X-RAY DIFFRACTION IN U. S. STEEL KOšICE RESEARCH LABORATORIES FOR PHASE ANALYSIS, TEXTURE AND RESIDUAL STRESS ESTIMATION

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Abstract

The X – ray laboratory long time oriented on texture analyses of rolled steel sheets has been working in U. S. Steel Košice, previously VSŽ since 1971. Many works were carried out during this period some of these were also published [1-4]. This laboratory has a specific focus on solving the problems of production and research. In the area of qualitative and quantitative phase analysis materials such as slags, raw materials, coatings, ceramics, dusts, sediments, ashes etc. were investigated. Possibility of X – ray laboratory are demonstrated on examples of annealed slag, damaged ceramic roller and brick exposed in furnace. Besides of rolled steel sheets texture also tin coating texture was analyzed recently in combination with EBSD method. The rolled steel sheets residual stresses estimation is part of analyses too; an example of annealed and rolled color coated sheets residual stresses investigation show the possibility of X- ray tenzometry.

Introduction

Analyses made in USSK Research Institute X-ray laboratory significantly contributed to research project oriented on new steels types development and technological procesess innovations. The X-ray workplace with orientation on qualitative phase analysis, residual stresses and texture has been in operation since 1971. For professional, accurate and fast solution of requirements in mentioned fields the analyzer furnished with special software and hardware are of high importance.

The first X-ray equipment was a powder diffraction X-ray analyzer equipped with Bragg-Brentan and Schultz texture goniometer, operated with Mo, Cu, Fe, Co and Cr X-ray tubes which allowed a variety of analyses. The originally manual equipment with a special texture graphic recorder for pole figure was fully automated in the nineties. Data collection and computer processing allowed new resolutions and improved results, which could not be provided by old manually controlled analyzer with special graphic recorder. The increased accuracy of results with good data evaluations allowed also their publication on international conferences and symposia [5-8].

New, modern specialized X-ray equipment with 3D goniometer was purchased in 2003 and the continuity of qualitative phase analysis, residual stresses and texture determination was maintained. The equipment was later supplemented by a movable sample holder, a set of Mo, Cu, Co and Cr X-ray tubes and with highly sensitive PDS detector of Meteor1D type. A part of equipment was basic Rayflex software, which was step by step completed by ZDS Search match with PDF2 database 2004, AutoQuant, MulTex and LaboTex. Together with older Rifran, popLA and BearTex software which has been used in previous analyzer, made the base to solve problems in X-ray laboratory. The job on Metallography Department combined with and X-ray laboratory supports the products quality control, cooperation with customers, contributes to the research experimental activities and is very useful for solving the failure analyses and urgent operational requirements. The qualitative and quantitative phase analysis include solution of materials such as: different types of hot and cold rolled steel, determination of retained austenite, various alloys, coatings, raw materials, slags, casting powders, sludges, refractories, ceramics dusts, sediments, ashes, and so on.

To continuously improve the quality of products it is necessary in many cases to know the texture and residual stresses in rolled steel sheets. The texture is directly related to anisotropy of mechanical and magnetic properties of hot rolled steel sheets of different grades. In this area the attention is concentrated mainly on deep drawing steels, IF steels, tin plates and non oriented steels. The actual research facilities enable modeling of technological processes: hot and cold rolling, batch and continuously annealing. Texture analysis of modeled samples enable to understand the physical and metallurgical processes and in such way to improve the quality of rolled products. Real results always have been obtained from real production processes for hot and cold mill and continuously annealing lines.

The database of collected and archived large amount of procedural and technological data is a source for products quality control. Data are stored in large files in computing centre of hot rolling mill and are used by quality control and research staff. Small database from measured and treated X-ray data was developed in Access program on the similar principle. The motivation for making the x-ray database was the large amount of archived data for last 10 years and laboratory diary archived since 1996. Nowadays the X-ray database serves not only as an electronic diary, but provides information about resolved diffraction records, which is helping to solve similar problems. About 1,350 samples data are recently recorded in database with stored basic information together with keywords. The database allows sorting the materials according to chemical and mineralogical names, keywords and mineralogical groups. The solution and results of each task are stored in metallographic protocols or reports. Many professional programs allow storage of results in some simple protocol which very often help operator or researcher in their work. It has a great significance in real operational practice, when analyzing the known or similar

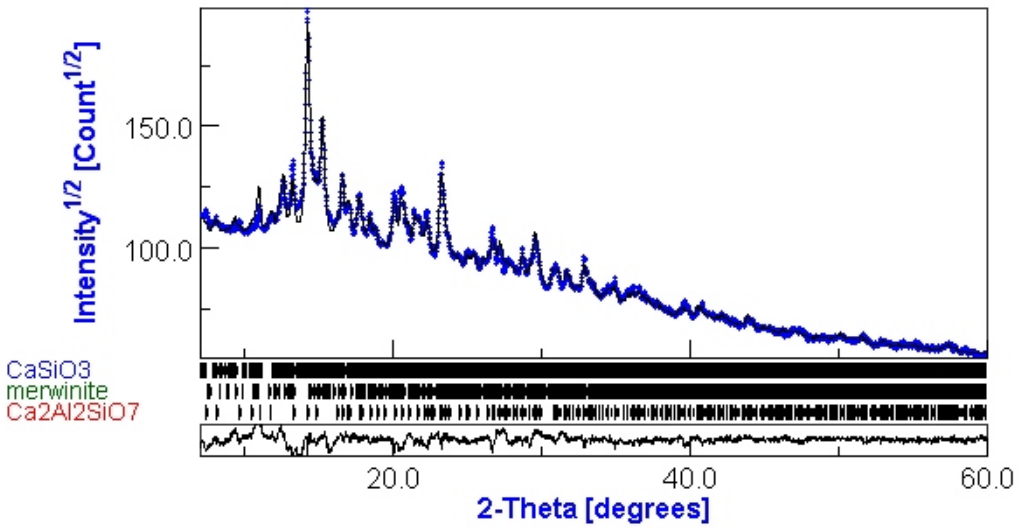

Figure 1. Fitted diffraction record of blast furnace slag annealing at 800 °C for 3 hours.

tasks the basic information about measured sample can be obtained in a very short time.

