Crystal phase analysis of by-products from NaBH₄ production via high-low pressure process by XRD

Muge Sarı, Aysel Kanturk, Ozgul Dere, Eren Halit Figen, Sabriye Piskin
Yıldız Technical University, Chemical Engineering Department, Davutpaşa Campus, Chemical Engineering Department, Esenler, Istanbul, 34210, Turkey, E-mail: mugesari@yildiz.edu.tr

Sodium borohydride (NaBH₄) is a safe and practical way of storing hydrogen due to its high hydrogen capacity (10.6 wt%). NaBH₄ is synthesized from different boron minerals by the thermal-chemical reactions. Constitution of by-product is little demand in NaBH₄ production process, because of requirements of its reuse or disposal issues. It is important to know the mineralogical properties of by-product for its disposal or reusing. In our study we investigated of characterization of by-products which were obtained from NaBH₄ production is based on the conversion reaction of borosilicate glass under high and low hydrogen pressure process (HPP-LPP) by XRD analysis. The XRD analysis was carried out at an ambient temperature by using Philips Panalytical X'Pert-Pro diffractometer in a range of diffraction angle from 10° to 90° with CuKα radiation λ=0.15418 nm at operating parameters of 40 mA and 45 kV with step size 0.02° and speed of 1°/min. According to the X-ray powder diffraction data, four crystal phases, Na₂SiO₃ (PDF Number: 00-016-0818), Na₂SiO₃·5H₂O (PDF Number: 00-003-0433), Na₃SiO₆·6H₂O (PDF Number: 00-018-1246) and Na₃Si(SiO₃)₂ (PDF Number: 01-088-1229) were defined for each by-products HPP and LPP obtained via HPP and LPP, respectively. By comparison XRD results of by-products were obtained under high and low hydrogen pressure processes, there are same crystallographic properties have been detected.

Keywords: X-ray diffractometers, crystal phases, inorganic compounds

A comparative study of two multiphasic alkal halide crystals: Quinary vs.exenary

Ricardo Rodríguez-Mijangos¹, Cordero-Borboa Adolfo²
¹Universidad de Sonora, Departamento de Investigacion en Fisica, P O Box 5-88, Hermosillo, Sonora, 83190, Mexico, ²Instituto de Fisica, Universidad Nacional Autonoma de Mexico, P O Box 20-364, Mexico D f 01000, Mexico, E-mail: mijangos@cajeme.cifus.uson.mx

A comparative study of two mixed crystals grown by a Czochralsky technique is realized: a quinary and a exenary. The first one is made by a melt of five salts: KBr, RbCl, RbBr, KI and RbI, mixed in equal molar composition and the second one made with the same salts, but adding KCl, being the six salts in equal molar composition. The X ray diffractometry technique, determine for the quinary three phases with NaCl structure type: a single, a binary and a ternary, each one with a lattice constant well defined, in the exenary determine two phases, also with NaCl type: a binary and a quaternary , each one with a lattice constant well defined. By using equations of mass balance is obtained for each phase the concentrations of the components in molar fraction in the quinary and exenary crystals. Is remarkable the KCl play role simplifying the phase number from three to two when increase the components number from five to six. A qualitative analysis is done explain this phenomena.

Keywords: inorganic materials, growth crystal, composition and structure of materials and alloys

From dimeric tantalopentatungstate to monomeric organosilyl Lindqvist type polyoxometalates

Mongi Debbabi¹, Fatma Bannani², Rene Thouvenot²
¹University of Monastir, ENIM, Monastir, Monastir, 5019, Tunisia, ²CM2, case courrier 42 University Pierre et Marie Curie, 4 place Jussieu, Paris cedex 05, E-mail: dmongi2@yahoo.it

Polyoxometalates (POMs) are a well-known class of inorganic metal-oxygen clusters with an unmatched structural variety combined with a multitude of properties. The search for novel POMs is predominantly driven by exciting catalytic, medicinal, material science and bioscience applications. However, the mechanism of action of most polyoxoanions is not selective towards a specific target. In order to improve selectivity it appears highly
desirable to attach organic functionalities covalently to the surface of polyoxoanions. As part of a broad program centered on the functionalization of polyoxometalates, we have been interested in the derivatisation of Lindqvist type polyoxoanions with organosilyl moieties. The condensed polyoxometalate (nBu4N)4[(TaW5O18)2O] which is synthesized by reacting [TaW10O40]6− with BuSnCl5, crystallises in the orthorhombic space group Pbna with lattice parameters a = 15.7981(14), b = 17.939(3), c = 35.216(6) Å, V = 9980 Å3 and Z = 4. The crystallographic study of (nBu4N)4[(TaW10O39)O] shows that the dimer is composed from two polyoxoanion fragments linked by linear Ta-O-Ta bridge. Such a linkage readily reacts with organosilyl (Lewis electrophilic reagents), such as R′R″SiOH (R′ = R″ = Et, iPr, OtBu, Ph; R = tBu, R″ = Me) to yield monomeric plenary Lindqvist derivatives (nBu4N)4[W5O18(Ta(O)SiR′R″)]. These derivatives are characterized in the solid state by IR and in solution by multinuclear NMR (13C, 29Si, 183W). The crystallographic study of (nBu4N)4[(W3O10)2(Ta(O)SiPh)] indicates that {SiPh3}+ is grafted on the surface of the polyanion through the terminal O-Ta oxygen atom.

