Al substitution in MgB₂ single crystals: influence on structural properties and phase separation

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Superconducting single crystals of Mg_{1-x}Al_xB₂ 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 MgAlB₄ layers parallel to (hk0) plane of Mg_{1-x}Al_xB₂. 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_{1-x}Al_xB₂ and the MgAlB₄ crystal structure using image plate intensity data. Both phases are hexagonal, space group P6/mmm. MgAlB₄ is a superstructure of MgB₂ 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_c 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.