A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 53 2008 Issue 1

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POSSIBILITIES OF APPLICATION OF PHASE IDENTIFICATION BY ELECTRON BACKSCATTER DIFFRACTION FOR DETERMINATION OF PHASE COMPOSITION OF MULTILAYER SCALES

MOŻLIWOŚCI ZASTOSOWANIE IDENTYFIKACJI FAZ METODĄ DYFRAKCJI ELEKTRONÓW WSTECZNIE ROZPROSZONYCH DO OKREŚLANIA SKŁADU FAZOWEGO ZGORZELIN WIELOWARSTWOWYCH

In this paper the application of electron backscatter diffraction for the phase identification of complex layers is presented. The obtained results were compared with results of X-ray diffraction and EDS X-ray microanalysis obtained for the same layer. The conducted research shows that X-ray diffraction not always gives full description of the examined layers (especially when layers are relatively thick). Also X-ray microanalysis does not assure full description of the examined layers, since in most cases the phases are disequilibrium. Identification of particular phases can be obtained by electron backscatter diffraction. It appears that presently combination of the three research techniques provides the most complete description during examination of layers.

Keywords: EBSD, gamma TiAl, oxidation, scale

W pracy przedstawiono zastosowanie dyfrakcji elektronów wstecznie rozproszonych do identyfikacji fazowej warstw o złożonej budowie. Otrzymane wyniki porównano z wynikami dyfrakcji rentgenowskiej oraz mikroanalizy rentgenowskiej EDS uzyskanymi dla tej samej warstwy. Z przeprowadzonych badań wynika, że dyfrakcja rentgenowska nie zawsze daje pełny opis badanych warstw (szczególnie gdy warstwy są stosunkowo grube). Także mikroanaliza rentgenowska nie daje pełnego opisu badanych warstw m.in. dlatego, że w wielu przypadkach fazy wchodzące w ich skład są fazami nierównowagowymi. Identyfikację poszczególnych składników fazowych można uzyskać za pomocą dyfrakcji elektronów wstecznie rozproszonych. Wydaje się, że w chwili obecnej połączenie tych trzech technik badawczych daje najpełniejszy opis podczas badania warstw.

1. Introduction

Current technique provides vast application for the elements with surfaces covered with a variety of layers. Properties of these layers mainly depend on their phase composition. In order to predict utilisation properties of the designed layers it is necessary to know phases being a part of them.

A number of research techniques is used to identify phases [1]. These could be e.g. techniques basing on chemical composition (EDS, WDS, AES, XPS, ISS, SIMS). They let determine quantitative chemical composition or participation of each element being a part of examined phases, what allows finding out their chemical formula.

This kind of examinations does not give full description. There is lack of such important information as crystallographic structure. It is particularly important in the following situations:

- when a phase may occur in many crystallographic systems,
- when a phase is non-equilibrium,
- when the accuracy of chemical analysis is too low.

In modern metallography, in order to determine a crystallographic structure, electron diffraction in transmission electron microscope on extraction replicas or thin foils as well as X-ray microdiffraction (μXRD) are applied. The former requires exceptionally difficult and time-consuming procedure of thin foil or replica preparation, what really reduces its field of application. The latter has limited resolution: usually 10-50 μm. Both traditional research techniques for examination of structure of microscopic area lead to difficulties as far as the identification of the diffraction patterns is concerned. Rel-

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atively new method is electron backscatter diffraction (EBSD) [2-4].

2. Aim of the work

The aim of this work is to present the possibilities provided by the phase identification with electron backscatter diffraction (EBSD) for determining phases present in complex surface layers.

3. Material and method

The researches were conducted on γ-TiAl alloy oxidised in 1173 K in air atmosphere during 120 hours.

An X-ray diffraction on the X-ray diffractometer SIEMENS D300 was performed on the samples obtained this way. Next, metallographic microsections were created transversally with respect to layers and analysed under HITACHI S- 3000N scanning electron microscope. The analysis consisted of microscopic observations of the obtained layer, determination of its chemical composition by energy dispersive spectroscopy (EDS) and finally the identification of phases appearing in the layer by electron backscatter diffraction (EBSD).

4. Discussion on the results

Fig. 1. presents diffraction X-Ray spectrum, both with identification of the reflexes, obtained from the surface of the examined sample. Table presents the results of chemical composition from areas marked from 1 to 5 in the Fig. 2. Figs. 3-5 present the results of electron backscatter diffraction from these areas.

