

STRUKTURA 2007

Penzion za vodou, Dvůr Králové, 18. 6. - 21. 6, 2007

Lectures - Monday, June 18

L1 - L4

STRUCTURAL DATABASES

see pages 89 - 101

L5

TEXTURE ANALYSIS OF ROLLED STEEL SHEETS BY X-RAY AND ELECTRON DIFFRACTION (EBSD)

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The rolled IF and TRIP steel sheets were evaluated by X-ray and electron diffraction methods. Textures were measured by X-ray diffraction using texture goniometer with Mo X-ray tube, employing (110), (200) (112) and (103) crystallography planes. EBSD camera was used for texture measurements by electron diffraction. ODF – orientation distribution functions were calculated from texture-measured data by use of shf – spherical harmonic function, WIMV and ADC methods. Both, X-ray and EBSD data obtained very good agreements on texture characterizations of non-grain oriented (NGO) steels in relation to their magnetic properties. The EBSD methods allow obtaining more structural information: IPF maps, grain shape, size, crystal orientation of selected grains and others.

The partially annealed IF steel sheet sample EBSD analysis shows directly the texture of individual grains, X-ray diffraction method gives the material complex texture. EBSD method allows categorizing grains according to proper criteria. The criteria "Grain average disorientation" was used for grains categorizing into two groups; re-crystallized and deformed grains.

The rolled IF steel sample texture was formed by alpha and gamma fibers. The final texture was the superposition

of the deformation and recrystallization texture. The deformation texture was formed by uncompleted alpha fiber with (001) 011, (113) 011, (223) 011 texture elements. The recrystallization texture was formed by gamma fiber with (111) 011 to (111) 112 texture components.

The EBSD method allows analyzing samples with complex phase composition. Comparing the X-ray diffraction method, which is limited by material texture, the retained austenite content in rolled steel sheets can be directly and exactly determined by EBSD method. At the same time the texture is obtained for particular phases too, in this case the texture of ferrite and residual austenite. The EBSD method provides data about grain size and shape, boundaries characteristics for all present phases.

The rolled TRIP steel sample ferritic phase was formed by strong gamma fiber (111) 011 to (111) 112 and by small amount of (100) 011 and (110) 001 texture components. The retained austenite content was estimated for 5,2%. The residual austenite texture was formed by Goss - (110) 001, Brass - (110) 112 texture components and a small amount of Copper - (112) 111 component.



Lectures - Tuesday, June 19

L6

PHASE TRANSFORMATIONS, PHASE DIAGRAMS, DIFFERENTIAL SCANNING CALORIMETRY

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Thermodynamical basis of phase transformations. Anomalies of thermophysical quantities. Classification of phase transformations. Equilibrium in heterogeneous systems. Gibbs phase rule. Common tangent construction. Lever rule. Tangential plane construction. Binary and ternary phase diagrams. Basic types of binary phase diagrams.

Thermal analysis – differential thermal analysis (DTA), differential scanning calorimetry (DSC).

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L7

DATABASES OF CRYSTALS WITH STRUCTURAL PHASE TRANSITIONS

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Crystals with structural phase transitions (SPT's) exhibit unusual physical properties that are utilized in many technical applications (e.g. anisotropic ceramics, memories, sensors, transducers) and in other fields (e.g. earth sciences). Though basic structural data on SPT's can be found in existing structural databases (ICSD, CSD) special features and advanced theoretical background of SPT's call for a specialized database.

The most comprehensive listing of non-metallic crystals with SPT's is available in *Tomaszewski's database of structural phase transitions* [1,2]. This is a printed table in which each row (record) is related to one chemical formula of a crystal and columns (fields) give transition temperatures as well as the main available structural data of corresponding phases in between two neighbouring transitions. In updated version [2] about 4 300 crystals are recorded with more than 6 300 phase transitions and about 10 000 crystal phases observed at normal pressure. This presentation provides useful information about the appearance of SPT's in crystals but does not allow an efficient search.

