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Phase Transformations Induced by Mechanical Milling and Sintering in the (Fe, Al)-Fullerenes Composites

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The present work deals with phase transformations in (Fe, Al)-fullerenes composites with special attention in the changes produced in the fullerene structure during mechanical milling (MM) and sintering. MM is described as a high energy milling process in which powder particles are subject to repeated cold welding, fracturing, and rewelding producing non equilibrium structures and nanocrystalline materials. The Spark Plasma Sintering (SPS) technique is commonly used to preserve the nanostructure achieved during MM, applying high pressures and electric pulse current through the sintering mixture for short periods of time.

Mixtures of (Fe, Al)-2.5% C_{60} are prepared by MM in a high-energy SPEX mill. Elemental powders of fullerene C_{60} (purity 99.99%) and Fe matrix (purity 99.99% and an average particle size of 50 µm) or Al matrix (99.9% and 30 µm) are used. Milling time has been varied from 0.5 to 2 hours in order to follow the alloying process. A variety of ball mills is used in the experiments (4.9 and 9.6 mm in diameter). The consolidation of powders has been performed by SPS at 775 K and 650 K for Fe-C_{60} and Al-C_{60} system respectively, with an external pressure equal to 15 kN. The phase transformations and structural changes occurring in the studied material during MM and subsequent sintering process are investigated by Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), X-ray diffraction (XRD) and Raman spectroscopy.

XRD shows that by using balls of 9.6 mm in diameter, the fullerene can be transformed during MM, whereas milling with balls of 4.9 mm leaves the fullerene structure apparently unchanged [1, 2]. Raman spectroscopy confirms the phase transformation from fullerenes to carbides and diamonds in the milled powders using the large balls. Fig. 1 shows the characteristic spectrum of pure fullerene (1a) and the spectrum of the milled Fe-C_{60} powders after 2 h of MM (1b). The small peak around 1330-1340 cm^{-1} (as indicated by the dotted line in Fig. 1b) indicates the position expected for diamond which is unique for this phase [3]. Fig. 2 shows TEM and dark field-TEM images with the corresponding diffraction pattern obtained with a fullerene reflection of resulting powders after 2 h of MM to achieve a massive nanostructure. Furthermore some areas (indicated by arrows) appear in the dark field image with a different contrast and shape corresponding more likely to other phase. The HRTEM image confirms the existence of diamond in the milled powders of Fe-C_{60} system by 2 h of milling (see Fig. 3). The lattice images in Fig. 3 corresponding with a nanocrystalline rhombohedral diamond structure and the Fourier transform agrees with the value for a diamond phase (0.251 nm) [4]. Fig. 4 shows SEM images of Fe-fullerene milled powders by 2 h. Fig. 4a shows particles with irregular morphologies with some possibility to comminute further. SEM images analysis of the sintered products (Fig. 4b) shows that longer milling times promote higher densification, since powder particles become smaller and more regular as the milling time increases. The transfer of mechanical energy to the powder particles results in the phase transformation of fullerene to hard phases like diamonds and carbides when the energy supplied by processing is large enough and also by SPS technique to Fe-C_{60} and Al-C_{60} systems.

References

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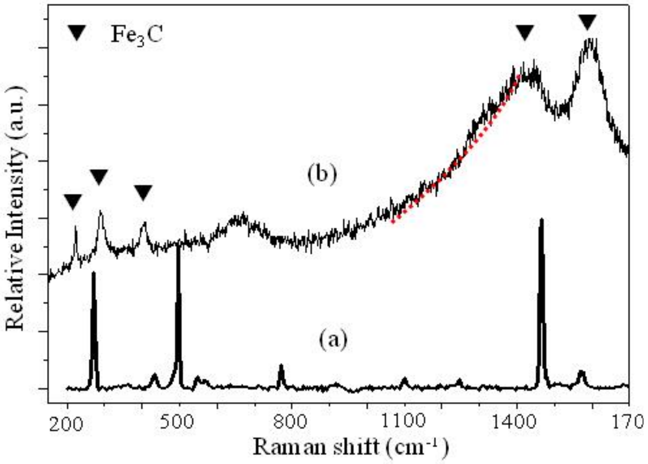

Fig. 1- Raman spectra for (a) pure fullerene and (b) Fe-C_{60} milled powders by 2 h.

