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

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The X-ray laboratory has been established as part of the Metallography Department in the Research and Testing Institute VSŽ (East Slovak Steekworks) since 1971. The first X-ray diffraction equipment was Siemens Kristaloflex IV with two Bragg-Brentano and Schultz texture goniometers. The originally manual equipment was step by step fully automated in the early nineties. The detector was connected through interface to computer for data collection and the analog motors were replaced by step motion. Special gear was added to textural goniometer for theta motion; other gear units were functional and could be used for fully automated regime. The original special analog recorder drawing pole pictures was replaced by a computer programs. Evaluation procedure of measured data was changed from paper record to software mode. From information provided by Prof. Jaroslav Fiala, VSŽ was the first to used PDF2 diffraction database in Czechoslovakia. It was installed in the Hot strip mill Computer center. In the nineties the fully automated X-ray equipment was gradually completed by purchasing the (ZDS, RIFRAN, BearTex) software and by free available software like popLA, GSAS, etc. 1 .

After the plant acquisition by U. S. Steel, the laboratory was refurbished by new Seifert XRD 3003 PTS equipment with 3D goniometer in the late 2003. The equipment allows measuring and analyzing also the texture and residual stresses in rolled steel sheets. The equipment was upgraded by XY positional stage and highly sensitive PSD Meteor1D type detector, which reduced the measurement time from about 10 hours to 25 minutes. The point focus is used for textural and residual stresses analyses and line focus for phase analysis. X-ray analyzer is equipped by Rayflex software, which does not cover all analyzed areas. MulTex and LaboTex systems were purchased for orientation distribution function (ODF) calculations. The popLA mode is still used for the textural analysis, since 2006 the EBSD analyzer is in operation too. Rayflex – residual

stress program is sufficient for residual stress analyses by use of  $\sin^2$  method. The ZDS search match software with PDF2 2004 diffraction database is used for qualitative and quantitative phase analysis. Refinement of diffraction records is performed by AutoQuant program, which uses the Rietveld method. For more complex analyses, which are solved in cooperation with Institutes of SAS, TU and UPJŠ Universities in Košice, programs GSAS 2 , FullProf 3 a MAUD are used 4 . The structural data can be obtained also on the website COD – Crystallography Open Database 5 or AMCSD – American Mineralogist Crystal Structure Database 6 .

Measured and evaluated diffraction records are systematically processed in the database which provides a great amount of valuable information. According to the keywords, it can be easily identified which analyses are most often required and resolved. Besides to rolled steel sheets also coatings, ceramics, sediments, dusts, sludges, different slags, casting powders, entry ores, etc. are analyzed. The database, which is the enlarged electronic diary, contains approximately 1300 samples records with approximately 260 identified minerals. The stored solutions data are valuable information used with new analyses.

Examples of selected phase analysis, residual stress and texture determinations performed in metallurgical operation laboratory are included to this contribution.

1. D. K. Smith, S. Gorter, *J. Appl. Cryst.*, 24 (1991) 369-402.
2. <http://www.ncnr.nist.gov/programs/crystallography/software/downloads.html>.
3. <http://www.ill.eu/sites/fullprof/php/downloads.html>.
4. <http://www.ing.unitn.it/~maud/index.html>.
5. <http://www.crystallography.net>.
6. <http://ruff.geo.arizona.edu/AMS/amcsd.php>.