This drawback can be partially removed if the records are arranged according to the symmetry changes at the SPT. Such tables have been created from the updated Tomaszewski's database [2]. In the first step only crystals which exhibit just a single SPT associated with a dissymmetrization (reduction of the space group symmetry) have been included. To increase the reliability of data only those SPT's have been taken into account for which structures of both phases are commensurate and are recorded in

the Inorganic Crystal Structure Database (ICSD). These restrictions drastically reduce the number of considered crystals (only 20% of crystal phases that appear in [2] have a structure solved and recorded in ICSD!). A table formed from these selected data constitutes a simple *Ferrous Phase Transition Database* which can provide answers to useful simple queries and from which some statistical conclusions can already be deduced [3].

Present effort concentrates on creation of *computerized relational databases of SPT's*. This approach makes use of recent advances in database techniques and can utilize the extensive theoretical knowledge on SPT's (see, e.g. [4]).

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2. P. Tomaszewski, *Golden Book of Phase Transitions*, Phase transitions database PTDB, manuscript, 2002.
3. V. Janovec, P. Tomaszewski, L. Richterová, Z. Kluiber, Inverse database of phase transitions in crystals with a single phase transition. *Ferroelectrics*, **301**, (2004), 169.
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Acknowledgement

This work has been partially supported by the grant 202/07/1289 of the Grant Agency of the Czech Republic..

L8

MEASUREMENT WITH VT-RH CHAMBER

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Powder diffraction patterns are the most fundamental, yet crucially important; application of XRPD is in identification or “fingerprinting” of crystalline phases in pharmaceutical industry [1]. Function of temperature and/or relative humidity can provide a direct means of characterizing the stability of a pharmaceutical material at defined temperature and relative humidity and the occurrence of hydration/dehydration processes [2]. Such a non-ambient diffraction experiments can be performed at any stage of the drug development process (API production, stability testing, formulation, storage...) to avoid further complications.

The object of presented study is to demonstrate the possible utilization of variable temperature and relative humidity XRPD to investigate hydration/dehydration process of the pharmaceutical material.

The behaviour of pharmaceutical hydrates has become the object of increasing interest over last two decades, primarily due to the potential impact of hydrates on development process and dosage form performance. Hydration of

the material also plays a role in bioavailability, influences dissolution, hardness of tablets or even processability. Interconversion between polymorphs and hydrates may occur as a function of temperature or of relative humidity or of both. During and after manufacturing even air moisture from the environment may change the hydration state of API in dosage form.

X-ray diffraction system with variable temperature and relative humidity was used. Instrumentation as well as obtained results will be discussed. The potential for interconversion during development was studied. Just different powder diffraction patterns can be used as an evidenced of change in the structure. Anyway, DSC and IR were used as complementary techniques to XRPD.

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L9

THERMAL EXPANSION OF TIS: ASSESSMENT OF MISCIBILITY WITH TROILITE (FES)

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From meteorites - aubrites, in which otherwise lithophile elements behave as siderophiles due to strongly reducing conditions, titanium-rich iron monosulfides were reported in literature. For example, in the Bustee aubrite, the titanium-bearing troilite, associated with osbornite (TiN), heideite (FeTi₂S₄) and oldhamite (CaS), was found to contain 17.2 to 25.2 wt % Ti. In the Bishopville aubrite, the content of titanium in troilite is reported to be up to 5.7 wt % [1]. The crystal structures of troilite and TiS are not identical under ambient conditions. While troilite is hexagonal with space group is *P2c* and unit-cell dimensions $a \sim 5.97 \text{ \AA}$, $c \sim 11.76 \text{ \AA}$, $V \sim 362 \text{ \AA}^3$ [2], TiS adopts NiAs-type structure with space group *P6₃/mmc* and the unit cell parameters $a \sim 3.31 \text{ \AA}$, $c \sim 6.34 \text{ \AA}$, $V \sim 60.2 \text{ \AA}^3$ [3]. At elevated temperatures, however, the troilite structure transforms to NiAs-type structure as well [4]. Consequently, a relatively significant mutual solubility can be expected between FeS and TiS at temperatures above this phase transition. To evaluate the crystallographic limits for the TiS miscibility

in FeS we carried out a series of high-temperature unit-cell refinements for the former phase.

