Amorphous Iron–Boron Powders prepared by Chemical Reduction of Mixed-metal Cation Solutions: Dependence of Composition upon Reaction Temperature

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By altering reaction temperature from −7 to 30 °C, the boron content in Fe–B amorphous powders can be regulated over a wide range, from 23 to 40 atom%; this results from the difference in the apparent activation energies for the deposition of iron and boron atoms.

In recent years, chemically prepared amorphous Fe–B powders have attracted much attention due to the combination of their ultrafine size and amorphous structure which can lead to potential applications.[1,2] Obviously, the properties of the powders will mainly be determined by the atomic ratio of iron to boron and so, much effort has been devoted to preparing powders with a widest possible composition range by altering preparation conditions in terms of pH, concentration and relative ratios of reaction solutions.[1–3] The available range of the boron atom incorporation is from 15 to 40 atom%. However, the composition range is relatively narrow if one simply regulates a single experimental parameter. To our knowledge, the influence of temperature has not been emphasized in previous studies, which may lead to possible discrepancies of the experimental data in the literature.[1–4] Evidently, the temperature is a very important factor in reaction rates, and will greatly affect the nucleation conditions, and thereby the composition of the products. In this work, the regulation of boron content over a wide range through altering reaction temperature is reported.

The samples were prepared by dropwise addition of 0.1 mol dm^{-3} aq. $FeSO_4$ (50 ml) to 1 mol dm^{-3} KBH_4 (50 ml) within *ca.* 7 min under strong agitation at −7, −3, 1, 7, 13, 21 and 30 °C (NaCl was employed below 0 °C). Other procedures were similar to those described elsewhere.[3] The compositions of the samples were analysed by an inductively coupled plasma method. The amorphous structure was confirmed by X-ray diffraction using Fe-Kα radiation and the pattern shows a halo peak at 2θ *ca.* 52°. The particle sizes ranged from 20 to 100 nm as determined by transmission electron microscopy. The typical Mössbauer spectra of samples consist of a very broadened sextet and a paramagnetic doublet, fairly similar to the spectra for previously reported amorphous Fe–B powders.[1–4] Fig. 1 shows the temperature dependence of the compositions. On increasing the reaction temperature from −7 to 30 °C the boron content increases from 23 to 40 atom% correspondingly, indicating a rather sensitive relationship.

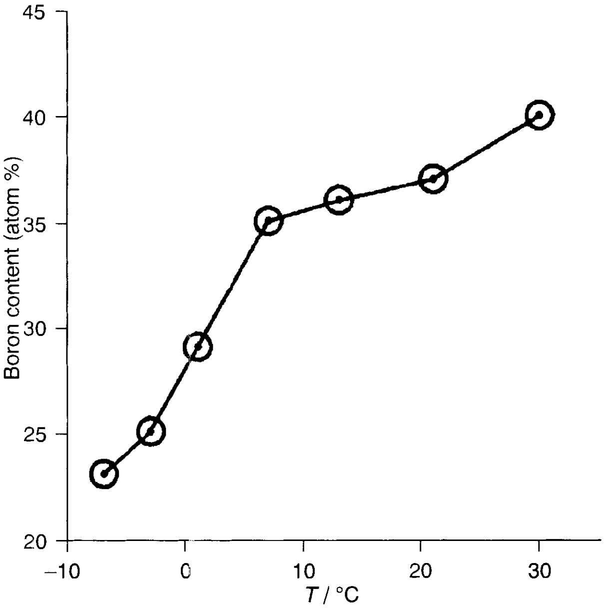

Fig. 1 Dependence of boron content on reaction temperature for Fe–B amorphous powders

The formation of Fe–B amorphous powders through reaction of Fe^{2+} with KBH_4 can be represented by eqns. (1) and (2).[5]

$$BH_4^- + Fe^{2+} + 2H_2O \rightarrow Fe + BO_2^- + 2H^+ + 3H_2\uparrow \quad (1)$$

$$2BH_4^- + 2H_2O \rightarrow 2B + 2OH^- + 5H_2\uparrow \quad (2)$$

The resulting small particle size and the amorphous nature of the deposited particles may be related to rapid nucleation processes in aqueous solution,[6] while the B atoms sustain the stability of the amorphous structure below the glass transition temperature.

From Fig. 1 it is seen that the relative rates of formation of Fe and B through the very complex reactions (1) and (2) vary appreciably with temperature. This fact must be related in turn to the different activation energies of these two reactions.

In conclusion, the boron content for Fe–B amorphous powders can be regulated over a wide range by altering reaction temperature.

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