examples of application of the methodology will be presented as well.

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Keywords: structure determination, texture, Rietveld method

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New Tetragonal Phase in Al-Fe-U System

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Ternary aluminides in the Al-rich region of U-Fe-Al system have received considerable attention due to their interesting magnetic properties. Most of the structural studies of these materials were concentrated on UFe_xAl_{12-x} ($4 \le x \le 6$) compounds of $ThMn_{12}$ type described by the *I4/mmm* space group [1]. Recently it was reported about another type of a ternary intermetallic phase with a composition UFe_2Al_{10} that could be formed in the U-Fe-Al system. Its structure was determined as orthorhombic, belonging to the space group *Cmcm* [2]. In the present work we report our results on structural characterization of a new ternary aluminide observed in Al-rich corner of U-Fe-Al system. Its approximate composition is Al-4.2at%Fe-8.5at%U suggesting the provisional stoichiometry of U_2FeAl_{20}.

Using transmission electron microscopy and electron microdiffraction technique [3] the structure of the new phase was established as tetragonal with the unit cell parameters a=12.41Å and c=10.30Å. The space group describing the structure is $I\bar{4}2m$. The atomic model of the structure was developed using direct methods applied to the data taken from X-ray powder diffraction. The reliability factors characterizing the Rietveld refinement are: Rp=12.9%, Rwp= 15.5%, Rbragg=7.07% and Rf=3.7%.

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Magnetic Structure of $BaFe_{12}O_{19}$ Determined by Resonant X-ray Magnetic Scattering

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Resonant X-ray magnetic scattering (RXMS) has attracted much interest as a useful tool to determine the magnetic structures associated with specific electronic states such as 3*d*-4*p* interactions. The resonant enhancement in the Bragg intensity between charge and magnetic scatterings [1] makes it possible for a tiny single-crystal to give a complete determination of the crystal structure and spin arrangement. M-type $BaFe_{12}O_{19}$ has been examined in this study, because there are five independent Fe sites in a hexagonal-ferrite structure, which are tetrahedral 4f_1, bipyramidal 2*b*, and octahedral 2*a*, 4f_2 and 12*k* sites.

RXMS experiments were performed at the Fe *K* absorption edge at BL-3A, Photon Factory. Diffraction profiles for more than 30 reflections of a single crystal of 0.07 mm in diameter were measured with right- and left-circularly polarized X-rays, which were produced passing through a diamond (001) phase retarder. The magnetic anomalous scattering factors were estimated in the structure-refinement procedure. The observed asymmetry ratios were in agreement with those made for the most appropriate spin-orientation.

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Neutron Diffraction Analysis of Photoisomerization of δ-Cyanobutyl Cobaloxime

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δ-Cyanobutyl groups in cobaloxime complexes were known to be isomerized to γ-, β-, α-cyanobutyl groups successively on exposure to visible light with retention of single crystal form. However, in case of the photoisomerization of δ-cyanobutyl cobaloxime with bulky diphenylborone in the neighborhood of alkyl group, Ph_2B-cobaloxime, only α-form was observed by X-rays as photoproduct and FT-IR experiments showed that β-form was produced with decreasing α-form and gradually increased, *i.e.*, δ→α→β reaction path not the "slide type" reaction δ→γ→β→α previously observed. This study was carried out to elucidate the detailed reaction mechanism by tracking the transfer of replaced two deuterium atoms. We prepared a large single crystal of δ-cyanobutyl-*2,2-d_2* Ph_2B-cobaloxime complex and carried out a single crystal neutron diffraction measurement after 2 days irradiation with BIX-3 diffractometer. The final R factor was 16.36 %. The result showed that a half of the δ-form was isomerized to α-form and one of the two deuterium atoms bonded to α position of δ-cyanobutyl group was transferred to δ position, *i.e.*, from Co–$CH_2CH_2CH_2C\mathbf{D}_2CN$ to Co–$C\mathbf{D}(CN)CH_2CH_2CH_2\mathbf{D}$. This result indicated the cyanobutyl group turned upside down after homolytic cleavage of Co-C bond and directly isomerized from δ-form to α-form.

Keywords: neutron diffraction, hydrogen transfer, crystalline state reactions

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***Ab-intio* Structure Determination of a Metal Complex from Laboratory X-ray Powder Data**

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The crystal structure of Praseodymium tartrate, $Pr(C_4H_4O_6)_2.H_2O$, has been determined *ab initio* from laboratory X-ray powder diffraction data. Diffraction data were collected using monochromatized Cu Kα1 radiation (λ=1.5406Å) in the 2θ range 7.00-86.26º with a step width 0.02º and Bruker D8 Advance X-ray powder diffractometer. Indexing of the pattern was carried out using the program *NTREOR*. The solution with highest figure of merit [M_{20}=47.0, F_{20}=100.0] in the orthorhombic system having a=21.98(4), b=7.59(3), c=5.96(3) Å, agreed with the possible solutions obtained from the *DICVOL91* and *TREOR90* programs. Fitting the pattern with different possible space groups and a pseudo-Voigt function for the peak shape, the integrated intensities of 498 reflections were extracted based on the best fitted space group $P2_12_12_1$ using the program *TOPAS*. The position of the heavy atom was found from the Patterson map calculations. Attempts to build the structure with successive Fourier method were not successful. Finally the direct space approach using the program *FOX* with different input options led to a model structure containing one Praseodymium, two tartrate ions and one water molecule in the asymmetric unit. Rietveld refinement of the model structure with restraints using the program *EXPO2004* converged to Rp = 0.088, Rwp = 0.113, Re = 0.074 and GoF = 1.543, respectively. The final structure reveals that the Praseodymium atom is coordinated by nine O atoms of the ligands lying in the range 2.30 to 2.95 Å.

Keywords: ab-initio powder structure determination, direct space method, Rietveld refinement