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Reactive synthesis of Ti–Al intermetallics during microwave heating in an E-field maximum

S. Vaucher[a,*], M. Stir[a], K. Ishizaki[a], J.-M. Catala-Civera[b], R. Nicula[a]

[a] Swiss Federal Laboratories for Materials Testing and Research – Empa, Feuerwerkerstr. 39, CH-3602 Thun, Switzerland
[b] Universidad Politecnica de Valencia, Camino Vera s/n, E-46022 Valencia, Spain

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ABSTRACT

The time-resolved X-ray diffraction synchrotron radiation technique was used in combination with E-field microwave heating to study *in situ* the kinetics of intermetallic phase formation in the Ti–Al system. The reaction of Ti with Al is triggered by the melting and spreading of Al onto the surface of Ti particles. The tetragonal $TiAl_3$ phase is the primary reaction product, formed by instantaneous nucleation at the interface between the unreacted Ti cores and the Al melt. The growth of $TiAl_3$ layers is diffusion-controlled. These preliminary results demonstrate that microwave heating can be used to rapidly synthesise intermetallic phases from high-purity elemental powders.

1. Introduction

Titanium aluminides are an important family of intermetallic compounds nowadays widely used in structural, automotive or aerospace applications [1–3]. Typical applications range from lightweight airframe structural materials to compressor blades, rotors, and many other components for automobile or aircraft engines. The special combination of low density, high resistance to high-temperature corrosion, reasonable oxidation resistance and high specific yield strength is more pregnant in the γ-TiAl and α_2-Ti_3Al intermetallic phases. Consequently modern industrial TiAl alloys commonly have lamellar microstructures that alternate the γ and α_2 phases. Such microstructures form by a eutectoid decomposition of the α-TiAl phase upon melt-solidification.

The binary phase diagrams of aluminium with transition metals are abundant in intermetallic phases with similar formation enthalpies. 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 γ-TiAl or α_2-Ti_3Al phases [4]. Combustion synthesis (CS), pioneered for intermetallics during the 1970s [5], has become within just a few years the method of choice for the rapid synthesis of intermetallic compounds [4–6]. The CS method is well-suited for materials that are difficult to shape due to their limited ductility at low temperature and difficult to cast due to their high melting point and reactivity. The pro's and contra's of the CS technique were already reviewed in numerous occasions [4–11]. Synthesis by combustion reactions relies on the exothermal energy release from the reactant materials. Self-propagating high-temperature synthesis (SHS) together with reactive synthesis (RS, also termed simultaneous combustion, volumetric combustion or thermal explosion mode) are the two major CS processes for the case of solid-state reactant materials [4]. The exothermal reaction is initiated without activation and proceeds in a self-sustained manner only above a certain limit temperature, namely the adiabatic temperature (T_{ad}). The adiabatic temperature T_{ad} equals 1493 K for γ-TiAl and 1517 K for the α_2-Ti_3Al phase. For weak exothermic systems with $T_{ad} < 1800$ K [6], the reaction must be activated, as is the case for the Ti–Al system. The activation can either be performed by mechanical milling [12–14], pre-heating using a conventional radiant-type furnace (thermal activation, TACS) [15] or by the application of electric fields or currents (field-assisted CS or FACS) [16–18], with or without the simultaneous use of hydrostatic or uniaxial pressure [4]. An alternative method for the activation of reactive synthesis is the use of microwaves (MACS) [19–22].

The role and importance of time-resolved studies of phase transformation kinetics of heterogeneous materials during SHS or RS was made clear by many authors [16,17,23–25]. Recently, microwave heating was used in combination with the synchrotron radiation powder diffraction technique to investigate the ultrafast kinetics of phase transitions and chemical reactions in various metallic alloys and oxide materials [26–30]. In the present work, the formation of titanium aluminide phases from elemental powders during microwave heating is investigated *in situ* using the time-resolved

* Corresponding author. Tel.: +41 33 228 2949; fax: +41 33 228 4490.
E-mail address: sebastien.vaucher@empa.ch (S. Vaucher).

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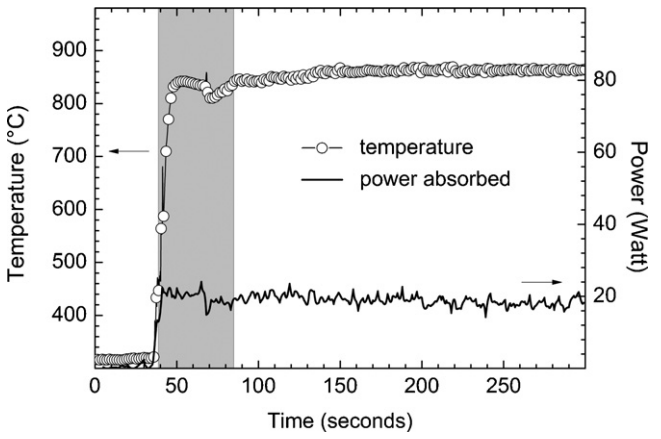

Fig. 1. The temperature profile (PhotriX LP) and absorbed power recorded during E-field microwave heating at 2.45 GHz.