Qualitative and Quantitative Phase Analysis

In the last 20 years the work in X-ray laboratory has changed significantly. The original paper diffraction records which were made by plotter were replaced by electronic form and manual search was replaced by software search – match programs with rich diffraction database, which contains thousands of data. This requires certain knowledge and experiences, because the statistical equivalence of measured record with found in PDF2 database can lead also to erroneous results. Therefore, in many cases it is very suitable to confirm the obtained result with some of specifying program that uses the Rietveld method for fitting. The best known are the GSAS, FullProf, MAUD and BGMN programs, all these programs need the structural data for fitting. The data are stored in different formats; the most common format is .cif. The commercial program in this field, used in our X-ray laboratory is a AutoQuant program, that use .str format structural data, but has a limited number of this data.

Samples, such as slags, sediments, deposits, sludges etc., which are analyzed in X-ray laboratory in most cases are not perfect because they have a real structure. They can be fully or partially amorphous, textured and their structure can not coincide with the structure described in the diffraction and structural databases. To determine the phase composition of amorphous material by common X-ray procedure is not possible, but such material is possible to anneal in order allow crystallization. The blast furnace slag is an example of amorphous material which after annealing at 800°C for 3 hours transform to crystallized material and than is possible to determine its phase composition. Figure 1 shows the refined diffraction record measured by using Mo X-ray tube, where it was identified 53,4% weight content of $Ca_2Al_2SiO_7$ – Akermanite, 29,2% of Ca_3MgSiO_8 – Merwinite and 17,4% of $CaSiO_3$ – Pseudowollastonite. The same minerals were determined in the slowly cooled blast furnace slag brash.

The example of operation with X-ray database and real structured materials was the damaged ceramic roller which was analyzed about six years ago. At that time it was not possible to refine the diffraction record using AutoQuant and MAUD programs, these records were fitted recently as an example for fitting accuracy possibilities by means of those programs. The original SiO_2 roller consists of fine amorphous structure which was confirmed in the roll middle and about 0.7 mm below surface. After rolling of steel sheets the $FeSiO_4$ – Fayalite, SiO_2 – Quartz, SiO_2 – Cristobalite, SiO_2 – Tridimite a SiO_2 – Stishovite phases were detected on the roller surface. The compact sample was gradually ground and measured, for data processing by their fitting; the sample texture should be taken into account. Therefore the diffraction record was fitted by MAUD program which fits the textured records very well, figure 2, [9-12]. The program MAUD provides tools, which effectively calculate texture in sample different phases. These recalculated textures can be displayed with information about preferred orientation in different phases. The result of quantitative phase analysis is a graph in figure 3, which shows the changes in phase composition from ceramic roller surface to the depth of about 0.7 mm below the surface.

The example, where the AutoQuant program together with structural data can be used very effectively is the field of ceramics materials. The samples of bricks, which were exposed in the furnace, were submitted for examination by X-ray equipment. Analysis allowed obtaining valuable information about structural changes in different types of materials used for furnace lining. Several measurements were realized on the compact sample by using of movable holder. Fe - iron, Al_2O_3 - Corundum, $CaAl_{12}O_{19}$ - Hibonite, $FeAl_2O_4$ - Hercynite and $MgAl_2O_4$ - Spinel phases were identified in the diffraction records, which were fitted by AutoQuant and MAUD programs. The fitted record from affected area is shown on figure 4. The change in phase

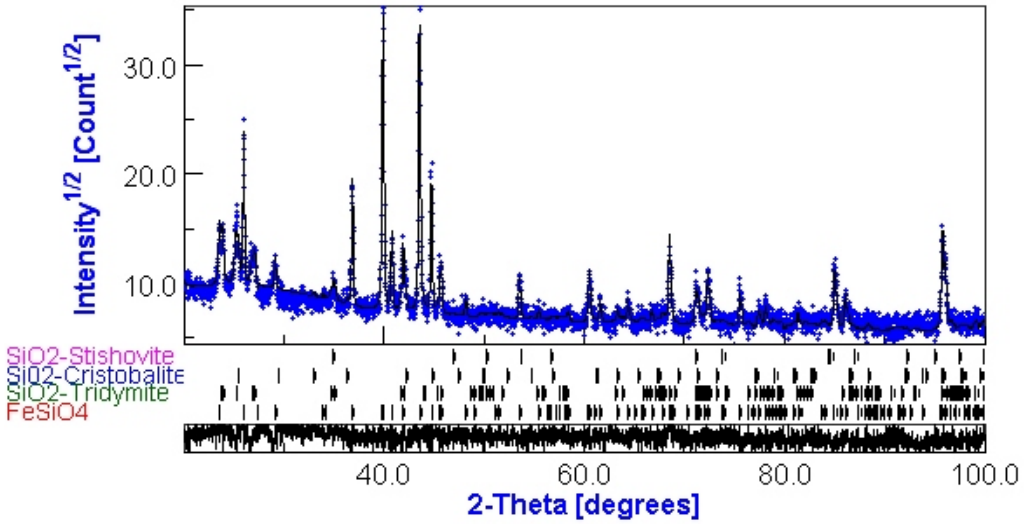

Figure 2. Fitted diffraction record of expanded ceramic roller.

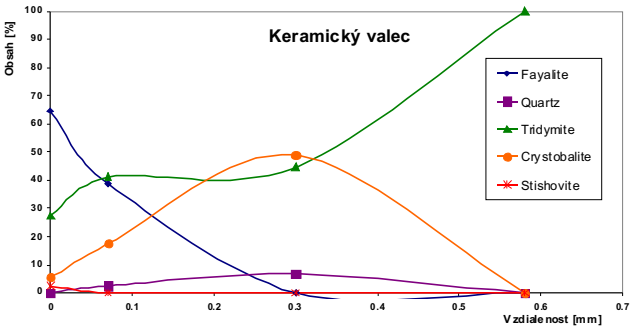

Figure 3. The phase contents dependence on distance from surface in expanded ceramic roller.

composition of refractory brick from the surface to a depth of 23 mm below surface is shown in figure 5. Because such amount of Spinel was not found in original material, the graph suggested, that the affected zone is deeper than removed and measured sample, deeper than 23 mm below the surface.