To collect the diffraction data we used an X'Pert PRO MPD Alpha-1 multi-purpose X-ray diffraction system equipped with an incident beam monochromator, Co tube, and X'Celerator detector. The material was a synthetic TiS prepared under controlled condition in an evacuated silica tube. The sample was heated in an HTK 1200 high-temperature chamber in an alumina sample holder. The holder spun. The NIST silicon internal standard was used for calibration. To prevent oxidation of the sample helium protecting atmosphere was utilized.

In Figure 1 we present results of high-temperature diffraction study of TiS for temperature range from 20 to 400 °C. Within the interval the parameter a and the cell volume increase monotonically whereas cell edge c has the opposite trend. This behavior is identical to that observed for (close-)stoichiometric FeS above the temperature of transition between 2H and 1C polytypes [4]. This observa-



tion substantiates a broad field of mutual solubility along FeS - TiS join at elevated temperatures. On the contrary, these results do not corroborate that phases described from aubrites possess the troilite type 2H structure; most probably the transformation which troilite undergoes at ca 100 °C causes the change in symmetry in Ti-containing minerals.

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2. H.T. Evans *Science*, **167**, (1970), 621.
3. H. Hahn & B. Harde *Zeit. Anorg. Allgem. Chemie* **288**, (1956), 241.
4. E.N. Selivanov, A.D. Vershinin, R.I. Gulyaeva *Inorg. Materials*, **39**, (2003), 1097.

L10

THE STRUCTURE ANALYSIS USING PROGRAM MAUD

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The structure determination from powder diffraction data using the Rietveld approach is in most cases tedious work. However, due to suitable software available long lasting calculations can be significantly shortened. Currently, variety of computer programs for this purpose is available either as freeware or on a commercial base. Nevertheless, the degree these programs help user in extracting the structural data may vary significantly. The shared computer programs based on the Rietveld method can be divided into two general categories from the point of processing basic Rietveld refinable parameters. First category contains number of examples of programs which provide user with a value of refined parameters, for example GSAS, DBWS or FullProf. On the other hand only few programs are available which directly list values of structural parameters determined after each refinement cycle. One of them is a Rietveld method based computer program MAUD.

Both approaches have advantages as well as drawbacks. The first group of programs necessarily needs to employ further processing of extracted parameters. On the other hand it means also opportunity for operator to treat and manipulate the raw parameters in order to extract specific structural information. Second group offers instant information about structural parameters which are considered as refined values. However, these are provided without possibility to interact with basic parameters known from the Rietveld approach. Certainly, number of supporters can be found for both categories of programs. This contribution is not aimed at finding the best group of programs. It would rather concentrate and highlight some of valuable functions and properties the computer program MAUD offers to an operator.

Maud – stands for Materials Analysis Using Diffraction is diffraction/reflectivity analysis program developed on

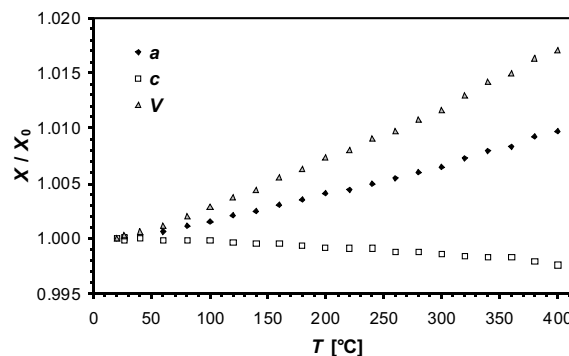


Figure 1. Relative changes of unit-cell dimensions in TiS at elevated temperature.

the basis of Rietveld method by Luca Lutterotti from University of Trento in Italy. This program is available since 1997. Maud is written in Java and can be executable in Windows, Mac OSX, Linux or Unix with pre-installed Java VM 1.4.

