



Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

<http://193.49.43.2:8080/smis/servlet/UserUtils?start>

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.



	Experiment title: High-pressure studies on a nitridosilicate and an isotopic oxonitridoaluminosilicate	Experiment number: HS-2364
Beamline: ID09A	Date of experiment: from: 14.04.2004 to: 16.04.2004	Date of report: 31.08.2004
Shifts: 9	Local contact(s): Monica Ambouage, Michael Hanfland	<i>Received at ESRF:</i>

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Report:

A new exciting field in materials science was opened by the development of nitridosilicates and oxonitridosilicates. They can be derived from oxosilicates by a total or partial exchange of oxygen with more covalently behaving nitrogen. This replacement extends the structural possibilities of conventional oxosilicates significantly. Furthermore, the exceptional thermal and chemical stability combined with high mechanical strength make them materials with interesting properties for the materials science community.

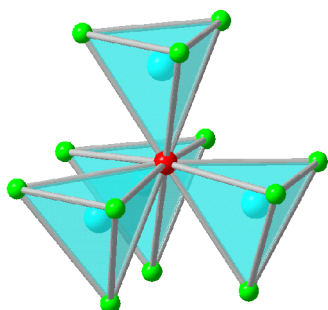


Figure 1: Star-like $[C^{4+}(SiN_3^{2-})_4]$ unit.

Very recently, new nitridosilicates with star-like units $[N^{4+}(SiN_3^{2-})_4]$ of four SiN_4 -tetrahedra sharing a common corner were found [1]. These units are connected to form a condensed three-dimensional network. When replacing the central nitrogen by carbon, carbidonitridosilicates with $[C^{4+}(SiN_3^{2-})_4]$ units are formed [2] (Figure 1). It is expected that these star-like units are not stable at simultaneously high pressures and temperatures and a phase transition that changes the coordination of nitrogen from four to six may occur. This would be the first example of nitrogen being arranged in 6-fold coordination around silicon within a high-pressure nitridosilicate structure.

We planned to measure BaYb[Si₄N₇] and BaYb[SiAl₃O₃N₄] at high pressures after off-line laser annealing the compressed structures at about 2500 K. Unfortunately, laser heating was not available during the experiment due to malfunction of the system. Alternatively, we measured at ambient temperature SrYb[Si₄N₇] and MYb[SiAl₃O₃N₄], M= Sr, Ba, up to pressures of 41 GPa in order to evaluate the effects of cation and anion substitution on the stability and mechanical properties of isotypic nitridosilicate and carbidoaluminonitridosilicates.

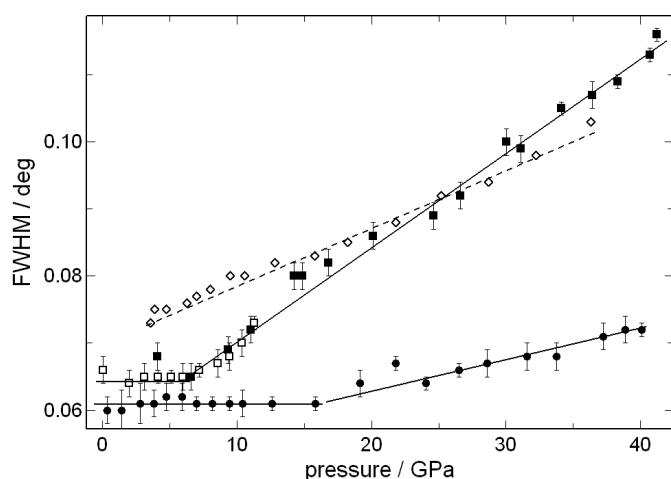


Figure 2: Change of the peak half width with pressure for SrYb[Si₄N₇] (filled circles), SrYb[SiAl₃O₃N₄] (squares), and BaYb[SiAl₃O₃N₄] (diamonds). Open squares correspond to a measurement in N₂, all others were measured in Ne.

The three samples studied showed an exceptional stability of the crystal lattice. No sign of a structural phase transition could be observed. Fig. 2 shows the change of the peak half width with pressure for all samples studied. The main result is a peak broadening above 7 GPa that increases linearly with pressure in SrYb[SiAl₃O₃N₄]. This is interpreted as a strain and seems to be related to the substitution in the anion sublattice since a corresponding effect was not observed in the isotypic compound SrYb[Si₄N₇]. Deviatoric stress, caused by the pressure medium can be excluded as the origin of the peak broadening since the change in the slope of the peak half width was observed with N₂ as well as with Ne as pressure transmitting medium.

The here observed response of the structure to pressure can be seen as a potential structural instability. That may make the SiAlON compound a promising candidate for the observation of the proposed breakdown of the star-like units at higher temperatures, which are supposed to result in a change of the N-coordination from four to six.

- [1] a.) Huppertz, H. and Schnick, W. (1996): *Angew. Chem. Int. Ed. Engl.*, 35, 1983.
 b.) Huppertz, H. and Schnick, W. (1997): *Z. Anorg. Allg. Chem.*, 623, 212.
 c.) Huppertz, H. and Schnick, W. (1997): *Acta Crystallogr. C*53, 1751.

- [2] Höpfe, A.H., Kotzyba, G., Pöttgen, R., and Schnick, W. (2001): *J. Mater. Chem.*, 11, 3300 – 3306.

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