ESRF	**Experiment title:** Structure of Amorphous Si-C-N Ceramics	**Experiment number:** HS-1594
Beamline: ID15B	**Date of experiment:** from: 03.04.02 to: 06.04.02	**Date of report:** 01.08.02
Shifts: 9	**Local contact(s):** Veijo Honkimaki	*Received at ESRF:*

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Report:

The aim of the experiment was to get information about the atomic structure in various amorphous Si-(B)-C-N ceramics produced by thermolysis of suitable precursors such as polysilylcarbodiimide or polysilazane [1]. About 30 Si-C-N and Si-B-C-N ceramics with different chemical compositions and ceramics annealed up to the crystallization temperature were measured. The results from this experiments will be compared with the results from neutron scattering at ISIS and X-ray scattering with Ag-Kα radiation in our Lab [2]. Contrast variation between X-rays and neutrons should be applied because with X-rays mainly the Si-N and Si-Si correlations are observed whereas neutrons show the Si-N, N-N and C-C correlations. Because of the low quality of the measurements with Ag-Kα radiation it is necessary to use synchrotron radiation for the contrast variation.

High intensity synchrotron radiation (89.031 keV, 0.139 Å) was used to get information with high accuracy in short acquisition times. The powder samples were studied in transmission geometry in a 1 mm sample holder with mylar windows. The detection system was a MAR2300 online image plate scanner with a pixel size of 0.15 mm. The resolution and angular range covered could be modified by changing sample-detector distance. Using two positions, 172.5 mm and 332.5 mm, a q-range from 0.7 Å $\leq$ q $\leq$ 34.5 Å could be investigated.

The 1d diffraction patterns were obtained by integration of the 2d diffraction patterns. The measured intensities were corrected for background, compton scattering, absorption and geometrical factors and then normalized to get the structure factor $S(q)$.

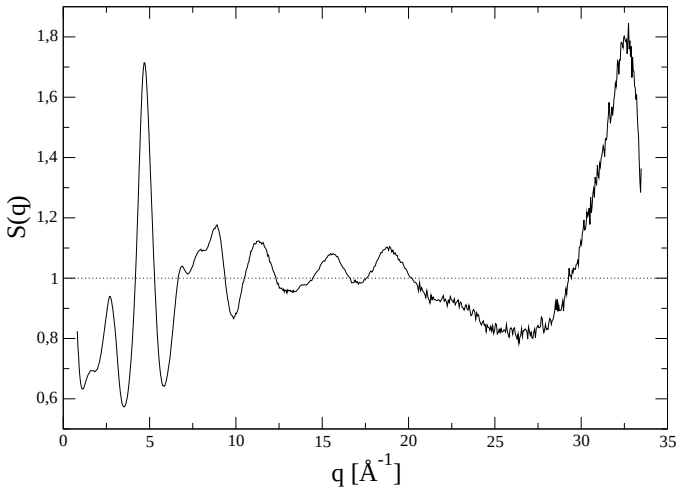

Fig. 1: Structure factor $S(q)$ measured at the ESRF beamline ID15B.

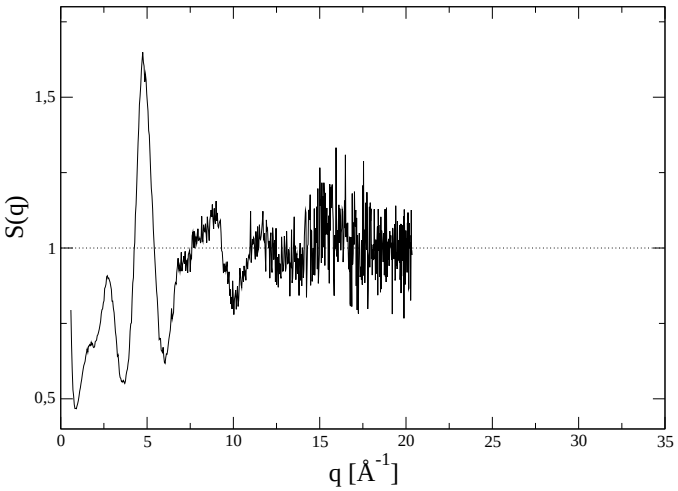

Fig. 2: Structure factor $S(q)$ measured at the MPI Stuttgart with Ag-Kα radiation.

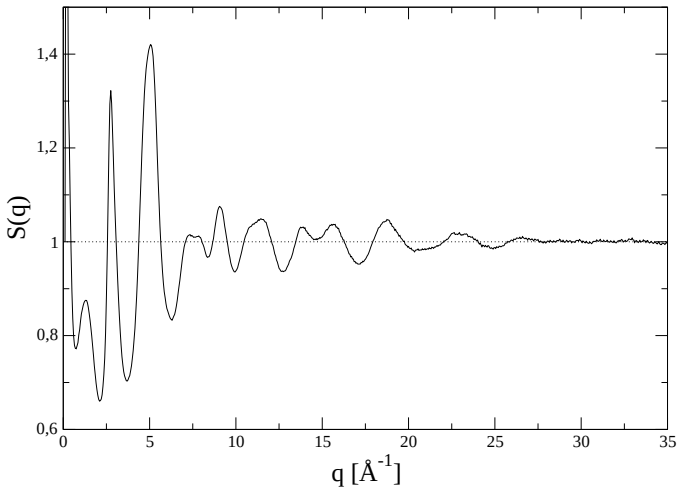

Fig. 3: Structure factor $S(q)$ measured at ISIS with neutrons.

Fig. 1 shows the structure factor $S(q)$ of a Si-C-N ceramic measured at the ESRF. Presently there are still problems to normalize the data with our program at high q values. Comparison with the measurement at MPI (Fig. 2) indicates that it is necessary to use syncrotron radiation for this measurements because $S(q)$ shows structures up to at least q=25 Å^{-1}. Also the acquisition time is very short: At the ESRF we need only about one hour measuring time, with Ag-Kα radiation we need about 4 weeks and the accuracy is still not sufficient. The $S(q)$ of the neutron scattering (Fig. 3) shows the contrast to the X-ray scattering, which will be used to obtain information on partial correlation functions and thus get a better understanding of the atomic short range order in amorphous Si-(B)-C-N ceramics.

References

[1] J. Bill, F. Aldinger, *"Precursor-Derived Covalent Ceramics"*, Adv. Matter 7 (9) 1995, 775-787

[2] S. Schempp, J. Duerr, P. Lamparter, J. Bill, F. Aldinger, *"Study of the Atomic Structure and Phase Separation in Amorphous Si-C-N Ceramics by X-ray and Neutron Diffraction"*, Z. Naturforsch. 53a (3-4) 1998, 127-133