## UB02

# Hydrogenation-assisted Crystallisation of Amorphous Nd-Fe-B Material

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In the manufacturing route of Nd-Fe-B-type magnet, in particular, in the melt-spinning route of the $Nd_2Fe_{14}B$ single phase material and $Nd_2Fe_{14}B/Fe$ nanocomposite materials, achieving a fine microstructure is a key factor for improving magnetic performance. Meanwhile, a hydrogen gas has been used effectively for achieving a fine-microstructure in the Nd-Fe-B-type material. The widely used hydrogen-related processes are, so-called, the HD (hydrogen decrepitation) [1] and HDDR (hydrogenation, disproportionation, desorption and recombination) [2, 3] processes. In these hydrogen-related processes the hydrogen gas is reacted with the phases like Nd-rich grain boundary phase and $Nd_2Fe_{14}B$ matrix phase in the Nd-Fe-B material and the reactions have a huge positive reaction enthalpy, thus evolving a huge heat. The present authors have extended their idea to the thought that this huge heat can be used to crystallise an amorphous Nd-Fe-B material, and this hydrogenation-assisted crystallisation may be developed as a new processing route for achieving a fine microstructure in the Nd-Fe-B material. In the present study the mechanically milled Nd-Fe-B amorphous material was reacted with hydrogen gas and the crystallisation assisted by the hydrogenation was studied.

The starting alloy of $Nd_{15}Fe_{77}B_8$ alloy was pulverized and then subjected to a high energy mechanical milling using a shaker mill in order to get an amorphous material. The milled amorphous material was reacted with hydrogen gas (1 kgf/cm$^2$) at various temperatures. The material was first heated at desired temperature and then the hydrogen gas was introduced into the reaction chamber and held for 5 min. Some of the hydrogenated material was heated under vacuum to desorb the hydrogen and induce a combination between the constituent phases. Phase analysis of the materials under various conditions was performed by an x-ray diffraction (XRD) using CuK$\alpha$ radiation. A differential thermal analysis (DTA) was also performed for the milled material to examine crystallization and hydrogen reactivity. Magnetic characterisation of the materials was performed by a vibrating sample magnetometer (VSM). For the VSM measurement the particulate sample was fixed with a wax and then pre-magnetised with 6 T pulsing field.

It was found that the hydrogenation facilitated significantly the crystallisation of the amorphous $Nd_{15}Fe_{77}B_8$ alloy, and the crystallisation was completed at the temperatures much lower than the ordinary thermal crystallisation temperature. The huge heat evolved from the hydrogenation was believed to assist the crystallisation of the amorphous. The phase analysis study revealed that the $Nd_{15}Fe_{77}B_8$ material crystallised from the amorphous by the hydrogenation consisted of a mixture of $NdH_x$, Fe and $Nd_{1+x}Fe_4B_4$ phases. It was also revealed that the desorption of hydrogen from the hydrogenated material under vacuum at an elevated temperature led to a combination of Nd, Fe and $Nd_{1+x}Fe_4B_4$ phases into the $Nd_2Fe_{14}B$ phase. In this article the phase constitution and magnetic properties of the Nd-Fe-B material prepared from the amorphous material by the hydrogen-assisted crystallisation is to be discussed.

### REFERENCES

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## UB03

# Corrosion Kinetics of Spark Plasma Sintered NdFeB Permanent Magnets in Different Solutions

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In present study, spark plasma sintering (SPS) technology was applied to fabricate a new kind of NdFeB permanent magnetic materials with original microstructure that is different from that of conventional sintered magnets. The corrosion behaviors of both magnets were studied in NaCl, NaOH, and $H_2SO_4$ solution. The corrosion kinetic curves indicated that both magnets exhibited evidently passive behavior in alkaline solution and processed activation dissolution in acid solution. In comparison with the conventional sintered NdFeB magnets, SPS magnets processed much lower corrosion rate in different solutions. SEM observation showed that conventional sintered NdFeB magnets exhibited typical inter-granular corrosion process (Fig. 1). In the SPS NdFeB magnets, however, the inter-granular corrosion process though Nd-rich phase were suppressed effectively due to the unique microstructure of the magnets, i.e. the grain size of the $Nd_2Fe_{14}B$ main phase is fine and uniform, and the fine Nd-rich phase does not form along the grain boundaries of main phase, but agglomerates into the triple junctions when compared with conventional magnets (Fig. 2). As a result, the SPS NdFeB magnet possesses excellent corrosion resistance.

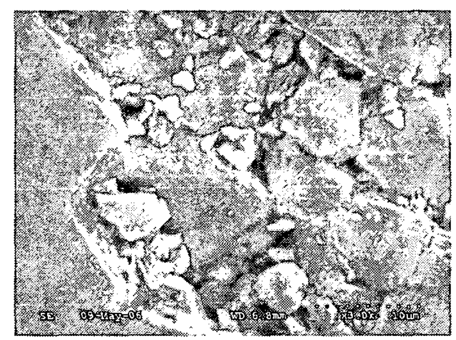

**Fig. 1.** SEM micrograph of conventional sintered NdFeB magnets in 0.1% $H_2SO_4$ solution at 10 minute

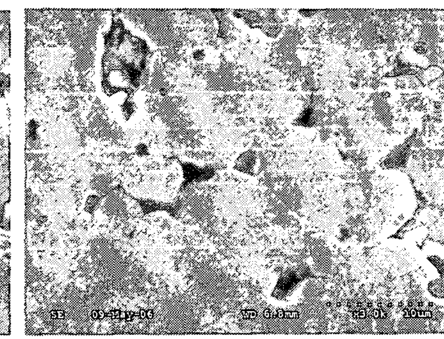

**Fig. 2.** SEM micrograph of SPS NdFeB magnets in 0.1% $H_2SO_4$ solution at 10 minute

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