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Past and Present of X-ray Residual Stress Measurement in Czech Republic

"Stress Problems" Solved for Machine Industry

Stress measurement, especially that of residual stresses, represents one of the most wide-spread and technically most important applications of X-ray diffraction. In Czech Countries this area of experimental physics has almost reached a 60 year old tradition. From the point of view of national machinery traditionally much attention has been paid to the X-ray stress analysis, namely for its possibility of reliable and effective checking of metal and alloy surface treatment technologies. That is why all the Czech X-ray laboratories are trying to keep pace with country powers of X-ray tensometry such as Germany, U.S.A., Great Britain, France, Japan and Russia, both in computer assisted control of experiments and in finding new ways of interpretation of measured strains in interatomic distances.

The oldest information about the diffraction research on changes induced in solid state substance structure by exterior forces comes from Roentgenological and Radiological Institute in Petersburg, where shortly after the World War I A.J.Joffe applied Laue's method for assessing the anisotropy of elastic limit dependence on temperature in monocrystals of NaCl, CaSO₄, and some natural minerals. In polycrystalline materials the primacy in this area of experimental physics is shared between Americans H.H.Lester and R.H. Aborn, who in 1925 measured the lattice strain of metal crystals in tensile stressed steel sample using the Debye-Scherrer method; it was shown that interplanar distances change linearly with the stress. The problem of the X-ray assessment of stress was treated theoretically by the Russian G.J.Aksjonov in 1929. However, the diffraction method gained practical meaning for stress measurement owing to the back reflection arrangement of the Debye-Scherrer method introduced in the early thirties in Germany.

The Czechoslovak scientific world was presented with the concept of X-ray radiation issues applied to the research in the quality of metals as early as 1931 in a comprehensive article named "Materials testing by means of X-rays". The authors of this article, P.Skulari and V.Miklenda, did not only work out a thorough survey of technical applications of X-ray radiation known so far, but pointed out at the same time the strong support given to the X-ray crack detection, spectral and structure analysis in Germany, the U.S.A., France and the U.S.S.R.

In Czechoslovakia, the first measurements of residual stress by means of X-rays were probably performed by A.Kochanovská in 1936. She investigated the origin of cracking in the cover of shells. This research was of military character and therefore its results could not be published. Between 1937-1939 the Czech physicist P.Skulari paid attention to X-ray stress analysis of forged aluminium, to head treated iron and steel, and to residual stress non-homogeneities near welded seams. Kochanovská and Skulari are credited with introducing diffraction stress

analysis into X-ray laboratories of Czechoslovak universities as well as research institutes. The industry was in this country interested mainly in the following topics:

- residual stress distribution in welds of steel tubes,
- macroscopic residual stress on the surface of hardened high-strength steel,
- residual stress measurement in front of the fatigue crack.

At present X-ray diffraction for residual stress investigation is used in several Czech and Slovak laboratories. However, the most systematic development in this field of experimental stress analysis is concentrated at the Faculty of Nuclear Sciences and Physical Engineering (FNSPE) of the Czech Technical University in Prague.

In the early sixties research in the X-ray laboratory of the Department of Solid State Engineering of the FNSPE specialized in X-ray investigation of sintered carbides of the system WC-Co and the following problems were studied, e.g.:

- The dependence of thermal microstresses in WC-Co system on the content of Co.
- The influence of the average grain size of WC and the content of the binding cobalt phase on X-ray measurement of macrostresses in sintered carbides of the WC-Co system.
- The theoretical model of the WC-Co system structure.
- The study of factors undesirably affecting the strength of hard metal tips.
- The Czech industry of powder metallurgy was also interested in properties of nickel powder and carbonyl iron powder compacts. Residual macrostresses were measured by X-ray diffraction

- on plastically deformed samples of nickel powder, in dependence upon the applied pressure,
- in samples of cylindrical shape produced by one-side and isostatic pressing of powder carbonyl iron.

It was shown, that the interpretation of X-ray determined changes of interplanar distances due to the macroscopic stresses using the elasticity theory is justified in the plastically deformed samples only if the density of stampings is approaching that of a compact material. The higher the material's plasticity, the lower the pressure needed to satisfy this condition.

In the past twenty years these "stress" problems were solved in the X-ray laboratory of the FNSPE of Czech Technical University in Prague:

X-ray diffraction analysis of oxide films on zirconium alloys obtained by high-temperature oxidation in water, steam, or air,

residual stress in surface layers of the Cr-Ni steel of turbocompressor rotor blades,

X-ray determination of residual stresses due to grinding by cubic boron carbide,

stress analysis on the surface of large parts of a long-

distance austenitic steel gas pipeline,

X-ray stress analysis of cut thread surfaces,

X-ray residual stress measurement in gray cast iron.

Analysis of the state of stress on the surface of shot-peened samples of carbon steel by means of three different X-ray methods (by ψ -goniometer, by one-tilt method with no reference substance, and by two-exposure technique).

