

s6.m21.p6 **Al substitution in MgB<sub>2</sub> single crystals: influence on structural properties and phase separation.** G. Schuck, N.D.Zhigadlo, S.M. Kazakov, J. Jun, J. Karpinski, *Solid State Physics Laboratory ETH 8093 Zürich, Switzerland.* E-mail: schuck@solid.phys.ethz.ch

**Keywords: Superconductivity; Phase separation; Image plate**

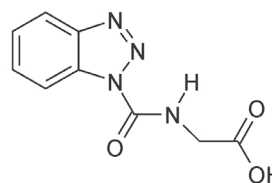
Superconducting single crystals of Mg<sub>1-x</sub>Al<sub>x</sub>B<sub>2</sub> phase have been grown at a pressure of 30 kbar using cubic anvil technique [1]. Single crystal X-ray and HRTEM investigations show the existence of macroscopic phase separation in some crystals. This is in a form of non-superconducting MgAlB<sub>4</sub> layers parallel to (hk0) plane of Mg<sub>1-x</sub>Al<sub>x</sub>B<sub>2</sub>. In some crystals with high Aluminum content also a third phase is observed in (0kl) projections of the reciprocal space. For crystals with high aluminum content we were able to refine simultaneously the Mg<sub>1-x</sub>Al<sub>x</sub>B<sub>2</sub> and the MgAlB<sub>4</sub> crystal structure using image plate intensity data. Both phases are hexagonal, space group P6/mmm. MgAlB<sub>4</sub> is a superstructure of MgB<sub>2</sub> accompanied by doubling of the c axis and ordering of Magnesium and Aluminum in alternating layers. The third phase is also hexagonal; a = 2.9 Å and c = 7.1 Å. Most of the x-ray measurements were carried out on a Mar-300 Image Plate system; additional measurements with a CAD-4 and a Siemens P4 diffractometer were carried out. Precipitation free crystals were grown with modified crystal growth procedure. Critical temperature versus Aluminum content dependence of single crystals show different behavior from that of polycrystalline samples, because even small substitution of 1 % Aluminum decreases T<sub>c</sub> by about 3 K. Lattice parameter c decreases with Aluminum content more than a parameter, however a miscibility gap x > 0.1 is observed.

[1] J. Karpinski, S.M. Kazakov, J. Jun, N.D. Zhigadlo, M. Angst, R. Puzniak, A. Wisniewski, *Physica C* in print, cond mat/0304658.

s6.m22.p1 **Polymorphism of N-(1-Benzotriazolylcarbonyl)-Glycine.** Miroslav Zegarac,<sup>a</sup> Marijana Zovko,<sup>b</sup> Nada Kosutic-Hulita,<sup>a</sup> Biserka Prugovecki,<sup>c</sup> Ivan Butula<sup>b</sup> and Miljenko Dumic<sup>a</sup>, <sup>a</sup>PLIVA - Research Institute Ltd., Prilaz baruna Filipovica 29, 10 000 Zagreb, Croatia, <sup>b</sup>Faculty of Pharmacy and Biochemistry, University of Zagreb, Ante Kovačevića 1, 10 000 Zagreb, Croatia, and <sup>c</sup>Laboratory for General and Inorganic Chemistry, Faculty of Science, University of Zagreb, Kralja Zvonimira 8, 10 000 Zagreb, Croatia. E-mail: miroslav.zegarac@pliva.hr

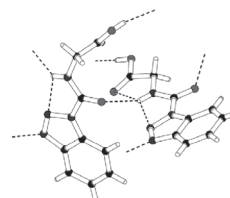
**Keywords: Polymorphism; N-(1-benzotriazolylcarbonyl)-glycine; Solvates**

As a part of an ongoing study on small peptides, we used N-(1-benzotriazolylcarbonyl)-glycine (**N-BtCO-Gly-OH**) as a valuable building block.[1]- [3] It is the only one, from the series of such N-BtCO-amino acids and other N-(1-azolylcarbonyl)- amino acids investigated, that gave hydrated product by crystallization from aqueous solvents.



N-BtCO-Gly-OH

Performed structural studies (IR, XRPD, DSC) showed that anhydrous **N-BtCO-Gly-OH** was obtained in two polymorphic forms: microcrystalline needles obtained by crystallization from toluene (form A) and plates obtained by saturation of **N-BtCO-Gly-OH** acetonitrile solution with water (form B). Single-crystal x-ray diffraction of later showed strong intra- and intermolecular hydrogen bonding.



Hydrogen bonding of **N-BtCO-Gly-OH** (form B)

Essential crystallographic data for the anhydrous **N-BtCO-Gly-OH** (form B):

C<sub>9</sub>H<sub>8</sub>N<sub>4</sub>O<sub>3</sub>, M<sub>r</sub> = 220.19, Monoclinic P2<sub>1</sub>/n, Z = 4 × 2, a = 7.456(2), b = 15.860(2), c = 16.625(3) Å, β = 95.31(2)°, V = 1957.5(7) Å<sup>3</sup>, λ(Mo Kα) = 0.7107 Å, R<sub>F</sub> = 0.0526, wR<sub>F</sub><sup>2</sup> = 0.1241 and S = 1.008 for 297 parameters and 3661 observed reflection with I > 2σ(I).

However, crystallization of **N-BtCO-Gly-OH** from acetone-water leads to the formation of monohydrated product (form H), which loses water on heating at 86-115 °C. Form H crystallize in orthorhombic space group P2<sub>1</sub>2<sub>1</sub>2<sub>1</sub> with the unit cell parameters a = 4.823(5) Å, b = 14.0493(2) Å, c = 15.2980(8) Å, and α = β = γ = 90°, characterized by strong intra- and intermolecular hydrogen bonding and IR bands ν(-CO-) at 1711 and ν(NH) at 3357 cm<sup>-1</sup> and two sharp n(OH) bands at 3573 and 3455 cm<sup>-1</sup>, respectively.

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[2] I. Butula, B. Zorc, M. Ljubic, *Synthesis*, 1983, 327-329.

[3] I. Kalcic, M. Zovko, M. Jadrijevic-Mladar Takac, B. Zorc, I. Butula, *Croat. Chem. Acta*, **76** (2003), 217-228.