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STRUCTURE OF POWDERS FROM NI-AI SYSTEM AND THESE MODIFIED BY CERAMIC PHASES

Microstructure, morphology and phase composition of powders from the Ni-Al system is characterised. Atomisation method of melted Ni-Al alloys was applied for the preparation of the Ni₃Al powders. Powders of NiAl and Ni₂Al₃-NiAl phases and these modified by ceramic phases were prepared in the process of self-propagation, high-temperature synthesis. The structure of NiAl-Al₂O₃ and NiAl-Al₂O₃-AlN composite powders was studied by light and scanning microscopy as well as by X-ray diffraction methods. The selected powders are destined for thermal spraying of coatings.

STRUKTURA PROSZKÓW Z UKŁADU NI-AI ORAZ ICH MODYFIKACJA FAZAMI CERAMICZNYMI

Przedstawiono wyniki badań składu fazowego i morfologii wybranych proszków z układu Ni-Al. Mieszaniny proszków wyjściowych niklu i aluminium są substratami do wytwarzania materiałów kompozytowych z fazami międzymetalicznymi z wykorzystaniem procesów metalurgii proszków i odlewniczych (tab. 1). Proszki faz międzymetalicznych z układu NiAl wytwarzano metodą rozpylania lub samorozwijającej się syntezy wysokotemperaturowej SHS. Dyfraktogramy syntezowanych proszków, analizowane metodą Toraya, przedstawione na rysunkach 1-4 wykazują pełną zgodność z kartami ASTM. Charakterystyczną morfologię proszków NiAl 70-30 i NiAl 95-5, ujawnioną na mikroskopie skaningowym, przedstawiono na rysunkach 6 i 7. Skład chemiczny wybranych materiałów NiAl określony metodą EDX podano w tabeli 2. Dyfraktogram kompozytowego proszku zawierającego fazy: NiAl, tlenków aluminium i żelazo oraz azotek aluminium charakteryzuje proszek otrzymany w procesie SHS (rys. 5). Złożony skład fazowy proszku wskazuje na potrzebę dopasowania parametrów technologicznych procesu wytwarzania kompozytowych proszków o zadanym składzie fazowym. Wytwarzane proszki są przeznaczone dla procesu natryskiwania cieplnego powłok.

INTRODUCTION

Intermetallic NiAl alloys have potential high temperature structural materials because of their attractive properties such as low density, high melting temperature, high thermal conductivity, very good oxidation and hot corrosion resistance and good mechanical properties [1, 2]. Metallurgy of powders completes the founding methods of intermetallic phases production of nickel and aluminium. It constitutes a technological alternative for a production of materials of high dispersion of reinforcing phases. Examples of results for Ni-Al powders and Ni-Al composite materials produced by selected reaction synthesis process is presented in Table 1.

AIM AND SCOPE OF INVESTIGATIONS

The main aim of present studies was the verification of the technology procedures through the chemical and phase composition analysis of the powders of the phases from Ni-Al system and composite powders of these phaFor the realisation of the assumed goal of studies the following scope of investigations was chosen:

- powder preparation of intermetallic phases from the Ni-Al system as well as a preparation of composite powders,
- analysis of chemical and phase compositions of the synthesised powders,
- determination of the powders morphology.

MATERIAL AND EXPERIMENTAL PROCEDURE

Powders from Ni-Al system of definite chemical composition were prepared. The method of alloys atomisation and self-developing, high-temperature synthesis were applied for the preparation of these powders. The

ses modified by ceramic phases. The preparation of Xray standards were necessary for a quick phase analysis of synthesized, modified or composite powders with desired intermetallic phases.

