ESRF	**Experiment title:** Combined high pressure and low temperature powder diffraction studies of tetrakistrimethylsilylsilane	**Experiment number:** HS-1094
Beamline: ID 9	**Date of experiment:** from: 25.1.2000 to: 1.2.2000	**Date of report:** 1 July 2000
Shifts: 18	**Local contact(s):** Michael Hanfland	*Received at ESRF:*

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Report:

The compound tetrakistrimethylsilylsilane $Si(Si(CH_3)_3)_4$ (TSi) belongs to a class of crystalline molecular compounds with nearly spherically shaped molecular units (Fig. 1) in a closed packed stacking. At ambient conditions, the crystal structure has space group $Fm\bar{3}m$ with molecules showing approximately 6-fold orientational disorder (Fig. 2). Upon cooling a first-order phase transitions occurs at T_c= 225 K to space group $P2_13$ with fully ordered molecules due to steric interactions between neighboring molecules [1].

In this work, *in situ* measurements were performed at high pressure as well as at high pressure and low temperature for TSi using a DAC and a closed cycle helium cryostat. An angle dispersive powder diffraction technique at λ= 0.43133 Å with an online fast readout 2D MAR345 detector was used. Data reduction was performed using the programs FIT2D (by Andy Hammersley, ESRF) and GUFI (by Dinnebier).

In contrast to the phase transitions of TSi which occur at low temperature [1] and in contrast to the phase transitions of the similar compound $C(Si(CH_3)_3)_4$ (TC) at low temperature [1] or high pressure [2], TSi undergoes two phase transitions from the cubic fcc phase (Fig. 2) to a monoclinic phase (Fig. 3) and at higher pressure to a rhombohedral phase (Fig. 4).

It is interesting to note that the quality of the powder patterns of TSi at high pressure *and* low temperature was high enough to index the powder pattern of the monoclinic phase with a cell volume of more than **2200** Å3 (Fig. 3b.).

[1]
R. E. Dinnebier, W. A. Dollase, X. Helluy, J. Kümmerlen, A. Sebald, M. U. Schmidt, S. Pagola, P. W. Stephens & S. van Smaalen (1999). Acta Cryst. B55, 1014-1029;
[2]
R. E. Dinnebier, S. Carlson & S. van Smaalen (1999). Acta Cryst. B 56, 10-316.

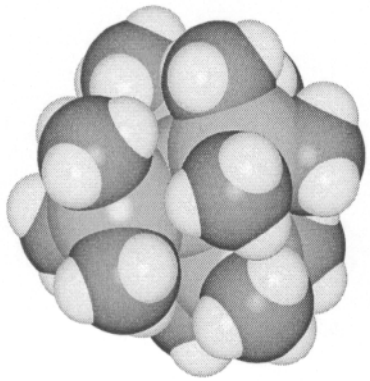

Fig. 1:
Cup model of the molecular structure of $Si(Si(CH_3)_3)_4$ at ambient conditions.

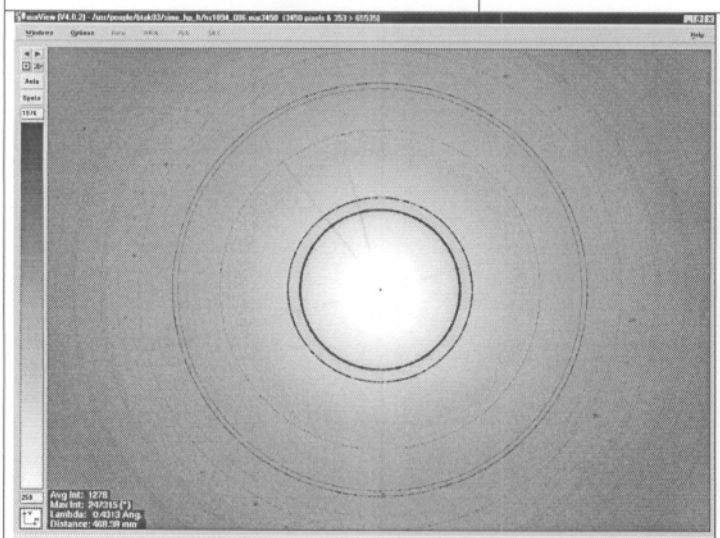

Fig.2:
Image plate of the cubic fcc of phase of $Si(Si(CH_3)_3)_4$ at 1 kbar and 295 K.

