

The appearance of these inclusions allow us to estimate the temperature of the fusion which did not reach the temperature of hematite melting. Besides, in the glass, we find the inclusions of hematite with the tracks of partial fusion.

Appearance of a great amount skeleton formations with the composition such as FeO is an evidence of a great fusion cooling speed. Such structures forms because of dissociation on the stage of cooling.

Besides we find the numerous inclusions corresponding by structure to baddeleyite glass in the form of thin thready formations, specifying on processes of segregation proceeding in the time of hardening of fusion. These processes of segregation were not observed before in fulgurites. Also, inclusions of baddeleyite glass meets and as separate grains with crystallographic facet and with the raised contents of uranium. Besides there are aluminosilicate inclusions enriched with the titan and phosphorus. In such inclusions are widely used the clusters of ferrian composition.

**Keywords:** fulgurite, hematite, segregation

#### P.10.04.4

*Acta Cryst.* (2005). A61, C373

#### Synthesis and X-ray Study of [Pt(NH<sub>3</sub>)<sub>4</sub>](ReO<sub>4</sub>)<sub>2</sub> Thermolysis Products

Andrey Zadesenets, Ilya Korolkov, Iraida Baidina, Sergey Gromilov, <sup>a</sup>Nikolaev Institute of Inorganic Chemistry SB RAS, Novosibirsk, Russia. E-mail: zadesenez@ngs.ru

Catalysts based on the Pt and Re play an essential role in reforming processes. About 65% of all the produced rhenium is used for these needs. One of the methods to prepare catalysts is the thermolysis of inorganic complex salts, containing two required metals. In this work we have studied the precursor complex [Pt(NH<sub>3</sub>)<sub>4</sub>](ReO<sub>4</sub>)<sub>2</sub> and obtained a solid solution Pt<sub>0.33</sub>Re<sub>0.67</sub>.

A synthesis of the precursor complex [Pt(NH<sub>3</sub>)<sub>4</sub>](ReO<sub>4</sub>)<sub>2</sub>, was held in the following way: 0.2 M water solutions of [Pt(NH<sub>3</sub>)<sub>4</sub>]Cl<sub>2</sub> and NaReO<sub>4</sub> were mixed at 50°C, then kept for an hour at room temperature. A white residue was separated and washed away with water and acetone. The yield is 75%. The crystal structure of [Pt(NH<sub>3</sub>)<sub>4</sub>](ReO<sub>4</sub>)<sub>2</sub> was determined with a X8APEX Bruker diffractometer (MoK $\alpha$ -radiation,  $\theta$  range 2.85–32.59°, 2007 independent reflections), R = 2.11%. Crystal data: a = 5.1847(6), b = 7.7397(8), c = 7.9540(9) Å,  $\alpha$  = 69.531(3),  $\beta$  = 79.656(3),  $\gamma$  = 77.649(3)°, V = 290.19(6) Å<sup>3</sup>, space group P-1, Z = 1. This complex is isostructural to [Pt(NH<sub>3</sub>)<sub>4</sub>](TeO<sub>4</sub>)<sub>2</sub> [ICSD Card 65-766]. The structure consists of the isolated complex [Pt(NH<sub>3</sub>)<sub>4</sub>]<sup>2+</sup> and ReO<sub>4</sub><sup>-</sup> ions.

The thermolysis of [Pt(NH<sub>3</sub>)<sub>4</sub>](ReO<sub>4</sub>)<sub>2</sub> was carried out at 900°C in H<sub>2</sub> atmosphere for 7 hours. According to X-ray analysis, the product is a single phase solid solution Pt<sub>0.33</sub>Re<sub>0.67</sub>. It is based on the hexagonal close-packed rhenium structure. Crystal data of the solid solution: a = 2.764(2), b = 4.415(3) Å, V = 29.21 Å<sup>3</sup>, space group P6<sub>3</sub>/mmc, Z = 2.

**Keywords:** platinum group, X-ray analysis, single-crystal structure analysis

#### P.10.04.5

*Acta Cryst.* (2005). A61, C373

#### Crystal Structure of KLiSO<sub>4</sub> at High Temperatures

Hyun Min Park, Yang Koo Cho, Su Jin Chung, *New Materials Evaluation Center, Korea Research Institute of Standards and Science, P.O. Box 102, Taejeon, 305-600 Korea.* E-mail: hmpark@kriss.re.kr

