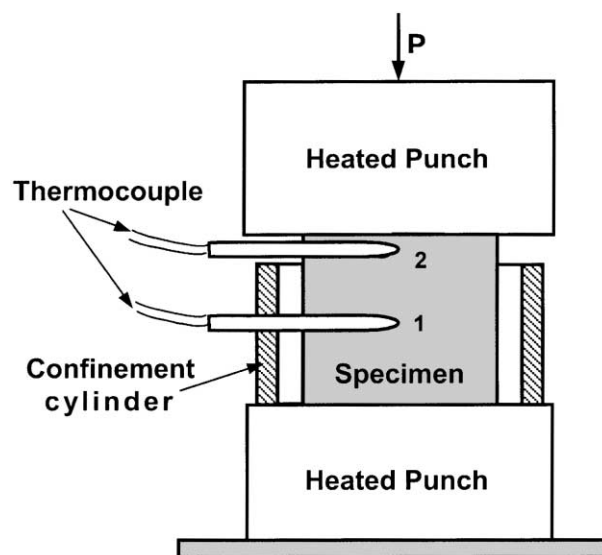


Fig. 2. A schematic of reactive forging—thermal explosion under pressure between preheated press rams.

When the preheating temperature (furnace or ram) was sufficiently high, thermal explosion (SHS) took place. For the compacts placed between the press rams,

a moderate uni-axial pressure of up to 60 MPa was applied immediately after the onset of thermal explosion. The sample was held under pressure for one minute and then unloaded. Plastic flow of the SHS reaction products under pressure at the high combustion temperature resulted in effective densification of the sample. The whole procedure of pressure-assisted thermal explosion between the press rams was performed in open air, and will be further referred to as reactive forging (RF). For the higher applied pressures, cylindrical samples were confined within hollow cylinders of the larger diameter (made of Inconel 718 superalloy) to limit outward flow of reaction product at the high combustion temperature and thereby prevent excessive flattening of the sample. The inner surface of the cylinder was sprayed with a Y_2O_3 /BN insulating layer to avoid reaction of cylinder walls with the sample. Due to the ease of RF processing and short exposure to pressure + temperature, up to 10 samples were fabricated in 30 min. If supplemented with forming dies, reactive forging will allow one to perform fast and effective reactive processing of dense near-net-shape in situ composites of complex shapes.

For comparison, a few experiments of thermal explosion in a constrained die under pressure¹⁶ were performed. A green compact was placed into a rigid die (Inconel 718) preheated to 950 °C after which a pressure of 50 MPa was immediately applied. Thermal explosion took place after pressure application when the sample was under the conditions of quasi-isostatic pressure. Compared to reactive forging, the processing cycle is much longer due to the need to assemble and heat the die prior to inserting the sample, and then cool and disassemble it in order to remove the sample after combustion synthesis.

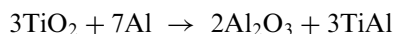
For a number of samples, reactive hot pressing (RHP) was performed at 950 °C under the pressure of 250 MPa. Green compacts were placed into a cold die (made of MA6000 Ni-based superalloy), loaded to 250 MPa and slowly heated (20°/min) to the synthesis temperature. After the processing temperature has been reached, the samples were held in the hot press for 1 h. As reported earlier, no thermal explosion of reactive blends takes place in the hot press, even if the RHP temperature is well above the SHS ignition temperature, T_{ig} .¹⁶ This is due to the partial transformation of reagents into products during the slow heating of the sample (and, correspondingly, long exposure at elevated temperatures below T_{ig}) typical of the hot pressing procedures. This reduces the exothermicity of the system and creates interfacial barriers, which, coupled with the enhanced heat transfer to the pressure die, prevents the synthesis reaction from becoming self-sustained thus making RHP a diffusion-controlled isothermal process.

After processing, the microstructure and phase composition of the synthesized composites were character-

ized employing X-ray diffraction (XRD) and scanning electron microscopy (SEM) with energy dispersive analysis (EDS). Density of the materials obtained was evaluated and when sufficiently high, measured by the Archimedes method.

