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
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L. KLIMEK*

PHASES IDENTIFICATION BY MEANS OF EBSD METHOD – NEW POSSIBILITIES OF MATERIALS RESEARCHES

IDENTYFIKACJI FAZ METODĄ DYFRAKCJI ELEKTRONÓW WSTECZNIE ROZPROSZONYCH – NOWE MOŻLIWOŚCI W BADANIACH MATERIAŁÓW

The examples of EBSD analysis of selected phases in steel are presented in this paper. The carbon steel, Co-Cr-Mo alloy and steel after termochemical treatment were examined. The obtained results confirm that the EBSD method can be a valuable tool used for recessive analysis of phases which occure in alloys with giving particular attention to theirs polymorphism and non stechiometry. It allows examining samples both as metallographic cross-section and fracturing as well as samples after surface treatment.

Keywords: electron backscatter diffraction, scanning electron microscopy, phase identification, X-ray microanalysis

W artykule przedstawiono przykładowe możliwości analizy składu fazowego materiałów. Badania wykonano na stopie Co-Cr-Mo, przełomie stali węglowej oraz stali po obróbce cieplnochemicznej. Z przytoczonych badań wynika, że dyfrakcja elektronów wstecznie rozproszonych może być cennym narzędziem służącym do precyzyjnej identyfikacji faz występujących w stopach z uwzględnieniem ich niestechiometryczności i polimorfizmu. Istnieje możliwość badań zarówno na zgładach metalograficznych , powierzchniach po obróbkach cieplnochemicznych oraz przełomach.

1. Introduction

Identification of phases in examined and utilised materials is significant in materials engineering. A large variety of research methods is available for these purposes [1]. They can be divided into two groups – based on determination of chemical composition and diffraction techniques. The former (e.g. X-ray microanalysis, Auger electron spectroscopy, mass spectrometry, ion spectrometry, etc.) lets to find out chemical composition quantitatively, by establishing the percentage of particular elements forming the examined phase. This allows discovering their chemical formula. However these examinations do not give the full description. When chemical composition inspection is inaccurate or the assessed phase is not in equilibrium, it cannot be often uniquely identified; similarly, when a phase can occur in a few crystallographic systems. These ambiguities result from lack of information about crystallographic structure.

Information about crystallographic structure can be obtained using electron diffraction in transmission electron microscope (on extraction replicas or thin foils) as well as using X-ray diffraction (μXRD). 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. In past years, several leading companies manufacturing research equipment elaborated a conception of PHASEID (*phase identification*) system, based on the phenomenon of electron backscatter diffraction (EBSD). This phenomenon is known from 1920s as Kikuchi diffraction lines. However, its first practical applications are dated back to 1970s – with construction of cameras with proper sensitivity [2] – primarily for determination of crystallographic orientation, then for phase identification [3, 4]. Phase identification set is composed of scanning electron microscope, X-ray microanalysis system (EDS or WDS), camera for collecting diffraction patterns and computer system for processing data (signals coming from both detectors), basing on the database with chemical and crystallographic classification of nearly 90000 phases. The criteria for phases recognition are: chemical composition, type of crystallographic lattice and volume of reduced cell [3, 5, 6].

* INSTITUTE OF MATERIALS ENGINEERING, LODZ UNIVERSITY OF TECHNOLOGY, 90-924 ŁÓDŹ, 1 STEFANOWSKIEGO STR., POLAND

2. Aim of the work

The aim of this paper is to show broad possibilities of application of identification of phases in metallographic examinations, offered by electron backscatter diffraction.

3. The idea of forming a diffraction image

Diffraction of backscatter electrons happens due to Bragg's Law, similarly as diffraction of passing electrons or X-rays.

The Kikuchi pattern is formed due to the fact that bent electron beam may interfere with "upper" or "lower" crystallographic plane (fig. 1). This provides two diffraction conditions and two diffraction lines appear in the figure. Since diffraction conditions in the crystal, on which a beam of primary electrons falls, may be satisfied for many crystallographic planes, many diffraction lines are obtained in the pattern.

