

## [C1]

### Influence of washing process on the magnetic properties of Nd-Fe-B powders prepared by thermochemical process.

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#### 1. Introduction

Modern permanent magnets, whether they are sintered or bonded, are produced by using magnetic powder. Accordingly, magnetic properties of the magnets primarily depend on the nature of the powder such as particle shape, size, purity, and composition. In order to develop high performance magnets, therefore, these powder parameters should be optimized. In this work, we fabricated Nd-Fe-B powder by thermochemical process, and investigated effect of washing step on the magnetic properties of the powder.

#### 2. Experimental

The Nd-Fe-B powder with the target composition of  $\text{Nd}_{15}\text{Fe}_{77}\text{B}_8$  was prepared by thermochemical process including spray dring, ball milling,  $\text{H}_2$  reduction, Ca reduction, (ball milling), washing, and drying. Phase identification of the powder was performed by Cu  $K_\alpha$  x-ray diffraction. Morphologies and microstructures of the powder were examined with a SEM. Magnetic properties of the final powder were measured with a VSM with a maximum applied field of 15 kOe.

#### 3. Results and discussion

The Nd-Fe-B powder prepared by the thermochemical process was mostly composed of  $\text{Nd}_2\text{Fe}_{14}\text{B}$ , but small amount of impurity CaO that was not removed during washing process was still found. Comparing with the powder water-washed only, there was no remarkable change in phase formation when the powder was milled for 3, 5, and 10 hours under the vacuum before washing. However, it was found by SEM observation that the distribution and the size of the powder became more uniform and smaller as the milling time increased, yielding  $\text{Nd}_2\text{Fe}_{14}\text{B}$  particle clusters of 3 - 4  $\mu\text{m}$  after 10 hour milling. Moreover, Ca content in the milled powder also reduced remarkably as the milling time increased, from 52.03 at.% in the powder only water-washed for 90 min. to 0.34 at.% in the powder milled for 10 hours before washing. Although the remenance of the powder increased due to the reduction of residual CaO, the overall magnetic properties, especially for  $iH_c$ , decreased with the increase of the milling time. When the powder milled for 10 hours was washed for 1 hour in dilute acetic acid with various concentration 0.1 - 3.0 wt.%, an abnormal lattice expansion of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  was found. The lattice expansion in the milled powder washed for 1 hour in 3 wt.% dilute acetic acid was measured as 0.12 Å along a-axis and 0.17 Å along c-axis of the  $\text{Nd}_2\text{Fe}_{14}\text{B}$  structure. The magnetic properties of the powder washed in dilute acetic acid were deteriorated as the concentration of the acid increased.

#### 4. Conclusions

When milling step was introduced before washing, not only the Nd-Fe-B powder became more uniform and finer, but also the residual CaO in the powder reduced remarkably with the increase of the milling time. However, the overall magnetic properties of the powder, especially  $iH_c$ , decreased with the increase of the milling time even though the remanence increased due to the reduction of CaO. Using dilute acetic acid as washing agent instead of water resulted in worse magnetic properties.

#### 5. Acknowledgements

This work is supported by dual use technology program (Grant No. 01-IT-MP-07) of Ministry of Commerce, Industry, and Energy.

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