3. Results and discussion

The 3:7 anatase-to-aluminum molar ratio in the reagent blend has been chosen to yield the Al_2O_3 -TiAl final product after the synthesis process has been completed according to reaction:



The reaction is moderately exothermic, and the calculation of the adiabatic temperature yields the value of $T_{ad} = 1733$ K, i.e. the melting temperature of TiAl. Since T_{ad} exceeds 1700 K, it is expected that combustion wave in this system can propagate at room temperature (without preheating).

According to the XRD analysis [Fig. 3(a)], the phase composition of the starting $3TiO_2$ (anatase)-7Al powder blend remains unchanged after attrition milling. In addition to the anatase and Al peaks, XRD pattern of the attrition milled blend contains a very small peak of Al_2O_3 suggesting that Al has been partially oxidized during milling.

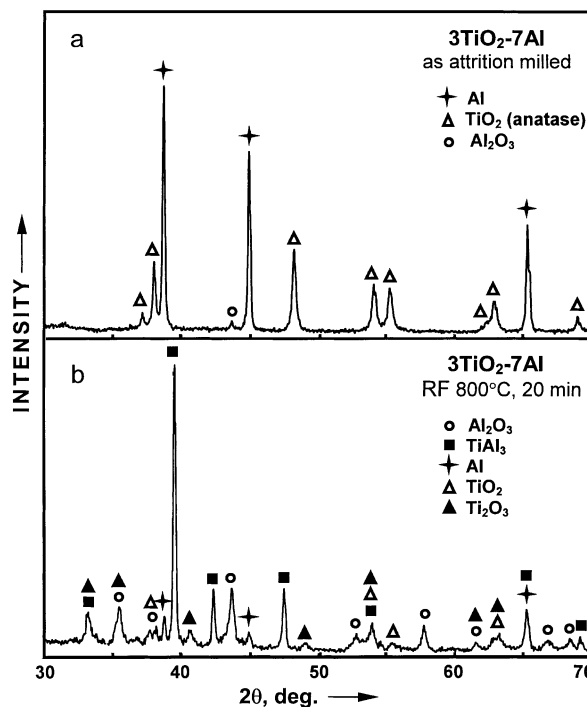


Fig. 3. X-ray diffraction pattern of a $3TiO_2$ -7Al compact: (a) as-compacted; (b) after a 20 min exposure between the press rams at 800 °C.

3.1. Combustion wave propagation

The ignition of the slightly compressed $3\text{TiO}_2\text{-7Al}$ powder blend on one end resulted in a stable combustion wave propagation at $v=0.07$ cm/s. This unusually low wave propagation rate must be due to the very high porosity of the reagent blend. As can be seen in XRD pattern (Fig. 4), the SHS product consisted of Al_2O_3 , $\gamma\text{-TiAl}$ and a small amount of Ti_3Al (α_2). The formation of a Ti-rich aluminide (Ti_3Al) indicates that there was not enough Al for the formation of TiAl , apparently due to the partial oxidation of Al during attrition milling. Some sintering took place during combustion, and the final product was a very porous composite sponge.

3.2. Thermal explosion in a furnace

When a free-standing $3\text{TiO}_2+7\text{Al}$ compact was placed into a preheated furnace, the lowest furnace temperature, T_f , at which self-sustained SHS reaction (thermal explosion) took place was 800°C , i.e. well above the melting point of Al. The measured combustion temperature (T_{comb}) was $\sim 1870^\circ\text{C}$, which is very close to the adiabatic temperature calculated for the reagents preheated to 800°C ($T_{\text{ad}}^{800} = 1881^\circ\text{C}$). As can be seen from the heating curve in Fig. 5, the melting of Al does not ignite thermal explosion, and abrupt self-heating of the sample occurs when it is overheated $\sim 250^\circ\text{C}$ above T_m of Al ($\sim 100^\circ\text{C}$ above T_f). After the aluminum has completely melted, it takes the sample almost 2 min to reach T_f , and two more minutes of slow self-heating to reach ignition. According to the model developed to describe temperature evolution in reactive powder blends,¹⁷ such delayed thermal explosion suggests the presence of a strong interfacial barrier that slows down diffusion-controlled precombustion processes that take place prior to the onset of TE. It is

