https://doi.org/10.62753/ctp.2002.03.5.5

Bolesław Formanek[1], Krzysztof Szymański[2], Bożena Szczucka-Lasota[3]
Politechnika Śląska, Katedra Nauki o Materiałach, ul. Krasińskiego 8, 40-019 Katowice

STRUCTURE OF COMPOSITE POWDERS WITH Ni-Cr MATRIX AND FeAl PHASES, CARBIDES AND ALUMINIUM OXIDE

The article presents the structure and phase composition of composite powders with the nickel-chromium 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 KOMPOZYTOWYCH PROSZKÓW Z OSNOWĄ NiCr, FAZĄ FeAl, WĘGLIKAMI I TLENKIEM GLINU

Przedstawiono strukturę kompozytowych proszków o osnowie NiCr z wprowadzoną fazą FeAl, węglikami chromu i tytanu oraz tlenkami aluminium. Proszki o złożonym składzie fazowym wytwarzano w procesie aktywowanej syntezy wysokotemperaturowej ASHS. Określona została procedura technologiczna wytwarzania takich proszków. Strukturę proszków analizowano metodami mikroskopii optycznej i skaningowej. Skład fazowy i chemiczny proszków określono metodami rentgenowskimi X-ray i EDX. Proszki charakteryzują się dyspersyjną strukturą (rys. 1). Wyniki badań składu fazowego proszków przedstawione na rysunkach 2-7 potwierdziły przyjęte założenia ich składu fazowego oraz właściwie dobrane parametry technologiczne syntezy. Wytworzone proszki są przeznaczone do natrysku cieplnego powłok.

INTRODUCTION

The intermetallic phases of iron, nickel and titanium with alumnium 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 Labora-tory in the United States. An application of the intermetallic phases from the Fe-Al system was discussed e.g. in publications of C. Testani and others [1]. Works of S.C. Deevi, 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. Shaw's publication [5]. The NiCr-FeAl-TiAl-Al_2O_3 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 NiCr-FeAl--CrAl-Al_2O_3, NiCr-FeAl-TiC-Al_2O_3, NiCr-FeAl-Cr_3C_2--Al_2O_3 systems, which besides phases of high hardness contain a soft nickel-chromium matrix. The powders manufactured are designed for the thermal spraying of coatings working at elevated temperatures [8, 9].

PURPOSE AND SCOPE OF RESEARCH

The main purpose of the study was to work out a high temperature synthesis of composite powders with a NiCr matrix and 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 introducing a plastic matrix powder NiCr 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%

[1] dr inż., [2] dr inż., [3] mgr inż.

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. [10].

The substrates, initial powders, were mechanically homogenized in a rotation-vibrating mill with steel grinding media or in a Turbulos 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 system.

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. [11, 12]. 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. 1) 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.

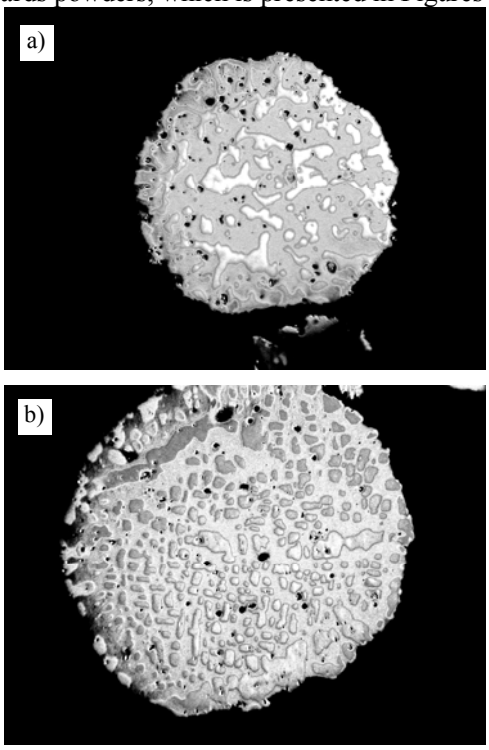


Fig. 1. Structure of composite powders: a) (NiCr)-FeAl-CrAl-Al_2O_3, b) (NiCr)-FeAl-Cr_3C_2-Al_2O_3