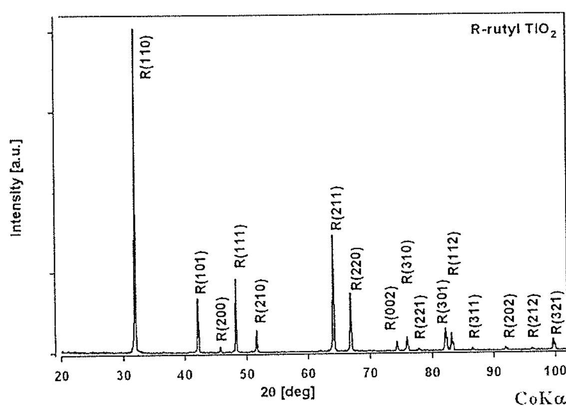

Fig. 1. Diffraction pattern from the surface of the scale formed on γ-TiAl base after oxidation in static air for 120 hours in 1173 K

In the diffraction spectrum shown above (Fig. 1) only lines coming from one of three forms of TiO_2 titanium oxide (rutile) are present [5]. Lack of lines coming from other phases could suggest that the obtained layer is monophase. However, both microscopic observations (Fig. 2.) and X-Ray microanalysis (Fig. 2, Table) show more complex structure of the oxide layer. On the scale microsection it is possible to distinguish five oxide interlayers (marked with 1 to 5).

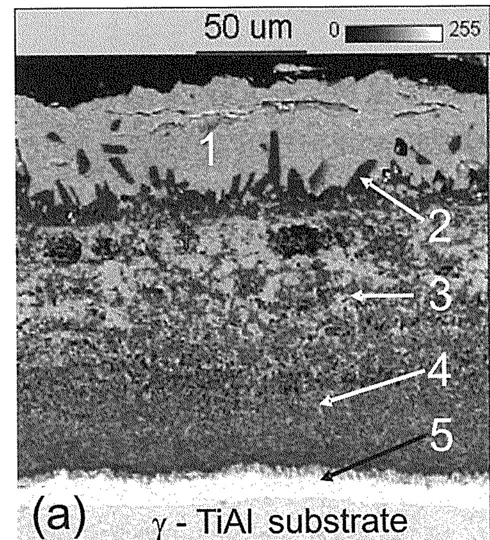

Fig. 2. Microscopic image of oxidised layers with marked areas of microanalysis and diffraction

TABLE

Presents the results of chemical composition from areas marked from 1 to 5 in the Fig. 2

Analysed area	Contents	Element		
		Al	Ti	O
1	%at.	0.2	47.0	52.8
	wt.	0.1	72.6	27.3
2	%at.	41.7	0.3	58.0
	wt.	54.4	0.7	44.9
3	%at.	33.1	62.7	4.2
	wt.	22.6	75.8	1.6
4	%at.	27.9	14.1	58.0
	wt.	31.9	28.8	39.3
5	%at.	28.8	71.2	0
	wt.	18.6	81.4	0

The elements distribution on the lateral microsection of the scale (Fig. 2.) as well as additionally performed quantitative chemical microanalysis of the marked areas (Table) show that the outer layer has about 60 μm (marked with 1 in Fig. 2.) and is enriched with titanium and oxygen. Phase identification from the same area was performed by electron backscatter diffraction (EBSD) and is presented in Fig. 3. Comparing the obtained image with the images indexed in The International Centre for Diffraction Data (ICDD database) that may contain Ti and O, it may be stated that crystallographic structure of

the examined phase corresponds to the crystallographic structure of polymorphic form of titanium oxide TiO_2 known as rutile. This confirms the result of the examination obtained by X-Ray microanalysis. Second sub-layer (marked with 2 in Fig. 2) is enriched with aluminium and oxygen. After analysis, carried on the same as previous, crystallographic structure of the examined phase corresponds to the crystallographic structure of α-Al_2O_3 aluminium oxide crystal (of a rhombohedral structure) basing on the card no. 83-2081 in ICDD database. Next sub-layers (marked with 3 and 4 in Fig. 2) consist of mix of those two phases TiO_2 and α-Al_2O_3 with different degree of dispersion. EBSD diffraction from sub-layer marked 5 in Fig. 2 shown that below all oxide phases formed Ti_3Al probably due to outward diffusion of aluminium from substrate to the scale during oxidation in 1173K.

