L7

THE EXPLOITATION OF X-RAY DIFFRACTION IN CHARACTERISATION OF STRENGTH OF HOT-ROLLED AND COLD-DRAWN FERRITIC-PEARLITIC STEEL

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A series of samples of the C45 (0.45 wt.% C) steel was prepared by hot rolling with different thermomechanical history in an industrial-type rolling stage (e.g. rolling temperature and speed, cooling rate). The microstructure of resulting material was ferritic-pearlitic with a pearlite volume fraction ranging from 57 to 90%; the mean interlamellar spacing in the pearlite varied between 180 and 270 nm. An equally-spaced arrangement of misfit dislocations was found at the ferrite/cementite interfaces. The microstrain they generate was observed in the X-ray diffraction by means of anisotropic line broadening; the density of the dislocations was proved to be proportional to the density of the lamellas. The dislocation density was found to correlate with the ultimate tensile strength (UTS) of the steel in the tensile test as well as the density of the lamellas. Upon gradual cold drawing through conical dies, the dislocation density observed in X-ray increased up to about 50% of elongation, the UTS was still well-following its dependence on the dislocation density, while the density of pearlitic lamellas remained intact. The X-ray diffraction can thus be utilised for an instant and non-destructive estimation of UTS of hot-rolled ferritic-pearlitic steels and cold-drawn steels with moderate grade of cold deformation.

Extended contribution submitted.

L8

RÖNTGENOVÁ DIFRAKČNÁ ANALÝZA TROSIEK. EBSD ANALÝZA OBALOVÝCH PLECHOV

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Extended contribution to be submitted.

L9

NOVÉ MOŽNOSTI DIFRAKTOMETRU RIGAKU SMARTLAB

Jiří Maršík

Rigaku Innovative Technologies Europe

Lectures - Session III, Tuesday, June 21

L10

KVALITATIVNÍ A KVANTITATIVNÍ FÁZOVÁ ANALÝZA

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str. 81-106

L11

ZKUŠENOSTI S PRÁŠKOVOU MIKRODIFRAKCÍ VE FORENZNÍ PRAXI

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Fázová analýza je ve forenzní oblasti jednou ze základních úloh. Většina expertiz v kriminalistice se zabývá určováním, popisem a komparacemi prakticky libovolných látek, které mohou přijít do styku s osobami či předměty. V obecném případě se ve forenzní laboratoři lze setkat s materiály jak přírodního původu, tak i libovolnými hmotami vzniklými činností člověka a laboratoř by měla být schopná fázi určit. V tomto kontextu jsou samozřejmě velmi významné i možnosti rentgenových metod.

Samozřejmě ale ani XRD metody nejsou samospasitelné a jsou obvykle používány v kombinacích s dalšími metodami (zejména SEM-EDS/WDS, Ramanova mikrospektrometrie, optická mikroskopie, XRF, FTIR apod.).

Klasické aplikace XRD metod však narážejí ve forenzní oblasti na jeden základní problém, kterým je množství materiálu, resp. potřeba analyzovat přesně definované místo, jehož velikost je alespoň řádově srovnatelná s velikostí plochy analyzované dalšími metodami – především optickou a elektronovou mikroskopií a mikroanalýzou (EDS/WDS/mikroXRF). Řešení tohoto problému přináší, alespoň částečně, systém rentgenové práškové mikrodifrakce. V Kriminalistickém ústavu Praha (KUP) je využíván systém kombinace běžné rentgenky, kapiláry fokusující rentgenový svazek do průměru okolo 0,1 mm, a pozičně citlivého detektoru.
Extended contribution to be submitted.

L12

HIGH-BRILLIANCE LOW-MAINTANACE MICROFOCUS SOURCES FOR DIFFRACTOMETRY

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Modern microfocus X-ray sources define the state-of-the-art for a number of applications such as protein crystallography and small-angle scattering in the home lab. These sources have small anode spots of 50 µm or less. They are usually combined with two dimensional multilayer mirrors as beam-shaping devices that image the source spot onto the sample position, magnified to a suitable size, and deliver a parallel or focused monochromatic beam.

The Incoatec microfocus source IµS incorporates the optimized combination of an extremely bright and very durable stationary air-cooled microfocus source and the latest type of two-dimensional beam shaping multilayer Montel optics, the Quazar optics. The source is available with copper, molybdenum, chrome and silver radiation; optics for focusing or collimating the beam are available.

The IµS is usable for single crystal diffraction, small angle x-ray scattering, materials characterization, powder diffraction, and other applications as well. We will show latest applications of the IµS equipped with different two-dimensional beam shaping multilayer optics.

