Preparation of porous Al$_2$O$_3$-Ti-C perform by combustion synthesis

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Abstract

Using combustion synthesis porous ceramic preforms for composite reinforcing were produced. Prepared mixture of alumina Saffil fibres, Ti powder and graphite flakes, after drying were placed in waveguide of microwave reactor. Supplied with constant power of 540W magnetron ignited and maintained reaction in flowing stream of CO$_2$ gas. Al$_2$O$_3$ fibres should improve preliminary endurance of perform, whereas Ti powder processed to hard titanium carbides and oxides. During microwave heating ignited plasma additionally improve process and partly fused metallic Ti.

Recorded temperature curves were similar for various samples. The highest synthesis temperature revealed samples containing 10% of Al$_2$O$_3$, 10% of Ti and 5% of graphite, all percentages atomic.

Microscopic observation showed considerable microstructure inhomogeneity of some samples. Both irregular component ordering and partly processed Ti particles inside preform exclude them for subsequent infiltration. Chemical analyze EDS of Ti based compounds partly confirmed work purpose, evidencing presence of Ti oxides and carbides. Independently of graphite content these compounds formed folded strips around solid or empty volume. Depends on CO$_2$ availability, reaction could be slowed down resulting in more compacted Ti compounds. Created as a result of combustion synthesis Ti compound after infiltration with liquid metal properly bounded with the matrix. It could be assumed that redox reaction proceeded and on surface of Ti compound alumina and Al-Ti compounds were created.

The preforms of proper strength and homogeneous structure were infiltrated with AlSi7Mg by squeeze casting method. In relation to typical composite reinforced only with fibres no significant increase of defects quantity was observed. Preliminary examination of mechanical properties confirmed that assumed work purpose is reasonable.

Key words: preform, combustion synthesis, composite, infiltration

1. Introduction

Manufacturing of composite materials by infiltration of porous ceramic preform allows to locally reinforce casting in the most loaded places. Properties of preform play fundamental role to achieve desired improve of composite quality. Presented in this work production method of preform utilizes combustion synthesis extensively developed also in the casting and metallurgy areas [1-4].

Natural brittleness of intermetallic compounds, often manufactured in these processes, can be reduced by means of additional components or creating laminates [5-9]. Assumed in this work conception consists in infiltration with relatively plastic aluminium alloy of preform pores built from hard and brittle phases. Used microwave reactor for igniting of plasma and combustion synthesis allows not only to improve the process but also create the structure and control reaction degree of initial mixture.
Benefits of microwave heating or plasma application have been already confirmed [10-12]. In the experiment a special reactor allowing for concentration of field and heating of metallic materials was built. Some of research results requires further additional analyses and therefore sometimes there were used general statements.

2. Experimental procedure

Saffil fibres and powders provided by Alfa Aesar company are used for production of preform. Granularity of Ti, C amounts 44 μm (-325 mesh) while Al₂O₃ +4% SiO₂ fiber diameter ranges between 4-6μm. A proper portions of powder and fiber were mixed in inorganic binder solution and next dried and pressed into perpendicular 1x4x6cm samples. Volume content of fiber was constant and amounted 10%, Ti powder ranged between 10-12% and graphite 5-15%. Simplified symbols informing about volume content of Al₂O₃ fiber-A, titanium -B and graphite -C were used. Exemplarily, sample marked with A10T10C6 symbol contains 10% of Al₂O₃, 10% of Ti and 6% of graphite.

Prepared samples were heated in microwave field to ignite combustion synthesis and process preform component with flowing CO₂ gas. Designed and built microwave furnace consists of rectangular waveguide, chamber with quartz tube and moveable short circuit at the end of the line. More detailed description was presented in [13]. Temperature was measured with a pyrometer Raytek Marathon MM with 500-3000°C temperature range and the measuring spot dia. 0.6 mm. Magnetron was supplied with 240 W power supporting plasma and synthesis development.

Preforms were infiltrated with EN-AC AlSi7Mg aluminium alloy by direct squeeze casting. Preheated to about 500°C preforms were put into die, then liquid metal was poured and immediately pressurised with 40 MPa. Microscopic observation were performed with an optical microscope and a scanning microscope Hitachi S-3400N equipped with a microanalyser EDS (4 nm, BSE detector). Phase identification was carried out using an X-ray diffractometer Rigaku Ultima IV with Cu Kα radiation at 40 kV and 40 mA and using ICDD (2008) cards.

3. Combustion synthesis

Comparing examined samples, course of combustion synthesis, temperatures changes and process dynamics were analyzed. The maximum temperature of the synthesis for samples without graphite usually ranged between 1560-1670°C, with 6% of C between 1500-1660°C and 1560-1670°C for samples containing 10% of C. Temperature curves of all samples were fairly similar (Fig.1), though material susceptibility to microwave heating was different. Presented curves do not reflect often observed fluctuation from discharges and unstable plasma. Operating microwaves resulted in slower cooling, and in 800-600°C range temperature was almost constant. Heated the A10T12C6 samples revealed sometimes second temperature increase presumably as a result of reaction of substrate remains.

Combustion synthesis with flowing CO₂ gas may include complex reactions with all system substrates. Probably proceeded intermediate stages related to adsorption and diffusion gas elements into solid particles. Taking into account EDS, XRD analyze results and literature investigation in Fig.2 is presented proposal of synthesis course.

CO₂ molecules are first of all oxygen source and by graphite or on Ti particle surface forms carbon monoxide. As a reaction result strongly increases temperature and molten Ti vigorously reacted creating spread and grained structure of Ti-O-C compounds. Component transport can be significantly disturbed by microwave field and induced plasma. Though Ti particles reflect microwave then on their surface, especially at sharp corners are induced currents resulting in discharges. This increases temperature and also disorders magnetron work. Analyze of microwave influence based on observation and comparison of samples produced in microwave field periodically disconnected have not clear explained this problem yet.
Microscopic observation showed poor homogeneity of samples and considerable complexity of its structure. Light areas in Fig. 3 present matrix of AlSi7Mg aluminium alloy. Irregular spreading phase are composed mainly from titanium oxide (Fig. 4). Properly embedded in the matrix, without reaction products or porosities at the phase boundary can efficiently enhance composite hardness [14]. Unfortunately, at limited gas availability inside perform or insufficient processed components there were formed impossible to infiltrate pores (Fig. 3).

Flaky structure of graphite despite of high temperature was maintained during synthesis (Fig. 5b). No reaction products at the boundary between graphite and Ti compound or aluminum alloy were observed. It is possible that they were displaced or only intermediate gas phase (CO) reacted.

Fig. 4 Ti based compound a) EDS analyze b)
uniform structure were used to locally reinforce aluminum casting. Presence of very hard compounds based on Ti and graphite as a lubricant could significantly improve wear resistance.

Fig. 5. Microstructure of the A10T10C10 sample a), visible graphite flakes with Ti compounds embedded in aluminum alloy matrix

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References