

## Synthesis of Ti-Al porous preform

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### Manufacturing and processing

#### ABSTRACT

**Purpose:** Article describes production method of intermetallic porous preform for reinforcing of composite materials. Usefulness of the high temperature self-propagating synthesis (SHS), with appropriate modification, to produce preform for production of composite was evaluated.

**Design/methodology/approach:** Mixture of aluminum and titanium powder was cold isostatically pressed (CIPed) and produced cylindrical pill was ignited in microwave field. Obtained structure usually have open porosity what enables for pressure infiltration with molten metal. The investigations of the structure of preforms on the scanning electron microscope (SEM) were made.

**Findings:** The produced preforms in most cases reveal open porosity, uniform morphology and are suitable for infiltration process. After reaction Al-Ti compounds form partly globular structure with microhardness much higher than substrates

**Research limitations/implications:** In the actual stages of this work proposed method can be used for manufacturing of porous preforms, which mainly contain  $Al_3Ti$ . During reaction, mixture compacts with the molar ratio of  $Al/Ti=1$  were transformed into inhomogeneous structure.

**Practical implications:** Obtained preform can be widely used as the reinforcement to produce hybrid composite materials by the infiltration method. Aluminum casting alloys can be locally reinforced to improve hardness and resistance to oxidation at high temperature.

**Originality/value:** Article is valuable for persons engaged in production of casting composite materials reinforced with porous preform. Proposed method allows to incorporate hard structures from Al-Ti compounds into aluminum casting.

**Keywords:** Composite; SHS; Preform; Infiltration

### 1. Introduction

Presently the most prospective methods of producing cast composite materials include squeeze casting process, where a liquid alloy infiltrates a porous preform, which exhibits a certain intrinsic structure and planned porosity. The matrices are typically made on the basis of ceramic or carbon fibres which are joined with silicon or phosphor binder. The last attempts concern manufacturing of materials using the high-temperature synthesis of metal powders, which reveals special properties. Unfortunately they usually exhibit large brittleness [1] and porous structures what limit its application. Amongst promising metals being fit for a synthesis are Al and Ti. Different methods were attempted for synthesis of Ti-aluminides. Depending on starting composition of stoichiometric ratio and

thermodynamic conditions several intermetallics are possible. For the Al-Ti system usually the atomic diffusion at the interface determines formation on Ti particles layer of  $Ti_3Al$ , next  $TiAl$  and finally beside aluminum particles  $Al_3Ti$  layer. However among various possible aluminides the formation of the intermetallic  $Al_3Ti$  is thermodynamically and kinetically favored [2-4]. Below about  $600^\circ C$ , i.e. below the aluminum melting point the interfacial diffusion reaction between solid Al and solid Ti is the prevalent formation mechanism. Above this temperature during an exothermic reaction, liquid Al infiltrates a Al-Ti compact [5]. Should be also emphasized significant influence of adiabatic and maximum temperature on structure of products. The properties for Al-Ti compounds are given in Table 1. Some parameters are determined in accordance with the assumptions used in the model and vary between the authors. Mechanical performance of Ti-Al alloys, depending on their

microstructure, makes this materials attractive for lightweight high-temperature application in aerospace and automotive industry, especially in turbine or valve elements submitted to oxidization at high temperature and in thermal protection systems. As low density materials reveal oxidization and corrosion resistance, good base-adherence of the scale, low thermal conductivity and high hardness [6-9]. Moreover by application of aluminum coating inducing  $\text{Al}_2\text{O}_3$  scale, the oxidation resistance can be significantly enhanced [10]

Table 1.  
Enthalpy and adiabatic temperature [11-13]

|                        | $\Delta H_{(298)}$ [kJ/mol] | $T_{ad(298)}$ [K] |
|------------------------|-----------------------------|-------------------|
| $\text{Al}_3\text{Ti}$ | -146.440                    | 1647              |
| $\text{AlTi}$          | -75.312                     | 1514              |
| $\text{AlTi}_3$        | -98.324                     | 1185              |

Also the appropriate infiltration pressure and the matrix temperature should be adjusted to create the correct link between the matrix and the reinforcing phase.

