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Magnetic Imaging of Nanocomposite Magnets

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Understanding the structure and magnetic behavior is crucial for optimization of nanocomposite magnets with high magnetic energy products. Many contributing factors such as phase composition, grain size distribution and specific domain configurations reflect a fine balance of magnetic energies at nanometer scale. For instance, magnetocrystalline anisotropy of grains and their orientations, degree of exchange coupling of magnetically soft and hard phases and specific energy of domain walls in a material.

Modern microscopy, including Lorentz microscopy, is powerful tool for visualization and microstructure studies of nanocomposite magnets. However, direct interpretation of magnetically sensitive Fresnel/Foucault images for nanomagnets is usually problematic, if not impossible, because of the complex image contrast due to small grain size and sophisticated domain structure. Recently we developed an imaging technique based on Lorentz phase microscopy [1-4], which allows bypassing many of these problems and get quantitative information through magnetic flux mapping at nanometer scale resolution with a magnetically calibrated TEM [5]. This is our first report on application of this technique to nanocomposite magnets.

In the present study we examine a nanocomposite magnet of nominal composition $Nd_2Fe_{14+\delta}B_{1.45}$ ($14+\delta=23.3$, i.e. "hard" $Nd_2Fe_{14}B$-phase and 47.8 wt% of "soft" α-Fe phase ($\delta=9.3$)), produced by Magnequench International, Inc. Conventional TEM/HREM study (Fig.1-2) suggests that material has a bimodal grain-size distribution with maximum at d_{max}=25 nm for $Nd_2Fe_{14}B$ phase and d_{max}=15 nm for α-Fe phase (Fig.1c, Fig.2) in agreement with synchrotron X-ray studies (d_{max}=23.5 nm for $Nd_2Fe_{14}B$ [6]). Lattice parameters for $Nd_2Fe_{14}B$ phase are a=8.80 and c=12.2 Å, as derived from SAED ring patterns (Fig.1a), again in good agreement with X-ray data. The fraction of large particles (of size ≥50 nm) is less then 5-7% of total amount of particles (Fig.1c, arrowed).

Our new imaging technique allows visualization of domain structure in nanomagnets (Fig.3) in color code. Both projected magnetization and magnetic flux maps (Fig.3) reconstructed using Lorentz phase microscopy suggest a complex domain structure with an average domain size about $100x(100\sim200)\ nm^2$ in a non-magnetized state. Large particles of darker contrast (of size ≥50 nm, presumably α-Fe precipitates) or clusters of such particles act as effective concentrators for magnetic flux in nanocomposite matrix. The measured relative local-flux concentration by factor of 1.28 agrees well with theoretical ratio $B_{sat}(\alpha\text{-Fe})/B_{sat}(Nd_2Fe_{14}B)=1.31$, strongly suggesting clustering of α-Fe particles that may have a detrimental effect on nanomagnet coercivity. Other smaller α-Fe particles (< 25 nm) do not disturb flux distribution, hence, they are magnetically coupled to Nd-Fe-B matrix grains as it was postulated by spring-exchange mechanism for nanocomposite magnets.

References

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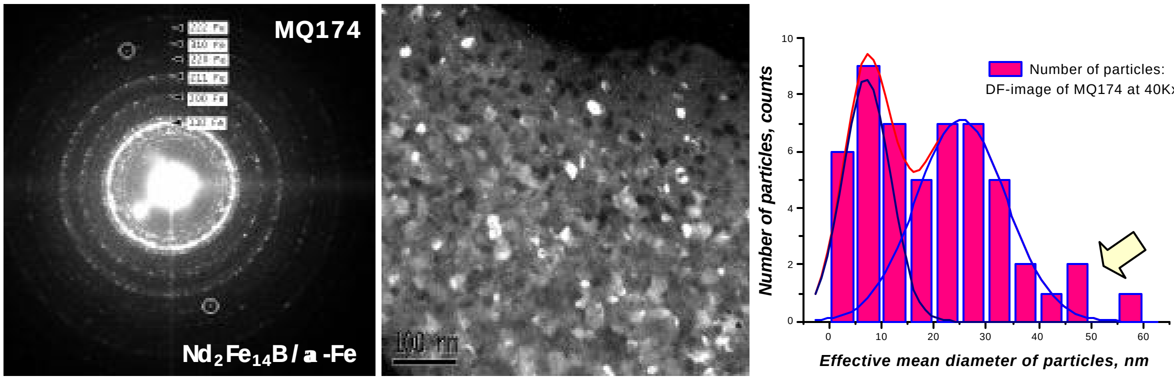

Fig.1 (a) SAED pattern of manocomposite magnet $Nd_2Fe_{23.3}B_{1.45}$ showing the presence of α-Fe particles, (b) dark field image and (c) particle size distribution derived from (b) with maximum at 15 nm for α-Fe phase and 25 nm for 2-14-1 hard magnetic phase. Notice presence of few particles with size about 50-60 nm (marked by arrow).

