Grain Boundary Wetting in the Nd-Fe-B-Based Alloy

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Abstract. The microstructure of Nd–Fe–B-based liquid-phase sintered alloy has been studied. The $Nd_2Fe_{14}B/Nd_2Fe_{14}B$ GBs can be pseudo-incompletely (or pseudo-partially) wetted by the Nd-rich melt. Such GBs form the non-zero contact angle with the melt in the triple junctions and contain the uniformly thin (7-10 nm) Nd-rich layer. Such GBs are different from the completely wetted as well as from partially wetted GBs. Most probably, such thin Nd-rich GB layers are responsible for the excellent magnetic properties of the NdFeB-base permanent magnets because these GB layers ensure the magnetic isolation between the $Nd_2Fe_{14}B$ grains needed for the high coercivity.

Introduction

It has been shown in the last years that properties of nanograined and fine-grained materials are strongly dependent on the structure of grain boundaries (GBs) and triple junctions (TJs) [1, 2]. It has been observed using the most advanced experimental methods like aberration-corrected high-resolution electron microscopy (HREM) and 3D-atom probe microscopy permitted to find that GBs and TJs are sometimes not atomically thin. They can contain the so-called complexions (or few nm thick layers with composition different from that of abutting grains) [3–15]. These layers can appear in equilibrium or non-equilibrium conditions [4–22]. Most interesting, from our point of view, is the phenomenon of the so-called pseudopartial (or pseudoincomplete) GB wetting [24–26]. It is marked as PPW in the generic phase diagram proposed in Ref. 23 (see Fig. 1g). Pseudopartial GB wetting is intermediate between complete (CW, Fig. 1g) and partial (PW, Fig. 1g) GB wetting.
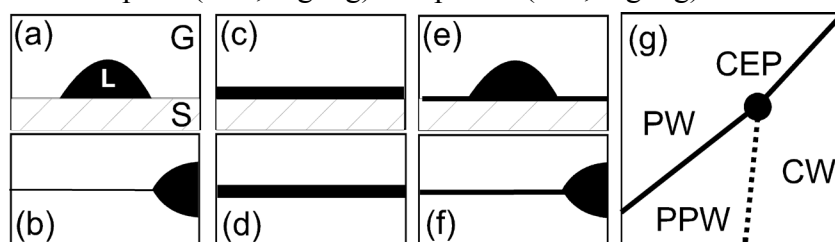

Fig. 1. The schemes for the wetting of free surfaces and GBs. (a) partial surface wetting, L – liquid phase, S – solid phase, G – gas phase; (b) partial GB wetting; (c) complete surface wetting; (d) complete GB wetting; (e) pseudopartial surface wetting; (f) pseudopartial GB wetting; (g) generic wetting phase diagram [23], PW – partial wetting, CW – complete wetting, PPW – pseudopartial wetting, CEP – critical end point, thick lines mark the discontinuous (first order) wetting transition, thin line mark the continuous (second order) wetting transition.

Consider the droplet of a liquid phase on the surface of a solid phase or between two solid grains. Usually one distinguishes partial (or incomplete) and complete wetting of surfaces or interfaces. If the liquid droplet partially wets a solid surface (Fig. 1a) then $\sigma_{sg} - \sigma_{sl} = \sigma_{lg} \cos\theta$, where σ_{sg} is the free energy of solid/gas interface, σ_{sl} is the free energy of solid/liquid interface, σ_{lg} is that of liquid/gas interface and θ is the contact angle. If the liquid droplet partially wets the boundary between two solid grains (Fig. 1b), then $\sigma_{gb} = 2 \sigma_{sl} \cos \theta$, where σ_{gb} is the free energy of a grain

