

# HEXAGONAL BORON NITRIDE AS A PRESSURE TRANSMITTING MEDIUM IN THE HIGH-PRESSURE HIGH-TEMPERATURE X-RAY DIFFRACTION EXPERIMENT

**P. Piszora**<sup>1\*</sup>, **W. Nowicki**<sup>1</sup>, **J. Darul**<sup>1</sup>, and **C. Lathe**<sup>2</sup>

<sup>1</sup> Faculty of Chemistry, Adam Mickiewicz University, Grunwaldzka 6, 60-780 Poznan, Poland

<sup>2</sup> Helmholtz Centre Potsdam, GFZ German Research Centre for Geosciences, Telegrafenberg, 14473 Potsdam, Germany

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\*) e-mail: pawel@amu.edu.pl

Hexagonal boron nitride (*h*-BN) powder is a solid lubricant that compares favourably with, and in some cases exceeds, the performance of other solid lubricants. Its high-temperature stability, low coefficient of friction, extreme pressure performance, high thermal conductivity, high electrical resistivity, inertness in a wide variety of chemical environments, and environmental friendliness make *h*-BN an ideal alternative to other solid lubricants in many high performance applications.

Boron nitride has long been used as a crucible material in inert environments at high temperatures. It has found application as a fibber coating in ceramic-matrix composites. In such applications, it is likely to be exposed to high-temperature oxidizing conditions. However, there are many questions about its oxidation behaviour. The literature contains numerous studies of BN oxidation [1,2], which indicate the oxidation behaviour of BN is complex and dependent on structure.

In our experiment, the hexagonal boron nitride was tested as the pressure-transmitting medium at high-pressure high-temperature conditions in the mixture with LiMn<sub>2</sub>O<sub>4</sub>.

Lithium-manganese oxide (*Fd3m* space group) sample was obtained by conventional solid-state reaction of Li<sub>2</sub>CO<sub>3</sub> with  $\alpha$ -Mn<sub>2</sub>O<sub>3</sub> (*Ia3*, bixbyite structure) at 1073 K. The synchrotron X-ray powder diffraction data, collected at beamline B2 (Hasylab), at ambient conditions confirmed the cubic spinel structure. The samples were mounted in *h*-BN cylinder, in boron-epoxy cube with the edge length 8 mm. The cube was located in between six anvils of the MAX80 press. In the high-pressure investigations a level of the pressure hydrostaticity due to applied pressure medium is very important [3]. The hexagonal boron nitride is considered to be a relatively 'soft' material and has been often used as the pressure medium; thereat we decided to perform high-pressure experiments on LiMn<sub>2</sub>O<sub>4</sub>/*h*-BN, with the weight ratio 1:2, in order to get quasihydrostatic conditions. The measurements were performed with the energy dispersive experimental setup and a white synchrotron radiation at F2.1 beamline (Hasylab). The

X-ray detection system was based on a germanium solid-state detector of resolution 135 eV at 6.3 keV. The sample was gradually compressed to required pressure of 3 kbar, at ambient temperature, and then it has been heated quickly to 500°C. After 15 minutes of heating, the sample was quenched by the switching off the power (the initial cooling rate was about 300°C/s). The structural changes have been monitored *in situ* with the energy dispersive XRD method. After the pressure releasing, the recovered sample has been analyzed by powder X-ray diffraction using a Huber image foil camera 670 with a capillary holder at I711 beamline in Max-lab (Lund).

Successful fitting of the XRD pattern of recovered sample with the Rietveld method revealed presence of LiMnBO<sub>3</sub>, *h*-BN and MnO crystal phases. This phase composition enables to imply conclusion that, at applied high-pressure/high-temperature conditions, *h*-BN is not chemically resistant to oxidation in the presence of oxide sample. Furthermore, the hexagonal LiMnBO<sub>3</sub> has been recently proposed as a new cathode material for lithium-ion batteries [4] and its HP/HT synthesis route can appear as technologically important. The sample container made of *h*-BN could be applied for oxides in HP/HT experiments only with a special precaution.

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