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Combustion synthesis and development of Ti–O–C aluminium composites

Krzysztof Naplocha*

Institute of Production Engineering and Automation, Wroclaw University of Technology, ul. Łukasiewicza 5, 50-371 Wroclaw, Poland

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ABSTRACT

Aluminium matrix composite reinforced with Ti compounds was successfully fabricated by SHS combustion synthesis and squeeze casting course. Prepared samples from mixture containing Ti, C and Al_2O_3 fibres were heated in microwave reactor to ignite synthesis and produce porous preform for subsequent infiltrating with liquid metal. Studies showed that synthesizing temperature has been remarkably increased by applying higher magnetron power and addition of graphite. Synthesis of specimens prepared from preliminary ball milled Ti and C powders proceeded at the highest propagation wave velocity. Microwave heating of metal Ti powder in the stream of CO_2 resulted in formation of corrugated precipitates composed of titanium oxide with carbon inclusions TiO(C) and Ti_2O_3. The produced preforms were impregnated by squeeze casting with the aluminium alloy AlSi7Mg. Proper interface with slight reduction of Ti oxide between the reinforcement and the matrix was developed. Subsequently, the samples were annealed at 500 and 1000 °C. Annealing at the lower temperature induced creation of Ti_3O_2(C) and Al_2O_3. This process was continued at 1000 °C, and additionally some $Ti(Al_{0,8}Si_{0,2})_3$ pellets appeared in the matrix. With prolonged annealing, oxygen was completely removed from Ti compound and oval grains of Ti(C) were created, enveloped with Al_2O_3. In the matrix, larger and numerous Ti_3AlSi_5 pellets were formed. Hardness examination showed that the best strengthening effect was achieved after annealing at 1000 °C.

1. Introduction

Carbides or oxycarbides attract wide interest because of their unique physical properties such as high hardness, corrosion and oxidation resistance at elevated temperatures, good thermal and electrical conductivity, low reactivity and high melting point [1–3]. However, due to their low fracture toughness, they reveal small resistivity to thermal shocks and usually require introducing additional components. Saturating a porous ceramic preform with ductile matrix may improve its resistance to initiation and propagation of cracks in various ways. Nowadays, variable methods of manufacturing ceramic skeletons and their subsequent impregnating with liquid alloys are elaborated and developed.

High-temperature synthesis SHS activated with microwaves, presented in [4] as MACS (microwave activated combustion synthesis), enables producing porous preforms to be reinforced with composite materials. Microwave heating improves the process as well as affects structure and conversion degree of the initial substrates mixture. Benefits of using microwaves or plasma have been already confirmed several times [5,6].

In the case of dielectric materials, the penetrating microwaves generate mainly internal electric field. It puts electrons or ions in oscillation and makes dipoles rotate. Striking elastically and rubbing each other they generate heat. The first parameter that determines ability of microwave absorption is the dielectric loss factor ε'' (very small for Al_2O_3 and TiO_2).

Falling radiation on non-ferromagnetic metals (Ti and Al) with solid compact structure, induces currents in macroscale, which in turn create opposed field blocking and reflecting microwaves. However, if the metal is in form of particles covered with insulating oxides, penetration of microwaves can be observed and the arising currents heat-up the material in microscale. The tangent component of magnetic field created by microwave radiation induces electric field in the particle skin, which thickness for metals is usually a 0.1–10 μm.

For fine metallic powders, the skin significantly affects heating of a particle core, especially that penetration depth increases with temperature [7]. The currents induced in this layer can change magnetic properties, even of paramagnetic materials (Al, Ti). In [8], the loss factor coefficients were determined on the ground of the Mie–Lorenz theory. It was found that with increasing thickness δ (more precisely r/δ ratio) the dielectric loss factor ε'' decreases dozens times, whereas coupling with the magnetic field becomes most intensive and the magnetic loss factor μ'' reaches its maximum at some average value of this ratio (r/δ = ca. 20).

The concept accepted in this work consists in impregnating a porous preform of hard and brittle material with a relatively ductile aluminium alloy. The Al_2O_3 fibres should enhance initial strength

* Tel.: +48 71 3204236.
E-mail address: krzysztof.naplocha@pwr.wroc.pl

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Table 1
Specimens composition with maximum combustion temperature T_m and wave velocity v.

Specimen symbol	Composition [vol%]			T_m [°C]		v [cm/s]
	Al_2O_3	Ti	C	at 250 W	at 500 W	
A10T5	10	5	–	1250	–	–
A10T10	10	10	–	1360–1410	1560–1660	–
A10T10C5	10	10	5	1400–1480	1840–1960	0.22–0.29
A10T10C10	10	10	10	–	1560–1670	0.18–0.26
A10TC15	10	15		–	1410–1540	0.32–0.43

of the preform, whereas the Ti powder is converted to hard carbides and oxides. Microwave heating of powdered mixture is a relatively new area of research. To gain insight into the phenomena that occur inside reactor chamber with heated material a detailed study of absorption power distribution was performed. Microwave first heats skin of paramagnetic Ti powder and subsequently partly dielectric Ti–O compound. This permits to ignite and support SHS synthesis of solid components with flowing CO_2 gas. Constructed reactor and developed method is an innovative way to heat materials, both ceramic and metal.

