

**P.11.01.1***Acta Cryst.* (2005). A61, C385**Structure/Properties Relationships in doped MgB<sub>2</sub> Single Crystals**Götz Schuck<sup>a</sup>, M. Wörle<sup>b</sup>, N.D. Zhigadlo<sup>a</sup>, K. Rogacki<sup>a</sup>, J. Karpinski<sup>a</sup>,  
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MgB<sub>2</sub> is a two electronic-bands, two energy gaps superconductor with a high T<sub>c</sub> of 39K and unusual properties such as temperature and field dependent anisotropy.

Superconducting hexagonal single crystals of pure and Al, C, Mn and Fe doped MgB<sub>2</sub> phase have been grown at a pressure of 30 kbar using cubic anvil technique [1-2] to study the intrinsic properties of MgB<sub>2</sub>. The superconducting transition of doped MgB<sub>2</sub> single crystals can be tuned in a wide temperature range between 10 and 39 K by adjustment of the nominal composition. Al [1], Mn and Fe are substituted on the Mg position of MgB<sub>2</sub> and C [2] on the B position. Introduction of disorder by substitution is partly observed.

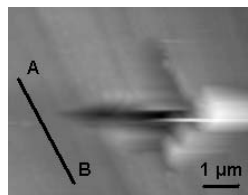
In order to elucidate structure/properties relationships we have carried out x-ray single crystal measurements on doped MgB<sub>2</sub> single crystals (the composition varies between 1 and 15 % doping material). Additional temperature dependent measurements on pure MgB<sub>2</sub> and Mn doped MgB<sub>2</sub> has been carried out.

[1] Karpinski J., Zhigadlo N. D., Schuck G., Kazakov S. M., Batlogg B., Rogacki K., Puzniak R., Jun J., Muller E., Wagli P., Gonnelli R., Daghero D., Ummarino G. A., Stepanov V. A., *Phys. Rev. B*, submitted, cond mat/0411449.  
[2] Kazakov S. M., Puzniak R., Rogacki K., Mironov A. V., Zhigadlo N. D., Jun J., Soltmann Ch., Batlogg B., Karpinski J., *Phys. Rev. B*, 2005, **71**, 024533.

**Keywords:** high-T<sub>c</sub> superconductivity, structure-physical properties relationships, X-ray crystal structure analysis

**P.11.01.2***Acta Cryst.* (2005). A61, C385**The Investigation of Crack Propagation in Cleavage Directions on the Surface of SiC by Sclerometry**Alexander Soshnikov, Kirill Gogolinsky, Vladimir Blank, *Technological Institute for Superhard and Novel Carbon Materials.* E-mail: soshn@yandex.ru.

The SiC crystals have been scratched using SPM-Nanoindenter Nanoscan [1]. The studied samples represent thin hexagonal plates with the natural grown surface. The values of hardness have been measured for "Si" (23±3 GPa) and "C" (34±4 GPa) sides of the H6-SiC samples with nitrogen impurity of 1x10<sup>18</sup> cm<sup>-3</sup>. The effect of microcracks along the cleavage direction {1 100} (marked AB) occurs with the load about 10 mN, that is shown on the image. Secondary microcracks propagate from the cracks in direction of secondary cleavage {11 20}. The width of cracks is in range of 300-600 nm, depth up to 60 nm. The cracks are developed during the scratching with the "face forward" indenter arrangement. The direction "edge forward" does not develop scratches with cracks, but the effect of periodic pile-up's is present. The period is in range of 300-600 nm for given loads. The samples have been turned to achieve



various direction of scratching. The differences found between the behaviours of cracks around the scratches made in various directions and different indenter orientations.

[1] Blank V., Popov M., Lvova N., Gogolinsky K., Reshetov V., *J. Mater. Res.*, 1997, **12**, 3109.

**Keywords:** scanning probe microscopy, hardness, structure defects

**P.11.01.3***Acta Cryst.* (2005). A61, C385**Superstructures of Pb-free and Pb-doped Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>2</sub>Cu<sub>3</sub>O<sub>10</sub> Superconducting Phases**Enrico Giannini<sup>a</sup>, Roman Gladyshevskii<sup>b</sup>, Radovan Cerny<sup>a</sup>, René Flükiger<sup>a</sup>, <sup>a</sup>*University of Geneva, Switzerland,* <sup>b</sup>*Ivan Franko Natl.*