The applications and results presented here are showing the versatility of the IµS. The source can be used for all kinds of experiments and can easily be integrated in all kind of setups.

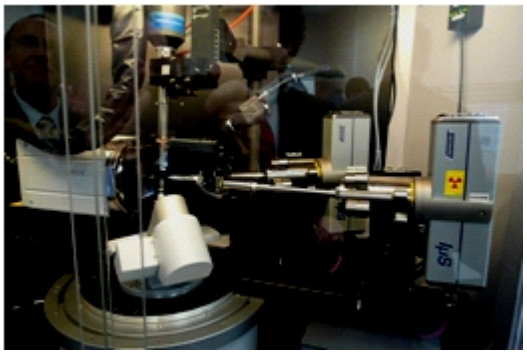

Figure 1. Bruker APEX DUO, equipped with Cu and Mo, IµS.

Figure 2. Upgrade of a Huber goniometer with a Ag , IµS.

Figure 3. Replacement of a Mo sealed tube with a Mo IµS on a Bruker AXS X8.

L13

HIGH-ENERGY MILLING INDUCED CALCITE TO ARAGONITE TRANSFORMATION IN EGGSHELL

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Eggshell biomaterial has been mechanically activated in a planetary mill Pulverisette 6 (Fritsch, Germany) in air for increasing time (15– 960 min.). X-ray diffraction study of the milling products has been carried out using a D8 Advance diffractometer (Bruker, Germany), the diffraction patterns were treated with the MAUD and Diffrac^{plus} Topas software.

The analysis has revealed an extensive and fast calcite-to-aragonite transformation, what is a polymorphous transformation of $CaCO_3$ from trigonal to orthorhombic crystal system. The process started after 15 min. of milling. The volume fraction of the aragonite phase reached maximum (~65%) after 240 min. of milling, the mechanochemical equilibrium between phases was established after 960 min.

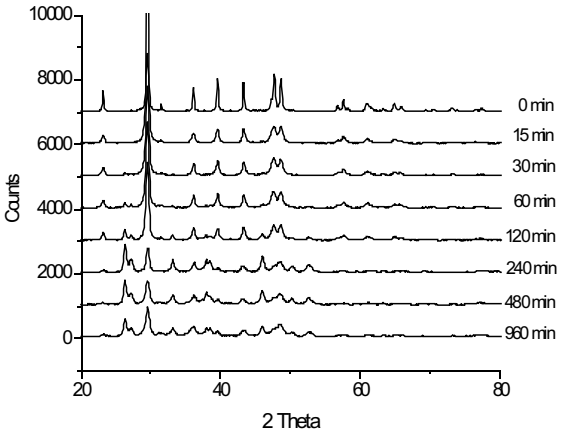

Figure 1. XRD spectra of high-energy milled eggshell. Duration of milling indicated in the graph.

The Slovak Grant Agency VEGA (project 2/0009/11), the Agency for Science and Development (project APVV-0189-10) and the project "Centre of Excellence of Advanced materials with Nano- and Submicron–Structure" ("nanoCEXmat") financed by the European Regional Development Fund are gratefully acknowledged. The SEM microphotographs have been kindly provided by the International Laser Center in Bratislava (J. Kováč, A. Šatka)

Extended contribution submitted..

L14

GROWTH OF SAPPHIRE PROFILES BY EFG METHOD AND THE USE IN STRUCTURAL ANALYSIS

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Single crystalline aluminum oxide - sapphire - belongs to one of the most important artificially produced material due to its unique physical and chemical properties. Sapphire has a high refractive index and a broad transmission band spanning the ultraviolet, visible and infrared bands. Sapphire is very hard material (Moh's 9) and has an extraordinary mechanical strength up to melting point 2053 °C, very good thermal conductivity, high electric resistivity and outstanding chemical resistance even to strong acids, bases and fluorination agents. All this makes sapphire a much sought-after material in industry and science.