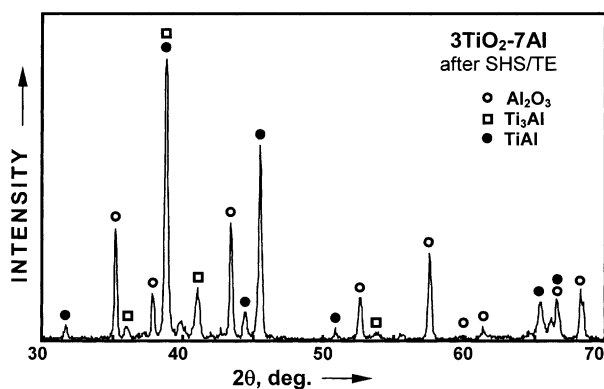


Fig. 4. X-ray diffraction pattern of $3\text{TiO}_2\text{-7Al}$ powder blend after combustion wave propagation (SHS). Identical XRD patterns were obtained from $3\text{TiO}_2\text{-7Al}$ compacts thermally exploded in the furnace (at $T \geq 800^\circ\text{C}$), between the press rams (at $T \geq 875^\circ\text{C}$) and in a rigid die (at $T = 950^\circ\text{C}$).

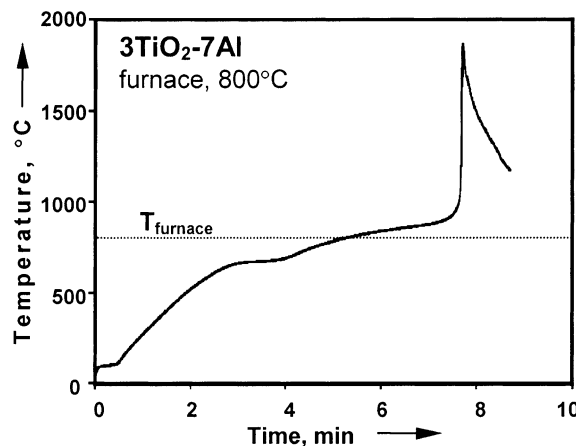


Fig. 5. Temperature evolution curve of a $3\text{TiO}_2\text{-7Al}$ compact placed in the furnace at 800°C .

believed that the previously mentioned partial oxidation of aluminum during attrition milling results in the formation of an Al_2O_3 layer on the Al powder surface that acts as a diffusion barrier.

The composition of the sample obtained by TE in the furnace is identical to that of the combustion wave propagation product (Fig. 4). After thermal explosion, the sample retained its initial shape and dimensions and contained many large unevenly distributed pores. To obtain denser samples reactive forging between press rams was performed.

3.3. Thermal explosion between the press rams (reactive forging)

In Fig. 6, temperature evolution curves of three $3\text{TiO}_2+7\text{Al}$ compacts placed between the rams of the Instron machine heated to 800 , 875 and 950°C (T_{ram}),

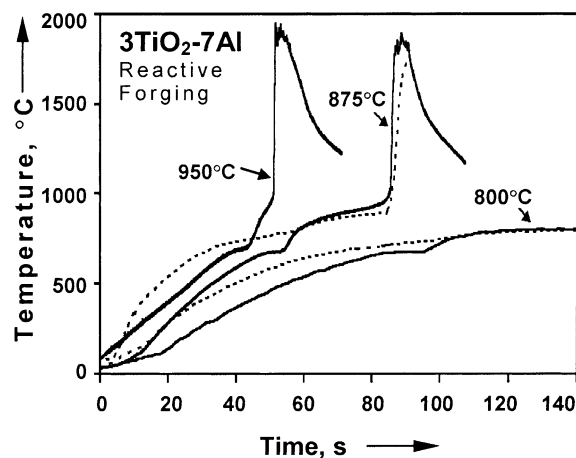


Fig. 6. Temperature evolution curves of $3\text{TiO}_2\text{-7Al}$ compacts placed between the press rams at $T_{\text{ram}} = 800$, 875 and 950°C , respectively. Solid curves correspond to the sample center (thermocouple 1), dashed lines correspond to the sample edge (thermocouple 2).

respectively, are shown. It can be seen that high heating rates (≥ 400 °C/min) have been achieved in all the samples. Initially, the temperature in the center lags far behind that close to the ram owing to the low overall heat conductivity of the sample dominated by the low conductivity of the continuous micro-porous TiO_2 ‘matrix’ [see Fig. 1(a)]. After the melting of Al in the sample interior (at ~ 670 °C), the temperature in the center quickly catches up with that close to the ram, and the two curves merge (for $T_{\text{ram}} = 800$ °C) or intersect (for $T_{\text{ram}} = 875$ °C) even before the ram temperature has been reached. Apparently, the sample’s heat conductivity has increased despite the drop of the heat conductivity of Al on melting. This increase in conductivity can possibly be explained by a better contact at the liquid Al/ TiO_2 interfaces (compared to the former solid Al/ TiO_2 interfaces) that allows the highly conductive Al component to participate more actively in the process of heat transfer.

