



Reactive melt infiltration of copper in Al–Cr preforms produced through combustion synthesis



Krzysztof Naplocha*, Kazimierz Granat, Jacek Kaczmar

Wroclaw University of Technology, ul. Lukasiewicza 5, 50-371 Wroclaw, Poland

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ABSTRACT

Combustion synthesis of Al–Cr preforms used for infiltration and reinforcing of composite materials was developed. Compacts of powdered Al and Cr with stoichiometric ratio Al/Cr equal to 2/1 were synthesized in a microwave reactor furnished with a pyrometer for controlling phase transformations. Due to low enthalpy of the reaction, green compacts were preheated and ignition occurred together with partial melting of Al at the interface with Cr particles. The synthesis proceeded by peritectic transformations $L + Al_7Cr \rightarrow L + Al_{11}Cr_2 \rightarrow L + Al_4Cr$, reaching maximum temperature of ca. 1000 °C. Porous structures including residual unprocessed Cr particles were soaked to homogenize them and to transform the phases into the stable intermetallic compound Al_9Cr_4 . Reactive infiltration of the preforms with molten Cu proceeds along with interfacial diffusion of Al that, released from a preform, infiltrates into the matrix changing its composition to $Cu_9Al_4(Cr)$. At the same time, the preform is decomposed and converted into a mixture of globular precipitates of $Cr_{52}Al_{35}Cu_{13}$ embedded in the $Cu_{47}Al_{41}Cr_{12}$ phase. The produced composite materials exhibit significant hardness and oxidation resistance at elevated temperatures. The protective layer is composed of oxides Al_2O_3 and $(AlCu)_2O_3$ created at parabolic constant oxidation rate (k_p) equal to $1.9 \times 10^{-6} \text{ g}^2 \text{ m}^{-4} \text{ s}^{-1}$.

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1. Introduction

Strengthening of copper brings the desired increase in hardness and abrasion resistance while maintaining its very good thermal and electrical conductivity. Thus, these materials are applied in manufacture of electrical components, contacts, pantograph linings and switches [1,2]. Cu–Cr alloys are used in construction of high voltage vacuum switches [3], whereas intermetallic compounds Al–Cu–Cr as quasicrystalline materials are characterized by high hardness, low coefficient of friction, resistance to heat and corrosion, and can be used for strengthening coatings or composite materials [4].

Manufacture of Cu-based composite materials by casting methods, with a part of liquid phase, is a relatively new technology. Typically, it is based on alloying the components or on pressureless infiltration. Kezheng et al. [5] infiltrated porous alumina preforms aiming at producing a wear resistant material with good thermal and electrical conductivity. In order to improve wetting of the reinforcement and to obtain good bonding with the matrix, an

alloying element containing 10% Ti was used. A similar method with alloying additives like Si, Al, Cr, Ti was applied in [6]. Although there was a significant amount of reaction products at the interface matrix/SiC, increase in flexural strength and Young's modulus was satisfactory. The frequently used chromium accumulates at the interface and as a reactive element forms a compound better wettable by the liquid Cu [7].

Recently, an intensively developing technology for Cu-based composite materials is the reactive infiltration processing (RIP). A porous material (preform) in contact with the liquid alloy is subject to transformation, changing its chemical composition and properties. Depending on kinetics of the reaction and rate of infiltration, the transformation can proceed simultaneously with infiltration, followed by complete saturation or during additional heat treatment. If the system is highly reactive and the preform exhibits low permeability, the reaction products may choke flow of the metal and complete conversion becomes impossible. From the other side, at significant size of pores, free flow and slow rate of the reaction, the transformation will be incomplete and annealing of composite materials is necessary. This can result in coarsening of the microstructure and forming porosity due to the change of lattice parameters of the substrate and product. Therefore, the process requires precise selection of chemical composition of the preform and parameters of infiltration.

* Corresponding author. Tel.: +48 71 3204235; fax: +48 71 3280670.

