pseudooctahedral coordination of the Co(III) atom is completed by one phenolato O and one amidic N atom of the same arm of the bridging *o-van-en* ligand. In addition, the asymmetric units of both polymorphs contain two acetonitrile solvate molecules. In polymorph **2**, consistent with the symmetry of the space group, the dinuclear $\{Co_2\}$ units are arranged in an alternating ABABAB fashion, in contrast to the AAA arrangement of the dinuclear units in polymorph **1**. As a consequence, both polymorphs differ in the positions of the acetonitrile solvate molecules and in the pattern of intermolecular interactions. Differences of some geometrical parameters, *e.g.,* torsion angles were observed, too.

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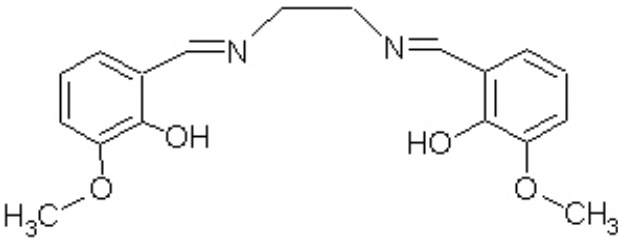

Scheme 1. Structural diagram of H_2(*o-van-en*).

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Session VIII, Thursday, September 15

L20

ELECTRON BACKSCATTER DIFFRACTION - EBSD

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The electron backscatter diffraction (EBSD) is the method widely used in materials science, nowadays. In past twenty years the instrumentation underwent extensive progress and the EBSD became standard laboratory technique. Significant progress in the instrumentation, the development of modern high resolution scanning electron microscopes and dual beam microscopes, as well as the fast EBSD detectors still offers the new possibilities of application of EBSD method for studying of various types of modern and perspective materials.

The Kikuchi pattern formation in Transmission Electron Microscope (TEM) was first observed and explained in 1928 by S. Kikuchi [1]. It was immediately found that the Kikuchi pattern is a powerful tool for the crystal orientation determination because "The Kikuchi diffraction pattern is a projection of the geometry of the crystal lattice from a volume of specimen in which this geometry is constant, or nearly so (Kikuchi [1])." In 1932 Meibon and Rupp observed high angle Kikuchi patterns from "reflected" electrons. Venables and Harland observed electron backscatter patterns in the Scanning Electron Microscope equipped with 30 mm diameter fluorescent imaging screen and television camera. This method allowed examination of specimens and the measurement of crystal orientation at high spatial resolution, which was significantly improved by the use of field emission gun scanning electron microscopes. The first on-line working (automated) EBSD systems were developed in 1980. In 1993 the Orientation Imaging Microscopy (OIM) or orientation mapping was established. The on-line orientation determination from the EBSD patterns is computationally time-consuming task, however within the last decades the EBSD technique underwent a great boom as a consequence of the computers hardware improvements and progresses in the scanning electron microscopes technique, as well.

The EBSD is surface sensitive method. Measured information come from the depth of several tenths of nm, depending on the measured material atomic number (the penetration depth of electrons decreases with increasing atomic number). The spatial resolution of the EBSD depends on the used electron microscope type (used electron source). In the case of scanning electron microscope equipped with field emission cathode it is in order of ~10 nm.

Two types of information are essentially held by the electron backscatter patterns. First is the Kikuchi pattern quality measure and the second is the orientation of irradiated volume. The Kikuchi pattern quality information, can be used for determination of the crystal "perfection", estimation of the crystal defects types and its densities because the presence of the lattice defects in irradiated volume has in general in consequence decrease of the Kikuchi pattern "sharpness" (blurring of the Kikuchi pattern). However, the Kikuchi pattern quality is strongly influenced by the specimen surface preparation. The surface area is in most cases of samples highly defective (from production or sample processing). The Kikuchi pattern from poorly prepared specimen is therefore not sharp and this effect correlates with influence of the lattice defects and imperfections. Therefore, the determination of the lattice defects and densities can be done only quasi-quantitatively.