For intermetallic preform other serious problem may occur during manufacturing. Very important is the uniform arrangement of all components inside the preform structure and its open porosity. In some cases different thermal properties of these components need special preparing operations.

## 2. Experimental procedure

The porous performs were made of elemental titanium (99,5%, -200 mesh) and aluminum (99,9%, -200 mesh) from AlfaAesar. The powders were mixed to ensure homogeneous microstructure. Cylindrical samples were cold isostatically pressed (CIPed) at 500 MPa with a diameter of 23-mm and a height of 4 mm. The samples were placed onto alumina fiber blanket with SiC susceptor, which couples microwave energy at wide range of temperature. Microwaves are reflected from the surface and therefore do not heat metals. Metal have high conductivity and are classed as conductors. Microwave heating of material depends on its dielectric loss factor, which is a measure of the ability of the material to dissipate the energy.

Preheated samples were next ignited in microwave head placed in waveguide steel sleeve. The microwave power is supplied from unit that contains a magnetron tube, transformer and controls. The system utilizes 0-900W power at 2,45 GHz. Continuous temperature measurement were made using one-color optical pyrometer (Raytek, model Marathon MM, temperature range: 540-3000°C). The measured spot on the surface was approximately 0,6 mm in diameter near the outer perimeter of the sample. Thermal activation was carried out in quartz tube under flowing argon.

Some of the prepared performs were subjected to infiltration by direct squeeze casting method. Before infiltration with pressure 75 MPa the performs were preheated to ca. 500°C.

## 3. Discussion of test results

Microscopic investigations were performed by using of optical and scanning electron microscopy with EDS analysis. Observation of microstructure after combustion synthesis revealed

good uniform arrangement of products and pores (Fig.1) only when stoichiometric molar ratio leads to  $\text{Al}_3\text{Ti}$  compounds.

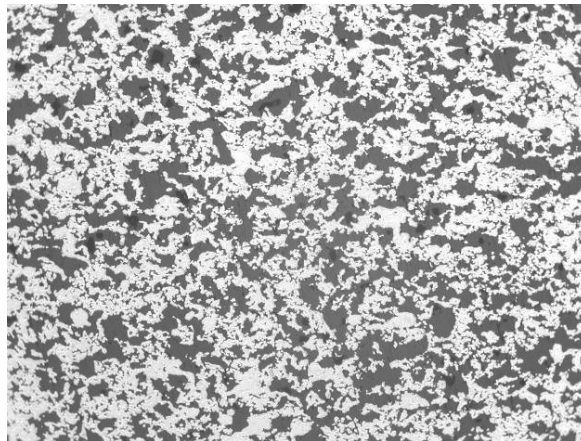


Fig. 1. Optical micrograph of porous preform from  $\text{Al}_3\text{Ti}$  compound (light area, dark – resin)

Homogeneous microstructure appears in entire volume of specimen independently of distance from ignition area or outer perimeter, probably faster cooled. Also for thicker pill shape specimens combustion synthesis allows to produce structure with regular porosity, unlike samples with  $\text{AlTi}$  compounds. In this case partly solid microstructure especially at external surface posses large and small pores (Fig.2). Probably partial melting of the specimen during combustion develops porosity into large voids within liquid, fully dense material.

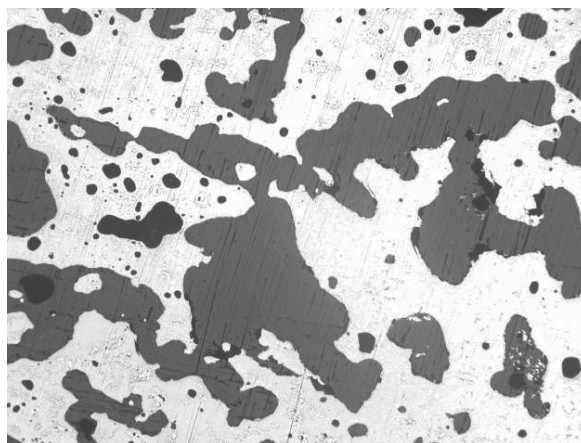


Fig. 2. The microstructure of porous preform from  $\text{AlTi}$  compound

Moreover, because resin under vacuum cannot fill some pores they seem to be closed, so infiltration with molten metal can be very difficult.