Author summarized the main features of the Maud program as follows:

- Easy to use, every action is controlled by a GUI
- Works with X-ray, synchrotron, Neutron, TOF
- Developed for Rietveld analysis, simultaneous multi spectra and different instruments/techniques supported
- Ab-initio structure solution integration, from peak finding, indexing to solving
- Different optimization algorithms available (LS, Evolutionary, Simulated Annealing, Metadynamics)
- Le Bail fitting
- Quantitative phase analysis
- Microstructure analysis (size-strain, anisotropy and distributions included)
- Texture and residual stress analysis using part or full spectra
- MEM algorithm for Electron Density Maps and fitting
- Thin film and multilayer aware; film thickness and absorption models
- Reflectivity fitting by different models, from Parratt (Matrix) to Discrete Born Approximation
- Several data files input formats
- Works and input images from 2D detectors (image plates, CCD)



- CIF compliance for input/output; import structures from databases

Our experience with the structure analysis of bulk steels or steel powders usually leads to application of quantitative phase analysis, microstructure analysis, texture analysis. In case of coated steel sheets, film thickness was determined successfully.

Due to simple user interface and available wizard the quantitative phase analysis can be performed using Maud in an intuitive way via releasing set of refinable parameters in five easy steps. Advanced users have opportunity to control each parameter separately.

For the purpose of microstructure analysis, Maud allows to use isotropic Delf model, anisotropic models with no rules and anisotropic model with Popa rules. Warren models for antiphase boundary and planar defects are also implemented in the code.

User can find also effective tools for compensation the texture of material. For this purpose, models such as March-Dollase, Harmonic functions, E-WIMV, WIMV are available. Experience shows that in most cases harmonic functions tends to be effective only when fiber symmetry as simplest one is engaged. The lower the symmetry the more of unstable parameters usually appears. E-WIMV and WIMV modules, allows the texture to be determined using Beartex from texture measurements and imported afterwards. Bearing in mind the drawback of harmonic func-

tions to import data from Beartex appears to be a safest way of texture compensation.

The Maud program provides user effective tool to characterize layers or multilayers deposited on single or multiphase substrate with possibility of thickness determination. For this purpose, the expected structure, phase constitution, ordering of layers and thickness is designed via available program interface. All mentioned layer properties can be during program execution handled as refinable parameters.

The Maud program is effective tool for structure analysis using diffraction. In general, it offers user friendly interface with the real-time pattern refinement monitor. Wider application of this program in scientific community is probably hindered with fact that there is no user guide available at the moment. Existing tutorials, Maud forum and Download section can be found at <http://www.ing.unitn.it/~maud/index.html>.

L. Lutterotti, S. Matthies and H. -R. Wenk, "MAUD (Material Analysis Using Diffraction): a user friendly Java program for Rietveld Texture Analysis and more", Proceeding of the Twelfth International Conference on Textures of Materials (ICOTOM-12), Vol. 1, 1599, (1999)

Acknowledgement

Financial support of the Slovak Grant Agency VEGA provided to project 1/4107/07 is acknowledged.

Lectures - Wednesday, June 20

L11

See page 103 for more

L12

SCANNING PROBE MICROSCOPY TECHNIQUES WITH ATOMIC RESOLUTION

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Scanning tunneling microscopy (STM), atomic force microscopy (AFM) and related scanning probe microscopy (SPM) techniques developed during the last two decades are widely used for studying material surfaces. Information on surface structure with atomic resolution provided by imaging in real space considerably helped to elucidate a number of open questions and introduced new concepts into studying surface processes and crystal growth. STM technique (limited on conductive materials) can be modified for tunneling electron spectroscopy and used for investigation of local electron structure at surface. Dynamical measurements – imaging the surface at changing sample temperature or even during deposition of growing material – allow direct studying of phase transitions, behavior of individual atoms at surface diffusion, nucleation and growth. STM installed into ultrahigh vacuum chamber with various deposi-

tion and other techniques for surface analysis represents a powerful tool for complex experiments in surface science. AFM is the most used SPM technique especially due to variability of measuring modes, possibility of using various interactions for imaging sample surface and measurement at ambient conditions. Well defined atomically resolved measurements require ultrahigh vacuum conditions. AFM images obtained in this way can be interpreted with the help of theoretical models and chemical resolution is possible.