boundary (GB). In case of incomplete surface or GB wetting, a surface or GB which is not covered by the liquid remains dry. It contains only the adsorbed atoms with surface or GB coverage below one monolayer. In this case the GB can exist in the equilibrium contact with the liquid phase. In the case of complete wetting (Figs. 1c,d) $\sigma_{sg} > \sigma_{lg} + \sigma_{sl}$ or $\sigma_{gb} > 2\,\sigma_{sl}$, the contact angle is zero, and liquid should spread between grains or over the free surface.
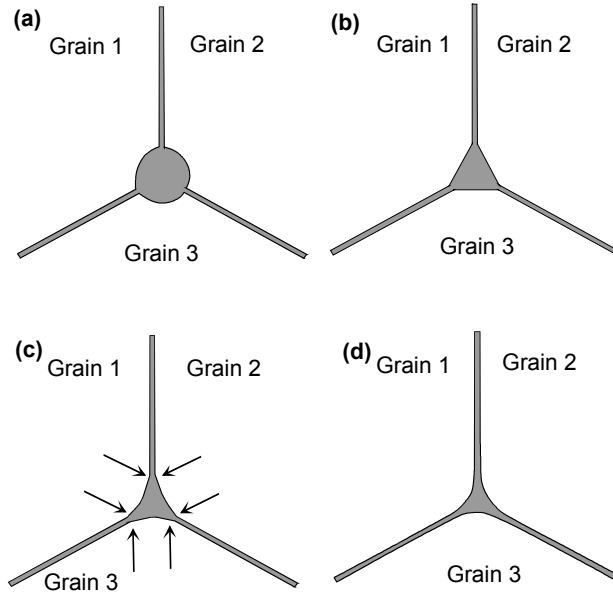

Fig. 2. Different configurations of liquid phase in the GB triple junction and thin quasi-liquid thin layers in the GB. (a) Pseudopartial GB wetting, $\theta > 60°$. (b) Pseudopartial GB wetting, $\theta = 60°$. (c) Pseudopartial GB wetting, $\theta < 60°$. The contact points between liquid phase in TJ and quasi-liquid layers in the GB are shown by arrows. (d) Complete GB wetting, $\theta = 0°$.

If the energy of two solid-liquid interfaces $2\,\sigma_{SL}$ becomes lower than the GB energy $\sigma_{GB} > 2\,\sigma_{SL}$, the transition from incomplete to complete (partial) GB wetting can take place. It proceeds then at a temperature T_w. Cahn [27] and Ebner and Saam [28] first showed that the (reversible) transition from incomplete to complete wetting can be observed with increasing temperature, and that it is a true surface phase transformation. The temperatures T_w of GB wetting transition depend both on GB energy and solid-liquid interfacial energy. They, in turn, depend on the crystallography of these interfaces [29–32]. The fraction of completely wetted GBs increases from 0 to 100% as the temperature increases from T_{wmin} to T_{wmax}. [33–39].

The contact angle is zero n the case of complete wetting (Figs. 1c,d). Thus, liquid spreads over the free surface or between grains. If the amount of liquid is small and surface (or GB) area is large, the liquid spreads until both solid grains or solid and gas begin to interact with each other through the liquid layer. Thus, liquid becomes a kind of "pancake" with a thickness e_s about 2-5 nm [8, 40]:

$$e_s = (A/4\pi S)^{1/2}. \tag{1}$$

Here A is the Hamaker constant and $S = \sigma_{sg} - \sigma_{sl} - \sigma_{lg}$ is the spreading coefficient on a strictly "dry" solid [40]. Such thin "pancake" on the free surface or between the grains is formed due to the deficit of a wetting phase. In a $\alpha+L$ two-phase area of a phase diagram this behaviour takes place very close to the solidus line.

In the most cases the partial wetting directly transforms into complete wetting, for example by increasing temperature [32,36,41] or decreasing pressure [42]. However, in some cases the state of pseudopartial wetting (PPW in Fig. 1g) can occur between partial and complete wetting. In case of pseudopartial wetting the contact angle $\theta > 0$, and liquid droplet does not spread over the substrate. However, the thin (few nm) precursor film spreads around the droplet and separates substrate and gas (Fig. 1e). Such precursor film is very similar for the liquid "pancake" which forms in case of complete wetting and deficit of the liquid phase. Pseudopartial wetting becomes possible if $A < 0$ and $S > 0$ [40].