In many cases the X-ray residual stress analysis performed at the FNSPE are a service for the Czechoslovak machine industry, e.g.:

efficiency checks of stress relief annealing; the investigated samples (pipes) were manufactured from austenitic steel,

determination of the state of stress due to roller burnishing in surface layers of the trailing axle,

residual stress measurement on the surface of the landing gear segment,

X-ray analysis of residual stress due to induction hardening,

investigation of residual stress on the surface of samples made by cutting the collector wall of steam generator.

Diffraction Analysis of Non-uniform Stress State

Recently we have been witnessing a growth of interest in the surface qualities of solids. However, this fact is not surprising, when we take into account that any interaction with material is being realized over its free surface. Surface layers can influence in a decisive way the employment on the whole volume of material. Surface layers are primarily important in processes of brittle and fatigue fracture and the like.

Various surface engineering procedures, as well as many conventional technologies, introduce stresses on particular engineering products either intentionally or involuntarily. These stresses are confined to shallow surface layers only several micrometers thick. In this way, considerable stress gradients may be created which influence significantly the different characteristics of the products, sometimes favourably, sometimes detrimentally. There is no analytical technique which allows us to evaluate such non-uniform stress fields as efficiently as X-ray diffraction.

Classical X-ray methods of stress measurement accept the premises that the state of stress is homogeneous within the whole measured volume or may be considered as such (respectively to the capacity of the measuring method). However, as the X-ray penetrates into a certain depth under the surface, the justification of homogeneity depends on the depth of penetration and on the rate of lattice strain gradient within the limits of irradiated volume. One of the reasons why the effective depth of penetration T_{ef} is being neglected is its negligible size compared to linear dimensions of irradiated surface (in most technical metals the effective depth being in the order of 10^0 - $10^1 \mu m$). Generally however, such an approach is incorrect; for example, it was observed that the residual stress on welded parts may change in the layer of several tenths of μm not only its size, but even the sign. It's only natural that a question arises about how such a non homogeneous state of stress can reflect on the results of X-ray stress measurements by classical tensometric methods.

In the simplest case we can assume that inside the layer equal in thickness to the effective depth of penetration T_{ef} the stress changes but not its gradient. If the stress change already develops within the range of T_{ef} in a random way, e.g. as a result of an occasional change of stress gradient, all evaluation of diffraction patterns is invalid. For this reason, a certain idea about the stress distribution is necessary. A linear course of stress σ at a distance T from

the surface is being supposed most frequently. Destructive diffraction stress analysis is carried out in principle in such a way that the layers of material are gradually taken off either by grinding or chemical or electrolytic etching. After the removal of each level, we can determine on the bare surface the phase distribution as well as the residual stress level or the crystal size (coherent areas). However, we must consider that grinding brings about an additional state of stress to the material. Whereas in etching, this factor doesn't exist but a problem of selective dissolving of the surface (prior dissolving of certain structure elements) arises. Electrolytic polishing has proved to be the best so far, where no additional structure defects develop, and a regular layer of metal is being taken off the whole surface. The main restriction in grinding and etching off the surface layers is the fact that the conditions on the surface and in the interior of studied objects are not identical. For this reason the removal of the layers leads to the stress redistribution in the sample volume. So, while evaluating the measurements we must make correction on the undergone relaxation of the stress. (The change of the original state of stress is negligible if the removed, e.g. etched off layer, is only a fraction of the overall cross section of the examined part.)

The traditional ways of X-ray stress measurements consider biaxial state of stress in surface layers into which X-rays penetrate. Classical diffractational methods are based on the stress calculation from the line slope $\varepsilon(\sin^2\psi)$; ε are experimentally determined lattice strains, ψ is the angle between the surface normal and the direction of the strain ε . If the components of the considered biaxial state of residual stress in irradiated volume have a steep gradient, the graph $\varepsilon(\sin^2\psi)$ will be curved.

The premise of classical methods, that the stress components σ_{13} , σ_{23} , σ_{33} in the direction of the surface normal are negligible, has proved to be incorrect. These components were detected in surface layers after repairs such as grinding, milling, shot peening, nitriding etc. Both the theory and the experiments from the last years have proven that the existence of the stress σ_{13} , σ_{23} results in splitting the course $\varepsilon(\sin^2\psi)$, that is, the graph $\varepsilon(\sin^2\psi)$ has an inverse curve for $\psi > 0^\circ$ than for $\psi < 0^\circ$.

If in the direction of the surface normal there is $\sigma_{33} \neq 0$, while $\sigma_{13} = 0 = \sigma_{23}$, the dependence of $\varepsilon(\sin^2\psi)$ will curve but remains unsplit. The degree of curvature depends on how steep the gradient $\partial\sigma_{33}/\partial T$ is.

As σ_{33} is the stress perpendicular to the surface, according to the definition in the case of residual stress on the surface it will be σ_{33} . In the case of triaxial state of stress, there always develops a gradient $\partial\sigma_{33}/\partial T$ between the surface and the surrounding layers. This fact can also be expressed by the statement that σ_{33} can exist in surface layers only as a gradient. (In the same way as σ_{33} , there exist in surface layers the components σ_{13} and σ_{23} also only as a gradient. It also applies to them that on the surface there is $\sigma_{13} = \sigma_{23} = 0$.)

