

# Experimental Report

07/07/2005

**Proposal:** 5-26-156                      **Council:** 12/2001  
**Title:** Texture Investigation of Hot Forged NdFeV Magnets  
**This proposal is a new proposal**  
**Research Area:** Materials

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**Samples:** Nd(Fe,V)12  
Nd(Fe,V)12

<b>Instrument</b>	<b>Req. Days</b>	<b>All. Days</b>	<b>From</b>	<b>To</b>
D20	2	2	18/03/2002	20/03/2002

**Abstract:**

## Texture development in Nd-Fe-V alloys by hot deformation in view of permanent magnet properties

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### Abstract.

In this study, a hot forging process is applied to Nd-Fe-V as cast alloys in order to develop both the microstructure and the crystallographic texture appropriate for permanent magnet properties. A neutron diffraction texture analysis is used to account for the extrinsic magnetic anisotropy: the stabilisation of the Nd(Fe,V)<sub>12</sub> hard magnetic phase has been achieved during forging but its extrinsic anisotropy level remains low. Attempts to understand this phenomenon are made through a discussion on the Nd-Fe-V alloy rheological and mechanical behaviour and a comparison with Nd-Fe-B permanent magnets.

### Introduction

Good levels of coercivity and enhanced magnetic anisotropy are the key for high performance magnets. In the field of rare earth - transition metal alloys, the development of anisotropic powders is needed for bonded magnet applications. Anisotropic Nd-Fe-B based alloys are already commercially available whereas, only recently some studies report on Nd-Fe-V-N alloys with potential permanent magnet properties.

The NdFe<sub>10.5</sub>V<sub>1.5</sub> compounds, one of the 1:12 phases RFe<sub>12-X</sub>M<sub>X</sub> (R = rare earth, M = transition metal), can be considered as potential materials for permanent bonded magnets because of their high intrinsic magnetic properties, higher than those of the Nd<sub>2</sub>Fe<sub>14</sub>B-type materials [1], which are presently the most used as permanent magnets. For this purpose, good extrinsic magnetic properties are also needed: coercivity, and extrinsic magnetic anisotropy. Up to now, mechanical alloying enabled to obtain a high coercivity in this type of materials ( $H_c = 872.17$  kA/m) [2].

However, large-scale production processes have to be considered. We proposed to apply a high-speed hot forging process to induce the microstructure suitable for permanent magnet properties in Nd-Fe-V alloys. In the case of Nd-Fe-B materials, coercivity and extrinsic anisotropy were already successfully induced in the bulk alloy by applying this hot forging process with a strain rate of  $125 \text{ s}^{-1}$  [3]. Coercivity resulted from grain size reduction. Extrinsic magnetic anisotropy was linked to a fibre texture of the c-axes, being the easy axes of magnetisation [4].

Besides the extrinsic magnetic properties, the major concern in the preparation of Nd-Fe-V alloys is the stabilisation of the  $\text{NdFe}_{10.5}\text{V}_{1.5}$  phase. The compounds can contain free iron, detrimental to the coercivity and the temperature and composition range of the 1:12 phase stability domain is limited. The high-speed hot forging process applied to as-cast Nd-Fe-V alloys, (containing only free iron and an intergranular Nd-rich phase) allowed the stabilisation of the 1:12 phase and the almost complete dissolution of free iron [1]. This revealed equivalent to a long annealing treatment of 24 hours at  $960^\circ\text{C}$ . The two main rheological parameters of the forging process were optimised, (the temperature and the Nd content). The best results were obtained by forging at  $930^\circ\text{C}$  samples prepared with 10% Nd-excess.

### Experimental details

Samples of the  $\text{NdFe}_{10.5}\text{V}_{1.5}$  composition with 10% excess of Nd were prepared by induction melting and then cast into cylindrical stainless steel tubes of 12 mm in diameter. The forging process was applied on these ingots under argon atmosphere at  $930^\circ\text{C}$ , either in the as-cast state, presenting free iron dendrites and the Nd-rich intergranular phase, or after an annealing treatment for 24 hours at  $960^\circ\text{C}$ , leading to the homogeneous 1:12 phase, without free iron traces. A third sample, also annealed, was prepared with 3% Nd-excess, in order to determine the influence of the intergranular phase content on the texture. At the forging temperature, the intergranular phase is liquid whereas the 1:12 phase or the free iron dendrites are solid.

The texture of the  $\text{NdFe}_{10.5}\text{V}_{1.5}$  alloys after the high-speed hot forging process was studied by neutron diffraction on the D20 powder diffractometer of the Laue-Langevin high flux nuclear reactor in Grenoble, France. For this purpose, an Eulerian circle was mounted on the diffractometer. The multidetector registers all the pole figures simultaneously for each  $(\chi, \varphi)$ -position.