For the first time, the sequence of discontinuous PW↔PPW and continuous PPW↔CW transitions has been observed in the mixture of alkanes and water [43]. The critical end point (CEP in Fig. 1f) was observed in a mixture of pentane and hexane which was deposited on an aqueous solution of glucose [23]. The first direct measurement of the contact angle in the intermediate wetting state (pseudopartial wetting) was for hexane on salt brine [43]. Later the formation of Pb, Bi and binary Pb–Bi precursors has been observed on the surface of solid copper [44]. They surrounded liquid or solidified Pb, Bi and Pb–Bi droplets. The pseudopartial wetting has been observed recently also for GBs (Fig. 1f) in Al–Zn [24,25] and W–C–Co [26] systems. The 2-4 nm thin layers of the soft Zn-rich phase between Al grains lead to the superductility of the ultrafine-grained Al–Zn alloys obtained by the high pressure torsion [20,21,45,46]. The analysis of existing literature permitted us to suppose that the pseudopartial GB wetting exists also in the Fe–Nd–B-based hard magnetic alloys [47]. The goal of this work is to experimentally observe this phenomenon.

Experimental

The Nd–Fe–B-based liquid-phase sintered alloy was purchased from the company Vacuumschmelze GmbH (Germany): it contained 74.5 wt.% Fe, 22.5 wt.% Nd, 10 wt.% Dy, 1.0 wt.% Co, 0.9 wt.% B, 0.1 wt.% Cu, 0.1 wt.% Al, 0.15 wt.% Ga, 0.04 wt.% C. The as-delivered samples were cut into 2×4×6 mm specimens. Transmission electron microscopy (TEM, HRTEM, STEM, EDXS) studies were carried out on the on the TECNAI instrument. X-ray diffraction (XRD) data were obtained on a Siemens diffractometer (Co Kα radiation). TEM lamellas were prepared on the STRATA dual beam facility. The magnetic properties were measured on a superconducting quantum interference device SQUID (Quantum Design MPMS-7 and MPMS-XL).
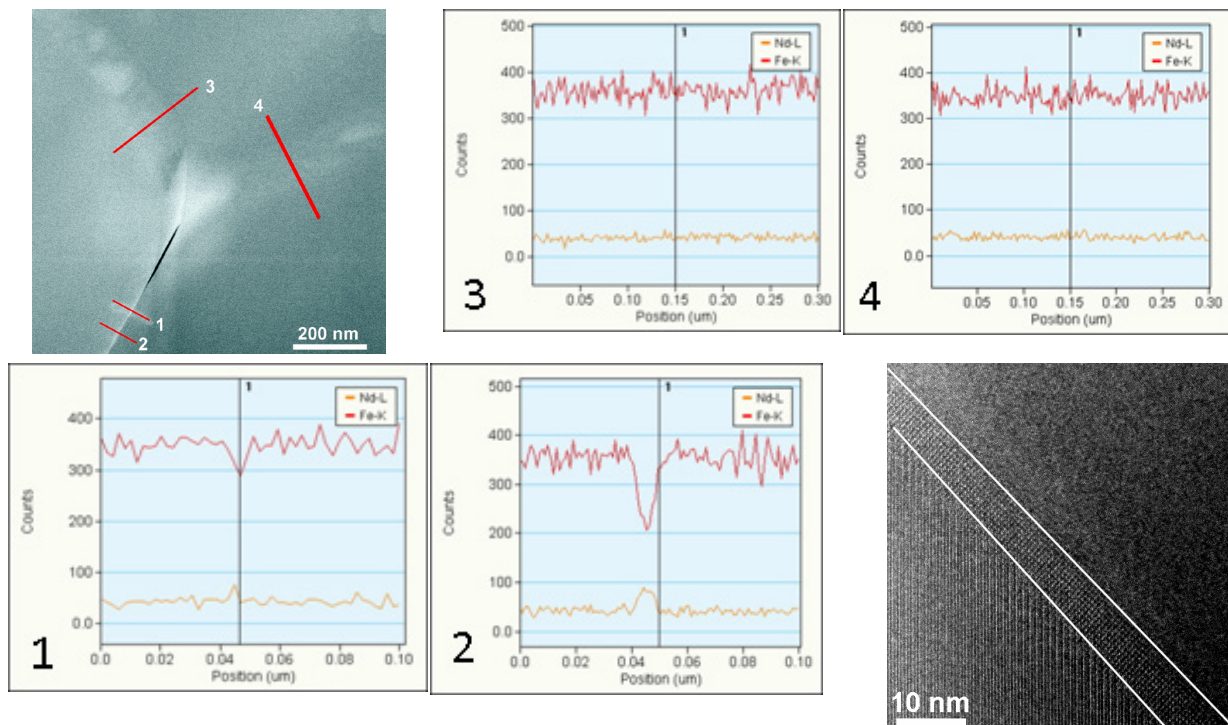

