Grain size reduction of $SmFe_{12}$-based powders; toward development of bulk high-performance permanent magnet

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Recent investigations on intrinsic magnetic properties of $SmFe_{12}$-based compounds with the $ThMn_{12}$ structure have shown that $Sm(Fe_{0.8}Co_{0.2})_{11}Ti$ and $Sm_{0.8}Zr_{0.2}(Fe_{0.8}Co_{0.2})_{11.5}Ti_{0.5}$ alloy compounds have comparable intrinsic hard magnetic properties and better temperature dependence with those of $Nd_2Fe_{14}B$ [1]. The remaining challenge is to further develop those intrinsic properties into practically useful extrinsic ones, particularly coercivity. In this work, we focused on reduction of the grain size of $SmFe_{12}$-based compounds by jet-milling and investigated the phase stability of the powders with different particle size [2,3]. We also introduce a few grain boundary phases that can exchange decouple $SmFe_{12}$-based grains in order to obtain high coercivity.

Starting alloys with compositions of $Sm(Fe_{0.8}Co_{0.2})_{11}Ti$, $Sm(Fe_{0.8}Co_{0.2})_{10.5}Ga_{0.5}Ti$, and $Sm(Fe_{0.8}Co_{0.2})_{10.5}Cu_{0.5}Ti$ were prepared by induction melting. The ingots were crushed into coarse powders and hydrogen decrepitated to reduce the particle size below 100 μm. The powders were jet-milled for the particle refinement study. The magnetic properties were measured using a SQUID-VSM. Microstructure of the samples were analyzed using SEM/FIB (Carl Zeiss 1540EsB) and TEM (Titan G2 80-200).

We used three different compositions of $SmFe_{12}$-based alloys and demonstrated fine, anisotropic, single-crystalline $SmFe_{11}Ti$-based micro-particles with high roundness by jet-milling using N_2 gas. The smallest particle size of 2.7±0.6 μm and roundness of 0.73±0.13 was achieved in the case of $Sm(Fe_{0.8}Co_{0.2})_{10.5}Cu_{0.5}Ti$, as shown in Fig. 1 (a). Although the $ThMn_{12}$-type structure is preserved even at high milling gas pressures, the coercivity of the powders remained below 0.2 T. The intergranular phase that originally exist in the as-cast alloy is removed or changed to an amorphous surface phase as shown in Fig. 1 (b). Detailed characterization of jet-milled powders using BF and HAADF-STEM showed that the $SmFe_{12}$-based jet-milled powders experience plastic deformation through crystallographic slip and the formation of slip bands during jet-milling process (Fig. 1(c)). Observed projection of the atoms in HAADF-STEM image (Fig. 1(c)) show that Sm atoms are missing in the slip bands indicating the crystal structure is damaged at the slip bands. These defects decrease local magnetocrystalline anisotropy compared to the matrix region that can be nucleation centers for the formation of reverse magnetic domains and thus limiting the coercivity. We will show that possible solutions for minimizing defect formation are, using lower milling gas pressure, performing a post-annealing step or using Ga doped starting alloy. Temperature-dependent XRD results showed that unlike large sized powders (>100μm), jet-milled powders with a size of smaller than 5μm decompose from the surface at temperatures above 600°C resulting in formation of α-Fe. We will also show low-melting alloys with good wettability with $SmFe_{12}$-powders that can be used for metal bonded magnets.

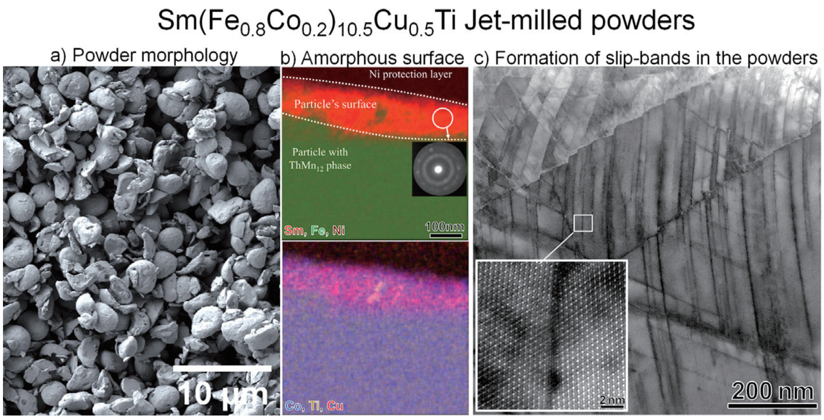

Figure 1: (a) Secondary electron (SE) SEM image showing morphology of jet-milled powders, (b) STEM-EDS maps and micro-diffraction pattern showing amorphous Sm-Cu rich phase at the surface of jet-milled powders, and (c) bright field (BF)-STEM and high resolution HAADF-STEM image (inset) obtained from inside of the jet-milled powders with composition of $Sm(Fe_{0.8}Co_{0.2})_{10.5}Cu_{0.5}Ti$.

Reference
[1] P. Tozman, H. Sepehri-Amin *et al.* Acta Mater. 153 (2018) 534.
[2] I. Dirba, H. Sepehri-Amin *et al.* Acta Mater. 165 (2019) 9. [3] I. Dirba, H. Sepehri-Amin *et al.* Submitted.