

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 $(n\text{Bu}_4\text{N})_4[(\text{TaW}_5\text{O}_{18})_2\text{O}]$ which is synthesized by reacting $[\text{TaW}_5\text{O}_{19}]^{3-}$ with BuSnCl_3 , crystallises in the orthorhombic system, space group Pbnm with lattice parameters $a = 15.7981(14)$, $b = 17.939(3)$, $c = 35.216(6)\text{\AA}$, $V = 9980\text{\AA}^3$ and $Z = 4$. The crystallographic study of $(n\text{Bu}_4\text{N})_4[(\text{TaW}_5\text{O}_{18})_2\text{O}]$ shows that the dimer is composed from two polyoxoanions fragments linked by linear Ta-O-Ta bridge. Such a linkage readily reacts with organosilyl (Lewis electrophilic reagents), such as $\text{RR}'_2\text{SiOH}$ ($\text{R} = \text{R}' = \text{Et}$, $i\text{Pr}$, OtBu , Ph ; $\text{R} = \text{tBu}$, $\text{R}' = \text{Me}$) to yield monomeric plenary Lindqvist derivatives $(n\text{Bu}_4\text{N})_2[\text{W}_5\text{O}_{18}\text{Ta}(\text{O})\text{SiR}'\text{R}_2]$. These derivatives are characterized in the solid state by IR and in solution by multinuclear NMR (^{13}C , ^{29}Si , ^{183}W). The crystallographic study of $(n\text{Bu}_4\text{N})_2[(\text{W}_5\text{O}_{18}\text{Ta}(\text{O})\text{SiPh}_3)]$ indicates that $\{\text{SiPh}_3\}^+$ is grafted on the surface of the polyanion through the terminal O-Ta oxygen atom.

Keywords: polyoxometalates, X-ray structure, NMR spectroscopy

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Octamolybdates - promising materials for industry and medicine

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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), $C 2/c$ 2/ Tetrakis(trimethylammonium) octamolybdate dihydrate; 10.433(3), 10.486(2), 10.708(4), 102.40(2), 101.78(3), 118.35(3), $P-1$ 3/ Tetrakis(naphthalen-1-aminium) octamolybdate tetra(1-naphthylamine); 15.561(5), 18.969(8), 7.54(3), 100.80(3), 103.04(3), 73.42(3), $P-1$ 4/ Bis(1-amino-1-phenyleneammonium) bis(2-methylbenzimidazolium) octamolybdate; 8.541(5), 10.293(5), 13.018(5), 80.03(5), 83.74(5), 75.12(5), $P-1$ 5/ Tetrakis(2,6-dimethylanilinium) 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 H_2MoO_4 , amine and H_2O . 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 isopolymolybdates, will be presented. Supported by ICDD and Polish MEiN grant 1T09A 07730

References:

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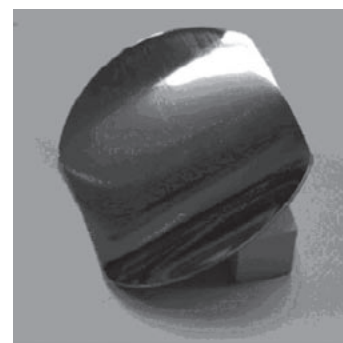
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Strongly and accurately shaped Ge crystal for non-scanning X-ray fluorescence spectrometer

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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 use 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

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

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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