Fig. 3. STEM micrograph of a triple joint between three $Nd_2Fe_{14}B$ grains filled by the Nd-rich phase. The positions of concentration profiles are shown (1, 2, 3 and 4). (1), (2), (3) and (4) are the Fe (top) and Nd (bottom) concentration profiles in the locations 1, 2, 3 and 4, respectively. HREM micrograph of the GB with profiles (1) and (2) with a thin layer of a Nd-rich phase.

Results

In Fig. 3 the STEM micrograph of a triple joint between three $Nd_2Fe_{14}B$ grains is shown (top left). This triple joint filled by the Nd-rich phase The chemical composition of these three grains measured by the EDXS in TEM corresponds to that of $Nd_2Fe_{14}B$ hard magnetic phase. Nd-rich phase in the triple joint was liquid during the liquid phase sintering [48–50]. Four lines in the STEM micrograph with digits 1, 2, 3 and 4 mark the positions of concentration profiles across all three $Nd_2Fe_{14}B/Nd_2Fe_{14}B$ GBs. These profiles (1), (2), (3) and (4) are also shown in Fig. 3. They contain Fe (top) and Nd (bottom) concentration profiles. The profiles (3) and (4) do not contain any Fe and/or Nd maxima or minima. It means that the respective GBs (top right and top left) remain "dry" and are not enriched (depleted) by the Fe and/or Nd. These two GBs have the non-zero contact angle with Nd-rich phase in the triple junction. In other words, they are incompletely (partially) wetted by the Nd-rich melt. This situation corresponds to the scheme shown in Fig. 1b.

The GB shown in the bottom of STEM micrograph is different. The concentration profiles (1) and (2) show that this GB is enriched by Nd and depleted by Fe. The thickness of the Nd maximum and Fe minimum is about 7-10 nm. The uniformly thin light-grey layer of a Nd-rich phase in GB is clearly visible in STEM micrograph in the locations of profiles (1) and (2). In the bottom of Fig. 3 the HREM micrograph of this GB is shown. A thin layer of a Nd-rich phase is visible. HREM also witness that the Nd-rich GB layer is uniformly thin and has a thickness of about 7-10 nm. The GB with profiles (1) and (2), similar to the GBs (3) and (4), also has the non-zero contact angle with Nd-rich phase in the triple junction. Since GB with profiles (1) and (2) is not "dry" and contains the thin Nd-rich layer, this situation corresponds to the scheme shown in Figs 1f and 2c. In other words, the GB with profiles (1) and (2) is pseudo-incompletely (or pseudo-partially) wetted by the Nd-rich melt.

Discussion

The NdFeB-based alloys were invented more than thirty years ago and remain the best hard magnetic alloys. They possess the highest magnetic energy product HB (B being the flux density and H being the field strength). The optimum magnetic properties of these alloys can be ensured when the grains of the hard magnetic $Nd_2Fe_{14}B$ phase are isolated from each other by a layer of a dia- or paramagnetic phase. The Nd-rich phase plays this role in most cases. It forms a liquid layer between $Nd_2Fe_{14}B$ grains during the liquid-phase sintering. It has been observed [51] that the minimum thickness of these layers for effective magnetic isolation of $Nd_2Fe_{14}B$ grains is only few nanometers [52]. If the portion a Nd-rich phase in an alloy is too high, it the saturation magnetization of an alloy decreases. Therefore, one has to keep the amount of the Nd-rich phase as low as possible. It has to be at the level is minimally needed for the effective magnetic isolation between $Nd_2Fe_{14}B$ grains.

