CHARACTERIZATION AND PRODUCTION OF STRUCTURAL CERAMICS IN THE SYSTEMS $Fe_{(1-x)}O$-Fe_3O_4 AND MgO-$MgFe_2O_4$.

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Ceramic materials are widely studied for their high temperature structural applications. In many crystalline ceramics the range of solid solution decreases with temperature and thus precipitation of a second phase occurs. Thus, ceramics can be hardened by precipitation of second phases. However little is known regarding the effect of precipitation and nanocrystalline grain structure in the ductility of ceramic materials. On the other hand, oxide ceramics are under intense investigation for their technological advantages in magnetization, dielectric response and chemical stability in such diverse uses as magnetic recording media, induction cores and microwave resonant circuits. This investigation has been undertaken to produce, characterize and measure the properties of ceramics that can be hardened by precipitation. The selected systems include $Fe_{(1-x)}O$-Fe_3O_4 and MgO-$MgFe_2O_4$. Mechanical milling is used to produce nanocrystalline ceramic oxides in the systems $Fe_{(1-x)}$-Fe_3O_4 and MgO-$MgFe_2O_4$ The mechanically alloyed powders are consolidated by means of spark plasma sintering (SPS) at temperatures ranging from 673 K to 1273 K and a pressure varying from 500 to 50 MPa in vacuum. This technique allows consolidation in sufficiently short times to preserve the nanocrystalline structure developed during the mechanical milling. The microstructural characterization has been carried out by X-ray diffraction (XRD), Mössbauer spectroscopy and conventional and high resolution transmission electron microscopy (TEM and HREM).

Figure 1a shows the XRD patterns of a sample composed of Fe_3O_4 after mechanical milling for different times. A change in the nature of the original component can be noticed as indicated by the broadening of the diffraction peaks. Fig. 1b is a Mössbauer spectrum showing that after 500 h of milling $Fe_{(1-x)}O$ becomes the predominant phase with minor amounts of Fe_3O_4 and Fe_α. During mechanical milling a nanocrystalline grain structure develops as is shown in Fig. 2 for the system MgO-$MgFe_2O_4$ after 20 h of high energy milling. The TEM dark field in Fig. 2 has been taken using a MgO reflection. In the case of the system $Fe_{(1-x)}O$-Fe_3O_4, the temperature of sinterization produces different volume fractions of $Fe_{(1-x)}O$, higher temperatures increase the final amount of Wüstite. Fig. 3 shows X-ray spectra of Fe_3O_4 specimens sintered at different temperatures. On the other hand the spatial distribution of the phases after sintering is relatively homogeneous as can be seen in Fig. 4. Two TEM dark fields are shown in this figure, taken with different reflections. Fig. 4a is produced by a reflection of $Fe_{(1-x)}O$ and Fig. 4b by using a Fe_3O_4 reflection. The nanocrystalline grains size is also apparent. HREM is also used to identify the phases in the produced nanocrystalline ceramics. Fig. 5 shows a nanosized grain of $Fe_{(1-x)}O$ in a specimen sintered at 1073 K and 50 MPa after 1000 h of milling.

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