The heating curve of the sample between the press rams at $T_{\text{ram}} = 800$ °C (Fig. 6) is drastically different from that of the sample in the furnace at the same temperature (Fig. 5). In the furnace, self-heating of the sample takes place followed by thermal explosion, whereas no self-heating is observed between the press rams. This difference clearly illustrates the negative effect of the enhanced heat transfer from the reaction zone to the rams on TE ignition. The result is also in agreement with the previously mentioned model of temperature evolution predicting that, for the thermal explosion mode of SHS, T_{ig} increases with the increasing rate of heat exchange with the environment.¹⁷

XRD pattern of the sample held between the rams until the temperature of 800 °C has been reached reveals almost no signs of reaction. The only difference compared to the unreacted Al– TiO_2 compact is the presence of several small peaks of Ti oxides with the stoichiometry close to TiO_2 but slightly more poor in oxygen. Apparently, TiO_2 loses some oxygen in reaction with Al, however the amount of the reaction products is too small to be detected by XRD. When the sample is held between the rams for additional 20 min, significant amounts of the reaction products (Al_2O_3 , Al_3Ti) as well as oxygen-poor Ti oxides (TiO , Ti_2O_3) are formed [Fig. 3(b)]. In spite of the presence of a liquid phase (Al) and the very fine size of anatase powder particles, the reaction at 800 °C seems to be very slow, and a measurable amount of Al remains in the sample after 20 min exposure. The rate of heat release is correspondingly low, and the sample temperature never rises above the ram temperature. The slow reaction kinetics could, again, be the result of the presence of the Al_2O_3 surface oxide that acts as a diffusion barrier, as well as prevents molten aluminum from effectively wetting the anatase powder.

$T_{\text{ram}} = 875$ °C was the lowest preheating temperature at which thermal explosion took place in $3\text{TiO}_2 + 7\text{Al}$ compacts placed between the press rams. Similarly to

the free-standing compact, thermal explosion in the samples between the rams was not ignited by the melting of Al, and significant overheating above T_m of Al was required for the reaction to become self-sustained (see Fig. 6). The phase composition of the samples thermally exploded between the press rams at 875 and 950 °C is identical to that of the sample thermally exploded in the furnace at 800 °C (Fig. 4), with the