In principle, every calculation based on biaxial stress method applied to the curved course $\varepsilon(\sin^2\psi)$ will lead to incorrect values of surface stresses.

The relations $\varepsilon(\sin^2\psi)$ in which the diversions from linearity couldn't have been interpreted as a result of random errors have been observed since the early 70's in connection with the improvement of measurements and computer techniques. By the systematic research in diversely curved courses $\varepsilon(\sin^2\psi)$ found especially in iron materials three main reasons of non linearity have been proven:

- the gradients of stress (in solid solutions even the gradients of concentration) in the direction from the surface to the interior of the samples,
- the angle between the plane of principal stresses and

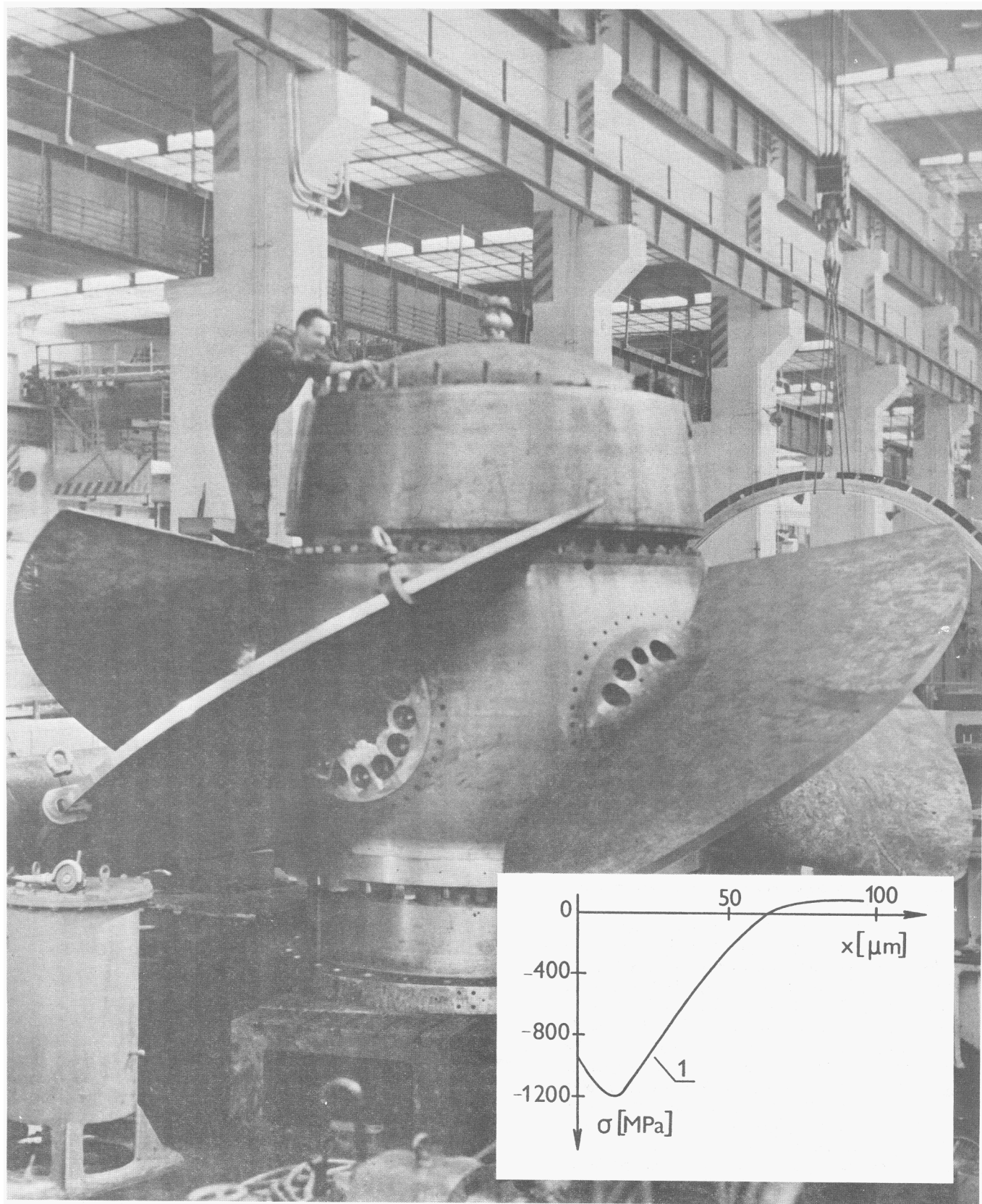


Fig. 1 Kaplan turbine runner assembly in CKD Blansko (Czech Republic) and residual stress depth distribution after shot peening.

the surface plane,

- strong surface textures.

The solution of the problems induced by these sources of systematic diversions from the linear course $\varepsilon(\sin^2\psi)$ influenced in a positive way improvement of existing and developing new methods of X-ray stress measurement.