E-mail address: krzysztof.naplocha@pwr.edu.pl (K. Naplocha).

Problems related to poor wettability or to choking infiltration by growing reaction products can be overcome by the use of high infiltration pressures typical in the squeeze casting method. In [8], distance of infiltration of an Al alloy and conversion degree of Fe-fiber preforms under pressure of 25 MPa were examined. Along with decreasing temperature or increasing fiber diameter, the reaction products in the form of various FeAl_{1-x} compounds grew on surfaces of the fibers, in some cases blocking the reaction front. Heat of the reaction was a key factor that, when dissipated, remelted the reaction products and, from the one side, widened the infiltration path but, from the other side, closed pores.

This paper presents a thorough study of a composite material together with its manufacturing technology based on reactive infiltration with pure Cu of porous preforms made of Al–Cr intermetallic compounds. The preforms were prepared by using a mixture of powders with stoichiometric ratio Al/Cr equal to 2/1 by the self-propagating high temperature (SHS) synthesis. The obtained open porosity, suitable morphology, chemical composition and strength enabled pressure infiltration by the squeeze casting method.

2. Materials and methodology

Production of composite materials employs following basic processes: microwave heating, self propagating SHS synthesis and squeeze casting infiltration. To produce porous preforms Al (99.9% Al, –325 mesh) and Cr (99.5% Cr, –200, –325 mesh) powders, provided by AlfaAesar, were mixed in stoichiometric ratio Al/Cr equals to 2:1. Subsequently portion of mixture was cold isostatically pressed to enhance contact between metallic particles and form cylindrical samples dia. 23 mm and 5 mm. The combustion synthesis was carried out in a specially designed microwave reactor with focused radiation in the heating chamber. As described in detail [9] samples were placed in a quartz tube filled with argon atmosphere and then the magnetron supplied with constant power of 240 W heated up samples with SiC washer strongly absorbing microwaves. Temperature of samples was measured by a pyrometer Raytek, model Marathon MM, with diameter of the measuring spot of 0.6 mm.

Infiltration of prepared preforms was performed in the squeeze casting mould made of hot-work tool steel Orvar supreme (5.2%Cr; 1.4%Mo) which can resist severe condition during casting of Cu. As a unique method special parameters were elaborated: temperature of preform, pouring and the die were respectively 1100 °C, 1150 °C and 600 °C. Pressure of infiltration was 90 MPa.

The structures were examined with an optical and scanning microscope Hitachi TM3000 and JEOL JSM-5800LV equipped with an energy dispersive spectrometer (EDS). Phase identification was carried out using an X-ray diffractometer (XRD) Rigaku Ultima IV with Cu K α radiation at 40 kV and 40 mA.

3. Examination results

Materials produced by the SHS synthesis are typically characterized by certain porosity and conversion degree. This is largely determined by the reaction enthalpy and the amount of heat that is divided into portions for preheating the substrates to the ignition temperature and preheating the synthesis products for their proper reacting. Because of low formation enthalpy of Al_4Cr and Al_9Cr_4 , respectively 16 and 17 kJ/mol [10], it is necessary to preheat the samples, in the case of this study up to about 600 °C. Presumably, at this temperature the reaction is ignited, which on a microscopic scale occurs at interrupted oxide layers and direct contact of metal powders. This is consistent with observations of other researchers at synthesis of compounds containing Al [11,12]. Dissipated energy preheats adjacent layers to the melting point of Al at which significant acceleration of the reaction was confirmed on the basis of dT/dt ratio analysis. This ratio reaches about 130 K/s at 670 °C, see Fig. 1b. Analysis of the derivative curve with reference to phase transformations of the binary Al–Cr system enables determining a sequence of reactions and phase transformations. The first arising phase is Al_7Cr that has minimum formation enthalpy of 22.6 at 710 °C [10] and the lowest liquidus temperature (661.5 °C) among all the existing compounds of the Al–Cr system. According to the EHF model (Effective Heat Formation) [13], the

