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STRUCTURE OF COMPOSITE POWDERS WITH Fe-Cr AND Fe-Al PHASES, DISPERSIVE CARBIDES AND ALUMINIUM OXIDES

The article presents the structure and phase composition of composite powders with the ferrochromium matrix and dispersion strengthening phases. The composite powders were obtained in the activated self-propagating high temperature synthesis. The complex phase composition of powders confirms an appropriate selection of technological parameters of SHS process.

STRUKTURA PROSZKÓW KOMPOZYTOWYCH Z FAZAMI Fe-Cr i Fe-Al, WĘGLIKÓW I TLENKÓW GLINU

Prezentowano strukturę i skład fazowy kompozytowych proszków z osnową żelazochromu i dyspersyjnych faz umacniających. Kompozytowe proszki z układów FeCr-FeAl-TiAl-Al₂O₃, FeCr-FeAl-TiC-Al₂O₃, FeCr-FeAl-Cr₃C₂-Al₂O₃ otrzymywano w procesie aktywowanej samorozwijającej się syntezy wysokotemperaturowej (ASHS). Ponieważ kompozytowe proszki zawierają twarde fazy międzymetaliczne z układu FeAl, węgliki i tlenki glinu, rozważono i doświadczalnie sprawdzono celowość wprowadzenia do ich składu chemicznego plastycznej osnowy FeCr. Metodami mikroskopii świetlnej i skaningowej określono: strukturę, kształt (rys. 1a, b) i morfologię proszków (rys. rys. 8 i 9). Skład chemiczny i fazowy kompozytowych proszków określono metodami analizy rentgenowskiej: X-ray oraz EDX (rys. rys. 2-7).

Specyficzny skład chemiczny (rys. rys. 2-4) oraz dyspersyjna struktura (rys. 1a, b) kompozytowych proszków zawierających twarde fazy oraz plastyczną osnowę pozwalają na ich wykorzystanie w procesach natryskiwania ciepłego powłok.

Wyniki badań potwierdzają słuszność założeń i koncepcji technologicznej wytwarzania kompozytowych proszków w procesie aktywowanej syntezy wysokotemperaturowej o założonym składzie chemicznym i fazowym.

INTRODUCTION

The intermetallic phases of iron, nickel and titanium with aluminium are the subject of research interest of many scientific centers and they are being manufactured by Exo-Melt method and applied more widely nowadays. An industrial application of intermetallic phase alloys is the main result of scientific and implementation works conducted at Oak Ridge National Laboratory in the United States. An application of the intermetallic phases from the Fe-Al system was discussed e.g. in publications of C. Teastani and others [1]. Works of S.C. Devy, V.K. Sikk, P.F. Tortorelli [2-4] show the basic technologies of the intermetallic phase manufacturing, they characterize their basic physico-chemical properties and trends of potential applications. The powders of nickel and iron intermetallic phases obtained by atomization and reactive synthesis methods were discussed in K.G. Show's publication [5]. The FeAl-TiAl-Al₂O₃ composite powders, also with the carbide phase content, were produced in the activated process of the self-propagating high temperature synthesis (ASHS) [6-7]. The present article characterizes composite powders from the FeCr-FeAl-TiAl-Al₂O₃, FeCr-FeAl-TiC-Al₂O₃, FeCr-Cr₃C₂-Al₂O₃ sys-

tems, which besides phases of high hardness contain a soft ferrochromium matrix. The powders manufactured are designed for the thermal spraying of coatings working at elevated temperatures.

PURPOSE AND SCOPE OF RESEARCH

The main purpose of the study was to work out a high temperature synthesis of composite powders with a FeAl phase which were additionally strengthened with hard phases of titanium or chromium carbides and aluminium oxide. It was assumed that the carbides and aluminium oxide formed during a high temperature synthesis would be an effective and functional filler of powders composite mixtures. It was also assumed that composite powders with an indicated phase composition can be obtained in an aluminocarbonthermal reduction of oxides mixtures or selected mixtures of titanium or chromium ores.

With respect to the fact that composite mixtures contain hard intermetallic phases from FeAl system as well as carbides and aluminium oxides, the purposefulness of

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introducing a plastic matrix powder FeCr into the chemical composition was given consideration and experimentally checked.