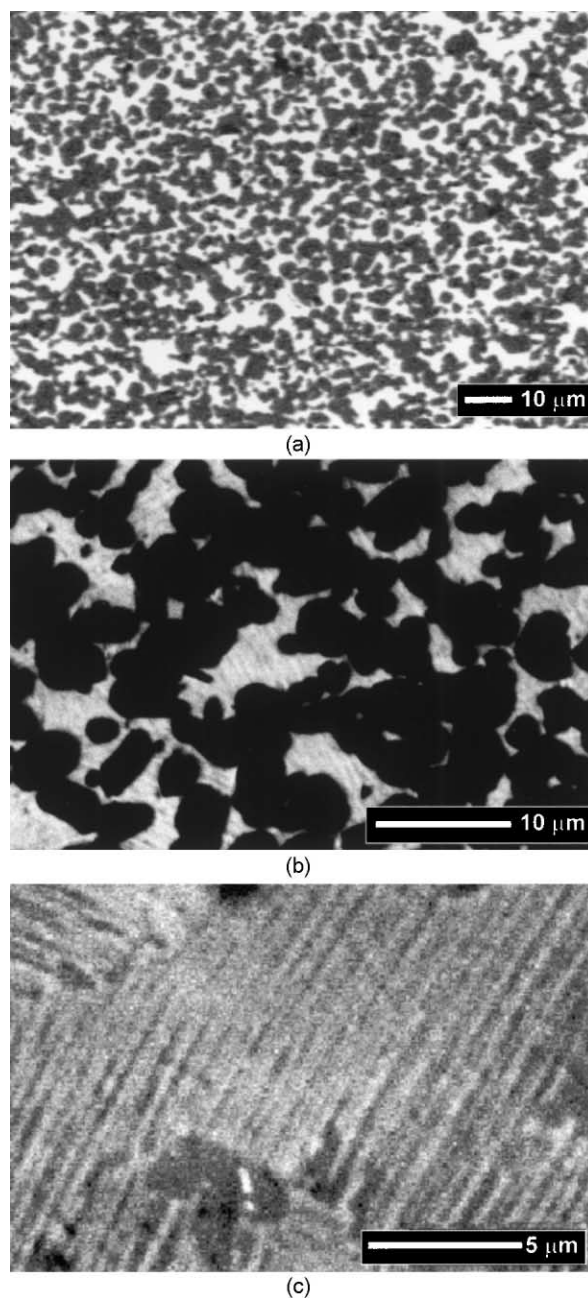


Fig. 7. SEM images (in backscattered electrons) of the Al_2O_3 – $\text{TiAl}/\text{Ti}_3\text{Al}$ composite synthesized by reactive forging at 950 °C, 40 MPa: (a) general view; (b) a higher magnification image; (c) a magnified aluminate region showing $\gamma + \alpha_2$ lamellar structure. In (a) and (b): dark phase— Al_2O_3 , bright phase—aluminide.

Al_2O_3 , AlTi and Ti_3Al compounds detected by XRD. Without pressure application, or at very low applied pressures (up to 4 MPa), reactively forged samples contained at least 15–20% homogeneously distributed pores, the pore size ranging from several microns to tens of microns. Apparently, higher pressures are required to close $\sim 20\%$ porosity carried over from the green compact plus $\sim 16\%$ intrinsic porosity associated with the negative volume change during the synthesis reaction.

According to the experimental measurements and thermodynamic estimations, the highest temperature reached in the course of the RF processing is in the range of 1900–2000 °C (Fig. 6). At this temperature, Al_2O_3 remains solid while TiAl is fully melted. When a unidirectional pressure exceeding 4 MPa was applied according to the scheme in Fig. 2, the semi-liquid product was spread over the lower ram and a very thin flat sample was obtained. In addition, liquid TiAl was squeezed out of the sample yielding highly inhomogeneous material with a high volume fraction of Al_2O_3 in the center and almost pure TiAl at the sample periphery. The use of the confinement cylinder as described in the Experimental Section, as well as optimization of the pressure application rate allowed us to avoid the excessive spreading of the sample and to obtain homogeneous samples in terms of Al_2O_3 and TiAl distribution.

The application of a moderate external pressure of 40–60 MPa during reactive forging (at 950 °C) yielded very high densities in the range of 95–98% TD without affecting the composition of the final product. As can be seen in Fig. 7(a,b), the Al_2O_3 (dark) and the aluminide (bright) phases form homogeneous networks with typical microstructural dimensions of a few microns. EDS analysis of the aluminide gives 42–48 at.% Al (balance Ti), which corresponds to the two-phase $\gamma + \alpha_2$ ($\text{TiAl} + \text{Ti}_3\text{Al}$) field of the Ti–Al diagram, quite in agreement with the above XRD results. SEM observation at higher magnifications, Fig. 7(b) and (c), reveals that the two-phase TiAl – Ti_3Al regions have a typical lamellar microstructure with alternating γ and α_2 plates $\sim 0.2 \mu\text{m}$ thick. The lamellar $\gamma + \alpha_2$ structure of Ti–Al alloys is tougher and more creep resistant than the pure γ -TiAl structure,^{18–23} and might beneficially affect the mechanical properties of the Al_2O_3 – $\text{TiAl}/\text{Ti}_3\text{Al}$ composite as a whole.

A representative fracture surface in Fig. 8 illustrates the fine grain size (2–5 μm) of both the ceramic and intermetallic components of the reactively synthesized Al_2O_3 – $\text{TiAl}/\text{Ti}_3\text{Al}$ composite. The lamellar morphology of the Ti–Al intermetallic phase can be clearly seen. The material is very dense with only a few fine pores observed in the field of view. Optimization of the processing parameters (especially pressure application rate) should allow the fabrication of pore-free Al_2O_3 – $\text{TiAl}/\text{Ti}_3\text{Al}$ composites with homogeneous distribution of the ceramic and intermetallic phases.

