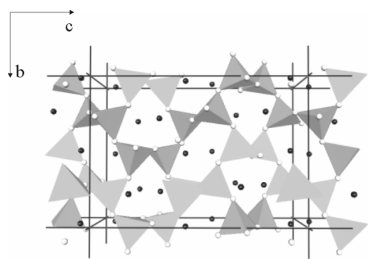


SYNTHESIS AND STRUCTURE OF CaSiN<sub>2</sub>

F. Ottinger R. Nesper

ETH Zurich Laboratorium Fur Anorganische Chemie Wolfgang-Pauli-Strasse  
10 Hci H106 ZURICH 8093 SWITZERLAND

Containing corner-sharing as well as edge-sharing Si<sub>4</sub>-tetrahedra as the main building blocks, very complex Si-N-networks are possible in ternary nitridosilicates. Therefore the structures of nitridosilicates are a significant extension to the structures found in the family of oxosilicates. Although the synthesis of CaSiN<sub>2</sub> has been reported in the 1960s, the structure of CaSiN<sub>2</sub> has not yet been clarified. Ca<sub>7</sub>Si<sub>4</sub>N<sub>10</sub> was received as the main product by reaction of CaSi<sub>2</sub> and Ca<sub>3</sub>N<sub>2</sub> at temperatures of 1400 °C in sealed Niobium ampoules. Crystals of transparent, orange CaSiN<sub>2</sub> were obtained as a by-product. These crystals are embedded in a Calcium matrix, as it is not possible to synthesize them stoichiometrically from only CaSi<sub>2</sub> and Ca<sub>3</sub>N<sub>2</sub>. Moreover, Calcium is acting as a flux and enables the formation of large single crystals. CaSiN<sub>2</sub> crystallizes in the orthorhombic space group *Pbca* (No. 61) with *a* = 5.129(3) Å, *b* = 10.224(1) Å, *c* = 14.821(4) Å, *Z* = 1, *R*(*F*<sub>o</sub>) = 0.0341. CaSiN<sub>2</sub> is isotypic with KFeO<sub>2</sub> and KGaO<sub>2</sub> building a 3-dimensional network of SiN<sub>4</sub>-tetrahedra forming sechser rings. All Si-N[2] distances are within the usual range for nitrido-silicates between 1.719 (6) - 1.778 (6) Å. The Calcium-Calcium distance is 3.052 (1) Å.



**Keywords:** NITRIDO SILICATES, NETWORK STRUCTURE, HIGH TEMPERATURE SYNTHESIS

11x

## OBSERVATION OF DYNAMIC LOCAL LAYER RESPONSE OF SMECTIC LIQUID CRYSTALS BY X-RAY MICRO-DIFFRACTION

A. Iida<sup>1</sup> Y. Takahashi<sup>2</sup> Y. Takanishi<sup>3</sup> M. Nakata<sup>3</sup> K. Ishikawa<sup>3</sup> H. Takezoe<sup>3</sup>

High Energy Accelerator Research Organization Institute of Materials

Structure Science 1-1 O-Ho TSUKUBA IBARAKI 305-0801 JAPAN

<sup>1</sup>Photon Factory, Institute of Materials Structure Science, High EnergyAccelerator Research Organization <sup>2</sup>The Graduate University for AdvancedStudies <sup>3</sup>Department of Organic and Polymer Materials, Tokyo Institute of

Technology

For the direct determination of the microscopic local layer structure in the smectic liquid crystal, the synchrotron X-ray microbeam diffraction technique becomes a powerful tool[1]. The dynamic local layer response of the stripe texture, which appears in the anti-ferro and ferroelectric liquid crystal (AFLC and FLC), was measured in detail under the high electric field.

The experiment was performed at the Photon Factory (BL4A) by the X-ray beam. The diffracted intensities were measured as functions of angles, which correspond to the layer orientation with respect to the normal to the rubbing direction and the surface normal, respectively. The time resolved measurements were carried out with the time resolution from a few fYs to ms.

The samples used were TK-C101(FLC) and TPMHPOBC (AFLC). The layer responses in the SmC\* and SmCa phase were measured for FLC and AFLC, respectively. The electroclinic effect is also measured in the SmA phase of both materials. For FLC cells, the reversible local layer change from the horizontal chevron to the quasi-bookshelf structure was confirmed under the triangular waveform. It was shown that the anchoring effect at the alignment film played an important role for the layer transformation. The local layer relaxation time for the step waveform was dependent on the applied electric field and the sample (AFLC/FLC). The relation of the layer response among samples and phases was discussed.