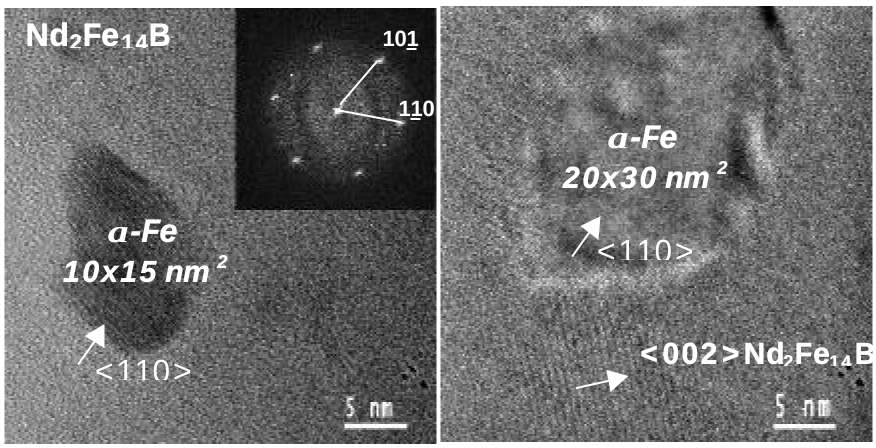

Fig.2 HREM images showing the presence of α-Fe particles in 2-14-1 matrix. Most of them are of 15 nm size (a) in agreement with histogram (Fig.1c), while some may be as large as 30-60 nm (b), resulting in local deviation from the matrix-averaged magnetic behavior, as proved by magnetic image analysis in Fig. 3.

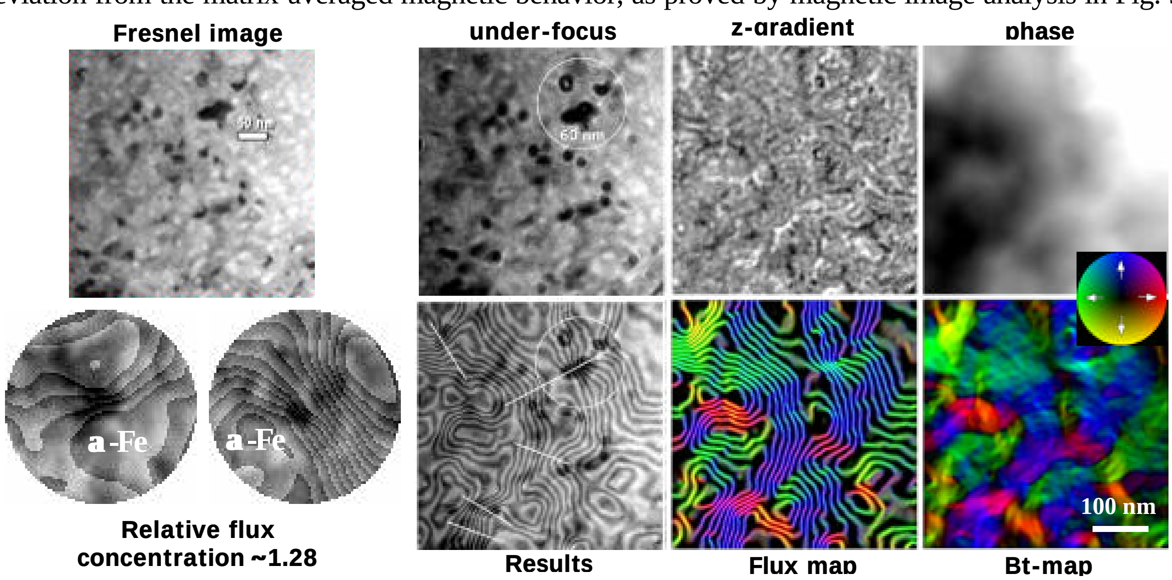

Fig.3 Lorentz phase microscopy used for magnetic flux and projected induction (Bt) mapping in nanocomposite magnet $Nd_2Fe_{23.3}B_{1.45}$ studied above by HREM/SAED (Fig.1-2). Reconstructed domain structure (of typical size 120-150 nm) is shown by color-code Bt-map. Some α-Fe particles (encircled) with size above 50 nm concentrate magnetic flux by measured factor 1.28 (theoret. 1.31) and, hence, may really deviate from spring-exchange mechanism postulated for nanomagnets.