The contribution is focused on STM and AFM methods with respect of imaging surfaces with atomic resolution. It provides information on conditions and limitations of imaging, a comparison with other techniques and new prospects.



L13

MICROSTRUCTURE ANALYSIS OF NANOCRYSTALLINE MATERIALS AND NANOCOMPOSITES USING THE COMBINATION OF X-RAY DIFFRACTION AND TRANSMISSION ELECTRON MICROSCOPY

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The capability of the combination of the X-ray diffraction and the transmission electron microscopy for the microstructure investigations on thin film and bulk nanocomposites are illustrated on three experimental examples: two Cr-Al-Si-N coatings with different chemical compositions and one BN bulk nanocomposite. Using a modified kinematical diffraction theory that describes and explains the phenomenon of the partial crystallographic

coherence of crystallites, we could show that the analysis of the X-ray diffraction line broadening is able to reveal nanocrystalline domains organised in semi-coherent clusters, to determine the size of the nanocrystalline domains and the clusters, and to quantify the mutual orientation of the partially coherent crystallites within these clusters. (see page 67 for more).

L14

INTERDIFFUSION IN SiGe ALLOYS STUDIED BY X-RAYS

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A growing importance of SiGe-based electronic and of optoelectronic devices in recent past is evident due to a significant progress in Si/SiGe band gap and strain engineering [1]. Devices such as MOSFETs are one of these, which attract their attention very intensively due to their application in CMOS circuits. Quantum cascade lasers and light emitters gain their interest for its current threshold and low power consumption. The production of light emitters have been already mastered to a certain extent in III-V materials, but making of optoelectronic devices on the basis of IV-IV materials is more challenging because of their compatibility to a standard Si technology [2].

A typical obstacle that has to be overcome in design, processing and operation of all electronic devices is the thermal load during fabrication and during operation. Due to heat dissipation in the circuits and operation of devices at high temperatures, the diffusion processes of the materials has to be taken into account. Thus a detailed knowledge of the diffusion in SiGe alloys is highly desirable. Unfortunately the interdiffusion process in SiGe is unlinear and strongly depends on Ge concentration [3]. The parameters describing precisely the SiGe intermixing are still under investigation.

The interdiffusion coefficient of mixtures can be described by Arrhenius equation $D = D_0 \exp(E_A/kT)$ given by

activation energy E_A and diffusion pre-exponential factor D_0 . The non-linearity of the diffusion process in SiGe alloys consists in the strong dependence of E_A and D_0 on the Ge concentration X_{Ge} in $Si_{1-x}Ge_x$ [3,4]. Up to now only activation energies E_A and diffusion prefactors D_0 for X_{Ge} up to Ge contents of 50% were reported with comparatively large error bars [4]. Recently the authors Aubertine et al. have performed a systematic measurement of interdiffusion in SiGe multilayers with estimation of E_A and D_0 for $0 < X_{Ge} < 0.20$ with relatively small experimental error [5]. To our knowledge the parameters E_A and D_0 for $Si_{1-x}Ge_x$ in the range of X_{Ge} from 0.5 to 1 are not well reported.