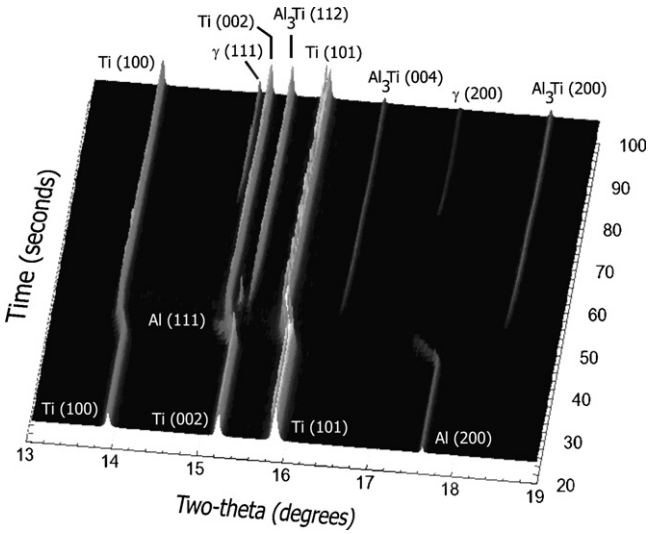

Fig. 2. Three-dimensional X-ray intensity map recorded during microwave heating.

X-ray diffraction (TRXRD) method. 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. The main reasons for the present study are as follows: it appears that some disagreement persists with respect to which intermetallic phases are formed first during CS [31]. Moreover, it was suggested in earlier works that with increasingly high heating rates, the Ti–Al reaction is no longer preceded by the melting of Al, but gradually takes the character of a solid-solid reaction similar to sintering [32]. In order to clarify these issues in more detail, we focus our study on the initial stage of the reaction between Al and Ti powders, and determine the kinetics of the reaction using the *in situ* time-resolved synchrotron radiation diffraction technique.

2. Experimental

Commercial Al (99.7%, MEPURA Metallpulver GmbH, Austria) and Ti (Pyrogenesis INC, Canada) powders were mixed in a Ti:Al 3:1 ratio, then mounted in sapphire capillaries. The filled sapphire capillary is placed in the resonant microwave cavity in the calculated position of the microwave E-field maximum. The TRXRD experiments were performed in transmission at the X04SA Materials Science beamline at the Swiss Light Source. High-resolution X-ray diffraction patterns were collected each second within a wide angular range using a fast MYTHEN II detector [33]. Microwave heating was carried out at 2.45 GHz using a portable monomode microwave applicator. A three-stub tuner, a sliding short and an iris are used to tune a WR-340 rectangular cavity (TE_{10n} resonant cavity). Both the forward and the reflected microwave power were monitored by an impedance analyser.

The specimen temperature was measured with an optical pyrometer (Raytek SXACLS) and also with a PhotriX IR fiber optic temperature system (Luxtron, Santa Clara, CA). The PhotriX light-pipe (LP) thermometer can be used for temperature measurements above 350 °C. The temperature profile (PhotriX LP) and the absorbed power recorded during the *in situ* microwave heating experiment are shown in Fig. 1. The forward microwave power is first set at 100 W. Resonant cavity tuning operations are stopped once the mix powder specimen couples to the microwave field and starts heating (at $t = 38$ s). Once the cavity is tuned, the absorbed power levels at 20 W and the temperature raises rapidly to nearly 850 °C (within 10 s). The non-isothermal region of interest (ROI) for the Ti reaction with Al is grey-shaded in Fig. 1.

3. Results and discussion

The TRXRD experiments were carried out using monochromatic synchrotron radiation ($\lambda = 0.62$ Å). The X-ray scattered intensity recorded during the microwave heating are shown as a 3D intensity map in Fig. 2. The initial powder diffraction patterns contain only the Bragg reflections of the starting Ti and Al powders (Fig. 3a). As seen from Fig. 2, at $t = 47$ s ($T = 819.3$ °C) the disappearance of both Bragg diffraction lines Al(1 1 1) and (2 0 0) indicates the melting of Al has occurred. The molten Al reacts immediately with the Ti particles to form the tetragonal $TiAl_3$ phase (space group S.G. 139, *I*4/*mmm*, tI8, PDF 37-1449). The present results therefore re-

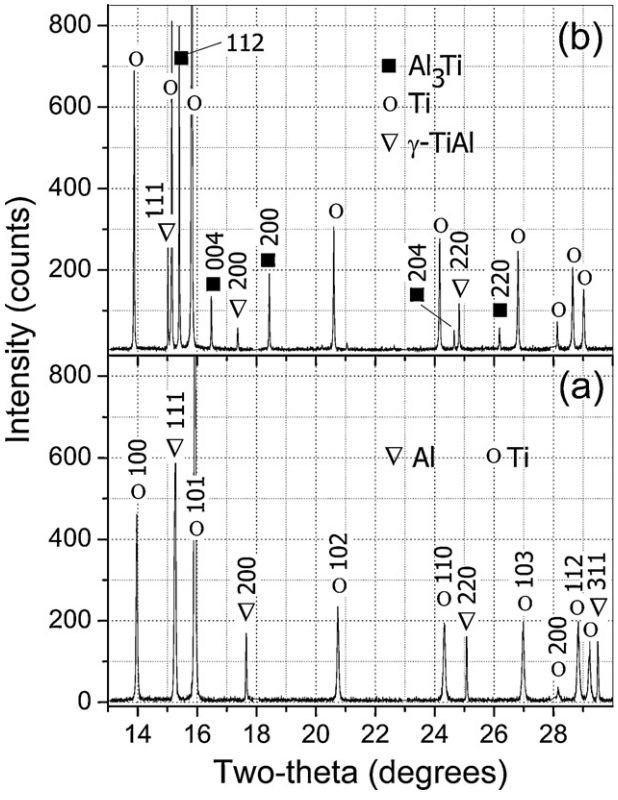

Fig. 3. Powder diffraction patterns collected at (a) $t = 1$ s and (b) $t = 80$ s.