The dependence of stress tensor components σ_{ij} on a distance T from the surface may often be more important for the predicted strength of the product than only the surface values $\sigma_{ij}(0)$. The knowledge of the function $\sigma_{ij}(T)$ makes it possible to predict the performance and reliability of the product, otherwise or to choose a convenient surface treatment in order to create a layer of appropriate thickness with the compressive prestressing strong enough to make a barrier against the cracks expanding from the surface to the interior of the volume. Such layers make then a reliable protection against product damage even if cracks reaching certain sub-critical levels are noticed.

X-ray analysis of non-uniform residual stresses lends generally a new dimension to the description of the structure of polycrystalline materials, promising a better insight into the mechanism of plastic deformation.

Among others, the following residual stress non-homogeneities at the Czech Technical University in Prague were solved:

Non-homogeneities of residual stresses on machined surfaces of iron materials

Residual macroscopic stress distribution was investigated on three steel samples prepared by three different technologies. Samples A and B were wet-ground and dry-ground, respectively. Sample D was ground, polished and further subjected to shot peening.

The results of X-ray analysis show that grinding may give rise to surface non-homogeneities of residual stresses. Individual measurements performed at random points of samples under study do not offer a representative picture of the mean value of stress. This finding reflects one of the specific characteristics of the diffraction method of determining stress, namely the possibility of mapping the investigated surfaces "point-after point". At the same time it also suggests new applications of X-ray tensometry in sophisticated studies of state of residual stress of machine parts.

Finally, it is to be stated that to obtain an overall picture of the state of stress on the surface of metallic parts, the local, i.e. point, finding should be confronted with the results of diffractometric measurements covering larger areas subject to measurement on sample surfaces.

Residual stresses induced by laser heat-treatment on surface layers of pure iron samples and carbon steels of various types

On melted surfaces, the distribution of macroscopic stresses across the heat-treated regions may be affected not only by thermal effects and phase transformations, but also by the non-uniform distribution of energy within the laser beam cross-section. Tensile stresses alongside the hardened areas may give rise to cracklets normal to the beam shift. Markedly widened diffraction lines detected from the hardened area of the surface testify to the originating changes in the structure being analogous to changes going on in cold-treated metallic materials where the plastic deformation induces a reduction in grain size as well as is the origin of microscopic stresses.

Investigation of residual stresses in electron beam hardened steel samples

Electron beam hardening is a highly efficient procedure of surface modification. In this process, non-uniform thermal strains and high temperature gradients lead to the generation of typical residual stress conditions for the investigation of which X-ray tensometry is suitable. Such investigations were carried out in electron beam hardened

samples of the 50CrV4 steel grade.

Residual state of stress with variable components of the stress tensor on the surface of a unidirectionally ground sample of martensitic steel

The lattice strains ε were determined by means of CoK_α radiation and θ -goniometer on the surface of a unidirectionally ground sample of the martensitic steel. Experimental values of ε were determined in the direction of grinding. It was proved that the experimental values correspond to the hypothesis that the components σ_{11} , σ_{12} , σ_{22} , σ_{13} , σ_{33} of the stress tensor represent a linear function of distance T from material surface inwards; $\sigma_{23} = 0$.

Analysis of non-uniform stresses in Al-alloys by TiK_α , CrK_α and CuK_α radiation

The aim of the research was to obtain basic information on the distribution of residual stress resulting from turning, polishing after the turning and shot peening in the surface layers of Al-alloys. The results of the measurements performed entitle to make the following conclusions:

- The state of macroscopic residual stresses is non-uniform; the stress gradient in the turned and polished samples is of opposite sign than in the shot-peened sample.

- Polishing of the turned samples did not lead to significant changes of residual macroscopic stresses.

- In all cases, the shot peening residual stresses are exclusively compressive.

X-ray stress measurement, study of textures, and qualitative and quantitative phase analysis are the most important applications of X-ray diffraction methods in Czech and Slovak Republic. The general engineering public is regularly informed about the possibilities of diffraction methods. These non-destructive ways of material testing are advertised in journals concerned with research in machinery and metallurgy.

In 1988 and 1990, two monographs on X-ray stress measurement appeared in Czechoslovakia: 1. Kraus and V. V. Trofimov, *Rentgenová tenzometrie (X-ray Tensometry)*, Academia, Prague 1988; I. Kraus, *Rentgenografie nehomogenních napetových polí (X-ray Stress Analysis of Non-uniform Stress Fields)*, Academia, Prague 1990. Besides German and English, Czech thus becomes the third language in which a manual is available on the X-ray analysis of residual stresses.

N. Ganev, I. Kraus

Three Times about Crystals from the Heart of Europe Vox audita perit, littera scripta manet

The language of old scholars was Latin. That is why many valuable old anterior texts are studied very seldom nowadays. Only exceptional ideas of great personalities have escaped oblivion. Mankind comes back to them now and then to find with astonishment that we are discovering what was discovered a long time ago. In order to understand correctly the contents of the ideas which resisted in the contest of time and develop our understanding of the world a little further it is important not to waste the opportunity we are offered by our ancestors.

