

P11.16.121*Acta Cryst.* (2008). A64, C544**X-ray crystallographic analysis of by-products produced in photochromism of dithienylcyclopentenes**Hiroko Fujiwara¹, Seiya Kobatake^{1,2}¹Osaka City University, Department of Applied Chemistry, Graduate School of Engineering, 3-3-138 Sugimoto, Sumiyoshi-ku, Osaka, Osaka, 558-8585, Japan, ²PRESTO, JST, Kawaguchi Center Building, 4-1-8 Honcho, Kawaguchi, Saitama, 332-0012, Japan, E-mail : kobatake@a-chem.eng.osaka-cu.ac.jp

Diarylethenes undergo thermally stable photochromic reactions even in the crystalline phase as well as in solution. Among the diarylethenes, dithienylperfluorocyclopentenes have a characteristic of fatigue resistance. However, dithienylcyclopentenes are not really investigated. Here we focused on analysis of side reactions in the photochromic reactions of 1,2-bis(2-methyl-5-phenyl-3-thienyl)cyclopentene (**1**), 1,2-bis(2-methyl-5-(p-methoxyphenyl)-3-thienyl)cyclopentene (**2**), and 1,2-bis(2,4-dimethyl-5-(p-methoxyphenyl)-3-thienyl)cyclopentene (**3**). Upon alternate irradiation with ultraviolet and visible light, the dithienylcyclopentenes (**1-3**) exhibited photochromism in hexane. When the solution was irradiated with ultraviolet light for more than 10 min, the coloration/decoloration performance was declined. By-products were isolated by HPLC, and their molecular structures were determined by ¹H-NMR spectra, Mass spectra, and X-ray crystallographic analysis of the single crystals. To know the relationship between the molecular structure of the dithienylcyclopentene and the formation rate of the by-product, we determined quantum yields of not only the photocyclization and photocycloreversion reactions but also the by-product formation. The quantum yields of the by-product formation of **1** and **2** were about 10 times larger than that of 1,2-bis(2-methyl-5-phenyl-3-thienyl)perfluorocyclopentene (**4**). This indicates that the dithienylcyclopentenes were more fatigable than the dithienylperfluorocyclopentenes. In contrast, by the introduction of methyl groups at the 4-positions of the thiophene in the dithienylcyclopentene, the coloration/decoloration cycles of **3** can be repeated as well as that of **4**. In conclusion, the side reactions were suppressed by the introduction of methyl groups.

Keywords: photochromism, diarylethene, photoreaction

P11.16.122*Acta Cryst.* (2008). A64, C544**X-ray diffraction of laser-heated silicon at high pressures**Laura Robin Benedetti¹, Daneiele Antonangeli¹, Chantel Aracne¹, Daniel L Farber¹, Mohamed Mezouar²¹LLNL, 7000 East Ave, L-206, Livermore, CA, 94550, USA, ²European Synchrotron Radiation Facility, 38000 Grenoble, France, E-mail : lrbenedetti@gmail.com

The phase diagram of silicon is complex : it exhibits seven distinct polymorphs at pressures below 100 GPa, and the melting curve has a minimum at ~10 GPa and ~1000 K. In order to explore the high temperature phase diagram, we performed simultaneous angular-dispersive x-ray diffraction and laser heating at beamline ID27 at the ESRF. We present x-ray diffraction of Si in the laser heating diamond cell between 30 and 70 GPa at temperatures between 1500 and 3000 K. Particular attention is paid to determination of the melting curve at high pressures.

Keywords: silicon, high pressure, laser heating

P11.16.123*Acta Cryst.* (2008). A64, C544**Rochelle salt thermal expansion coefficients determined by synchrotron radiation renninger scan**Adenilson O Dos Santos¹, Alan S de Menezes¹, Jose M Sasaki², Lisandro P Cardoso¹¹Universidade Estadual de Campinas, Instituto de Física Gleb Wataghin - Dep. Física Aplicada, CP 6165, Campinas, Sao Paulo, 13083-970, Brazil, ²DF, Universidade Federal do Ceara (UFC), CP 6030, 60455-760, Fortaleza, Ceara, Brazil, E-mail: adenilso@ififi.unicamp.br

The thermal expansion coefficients of Rochelle salt single crystal have been determined by using the Synchrotron radiation Renninger scan (RS) that acts as a 3D fine probe in these experiments. The angular peak shift clearly observed for sensitive secondary reflections in the RS due to the temperature variation allows for simultaneously detecting subtle distortions along the three crystallographic directions of the sample. These secondary reflections present a small angle between the entrance and exit position of the secondary reciprocal lattice point of the Ewald sphere by sample rotation around the diffraction vector (normal to the primary planes). The UNWEG program (Rossmannith, J. Appl. Cryst. (2003) 36, 1467) has allowed to calculate the Rochelle salt RS pattern by using the X-ray diffraction kinematical theory. The chosen sensitive secondary reflections were: (-2 4 1)(12 4 -1), (-2 5 2)(12 5 2) and (-1 4 1)(11 4 1), and from the peak shift measurements in the orthorhombic phase (T > 24°C) we were able to obtain the lattice parameters with very good resolution. The Rochelle salt thermal expansion coefficients determined from the lattice parameter variation ($\alpha_{[100]}=62(2)\times 10^{-6} \text{ C}^{-1}$, $\alpha_{[010]}=38(8)\times 10^{-6} \text{ C}^{-1}$ and $\alpha_{[001]}=45(5)\times 10^{-6} \text{ C}^{-1}$) are in very good agreement with the literature values ($\alpha_{[100]}=(58-62)\times 10^{-6} \text{ C}^{-1}$, $\alpha_{[010]}=(42-54)\times 10^{-6} \text{ C}^{-1}$ and $\alpha_{[001]}=(43-54)\times 10^{-6} \text{ C}^{-1}$).

Keywords: X-ray multiple diffraction, thermal expansion, inorganic crystals

P11.16.124*Acta Cryst.* (2008). A64, C544-545**Monocrystal like structural, stochastic and microstructural information from polycrystalline samples**Gavin B.M. Vaughan¹, Jonathon P Wright¹, Carsten Gundlach¹, Soeren Schmidt², Henning P Poulsen², Henning O Soerensen²¹ESRF, Materials Science, 6 rue Jules Horowitz BP 220, Grenoble Cedex, Isere, 38043, France, ²Risoe National Lab, Roskilde DK, E-mail : vaughan@esrf.fr

In the last several years we have demonstrated methods whereby single crystal quality data can be extracted from polycrystalline materials. These data can be used not only for high-quality structure solution and refinement, but also to study sample heterogeneity. By determining structural of each crystallite in a polycrystalline sample, we can characterise not only the average properties of samples by the distribution of these properties. As the individual orientation matrices of the crystallites are determined, it is furthermore possible to correlate chemical properties with microstructural properties of the crystals such as their strain state and orientation. In situ studies allow the dynamic behaviour to be correlated with all these properties, and finally spatially resolved studies allow the probing of inter-crystalline effects. We will provide examples from the latest results of these studies.

Keywords: crystal structure determination, materials