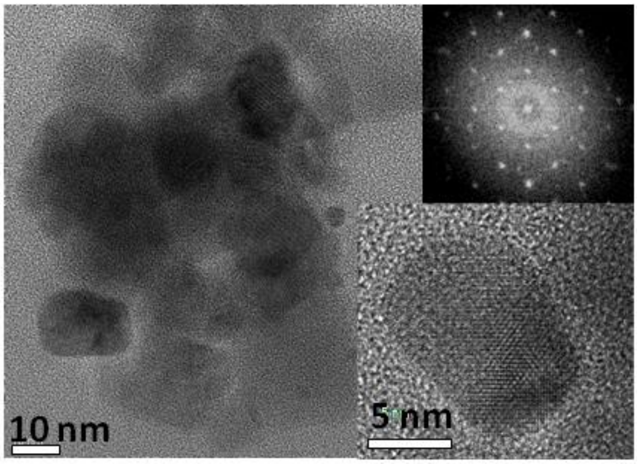

Fig. 3- Raman spectra for (a) pure fullerene and (b) Fe-C_{60} milled powders by 2 h.

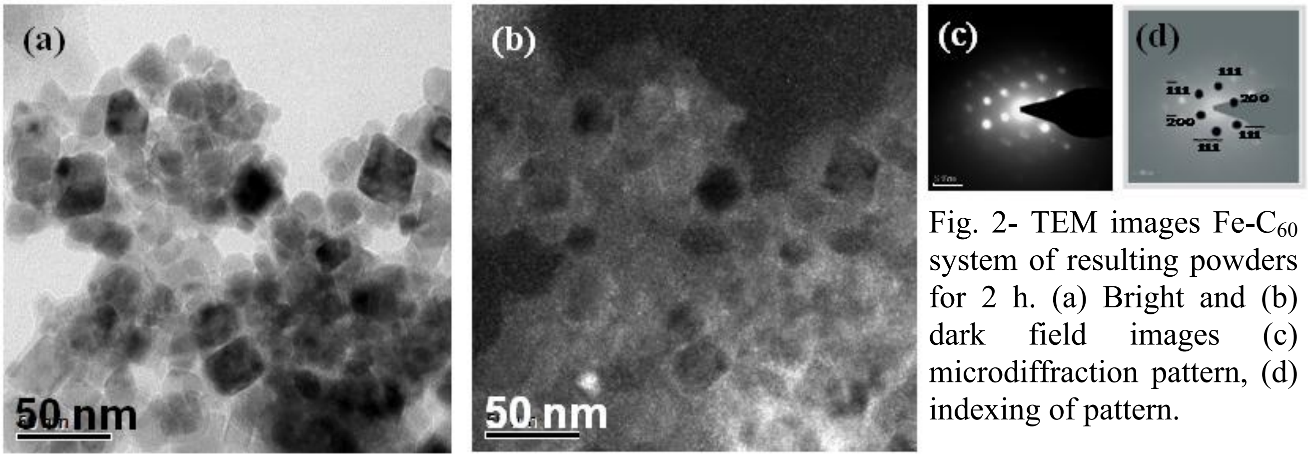

Fig. 2- TEM images Fe-C_{60} system of resulting powders for 2 h. (a) Bright and (b) dark field images (c) microdiffraction pattern, (d) indexing of pattern.

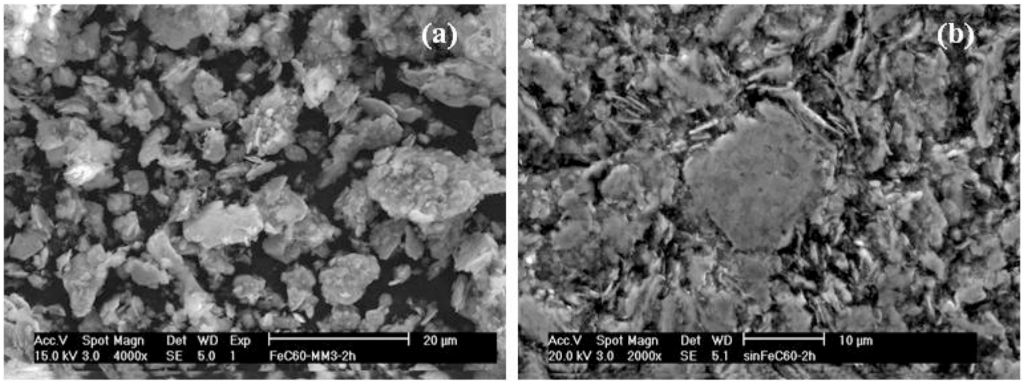

Fig. 4- SEM images of (a) milled powders and (b) sintered sample of Fe-C_{60} system by 2 h of MM.