Around the year 1500, there still prevailed in Bohemia the image of a scholar as a humanist writing Latin as well as Czech, with a profound knowledge of antiquity and ability of its literary emulation. Hardly half a century later a remarkable change started. Growing interest was being concentrated on theoretical aspects and practical application of natural sciences. Main impulses for scientific research came from undertaking and production. Jáchymov's silver fever in the 1st half of the 16th century may be quoted as an example of this. (In the 20th century the Czech town of Jáchymov experienced another one, this time uranium fever. It was from that place that Marie Curie-Sklodowska had

pitch blende delivered for her experiments.) Even at those times work in natural sciences, overreaching in importance the border of a small country in the heart of Europe, was accomplished. Among the best known are the books written by Georgius Agricola (about 1490 - 1555), a medical doctor and natural scientist and founder of geological sciences which also gathered all the knowledge in mineralogy of that time. Agricola's work *De re metallica libri XII* (1556) kept its value as a thorough encyclopaedia of mining and metallurgy for several next centuries.

Agricola paid much attention to the properties of minerals like colour, density, transparency, lustre and shape. He described especially this last characteristic in a very clear way, so that even a simple miner may have recognized common minerals with his very own eyes. Most frequently occurring shapes were hexahedrons found in mountain quartz crystal, plates found in mica and cubes in pyrite and fluorite. At the same time, he described shapes presently called dendrites and whiskers. Apart from practical instructions for crystal identification, Agricola showed that crystalline shapes of a certain substance depend on the type of the place of extraction and conditions under which crystals had developed. In his work we can also find "technical instructions" on how to artificially produce some crystals (e.g. rock salt, crystal carbonate, cupric sulphate, ferrous sulphate or alums).

Nature prefers simplicity and unity

This was the lifelong motto of Johann Kepler (1571-1630), who is known to us mainly through his astronomical discoveries. He is less known as an excellent mathematician and remained rather unknown as the founder of the theory of crystals.

The history of scientific crystallography began sometime during Kepler's Prague stay in the services of Rudolph II. When walking through winter Prague he suddenly noticed the geometrically unique shape of snowflakes. The famous astronomer resumed his own observations and considerations on why all the flakes possess the same six point star shape in his dissertation "*Strena seu de nive sexangula*", or in translation "A New Year's present ;on hexagonal snow", which he dedicated as a New Year present , in January 1611, to his Prague friend, imperial diplomat Jan Matoušj Wacker of Wackenfels.

In connection with meditations on the reason of cross hexagonal symmetry of snowflakes Kepler reached important conclusions about possible ways of close-packed spheres. Only nowadays we realise how close he was at his time to the current definition of space lattices. The result of his observations, that no snowflake possesses 5 or 7 points, was later deduced theoretically as a result of lattice structure of crystals. From the Kepler's pamphlet we can also draw empirical information that the angles of crystal faces of specific substances are constant. Hexagonal snow is an important part of Kepler's search for harmony in the world. However, he was well aware that this is a task for all the generations of mankind to come:

"The treasure of secrets in nature is inexhaustible, its riches are undescribable. Whoever brings something new from it to the daylight, has achieved no more than showing the way to others for further investigation."

Nature is mighty

Since the first examinations of crystals, sometimes systematic and thorough, another time only accidental, a number of millennia have passed. However, the beauty of crystalline shapes fascinates us in the same way as it fascinated our ancestors. Since Laue's discovery, in 1912, we have been and we continue to be amazed by the order and variety of their inner structure. Would anybody else be

able to make a more beautiful declaration on this subject than the great Czech writer K. Capek in his *Letters from England*?

"There are crystals as big as cathedral pillars, delicate as mildew and sharp as needles; plain, blue, green like nothing in the world, of fiery colours or black; mathematical, perfect, like the contrivances of queer and bewildered sages; or recalling livers, hearts, gigantic human organs and animal fluids. There are crystalline caves or spectral bubbles or mineral dough; there is mineral fermenting, grilling, growth, architecture and engineering; I vow that a Gothic church is not the most complicated of crystals. Even within us there persists a crystalline power; even Egypt crystallized in pyramids and obelisks, Greece in pillars, Gothic in pinaches and London in cubes of black mud; countless laws of structure and composition pass through matter like secret mathematical lightning flashes. We must be exact, mathematical and geometrical, in order to be equal to nature. Number and fantasy, law and abundance are the feverish forces of nature; not sitting down beneath a green tree, but creating crystals and ideas denotes becoming as nature; creating laws and forms; penetrating matter with glowing flashes of divine computation. Ah, how scantily eccentric, how scantily daring and precise is poetry!"

The science of Crystals was founded in Bohemia more than 450 years ago. Now, near the end of the 20th century, the study of crystals is not only an occupation but also the very *raison d'être* for scholars and researchers in several dozens of laboratories in universities, academy and industry throughout this country in the heart of Europe.