Keywords: polyoxometalates, X-ray structure, NMR spectroscopy

**Poster Sessions**

**P09.03.28**

Strongly and accurately shaped Ge crystal for non-scanning X-ray fluorescence spectrometer

Koichi Hayashi, Kazuo Nakajima, Fujiwara Kozo

Tohoku University, Institute for Materials Research, 2-1-1 Katahira, Aoba-ku, Sendai, Miyagi, 980-8577, Japan, E-mail: khayashi@imr.tohoku.ac.jp

Plastic deformation technique of Si and Ge single crystals, which enables us to obtain the various forms, makes impact upon the field of X-ray spectroscopy, because Si and Ge are commonly used as analyzing crystals for x rays. Recently, we developed a deformation technique for obtaining strongly and accurately shaped Si or Ge wafers of high crystal quality, although covalently bonded Si or Ge crystals have long been believed to be not deformable to various shapes. The demonstration of the deformed wafer made it possible to produce fine-focused x rays. In the present study, we prepared a cylindrical Ge wafer with a radius of curvature of 50 mm (Fig.), and acquired fluorescent x rays simultaneously from 4 elements by combining the cylindrical Ge wafer with a position-sensitive detector. The energy resolution of the x-ray fluorescence spectrum was as good as that obtained using a flat single crystal, and its gain was over 100. The demonstration of the simultaneous acquisition of high-resolution x-ray fluorescence spectra indicated various possibilities of x-ray spectrometry, such as one-shot x-ray spectroscopy and highly efficient wave-dispersive x-ray spectrometers.

Keywords: Ge wafer, X-ray spectrometer, plastic deformation

**P09.03.27**

Octamolybdates - promising materials for industry and medicine

Wieslaw Lasocha1,2, Maciej Gryzwa2, Wojciech Nitek1, Bartlomiej Lasocha3, Bartlomiej Gawel1, Alicja Rafalska-Lasocha1

1Jagiellonian University, Faculty of Chemistry, Ingardena 3, Krakow, woj. Malopolskie, 30-060, Poland, 2Institute of Catalysis PAS, Niezapominajek 8,30-239 Krakow, Poland, 3Collegium Medicum, Jagiellonian University, Krakow, Poland, E-mail: 1 lasocha@chemia.uj.edu.pl

Molybdates are interesting and perspective candidates for various applications in electronics and medicine (cancer therapy) [1], catalysis and environment protection. Polymolybdates are a numerous group of compounds and despite of enormous work done so far, synthesis of selected types of polymolybdates is still a challenging and demanding task, requiring experience, intuition and vast experimental work. Recently a group of 5 new beta-octamolybdates was obtained in our lab. We solved the crystal structures and investigated selected properties of: 1/ Ammonium tris(triethylammonium) octamolybdate, (a,b,c, alpha, beta, gamma, SG)25.230(5),10.859(2),19.033(3),121.92(1), C 2/ Tetrakis(triethylammonium) octamolybdate dihydrate; 1 0.433(3),10.486(2),10.708(4),121.78(3),118.35(3), P 0.433(3),10.486(2),10.708(4),121.78(3),118.35(3), P 3/ Tetrakis(naphthenal-1-aminium) octamolybdate tetra(1-naphthylamine); 15.561(5),18.969(8),7.54(3),100.80(3),103.04(3),73.42(3), P 1/ Bis(1-amino-1-phenyleanammonium) bis(2-methylbenzimidazoilium) octamolybdate; 8.541(5), 10.293(5), 13.018(5), 80.03(5), 83.74(5) 75.12(5), P-1 4/ Tetrakis(2,6-dimethylammonium) octamolybdate: 11.878(3), 10.533(3), 11.586(2), 101.12(2), 120.26(1), 75.46(2), P-1 Compounds 1 - 2 were obtained in hydrothermal conditions while 3-5 from hot mixtures of H3MoO4, amine and H2O. Most of these compounds crystallise in SG P-1, in compound 3 protonated and neutral amines are present, in 4 unexpected 2-methylbenzimidazolium cation was obtained. Based on the results of crystal structure determination of polymolybdates, some rules concerning the crystal engineering of iso polymolybdates, will be presented. Supported by ICDD and Polish MEiN grant 1T09A 07730

References:


**P09.03.29**

Determination of thermal treatment effect of plating sludge by phase identification: XRD technique

Ozgul Dere1, Mehmet Burcin Piskin2, Aysel Kanturk1, Muge Sari3

1Yildiz Technical University, Chemical Engineering, Davutpasa Campus, Chemical Engineering, Esenler, Istanbul, 34210, Turkey, 2Yildiz Technical University, Davutpasa Campus, BioEngineering, Esenler, Istanbul, 34210, Turkey, E-mail: odere@yildiz.edu.tr

Thermal treatment was used to recover the precious metals or stabilize solid industrial wastes such as metal plating sludge which includes Zinc (Zn), Chromium (Cr), Copper (Cu), Nickel (Ni), iron etc. compounds. After thermal treatment of the samples, metal compounds were converted to metal oxides and then they were leached with suitable reagents to recover the precious metals. Moreover treatment temperature is very important in order to determine the optimum conditions. Therefore, metal plating sludge’s composition changes due to operating process parameters. In addition to examined metal plating sludge’s crystal structure which were shown differences when compare with the others. These