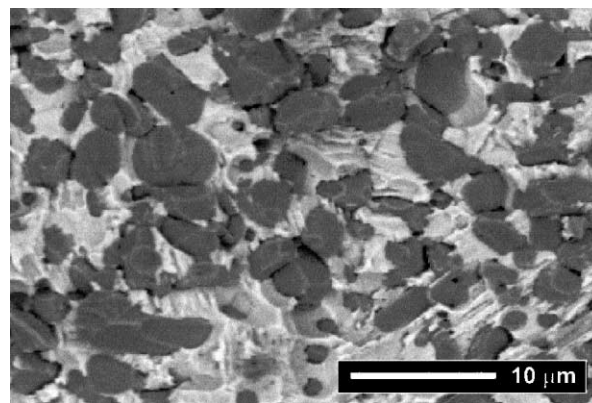


Fig. 8. Fracture surface of the Al_2O_3 – $\text{TiAl}/\text{Ti}_3\text{Al}$ composite synthesized by reactive forging at 950 °C, 40 MPa. Dark phase— Al_2O_3 , bright phase—aluminide. SEM, backscattered electrons.

3.4. Thermal explosion in a constrained die under pressure

The material obtained by thermal explosion of a $3\text{TiO}_2 + 7\text{Al}$ compact in a rigid die at 950 °C, 50 MPa contained Al_2O_3 , TiAl and some Ti_3Al , quite in agreement with our previously reported results.¹⁴ The microstructure and density of the sample were practically identical to those of the reactively forged samples (950 °C, ≥ 40 MPa), with the Al_2O_3 and TiAl phases distributed homogeneously across the sample. This homogeneous distribution is apparently due to the quasi-isostatic pressure in the constrained die that doesn't favor the directional flow of the liquid TiAl to the sample periphery at the high combustion temperature.

3.5. Reactive hot pressing

The synthesis reaction in 3TiO_2 – 7Al blend was far from being complete after 1 h RHP at 950 °C. This is not totally unexpected given the slow reaction rate observed in the samples placed between the press rams preheated to 800 °C (below the ignition temperature of TE). After RHP (950 °C, 1 h), neither TiO_2 nor Al is detected in the sample, however a large amount of intermediate compounds, such as TiO , Ti_2O and TiAl_2 , is revealed by XRD, in addition to the Al_2O_3 , TiAl and Ti_3Al product phases (see Fig. 9). The microstructure of such sample [Fig. 1(b)] is quite unlike the microstructure of the reactively forged sample (Fig. 7) and resembles that of the unreacted compact [Fig. 1(a)]. Former Al particles several microns in size having a rather regular shape can be seen in Fig. 1(b) separated from the former TiO_2 'matrix' by an Al_2O_3 layer. According to the EDS analysis, the particles' composition is close to TiAl_2 , whereas the 'matrix' is a mixture of TiO and Ti_2O . The morphology observed suggests that Al_2O_3 is formed by an interfacial reaction between TiO_2 and Al, whereas Ti–Al intermetallics are formed by the diffusion of Ti

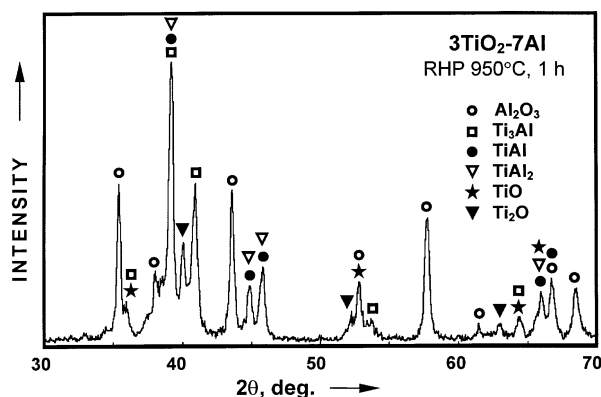


Fig. 9. X-ray diffraction pattern of a 3TiO₂–7Al compact reactively hot pressed at 950 °C for 1 h.

from TiO₂ into Al through the Al₂O₃ layer. In the course of this process TiO₂ is gradually transformed into oxygen-poor Ti–O compounds such as TiO and Ti₂O. The presence of the Al₂O₃ interfacial layer between the reagents must be responsible for the slow kinetics of the RHP reaction. Dark areas with a very fine dispersion of submicron Al₂O₃ particles [Fig. 1(b)] are an additional microstructural feature of the RHP sample. These areas, most probably, correspond to the locations of the smallest (0.5–1 μm) Al particles in the unreacted compact. This suggests that if micron or submicron starting Al powder is used, full conversion of reagents into products in RHP will be achieved over a much shorter time yielding an Al₂O₃–TiAl composite with extremely fine microstructure.