Rys. 1. Struktura proszków kompozytowych: a) (NiCr)-FeAl-CrAl-Al_2O_3, b) (NiCr)-FeAl-Cr_3C_2-Al_2O_3

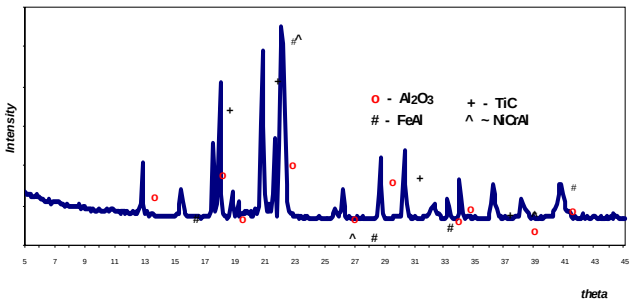


Fig. 2. Diffraction pattern of NiCr-FeAl-TiC-Al_2O_3 composite powders

Rys. 2. Dyfraktogram próbki NiCr-FeAl-TiC-Al_2O_3

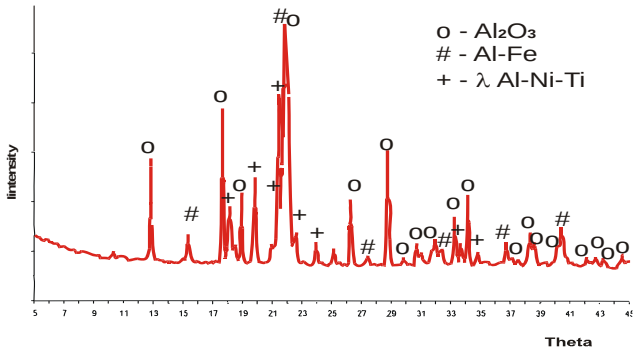


Fig. 3. Diffraction pattern of NiCr-FeAl-TiAl-Al_2O_3 composite powders

Rys. 3. Dyfraktogram proszku kompozytowego NiCr-FeAl-TiAl-Al_2O_3

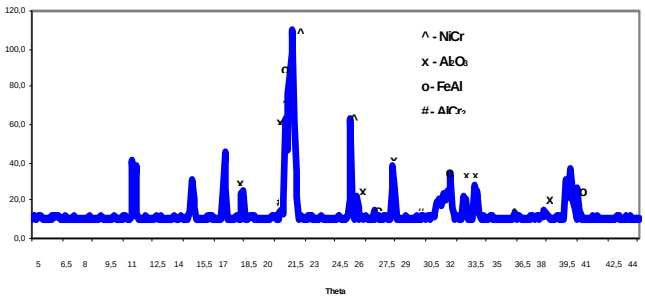


Fig. 4. Diffraction pattern of (NiCr)-FeAl-CrAl-Al_2O_3 composite powders

Rys. 4. Dyfraktogram próbki (NiCr)-FeAl-CrAl-Al_2O_3

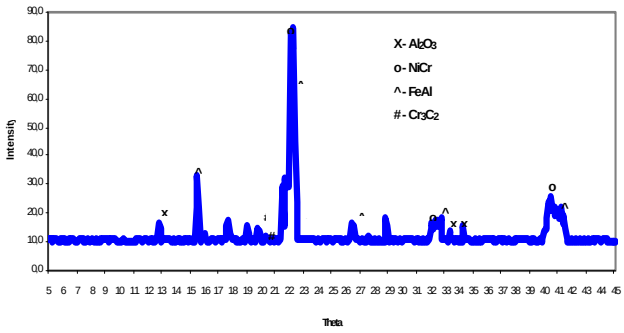
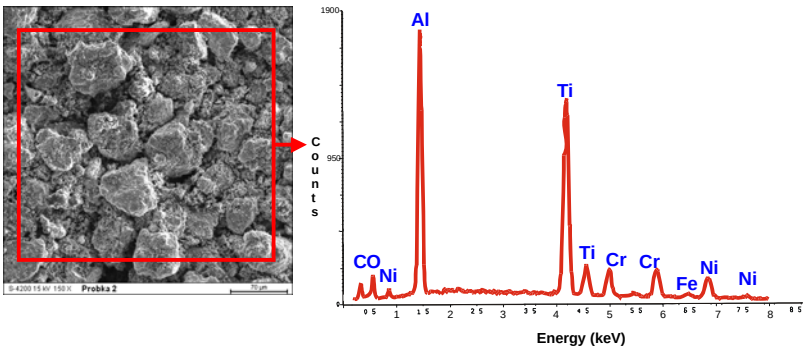


