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ELECTROCHEMICAL SYNTHESIS OF GdB_6 NANOTUBES

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Two types of nanotube materials in chemistry are of particular interest nowadays: carbon nanotubes and diborides like MgB_2, CaB_2, ZrB_2.

It is seen from literature [1] that electrosynthesis of rare earth metal borides is effective method, but there is no data concerning borides of gadolinium, which are widely used in special alloys metallurgy, laser technique and especially perspective for the neutron adsorption screens preparation. Herein the results are reported on the applicability of the high temperature electrochemical synthesis to gadolinium boride manufacture.

NaCl-KCl-NaF(10wt.%)-GdF_3-KBF_4 molten system was investigated in the 973-1023 K temperature range.

The nature of the separate and joint electrodeposition of the boride components was studied by means of linear sweep, cyclic voltammetry and steady-state voltammetry and parameters of gadolinium hexaboride synthesis were worked out.

Waves on the voltammograms in NaCl-KCl-NaF-GdF_3-KBF_4 melt (Fig. 1b) reflected the boron reduction from BF_4^- complexes (R_B , the least negative wave at about –0.8…-1.0 V) and gadolinium discharge on a boron deposit (R_{Gd-B}, at about –1.8 V). The most negative wave (R_{Gd} , at about -2.5 V) corresponded to that of gadolinium discharge on Mo electrode in electrolyte without boron (Fig.1a). This wave has disappeared soon after first additions of boron (when B/Gd=0.5). Since then only two waves presented on voltammograms within the studied boron to gadolinium molar ratio (i.e. 0.5 to 6). Semi-logarithmic dependencies analysis of the E versus $\log(I_d - I)$ and E versus $\log[(I_d - I)/I]$ indicated that the discharge followed the relationship first proposed by Frumkin [2] for irreversible process.

Potentiostatic electrolysis (7 hours duration) at the potential –1.7 V of Gd-B wave peak (Fig. 1b) yielded fine grayish-black powder.

X-ray analysis evidenced the homogeneity in structure and composition of the produced gadolinium boride. Its dispercity does not give in to that of the powders produced by more expensive gas-phase or plasma-chemical methods.

The morphology of the product has been defined by electronic scan microscopy (Fig.2) as follows:
- fine coral-like dendrites;
- micro- and nanotubes of the different diameter to length ratio.

1. G. Xie, K. Ema, Y. Ito, and Z.M. Shou, J. Appl. Electrochem., **23**, 1993, p.753.
2. Ya.Geirovskiy, Ya.Kuta, *Principles of polarography*, N.-Y.: Academic, 1966.

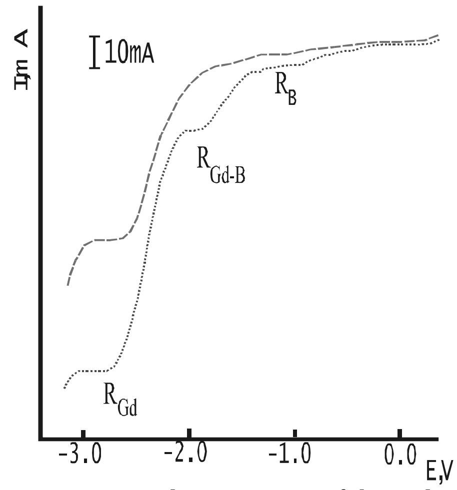

Figure 1. Voltammograms of the molten systems: a – NaCl-KCl-NaF (10wt.%)-GdF_3 ($1.6\cdot10^{-4}$ $mol\cdot cm^{-3}$); b – NaCl-KCl-NaF(10wt.%)-GdF_3-KBF_4 (molar B/Gd=0.25); Mo-working electrode, GC-counter electrode, GC-reference electrode; 1023 K, $\nu=0.1\ V\cdot s^{-1}$

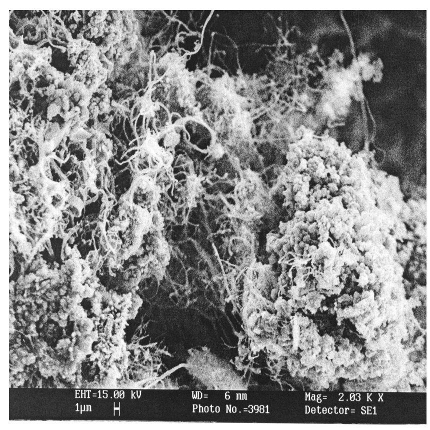

Fig.2 SEM of the electrosynthesized gadolinium boride