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Guillermo Esparza, Esparza
Harvey Mudd College

Patricia Kalita
University of Nevada, Las Vegas, patrizia@physics.unlv.edu

Andrew Cornelius
University of Nevada, Las Vegas, cornel@physics.unlv.edu

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Esparza, Guillermo Esparza,; Kalita, Patricia; and Cornelius, Andrew, "Neutron diffraction of NaBD4: Phase transition, Rietveld Structure refinements, and equation of state" (2011). *Undergraduate Research Opportunities Program (UROP)*. 6.
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Neutron Diffraction of $NaBD_4$: Phase Transition, Rietveld Structure Refinements, and Equation of State

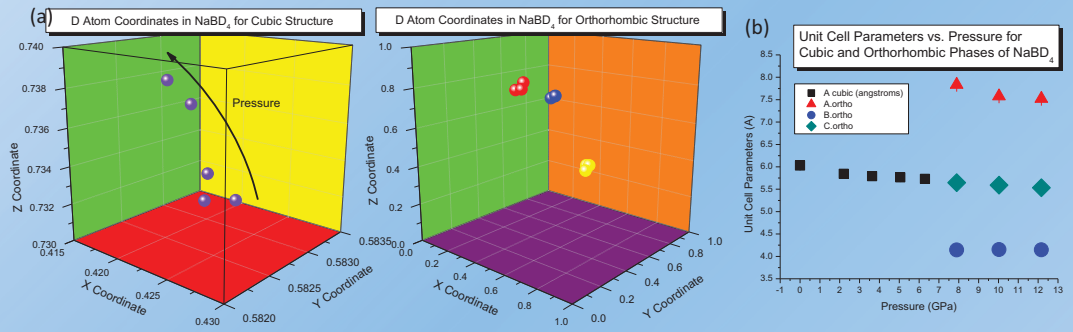
Guillermo Esparza, Patricia Kalita, Professor Andrew Cornelius
Department of Physics and Astronomy
University of Nevada Las Vegas, NV 89154

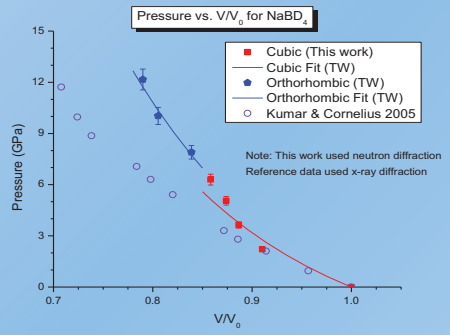
BACKGROUND

$NaBH_4$ is a hydride with possible applications as a hydrogen storage material for future renewable energy technologies. It's dehydrogenation properties are enhanced with the mixture of particular catalysts through ball-milling techniques during which local pressures may exceed several GPa's. It is for this reason that understanding the behavior of pressure induced phase changes of its crystalline unit cell is an area of interest.

RESULTS

Analysis of the neutron diffraction data using Rietveld Refinement showed a phase transition occurring into the orthorhombic phase between 6.3 and 7.9 GPa. However, this appeared to occur from the cubic phase, as the intermediate tetragonal structure mentioned in other literature to appear between 6.3 and 8.9 GPa was not observed. Atom coordinates were determined for deuterium, demonstrating only a small amount of drift, and are showcased in Fig.1 (a) for the cubic and orthorhombic structures. The Pressure vs. Volume data is also shown



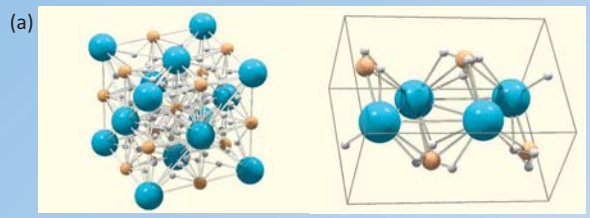


Structure	Reference	B_0 (GPa)	B'	V_0 (Å^3)
$NaBD_4$ Cubic	This work	24+/-2	4.5+/-1.4	218.0+/-1.6
	Kumar & Cornelius 2005	19.9	3.5	237.5
$NaBD_4$ Orthorhombic	This work	31+/-0.6	4	218.5+/-0.6
	Kumar & Cornelius 2005	31	3.9	

Fig.2 (a). 3D plots displaying the shift of deuterium atom positions within the unit cell. (b). A plot of unit cell parameters with a dependence on pressure for both observed phases of $NaBD_4$. (c) Plot of Pressure vs. volume data along with equations of state for both phases and a table displaying the determined values for B, B', and V_0 .

EXPERIMENTAL PROCEDURE & DATA ANALYSIS

This study makes use of neutron diffraction data collected from $NaBD_4$ up to about 12 GPa. The sample was held in a Paris-Edinburgh cell in non-hydrostatic pressure conditions. The program Topaz was used to perform Rietveld Refinement on the data, and external data on the structure and atom positions of Na and B was attained in order to determine the atom positions of hydrogen (in this case its isotope deuterium) within the unit cell. Volume vs. Pressure data was also collected in order to attain an appropriate equation of state and from it determine the compound's bulk modulus.



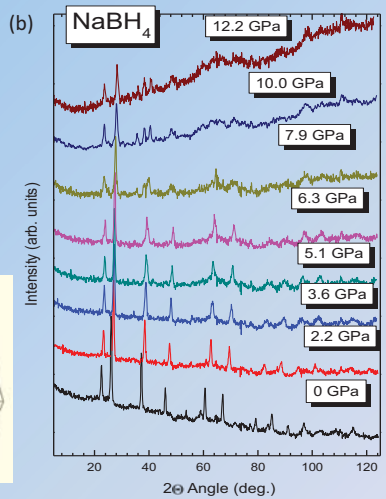


Fig.1 (a) Unit cell structure for the cubic phase (left) and orthorhombic phase (right). (b) Representative neutron diffraction patterns at various pressures up to 12.2 GPa for $NaBD_4$ in non-hydrostatic pressure conditions.

CONCLUSIONS AND SUMMARY

When $NaBD_4$ is studied at high pressures up through 12.2 GPa, shifting of peaks to a higher 2θ in neutron diffraction patterns indicates a smaller d-spacing as the unit cell is compressed. The appearance of new peaks also indicates at least one phase transition to lower symmetry somewhere between 6.3 and 7.9 GPa, determined to be from a cubic Fm-3m space group to an orthorhombic Pnma one. The resulting pressure vs. volume data was used to fit a 3rd order Birch-Murnaghan equation of state to the cubic phase and a 2nd order one to the orthorhombic phase, with the determined values of B, B', and V_0 displayed in Fig. 2 (c).

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ACKNOWLEDGEMENTS

Guillermo Esparza would like to thank Professor Ravhi S. Kumar, Daniel Antonio, and Jason Baker from UNLV for constant support and encouragement. The UNLV High Pressure Science and Engineering Center was supported by the U.S. Department of Energy, National Nuclear Security Administration, under cooperative agreement number DE-FC52-06NA27684. Support from the REU program of the National Science Foundation under grant DMR-1005247 is gratefully acknowledged.