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MANUFACTURING OF POROUS Al-Cr PREFORMS FOR COMPOSITE REINFORCING USING MICROWAVE ACTIVATED COMBUSTION SYNTHESIS

WYTWARZANIE POROWATYCH PREFORM Al-Cr DO UMACNIANIA KOMPOZYTÓW POPRZEZ SYNTEZĘ SPALENIOWĄ AKTYWOWANĄ MIKROFALAMI

The combustion synthesis of porous skeletons (preforms) of intermetallic Al-Cr compounds intended for metal matrix composite MMC reinforcing was developed. Mixture of Al and Cr powders with granularity of −10, −44, −74μm were cold isostatic pressed and next ignited and synthetized in a microwave reactor under argon atmosphere (microwave-activated combustion synthesis MACS). In order to ignite the synthesis, microwave energy was focused by a tuner on the specimen. The analysis of reaction temperature diagrams revealed that the synthesis proceeded through the following peritectic transformations: L(liquidus)+Al7Cr→L+Al11Cr2→L+Al4Cr. Moreover, EDS and XRD examinations showed that the reaction proceeded between a solid Cr and a liquid Al to create a distinct envelope of Al_9Cr_4 on Cr particle which next extended and spreaded over the entire structure. The produced preforms with uniform structure and interconnected porosity were infiltrated with liquid Cu and Al alloy. The obtained composite materials exhibited high hardness, wear and distinct temperature oxidation resistance.
Keywords: SHS, microwave, Al-Cr, preform, composite

Wytworzono poprzez syntezę spaleniową porowate preformy (szkielety) ze związków międzymetalicznych Al-Cr do umacniania materiałów kompozytowych o osnowie metalowej. Sprasowane na zimno, jednoosiowo (CIP) mieszaniny proszków Al i Cr o ziarnistości −10, −44, −74μm podgrzewano w reaktorze mikrofalowym w atmosferze argonu. W celu zainicjowania syntezy wykorzystano proces aktywacji mikrofalami MACS (microwave-activated combustion synthesis) skupiając promieniowanie na powierzchni próbki za pomocą stroika falowodowego. Na podstawie analizy krzywych temperatur ATD określono przebieg syntezy z następującymi przemianami perytektycznymi: L(liquidus)+Al7Cr→L+Al11Cr2→L+Al4Cr. Ponadto, analiza chemiczna EDS oraz fazowa XRD wykazały, że reakcja przebiega pomiędzy proszkiem Cr oraz ciekłym Al tworząc wyraźną otoczkę ze związku Al_9Cr_4 na cząstkach Cr, które z postępem reakcji rozszerzają się i zanikają. Wytworzone preformy o jednorodnej strukturze z otwartymi porowatościami użyto do infiltracji ciekłym Al oraz Cu i wytworzenia kompozytów. Charakteryzowały się one dużą twardością, odpornością na ścieranie oraz odpornością na utlenianie w wysokiej temperaturze.

1. Introduction

The metal matrix composites MMC are commonly reinforced with particles, fibers and recently with tailored structures formed from a ceramic or an intermetallic compounds. Depending on the physical and mechanical properties of reinforcement composites with relatively ductile metal matrix can exhibit desirable thermal and strength properties, the unique oxidation or the wear resistance [1,2]. The major sectors which used MMC are: military defence, aircraft and currently expansively growing automotive industry [3]. The applications such as the pistons engine where the strength and resistance to thermal fatigue are markedly improved, provide better fuel economy, engine performance and the lower exhaust emissions.

The production methods of composite materials are commonly based on the powder metallurgy or on the liquid metal infiltration of a preform made of the reinforcement. The metal penetrates porous preform which after solidification is embedded into the matrix. The reinforcement can be either continuous, or discontinuous.

In this work manufacturing of an intermetallic Al-Cr compound preforms, for a local reinforcement of composite materials, was developed. The combustion synthesis permits to obtain the structures with open porosity suitable for infiltration with liquid metal. The applied microwave energy activates and supports the process. A special microwave chamber with the focused radiation and the proper field distribution can be easily controlled depending on the charging material.