Above room temperature there are three modifications of KLiSO<sub>4</sub>; i.e. phase I, II and III. Phase transition temperatures between them are 435°C and 668°C respectively. The x-ray diffraction intensities of phase I at about 700°C and of phase II at about 600°C were collected on the CAD4 single crystal diffractometer equipped a self-made furnace. During collection of intensities data, a prominent decay of intensities was observed. The crystal structure of phase I is the tridymite derivatives with ordered arrangement of SO<sub>4</sub><sup>-</sup> and LiO<sub>4</sub><sup>-</sup> tetrahedra similar to that of phase III. The statistical data of the structure refinement for phase I with polar space group of P6<sub>3</sub>mc was R = 0.057, Rw = 0.056 and S (goodness-of-fit) = 1.859. The crystals

of phase II usually exhibit misleading hexagonal twinned cell which is composed of three orthorhombic twin domains in the temperature range between 435°C and 668°C. An almost twin-free single crystal of Phase II was observed at the elevated temperature. The crystal structure of phase II was refined with this crystal is orthorhombic with the polar space group Pc2<sub>1</sub>n. The final statistical data was R = 0.077, Rw = 0.073 and the S = 1.028 with ordered arrangement of atoms. This data was compared with that of the twinned crystal corrected with twin ratio and further discussed previously reported disordered model with the space group of Pmcn.

**Keywords:** KLiSO<sub>4</sub>, structure analysis, high temperature

#### P.10.04.6

*Acta Cryst.* (2005). A61, C373

#### Ab initio Treatment of Minerals at Extreme Conditions

Daniel Y. Jung<sup>a,b</sup>, Artem R. Oganov<sup>b</sup>, <sup>a</sup>Institut of mineralogy and petrology, Department of Earth science, ETH Zürich. <sup>b</sup>Laboratory of crystallography, Department of Materials, ETH Zürich, Switzerland. E-mail: daniel.jung@mat.ethz.ch

The mantle of the Earth extends from the depth of about 670 km to 2981 km. It consists mainly of MgSiO<sub>3</sub>-perovskite, (Mg,Fe)O magnesiowüstite and CaSiO<sub>3</sub>-perovskite. It is possible to calculate thermodynamic properties, structures and energetics of the separate minerals at extreme conditions of the mantle using *ab initio* methods, such as the density functional theory with the generalized gradient approximation (GGA) [1] and the projector augmented wave (PAW) method [2], which are included in the VASP [3] code. To get a better picture of the mantle it is necessary to not only look at chemically pure minerals, but to consider them as a solid solution, as it is the probable case in nature.

Using density functional theory the structure and the stability of the CaSiO<sub>3</sub> perovskite in the pressure range of the Earth's mantle (0-150 GPa) have been calculated [4]. Additionally we use the subregular solid solution model together with point defect calculations to model the solvus of the (Ca,Mg)-perovskite phase diagram at 25 GPa. This is a special case, because there is also a symmetry change from a tetragonal to an orthorhombic perovskite structure as you increase the concentration of Mg. This is the first work to treat this subject with *ab initio* methods.

[1] Perdew J.P., Burke K., Ernzerhof M., *Phys. Rev. Lett.*, 1996, **77**, 3865. [2] Blöchl P.E., *Phys. Rev. B.*, 1994, **50**, 17953. [3] Kresse G., Furthmüller J., *Phys. Rev. B.*, 1996, **54**, 11169. [4] Jung D.Y., Oganov A.R., *Phys. Chem. Mineral*, 2005, *in press*.

**Keywords:** (Ca,Mg)SiO<sub>3</sub> perovskite, Earth's mantle, solid solution

#### P.10.04.7

*Acta Cryst.* (2005). A61, C373-C374

#### Viscosity Measurements of Fe-FeS Melts under High Pressures

Ken-ichi Funakoshi, Akifumi Nozawa, *Japan Synchrotron Radiation Research Institute, SPring-8, Hyogo, Japan.* E-mail: funakosi@spring8.or.jp

The Fe-FeS melt is the important Earth's outer core material and its viscosity has been thought to be very low under high pressure and high temperature. Recent measurements of the Fe-FeS melt have showed the low viscosity values (~10<sup>-2</sup> Pa-s), however, the accuracies are not so good for determining the viscosity values. An x-ray radiography technique with synchrotron radiation is very useful for the falling sphere viscosity measurement, because it enables us in situ observation of the sinking process and determination of the reliable viscosity coefficient.

We measured the viscosities of Fe-FeS (Fe<sub>73</sub>S<sub>27</sub>, Fe<sub>80</sub>S<sub>20</sub>, Fe<sub>90</sub>S<sub>10</sub>) melts, combining the falling sphere method with the large volume press at the SPring-8. Precise viscosities have been obtained up to 9 GPa using Stoke's law. The pressure dependences of the viscosities are very small, however, the viscosities slightly increase with increasing pressures. The activation energies and the activation volumes have been determined from the dependences on pressure and temperature of the viscosities. The viscosities of the Earth's outer core have been calculated using the activation energies and the activation volumes of Fe<sub>90</sub>S<sub>10</sub>. The calculated viscosities of the Earth's outer core