Texture analysis

Texture of samples was until recently studied mainly by X-ray and neutron diffraction using the texture goniometers. Nowadays the texture can be examined very effectively by electron beam with EBSD method. Both methods have their advantages and limitation. The advantages of X-ray texture analysis are minimum requirement for sample preparation which is becoming a priority for the measurement of cold rolled sample, because the preparation of that sample for EBSD is very difficult. The advantage of EBSD method is a detailed examination of samples and acquiring of extra information which can not be obtained, by classic X-ray analysis such as: grain size, grain boundaries data, phase composition etc. For texture calculation by EBSD methods it is not exactly clear how many

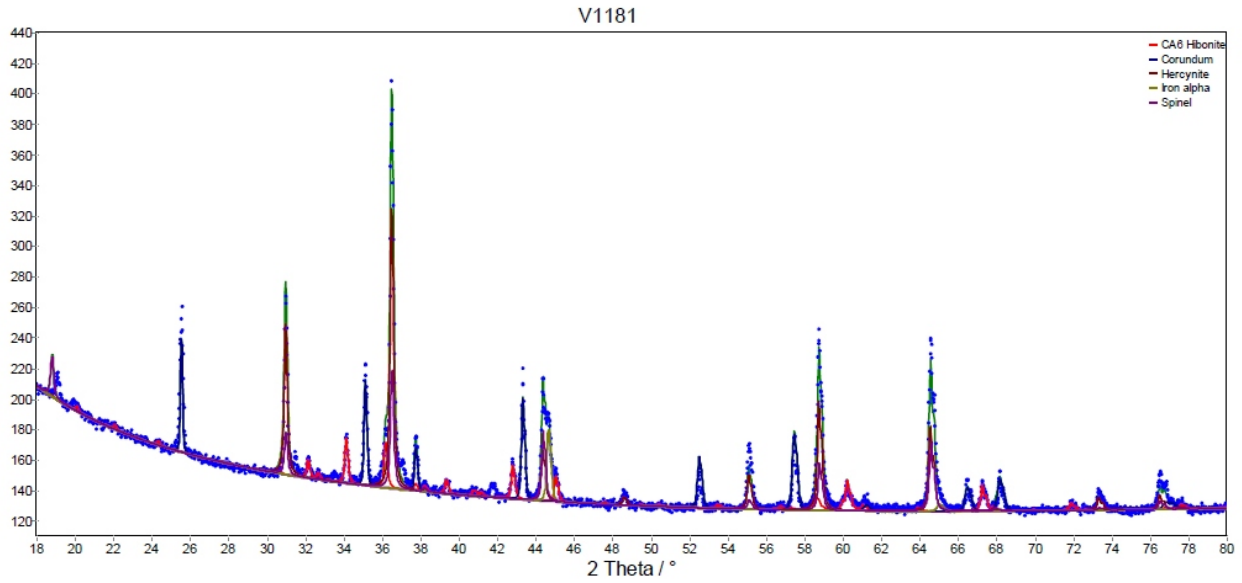

Figure 4. Fitted diffraction record of brick exposed in furnace.

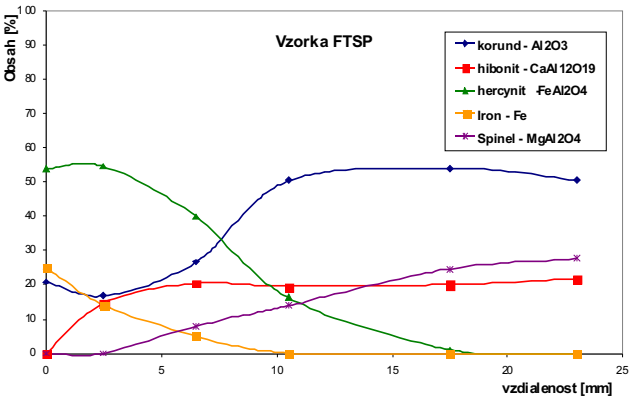

Figure 5. The phase contents dependence on distance from surface in brick exposed in furnace.

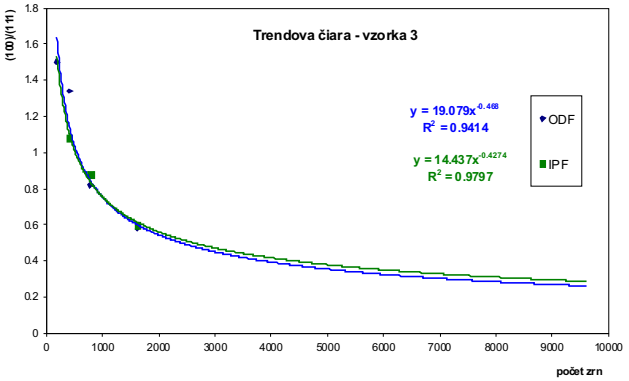

Figure 6. The effect of crystallographic ratio planes on number of grains.

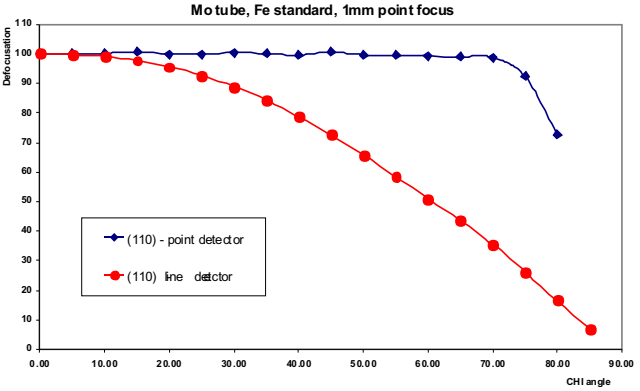

Figure 7. Defocusation curves for point and plane detector.

measurements should be made to guarantee sufficient accuracy for orientation distribution function – ODF calculation. The ODF calculation does not depends on the number of measured points, but depends on the number of measured grains, because they determine the preferred orientation in material. This problem is known from measuring of coarse grain material (oriented and non oriented electro technical steels) by X-ray beam. The EBSD methods enable many time repeated measurement with the same condition and used the sum of measurements for computation. The graph on the figure 6 shows the dependence of grain number on (100)/(111) planes crystallographic ratio calculated from the ODF and inverse pole figures – IPF which improve the result in dependence on number of measured crystallographic grain. The diagram is approaching in limit the value of 10 000 measured grains which is in practice difficult to achieved, but it is evidence that texture calculated only from few hundred grain could not be the representative texture characteristic of sample.

