



Rapid and direct reactive synthesis of Ti–Al intermetallics by microwave heating of TiH₂ and Al powder without microwave susceptor



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ABSTRACT

TiAl and TiAl₃ titanium aluminide intermetallic compounds are synthesized directly within a few ten seconds by microwave technique. An initial mixture of TiH₂ and Al powders has been used. Such mixture avoids the use of a microwaves susceptor. The hydrogen desorbed during the experiment keep the mixture in a reductive atmosphere thus avoiding possible oxidation. The role of TiH₂ is then: (i) to act as a microwave absorber, (ii) to provide a reductive atmosphere and (iii) to provide energy through the hydrogen release reaction. The formation of the various intermetallics has been highlighted by X-ray diffraction and EPMA analysis.

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1. Introduction

Recently, ordered Ti–Al intermetallic compounds, especially the stoichiometric compounds TiAl₃ and TiAl(γ), have attracted extensive attention of many researchers, because of their excellent properties, such as the superior specific stiffness, high melting point, good oxidation resistance at high temperature [1]. The binary phase diagrams of Al with transition metals are abundant in intermetallic phases with similar formation enthalpies. Achieving a single-phase product is, therefore, difficult. The formation of secondary phases, starting from stoichiometric ratio composition of constituent elements, can be controlled by the heating rate. Slow heating then favours the formation of brittle mixtures of such intermetallic phases. Rapid heating processing techniques can instead promote the formation of single-phase [2,3]. Numerous technologies have been developed to prepare this Ti–Al intermetallic compounds, mainly including γ-TiAl and TiAl₃.

Combustion synthesis (CS) or self-propagating high-temperature synthesis (SHS) is an effective, low-cost method for production of various Ti–Al phases. The synthesis by combustion reactions relies on the exothermal energy release from the reactant materials.

Ti–Al system is considered as weak exothermal system since its adiabatic temperature is less than 1800K [4]. For this reason, the combustion reaction must be activated. The activation can either be performed by mechanical milling [5], pre-heating using a conventional radiant-type furnace (thermal activation, TACS) [6] or by the application of electric fields or currents (field-assisted CS or FACS) [7,8], with or without the simultaneous use of hydrostatic or uni-axial pressure.

The combustion process itself can also be affected by microwave heating (Microwave Activated Combustion Synthesis or MACS) [9–11]. The processes differ in the method of application of the electric/electromagnetic field and the mechanism by which activation occurs. FACS utilizes a mechanism of joule heating due to electrical resistance (provides energy to the propagating reaction); while MACS relies on microwave absorption. Microwave activation is attractive for combustion synthesis reactions, allowing rapid heating (and cooling) rates, volumetric heating, and direct absorption of energy by the reaction zone [12,13]. In a combustion process, the hottest region occurs where the reaction is taking place. Additionally, microwave process has been shown to faster the formation of such hot spots that account, at least partially, for an increase in the kinetics of reaction noted for microwave processes, and a shift in the equilibrium of a reaction. It is important to be noted that the presence of microwave susceptor (e.g. silicon carbide) is mandatory in the case of microwave activated

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combustion synthesis. It preferentially absorbs microwaves in MACS, provides ignition, and activates combustion reactions. Furthermore, the susceptor can, if desired, provide continued post-combustion heating. Both processes are capable of rapid ignition and heating rates.

In general, the sintering process of titanium-based objects should be performed in an argon atmosphere or in vacuum because of titanium's high reactivity with oxygen, nitrogen, and water [14]. Therefore, cost reduction is a major driving force in the manufacturing and processing of porous Ti–Al alloys filters or even of dense TiAl alloys. On the other hand, the formation of titanium oxide or titanium nitride layers during sintering cannot be prevented completely under these neutral atmospheres. In order to prevent the formation of these compounds during the processing stage, the titanium hydride evolving hydrogen can appear as an attractive raw material [14,15]. More recently, porous TiAl alloys with different nominal powder compositions were fabricated by Q. Peng *et al.* [16] through a cost-effective powder metallurgy (PM) approach by sintering TiH₂ and Al mixed powders. It is found that the properties vary with the TiH₂ and Al contents (*e.g.* the nature of the pores can be improved by using different compositions). Furthermore, by detailed structural characterizations, four crystalline phases Ti₃Al, TiAl, TiAl₃, and Ti₂Al₅, were observed in the fabricated porous TiAl alloys with different compositions [16].

In this study, we introduce a new cost-effective Microwaves Reactive Synthesis (MRS) approach without microwave susceptor to synthesize a single phase of TiAl and TiAl₃ starting from TiH₂ and Al powders. In the Ti–Al phase diagram, both of these compounds have a broad single-phase field. So starting mixture compositions were adjusted across the single-phase field (TiH₂:Al molar ratio of 1:1 and 1:3). In contrast to MACS, in which microwaves are used just for the ignition of the CS process, in our experiment the microwave field is maintained during the entire duration of the high-temperature treatment.