4. Conclusions

1. Self-propagating high-temperature synthesis (SHS) of a compacted 3TiO₂–7Al powder blend was used to fabricate alumina–TiAl interpenetrating composites. Different SHS modes, namely combustion wave propagation, pressureless thermal explosion (TE) in a furnace, and pressure-assisted TE in a constrained die or between press rams (reactive forging) have been studied. Due to the fast kinetics of SHS reaction, processing cycles of several minutes were sufficient to achieve full conversion of reagents into products with the formation of fine micron size homogenous microstructures with interpenetrating Al₂O₃ and TiAl networks.

2. The Al₂O₃–TiAl phase composition of the final SHS product was identical for the different SHS modes applied. Due to partial oxidation of starting Al powder during blend preparation, a small amount of the Ti-rich Ti₃Al (α₂) aluminide was present in the SHS product forming a very fine submicron γ + α₂ lamellar microstructure with TiAl (γ).

3. The melting of Al did not ignite thermal explosion in the 3TiO₂–7Al blend, and significant overheating

above the melting temperature of Al was required for the reaction to become self-sustained. Due to the enhanced heat transfer to the press rams, a higher ignition temperature of TE was measured in Reactive Forging ($T_{ig}=875$ °C) compared to TE of the free-standing sample in the furnace ($T_{ig}=800$ °C).

4. The combustion temperature (T_{comb}) of thermal explosion ranged between ~1870 and 2000 °C, depending on the furnace or ram temperature. These values are very close to the predicted adiabatic temperature of 3TiO₂ + 7Al → 2Al₂O₃ + 3TiAl reaction calculated for the corresponding reagent temperatures.

5. The application of a moderate pressure (~50 MPa) during thermal explosion in a rigid die or between press rams (reactive forging) yielded near fully dense (up to 98% TD) interpenetrating Al₂O₃–TiAl composites. Since the processing cycle is much shorter for reactive forging than for thermal explosion in a constrained die, the former approach seems to be more technologically feasible and thus more attractive for industrial implementation. If supplemented with forming dies, the reactive forging facility can be used for the fabrication of dense near-net-shape in situ composites.

6. Reactive hot pressing of 3TiO₂–7Al blend at 950 °C, 1 h resulted in only partial conversion of reagents into the Al₂O₃–TiAl product. The slow kinetics of this solid-state reaction is explained by the formation of an Al₂O₃ layer on the surface of the former Al particles acting as a diffusion barrier. At the same time, the results of the RHP experiment suggest that the use of micron/submicron Al powder can significantly accelerate the process yielding an Al₂O₃–TiAl composite with extremely fine microstructure.

Acknowledgements

This work was supported by the German–Israeli Foundation for Scientific Research and Development (GIF) through research grant No. I-556-216.10/97.

References

1. Clarke, D. R., Interpenetrating phase composites. *J. Am. Ceram. Soc.*, 1992, **75**, 739–759.
2. Aghajanian, M. K., Rocazella, M. A., Burke, J. T. and Keck, S. D., The fabrication of metal matrix composites by a pressureless infiltration technique. *J. Mater. Sci.*, 1991, **26**, 447–454.
3. Subramanian, R., McKamey, C. G., Schneibel, J. H., Buck, L. R. and Menchhofer, P. A., Iron aluminide–Al₂O₃ composites by in situ displacement reactions: processing and mechanical properties. *Mater. Sci. Eng.*, 1998, **A254**, 119–128.
4. Claussen, N., Garcia, D. E. and Jansen, R., Reaction sintering of alumina–aluminide alloys (3A). *J. Mater. Res.*, 1996, **11**, 2884–2888.
5. Schicker, S., Garcia, D. E., Bruhn, J., Janssen, R. and Claussen, N., Reaction processing of Al₂O₃ composites containing iron and iron aluminides. *J. Am. Ceram. Soc.*, 1997, **80**, 2294–2298.

