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TEM studies of the $ThMn_{12}$–type and Th_2Zn_{17}–type phases in the Nd-Fe-Ti system

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Rare-earth intermetallic compounds with high Fe concentrations and adopting the $ThMn_{12}$–type and Th_2Zn_{17}-type structures have attracted considerable attention in the field of permanent magnets. A large number of experimental studies has been done on these compounds, which are usually measured on alloys structurally characterized only by powder X-ray diffraction (XRD). However, the materials are often multiphasic and their quenched or annealed microstructures evidence homogenization ranges, second phases (resulting from segregation) and on-going phase transformations that may not be easily detectable by XRD [1,2]. Additional microstructural studies on these systems are therefore required.

Among the Fe-based systems, the $ThMn_{12}$–type and Th_2Zn_{17}–type structures usually require stabilization by a third element, such as Ti. In the present work Nd:11Fe:Ti has been prepared by melting Nd, Fe and Ti in an arc furnace followed by subsequent splat-quenching and/or annealing treatments. The resulting materials have been characterized by XRD, scanning and transmission electron microscopy coupled with energy dispersive spectroscopy.

The results have shown that the presence of α-Fe(Ti) could not be avoided during the solidification of the Nd:11Fe:Ti alloy. Furthermore, the microstructure morphologies and elemental analyses showed that at moderate cooling rates a secondary crystallization phase of Th_2Zn_{17}–type appeared in the Nd:11Fe:Ti alloy as result of a peritectic reaction. This lower temperature phase was however not detected in the splat-quenched material, where the high cooling rate route suppressed its crystallization [3]. Nevertheless during a subsequent heat-treatment at 800 ºC the following decomposition took place: $NdFe_{11}Ti \rightarrow Nd_2(Fe,Ti)_{17} + \alpha\text{-Fe(Ti)} + Fe_2Ti$. This reaction had already been proposed by Jang and Stadelmaier [4], who suggested that the $NdFe_{11}Ti$ compound is unstable at temperatures below 1000 °C. The current study shows that this transformation results in the fine lamellar intergrowth of the Th_2Zn_{17}-type phase in $ThMn_{12}$-type grains, displaying random distribution of planar defects (Figure 1). The reciprocal space of the combined parent and intergrown phases has been mapped through a series of 3-D microdiffraction experiments (Figure 2). This allowed to establish that the preferred orientation relation between the two phases is $(020)_{1:12}//(003)_{2:17}$ and $[100]_{1:12}//[110]_{2:17}$, with the invariant plane sitting at $(022)_{1:12} // (\bar{3}33)_{2:17}$ planes.

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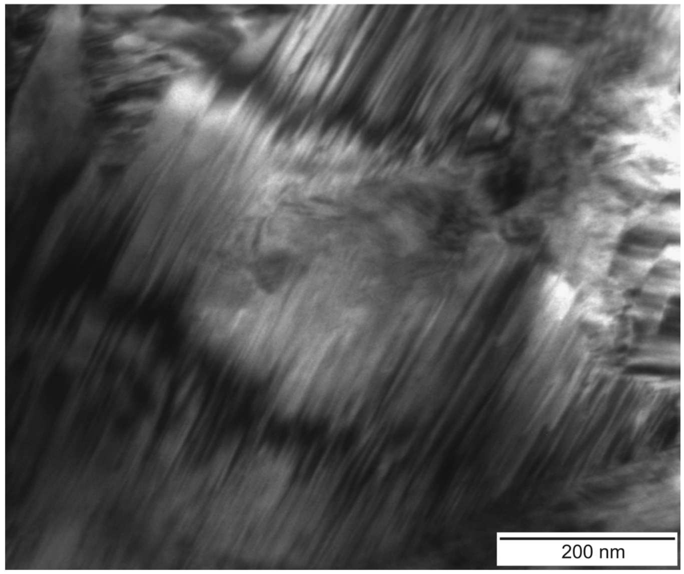

Figure 1. (a) Grains with a random distribution of planar defects.

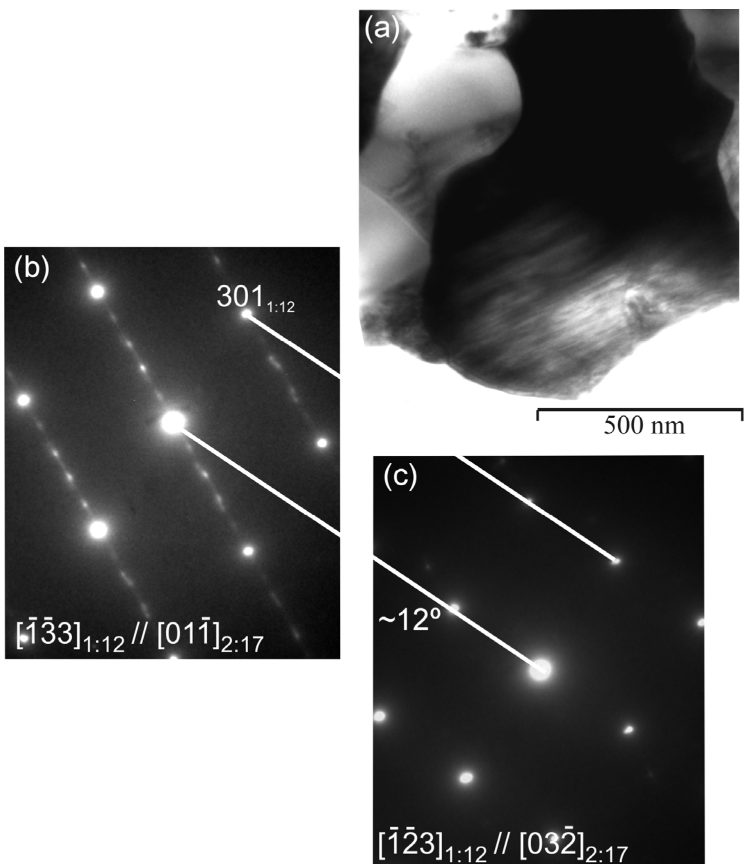

Figure 2. (a) Grain with a random distribution of planar defects. (b) Experimental microdiffraction pattern obtained along $[\bar{1}\bar{3}3]_{1:12}$ // $[01\bar{1}]_{2:17}$. (c) Experimental microdiffraction pattern obtained along $[\bar{1}\bar{2}3]_{1:12}$ // $[0\bar{3}\bar{2}]_{2:17}$ after tilting ~12° from the previous orientation while keeping excited the $(301)_{1:12}$ reflection.

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