Fig. 5. Diffraction pattern of NiCr-FeAl-Cr_3C_2-Al_2O_3 composite powders

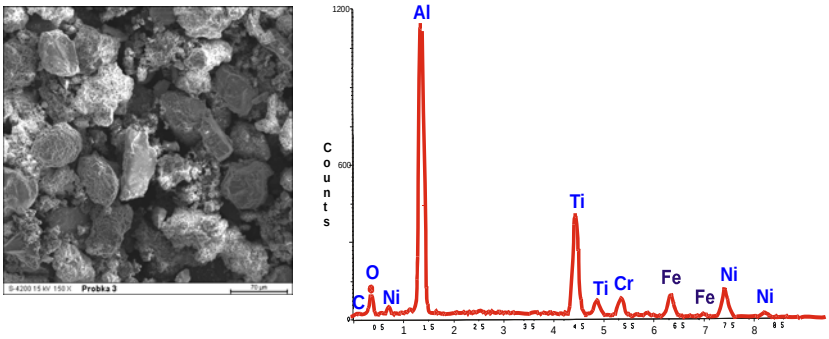
Rys. 5. Dyfraktogram próbki NiCr-FeAl-Cr_3C_2-Al_2O_3



Elements	Weight %
O	7.84
Al	25.03
Ti	37.32
Cr	6.39
Fe	10.50
Ni	12.93

Fig. 6. Morphology (SEM) and a spectrum of characteristic X-radiation (EDX) from NiCr-FeAl-TiC-Al_2O_3

Rys. 6. Morfologia (SEM) i EDX próbki NiCr-FeAl-TiC-Al_2O_3



Elements	Weight %
O	9.09
Al	33.15
Ni	20.33
Ti	22.96
Cr	5.43
Fe	9.04

Fig. 7. Morphology (SEM) and a spectrum of characteristic X-radiation (EDX) from NiCr-FeAl-TiAl-Al_2O_3

Rys. 7. Morfologia i EDX próbki NiCr-FeAl-TiAl-Al_2O_3

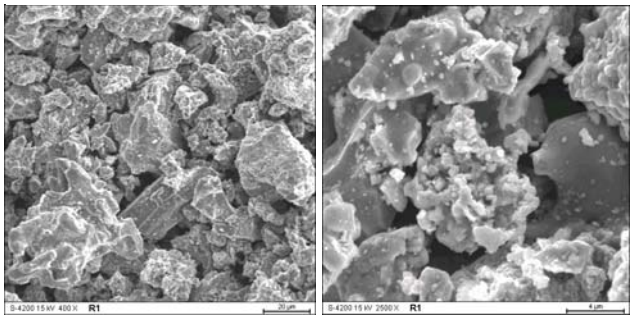


Fig. 8. Composite powder morphology of NiCr-FeAl-CrAl-Al_2O_3

Rys. 8. Morfologia kompozytowego proszku NiCr-CrAl-Al_2O_3

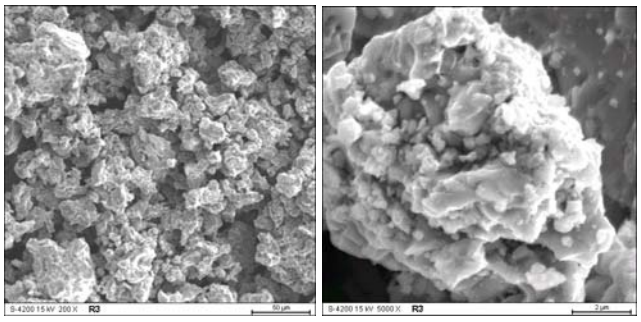


Fig. 9. Composite powder morphology NiCr-FeAl-Cr_3C_2-Al_2O_3

Rys. 9. Morfologia próbki NiCr-FeAl-Cr_3C_2-Al_2O_3

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 alumino-carbonthermal synthesis of composite powders is 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 Committee of Scientific Research, are used in the paper.