Typically is sapphire grown as bulk crystal by Czochralski or Kyropoulos method. However crystal machining is difficult in comparison to other materials due to its high hardness. First growth of profiled single crystal sapphire was published in 1971 by La Belle and Mlavsky (TYCO laboratories) [1]. EFG (Edge-defined Film-fed Growth) method is in principle similar to older one (1953) of A. V. Stepanov for growth of shaped single crystals of metals [2]. Review of modern modifications of EFG and related methods was published [3][4]. Main advantages of EFG method are growth of near net shape decreasing pro-

Table 1. Absorption coefficients and transmittance at important K lines

	K (keV)	μ/ (cm^2/g^{-1})	d (cm)	
Cu	8.04	3.24E+01	0.10	0.0%
Mo	17.441	3.09E+00	0.10	29.2%
W	58.856	2.46E-01	0.10	90.7%

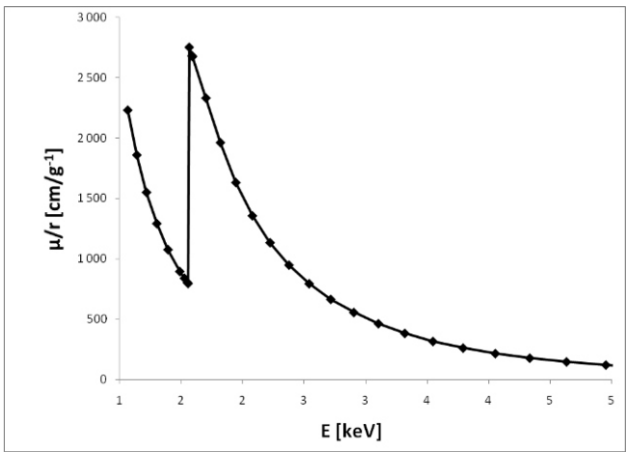

Figure 1. Absorption coefficients of Al_2O_3 - sapphire in dependence of radiation energy.

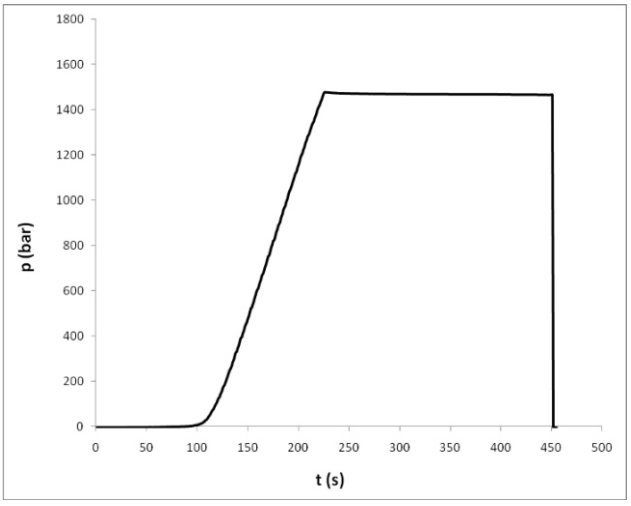

Figure 2. Pressure test of sapphire capillary OD/ID 1.0/0.6 mm, tube burst at 1550 bar after 7.5 min.

duction costs, possibility of growth of complicated shapes and large scale profiles, faster growth rate and also automated process control.

Crytur produces sapphire single crystals, rods, ribbons, tubes and fibers since 1978 and is the only EU manufacturer of the EFG sapphire today.

Sapphire tubes are used in various industrial and scientific applications demanding extraordinary material properties. High pressure, high temperature, together with harsh chemicals are the right reason to use sapphire tube instead of quartz or corundum ceramics. Tubes produced by EFG method can be designed to according to demands of specific applications like UV/VIS/IR measurements, nuclear magnetic (NMR) or X-ray (XRS) spectroscopy.

High pressure MNR tubes have to take hold of enough volume and typical outside diameter is 10 or 5 mm and wall thickness 1 - 1.5 mm. Pressures used during measurements are in range 10-150 MPa, the highest pressure require tubes with special collar design and post growth tube annealing [5].

In XRS high pressure XRS capillaries are used. Outside diameter of such a capillary is typically 1.57 mm (1/16" compatible with laboratory equipment) or 1.0 mm and wall thickness 0.2 - 0.4 mm are used. Crucial parameter for XRS is absorption coefficient of sapphire for K lines of X-ray sources.

The maximum pressure p_{max} is a function of the tensile strength of the material and the quotient of the outer and inner diameters d_o and d_i. According to [5] relation can be approximately expressed as

$$p_{\max} \quad \ln(d_0^2 / d_i^2) \qquad (1)$$

Due to possible presence of material defects like bubbles or material stress in sapphire cell the maximum operating pressure has to be verified by internal pressure test. Some tubes show time dependent pressure threshold, this process will be further studied. Also pressure test at elevated temperature will be important to set conditions which sapphire cell can withstand.

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My great acknowledgement to our colleagues M. Steinhart (University of Pardubice) and W. Kremer (University of Regensburg) for pressure tests of XRS and NMR sapphire cells.

L15

PRODUKTY FIRMY BRUKER

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Scientific Instruments Brno