The X-ray diffraction texture analysis remains an effective method for examining texture of rolled steels, different coatings etc. The older point detector measured a maximum intensity at the top of diffraction peak and this value was strongly dependent on the texture goniometer set up and on number of impulses. The measured defocusation curves were also very dependent on 2 theta angle and rapidly descended in dependence on sample tilt chi angle. The present fast plane detector allows measuring the integral value of the diffraction profile, hence the values of the defocusation curves are stabile up to 70 degree of chi angle and also the chi angle is independent on the 2 theta angle. In many cases the linear detector allows to measure more than one diffraction profiles at once, whereby accelerates and improves the process of measurement. The diagram on figure 7 is showing the defocusation curves for point and linear detector using Mo tube and Fe standard.

Determination of texture for rolled steels is a main area of analyses, also the specific analyses are solved occasionally, e.g. to determined the texture of Zn or Sn coatings. The task was solved by both methods together with qualitative phase analysis and results were compared. In determining the texture using EBSD it has been and remains the main problem in sample preparation. At last the samples were measured without any preparation, only with simple cleaning of samples. In comparison with accuracy attained when measuring the rolled steels, the confidence index CI was quite lower, but largest set of measured data allowed calculating of ODF. The results in the simple form of pole figures are shown in figure 8. On the samples which were measured by X-ray methods, figure 9, ODFs and volume fractions of texture crystallographic planes and orientations were calculated. Diffraction records of compact coatings were highly textured; therefore the texture correction was used. The best results for strong texture correction were obtained by using the spherical harmonic functions – SFH and computed model pole figures were obtained, figure 10. The pole figures form both methods are in good conformity and show the same texture of tin coatings.

Analysis of residual stresses

The residual stresses are present mainly in hot and cold rolled steels, but stresses may also occur after an incorrect heat treatment when the cooling process was not homogenous and a thermal temperature gradient might arise in different locations. The residual stresses in the steel sheets cause the spontaneous bending, mainly during sheet cutting to strip forms. The bending depends on the direction of stress which can be in random direction. In practice the bend is named as the longitudinal and transversal buckles. The source of residual stresses generation is the mechanical or thermal gradient during the production or processing. Besides the internal stresses measurement and determination of their source, their elimination from prod-

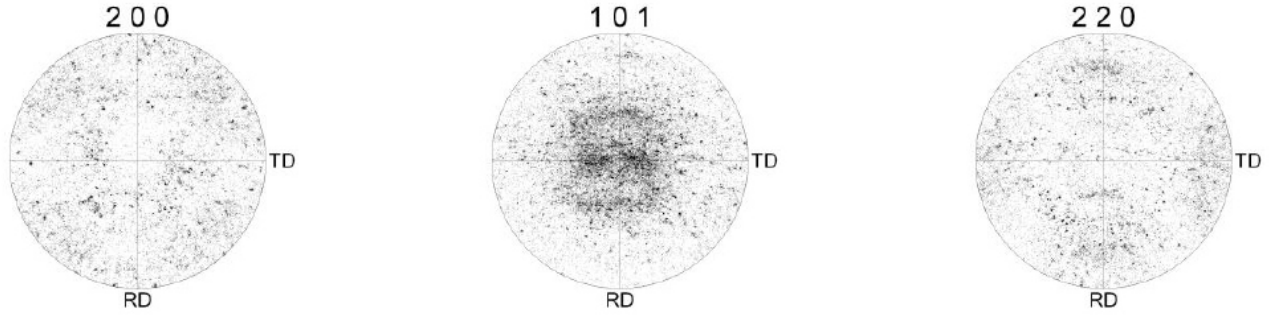

Figure 8. The pole figure of tin coating determined by EBSD methods.

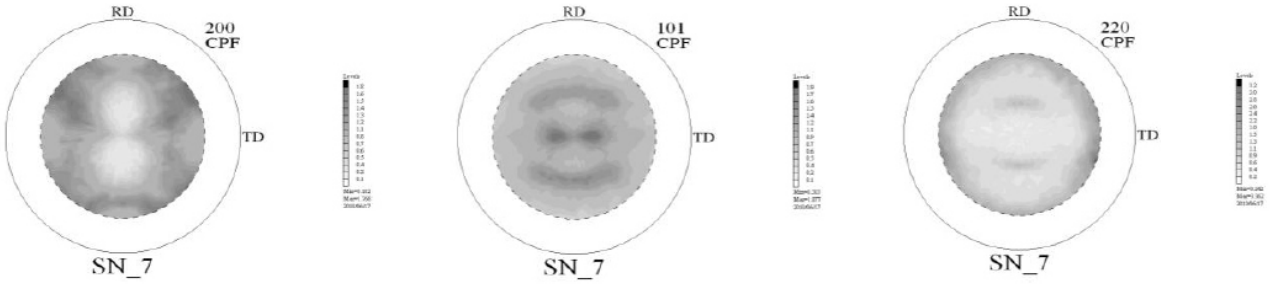

Figure 9. The pole figure of tin coating determined by X-ray methods.

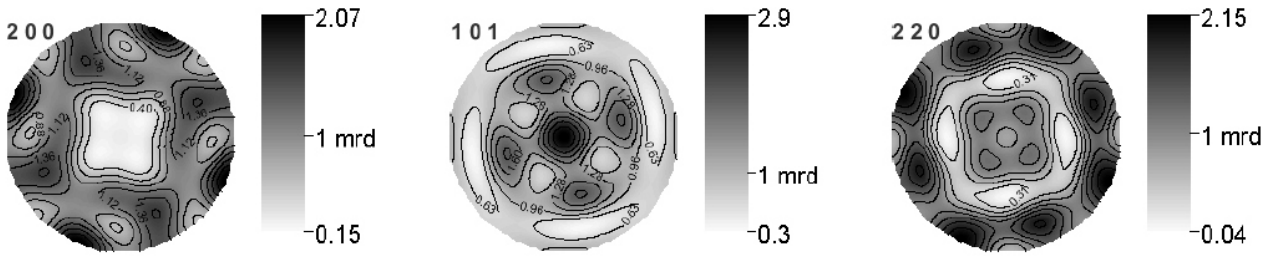

Figure 10. The pole figure modeled for correctet data by MAUD program.