The samples, of cubic shape of about 6mm, were put on the Eulerian circle in such a way that the forging direction was in a plane perpendicular to the  $\varphi$ -axis. The position of the circle was  $\omega = 34.2^\circ$  corresponding to the Bragg angle of the (400) peak of the 1:12 structure. Scans in  $\chi$  from  $0^\circ$  to  $90^\circ$  with a  $5^\circ$  step and in  $\varphi$  from  $0^\circ$  to  $355^\circ$  with a  $5^\circ$  step for each value of  $\chi$  were operated for all the analysed samples.

Data treatment was carried out using the following calculation codes: XRFIT (developed at ILL), POFINT [5], GOMAN [6] and Beartex [7].

## Results

For all the samples, a fibre texture component is observed, with the  $\langle 010 \rangle$  directions parallel to the forging direction Y, reinforced by two three-dimensional components. This is presented in Figure 1, in the case of the sample with 10% Nd-excess. The  $\{100\}$  and  $\{001\}$  pole figures illustrate the presence of a nearly fibre texture with  $\langle 010 \rangle // Y$  being the fibre axis. These figures also evidence two texture components with c-axes at about  $\chi = 60^\circ$ .

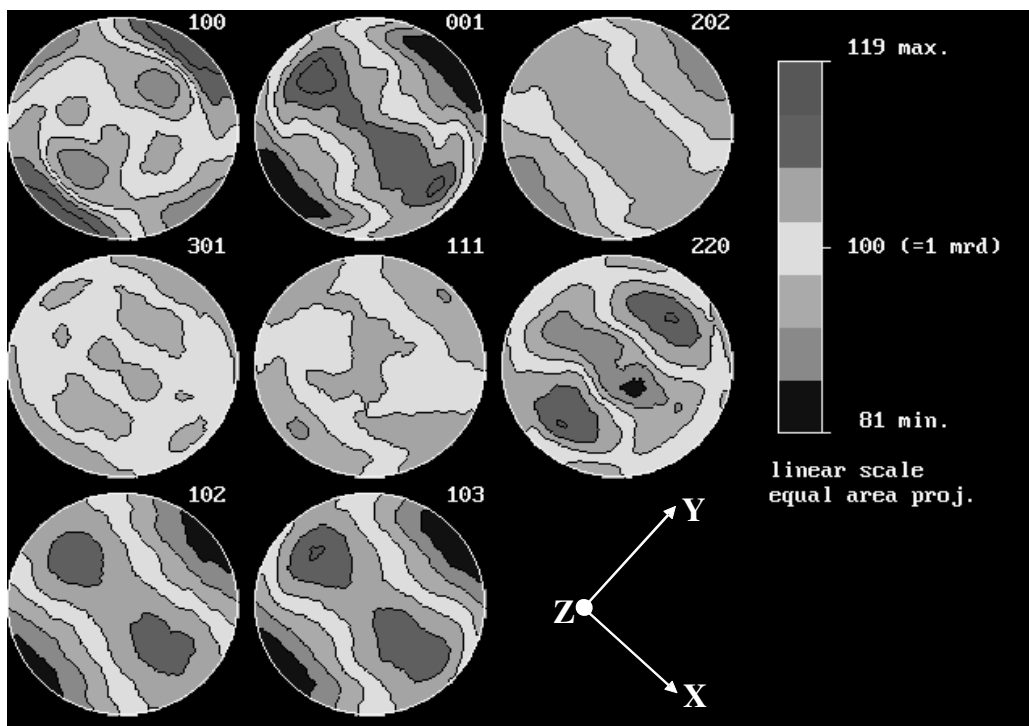


Figure 1. Low (hkl) poles figures recalculated from the ODF for  $\text{NdFe}_{10.5}\text{V}_{1.5} + 10\%\text{Nd}$

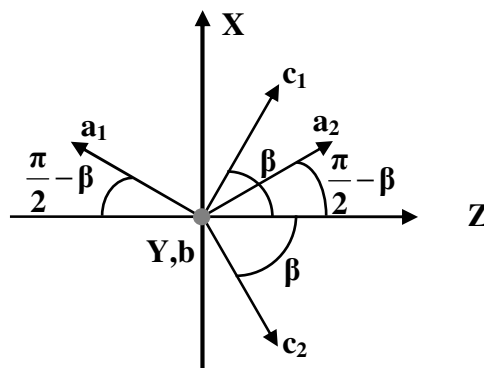


Figure 2. Three-dimensional texture components obtained after forging the  $\text{NdFe}_{10.5}\text{V}_{1.5} + 10\%\text{Nd}$  sample. Y is the forging direction and  $(a_1, b_1, c_1)$ ,  $(a_2, b_2, c_2)$  the two texture components

Using the Roe-Matthies convention [8, 9], the two three-dimensional components are defined by  $\{0, \beta, 0\}$  and  $\{\pi, \beta, 0\}$  ( $\beta \approx 60^\circ$ ).