References

[1] Y. Takahashi et al, Jpn. J. Appl. Phys. 40(2001)3294

**Keywords:** SMECTIC LIQUID CRYSTAL, X-RAY MICROBEAM, TIME-RESOLVED MEASUREMENT

CRYSTAL AND MAGNETIC STRUCTURES OF LAYERED Sr<sub>2</sub>MnGaO<sub>5+x</sub> OXIDESA.M. Balagurov<sup>1</sup> V.Yu. Pomjakushin<sup>1</sup> A.M. Abakumov<sup>2</sup> E.V. Antipov<sup>2</sup> M.V. Lobanov<sup>2</sup> P. Fischer<sup>3</sup> D.V. Sheptyakov<sup>1,3</sup><sup>1</sup>Joint Institut for Nuclear Research Frank Laboratory of Neutron Physics

Frank Laboratory of Neutron Physics Joint Institut for Nuclear Research

DUBNA MOSCOW REG. 141980 RUSSIA <sup>2</sup>Department of Chemistry,Moscow State University, 119899 Moscow <sup>3</sup>Laboratory of Neutron Scattering,

ETHZ&amp;PSI, CH-5232 Villigen, Switzerland

Crystal and magnetic structures of new complex manganese oxides Sr<sub>2</sub>GaMnO<sub>5+x</sub> (*x* = 0, 0.4 and 0.5) were studied by neutron powder diffraction. The crystal structure of the compound with *x* = 0 belongs to a brownmillerite type and consists of alternating SrO, MnO<sub>2</sub>, SrO and GaO<sub>1+x</sub> layers with *Ima2* space symmetry. Mn magnetic moments are aligned antiferromagnetically in all directions below *T*<sub>N</sub>=180 K (G-type order). The oxidized compound with *x* = 0.5 has a perovskite-like crystal structure with a tetragonal unit cell of *P4/mmm* space symmetry. The GaO<sub>1+x</sub> layers are partially filled. Its magnetic structure (*T*<sub>N</sub> = 108 K) preserves AFM ordering within the MnO<sub>2</sub> plane, however, the interlayer coupling becomes ferromagnetic (C-type order). The average crystal structure of *x* = 0.4 sample can be satisfactorily described in monoclinic *P2/m* space group but two-phase state could not be excluded. The latter is indicated by two-step magnetic phase transition: appearance G-type structure at 140 K and then C-type structure at 110 K. Despite the distances between neighbouring in-plane and out-of-plane Mn atoms are very different (3.8 Å and 8 Å, respectively) a magnetic structure of all compounds has a 3D character.

**Keywords:** MANGANITES, LAYERED STRUCTURE, NEUTRON DIFFRACTION

## DETERMINATION OF PHASES AND PHASE TRANSITIONS IN LIQUID CRYSTALS

A.K. Schaper<sup>1</sup> Z. Zhao<sup>1</sup> W. Ruland<sup>1</sup> B. Albert<sup>2</sup><sup>1</sup>Philipps University Materials Science Center Dept of Geosciences Hans-Meerwein-Str. MARBURG 35032 GERMANY <sup>2</sup>Department of Chemistry,

Justus Liebig University, 35392 Giessen, Germany

From certain liquid crystal (LC) compounds it is known that they pass through an intermediate hexatic-B (SmBHex) phase during transition from the crystalline smectic-B (SmB) phase to the liquid-like smectic-A (SmA) phase. This hexatic phase is characterized by long-range bond orientational order over macroscopic dimensions, but only short-range positional order propagating over not more than a few tens of nanometers. The hexagonal bond orientational order correlations extend over a three-dimensional stack of two-dimensional hexatic layers which exhibit only weak interlayer coupling. In this paper, direct proof of the hexatic order in a cyclohexyl derivative of a biphenylic type compound is provided by *in situ* low-dose selected area electron diffraction (SAED). Further information on the phase transitional behavior of the LC material with the bulk transition sequence SmB - SmBhex - SmA - N - I is gained from light microscopy observations, calorimetric measurements, small and wide angle x-ray scattering (SAXS, WAXS), and from x-ray density fluctuation measurements, in particular. The particle density fluctuation of condensed matter is caused by thermal motion of the atoms or molecules, and is influenced in a definite manner by the presence of structural defects and lattice disorder. Therefore, the relationship between the density fluctuation and the x-ray scattering intensity extrapolated toward zero angle may serve as quantitative measure of the state of order and of the strength of particular defect populations in solids. For the first time, we apply this method to characterizing the phase transitional behavior of an LC material.

**Keywords:** LIQUID CRYSTALS PHASE TRANSITIONS DIFFRACTION AND SCATTERING