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Displacement Fields Associated to Chemical Modulations in Sintered Ceramics After Spinodal Decomposition.

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Ceramics oxides in the system FeO-Fe_3O_4 have been produced by means of mechanical milling and sintering for application as electromagnetic wave absorbers. Starting from powder mixtures of Fe and Fe_3O_4 or Fe and Fe_2O_3, as well as pure Fe_3O_4, a metastable solid solution of Fe and O has been produced. A low energy mill produces a solid solution after around 1000 h of milling. X-ray diffraction patterns show the formation of the solid solution but it is unclear whether a Fe lean wüstite or an oxygen rich magnetite is formed. However use of Mössbauer spectroscopy reveals an increasingly higher amount of metastable wustite ($Fe_{(1-X)}O$) as a function of milling time i.e., 500 h and 1000 h of milling produce 67 and 74 mol % $Fe_{(1-X)}O$, respectively. Magnetic measurements also indicate the formation of a paramagnetic phase which corresponds most likely to wüstite. Consolidation of the mechanically milled powders has been achieved by means of plasma assisted sintering. Specimens sintered at relatively high temperature (T>1073 K) show characteristics of a spinodal decomposition. Lower sintering temperatures (673-773 K) give rise to microstructures similar to the as-milled powders.

Conventional TEM and high resolution electron microscopy (HREM) have been used to characterize the spinodally decomposed microstructure found after sintering [1]. Nevertheless such characterization can be extended with the measurement of the displacement fields produced by the chemical modulation of cations in the material. Wüstite and Magnetite have a unique Oxygen lattice and a different distribution of Fe cations [2]. Magnetite and wüstite have both a cubic structure (spinel, a_0 = 0.8411 nm and NaCl a_0=0.43538 nm, respectively). Wüstite can have large deviations from stoichiometry. During the spinodal decomposition of the supersaturated wüstite formed during milling, a periodic array of domains of the two phases can be observed. Figure 1a shows a representative experimental HREM image showing the two phases (B = [001]). The different domains can be recognized more easily in Fig 1b obtained by creating a Moire pattern. Fig. 2 shows the modulations in a different orientation (B = [111]). Fig. 2a shows a experimental HREM image with some areas corresponding to the two phases. Careful observation shows a modulation in the intensity of the interference maxima corresponding to the cations i.e., the characteristic chemical modulation of an spinodal decomposition. Figure 2b shows a reconstructed image from an area close to that in Fig. 2a. A through focal series has been used for the reconstruction process. The chemical modulation can also be observed in this case. However evaluation of the corresponding displacement fields has a higher accuracy than in the experimental images. Several strain maps have been produced in this manner.

References

1. A. Huerta, H.A: Calderon, M. Umemoto, M.E. Brito, Microcopy and Microanálisis 6 (Sup.2) (2000) 366.
2. M.E. Fine, Advances Mats. Research 4 (1970) 1.

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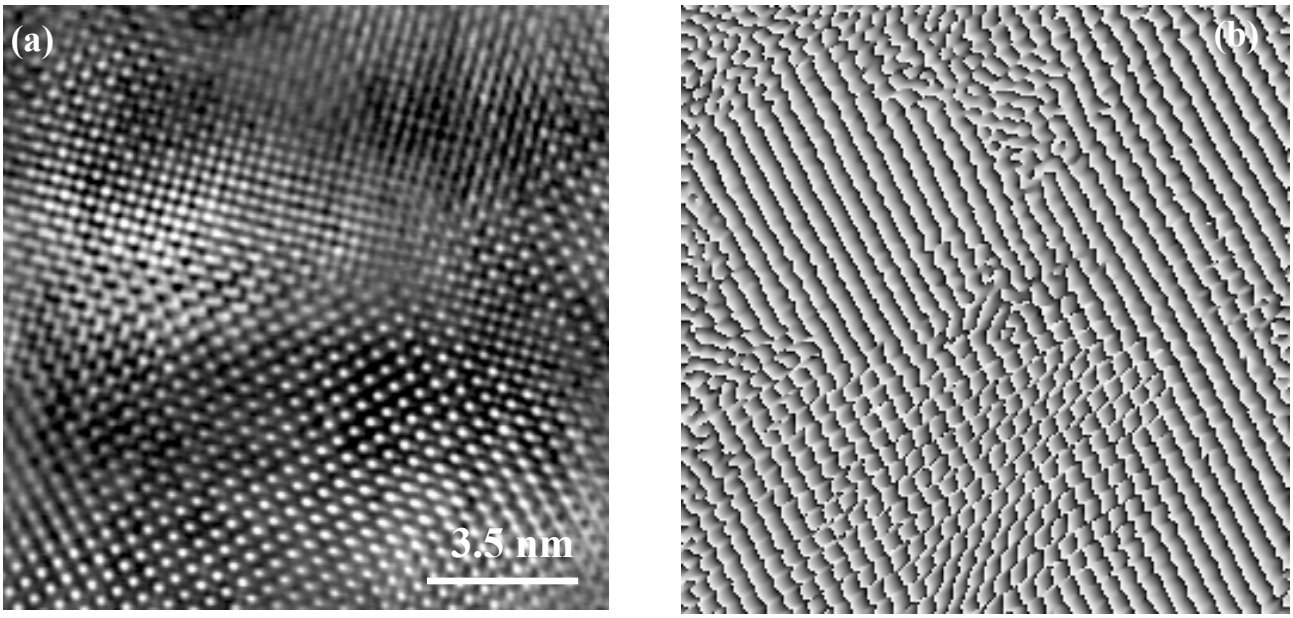