2. Experimental

Home-made titanium hydride was prepared from treatment of pure Ti sponge (Strem Chemicals 99.8%) at 450 °C under 40 bars of hydrogen for 2 h. The as prepared hydride and the Al powder (about 10 μm with spherical shape – Strem Chemicals 99.9%) were ball milled in a stainless steel vial under 10 bar of H₂ for 2 h with 30 min of continuous milling followed by 10 min pause (in order to allow the system to cool down). Ball milling was performed using a Fritsch P5 ball mill. The ball to powder weight ratio was 17:1 corresponding to 8 g of powder and 34 stainless steel balls (10 mm of diameter), and the rotation speed was 250 rpm. The milling vial was sealed and opened in a glove box filled with purified argon. Proper quantities (100 mg) of powders were isostatically cold pressed at 450 MPa for 3 min into cylindrical tablets (diameter 6 mm and about 2 mm high). Such pellets were used for microwaves (MW) experiments. The MW experiments were carried out in a MARS 6 – CEM microwave cavity with microwave frequency of 2.45 GHz. The MW power was fixed for all the experiments to a value of 1500 W. In the center of the microwave cavity, the pellet was placed in an airtight quartz tube connected to a vacuum pump (10⁻² MPa) through a hole at the top of the microwave cavity. The pellets before and after the microwaves treatment were characterized by X-ray diffraction. For diffraction measurements, a Philips PW 1050 diffractometer with Cu Kα₁ radiation (λ = 0.15405 nm) was used. The patterns were scanned in steps of 0.017° (2θ) from 8° to 80° with a constant counting time of 30 s. The samples were investigated ex-situ by electron probe microanalyses (CAMECA

SX100) to identify the phases obtained. The temperature was measured by using a pyrometer which is fixed outside the microwave oven cavity (behind the transparent door). The error bars were estimated by taking into account: (i) the measurement error of the pyrometer and (ii) the experimental error (the sample has a small size. In addition, it moves in the quartz tube during the irradiation).

3. Result

In order to check the decomposition of TiH₂ into Ti and the sufficient heat generation between TiH₂-Al powder mixture during microwave process, it is mandatory to first study the dehydrogenating reaction of pure titanium hydride by microwave heating. Such study has already been done by Luo and co workers [17] but as we are using ball milled TiH₂, it was important to check its dehydrogenating properties because it is known that ball milling can induce different dehydrogenating temperature and kinetics.

X-ray diffraction pattern of pure titanium hydride (ball milled for 2 h) after different durations of microwaves irradiation exposure at a fixed power of 1500 W are shown in Fig. 1 and the relative weight percentage of Ti estimated from X-Ray diffraction refinement after microwave irradiation is shown in Table 1. It is worth pointing out that, after 5 s of microwave irradiation, small peaks appear clearly at 38° and 40° (this last one appear as a shoulder on the (200) peak of TiH₂ at 40.5°). This two peaks can be attributed to the formation of Titanium indicating that the desorption of TiH₂ already started. After 10 s, the formation of Ti is even clearer and TiH₂ remained present at a very low level (*e.g.* shoulder on the (101) peak of Ti at 40.2°). After 20 s, TiH₂ is completely transformed into Ti. The temperature profile during microwave irradiation of pure TiH₂ powder as a function of time is shown in Fig. 2. As can be seen from Fig. 2, the temperature reaches 450, 770 and 900 °C after respectively 5, 10 and 20 s of microwave heating. The temperature fluctuation can be linked with the release of hydrogen which can generate low-temperature plasmas which reflect incident MWs, leading to these cyclic temperature fluctuations. The temperature reached is sufficient to induce (i) the desorption of hydrogen (the release of hydrogen start around 400 °C only) [18] and also (ii) the melting of Al powder. Then, the reaction between Ti and melted Al

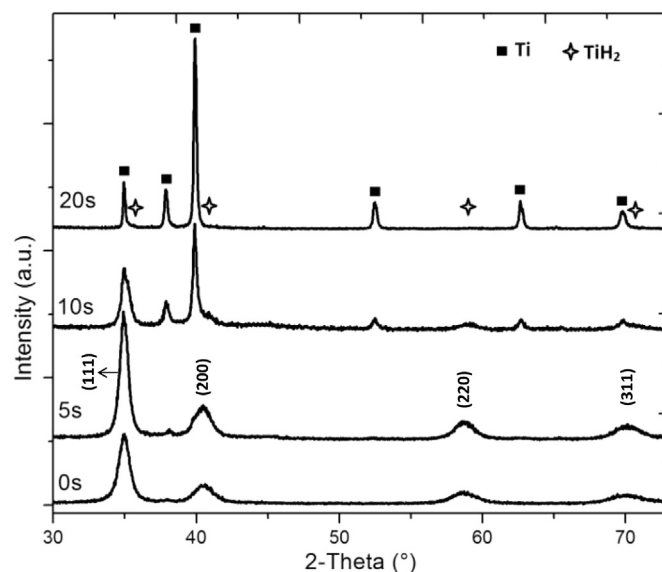


Fig. 1. X-ray diffraction profiles of Titanium hydride powder after different times of microwaves irradiation exposure with 1500 W input power.

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