2. Materials and methodology

For the Al-Cr system, the low enthalpy of forming intermetallic compounds requires preliminary preheating of the initial powder mixture. Thus, a cold pressed of powdered Al

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and Cr mixture was synthetized with a method defined as the microwave-activated combustion synthesis MACS [4]. At first, the proper quantities of Al and Cr powders produced by AlfaAesar with granularity of Al (99.9%Al) −325 mesh and three kinds of Cr (99.5% Cr) powder −200, −325 mesh and 10μm were mixed in the stoichiometric ratios 4:1 and 9:4. Next cylindrical samples dia. 23 mm and 4.5 mm high were cold isostatic pressed (CIP) at 484MPa. The combustion synthesis was performed in a microwave reactor with a tuner focusing radiation on the samples. A typical magnetron was supplied with a constant power of 240W which just before the ignition and the rapid temperature jump was disconnected. The temperature was controlled by the pyrometer Marathon MM Raytek with a measuring spot of ca. 0.6 mm. The sample, partially insulated with Al_2O_3 blanket, touched the SiC washer strongly absorbing the microwaves.

The structures were observed and analyzed with the optical and scanning microscope JEOL JSM-5800LV. The phase identification was carried out by the usage of the X-ray diffractometer (XRD) Rigaku Ultima IVwith Cu Ka radiation at 40 kV and 40mA.

The prepared porous skeletons (preforms) were infiltrated with a molten metal, using the squeeze casting method, to produce the composite materials. Preheated to 700°C or 1000°C, correspondingly for Al and Cu alloys, were placed in the metal die and after pouring immediately, the pressure of 90 MPa was exerted directly on the surface of metal. In the case of usage of high melting point Cu, induced reactive infiltration converted the structure of prefom before complete solidification.

3. Results and discussion

The prepared compacted powders were placed in the chamber of microwave reactor for the activation and supporting of the synthesis. The recorded temperature diagrams showed that the material was heated to the initial temperature of ca. 500°C and then the reaction was ignited causing a melting of Al powder. This corresponds with the data quoted in literature [4,5] where in aluminum-based systems liquid Al without oxides on its surface usually initiates SHS synthesis. In the performed investigation, on the contrary to the literature data [6-8], no significant effect of the powder granularity on the microstructure or on the reaction course was observed. Distinct differences were observed in the reaction velocity, maximum temperature and the volume of residues of unprocessed Cr particles. Only the synthesis with finer, 10μm Cr particles, provided the sufficient conversion of powders and the uniform microstructure. The analysis of the reaction course on the ground of temperature diagrams showed that the synthesis proceeded through the peritectic transformations L +Al7Cr→L+Al11Cr2→L+Al4Cr, see Fig. 1. On the ground of the differential thermal analysis and the usage of EHF [9] model it can be ascertained that the synthesis began with the formation of Al7Cr that is transformed into a relevant compound according to the contributions of substrates.

Depending on the powder granularity prefom, microstructure can comprise a few types of the intermetallic compounds with the unprocessed residues of the initial powders. Such "cored morphology" can be processed and reduced by heating at 520°C for 48h. Thus, the conversion of the substrates was completed, conserving the initial morphology and the porosity of the preform structure.

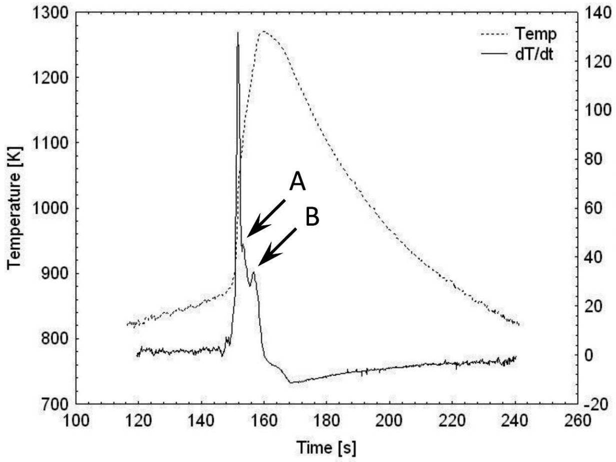

Fig. 1. Temperature profile and its differential for samples with granularity of Cr particles −44μm. Phase transformations are: L $+Al_7Cr \rightarrow L+Al_{11}Cr_2$ at A and L $+Al_{11}Cr_2 \rightarrow L+Al_4Cr$ at B

The microscopic observation of the preform fractures showed a porous structure formed from the dense compound covered with rounded Al-Cr precipitates, see Fig.2a. EDS analysis confirmed the reaction zones around Cr particles. An envelope of Al_9Cr_4 was enclosed in Al_4Cr compound. With the reaction progress, pure chromium slowly passed to the envelope which finally disappeared, see Fig. 2a.

a) b)