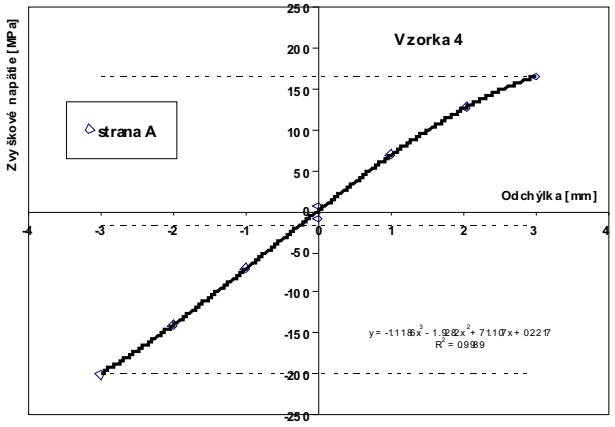

Figure 11. The dependence of generated stresses in color coated batch annealed steel sheet.

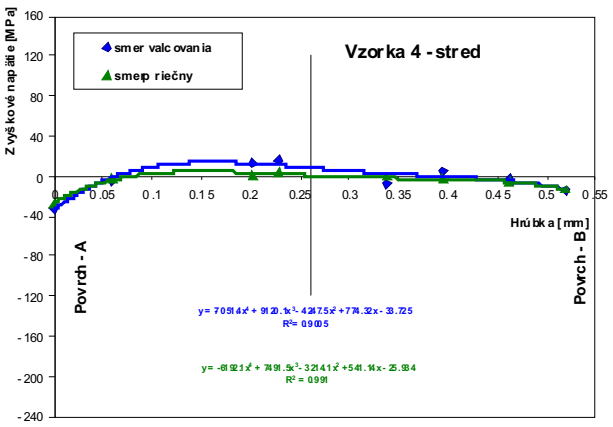

Figure 12. The stress profiles in color coated batch annealed steel sheets measured through thickness of sheet.

ucts is important too. This could by done in two basic ways: by heat treatment and by mechanical straitening. Both of them increase the economy of the production and therefore they are employed only in necessary cases. The main attention is paid on observing the technological parameters to prevent the residual stresses formation.

The $\sin^2$ X-ray tensometry method, most frequently with Cr X-ray tube, is used for determination of re-

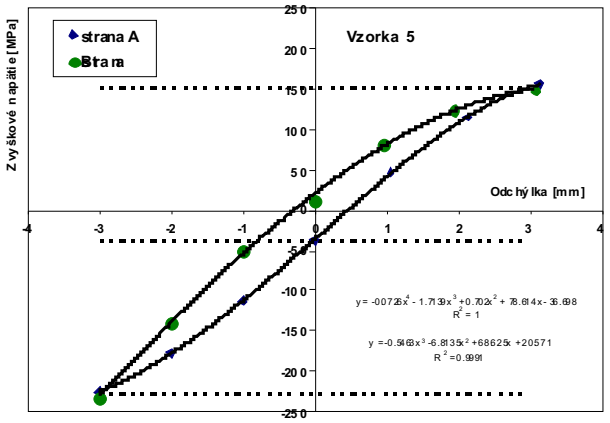

Figure 13. The dependence of generated stresses in color coated cold rolled steel sheet.

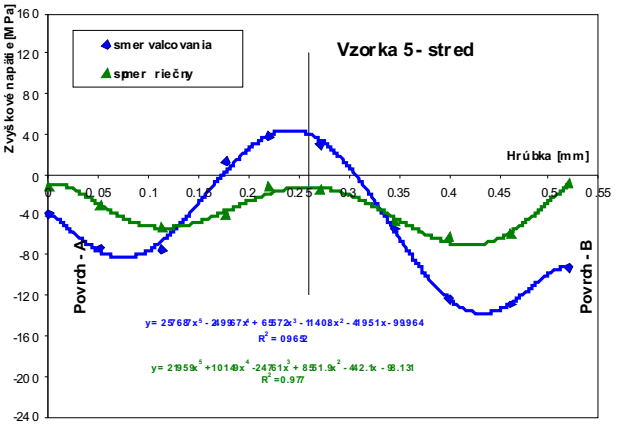

Figure 14. The stress profiles in color coated cold rolled steel sheets measured through thickness of sheet.

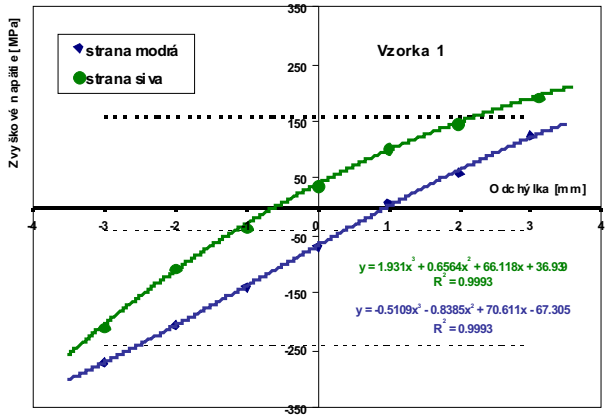

Figure 15. The dependence of generated stresses in color coated cold rolled steel sheet.

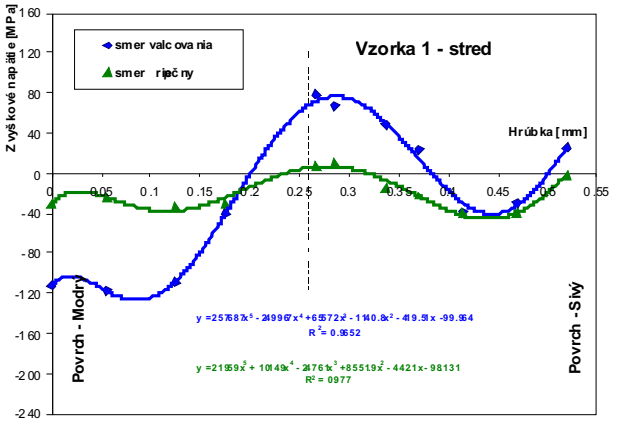

Figure 16. The stress profiles in color coated cold rolled steel sheets measured through thickness of sheet.

sidual stresses in the material. Samples are commonly measured in three direction (the rolling, transversal and at 45 degree direction to rolling) which allows to calculated the stress tensor in measured point.