Figure 1. Fe_3O_4 –15 at.% Fe milled for 200 h and sintered at 1273 K. (a) Experimental HREM image with B = [001]. (b) Moire image formed with interference every two lines.

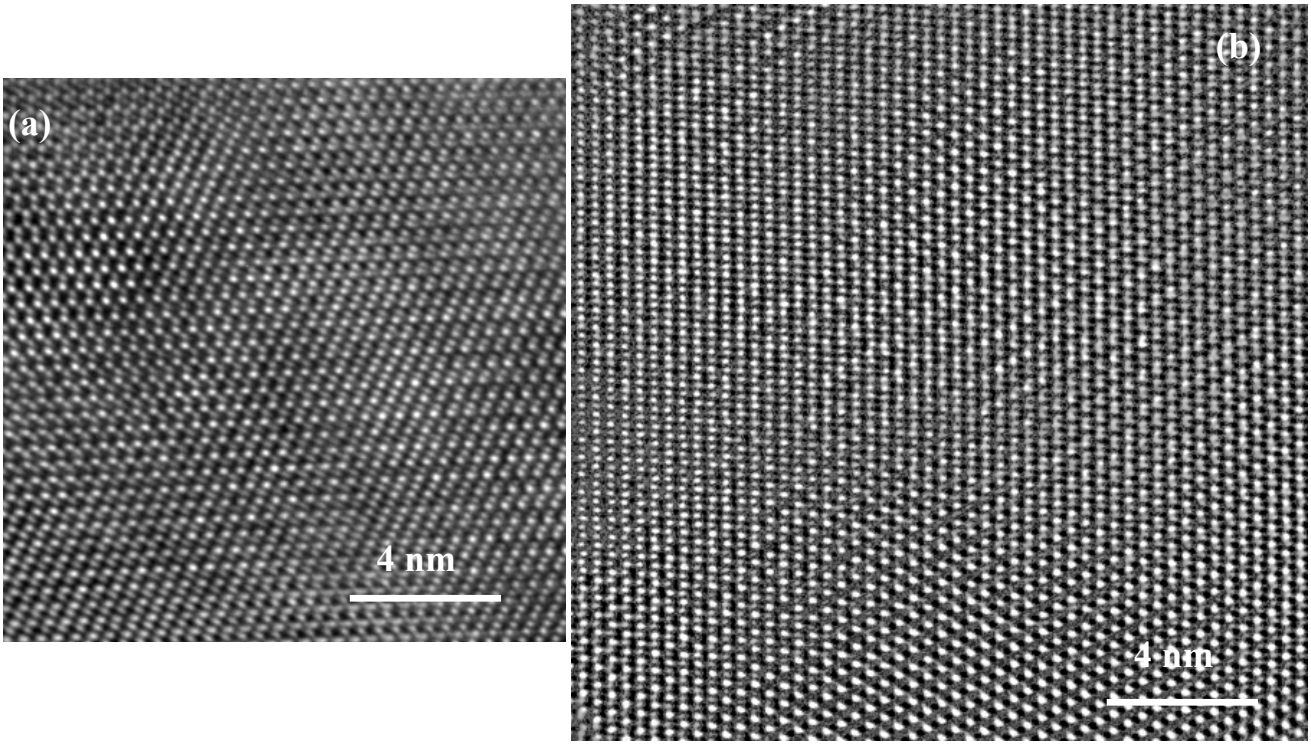

Fig. 2. (a) Experimental image corresponding to a Δf = -200 nm. (b) Reconstructed image showing two phases and the corresponding chemical modulation. The nominal composition of the sample is Fe_3O_4 – 15 at.% Fe milled for 600 h and sintered at 1273 K.

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