ESRF	**Experiment title:** Triclinic type F structure of $Sm_2Si_2O_7$ and $Eu_2Si_2O_7$; geometry of the $Si_2O_7^{6-}$ ion in $La_2Si_2O_7$ at high pressure.	**Experiment number:** CH-88
Beamline: BM3/ID09	**Date of experiment:** from: 1 November 95 to: 3 November 95	**Date of report:** 31 July 1996
Shifts: 6	**Local contact(s):** Michael Hanfland	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Axel Nørlund Christensen,* Chemistry Department, Aarhus University, DK-8000 Aarhus C, Denmark.

Brian Themsen,* Chemistry Department, Aarhus University, DK-8000 Aarhus C, Denmark.

Anette Frost Jensen,* Chemistry Laboratory IV, H.C. Ørsted Institute, Universitetsparken 5, DK-2100 Copenhagen Ø, Denmark.

Michael Hanfland,* ESRF, Experiment Division, B.P. 220, F.38043 Grenoble Cedex, France.

Part B: Investigation of the structure of $La_2Si_2O_7$ at pressures up to 212 kbar.

The rare earth disilicates $RE2Si_2O_7$ can exist in seven different phases of which four are high temperature phases. $La_2Si_2O_7$ exists in a low temperature phase, structure type A, and in a high temperature phase, structure type G. The present investigation concerns possible phase transitions of $La_2Si_2O_7$, structure type G, at 300 K and at pressures up to 212 kbar, and an investigation of the geometry of the silicate ion in dependence of the pressure.

The sample of $La_2Si_2O_7$ was made in a solid state synthesis from a stoichiometric mixture of La_2O_3 (Johnson Matthey) and SiO_2 (Kieselgur, Merck), pressed into pellets and kept twice at 1400°C for 75 h. X-Ray powder patterns of the sample showed that it was $La_2Si_2O_7$, structure type G, with a minor impurity of $La_{9.33}[\ ]_{0.67}(SiO_4)_6O_2$.

The sample used in the high pressure experiments was ground in a B_4C mortar and dispersed in ethanol. After a partial sedimentation of the solid for 5 minutes, the upper half of the suspension was sampled, and the solid $La_2Si_2O_7$ left after evaporation of the ethanol was used in the experiment. Two experiments were performed, one with a methanol-ethanol mixture, and one with nitrogen as the pressure transmitting medium.

The X-ray wave length used was $\lambda = 0.4664$ Å. The powder patterns recorded were used in profile refinements of the structure of $La_2Si_2O_7$. It was possible to fit the patterns to the structure model of $La_2Si_2O_7$, type G, and to the model of the impurity phase $La_{9.33}[\ ]_{0.67}(SiO_4)_6O_2$. Fig. 1 shows observed and calculated powder patterns and a difference plot. However, the data could not yield a detailed description of the geometry of the disilicate ion in dependence of the applied pressure due to the low scattering contributions of the oxygen atoms, compared to those of the lanthanum and the silicon atoms. The Si-Si distance in the disilicate ions shows, however, a significant decrease with pressure.

With applied pressures at and over 166 kbar a significant change in the powder patterns were observed. The background increased, and new diffraction lines appeared. Fig. 2 displays the powder pattern recorded at 212 kbar. This pattern is not sufficiently resolved to be used for a determination of the structure of this phase.

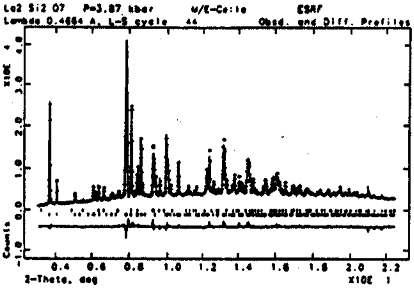

Fig. 1. Powder pattern of $La_2Si_2O_7$ at 3.87 kbar.

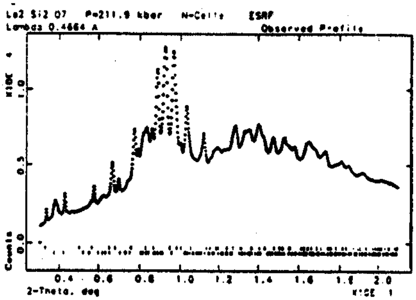

Fig. 2. Powder pattern of $La_2Si_2O_7$ at 211.9 kbar.