I.Kraus

STATUS OF CPD PROJECTS

A book on Rietveld method

The CPD project to produce a book on the Rietveld method has just been completed. The multi-author book is titled "*The Rietveld Method*", is edited by R.A.Young, and is published by the Oxford University Press, Walton Street, Oxford OX2 6DP, England.

Advances in Powder Diffraction, Hangzhou, China

It is a satellite meeting of the XVIth IUCr Congress and General Assembly, Beijing, China, and will be held at the Shangri-La Conference Centre, Hangzhou, 31 August - 3 September 1993. The main aim of the meeting is to present recent developments in powder diffraction theory and practice and to emphasise the power of modern diffraction methods in materials science. The topics to be covered in the scientific programme include structural studies from powder data, instrumentation, standards and databases, non-ambient and time-resolved diffraction and studies of microstructure, thin films and surface layers.

J. Ian Langford, Programme Chairman

MEETING REPORTS

12-th Russian Conference on X-ray Diffraction

Analysis of Raw Materials, Sochi, March 23-27,1992

This conference was organized - like the 11 previous conferences of this series which took place on different spots of the late USSR - by the X-ray Raw-Materials Commission of the Mineralogical Society jointly with the Geological Institute of the Russian Academy of Sciences and the Sankt-Petersburg University. The meeting was sponsored by Science - Production Association "Burevestnik", Joint-Stock Company "AYAKS" (Sanct-Peterburg) and the Science-production Cooperative "KHROS" (Moscow), Chairman of the Organizing Committee was Prof. V.A. Frank- Kamenetsky. The scientific program of the conference addressed the following topics:

- crystallochemistry: new facts, systematization, crystal structure determination and refinements, modular structures;
- real structure analysis of minerals;
- layer silicates: crystallochemistry of octahedral cations, variations of structure of mica in different deposits, heterometry in micas, mechanisms of structure inheritance;
- instrumentation and automation of measurement, software systems for the estimation of thin structure, calculation of the "true" profile, displacement and area of diffraction peaks;
- data acquisition in X-ray powder diffractometry;
- Rietveld technique;
- phase identification and quantitative analysis;
- application of the X-ray powder diffraction to problems of genesis, technology and synthesis of crystalline materials, studies under various P-T-C conditions.

The volume of abstracts of conference papers contains 214 items, including abstracts of 12 plenary lectures. Some 70 participants from Russia, Belorussia, Ukraine, Georgia, Lithuania and Tatarstan attended the conference. This number is in contrast with the attendance of the previous conferences of the series which amounted to some 250 people.

Among the most remarkable contributions were the opening speech of the senior of Russian crystallographers, Prof. G. B. Bookii, the lecture "80 years of X-ray diffraction in geological sciences" delivered by Prof. V. A. Frank-Kamenetsky and the following papers:

S.K.Filatov, T.F.Semenova: Commencement of crystallochemistry combination in tetrahedra (OT4).

B.B.Zvyagin: Modular structures: theory and practice.

R.K.Rastsvetaeva et al.: The structure of Ba-Ti hollandite, a new natural Ca-borate, Ba-Mn titanosilicate type silicate - Yershovite

E.A.Goilov: Crystallochemistry of octahedral cations Fe, Mg, Al in layer silicates.

V.A.Liopo et al.: Variations of structure and composition of micas from different deposits. X-ray diffraction study of Belorussian ambers.

A.L.Salyn', V.A.Drits: A complex method of quantitative X-ray analysis.

Yu.Z.Chereiskaya et al.: An investigation of microstructure and phase-formation in wollastonite-bearing ceramics by method electron microscopy and x-ray powder diffraction.

E.K.Vasil'ev: Databases for qualitative X-ray phase analysis - a historical aspect.

A.A.Kashev & E.K.Vasil'ev, Irkutsk, Russia

First-night Regional Powder Diffraction Meeting in Czechoslovakia, Liptovský Mikuláš, September 29 - October 1, 1992

The 211th Seminar of the Czechoslovak Crystallographic Society in Liptovský Mikuláš, September 29 - October 1, 1992, was devoted almost exclusively to x-ray powder diffraction and its various applications in materials science and engineering. The program featured 29 lectures and 13 posters covering a wide spectrum of topics: size/strain analysis, residual stress measurements, solid solution studies, layer structures, martensite transformation, phase abundance analysis, electronic materials and ceramics. Among the most exciting were the lectures given by D.Havlicek (pitfalls in air pollutants locating and quantification), A.Buchal (open questions on ion nitriding of steels) and F.Hanic (new theory of high temperature superconductivity). A separate group of contributions dealt with computer programs in powder diffractometry. A palpable drift to practical applications was felt at the seminar, reflecting probably the new economic trends in the country. Another feature highlighting

this tendency was a lively interest of manufacturers of scientific equipment, of which Philips, Siemens, Inel, Minpepa (Prague) and High Screen (Liptovský Mikuláš) sponsored the meeting. The seminar was very successful thanks primarily to the endeavour of the chairmen of the program and organizing committees, T.Havlik and P.Sutta, as well as to the hospitality of the Technical Military College in Liptovský Mikuláš, where the meeting took place. In fact, this was the first powder diffraction meeting of such a big size run in Czechoslovakia and at the same time the best, and hopefully, the founder of a tradition of regional powder diffraction meetings in the district of both Czech and Slovak Republics.

