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Metal (Fe, Al)-Fullerene Nanocomposites: Synthesis and Characterization.

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Recently the production of composites with a metallic matrix and nanostructures of carbon has become an interesting subject of investigation due the discovery of better mechanical properties [1]. In the present work the objective is to form solid composites of fullerenes inside a metallic matrix by powder metallurgy methods. A higher material strength is expected via the formation of hard phases in the composites. Fe-2.5 mol % C_{60} and Al-2.5 mol % C_{60} composites have been prepared by mechanical alloying of pure powders with a high energy Spex mill. Milling has been varied from 0.5 to 2 hours in order to follow the alloying process. Spark Plasma Sintering (SPS) has been used to consolidate the resulting powders. This technique helps to preserve the nanostructure of the composite (developed during milling) owing to the low temperatures and short periods required by the process. Results of X-Ray Diffraction and Raman Spectroscopy show that by using balls of 10 mm diameter, the fullerenes are destroyed during mechanical milling, whereas milling with balls of 5 mm does not affect the structure of the C_{60} fullerenes. SEM shows homogeneous powders with different particle sizes depending on milling times. Sintering has been performed by using SPS at 773 K and applying 15 kN. Samples with 20 mm in diameter and 2 mm in thickness have been produced.

X-ray diffraction patterns show the development phases present after milling and sintering in Fig. 1. As can be seen, C_{60} fullerenes withstand the process but there is also formation of carbide phases, particularly after sintering. Milling depending on energy input, can alter the development of phases. However for the following results, proper conditions are found to preserve fullerenes from reacting during milling. The dispersion of fullerenes depends on the nature of the metal. Fe powders become nanocrystalline from very early milling times as can be seen in Fig. 2. The corresponding dark field image (taken with a fullerene reflection) shows a fine crystallite size and a rather good dispersion of fullerenes. Fig 3 shows an Fe-C_{60} composite powder particle after 2 h of milling in a bright field image with an Fe crystallite average size below 20 nm. In the case of Al, the dispersion is equally good but longer milling times are required. Fig. 4 shows a bright-dark field image combination with the details of the nanostructure. Figure 5 shows a lattice image of a Fe-C_{60} powder particle after 2 h of milling. Although the Fe lattice is not on axis and thus only faintly resolved, a rather fine C60 crystallite can be imaged almost on axis, surprisingly, the particle interface has no apparent holes meaning that the composite has formed with rather good cohesion. Both Fe and C_{60} retain their structure and at the same time a fine nanostructure is developed during milling. Sintering by SPS promotes development of carbide phases (see Fig. 1) but without a full reaction of the fullerene phases, together with a slight grain growth and a considerable strengthening.

References

[1] F.C. Robles-Hernandez, H. A. Calderon, JOM 62 (2010) 63.
[2] Financial support from IPN (COFAA-SIP) and CONACYT (Grant 58133) are gratefully acknowledged.

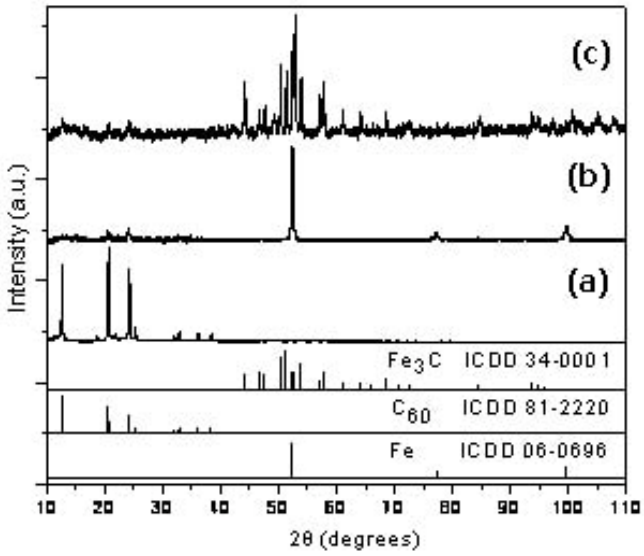

Fig. 1. X-ray diffraction patterns of Fe-C_{60}. (a) Pure C_{60}. (b) As milled (2 h) composite powders. (c) As sintered material.