Fracture of perform made from  $\text{Al}_3\text{Ti}$  compound during scanning microscopy observation also confirmed regular morphology and sufficient porosity enables infiltration (Fig.3). Pores are evenly distributed and uniform in size.

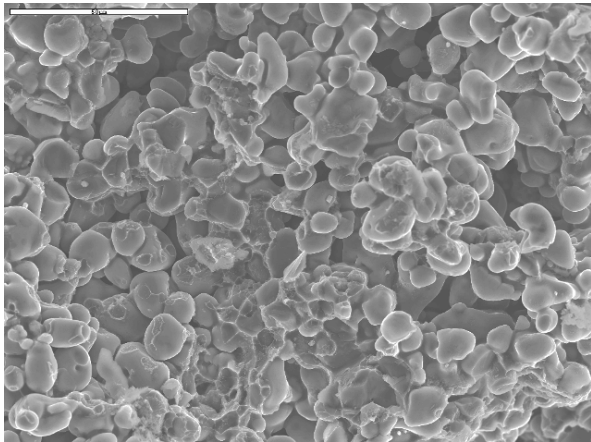


Fig. 3. SEM micrograph showing morphology of  $Al_3Ti$  porous structure after combustion

Combustion aluminide products usually tend to globular shape (Fig.4). The conversion of reactants to products with change in molar volume, producing pores and new morphology. Very important is intensity of exothermic reaction. Observed globules probably arise at presence of partly liquid products which flow away from the centre of combustion reaction.

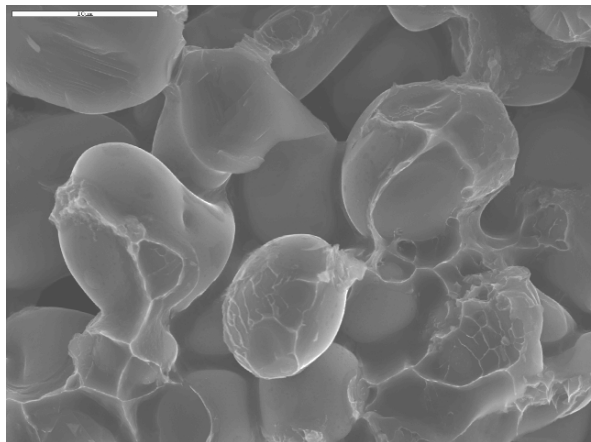


Fig. 4. Fracture surface (SEM) with  $Al_3Ti$  globules

EDX analysis of the fracture surfaces shows high Al and Ti peaks (Fig.5). The composition of various areas, smaller or larger, was similar. In some cases oxide impurities from Ti powders or due to leak in argon protection atmosphere system occurred. Generally atomic ratio of 3,16 indicates formation of  $Al_3Ti$  compound (Table 2).

Table 2.

Results of EDS analysis from area presented in Fig 3

| Element | Al    | Ti    | O     |
|---------|-------|-------|-------|
| % at.   | 63.79 | 20.18 | 16.03 |
| % wt.   | 58.46 | 32.82 | 8.71  |

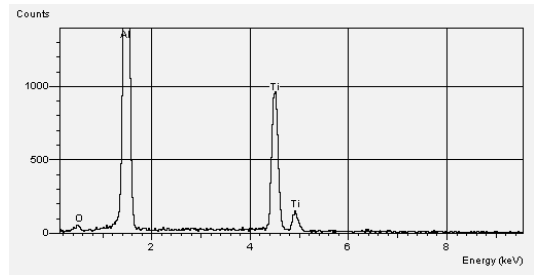


Fig. 5. EDS analysis of the preform

Because of the low exothermic character of the reactions of Ti and Al to synthesize intermetallic compounds by SHS, cylindrical specimens were preheated. Probably at ignition temperature aluminum powder melts and combustion reaction develops through entire specimen. In the case of  $Al_3Ti$  compounds there was a wide propagation front, which reaches ends of sample and next reaction flamed up the sample to very bright colour. As a result no sharp peak of maximum temperature ( $T_m$ ) is observed (Fig.6). Powder mixture enables to synthesize  $AlTi$ , which burned more vigorously. Specimen was also preheated but propagation front started rapidly. Detection of temperature by means 0.6mm spot of pyrometer recorded sharp peak of  $T_m$  temperature (Fig.7).