J.Fiala

Rietveld Summer Schools in South America, December 1992

Two December 1992 Rietveld Summer Schools (RSS's) were organized by the CPD in cooperation with the local organizers. The first was in La Plata, Argentina on 8-10 December 1992 with Prof. Graciela Punte (National University of La Plata) as chair of the local organizing committee. The second was held in Sao Paulo, Brazil on 14-16 December with Professors Yvonne Mascarenhas and Lia Amaral (University of Sao Paulo at San Carlos and at Sao Paulo) in charge of the local organization. The main lecturers/teachers were the three professors who had the same roles in the previous two RSS's held in Poland, A.K.Cheetham, R.B. Von Dreele, and R.A.Young. Thirty students, out of 50 applicants, were accommodated for the La Plata school. In the Sao Paulo school, 42 students were accommodated. The students generally found their Rietveld analysis skills to be markedly improved at the end of the three days and so indicated. Through the host groups, the schools received financial sponsorship from CONICET (Argentina) and CNPQ (Brazil). The full travel expenses of the three lecturers from the northern hemisphere were funded by the Cooperative Science programs of the US NSF with CONICET and CNPQ. The Schools were also sponsored in name and underwritten by the IUCr through its Visiting Professor Program and Commission on Teaching. The underwriting by the IUCr was crucial; the schools could not have been held without it: The NSF funding actually came through only after all plans and local arrangements, including local financial commitments, had been made and travel tickets had been bought. A more extensive report may appear in the next CPD Newsletter, if there is one.

R. A. Young

XRD on Polycrystalline Materials, Pisa, December 1992

A meeting addressed to young researchers has been organized (Pisa 14-16 December 1992) by the Consorzio Pisa Ricerche and the Associazione Italiana di Cristallografia. The main topics discussed at the meeting were theory and measurement of diffraction and powder patterns interpretation. Lectures were held by C. Giacobozzo, G. Fagherazzi, G. Berti, P. Scardi, N.Masciocchi, J.W.Visser, M. Belotto and R.L.Snyder. A workshop on the use of the ICDD Powder Diffraction File was arranged with R.L.Snyder and J.W.Visser as teachers. Some 60 participants attended this successful and valuable meeting.

B. Giovanni

Computational Methods in X-ray Powder Diffraction Analysis

The 4th Egyptian International School and Workshop in Crystallography was held in Amon Village, near Aswan, 16-25 January 1993, and was devoted to computational

methods in powder diffraction. The School was organized by various Egyptian learned societies, under the auspices of the IUCr, and the main objectives were to inform scientists in the Middle East of the latest advances in powder diffraction and to give instruction in modern methods and practices. The school received financial support from the IUCr, the Ambassadors of France and the Netherlands in Cairo, the British Council and the German Academic Exchange Service (DAAD).

The scientific programme was arranged by a committee of the Commission on Powder Diffraction. The main topics were collecting data worthy of computer analysis, data reduction, phase identification, crystal structure and microstructure analysis, quantitative analysis, computer programs and non-crystalline materials. Plenary lectures were given by Prof. A. Authier and Prof. F. Ahmed. There were about 60 participants, the majority from Egypt and others from Saudi Arabia, India and the USA.

J. Ian Langford

"Modern Methods Techniques and Application of Single Crystal and Powder Diffraction", Prague, Czech Republic, 19-20 January, 1993

This meeting was organized by the Czechoslovak Crystallographical Society and kindly sponsored by the firm ENRAF-NONIUS, a well known producer of X-ray diffraction equipment.

Czechoslovak Crystallographical Society has some 500 members, 400 from Czech republic and 100 from Slovakia, respectively. There are also some members from abroad, mostly former emigres. It still remains the common society for both successive states of former Czechoslovakia. Its main activity is the organization of scientific exchange, mostly in form of one-day meetings, the so called "Talks on problems of the structure analysis". Every year, a week domestic or international conference is organized for some 100 participants, dealing with various topics. From time to time, a two-day meeting, like this one, in Prague in January 1993, is organized, too. Since the history of activities of the Czechoslovak Crystallographic Society, which survived successfully many political and economic changes, is rather long dating back to the early post-war years, the meeting in Prague in January was registered as the 214-th "Talks".

The seminar took place in the hotel POSISTA at the western outskirts of Prague. Almost 100 persons had returned application form and about 90 of them actually came. Despite of recent splitting of the common state, about 20 participants from Slovakia were also welcomed.

The first day was dedicated to the single crystal diffractometry while the program of the next day was of special interest for people working in the field of powder diffraction: texture correction (V. Valvoda), ENRAF-NONIUS diffractometer PDS120 (P.U. Pennartz), phase analysis (J. Fiala) and diffraction techniques in mineralogy (M. Rieder).