The most broadly used technology for the production of NdFeB-base hard magnetic alloys is the liquid phase sintering. The rather coarse-grained (10-20 μm) $Nd_2Fe_{14}B$ powders are usually sintered close to 1100°C [53, 54]. One measured the contact angles between $Nd_2Fe_{14}B/Nd_2Fe_{14}B$ GBs and liquid phase between 700 and 1100°C [55]. Even at highest studied temperature of 1100°C the portion of completely wetted $Nd_2Fe_{14}B/Nd_2Fe_{14}B$ GBs was not 100% (namely, slightly above 80%). It quickly decreased with decreasing temperature. At 700°C it was only around 10%. These results were obtained using the „pure" three-component Nd–Fe–B alloys. The micrographs published in the literature gave the possibility to estimate the portion of completely wetted $Nd_2Fe_{14}B/Nd_2Fe_{14}B$ GBs in the alloys with different doping elements (like Dy, Pr, Al, Cu, Co etc.) in addition to Nd, Fe and B. Only very seldom the values for portion of completely wetted GBs were above the line for the „pure" three-component Nd–Fe–B alloys. Thus, the portion of completely wetted $Nd_2Fe_{14}B/Nd_2Fe_{14}B$ GBs is low and the amount of wetting phase is high. In this case, what is the mechanism for the formation of magnetically isolating Nd-rich layers between $Nd_2Fe_{14}B$ grains? And this magnetic isolation is needed for high performance of the NdFeB-based alloys for permanent magnets. Maybe the pseudopartial GB wetting is the explanation?

In case if the thin GB layers of a constant thickness (or complexions) exist in a polycsrystal, it is not easy to judge, whether it is (1) prewetting/prewetting in the one-phase area of a bulk phase diagram; (2) thin GB "pancake" in the two-phase area of a bulk phase diagram close to solidus linee or (3) pseudopartial wetting. The huge trouble is that usually the liquid or solid phase can be found only in the GB triple junctions (TJ) or in so-called "pockets". The pseudopartial wetting can be clearly identified only if the solid/liquid interface is convex and the contact angle is $\theta \geq 60°$ (Figs. 2a,b). If the solid/liquid interface is concave and the contact angle is low $\theta < 60°$ (Figs. 2c,d), the difference becomes tiny. The pseudopartial wetting takes place in case when the solid/liquid interface has a discontinuity (i.e. two intersecting tangentials) in the point between TJ pocket and GB layer (Fig. 2c). If the solid/liquid interface is continuous (one tangential) in all points between TJ pocket and GB layer (Fig. 2d), we observe not the pseudopartial wetting but complete wetting with the deficit of a wetting phase.

Can the $Nd_2Fe_{14}B/Nd_2Fe_{14}B$ GBs contain the few nanometer thick Nd-rich layers like the prewetting GB layers in metals [17–22, 42, 56–62] or oxides [5–16]? Indeed, in early 1990-ies such few nm thin GB layers of a Nd-rich phase were experimentally found in the NdFeB-based alloys [63]. Later the Nd-rich uniform thin (< 5 nm) layers between $Nd_2Fe_{14}B$ grains were observed in many experimental works in various alloys [63–76]. What is the physical reason for the formation of such uniformly thin GB layers? As we saw above, two possibilities exist. First is the complete GB wetting and a deficit of a melt. In this case the liquid in the GB TJs could completely wet the TJs and form the continuous network along TJs. In this case, the "wet" TJ pockets make an impression that the amount of a liquid phase is high. Nevertheless, the liquid phase distributes in TJs in such a way that the pockets in TJs remain macroscopic (i.e. few μm in diameter), however, the melt in GBs is in a deficit. It is able to cover the GBs only with very thin films. In this case the liquid in the TG pocket should undergo into a thin GB layer without any shape discontinuity (like it is shown in the scheme in Fig. 2c). Indeed, the continuous shape transition between GB film and TJ pocket has been observed many times [53, 65, 66, 77–80]. One can see in those micrographs simultaneously TJs with (solidified during cooling) liquid inside and GB thin films [53, 63, 65–67, 76–81].

