Interaction between exsolution microstructures and magnetic properties of the magnetite-spinel solid solution

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ABSTRACT

Magnetic properties of exsolved samples of the (Fe₃O₄)(MgAl₂O₄)ₓ solid solution were measured over the temperature range 4–923 K and were correlated with microstructures observed by transmission electron microscopy. Several stages of microstructural development were identified. The early stages of exsolution are characteristic of spinodal decomposition with sinusoidal fluctuations in composition occurring parallel to (100). Satellite peaks observed in both electron and X-ray diffraction patterns indicate an average wavelength for the fluctuations of 100 Å. Further development of this microstructure is characterized by an increasing amplitude of the fluctuations and a sharpening of the compositional profile. A fully exsolved sample produced at 700 °C consists of two phases with approximate compositions x = 0.19 and x = 0.92.

Saturation magnetization (Mₛ), saturation remanent magnetization (Mᵣ), and coercivity (Hᵥ) were determined from hysteresis loops measured at temperatures in the range 4–300 K. Alternating-field susceptibility (χₐ) was measured at high temperatures up to 923 K. The temperature dependence of magnetic properties was found to be a complex function of the intrinsic properties of the homogeneous solid solution and the observed microstructure. A common feature of all exsolved samples is a pronounced decrease in Mₛ at temperatures below the Curie temperature of material with x < xₑ, where xₑ is the compensation point identified by Harrison and Putnis (1995). In the early stages of spinodal decomposition we observed samples with pronounced coercivity (Hᵥ = 173 mT) and unusually high remanence (Mᵣ/Mₛ = 0.82) at 4 K, characteristic of magnetization reversal by strongly pinned conventional domain walls. At temperatures in the range 50–100 K the coercivity and remanence decrease rapidly, with hysteresis loops characteristic of magnetization reversal by weakly pinned interaction domain walls above 100 K. This transition in domain state is related to the development of paramagnetic boundaries around the Fe-rich components of the compositional fluctuations.

In the advanced stages of exsolution we observed coercivity and remanence more consistent with single-domain behavior. This is related to the sharpening of the compositional profile and the development of distinct lamellae with high shape anisotropy.

INTRODUCTION

The interaction between magnetism and microstructure is arguably one of the most important mechanisms for increasing the stability of a natural remanent magnetization (NRM) over geological time. A wide range of microstructures exists in natural magnetic minerals associated with high-temperature oxidation (Tucker and O’Reilly 1980), cation ordering (Nord and Lawson 1989), spinodal decomposition (Smith 1980), and even lightning strikes (Banfield et al. 1994). A common cause of increased coercivity and remanence is the subdivision of large multidomain grains into fine-scale arrays of single-domain grains separated by exsolved lamellae of a non-magnetic phase. In natural titanomagnetite, for example, it is commonly proposed that exsolved lamellae of ilmenite, which are formed during oxidation at high temperatures, lead to enhanced remanence properties of the host spinel phase (Graham 1953; Larson et al. 1969; Strangway et al. 1968). Some workers suggest, however, that the scales of these oxidation-exsolution textures are too large to yield single-domain properties (for example, Hargraves and Banerjee 1973). A potential source of much finer-scale exsolution textures is subsolvus exsolution, which occurs in titanomagnetite at temperatures <500 °C (Price 1981). This phenomenon has been exploited for many years in the production of commercial permanent magnet materials (Livingston 1981). Typically exsolution occurs by a mechanism of spinodal decomposition, which yields a tweed-type microstructure consisting of sinusoidal fluctuations in composition with a