Jiri Hybler

Workshop in X-ray Powder Diffractometry

A workshop on modern X-ray powder diffractometry was held at The University of Zimbabwe, Harare, 25 to 31 March 1993. The course was organised and hosted by the University's Institute of Mining Research (IMR) and was sponsored by the Swedish Agency for Research Cooperation with Developing Countries (SAREC). There were 24 participants drawn from the University, local industry and government departments, with interests in geology, geophysics, metallurgy, mineralogy and soil science. The main objectives were to give instruction in

making optimal use of conventional powder diffractometers, for the collection of high quality data, and in modern methods for characterising materials by means of X-ray diffraction.

J. Ian Langford

NEWS FROM ICDD

The International Centre for Diffraction Data has awarded two Crystallography Scholarships for 1993:

Janet M. S. Skakle, University of Aberdeen, Scotland and Peter C. Burns, The University of Manitoba, Canada have been selected as the recipients by the ICDD Scholarship Award Selection Committee. Janet Skakle is pursuing a Ph.D. degree studying structures of a variety of barium rare-earth titanates using neutron and x-ray diffraction. Peter Burns is conducting graduate studies on the stereochemistry of Cu^{2+} oxysalt minerals.

Ludo Frevel

FUTURE MEETINGS

May 23-28, 1993: **American Crystallographic Association Meeting**, Albuquerque, New Mexico, USA. (Dr. A. Larson, LANSCE H805, Los Alamos National Laboratory, Los Alamos, NM 87455, USA; tel: 505-667-2942).

May 31-June 2, 1993: **International Conference for Applied Mineralogy**, Perth, Western Australia. (Secretary ICAM 93, Congress West, POB 1248, West Perth, Western Australia 6005, Australia; tel: 6 19 322 6909).

June 8-10, 1993: **Second Regional Czech and Slovak Powder Diffraction Conference**, Liptovský Mikuláš, Slovakia. (Dr. P. Šutta, Dept. of Physics, Military Technical University, 03119 Liptovský Mikuláš, Slovakia; tel: 42 0849 25241 ext. 2475).

July 18-26, 1993: **10th International Clay Conference**, Adelaide, Australia. (10th ICC Secretariat, Elliservice Convention Management, POB 753, Norwood, South Australia, Australia 5067).

Aug 2-6, 1993: **Annual Denver Conference on Applications of X-ray Analysis**, Denver, Colorado, USA. (L. Bonno, Conference Secretary, Dept. of Engineering, University of Denver, Denver, Colorado 80208, USA; tel: 303 871-3515).

Aug 21-29, 1993: **XVIth IUCr General Assembly and International Congress of Crystallography**, Beijing, China. (Prof. M.-C. Shao, Dept. of Chemistry, Institute of Physical Chemistry, Peking University, Beijing 100871, China).

Aug 31- Sep 3, 1993: **Satellite Meeting on Powder Diffraction**, Hangzhou, China. (Prof. R.-G. Ling, Central Laboratory, Hangzhou University, Hangzhou, China 310028; fax: 86-571-870107).

Sep 20-24, 1993: **Tenth International Conference on Textures of Materials**, Clausthal, Germany. (Institut für Metallkunde und Metallphysik der Technischen Universität Clausthal, ICOTOM 10, Grosser Bruch 23, D-3392 Clausthal-Zellerfeld, Germany).

Sep 21-24, 1993: **Annual Meeting of the Regional Czech and Slovak Crystallographic Society**, Gabčíkovo, Slovakia (Doc. M. Dunaj-Jurco, Dept. of Inorganic Chemistry, Faculty of Chemical Technology, Slovak Technical University, Radlinského 9, 81237 Bratislava, Slovakia).

Sep 25-28, 1993: **Third European Powder Diffraction Conference EPDIC-3**, Vienna, Austria. (Prof. A. Preisinger, Technische Universität Wien, Getreidemarkt 9/171, A-1060 Wien, Austria; tel: 431-58801-4749, fax: 431-568136, e-mail: el711dab@awiunill.edvz.univie.ac.at).

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If you would like to receive a personal copy of future issues of this Newsletter, please make sure that your name is on our mailing list by completing a copy of this form and mailing it to the CPD Secretary, Dr. R. J. Hill, at his address shown below. You may also use this form to notify us of a change of address, or to let us know of anyone else who might like to receive the Newsletter.

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This issue of the CPD Newsletter has been edited by J.Fiala with a considerable technical assistance of N.Ganev from Prague and a generous support of the Chairman of the CPD, Prof.R.A.Young, who let the issue printed in Atlanta. The next issue of the CPD Newsletter is to appear in the autumn of 1993 in case that it is not incorporated into the new general Newsletter of the IUCr. This 11th issue will be edited by L. Frevel. He would greatly appreciate contributions from readers on matters of interest to the powder diffraction community, such as meeting reports, notices of future meetings, developments in instrumentation, techniques and computer programs, and news of general interest. Please, send articles and suggestions directly to him at his address given below. Thank you.

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