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No.	Chemical composition Ni-at% Al	Process	Conditions	Phase composition	Ref.
1 2 3 4	25Al 25Al 25Al 25Al	SRS	Flaky Ni powder + Al powder \checkmark 0,9 kms ⁻¹ impact \checkmark 1,37 kms ⁻¹ impact \checkmark 1,60 kms ⁻¹ impact GD - 54%	Ni ₃ Al, Ni, NiAl, NiAl, Ni ₂ Al ₃ , Ni ₃ Al NiAl Ni, Ni ₃ Al, Al rich NiAl	[3]
5 6	25AI 25AI	SHS	GD - 72% GD - 80%	N ₁₃ Al, Al rich NiAl Ni ₃ Al	
7	25A1	RI	<i>T</i> - 1073 К, <i>P</i> - 41 МРа	Ni ₃ Al	[5]
8 9	molten Al molten Al	РС	55% D Ni perform 50 MPa 66% D Ni perform <i>T</i> - 8073 K	NiAl,Ni3Al Ni3Al	[6]
10 11 12	40Al 50Al 50Al + 15÷30 TiB ₂	HPRS HPRS	<i>T</i> - 973 K, <i>P</i> - 1 GPa <i>T</i> - 1173 K, <i>P</i> - 3 GPa <i>T</i> - 1173 K, <i>P</i> - 3 GPa	NiAl, Ni ₃ Al, Ni ₂ Al ₃ , NiAl-Ni ₃ Al, Ni ₂ Al ₃ TiB ₂ + Ni ₃ Al + NiAl	[7]
13		RHP	<i>T</i> - 1630 K, <i>P</i> - 20 GPa	TiB ₂ + NiAl+ Ni ₃ Al Ni ₂ B, Al ₂ B, NiT ₂ ,	
14	49Al+15 NiAl+15TiB ₂	RS	T - 973, vacuum	NiAl, TiB ₂	[8]
SRS SHS RI PC HPRS RHP RS	shock-inducted reaction synthesis self-propagating high temperature synthesis reaction infiltration pressure casting high-pressure reaction sintering reaction hot pressing reaction synthesis				

 TABLE 1. Characteristics of intermetallic NiAl phases formed by reactive processes

 TABELA 1. Charakterystyka faz NiAl otrzymanych za pomocą reaktywnych metod

studies on Ni₅Al, Ni₃Al, NiAl, Ni₃Al₂-NiAl, NiAl--Al₂O₃, NiAl-Al₂O₃-AlN were performed. The microstructure of etched powder samples was determined by Reichert MF-2 light microscope whereas powders morphology on Hitachi 4200 scanning microscope. The phase composition of powders was analysed from X-ray diffraction patterns taken on Philips diffractometer equipped with copper tube and graphite monochromator on a diffracted beam. The chemical composition of powders was determined by means of the EDX method using the voyager system and the equipment of a scanning microscope.

RESULTS

Toraya procedure [9] of whole powder pattern fitting (WPPF) was applied for testing of the quality of the experimental patterns. Good agreement between experimental and calculated patterns was obtained (Figs 1-4). Moreover Toraya procedure enabled the finding of the second NiAl phase beside Ni₂AL₃ one (Fig. 4).

The complex powder prepared by SHS method is of complex composition and contain NiAl, NiAL₃, Al₂O₃, Ni₂Al₃, AlN, FeO phases (Fig. 5). The presents of FeO

phase is the results of the high energy mill preparation conditions.

The chemical composition estimated by EDX method and morphology of powders is presented is given in Table 2 and in Figures 6 and 7 respectively.

TABLE 2. The chemical composition of NiAl powdersTABELA 2. Skład chemiczny proszku kompozytowego NiAl

	Al (wt. %)	Ni (wt. %)			
NiAl 70/30*	27.52	69.74			
NiAl 95/5	4.64	95.36			
* Oxygen - 2.74					

CONCLUSION

Powders of phases from the Ni-Al system have a different morphology depending on their preparation method. A verification of a phase composition of powders from X-ray diffraction patterns indicates that the technological parameters of the powder preparation were correctly chosen. Preparation of composite powders of Ni-Al phases with a definite volume fraction of ceramic phases is connected with obeying of a proper procedure and technological parameters. The selected powders of definite chemical and phase compo-



sitions are destined for spraying of bond coatings or of concentra coatings with intermetallic phases of nickel and higher

concentration of aluminium.



Fig. 1. X-ray diffraction pattern of NiAl 95-5 powder Rys. 1. Rentgenogram proszku NiAl 95-5



Fig. 2. X-ray diffraction pattern of Ni₃Al powders Rys. 2. Rentgenogram proszku Ni₃Al



Fig. 3. X-ray diffraction pattern of NiAl powders Rys. 3. Rentgenogram proszku NiAl



Fig. 5. X-ray diffraction pattern of composite powder Rys. 5. Rentgenogram proszku kompozytowego



Fig. 6. Morphology of NiAl 70/30 powder Rys. 6. Morfologia proszku NiAl 70/30



Fig. 7. Morphology of NiAl 95/5 powder Rys. 7. Morfologia proszku NiAl 95/5

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