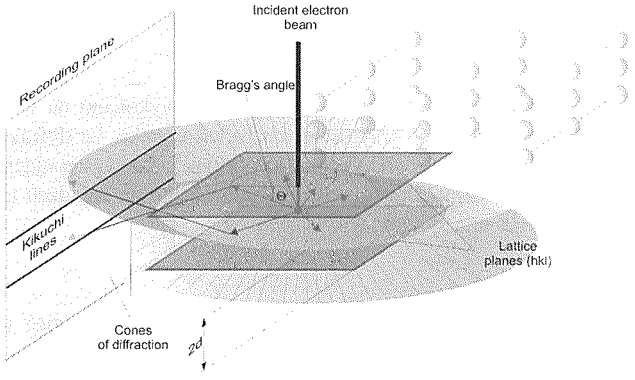


Fig. 1. Electron backscatter diffraction on crystallographic plane

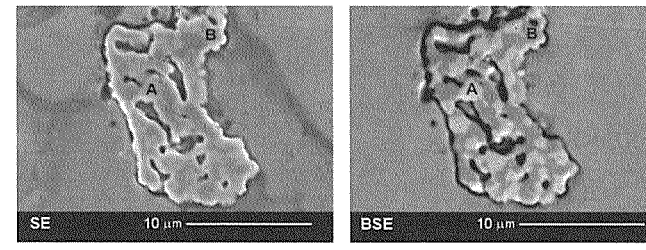
4. Research methods and results

In the present paper, examples of precipitations identification in different materials are presented. The following samples were used for the research purposes:

a) Co-Cr-Mo alloy sample – microsection,
b) carbon steel sample – fracture,
c) sample after thermochemical treatment SULFINUS® [7] – external surface

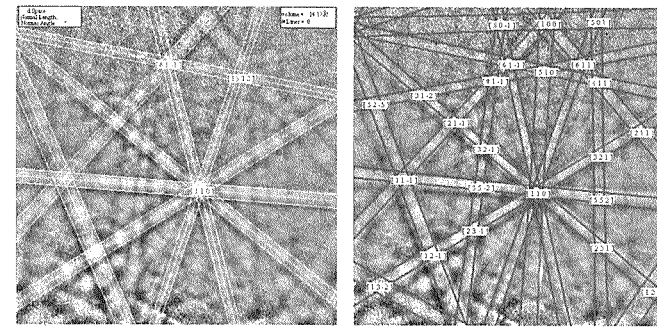
Each of the samples was examined under HITACHI S-3000N scanning electron microscope. Next, in order to determine chemical composition, energy dispersive spectroscopy (EDS) was performed on selected phases. The following stage of the research was to create a diffraction pattern of backscatter electrons and consequently phase identification.

Co-Cr-Mo alloy sample



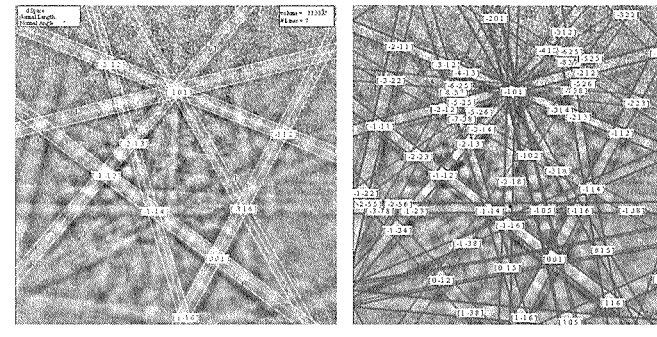
a) b)

Fig. 2. Co-Cr-Mo alloy sample: a) carbide – figure in secondary electrons, b) carbide – figure in backscatter electrons



a) b)

Fig. 3. Co-Cr-Mo alloy sample – diffraction in the place marked A in Fig. 2: a) solution of obtained diffraction pattern, b) simulation for CrC carbide described in ICDD database



a) b)

Fig. 4. Co-Cr-Mo alloy sample – diffraction in the place marked B in Fig. 2: a) solution of obtained diffraction pattern, b) simulation for $(Cr_{0.77}Co_{0.15}Mo_{0.08})_{23}C_6$ carbide described in ICDD database

Carbon steel sample

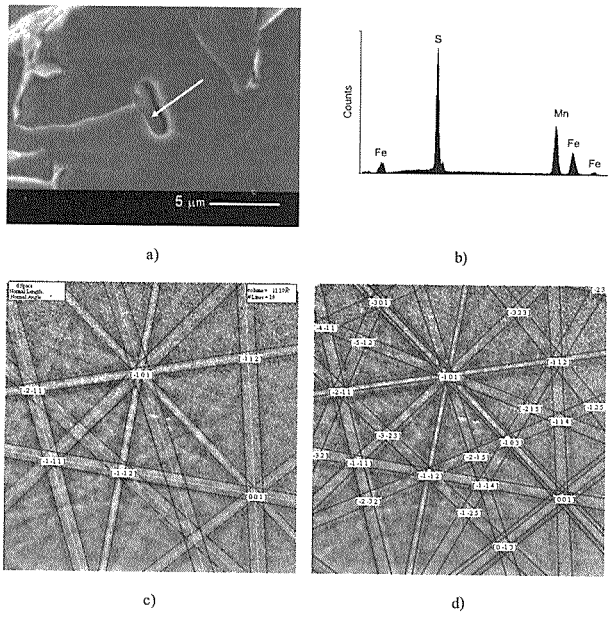


Fig. 5. Carbon steel sample: a) fracture – precipitation being identified marked with arrow, b) EDS spectrogram from marked precipitation, c) solution of obtained diffraction pattern, d) simulation for MnS sulphide described in ICDD database