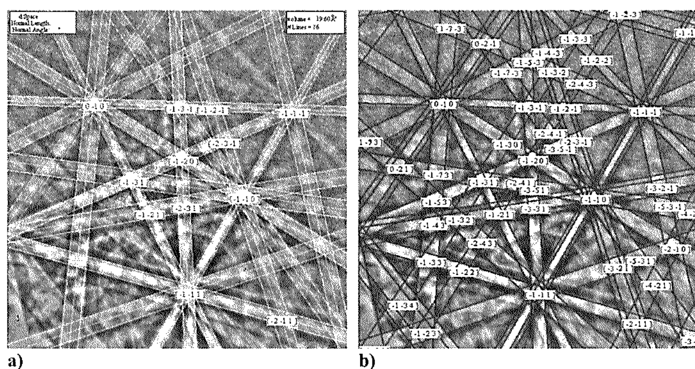

Fig. 3. EBSD diffraction pattern of the area marked with 1 in Fig. 2: a) found lines and surfaces; b) simulation for TiO_2 titanium oxide crystal (rutile of a tetragonal structure) basing on ICDD data in the card no. 77-0441

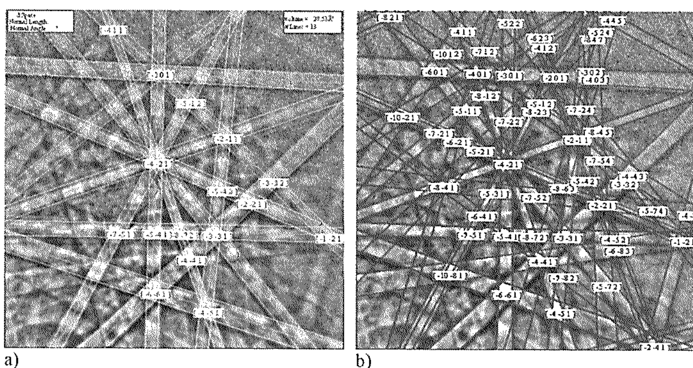

Fig. 4. EBSD diffraction patterns obtained from the area marked with 2 in Fig. 2: a) found lines and surfaces; b) simulation for α-Al_2O_3 aluminium oxide crystal (of a rhombohedral structure) basing on the card no. 83-2081 in ICDD database

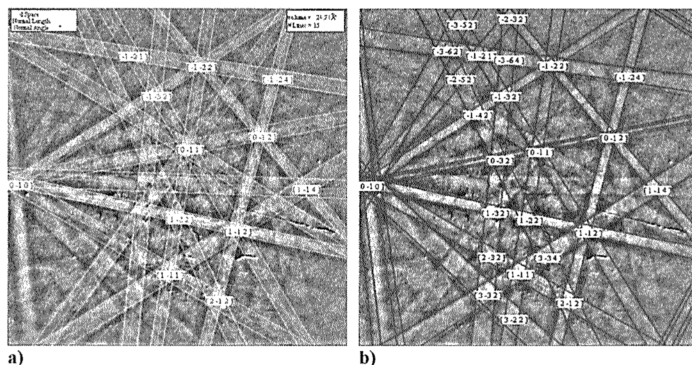

Fig. 5. EBSD diffraction patterns obtained from the area marked with 5 in Fig. 2: a) found lines and surfaces; b) simulation for Ti_3Al crystal (of a hexagonal structure) according to data from the ICDD card no. 09-0098

Acknowledgements

A financial support of the Polish State Committee for Scientific Research in the frame of the project No. PBZ-KBN-100/7/T08/2004 is gratefully acknowledged.

REFERENCES

[1] S. Kikuchi, Diffraction of cathode rays by mica. Jap. J. Phys. **5**, 83 (1928).

[2] A. J. Schwartz, M. Kumar, B. L. Adams, Electron Backscatter Diffraction in Materials Science. Kluwer Academic/plenum Publishers233 Spring Street, New York, N.Y.10013.

[3] J. F. Michael, R. P. Goehner, Crystallographic phase identification in the scanning electron microscope backscattered electron Kikuchi patterns imaged with a CCD based detector. MSA bulletin 23-168 (1993).

[4] L. Klimek, P. Kula, K. Jakubowski, Metodyczne aspekty identyfikacji faz metodą dyfrakcji elektronów wstecznie rozproszonych Polska Metalurgia w latach 1998-2002 Komitet Metalurgii Polskiej Akademii Nauk Wydawnictwo Naukowe "AKAPIT" **2**, 418-424, Kraków 2002.

[5] L. Kaczmarek, Oxidation resistance intermetallic γ-TiAl coatings to protect titanium alloys against oxidation in high temperature, Ph.D. Thesis, Technical University of Lodz, 2006.

Received: 3 December 2007.