It was assumed that the plastic mass fraction of powders matrix should not be higher than 35% taking into account the prospective application of synthesized powders as well as the requirements set to the structure and properties of thermally sprayed coatings.

On the basis of theoretical discussions and the experience following from the undertaken investigations a thesis has been formulated that it will be possible to obtain composite powders with complex chemical and phase composition designed for thermal spraying of coatings in a self-propagating high temperature synthesis, by applying powder mixture activation processes (ASHS).

The scope of the research covered:

- development of a technological procedure of composite powders production from sinters obtained by a self-propagating synthesis method,
- determination of technological parameters of the process,
- classification and disclosure of the produced powders' structure.

MATERIALS AND RESEARCH METHODOLOGY

For the fabrication of reaction mixtures selected oxides were used: Fe_2O_3 , TiO_2 and Cr_2O_3 in their pure form or ground metal ores of titanium and chromium.

Stoichiometric chemical compositions of mixtures were prepared or ones departing from stoichiometry and resulting from basic synthesis reactions, e.g. [8].

The substrates, initial powders, were mechanically homogenized in a rotation-vibrating mill with steel grinding media or in a Turbul's mixer for 0.5÷1 h. The synthesis, depending on the needs, was carried out in steel or ceramic containers with the application of ignition initiation with a resistance spiral or from the container's edge. The slightly sintered synthesis products obtained were broken up in a Fritsch crusher and then milled in a rotation-vibrating mill. Powders were separated on sieves in a vibrating separator.

The powders structure and their shape were observed with a Reichert MFZ light microscope. The morphology and chemical composition of the composite powders produced were determined with a Hitachi S-4200 scanning microscope with an analysis system of the characteristic X-radiation of elements by Noran.

The analysis of ASHS products phase composition was conducted by an X-rays diffraction method on a Philips device with a carbon monochromator of deflected beam, with the application of a copper lamp. The peaks of the phases obtained were identified on the basis of ASTM cards. The results were analysed in order to

correct the chemical composition of the systems synthesized and the conditions of their high temperature synthesis. The technological procedure of composite powders production has been broadly presented in the authors' publications, e.g. [9, 10]. The authors tried to choose such technological conditions of powders fabrication that it would be possible to obtain composite powders of an indicated structure and phase composition which could be transformed for the technological processes of their fabrication on a production scale.

CHARACTERISTICS OF SINTERS SYNTHESIS AND POWDERS PRODUCTION CONDITIONS

In the investigated systems of oxides or metal ores and aluminium and carbon powders used as reducers there is a wide range of compositions similar to stoichiometric ones for which the SHS and ASHS types can be initiated at a room temperature by means of a high temperature heat source. The thermal differential analysis of reaction mixtures presented in the paper showed that all the differential curves have an endothermic effect which is the result of melting aluminium. Exothermic effects of the synthesis are characterized by peaks of different height and width which also determine characteristic temperatures of the synthesis of intermetallic phases with aluminium oxide or the FeAl phase of carbides or aluminium.

Favourable conditions of producing composite sinters with an indicated phase composition are obtained at an initial concentration of the bed and its diversified granulation. The synthesis processes must be carried out in such a way that a „homogeneous” bed structure could be obtained without delamination and globular precipitations of intermetallic phases. The thermal effect of the synthesis can be also regulated by introducing inert additions or additions of lower reactivity to base mixtures.

The self-propagating high temperature synthesis must be conducted so as to obtain a slightly sintered bed of low porosity which is afterwards subjected to mechanical breaking up.

MORPHOLOGY AND PHASE COMPOSITION OF COMPOSITE POWDERS

The observations of powders microstructure made with a light (Fig. 1a, b) and scanning microscope showed that the powders are characterized by a diverse structure (Figs 6-9).

This results from the SHS process specificity, diversified bed granulation and the thermal wave propagation in the bed. The powders are characterized by a depressive structure with precipitations of relevant phases or aluminium oxide. Full interpretation of composite

powders structures goes beyond the scope of the article presented. What deserves attention is the phase and chemical composition of the produced sinters, and afterwards powders, which is presented in Figures 2-5.