Sample after thermochemical treatment SULFINUS®

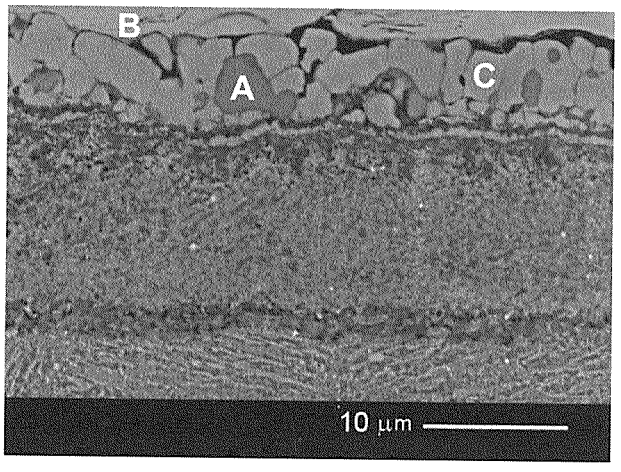


Fig. 6. Steel after thermochemical treatment SULFINUS® – nitrosulphided layer with marked places of performed diffraction in secondary electrons – SE

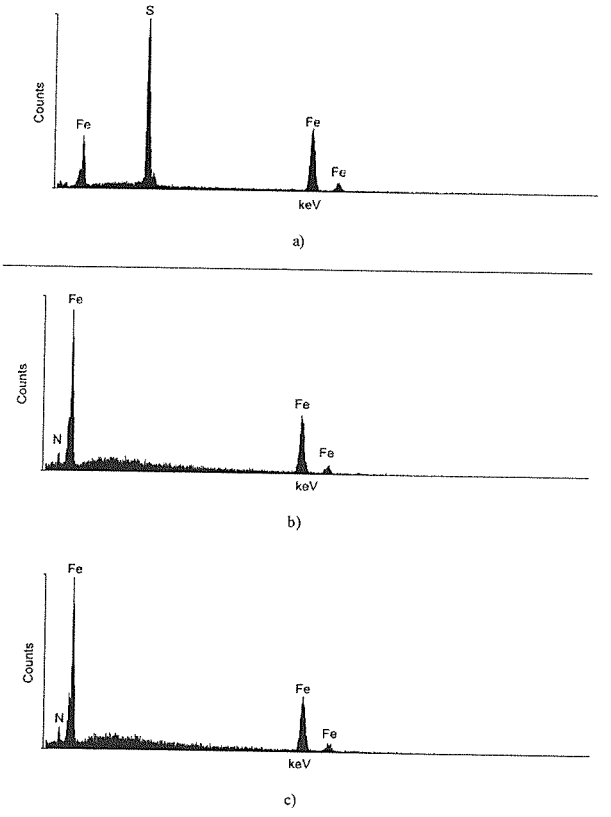


Fig. 7. EDS spectrograms of phases marked in Fig. 6: a) place A, b) place B, c) place C

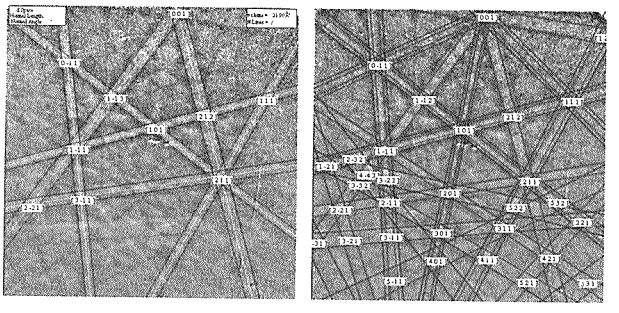


Fig. 8. Place marked A in Fig. 6: a) solution of obtained diffraction pattern, b) simulation for FeS sulphide described in ICDD database

