ESRF

ESRF	**Experiment title:** Crystal structure of methane and silane at very high pressures	**Experiment number**: HS3106
Beamline: ID09	**Date of experiment**: from: 2/11/2006 to: 7/11/2006	**Date of report**: 28/02/2007
Shifts: 15	**Local contact(s)**: M. Hanfland	*Received at ESRF:*

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Report:

Methane (CH_4) was studied up to 35 GPa using several sample loadings. We have collected diffraction patterns of phase B, stable between 9 and 25 GPa, and the so-called HP phase, stable above 25 GPa. We obtained a very high quality diffraction pattern of the phase B on pressure release (Figure 1). The cubic cell, proposed earlier by Umemoto et al [1], is shown to be incorrect, as there are two observed diffraction reflections that cannot be explained with this cell (Figure 1, inset). The high quality of the data allowed us to propose a new, larger, cubic unit cell that would explain all the observed diffraction peaks (Figure 2). The refined lattice parameter at 8.0 GPa is 12.0014(1) Å. The structure solution is in progress.

We have encountered significant difficulties with loading silane (SiH_4), which is poisonous and explosive substance. As the result we have not obtained any meaningful data on silane. During last several months we have constructed special glove box equipped with the proper silane resistant valve and at the moment we are capable of loading SiH_4.

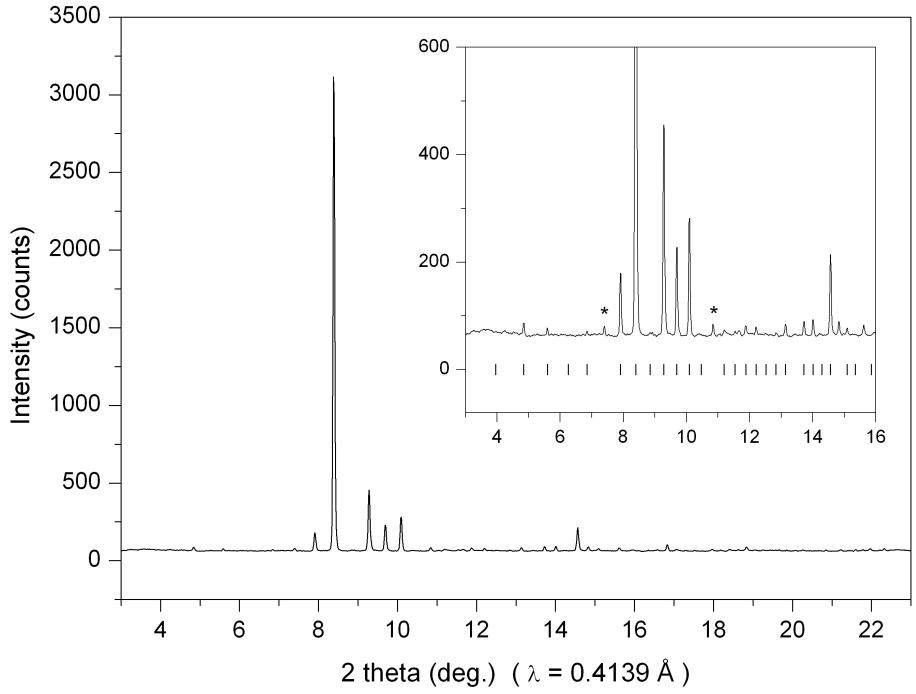

Figure 1. Powder diffraction pattern of methane in phase B, collected at 8.0 GPa on pressure decrease. Inset shows the same pattern fitted with the primitive cubic cell (8.486 Å), proposed by Umemoto et al [1], with tick marks showing the calculated peak positions. The asterisks show the two diffraction peaks that are unexplained within the proposed cell.

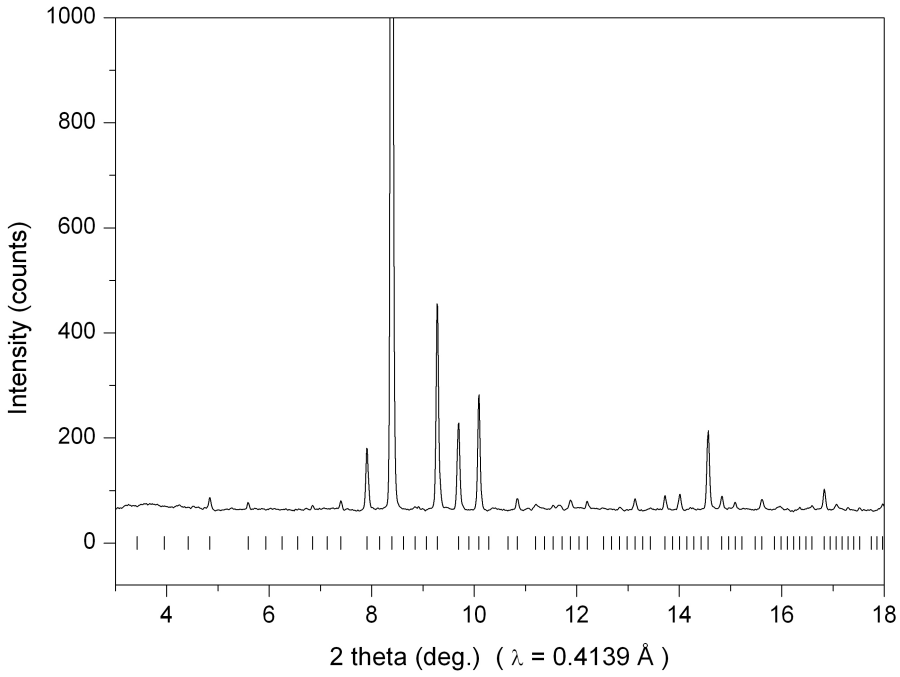

Figure 2. Powder diffraction pattern of methane in phase B, collected at 8.0 GPa on pressure decrease, fitted with the newly proposed cubic cell (12.001 Å). Tick marks show the calculated peak positions.

[1] S. Umemoto, T. Yoshii, Y. Akahama and H. Kawamura, J. Phys.: Condens. Matter 14 (2002) 10675.