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Microstructural Development in Melt-spun $Nd_2Fe_{14}B$ Under High Magnetic Field Annealing

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Using an external magnetic field during heat-treatment can stimulate formation of texture, bias spinodally decomposed phase morphologies, promote martensitic transformation, as well as improve coercivity of permanent magnets. This is because magnetic field changes the energy of the system and thus its equilibrium state. It has been demonstrated that magnetic field has the strongest effect on materials structure and properties when the heat treatment was performed around the phase transformation temperature. [1,2] Here, field effect was studied on rapidly quenched amorphous melt-spun $Nd_2Fe_{14}B$ + TiC ribbons. Suppression of crystallization was observed for samples treated below 660°C with a 9 T field, while the field effect is much less distinctive for samples treated at 675°C. This work demonstrates that high field annealing is most effective in controlling microstructure of rare-earth permanent magnets close to the onset of a phase transition.

The $Nd_2Fe_{14}B$ + TiC ribbons were prepared by melt spinning at 25 m/s with a chamber pressure at 1/3 atm, helium ejection pressure of 120 Torr, and an ejection temperature of 1375°C. Magnetic properties were measured with a Quantum Design VersaLabTM VSM at 300 K. TEM samples were prepared in cross-section by mechanical polishing followed by low voltage ion-mill with liquid nitrogen cold stage. TEM was performed using a Titan Themis with Super-X energy-dispersive X-ray detector.

Figure 1a shows DSC curve of the as-spun ribbon. The ribbons were crushed into sub-millimeter flakes and firmly pressed into a DSC cup. At a heating rate of 10 K/min the crystallization process starts at ~625°C and ends at ~650°C. As-spun ribbons were annealed between slightly under the onset of crystallization, and 15°C above the completion of crystallization. Figure 1b and 1c show magnetic properties of the as-spun material after heat-treatment for 15 min between 600°C and 675°C in applied magnetic fields of 0 and 9 T, respectively. Gradual increase of crystalline phase fraction was observed with increasing annealing temperature. The 9T field suppresses grain growth as seen by a larger phase fraction of soft magnetic phases at 600°C, 630°C, and 660°C.

Figure 2 shows cross-sectional TEM image of the ribbon treated at 630°C. Sample annealed at 630°C, 0 T has uniform $Nd_2Fe_{14}B$ grains of 10-15 nm, as shown in Fig. 2a. TiC precipitated out as nano-clusters (<5 nm) and are distributed mostly at triple junctions and grain boundaries. Corresponding SAED pattern (Fig. 2b) shows well-defined rings which was indexed as crystalline $Nd_2Fe_{14}B$. However, sample annealed at 630°C, 9 T remained amorphous and showed no phase separation (Fig. 2c), which is consistent with its SAED pattern with diffuse rings (Fig. 2d). EDS analysis on grain boundary phases and Lorentz microscopy study on magnetic domain structure evolution is ongoing [3].

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References:

[1] Lin Zhou *et al,* Acta Materilia **133** (2017), p. 73.
[2] Michael A.McGuire *et al,* Journal of Magnetism and Magnetic Materials **430** (2017), p. 85.
[3] This research was supported by the Critical Materials Institute, an Energy Innovation Hub funded by the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, Advanced Manufacturing Office.

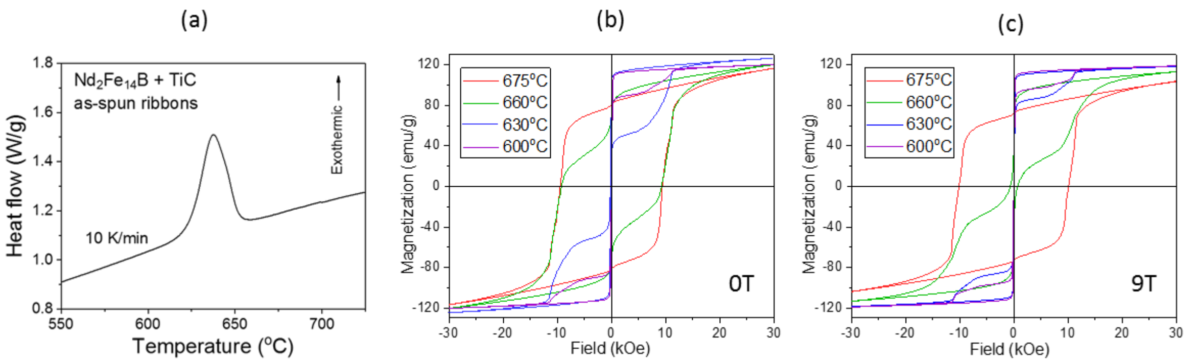

Figure 1. (a) DSC curve of as-spun ribbon; Hysteresis loop measured by VSM of heat-treated ribbons at different temperature under no external field (b) or 9T field (c).

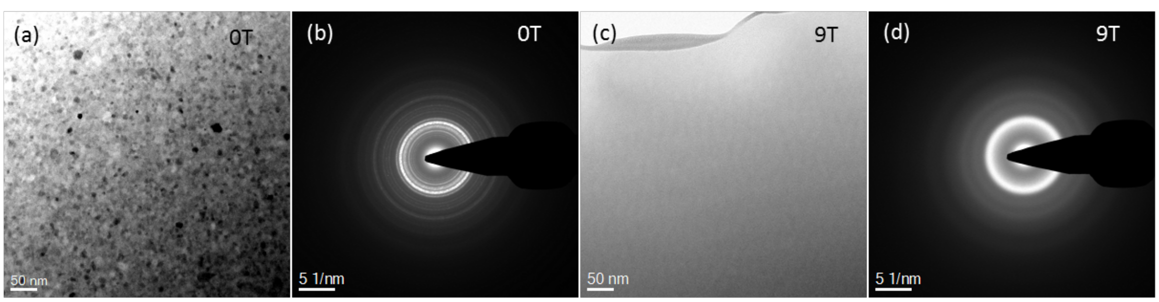

Figure 2. BF TEM image and corresponding SAED pattern of the ribbon treated at 630°C with no field (a, b) and with a 9T field (c,d).

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