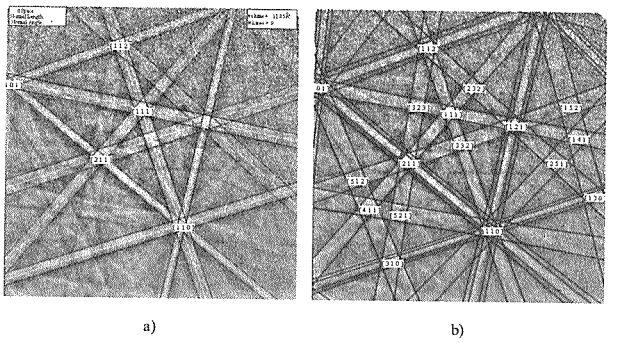
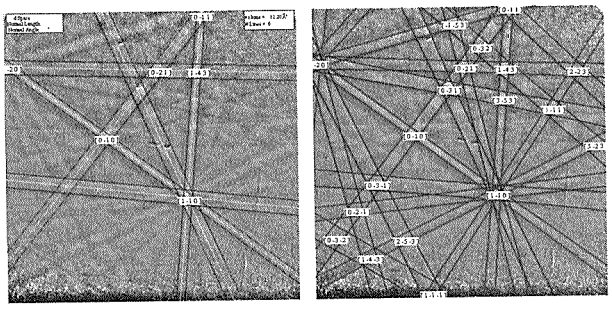


Fig. 9. Place marked B in Fig. 6: a) solution of obtained diffraction pattern, b) simulation for Fe4N nitride described in ICDD database



a) b)

Fig. 10. Place marked C in Fig. 6: a) solution of obtained diffraction pattern, b) simulation for Fe3N nitride described in ICDD database

TABLE

Results of analysis of chemical composition of phases marked in Fig. 2 obtained using X-ray microanalysis

Place of analysis	Elements content [wt. %]			
	Cr	Mo	C	Co
Phase A	50,5	2,1	38,7	8,7
Phase B	37.7	14,8	18,9	28,6

5. Discussion of results

Chromium-nickel steel

The conducted analysis of map of elements distribution shows that in the examined area two kinds of carbides are present. One of them is richer in chromium. Results of electron backscatter diffraction allowed identification of these carbides. The one poorer in chromium is the carbide of $(Cr_{0.77}Co_{0.15}Mo_{0.08})_{23}C_6$ type, described in ICDD database in the card no. 37-1229, whereas the carbide richer in chromium has a structure of CrC carbide, described in ICDD database in the card no. 47-1424. Observed differences between the chemical composition obtained by EDS analysis and stechiometric composition may result from small size of examined carbides and because of that larger area of analysis than the inspected carbide.

Carbon steel

Analysing the chemical composition of the examined precipitation it is possible to state that it is complex iron and manganese sulphide. Crystallographic database contains about a dozen of cards describing sulphides of these elements, differing in contents of Fe and Mn, as well as in crystallographic net structure. After having analysed the obtained diffraction pattern and after having compared it with patterns of all the sulphides in database, it was decided that the most accurate with the obtained diffraction pattern is the one of MnS sulphide, described in card no. 6-0518, having regular structure and Fm3m group. It may be derived that the investigated precipitation is a manganese sulphide having the same structure as MnS, where part of the manganese atoms was substituted with atoms of iron. Inconsistency of the lattice as well as the result of chemical analysis let prove that this is a non stechiometry sulphide.

Steel after thermochemical treatment SULFINUS®

Images in scanning electron microscope demonstrated that external layer (so called compounds zone) is composed of three kinds of phases, what is clearly visible in microscopic pictures. The identifications carried out showed that these are: iron nitride – brighter phase and iron sulphide – darker phase. Chemical microanalysis revealed that these are iron sulphide and two phases of iron nitride. However quantitative results do not let to uniquely identify the types of the nitrides. Conducted diffraction examinations showed that in external layer there exists Fe_4N nitride of a cubic structure and card no. 77-2006 Below it, there appears FeN nitride of a hexagonal structure and card no. 83-0879. Whereas the examined sulphide is $Fe_{0,95}S_{1,05}$ of a cubic structure and card no 75-0600. It would be expected that on the surface the layer is richer in nitrogen and going deeper the layers have smaller nitrogen contents. Similarly, after SULFINUS® treatment, iron sulphides would be supposed to appear on the surface. Explanation of this phenomenon could be found in researches of A. Rzepkowski [8]. It should be stressed that using electron diffraction it would not be possible to determine the structure of this layer, since only existing phases would be obtained, without information of their mutual distribution.

6. Conclusions

1. Phase identification by electron backscatter diffraction may be a valuable tool for a precise characterisation of phases in alloys including their non stechiometry and polymorphism.
2. It is possible to conduct research not only on metallographic microsections, but also on the outer surfaces and fractures, what significantly extends the area of application of this technique (as it is well-known, chemical microanalysis from the fracture surfaces is not reliable).
3. Phase identification by electron backscatter diffraction does not require complicated procedure of sample preparation, what makes this method even more attractive.
4. Electron backscatter diffraction allows not only identification of phases, but also their mutual distribution, which is significant for surface treatment.

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