The aim of our investigation was to extend the knowledge about SiGe interdiffusion process also for the range of Ge content from 25 % to 50 % and to determine new values of E_A and D_0 . We have annealed simple $Si_{1-x}Ge_x$ multilayers grown by molecular beam epitaxy on relaxed SiGe pseudosubstrate with graded Ge content from pure Si up to constant composition top layer. The multilayer itself consisted nominally from 30 periods of SiGe/Si bilayers sandwiched in between a 20 nm thick step graded layer system and covered by an additional SiGe cap layer due to strain symmetrization. More about the sample structure and

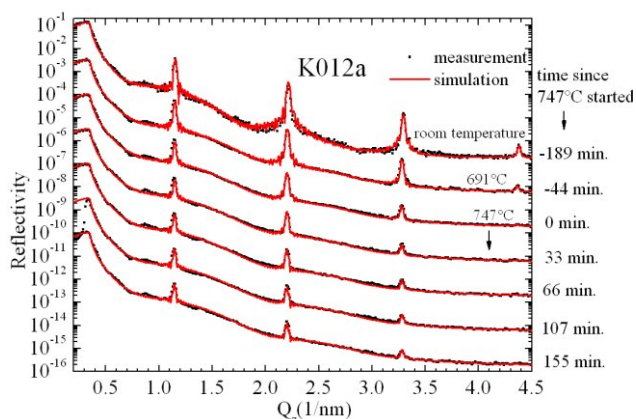


Figure 1. Evolution of x-ray reflectivity during annealing of $\text{Si}_{0.7}\text{Ge}_{0.3}/\text{Si}$ multilayer at $747\text{ }^\circ\text{C}$.

about the experiment can be found in our recent publication [6].

In order to study the diffusion properties of SiGe alloys in multilayer structures, we have used in-situ x-ray reflectivity and diffraction techniques performed at ESRF at beamline BM20. The critical temperature, where the interdiffusion started to be observable in our multilayers, was around $700\text{ }^\circ\text{C}$ and thus we have performed several isothermal annealing measurements above this temperature in order to obtain activation energy E_A and diffusion pre-exponential factor D_0 . Reciprocal space maps were recorded at several stages of annealing and for several temperatures mostly around $800\text{ }^\circ\text{C}$. The diffusion coefficients were obtained from the Ge content profile and from the profile of electron density, which were determined from

simulations of specular reflectivity and diffraction data, see Fig. 1.

The results were compared with data recently published by Aubertine *et al.* [5], who report diffusion coefficients showing linear decrease of activation energy and exponential decrease with Ge concentration. Our experiments show good agreement with extrapolation of these data [6] for Ge contents 50 % and 25 %. This statement suggests further generalization whether the dependency of E_A or D_0 is linear or exponential for the whole range of Ge content.

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4. D.B. Aubertine, M.A. Mander, N. Ozguven, A.F. Marshall, P.C. McIntyre, J. O. Chu, P. M. Mooney, *J.Appl. Phys.*, **92**, (2002), 5027.
5. D.B. Aubertine & P.C. McIntyre, *J.Appl. Phys.*, **97**, (2005), 13531.
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Acknowledgements

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Lectures - Thursday, June 21

L15

See page 108 for more

L16

See page 110 for more



L17

ANALÝZA VPLYVU PARAMETROV NAPRAŠOVANIA NA KVALITATÍVNE ZLOŽENIE TVRDÝCH POVLAKOV

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V odbornej literatúre sme nenašli príspevky, ktoré by boli zamerané na sledovanie parametrov PVD naprašovania na tvorbu vrstvy. Väčšina autorov sa venuje kvalitatívnemu a kvantitatívnemu hodnoteniu PVD vrstiev vytvorených za predom definovaných podmienok [1-4]. V príspevku sa preto hodnotil vplyv predpätia a pracovného tlaku na kvalitatívne zmeny tvrdých vrstiev TiN/TiAlN vytvorených metódou PVD (konvenčný katódový oblúk) v zariadení PLATIT 1000. Rozsah použitých tlakov bol od 0,5 Pa do 5 Pa a hodnoty predpätia sa menili od 5 V do 500 V. Získané povlaky vytvorené na substráte spekaného karbidu (WC/Co) sa analyzovali pomocou rtg. difrakcie a to kvalitatívnou analýzou určujúcou fázové zloženie vytvorených vrstiev ako aj kvantitatívnym hodnotením difrakčných profilov, z ktorých sa analyzovala textúra povlakov a hodnota štruktúrnych napätí vo vrstvách.