Second possible explanation for the observation of uniformly thin GB layers is the pseudopartial GB wetting (Figs. 2a,b,c). In this case one hast to observe in the contact point between liquid TJ pocket and thin GB layer a certain shape discontinuity. In many papers the TEM micrographs were published where the pseudopartial GB wetting obviously takes place [63, 66, 67, 73, 76, 78, 79, 81]. In any case, one needs the micrographs where both TJ pockets and GB layers are present together, in order to judge about the nature of thin GB layers. Unfortunately, usually in published micrographs of thin Nd-rich GB layers we cannot see the locations where the GB contacts with a TJ Nd-rich. In such cases it is hard or even impossible to judge, whether one observes the complete or pseudopartial GB wetting.

The results obtained in this work demonstrate again that the NdFeB-based alloys, can contain GBs either incompletely or pseudoincompletely wetted by the Nd-rich melt. In first case (GB profiles 3 and 4 in Fig. 3), the GBs remain "dry" and are not enriched (depleted) by the Fe and/or Nd (profiles 3 and 4). The GBs with profiles 3 and 4 have the non-zero contact angle with Nd-rich phase in the GB TJ. Thus, these GBs are incompletely (or partially) wetted by the Nd-rich melt, similar to the scheme shown in Fig. 1b. The pseudo-incompletely (or pseudo-partially) wetted "bottom" GB with profiles 1 and 2 also has the non-zero contact angle with Nd-rich phase in GB TJ. However, this GB contains the thin layer which is enriched by Nd and depleted by Fe (see profiles 1 and 2). This layer is uniformly thin (about 7-10 nm). It is clearly visible in TEM and HREM micrographs. The GB with profiles 1 and 2 is not "dry" and contains the thin Nd-rich layer. Therefore, this situation corresponds to the scheme shown in Figs 1f and 2c. In other words, the "bottom" GB with profiles 1 and 2 is pseudo-incompletely (or pseudo-partially) wetted by the Nd-rich melt.

The completely, partially and pseudopartially wetted $Nd_2Fe_{14}B/Nd_2Fe_{14}B$ GBs as well as their TJs (filled by the Nd-rich melt) form the continous network with a complicated topology. The

contiguity of this network ensure the ability of $Nd_2Fe_{14}B/Nd_2Fe_{14}B$ GBs to fix the magnetic domain walls and to prevent thus their movement. This behaviour increases the magnetic energy product *HB* of NdFeB-based hard magnetic alloys.

Conclusions

In this work we observed that the $Nd_2Fe_{14}B/Nd_2Fe_{14}B$ GBs in the NdFeB-based permanent magnets can be pseudo-incompletely (or pseudo-partially) wetted by the Nd-rich melt. Such GBs form the non-zero contact angle with the melt in the triple junctions. Simultaneously, they contain the uniformly thin (about 7-10 nm) Nd-rich layer. Therefore, such GBs are different from the completely wetted GBs (zero contact angle and non-unifom and thick Nd-rich layer with thickness above 100 nm), as well as from partially wetted GBs (non-zero contact angle, no Nd-rich layer). Most probably, such thin Nd-rich GB layers in the pseudo-incompletely (pseudo-partially) wetted $Nd_2Fe_{14}B/Nd_2Fe_{14}B$ GBs are responsible for the excellent magnetic properties of the NdFeB-base permanent magnets. It is because these layers can ensure the magnetic isolation between the $Nd_2Fe_{14}B$ grains which in turn is needed for the high coercivity.

Acknowledgements

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