

**P08.10.110***Acta Cryst.* (2008). A64, C452**New  $M_{1/3}Re_{2/3}O_2$  (M = Fe, Co, Ni) oxides with rutile-like structures**Daria A. Mikhailova<sup>1,2</sup>, Helmut Ehrenberg<sup>1</sup>, Gerhard Brey<sup>3</sup>, Hartmut Fuess<sup>2</sup>

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For the system  $M_{1/3}Re_{2/3}O_2$  (M = Fe, Co, Ni) different rutile or rutile-derived structural modifications were found depending on the temperature during high-pressure high-temperature syntheses. The crystal structures have been determined by single crystal X-ray diffraction. For synthesis temperature higher than 1300 °C, phases with tetragonal rutile aristotype structure (S. G.  $P4_2/mnm$ ) and a statistical distribution of M- and Re-atoms on the metal site in the structure were obtained. At lower synthesis temperature, rutile-like structures with orthorhombic or monoclinic symmetry, partial order on M- and Re-sites and metallic Re-Re bonds were observed for  $Co_{1/3}Re_{2/3}O_2$  and  $Ni_{1/3}Re_{2/3}O_2$ . A monoclinic  $CrWO_4$ -type structure (S. G.  $C2/m$ ) with only Re-atoms on the 4g site and statistically distributed Ni- and Re-atoms on the 4i site was adopted by  $Ni_{1/3}Re_{2/3}O_2$ . Single crystals  $Co_{1/3}Re_{2/3}O_2$  with orthorhombic  $CrWO_4$ -type structure (S. G.  $F222$ ) and also partially mixed Co, Re-positions were found together with a monoclinic modification, which is isotypic to monoclinic  $Ni_{1/3}Re_{2/3}O_2$ . Note, that another orthorhombic modification (S. G.  $Cmmm$ ) was reported for the composition  $Co_{1/2}Re_{1/2}O_2$  with distinct positions of Co and Re and without any metallic Re-Re-bond [1]. Unfortunately, the synthesis conditions were not described in sufficient detail to allow a comparison with our preparation method.  $Ni_{1/3}Re_{2/3}O_2$  with the rutile-structure orders ferro- or ferrimagnetically at 23.5 K, whereas tetragonal  $Co_{1/3}Re_{2/3}O_2$  shows two magnetic transitions: below 17.5 K the ferromagnetic component dominates in contrast to an antiferromagnetic order between 27.0 and 17.5 K.

[1] W. H. Baur, W. Joswig, G. Pieper, D. Kassner, J. Solid State Chem. 99 (1992) 207-211.

Keywords: high-pressure high-temperature synthesis, rutile-like structure, metallic Re-Re bond

**P08.10.111***Acta Cryst.* (2008). A64, C452**Compression and thermal behavior of  $GeO_2$  glass**

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One fascinating high-pressure behavior of tetrahedral glasses and melts is the local coordination change with increasing pressure, which provides a structural basis for understanding numerous anomalies in their high-pressure properties. Because the coordination change is often not retained upon decompression, studies must be conducted in situ. Previous in situ studies have revealed that the short-range order of tetrahedrally structured glasses and melts changes above a threshold pressure and gradually transforms to an octahedral form with further pressure increase. We report compression behavior of  $GeO_2$  glass up to 35 GPa, and a thermal effect associated with the coordination change at given pressures.

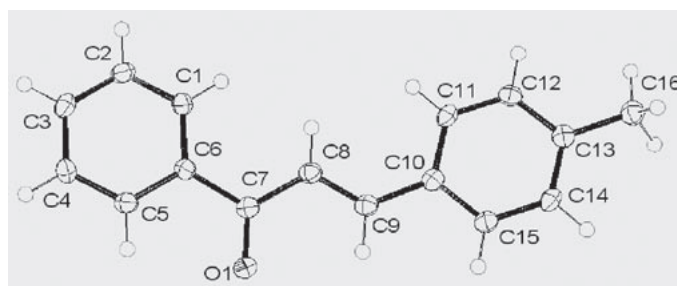
The results show a clear anomaly in compression curve associated with the coordination change, as well as distinct thermal behaviors of  $GeO_2$  glass in tetrahedral, octahedral, and their intermediate forms. An unusual thermally induced densification, as large as 16%, was observed on a  $GeO_2$  glass at a pressure of 5.5 GPa. The large thermal densification at high pressure was found to be associated with the 4- to 6-fold coordination increase. Experiments at other pressures show that the tetrahedral  $GeO_2$  glass displayed small thermal densification at 3.3 GPa arising from the relaxation of intermediate range structure, whereas the octahedral glass at 12.3 GPa did not display any detectable thermal effects.

Keywords: high pressure, coordination change, compression

**P08.10.112***Acta Cryst.* (2008). A64, C452**Disappearing and reappearing polymorphism in *p*-methylchalcone**Inna Barsky<sup>1</sup>, Bernstein Joel<sup>1</sup>, Peter W Stephens<sup>2</sup>, Kevin H Stone<sup>2</sup>, Eugene Cheung<sup>3</sup>, Magali B Hickey<sup>3</sup>

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The polymorphism of *p*-methylchalcone (*p*-MC) has been the subject of interest for about 80 years. One stable form (Form I) and two metastable forms (Form II and Form IV) of *p*-methylchalcone have been obtained, with Form II having previously been reported to be a disappearing polymorph. To prevent the conversion of Form II, which has a melting point of 89-91 °C, into the stable form it was produced in conditions free from seeds of the stable form. Form IV was discovered serendipitously as a single crystal that appeared in the preparation of a powder of Form II. Forms I and II have also been characterized by optical microscopy, differential scanning calorimetry (DSC), variable temperature powder X-ray diffraction and spectroscopic analysis. The crystal structures of these three forms have been solved, Forms I and IV by single-crystal methods and Form II from synchrotron powder diffraction.



Keywords: *p*-Methylchalcone, polymorphism, X-ray structure determination

**P08.11.113***Acta Cryst.* (2008). A64, C452-453**Switchable polymorph-dependent luminescence of terpyridine**Toshiki Mutai<sup>1</sup>, Yasuhiro Shigemitsu<sup>2</sup>, Hirohiko Houjou<sup>1</sup>, Koji Araki<sup>1</sup>

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