More interesting information about the investigated specimen are provided by the orientation of each infinitesimal measured sample volume which can be calculated from respective Kikuchi pattern. From the orientation map we can obtain information on the specimen morphology, grains and sub-grains shapes and grain and sub-grain size distributions. Measured orientation information allows us to calculate the misorientation [2] between different measured points and to describe the properties and the character of grain boundaries (GBs), to quantify fractions of high/low angle grain boundaries, observe and investigate occurrence of special grain boundaries (for instance CSL grain boundaries).

Orientation information yields the details on the preferred orientation of crystallites, where we are not restricted to the measurements of distribution of one (or several) lattice planes in different direction in sample, which is the case of texture measurements using the X-ray diffraction, but we simply determine the distribution of all possible crystal orientations in given direction in sample.

Measured orientation data can be used for calculation of the orientation distribution function (ODF) or misorientation distribution function (MODF). In specimen containing more phases, quantitative (volume averaged) phase analysis can be done, and above described details can be constructed for each individual phase present in sample. Moreover, we can investigate mutual orientation dependences between different phases in the specimen.

The possibilities and advantages of several different program packages for measurement, data processing and evaluation will be discussed in the talk. The particular EBSD results will be demonstrated for various type of investigated materials – severally plastically deformed (SPD) materials, hardmetals, cermets, ceramics, thin coatings, single crystalline samples and epitaxial thin films.

This work was supported by the Grant Agency of the Czech Republic under Grant No. 14-18392S.

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L21

TEXTURAL AND MICROSTRUCTURAL ANALYSIS BY EBSD METHOD IN THE USSK CONDITIONS

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The analysis of rolled steel plates is significant part of the metallographic analyses. Since the 1971 up to 2006 the textures have been analyzed only by methods of X-ray diffraction (XRD) using XRD device with texture goniometer. Important knowledge extension about the textures occurred with application of the diffraction methods of back-scattered electrons EBSD. This method offers possibility to analyze the sample not only from the texture point of view, but also from the microstructure point of view (grain size, high and low angle grain boundaries, grain misorientation, twins).

Database, which includes cold and hot rolled sheets, continual and batch annealed samples, double reduced samples, was created by analyzing many samples (ca. 2000) of steel plates. From the material point of view these are mainly samples of tinplates, dynamo sheets, automotive sheets, deep-drawn, IF plates. Except of these materials also plates from austenitic steels, plates with residual austenite in TRIP steels were also analyzed.

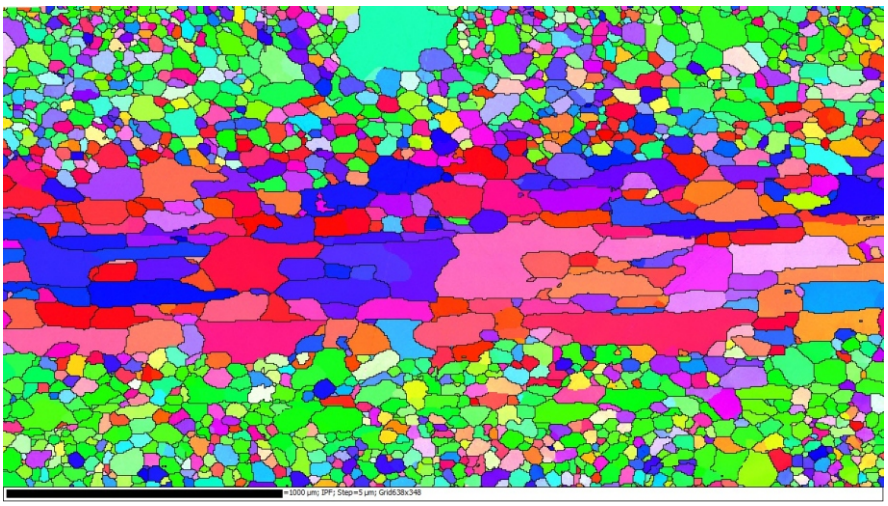

Figure 1. IPF map of hot rolled non-oriented steel sheet.