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The structures of Bi-2223 and Bi,Pb-2223 were studied by single-crystal XRD. The structures are characterized by incommensurate modulations, however, they can be conveniently described in 5-fold supercells. An additional O atom was found to be inserted in the Bi-O chains, at the level of approximately every 10<sup>th</sup> cation, which defines the translation unit of the modulation wave. A partial substitution of Ca by Bi was also observed in both crystals, and the actual compositions of the crystals were Bi<sub>2.16</sub>Sr<sub>2</sub>Ca<sub>1.84</sub>Cu<sub>3</sub>O<sub>10.17</sub> (*P222*, *a* = 27.105(4), *b* = 5.4133(6), *c* = 37.010(7) Å) and (Bi<sub>1.89</sub>Pb<sub>0.22</sub>)Sr<sub>2</sub>Ca<sub>1.89</sub>Cu<sub>3</sub>O<sub>10.18</sub> (*Pnnm*, *a* = 26.976(7), *b* = 5.4130(10), *c* = 37.042(11) Å), respectively. The structures of the Bi<sub>2</sub>Sr<sub>2</sub>Ca<sub>n-1</sub>Cu<sub>n</sub>O<sub>4+2n+δ</sub> series have a strong 2D character with weak interactions between the BiO layers. In the orthorhombic superstructure of Bi-2223 (*n* = 3), the longitudinal displacement waves of the Bi atoms in two neighboring BiO layers are in phase and, consequently, the transverse waves are out of phase (shifted by 180°). For the monoclinic superstructures of Bi-2212 (*n* = 2) and Bi-2201 (*n* = 1), the phase differences between the transverse waves of the Bi atoms in consecutive slabs are 160 and 72°, respectively. The amplitude of the transverse displacement wave increases with decreasing thickness of the slabs (0.139(7) Å in Bi-2223, 0.156(6) Å in Bi-2212, and 0.310(7) Å in Bi-2201).

**Keywords:** high-T<sub>c</sub> superconductor, modulated structure, supercell

**P.11.01.4***Acta Cryst.* (2005). A61, C385**Presence of an Ionic Charge ordering at the Verwey Transition in Fe<sub>3</sub>O<sub>4</sub>: A Resonant X-ray Diffraction Study**J. Emilio Lorenzo<sup>a</sup>, E. Nazarenko<sup>a</sup>, Y. Joly<sup>a</sup>, J.L. Hodeau<sup>a</sup>, D. Mannix<sup>b</sup>, C. Marin<sup>c</sup>. <sup>a</sup>*Lab. Cristallographie, CNRS, Grenoble, France.* <sup>b</sup>*Xmas, ESRF, Grenoble.* <sup>c</sup>*DRFMC-SPSMS, CEA-Grenoble, France.* E-mail: emilio.lorenzo@grenoble.cnrs.fr

Magnetite, Fe<sub>3</sub>O<sub>4</sub>, is a mixed valence system that exhibits many interesting properties, some of them known since the early times. Moreover, magnetite is also on the spot of physicists because of the lack of consensus as to the nature of the metal-insulator transition occurring at T<sub>v</sub>=120K (Verwey transition). The real question that remains largely open is the amount of charge, δ, that is going to localize at the octahedral metal sites giving rise to iron charge states of the type Fe<sup>2.5±δ</sup>. To this end we have carried out a series of resonant X-ray diffraction (RXD) experiments in the neighborhood of the Fe K-edge that have revealed distinct signatures of a small charge ordering (CO) compatible with the symmetry of the low temperature structure. The magnitude of the charges, δ ≈ 0.15 e<sup>-</sup>, has been determined through a refinement of the energy dependence around the of the Fe K-edge of the line shape of 30 selected reflections of the low temperature structure. Our results, in agreement with bond valence sums calculations, are in striking contradiction with previous RXD experiments that have concluded on the absence of any CO in magnetite. This small value of the charge is of fundamental importance and strengthen the argument that covalency effects play a major role in the physics of these strongly correlated compounds. In this paper the strength and limitations of RXD will be discussed as well as, hinting why CO was not observed in previous experiments.

**Keywords:** charge transfer, iron oxides, DAFS

**P.11.01.5***Acta Cryst.* (2005). A61, C385-C386**Crystal Chemistry and Crystallography of the Ba<sub>2</sub>RCu<sub>3</sub>O<sub>6+x</sub>-SrTiO<sub>3</sub> System**Winnie Wong-Ng<sup>a</sup>, Zhi Yang<sup>a</sup>, James Kaduk<sup>b</sup>, Qing Huang<sup>a</sup>, Lawrence Cook<sup>a</sup>, <sup>a</sup>*Materials Science and Engineering Laboratory, NIST, Gaithersburg, MD.* <sup>b</sup>*BP-Amoco, Naperville, IL, USA.* E-mail: winnie.wong-ng@nist.gov

Continued world-wide research in high T<sub>c</sub> superconductors has led to the promise of a wide variety of industrial applications. To