2. Experimental

Powders provided by AlfaAesar company and Saffil fibres were used for production of the preforms. Average size of Ti and C particles amounted to 44 μm (−325 mesh), whereas diameter of fibres (96% Al_2O_3 + 4% SiO_2) ranged between 4 and 6 μm. Specimens were prepared by a three-step process: production of preforms by SHS synthesis, squeeze casting infiltration and thermal annealing. In the first stage, proper quantities of powders and fibres were mixed for 15 min with 3% water solution of silica binders to obtain homogeneous mixture. Next, the mixture of powders and fibres was drained-off and formed to rectangular preforms 6 × 4 × 1 cm. Volume fractions amounted to 5–10% of Ti powder and to 5–10% of graphite. Simplified symbols informing about volume fractions of Al_2O_3 fibres (A), titanium (T), and graphite (C) were established (Table 1). For example, the specimen A10T10C5 contained 10% of Al_2O_3, 10% of Ti and 5% of C (all percentages volumetric). Specimens with symbol TC were prepared of Ti and C powders comminuted in a ball mill for 14 h. The milling process was performed under argon atmosphere in an attritor containing hard steel balls of 11 mm in diameter. The following operation parameters: ball to powder ratio (BPR) = 20:1; rotational speed = 80 rpm were used.

The prepared preforms were heated-up in microwave field in order to ignite the combustion synthesis and to process the components with flowing CO_2. The specially designed microwave furnace comprised a rectangular waveguide, a chamber

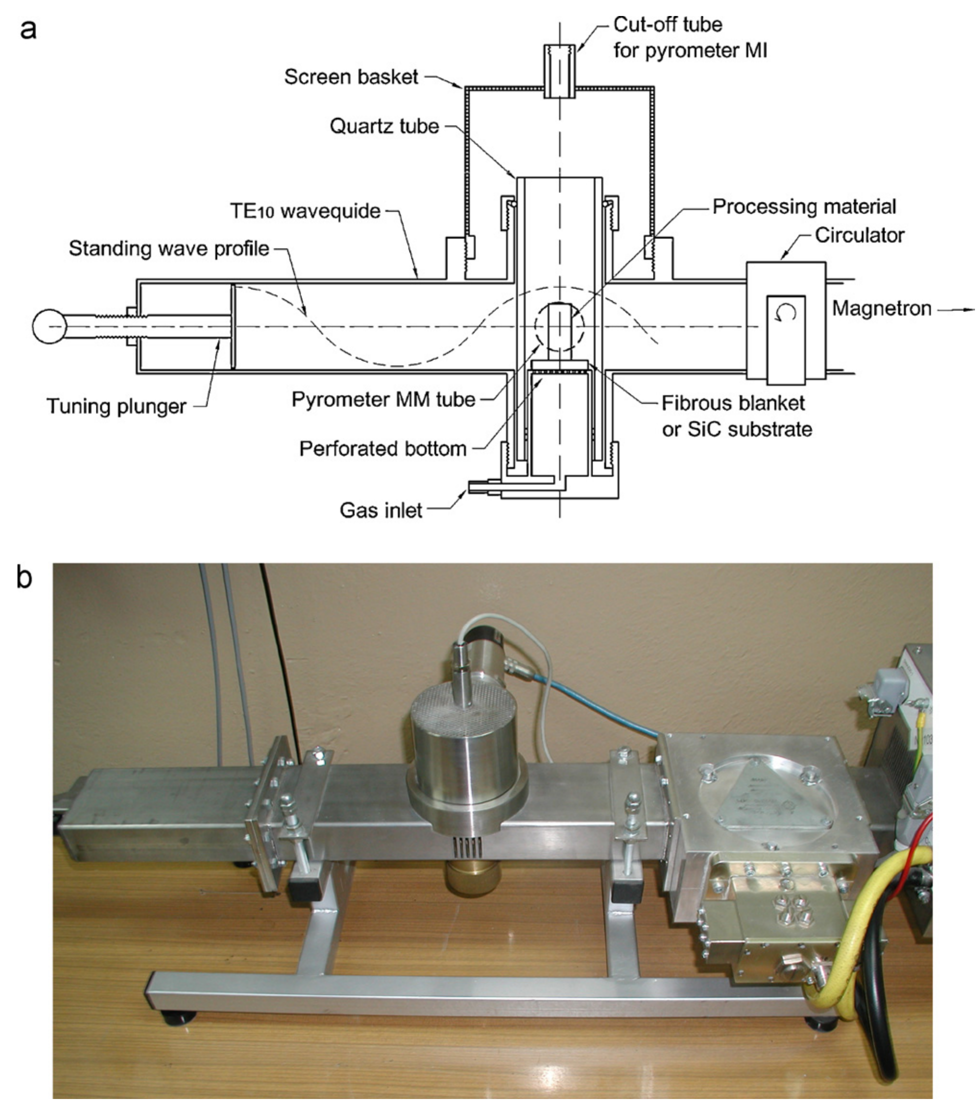

Fig. 1. (a) Diagram of single mode microwave reactor, and (b) a photograph of the reactor with circulator to protect 2.45 GHz magnetron.