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HIGH-PRESSURE SYNTHESIS OF NANOSTRUCTURED SUPERCONDUCTING MATERIALS BASED ON MAGNESIUM DIBORIDE

Correlations between a nonuniform nanostructure state and superconducting properties have been studied for the MgB_2-based material fabricated using a high-pressure synthesis. The investigated samples were fabricated from the Mg and B powder mixtures (with or without Ti, Ta and SiC admixtures) in recessed-anvil high-pressure apparatuses with a volume close to 330 cm^3. The temperature interval of the synthesis was 600–1200°C, the pressure was up to 2 GPa. The high-pressure synthesis of the MgB_2 ceramic provides possibility to solve the problem of the unwanted magnesium volatility during the synthesis and, as a result, to fabricate high-strength magnesium-diboride specimens with specific density close to the theoretical one and exhibiting high superconducting characteristics.

Mechanisms of the structure formation from the point of view of their responsibility for the pinning mechanism of the Abrikosov vortices in the investigated materials are discussed. The observed change of the oxygen distribution in the MgB_2 matrix structure synthesized under various temperatures is followed by the corresponding change of the external magnetic field dependence of the critical current density. Clear correlations between the synthesis conditions, crystal structure of MgB_2 and MgO phases, as well as between superconducting properties of the fabricated samples is observed.

The structure formation mechanism (according to the multistep crystallization scheme) creates nonuniform nanostructure state of the massive samples. Various structure investigations (complementing each other) give us a possibility to understand the high pressure synthesis peculiarities of the MgB_2 material. The existence of two phases in the massive superconducting material (synthesized under high pressures) does nor create more difficulties for fundamental investigations but gives some advantageous for realization of applied tasks in the field of the large current density increase.

Keywords: magnesium diboride, high pressure synthesis, nanostructural inclusions, critical current density

Fig. 1. Magnetic field dependences of the critical current density j_c of the samples synthesized at 2 GPa for 1 h from Mg and B, taken in the MgB_2 ratio: *а* – from B(I) at 1050°C (■) and from B(II) at 600°C (□); *б* – from B(I) at 1050°C (■) and at 800°C (□); *в* – from B(I) with 10 mass% SiC at 1050°C (■) and from B(II) at 600°C (□); *г* – from B(III) with 10 mass% Ti at 1050°C (■) and at 800°C (□). Working temperature *T*, K: *1* – 10, *2* – 20, *3* – 25, *4* – 30, *5* – 33, *6* – 35

Fig. 2. Composition images (backscattering electron images) of HPS MgB_2-based materials synthesized from Mg and B taken in MgB_2 stoichiometry at 2 GPa for 1 h: *а*, *б* – from B(III) at 800 and 1050°C, respectively; *в* – from B(III) with 10 mass% Ti at 800°C; *г* – from B(III) with 10 mass% Ta at 1050°C; *д*, *е* – from B (III) with 10 mass% Ti at 1050°C under different magnification; *ж*, *з* – from B(I) with 10 mass% SiC at 1050°C under different magnification

Fig. 3. Backscattering electron image and analysis of elements distribution over the area of the HPS MgB_2-based sample from Mg and B(III) with 10 mass% of Ti taken in MgB_2 stoichiometry at 2 GPa, 1050°C, for 1 h: *а* – electron image; *б*, *в*, *г* – distribution of boron, oxygen and magnesium, respectively. The brighter area, the higher is amount of the studied element

Fig. 4. X-ray patterns of the MgB_2 sample synthesized from B(I) with 10 mass% SiC (*а*) and without (*б*): ■ – MgB_2, ● – MgO, ◆ – SiC, ▲ – MgB_{12}

Fig. 5. Experimental data of the HPS Mg–B based sample synthesized from Mg and B taken in 1:8 ratio at 2 GPa, 1200°C for 1 h: *а* – backscattering electron images; *б* – magnetic field dependence of critical current density j_c of the sample at different temperatures T, K: *1* – 10, *2* – 20, *3* – 25, *4* – 30, *5* – 33, *6* – 35; *в* – X-ray patterns of the sample: 1, 2, 3 – phase reflexes of MgB_2, MgO, MgB_{12}, respectively (in the insert, the temperature dependence of the sample magnetic susceptibility)