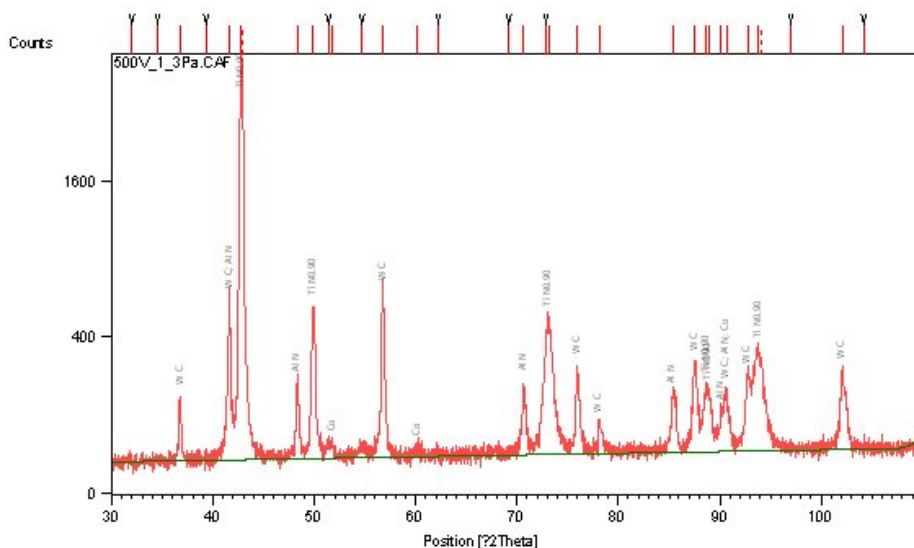
Ukázalo sa, že zmena parametrov naprašovania nemenila fázové zloženie vzniknutých vrstiev. Vo všetkých prípadoch sme detekovali prítomnosť nitridu titánu TiN_{0,9} a nitridu hliníka AlN, (AlTi)N. Substrát tvorila zmes karbidu volfrámu WC a Co. Difrakčný obraz pre parametre naprašovania (BIAS 500 V, p = 1,3 Pa) je na obr.1.

Zásadný vplyv zmeny predpätia sa prejavil v kvantitatívnom zložení vrstiev, stupni textúry a hodnote vnútorných štruktúrnych napätí vo vrstve. Kým nízke predpätia (5 a 25 V) vytvárali tenké vrstvy z relatívne nízkymi štruktúrnymi napätiami, pri dosiahnutí 75 V (100 V) bola vrstva optimálna z hľadiska hrúbky, ale mala

najvyššie hodnoty štruktúrnych napätí. Ďalšie zvyšovanie predpätia spôsobilo pokles hrúbky vrstvy v dôsledku vyššej kinetickej energie dopadajúcich iónov pôsobiacich odprašujúcim účinkom. Súčasne mierne klesala hodnota štruktúrnych napätí a textúry.

Zmena pracovného tlaku v naprašovacej komore sa výrazne neprejavila na nameraných difrakčných obrazoch vytvorených vrstiev.

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Obr. 1 Difrakčný obraz vytvorenej vrstvy na substráte spekaného karbidu

L18

KVANTITATIVNÍ RENTGENOVÁ DIFRAKČNÍ ANALÝZA DVOUFÁZOVÝCH TEXTUROVANÝCH VZORKŮ

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Základním problémem metodiky kvantitativní rentgenové difrakční fázové analýzy je texturovanost měřených vzorků, která mění intenzity difrakčních píků.

Pro fázovou analýzu byla použita bezstandardová metoda [1] pro dvoufázové netexturované vzorky. Texturovanost integrálních intenzit píků (hkl) obou fází je korigována pomocí faktorů $P(hkl)$ vypočtených Harrisovou metodou [2, 3]. Pro tuto metodiku jsou uvedeny matematické vztahy, výpočetní programy a jejich výsledky. Jsou uvedeny analýzy práškových vzorků a vzorků

s vysokým a nízkým stupněm texturovanosti a diskuse výsledků.