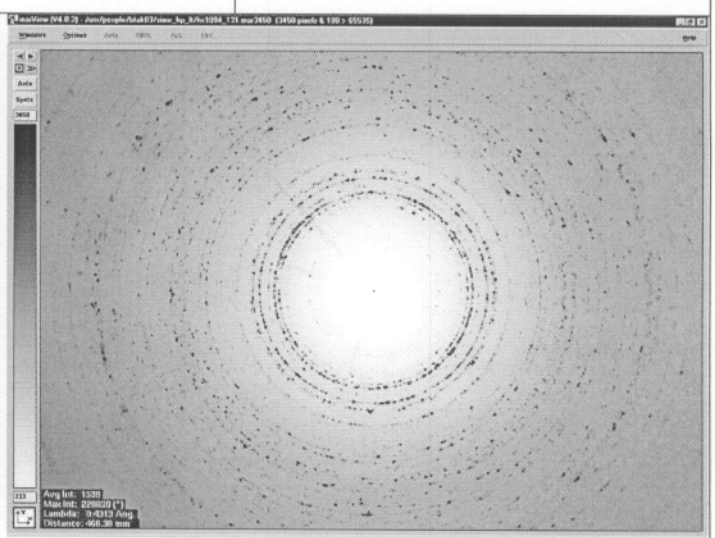

Fig. 3a:
Image plate of the monoclinic phase of $Si(Si(CH_3)_3)_4$ 2.1 kbar and 240 K.

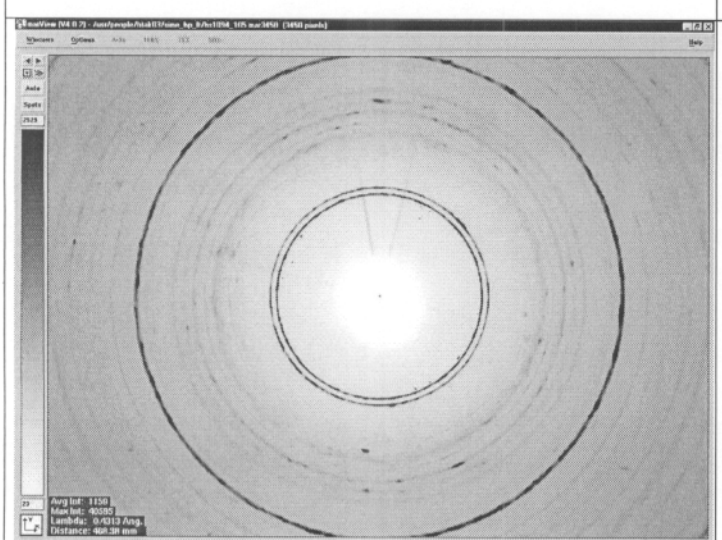

Fig. 4:
Image plate of the rhombohedral phase of $Si(Si(CH_3)_3)_4$ at 22 kbar and 295 K.

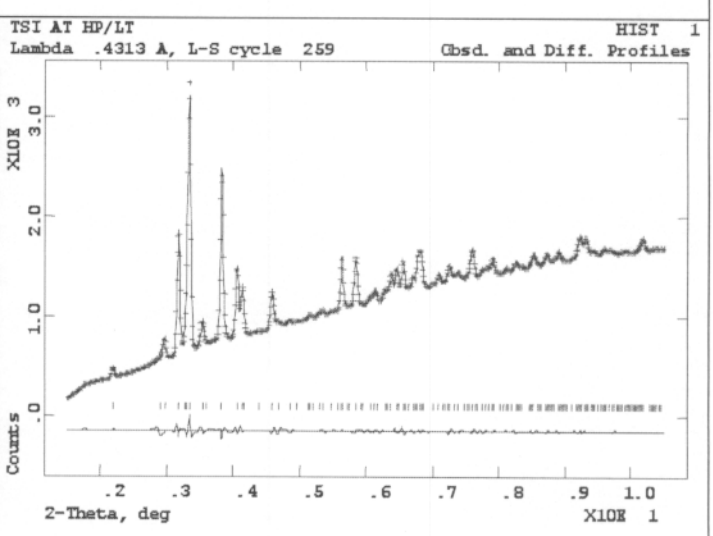

Fig. 3b:
*LeBail plot of the monoclinic phase of $Si(Si(CH_3)_3)_4$ at 2.1 kbar and 240 K (a= 15.380, b= 16.942, c=8.917 Å, β= 101.62°, V= **2275.85** Å^3, P $2_1/n$).*