The stresses in materials can also be generated by tension and bending. Simple tool was made on principle of four points bending that allows generating stresses, which depend on the bend intensity. The dependences of generated stresses in color coated, batch annealed and cold rolled steel sheets on bend intensity are graphically displayed on figures 11, 13 and 15. The color coated steel after annealing has zero stress, but bend generate tension or pressure stress in dependence on convex or concave bend, figure 11. The intensity of the bending deflection was measured by micrometric gauge used in setting the zero position of the sample in analyzer stand. A similar dependence can be obtained on cold rolled steel; figures 13 and 15 where residual stresses are not zero, therefore the shape of the curve have hysteresis loop form, which can be measured after cyclic loading or by measuring the magnetic properties. The "stress strain" curves on figures 11, 13 and 15 are shifted to the compression field, and the largest shift was measured on cold rolled samples, for annealed sample it was close to zero. The stress profiles measured through thickness of sheets in longitudinal and transversal direction are shown in figures 12, 14 and 16; stress curves in the longitudinal and transversal direction are not identical. The curves in the longitudinal direction are asymmetrical compared with curves in the transversal direction. A significant differences of residual stresses in rolling and transversal directions which are the largest in the sheet thickness center and in subsurface, the asymmetrical profiles of residual stresses through thickness of sheet and the shift of "stress strain" hysteresis loops caused by residual stresses are probably the reason of macroscopic effects of residual stresses (different bending of sheets, waves, etc.).

Conclusion

Nearly 40 years the functional X-Ray laboratory continuously supports the production and research by various analyses on different materials. In this period the laboratory many times updated its equipments, methods, hardware, software etc. The first originally manual equipment was automated and later replaced by new powerful one with new software which was gradually completed by specific software. The current hardware and software furnishing allows solving many tasks of qualitative and quantitative phase analysis, textures and residual stresses.

Most of the investigated materials has a real structure and are partially or fully amorphous. The described example was primarily the amorphous slag, which crystallized during annealing and phase components were determined by using AutoQuant and Maud programs.

Other presented examples represented a ceramic roller and ceramic brick exposed in furnace. After the identification of phase composition by PDF2 diffraction database, recorded data were fitted by programs using Rietveld method to determine the weight portion of phase components. Structures that can not be determined by chemical methods were found and described.

However the texture of tin coatings was determined by EBSD method, but as the sample preparation was and still is very difficult, the X-ray texture analysis was an effective and rapid solution. Obtained results showed good agreement with EBSD and also with corrected pole figures generated by MAUD program.

The measurement of residual stresses in color coated steels showed different values in various directions and asymmetric dependence through thickness of steels. Internal stresses were generated on the principle of four – point bending, which were displayed by "stress – strain" curves in form of hysteresis loop.

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CRYSTALLOGRAPHY AT THE INSTITUTE OF INORGANIC CHEMISTRY OF THE SLOVAK ACADEMY OF SCIENCES, BRATISLAVA

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The Slovak Academy of Sciences (SAS) has been founded 18 June 18th, 1953. The Chemical Institute was one of its newly created Institutes and, within it, a Department of Inorganic Chemistry was established. A three-member crystallographic group headed by F. Hanic started working at this Department, which was later transformed into a separate Institute of Inorganic Chemistry (IICH), and the crystallographic group became a basis for a Laboratory for Structural Research (*Laboratory* in the following). Because of lack of appropriate premises at the beginning, the individual Laboratories of the Department were "hosted" at various Chairs of the Universities in Bratislava. F. Hanic, together with I. Čakajdová and T. Veselovská were "hosted" at the Chair of Physics of the Faculty of Natural Sciences of the Comenius University (FNS CU), in the laboratory of the late J. Maďar. D. Štempelová (later Gyepesová) joined this group in 1960.

F. Hanic attended in 1951 a lecture course in "Radiocrystallography" lead by Assoc.Prof. A. Kochanovská from the Institute of Technical Physics of the Czechoslovak Academy of Sciences (ITPH CAS) in Prague, who accepted an invitation by Prof. Dr. R. Lukáč, who headed the Chair of Mineralogy and Petrography (CMP) of the Slovak Technical University in Bratislava (STU). F. Hanic, who was at the time finishing his studies at the Faculty of Chemical Technology (FCHT) STU became interested in this field and he became a PhD student of A. Kochanovská at her Institute in Prague. Under the supervision of Dr. V. Syneček, he gained a solid basis for his future work in the single-crystal X-ray structure analysis at the SAS. The Laboratory was later expanded by M. Handlovič, I. Kaprálik, and by technicians K. Jurčo, Z. Klčovanská and O. Šályová. F. Hanic headed this group until 1961 when he was appointed as a director of the IICH. F.Hanic supervised eight PhD students, who then started the X-ray structural research at various Faculties in Bratislava, but also in Brno. Together with his team he solved about 35 crystal structures, mainly those of coordination compounds but also of organic structures, the vitamin B6 being among them. It is worthwhile mentioning that, at the beginning, the structures were solved virtually "by hand". The intensities of individual diffractions on Weissenberg patterns were estimated visually and later from integrated patterns, photometrically. The Fourier syntheses were calculated with the help of Beevers-Lipson strips and a desk calculator. The situation significantly improved in 1962 when SAS purchased an East-German (DDR) computer ZRA-1, which used punched cards. Still during "hosting" at the Chair of Physics FNS CU he, together with J. Maďar and mechanician A.Kiss, constructed a prototype of a precession camera, which differed from the Buerger model by some significant (and patentable) details. This camera was later produced also commercially. In 1970 he was expelled from the Communist party and, consequently, he lost also

his position as Director of the Institute. He worked afterwards as a rank-and-file scientist at another Department of the Institute.