New microscope with EBSD camera with the speed of nearly 1000 frames per second for the perfectly prepared sample was purchased last year. The older camera reached only 40 frames per second at the same quality of the measurement. The advantage of the new system is also a possibility of continual sample scanning (large area mapping). Thanks to this property it is possible to measure samples with big grains, ca. 300 microns. Samples with such structure are unable to be measured via classic XRD device, as there is insufficient amount of grains for orientation distribution function (ODF) calculations on the measured area. These samples are only possible to be measured by neutron radiation, as the sample volume and penetration of neutron radiation is dimensionally in cm.

Powerful software for the processing of the measured EBSD data allows processing of large files of measured data ca. 5 millions, the ODF calculation, texture fibers, inverse pole figure map, grain size distribution, misorientation, grain boundaries, twins etc. The main problem of this method is sample preparation. For common ferritic steels the standard metallographic procedure is enough – grinding, polishing on diamond paste and final polishing in vibration polisher in colloid solution with nano particles of ca. 40 – 50 nm size. Such procedure is however insufficient for samples containing austenite. At our department the procedure with finishing by electrolytical polishing was approved as sufficient. The best results were obtained for cooling the electrolyte to ca. 0 – 10°C, voltage of ca. 10 V in the perchloric acid and ethanol in the 1:10 ratio.

An example of dynamo sheet with coarse-grained structure, which was measured by sequential scanning of the pictures through the whole sample area, is given in Fig. 1. The dimensions of the sample are approximately 25 mm of length and 2 mm of thickness. The results are comparable to the results measured using the neutron radiation.

SL13

VYUŽITÍ EBSD PŘI STUDIU KOVŮ S TVAROVOU PAMĚTÍ

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Metoda „difrakce zpětně odražených elektronů“, letos osmaosmdesátiletá, ukázala se býti vzorovou ukázkou bonmotu stárnoucího Západu, že život začíná po padesátce. Celkem bizarní příklad úhlové závislosti odražených elektronů, považovaný ještě ve druhé polovině devadesátých let za rozmazanou sestru kanálovacích obrazců koopcí špionážní Houghovy transformace v roce 1992 a malého interakčního objemu elektronů s látkou zesílil v univerzální a neočekávaně robustní metodu, která se stala běžnou výbavou analytických SEM a standardem pro určování orientace mříže a věcí s tím souvisejících.

Prezentované aplikace metody EBSD ve studiu kovů s tvarovou pamětí budou – doufejme – rozšířením přehledových přednášek na dané téma. Vcelku komplikovaná 3D-EBSD je vázaná na kombinaci EBSD detektoru s fokusovaným iontovým svazkem. Poskytuje však komplexní informaci o distribuci zrn v materiálu. Použití bude prezentováno na nedávno publikovaných výsledcích ze superelastických NiTi drátů.

Podstatně obecnější je téma přípravy vzorků. Na slitině Co-Ni-Al jsme provedli studii vlivu použitých leštících substancí na kvalitu výsledných Kikuchiho obrazců, která sice nepřináší průlom do metalografické metodologie, ale ozřejmuje některé fráze z příruček o přípravě vzorků.

SL14

PŘÍPRAVA A CHARAKTERIZACE Ni_2MnGa DOPOVANÉHO INDIEM

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Slitiny s tvarovou pamětí patří mezi Heuslerovy slitiny vykazující řadu zajímavých elektronických a magnetických vlastností, které podstatně závisí na krystalové struktuře. Magnetická tvarová pamě a magneticky indukovaná reorientace (MIR) jsou vlastnosti, které umožňují snadné namáhání těchto materiálů v magnetických polích < 1 T. Klíčovým pojmem k pochopení těchto efektů je martenzitická transformace a její studie je tedy nezbytná pro budoucí aplikace těchto slitin.

Ni_2MnGa je dobrý modelový systém, ve kterém lze výše zmíněné efekty dobře pozorovat. Navíc, odlišné chování materiálu může být dosaženo také díky nestechiometrické kompozici vzhledem k výchozí 2:1:1 a to vše může být navíc doprovázeno vznikem modulované struktury. Oproti tomu, další z Heuslerových slitin, Ni_2MnIn, nemusí vykazovat martenzitickou transformaci. Studie vzorků dopovaných indiem namísto Ga bude tedy klíčovým přínosem k pochopení efektů spojených s martenzitickou transformací.