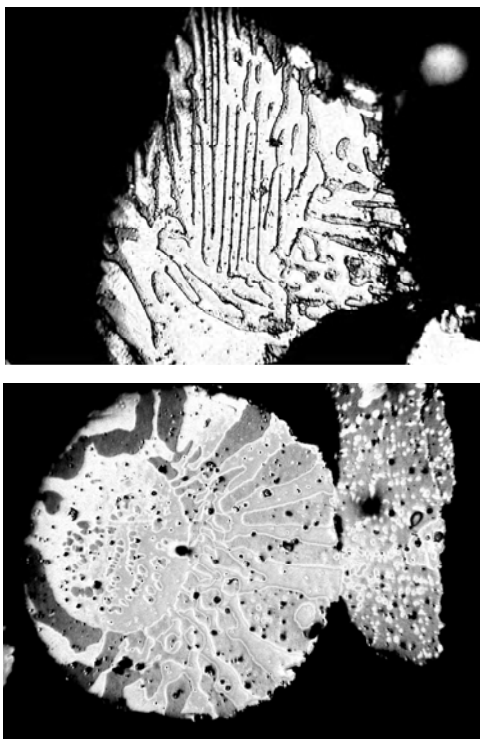


Fig. 1. Structure of composite powders: a) (FeAl)CrAl- Al_2O_3 , b) (Fe-CrAl)-FeAl- Cr_3C_2 - Al_2O_3

Rys. 1. Struktura proszków kompozytowych: a) (FeAl)CrAl- Al_2O_3 ,

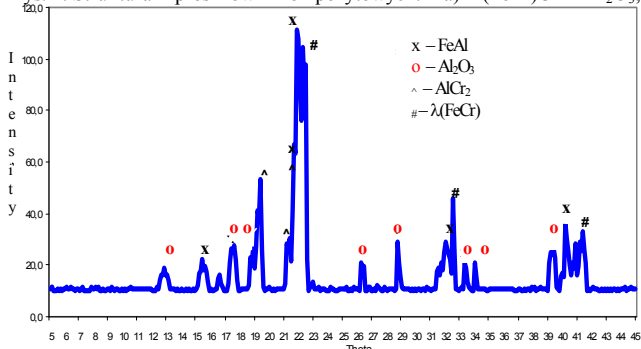


Fig. 2. Diffraction pattern of (FeAl)CrAl- Al_2O_3 composite powders

Rys. 2. Dyfraktogram próbki (FeAl)CrAl- Al_2O_3

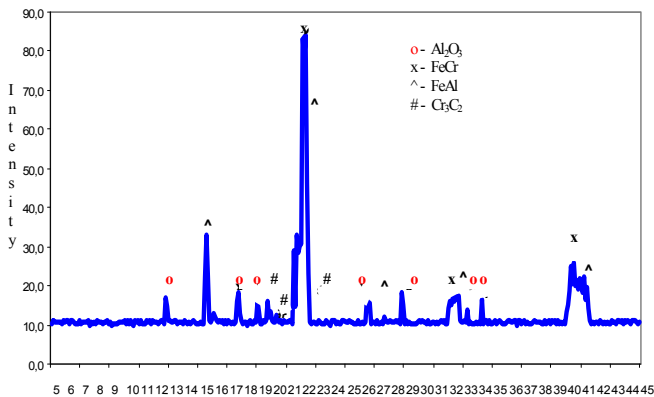


Fig. 3. Diffraction pattern of (FeCrAl)-FeAl- Cr_3C_2 - Al_2O_3 composite powders