REFERENCES

[1] Testani C., Di Giah Francesco A., Tassa O., FeAl intermetallic and application - Pocci D. tines.: An overview, Proc. of International Symposium on Nickel and Iron Aluminides Processing, Properties and Applications, ed. S.C. Deevi, P.J., Maziasz, V.K. Sikka, R.W. Cehh, 7-9 October 1996, Cincinati, 213-222.

[2] Notesan K., Tortorelli P.F., High temperature corrosion and applications of nickel and iron aluminides in coal-conversion power system, Proc. of International Symposium on Nickel and Iron Aluminides Processing, Properties and Applications, ed. S.C., Deevi, P.J. Maziasz, V.K. Sikka, R.W. Cehh, 7-9 October 1996, Cincinati, 265-280.

[3] Sikk V.K., Commercialization of nickel and iron aluminides, Proc. of International Symposium on Nickel and Iron Aluminides Processing, Properties and Applications, ed. S.C., Deevi, P.J. Maziasz, V.K. Sikka, R.W. Cehh, 7-9 October 1996, Cincinati, 361-375.

[4] Deevi S.C., Sikka V.K., Reaction synthesis and processing of nickiel and iron aluminides, Proc. of International Sympo- sium on Nickel and Iron Aluminides Processing, Properties and Applications, ed. S.C. Deevi, P.J. Maziasz,

V.K. Sikka, R.W. Cehh, 7-9 October 1996, Cincinati, 283-299.

[5] Shaw K.G., Reinshagen J.H., Powders production of nickel and iron aluminides, Proc. of International Symposium on Nickel and Iron Aluminides Processing, Properties and Applications, ed. S.C. Deevi, P.J. Maziasz, V.K. Sikka, R.W. Cehh, 7-9 October 1996, Cincinati, 301-310.

[6] Formanek B., Hetmańczyk M., Szymański K., Struktura natryskiwanych naddźwiękowo powłok z kompozytowych proszków aluminidków i tlenków, Konf. Naukowo-Techniczna nt. Nowe materiały - nowe technologie w przemyśle okrętowym i maszynowym, Szczecin-Świnoujście 10-13 września 1998, 317-322.

[7] Formanek B., Hetmańczyk M., Szymański, Przeliorz R., Struktura i własności natryskiwanych naddźwiękowo powłok z kompozytowych proszków FeAl-TiAl-Al_2O_3, FeAl--TiC-Al_2O_3, III Sem. Kompozyty - teoria i praktyka, Warszawa 1998, 85-93.

[8] Formanek B., Szymański K., Szala J., Szczucka-Lasota B., Structure of composite powders with Fe-Cr and Fe-Al phases, depressive carbides and aluminium oxide, Kompozyty (Composites) 2001, 1, 1, 118-122.

[9] Formanek B., Szczucka-Lasota B., Pająk L., Structure of oxide layers on plasma sprayed coatings with Fe-Al intermetallic alloys, AMME 2001, 651-656.

[10] Formanek B., Aluminocarbothermal synthesis of composite powders with intermetallic phases and dispersion oxides, AMME 2000, 191-195.

[11] Formanek B., Kompozytowe proszki z fazami międzymetalicznymi i ceramicznymi, Ogólnopolskie Sympozjum Naukowe nt. Stopy na osnowie faz międzymetalicznych, Warszawa 19-22 października 2000.

[12] Formanek B., Szymański K., Szala J., Szczucka-Lasota B., Structure of composite powders with Fe-Cr and Fe-Al phases, dispersive carbides and aluminium oxides, 2001.

Recenzent
Stefan Szczepanik