SL15

STRUCTURES OF Sr-PHOSPHONATE LAYERS INTERCALATED BY ALKANEDIOLES SOLVED BY MOLECULAR SIMULATIONS

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Phosphonate layers represent group of compounds playing an important role in the design of new two-dimensional inorganic-organic hybrid materials. The usability of the layered phosphonates can be increased by intercalation of various organic species. We will show influence of intercalated 1,2 alkanediols [1] and 1,n alkanediols [2] on structural changes of Sr-phenylphosphonate layers. Intercalates are analysed by experimental techniques like X-ray diffraction, thermogravimetry, chemical analysis in combination with molecular modelling methods. Synthesized strontium phenylphosphonate intercalates with 1,2- and 1,n-diols (from 1,2-ethanediol to 1,2-hexanediol/ 1,n octanediol) show very good stability at ambient conditions. Calculated and experimental basal spacing are in very good agreement for both types of intercalates.

Models for 1,2 alkanediols suppose creation of cavities surrounded by six benzene rings. Every cavity contains one water molecule and one molecule of the diol for the 1,2-ethandiol to 1,2-butanediol intercalates. Two types of cavities are calculated for 1,2-pentanediol one with water molecule and another one with two water molecules. In the case 1,2-hexanediol intercalate two types of cavities are calculated containing one or two diol molecules. This variability causes more disordered structural models with longer alkyl chains with respect to models with shorter alkyl chains.

Another presented structure models are strontium phenylphosphonates intercalated with 1,n diols. Simulations show how the molecule conformation can be influenced by a shape of the host layer. On the base of experimental and calculation results, diols can be divided for two sets 1,2-to 1,4- diols and 1,6- to 1,8-diols. Diols with shorter alkyl chains are immersed between benzene groups and both OH groups are bonded to one host layer. Longer alkyl chains allow connection between two neighbouring layers to form nearly pillared structure in the interlayer and they are bonded by hydrogen bonds through water molecules. 1,5-diol was not prepared experimentally and simulations showed that the reason of this instability is the length of the alkyl chain.

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L22

STUDY OF ORIENTED FILMS OF HEXAGONAL FERRITES GROWN BY CHEMICAL SOLUTION DEPOSITION METHOD

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Hexagonal ferrites were discovered more than 60 years ago but only quite recently two effects were found - magnetoelectric (ME) effect and spin Seebeck effect. There is a need for preparation of epitaxial or strongly oriented thin films. This is possible even in the case the thin film and substrate have different structures or large mismatch in the lattice parameters. Then the strong orientation can be achieved if a polycrystalline film is deposited first and then heated to break up into isolated grains (seeds). This is followed by deposition of thicker layer when the isolated islands can act as nucleation centers for the growth of a highly oriented film.

In this work, thin films of $Ba_2Zn_2Fe_{12}O_{22}$ (Y) ferrite were prepared through the chemical solution deposition method using $SrTiO_3$(111) (STO) substrates and the above two-step process. As seeding layers two types of materials were used - oriented films with hexagonal symmetry in layer of O^{2-}: cubic 111-oriented cobaltates, namely $CoFe_2O_4$ and hexagonal 00l-oriented M-ferrites. The former were prepared through the transformation of 00l-oriented layered cobaltates $Na(CoFe)O_2$ [1] and for the choice of the latter a set of M-ferrites with different lattice mismatch with respect to the STO substrate and also to the Y-ferrite top films, different chemical composition and different magnetic properties were tested. The best orientation of this Y-ferrite film achieved for hexagonal M-seed layer with composition $(BaSr)(GaAl)_{12}O_{19}$, $Ba(FeAl)_{12}O_{19}$ [2].

The samples were studied by X-ray diffraction (XRD), electron back-scatter diffraction (EBSD) and atomic force microscopy. XRD analysis was performed in both conventional Bragg-Brentano symmetric setup as well as in parallel beam with the Eulerian cradle. In order to find appropriate reference value of lattice parameters of different used seed layers, some data analysis of PDF-4 database had to be performed with respect to the statistics and/or also to the stoichiometry. Structural studies were focused to investigation of preferred orientation by symmetric and asymmetric XRD scans and by EBSD and also to the analysis of possible residual stresses.

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