6. Wagner, F., Garcia, D. E., Krupp, A. and Claussen, N., Interpenetrating Al_2O_3 - TiAl_3 alloys produced by reactive infiltration. *J. Eur. Ceram. Soc.*, 1999, **19**, 2449–2453.
7. Munir, Z. A., Synthesis of high-temperature materials by self-propagating combustion methods. *Ceram. Bull.*, 1988, **67**, 342–349.
8. Munir, Z. A. and Anselmi-Tamburini, U., Self-propagating exothermic reactions: the synthesis of high-temperature materials by combustion. *Mater. Sci. Rep.*, 1989, **3**(7–8), 277–365.
9. Holt, J. B., The use of exothermic reactions in the synthesis and densification of ceramic materials. *MRS Bull.*, 1987, **12**, 60–64.
10. Grebe, H. A., Thadhani, N. N. and Kottke, T., Combustion synthesis and subsequent explosive densification of titanium carbide ceramics. *Metall. Trans.*, 1992, **23A**, 2365–2372.
11. LaSalvia, J. C., Meyer, L. W. and Meyers, M. A., Densification of reaction-synthesized titanium carbide by high-velocity forging. *J. Am. Ceram. Soc.*, 1992, **75**, 592–602.
12. Hoke, D. A. and Meyers, M. A., Consolidation of combustion-synthesized titanium diboride-based materials. *J. Am. Ceram. Soc.*, 1995, **78**, 275–284.
13. Choi, Y., Lee, J. K. and Mullins, E., Densification process of TiC_x -Ni composites formed by self-propagating high-temperature synthesis reaction. *J. Mater. Sci.*, 1997, **32**, 1717–1724.
14. Gutmanas, E. Y. and Gotman, I., Dense high-temperature ceramics by thermal explosion under pressure. *J. Eur. Ceram. Soc.*, 1999, **19**, 2381–2393.
15. Klinger, L., Gotman, I. and Horvitz, D., *In situ* processing of composite TiB_2/TiC ceramics by thermal explosion under pressure: experimental study and modeling. *Mater. Sci. Eng. A*, 2001, **302/1**, 92–99.
16. Gotman, I., Travitzky, N. and Gutmanas, E. Y., Dense *in-situ* TiB_2/TiN and TiB_2/TiC CMCs: reactive synthesis and properties. *Mater. Sci. Eng.*, 1998, **A244**, 127–137.
17. Farber, L., Klinger, L. and Gotman, I., Modeling of reactive synthesis in consolidated blends of fine Ni and Al powders. *Mater. Sci. Eng.*, 1998, **A254**, 155–165.
18. Huang, S. C. and Chesnutt, J. C., Gamma TiAl and its alloys. In *Intermetallic Compounds: Vol. 2, Practice*, ed. J. H. Westbrook and R. L. Fleisher. John Wiley and Sons, Chichester, 1994, pp. 73–90.
19. Chan, K. S. and Shih, D. S., Fatigue and fracture behavior of a fine-grained lamellar TiAl. *Metal. Mater. Trans.*, 1997, **28A**, 79–90.
20. Chen, W. Z., Song, X. P., Qian, K. W. and Gu, H. C., The lamellar microstructure and fracture behavior of gamma-based TiAl alloy produced by centrifugal spray deposition. *Mater. Sci. Eng.*, 1998, **A247**, 126–134.
21. Kuang, J. P., Harding, R. A. and Campbell, J., Microstructures and mechanical properties of an investment cast gamma titanium aluminide. *Mater. Sci. Technol.*, 1999, **15**, 840–850.
22. Campbell, J. P., Venkateswara-Rao, K. T. and Ritchie, R. O., The effect of microstructure on fracture toughness and fatigue crack growth behavior in gamma-titanium aluminide based intermetallics. *Metal. Mater. Trans.*, 1999, **30A**, 563–577.
23. Wang, J. N. and Xie, K., Refining of coarse lamellar microstructure of TiAl alloys by rapid heat treatment. *Intermetallics*, 2000, **8**, 545–548.