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Crystallization of $Nd_2Fe_{17}B_x$ from stoichiometric melt composition

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Abstract

Containerless solidification of gas-atomized $Nd_{10}Fe_{85}B_5$ melt droplets was carried out using the drop tube technique. The phase constituents and microstructure of the solidified samples were investigated by means of powder X-ray diffraction analysis, thermomagnetic analysis, and scanning electron microscopy. Besides α-Fe and $Nd_2Fe_{14}B$, non-equilibrium phases such as $Nd_2Fe_{17}B_x$, ε-Nd, and $Nd_{1.1}Fe_4B_4$ were identified. The microstructure of the samples was categorized into three types, including a quasi-single phase one that consists mainly of $Nd_2Fe_{17}B_x$ dendrites. The lattice parameters and Curie temperature of $Nd_2Fe_{17}B_x$ were determined, and compared with those of the same type phase crystallized in Nd-rich Nd–Fe–B melt compositions. The results were discussed with respect to the stoichiometry, formation as well as potential application of $Nd_2Fe_{17}B_x$.

Keywords: Rare earth alloys; Rapid-solidification; Microstructure; SEM; X-ray diffraction

1. Introduction

Solidification of commercial Nd–Fe–B alloys involves primary crystallization of Fe-rich solid solution (γ-phase) as well as subsequent peritectic formation of the $Nd_2Fe_{14}B$ compound (ϕ-phase) from the liquid phase [1]. Previous work [2,3] has shown that liquid undercooling, achieved by electromagnetic levitation or by drop tube processing, can alter the solidification pathway of Nd–Fe–B alloys drastically. On the one hand, liquid undercooling can suppress primary γ-Fe crystallization in favor of direct crystallization of $Nd_2Fe_{14}B$. On the other hand, liquid undercooling can induce crystallization of a metastable intermetallic compound, χ-$Nd_2Fe_{17}B_x$ ($x \sim 1$), either as a primary phase or as an intermediate peritectic phase following primary γ-Fe formation. The grains of $Nd_2Fe_{17}B_x$ are decomposed into a fine mixture of γ-Fe plus $Nd_2Fe_{14}B$ in electromagnetically levitated bulk samples, but are preserved at least partially in drop tube-solidified small samples. The measurements on the drop tube-solidified samples have shown that $Nd_2Fe_{17}B_x$ orders ferromagnetically below 373 K with a $TbCu_7$-type hexagonal structure. In terms of recent in situ synchrotron radiation diffraction analyses on electromagnetically levitated bulk samples [4], the structure of $Nd_2Fe_{17}B_x$ has been refined to a Th_2Zn_{17}-type rhombohedral one. The difference between the two structural types lies in the degree of order of rare earth atom and iron atom-pairs [5]. The former has a lower order than that of the latter, and is usually regarded as a disordered variant of the latter. In both structures, boron atoms have been assumed to occupy interstitial sites. Ozawa et al. [6,7] have also reported crystallization of a metastable intermetallic phase of a Nd_2Fe_{17}-type structure from undercooled Nd–Fe–B melts, which is assumed to be identical to $Nd_2Fe_{17}B_x$. In the present work, gas-atomized $Nd_{10}Fe_{85}B_5$ melt droplets were containerlessly undercooled and solidified using the drop tube technique in order to check if $Nd_2Fe_{17}B_x$ can be crystallized from stoichiometric melt composition.

2. Experimental

Alloys with atomic composition of $Nd_{10}Fe_{85}B_5$ were prepared by arc-melting elemental Nd (99.9% purity), Fe (99.995 purity) and B (99.995% purity) under the protection of an argon atmosphere (99.999% purity). In order to compensate for mass loss during arc-melting and subsequent induction melting, an

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