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# TEM studies of TbNiAl in the Disproportionation Stage of the HDDR Process

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The hydrogenation disproportionation desorption recombination (HDDR) process is an effective way to obtain highly coercive magnets by refining originally coarse grains to fine grains [1,2]. The intermetallic compound TbNiAl can absorb up to 1.4 deuterium atoms per formula unit at room temperature and 1 bar $H_2/D_2$ pressure [3,4]. In the present work we have investigated the microstructural transformations during the disproportionation stage of the HDDR process in TbNiAl by transmission electron microscopy (TEM).

TbNiAl was prepared from Tb (purity 99,8%), Ni (99,9%) and Al (99,9%) by arc melting in an argon atmosphere. The ingots were remelted several times to increase their homogenity and annealed at 800°C. The material was first heated to 600°C in vacuum and the atmosphere was switched to 4 bar $D_2$. X-ray diffraction (XRD) showed that the sample was disproportionated into two phases, $TbD_{2.2}$ and NiAl. This is the so-called solid-HD(DR) experiment since the lumps of the starting material are maintained. TEM samples were prepared by mechanical grinding in a silicate crucible and spreading the dry powder on a holy carbon film supported on a titanium grid. Electron microscops was performed in a Philips CM 30 operated at 300 kV .

A dark field micrograph from the sample is shown in FIG 1a. This indicates that the structure consists of nanocrystalline regions with average sizes down to a few nanometers. Selected area diffraction shows the existence of both phases, the measured d spacing closely matching those of the $TbD_{2.2}$ and NiAl phases from the XRD data. Some texture is evident in the diffraction pattern. For example, the third ring marked in FIG 1b, corresponding to the NiAl phase (110), is not continuous. The other rings, which are more continuous, correspond to the $TbD_{2.2}$ phase. One method for studying the distribution of the phases in the disproportionated state is lattice resolution imaging, FIG 2. The detailed structure is shown in FIG 3 in which both the $TbD_{2.2}$ and NiAl phases are present. The dominant $TbD_{2.2}$ phase has the measured lattice spacing $d_{112}$=3.14Å. Using the $TbD_{2.2}$ as internal reference we measured a particle region with a lattice spacing of 2.04Å which corresponds well with the NiAl phase (110) spacing [5].

References

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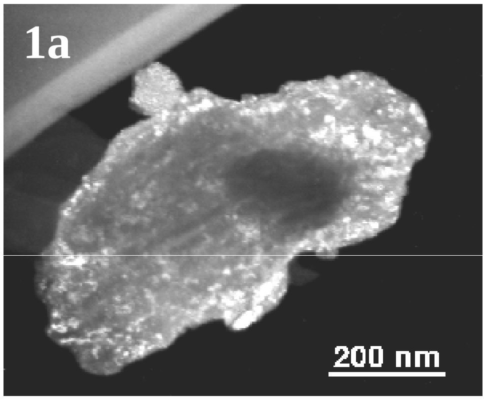


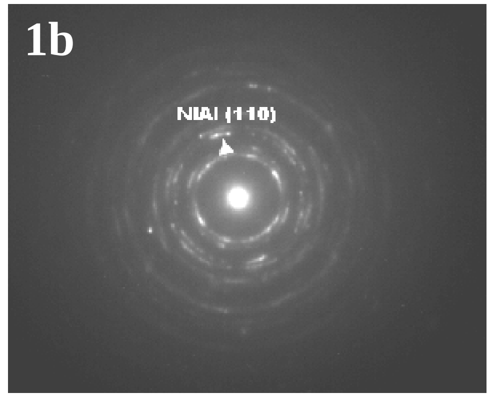
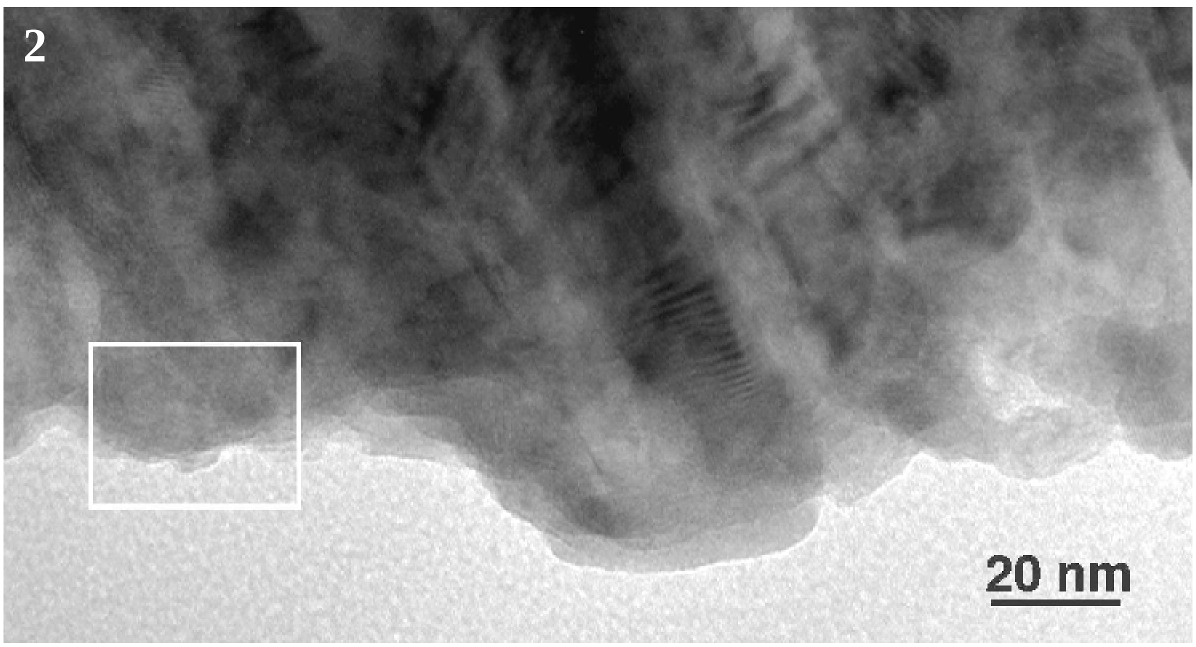




FIG 1a TEM dark field image and 1b selected area diffraction pattern of the TbNiAl disproportionated state



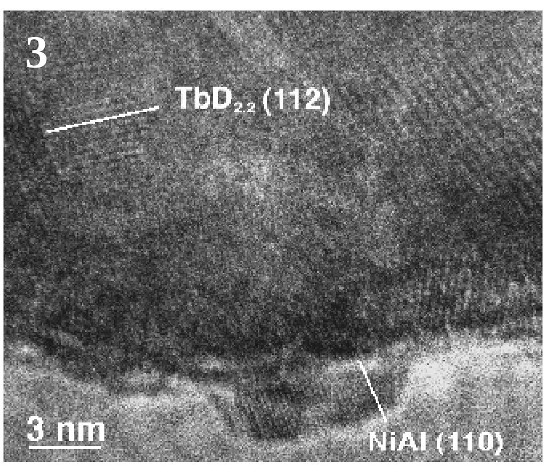




FIG 2 HRTEM image from the edge of a particle in the disproportionated state.

FIG 3 HRTEM image from small area in FIG 2 showing lattice fringes from the two phases.

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