Rys. 3. Dyfraktogram próbki (FeCrAl)-FeAl- Cr_3C_2 - Al_2O_3

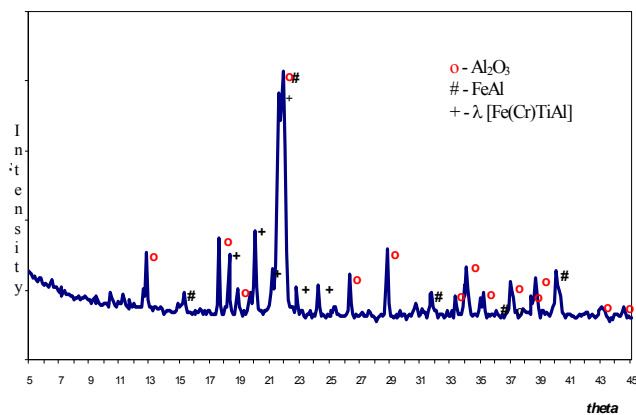
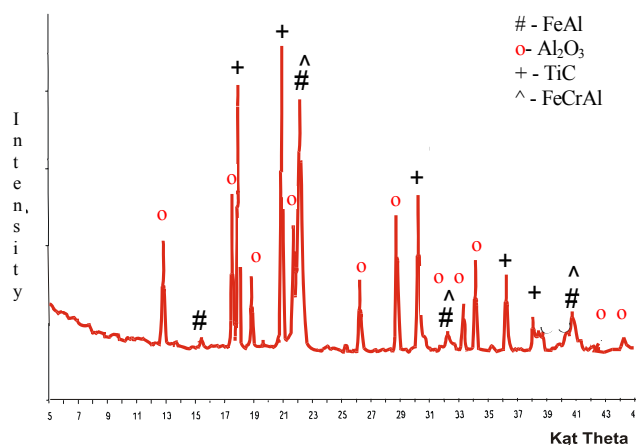


Fig. 4. Diffraction pattern of FeCrAl-FeAl-TiAl- Al_2O_3 composite powders
Rys. 4. Dyfraktogram proszku kompozytowego FeCrAl-FeAl-TiAl- Al_2O_3

Fig. 5. Diffraction pattern of (FeCrAl-FeAl-TiC- Al_2O_3) composite powders



Rys. 5. Dyfraktogram proszku kompozytowego (FeCrAl-FeAl-TiC- Al_2O_3)

It is consistent with the preliminary assumptions made as to the initial chemical compositions of the mixtures.

The morphology of all the synthesized composite powders is characteristic of the method of obtaining them. The powders chemical composition examined in selected places is different, which results from their structure. An average content of elements in the analysed powders surfaces is consistent with the chemical composition of the phases identified on diffraction patterns.

CONCLUSION

The obtained results of the research on composite powders production in an activated high temperature synthesis with an assumed phased composition have acknowledged correctness of the assumptions and the technological conception of their production.

A specific chemical composition and a depressive structure of composite powders which contain hard phases and plastic matrix allow to use these powders in the process of thermal spraying of coatings. The aluminocarbonthermal synthesis of composite powders is

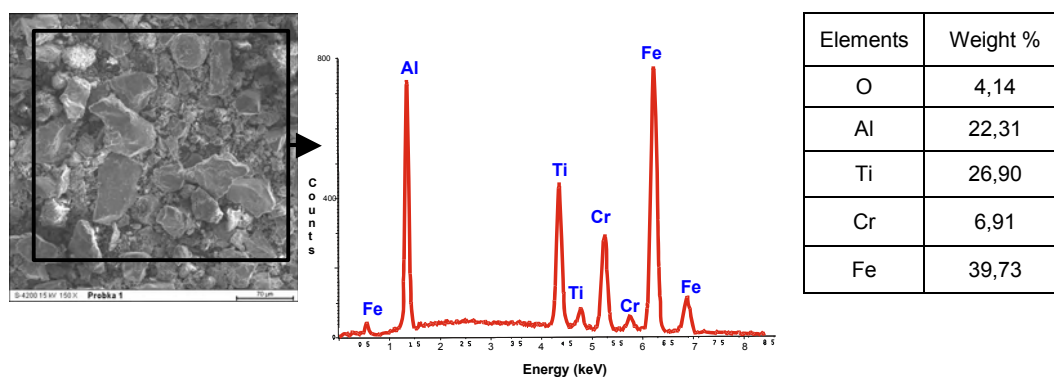


Fig. 6. Morphology (SEM) and a spectrum of characteristic X-radiation (EDX) from FeCrAl-FeAl-TiAl-Al₂O₃
Rys. 6. Morfologia (SEM) i EDX próbki FeCrAl-FeAl-TiAl-Al₂O₃