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L19

SEMIQUANTITATIVE METHOD FOR THE MONITORING OF THE GEOPOLYMER TRANSFORMATION PRODUCTS

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Synthetic geopolymers appear to be promising material applicable to a wide range of industrial branches, namely in building industry. Nevertheless products of geopolymer transformation can deteriorate properties of production. X-Ray phase analysis of geopolymers originating from thermal treated kaoline /metakaoline/ by alkaline treatment /NaOH and water glass/ has been carried out. Following products of the transformation have been identified: zeolite P, zeolite X, zeolite A, sodalite H and analcim T. A method for relative determination of the sum of the referred-to

phases has been worked out for the synthetic geopolymers, with respect to required quick determination and adequate accuracy. Data obtained in such way were collated with mechanical properties of the geopolymers arising from metakaolines at various temperatures of calcination and different ageing period, both at laboratory and 100°C temperature. Compression strength results correlated to the sums of the scaled intensities are presented proving applicability of the worked-out method.



L20

ČTVRTÉ MOMENTY V PROFILOVÉ ANALÝZE

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K popisu difrakčních profilů byly z matematické statistiky formálně převzaty některé veličiny charakterizující hustotu (rozložení) pravděpodobnosti $f(x)$ náhodné veličiny x . Motivem byla vnější podobnost tvarů hustot. Jednalo se zejména o momenty hustoty pravděpodobnosti, které jistým zhušňujícím způsobem charakterizují průběh, resp. tvar hustoty pravděpodobnosti:

$$M_0 = \int f(x) dx \quad \text{integrální intenzita,} \quad (1)$$

kteřou je rozumné normovat k 1, podobně jako je normovaná hustota pravděpodobnosti v matematické statistice. To umožňuje dále používat méně komplikované vztahy pro normované resp. redukované momenty (kolem počátku), které jsou definovány rovnicemi [1]

$$\begin{aligned} m_0 &= \int f(x) dx = 1 \\ m_1 &= \int x f(x) dx = T - \text{těžiště} \\ m_2 &= \int x^2 f(x) dx \\ m_3 &= \int x^3 f(x) dx \\ m_4 &= \int x^4 f(x) dx \end{aligned} \quad (2)$$

Centrální momenty, vztažené k těžišti, pak vycházejí [1]

$$\begin{aligned} 0 & \quad 1 \\ 1 & \quad 0 \\ 2 & \quad (x - m_1)^2 f(x) dx \quad m_2 - (m_1)^2 \quad W - \text{variance} \\ 3 & \quad m_3 - 3m_1 m_2 \quad (m_1)^3 \\ 3 & \quad m_4 - 4m_1 m_3 - 6(m_1)^2 m_2 + 3(m_1)^4 \end{aligned}$$

Důležitou vlastností momentů je, že pokud funkce h je konvolucí funkcí f a g , pak pro těžiště a pro variance platí [2]

$$T_h = T_f + T_g \quad (4)$$

a

$$W_h = W_f + W_g \quad (5)$$

Podobný jednoduchý vztah aditivity platí ještě pro třetí centrální momenty, zatímco pro čtvrté momenty platí [2]

$$m_{4,h} = m_{4,f} + 6 m_{2,f} m_{2,g} + m_{4,g} \quad (6)$$

V analýze difrakčních profilů se velmi často užívá druhý centrální moment – variance, nejen díky rovnici (5), ale taky díky své přímé fyzikální interpretaci [3]. Na užitečnost čtvrtých momentů v profilové analýze bylo upozorněno v [4]. Více pozornosti věnovali čtvrtým momentům autoři [5], zejména k určování velikosti částic, mikrodeformací a obsahu uhlíku v martenzitu z jedné difrakční linie. Jejich postup byl použitý k určení velikosti částic a mikrodeformací z jedné linie při studiu plastické deformace kovů [6]. V příspěvku bude pozornost zaměřena zejména na tuto problematiku.

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