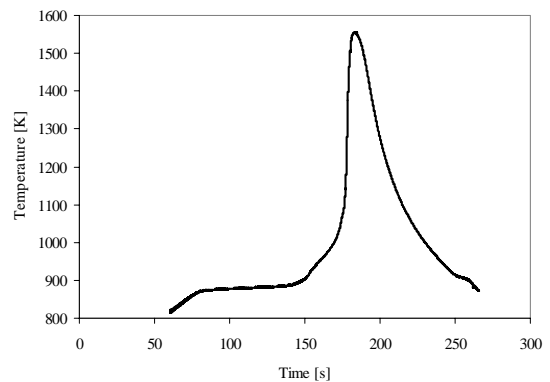


Fig. 6. Temperature profiles for  $Al_3Ti$  in microwave activated combustion synthesis

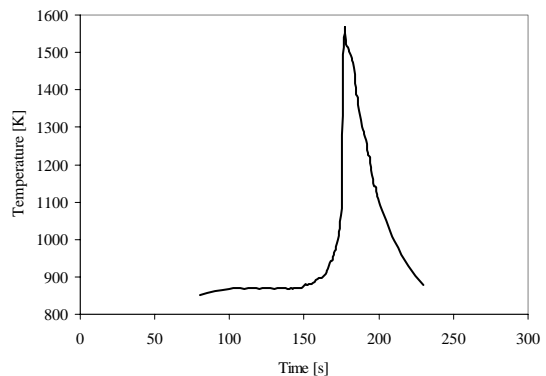


Fig. 7. Temperature profiles for  $TiAl$  compound

Produced porous perform were infiltrated with aluminum alloy by direct squeeze casting method under 75 MPa pressure. Using standard temperature parameters and preheating perform before infiltration, porous structure was filled with molten metal to maximum density composite (Fig.8). Microscopic observation revealed small amount of porosity probably due to incorrect pressure parameters, which were used to infiltrate fibre perform [14]. These preliminary attempts confirmed usefulness of perform for reinforcing casting in required areas.

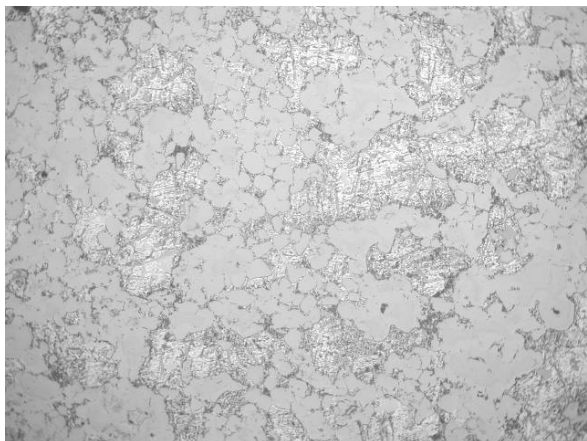


Fig. 8. Optical microstructure as cast aluminum alloy composite reinforced with  $Al_3Ti$  perform

#### 4. Conclusions

The manufacturing process results in the production of porous Al-Ti intermetallic preform suitable for infiltration by liquid metal alloys.

Prepared from proper amounts of Al and Ti powders compacts were burnt in microwave field. The high-temperature synthesis permitted to manufacture porous intermetallic structure with relatively regular and uniform morphology. When stoichiometric molar ratio Al/Ti=1 then produced AlTi sample synthesized violently and structure of such perform was irregular and pores with different shape segregated in the centre of the perform. These perform were not subjected to infiltration.

Fractures of  $Al_3Ti$  performs were observed by means of scanning electron microscopy (SEM) with EDS analyzer. They are characterized by open porosity and globular combustion products. Chemical composition of all areas was similar and proved that combustion carried out completely. Produced  $Al_3Ti$  performs were next, using squeeze casting method, infiltrated with casting aluminum alloy. Optical microscopic observation of microstructure showed that composites have no porosity and the performs can be used to reinforce locally casting parts.

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