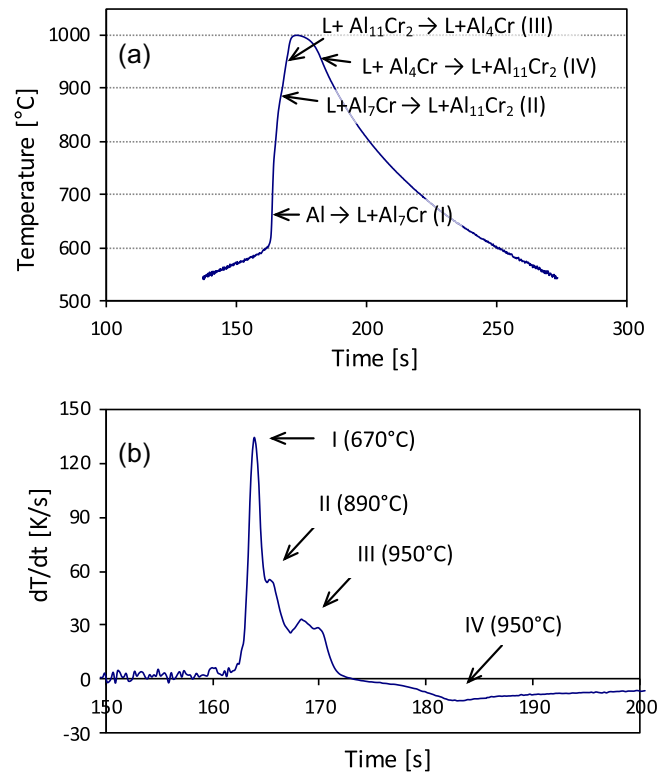


Fig. 1. Time–temperature profile for combustion synthesis of 2Al + Cr specimens (a) and derivate of temperature in time (b) with marked phase transformations.

largest atomic mobility of the deficient element Cr (2%) occurs then at the interface and the compound is formed in the diffusion zone.

With increase of temperature, the created compound Al_7Cr together with liquid Al(L) undergo a transition. Based on the dT/dt analysis, inflections evidencing peritectic transformations $\text{L} + \text{Al}_7\text{Cr} \rightarrow \text{L} + \text{Al}_{11}\text{Cr}_2 \rightarrow \text{L} + \text{Al}_4\text{Cr}$ can be found, as marked in Fig. 1a, at the temperatures of ca. 890 °C (II) and 950 °C (III), resp. The maximum synthesis temperature T_c or adiabatic temperature T_{ad} in the case of no heat loss ($\Delta Q = 0$) can be determined from the relation (1):

$$-\Delta H_{298} = \int_{298}^{T_c} C_p(A_x B_{1-x}) dT + \Delta Q, \quad (1)$$

where $C_p(A_x B_{1-x})$ is specific heat of the product. For manufacturing the preforms composed of Al_9Cr_4 , the adiabatic synthesis temperature should be ca. 1070 °C [9]. The recorded maximum temperature for the samples with similar chemical composition was about 1000 °C, which indicates relatively small heat losses as well as residues of unprocessed substrates. Typically, self-annealing of the sample, progress of the reaction and final formation of the material proceed during cooling. The thermal effect (IV) observed on the cooling segment of the curve represents a reaction inverse to the peritectic transformation (III) and probably it is not the last step of the synthesis.

This is confirmed by XRD phase analysis of the synthesis products, where the phase $\text{Al}_{11}\text{Cr}_2$ was not recognized, see Fig. 2. During the temperature decrease from the point IV (950 °C), during about 100 s nearly final microstructure of the preforms containing Al_4Cr and Al_9Cr_4 was developed. Phase compositions of the materials produced of powders with different granularity were similar, with slightly better visible peaks corresponding to unprocessed Cr powder in the case of thicker particles 74 μm (–200 mesh).

Microscopic observations of the produced structures revealed their significant open porosity suitable for infiltration with liquid

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