His successor at the Laboratory became S. Ďurovič, who became interested in structural crystallography in 1949 as a student at the FCHT STU. He attended a lecture course in structural crystallography lead by Prof. Dr. J. Novák from the Faculty of Natural Sciences Charles University in Prague. After having finished his studies, S. Ďurovič became an assistant at the Chair of Prof. Lukáč. He obtained a basic knowledge in single-crystal X-ray structure analysis from V. Syneček during summer praxis at the ITPH CAS in Prague. Together with his former student V. Kupčík, he started an experimental work. Later also E. Makovický, another former student of S. Ďurovič, joined them. V. Kupčík and E. Makovický started the structural investigations of sulfosalt minerals and laid down the basis for their classification. They continued in this direction also during their stays in West Germany and Canada, respectively, but, unfortunately, they could not return home during the so-called "normalization" after the Soviet-lead invasion of Czechoslovakia in August 1968. Both of them were later appointed as Professors at the Universities of Göttingen (Kupčík) and Copenhagen (Makovický).

F. Hanic started also collaboration with the Institute for Structural Research of the Academy od Sciences of the German Democratic Republic in Berlin, mainly with its Director, Prof. Dr. K. Dornberger Schiff, the founder of the theory of OD (Order-Disorder) structures. They decided that somebody from the Laboratory should learn this "craftsmanship" and have chosen S. Ďurovič, who spent the three months in Berlin to learn the basic ideas and assisted afterwards at the Summer School on OD Structures. The experience thus gained helped him to solve an OD structure of -$Hg_3S_2Cl_2$ using diffraction data from disordered crystals, during his two-year (1966-7) Postdoctorate Fellowship at the Department of Geology McGill University in Montreal (Canada). In Montreal he also refined the crystal structure of mullite, which he solved as first in 1962. Successful collaboration with K. Dornberger-Schiff resulted in an OD interpretation of basic types of sheet silicates later followed, in an efficient partnership with Z.Weiss from the Coal Reserch Institute in Ostrava, by an elaboration of a unitary geometrical theory of the polytypism of sheet silicates.

In 1979 the Laboratory was joined by Ľ. Smrčok who just finished his studies at the FNS CU, where he was attracted to structural crystallography by Ľ. Ulická. His main field of interest became X-ray powder structure analysis and crystallographic computing. In 1981 D. Mikloš, who joined the Laboratory in 1968, replaced S. Ďurovič as the Head of the Laboratory. After 1981, three scientists of the Laboratory became functionaries of the Institute, which had a negative impact on the research work and in 1990 the Laboratory was eventually included into the Department of Theoretical Chemistry. In the nineties the Laboratory hosted two young post-doc scientists (E. Morháčová and J. Kečkéš) and later also three PhD students (M. Ďurík, O. Pritula, M. Sládkovičová).

Among the most significant achievements of the Laboratory belong solution of the crystal structure of Ge-mullite and a single-crystal study of thermal transformation of sillimanite, structure solution of $Ca_{10}(Cr^{V}O_4)_6(Cr^{VI}O_4)$, of $TiCl_3.3C_4H_8O$ and of several oxoperoxo complexes of vanadium (V), particularly sensitive to temperature. Later, in the nineties, the Laboratory started a collaboration with Prof. V.Langer (Göteborg) on crystal and electronic structure determination of chiral derivatives of saccharides, largely the aminoderivatives, which are potent ional efficacious compounds in pharmacology as well of some copper(II) complexes containing Schiff base, derived from salicylaldehyde and *L*-glutamic acid and *N*-, or *O*- donor neutral ligands. An official collaboration has also been started with Institute of Physics CAS in Prague, the result was a refinement of the crystal structure of cronstedtite-3*T* and later also of the 1*T* and $2H_2$ polytypes of this mineral, with an efficient partnership by V. Petříček, but mainly by J. Hybler. An OD interpretation of the cronstedtite structure(s) was decisive in the explanation of its twinning and parallel intergrowths. The X-ray work was greatly corroborated by a high-resolution transmission electron microscopy (HTEM) carried out by T. Kogure from Japan. Also a collaboration with M. Nespolo (Japan, France) on a relation of OD twins to general twins can be mentioned here.

In the course of years the Laboratory coordinated an extensive work on Czech and Slovak crystallographic nomenclature, S. Ďurovič, D. Mikloš prepared the screenplay for educational movie "Crystals and Structure Analysis" (director J. Kořán, Krátký film, Prague). The members of the Laboratory also organized short several courses (a course on OD structures, courses on crystallography and crystal chemistry of silicates) and two annual meetings of Czechoslovak Crystallographers. S. Ďurovič was a members of an *ad-hoc* Commission of the International Union of Crystallography for the Nomenclature of Modulated, Disordered and Polytypic Structures and participated in elaboration a proposal for the construction of descriptive polytype symbols. However, the most important contribution of a member of the Laboratory to crystallography is no doubt the chapter "Layer Stacking in General Polytypic structures" for International Tables for Crystallography, Vol. C written by S.Ďurovič.

Application of X-ray Diffraction Methods on the Department of Inorganic Chemistry, Institute of Chemical Sciences, Faculty of Science, P. J. Safarik University in Košice

APLIKÁCIA METÓD RTG. DIFRAKCIE NA KATEDRE ANORGANICKEJ CHÉMIE ÚSTAVU CHEMICKÝCH VIED PRÍRODOVEDECKEJ FAKULTY UNIVERZITY P. J. ŠAFÁRIKA V KOšICIACH

Juraj Černák

Katedra anorganickej chémie vznikla v roku 1965 rozdelením spoločnej Katedry chémie [1]. Vlastné priestory získala rekonštrukciou budovy na Moyzesovej ulici č. 11 v Košiciach, kam sa pres ahovala v akademickom roku 1966/7 a tu sídli do dnešného dňa.