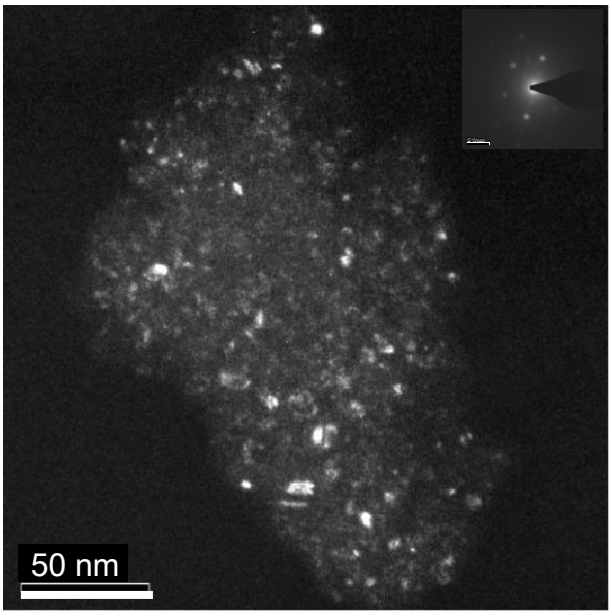

Fig. 2. TEM dark field image from fullerene reflection of as milled (30 min) Fe- C_{60} composite powders. Insert shows fullerene microdiffraction pattern. Tecnai 20 (FEI) microscope

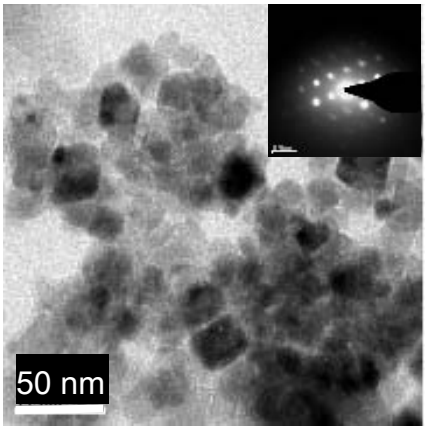

Fig. 3. Bright field image of as milled (2 h) Fe-C_{60} nanocomposite powders. Insert shows fullerene microdiffraction pattern.

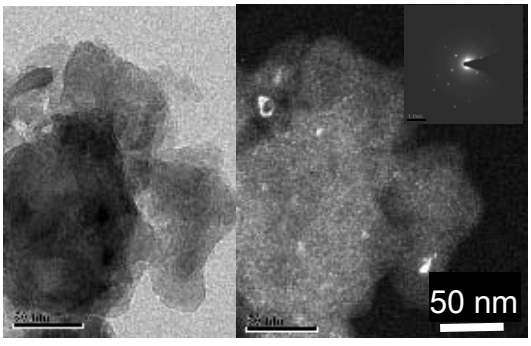

Fig. 4. Bright and dark field images of as milled (2 h) Al-C_{60} nanocomposite powders. Insert shows fullerene microdiffraction pattern.

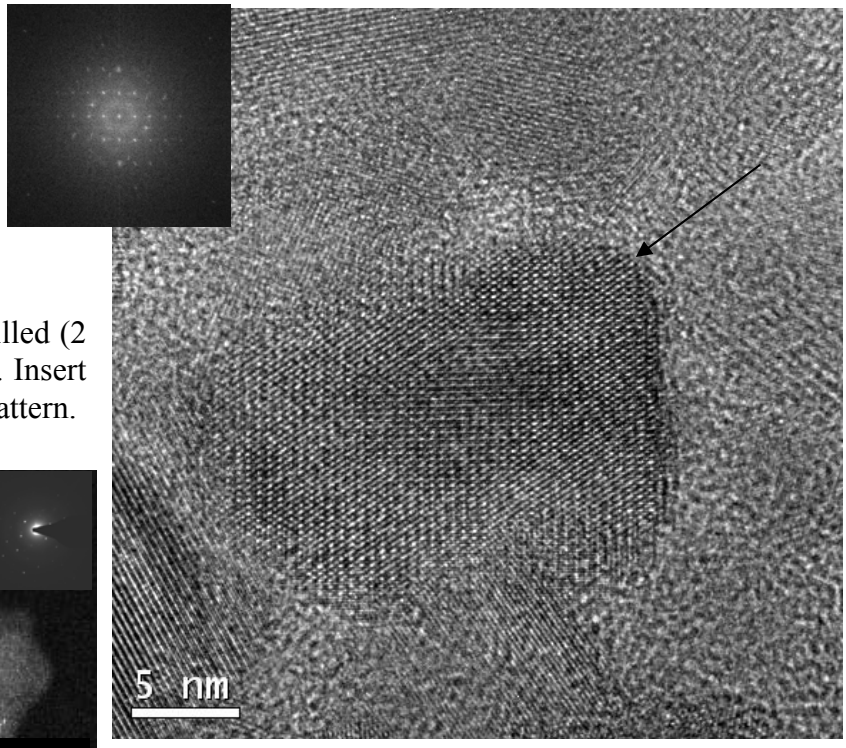

Fig. 5. High resolution TEM image of as milled (2 h) Fe-C_{60} nanocomposite powders. The arrow indicates a fullerene particle surrounded by the Fe matrix. Titan microscope (FEI). Insert shows FFT of C_{60} particle.

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