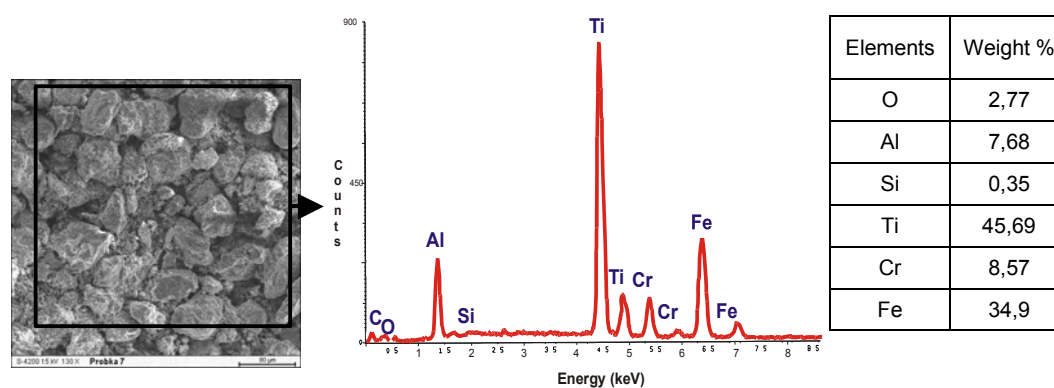


Fig. 7. Morphology (SEM) and a spectrum of characteristic X-radiation (EDX) from FeCrAl-FeAl-TiC-Al₂O₃
Rys. 7. Morfologia i EDX próbki FeCrAl-FeAl-TiC-Al₂O₃

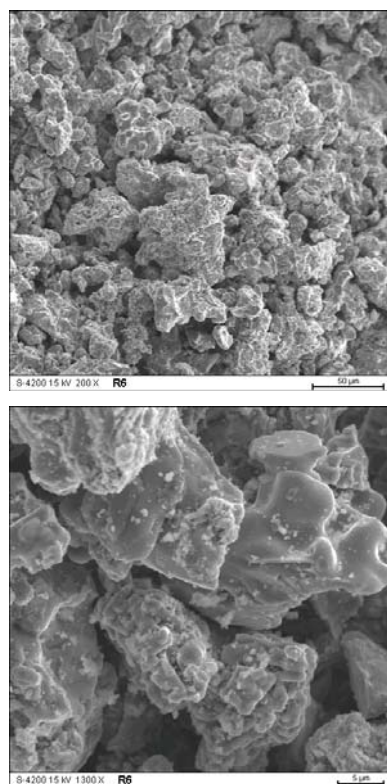


Fig. 8. Composite powder morphology of (FeAl)-CrAl-Al₂O₃
Rys. 8. Morfologia kompozytowego proszku (FeAl)-CrAl-Al₂O₃

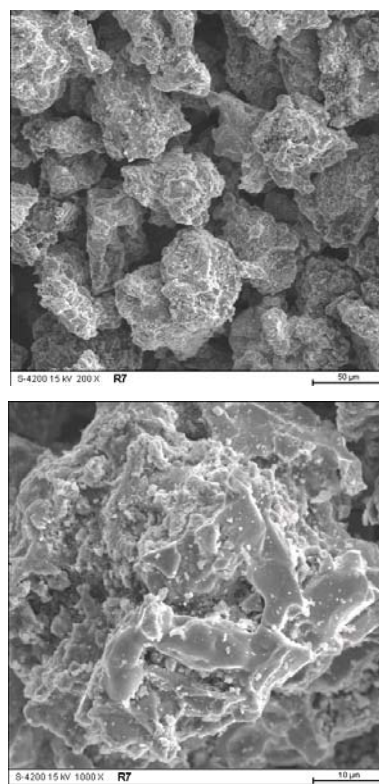


Fig. 9. Composite powder morphology (FeCrAl)-FeAl-Cr₃C₂-Al₂O₃
Rys. 9. Morfologia próbki (FeCrAl)-FeAl-Cr₃C₂-Al₂O₃

a strongly exothermic and complex process in which the selection of technological parameters and the way of synthesis condition homogeneity of the phase and chemical composition of the products. A technological achievement in the conducted research is the properly selected production procedure of composite powders with a complex phase composition.

The paper presents a selected part of the investigations within an extensive research programme concentrating on composite powders production, realized under national and international projects. Under the project ordered by PBZ, works are conducted on the transformation of composite powders synthesis laboratory results and adjusting them to the technological conditions of powders production on a large production scale.

Selected results of investigations carried out under the research projects sponsored by the Scientific Research Committee, are used in the paper.

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Recenzent
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