Počiatky používania difrakčných metód štúdia je možné položi do roku 1971, keď sa začal prednáša predmet Štruktúrna analýza. Tento predmet v tom čase viac rokov zabezpečoval externý učiteľ katedry, ešte stále aktívny pán prof. Ing. Ján Garaj, DrSc. Neskôr v roku 1979 ho v pozícii externého učiteľa nahradil pán doc. Ing. Dunaj-Jurčo, CSc., obaja z Katedry anorganickej chémie CHT STU v Bratislave. Od roku 1982 predmet zabezpečovali interní učitelia katedry, najprv doc. RNDr. Jozef Chomič, CSc. a o rok neskôr ho začal zabezpečova prof. RNDr. Juraj Černák, CSc., od roku 1998 v spolupráci s doc. RNDr. Ivanom Potočňákom, PhD. V roku 2000 k základnému predmetu pribudol aj nadstavbový predmet určený predovšetkým pre diplomantov katedry s názvom Výpočtové metódy v štruktúrnej analýze, ktoré mali charakter interaktívneho seminára spojeného s využitím kryštalografického softvéru a prácou na počítači. Oba tieto predmety sa vyučujú doteraz.

Experimentálne vybavenie vzhľadom na jeho finančnú náročnos bolo dlhodobo na katedre skromné. V Laboratóriu štruktúrnej analýzy sa nachádzal štandardný práškový difraktometer Mikrometa 2 s goniometrom GON 3, ktorý umožnil osvoji si experimentálnu prácu spojenú s rtg. difrakciou. Prístroj sa využíval hlavne na stanovenie fázovej identity medziproduktov a konečných produktov termického rozkladu.

Neskôr k tejto Mikromete v roku 1978 pribudol druhý vysokonapä ový zdroj s rtg. lampou, na ktorý sa ako nadstavba použil Weissenbergov goniometer. Tento sa využíval na overenie monokryštálového charakteru pripravených kryštálov a získanie predbežných kryštalografických údajov študovaných kryštálov (mriežkové parametre, priestorová grupa, spresnenie vzorcovej jednotky). S tým súvisela aj prevádzka tmavej komory, v ktorej sa spracovávali exponované filmy (z finančných dôvodov sa používali filmy pre lekárske účely). Takto študované kryštály sa spočiatku odosielali na zber dát (intenzít) na difraktometer Syntex P21 na Katedre anorganickej chémie Chemickotechnologickej fakulty STU v Bratislave, alebo sa snímkovanie realizovalo na zahraničných pracoviskách v rámci vedeckej spolupráce. Prvou takto študovanou látkou bola komplexná zlúčenina $[Zn(en)_3][Ni(CN)_4]\cdot H_2O$ [2]. Namerané dáta sa nakoniec spracovávali, teda riešenie štruktúry a jej upresňovanie sa realizovalo na počítačoch najprv v Bratislave, neskôr od roku 1980 aj v Košiciach na počítačoch SMEP na UPJŠ, resp. EC 1045 na TU v Košiciach po príslušnej implementácii kryštalografického softvéru. Podmienky na používanie kryštalografického softvéru sa výrazne zlepšili po nástupe éry PC počítačov. Uvedené prinieslo implementáciu sady kryštalografických programov SHELX do každého PC a on-line prístup do Cambridgskej kryštalografickej databázy (od roku 2001).

Metódy štruktúrnej analýzy sa hojne využívali aj v pedagogickom procese pri vypracovaní diplomových prác. V ďalšom období sa postupne zlepšovala know-how zamestnancov katedry, keď viacerí absolvovali zahraničné študijné pobyty, počas ktorých sa zdokonalili v používaní difrakčných metód a počas ktorých sa merali difrakčné dáta. Rozvinula sa aj vedecká spolupráca, najmä s partnerskou Katedrou anorganickej chémie CHTF STU v Bratislave (doc. Ing. M. Dunaj-Jurčo, CSc.), ako aj ďalšími pracoviskami: KACH MU v Brne (prof. Z. Žák), s FZÚ AVČR v Prahe (Dr. Petříček, Dr. Dušek), s Univerzitami v Poitiers (prof. C. Kappenstein), Parme (prof. P. Domiano), MLU Halle (prof. Steinborn) s Karlovou univerzitou v Prahe (Dr. I. Císařová), s Univerzitou v Gainesville, Florida (Dr. K. Abboud), s Palackého Univerzitou v Olomouci (prof. Z. Trávníček), s Philipps-Universität v Marburgu (prof. W. Massa, Dr. K. Harms), s Universidad de Zaragoza (prof. L.R. Falvello) a ďalšími. V spolupráci s týmito pracoviskami sa publikovalo podľa výsledkov rešerše v databáze WoK vyše 100 pôvodných vedeckých prác v karentovaných časopisoch, v ktorých sa prezentovali výsledky štruktúrnych analýz.

Od roku 1998 Katedra získala právo uskutočňova doktorandské štúdium. Prakticky každý študent doktorandského štúdia použil vo svoje dizertačnej práci rtg. difrakčné metódy; niektorí používali túto metódu okrajovo, ale väčšina prác bola výrazne orientovaná na používanie difrakčných metód štúdia, najmä na kompletné stanovenie kryštálovej štruktúry na báze monokryštálov. S využitím difrakčných prác bolo obhájených 7 dizertačných prác vypracovaných na katedre a v súčasnosti sa realizuje doktorandské štúdium 10 študentov.

Výrazná zmena experimentálneho vybavenia Laboratória štruktúrnej analýzy nastala v roku 2007, keď Dr. V. Petříček z Fyzikálního ústavu AV ČR v Prahe ponúkol starší monokryštálový difraktometer Oxford Diffraction s CCD detektorom. Po jeho inštalácii, s výraznou pomocou Dr. Dušeka z toho istého ústavu výrazne narástol počet študovaných kryštálov. Metóda štruktúrnej analýzy sa stala štandardnou metódou používanou v rámci magisterských a doktorandských záverečných prác, ako aj pri riešení grantových projektov zameraných na štúdium nových koordinačných zlúčenín s biologickou aktivitou, nízkorozmerných magnetík, mikro- a mezopórovitých látok ako aj organických zlúčenín. Z personálneho hľadiska merania na difraktometri zabezpečujú kolegovia doc. RNDr. Ivan

Potočňák, PhD., RNDr. Juraj Kuchár, PhD. a RNDr. Martin Vavra, PhD. Už v tomto roku by sa malo dôjs k výraznému vylepšenie podmienok zberu dát inštaláciou chladiča kryštálov z prostriedkov Operačného programu Výskum a veda, čím by sa umožnilo štúdium pri nízkych teplotách a rozšíril diapazón študovaných látok o tie, ktoré pri laboratórnej teplote sú nestále.

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