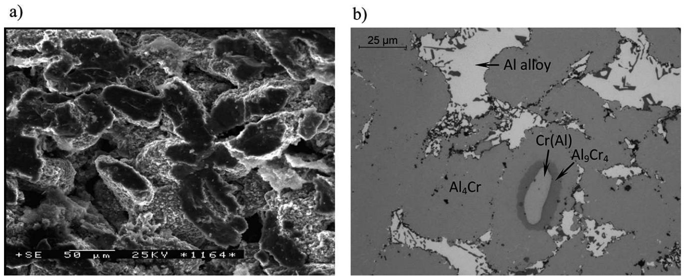

Fig. 2. Fracture of a preform produced from the mixture of 4Al + Cr(−74μm) powders (a), cast composite material obtained by infiltration of the preform with aluminum alloy (light background)

The performed XRD examinations showed that the reaction products included intermetallic phases with the residue of the starting and the transient phases, see Fig. 3. With decrease of Cr granularity, to -10μm, the conversion degree was higher though the existence of Al and Cr was still detected. The prevailing phase of Al_9Cr_4 was accompanied by some amount of Al_4Cr.

The produced porous materials (preforms) are characterised by appropriate open porosity for pressure infiltration. Using the direct-squeeze casting method, the preforms were saturated with liquid Al or Cu to locally reinforce the castings. The examinations of the composite structures showed thorough infiltration, the satisfactory homogeneity and a low residual porosity. The infiltration of the preform with liquid Cu proceeded with the reaction at the interface and interdiffusion of the elements. The intermetallic Al-Cr preforms were decomposed releasing Al which passed into Cu matrix. The morphology of the preform was unchanged though on the base of EDS analysis new phases comprised mixture of globular $Cr_{52}Al_{35}Cu_{13}$ embedded in $Cu_{47}Al_{41}Cr_{12}$ were detected, see Fig. 4. The produced composite revealed a significant increase of hardness and improved wear resistance. Hardness of transformed preform increased from ca. 450 to 700HV0.2 whereas Cu based matrix from ca. 90 to 500 HV0.2.

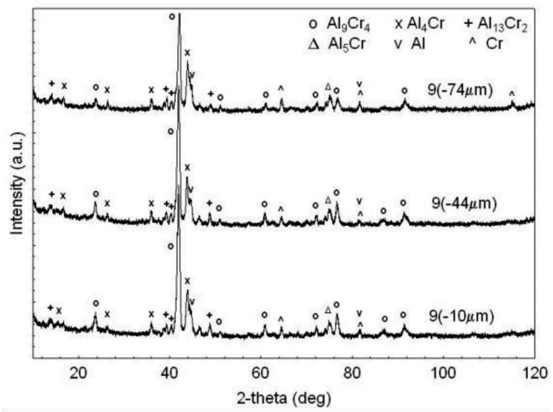

Fig. 3. XRD patterns of 9Al + 4Cr combustion products. The granularity of Cr powder (−10, −44 and −74μm) induces relatively small changes in the product phase composition

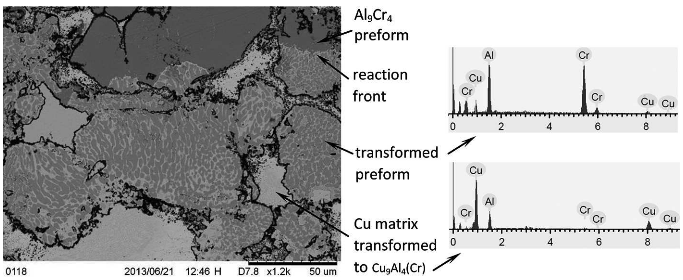

Fig. 4. The front of reactive infiltration of Al_9Cr_4 preform with Cu. EDS analysis of the transformed phase composition for preform and Cu matrix

4. Conclusions

The porous preform of Al-Cr compounds for composite reinforcing were produced using the microwave-activated combustion synthesis (MACS). The compacted mixture of Al and Cr powders were preheated to 500-510°C and ignited with a locally melting aluminium. Synthesis proceeded through the peritectic transformations: L+Al7Cr→L+Al11Cr2→L+Al4Cr leaving, especially in the case of coarse Cr particles, a distinct envelope of Al_9Cr_4 compounds on Cr partly processed particles. The reaction products comprised intermetallic compounds formed interconnected porous structure (preforms) suitable for infiltration with molten metal. Using a squeeze casting pressure infiltration Al and Cu composite materials were reinforced with Al-Cr preform improving hardness and wear resistance. Moreover, the infiltration with molten Cu induced the reaction with a preform which transformed a chemical and a phase composition both of the matrix and the reinforcement.

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