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The survivability of polycrystalline C_{60} to high speed vibration milling

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Abstract

For the solvent free mechanochemical synthesis of fullerene compounds as initiated by us in 1994, it became necessary to know how the polycrystalline C_{60} structures resist to high speed vibration milling (HSVM). We have shown that there is no mechanical damage in the C_{60} molecule up to a HSVM time of 30 min.

1. Introduction

In a paper published in 1994 we were able to show that some fullerene compounds are easier synthesized in a solvent free system than in solution, by mechanochemical ball milling [1]. This procedure has been further extended and the procedure's efficiency largely enhanced by replacing ball milling by high speed vibration milling (HSVM) [2].

Although our mechanochemical syntheses produce new C_{60} compounds at a quite acceptable output, we were now interested to know how the C_{60} crystals and molecules themselves are facing the intensive mechanical effect during the HSVM process, i.e., what is their survival during the mechanochemical synthesis.

This note deals with the above-mentioned topic.

2. Experimental

All the measurements were made with Gold Grade polycrystalline C_{60} (Hoechst A.G Frankfurt).

a. *Sample preparation*: Mass aliquots of about 60–80 mg of Hoechst Gold Grade C_{60} samples have been mechanically milled in an 5100-230 type SPEX Mixer/Mill using a hardened steel vial set (i.e., 1/2 in., diameter x 1 in. long with one steel ball) which provide a grinding load of 0.2–0.6 ml. The tray type clamp of the Mixer/

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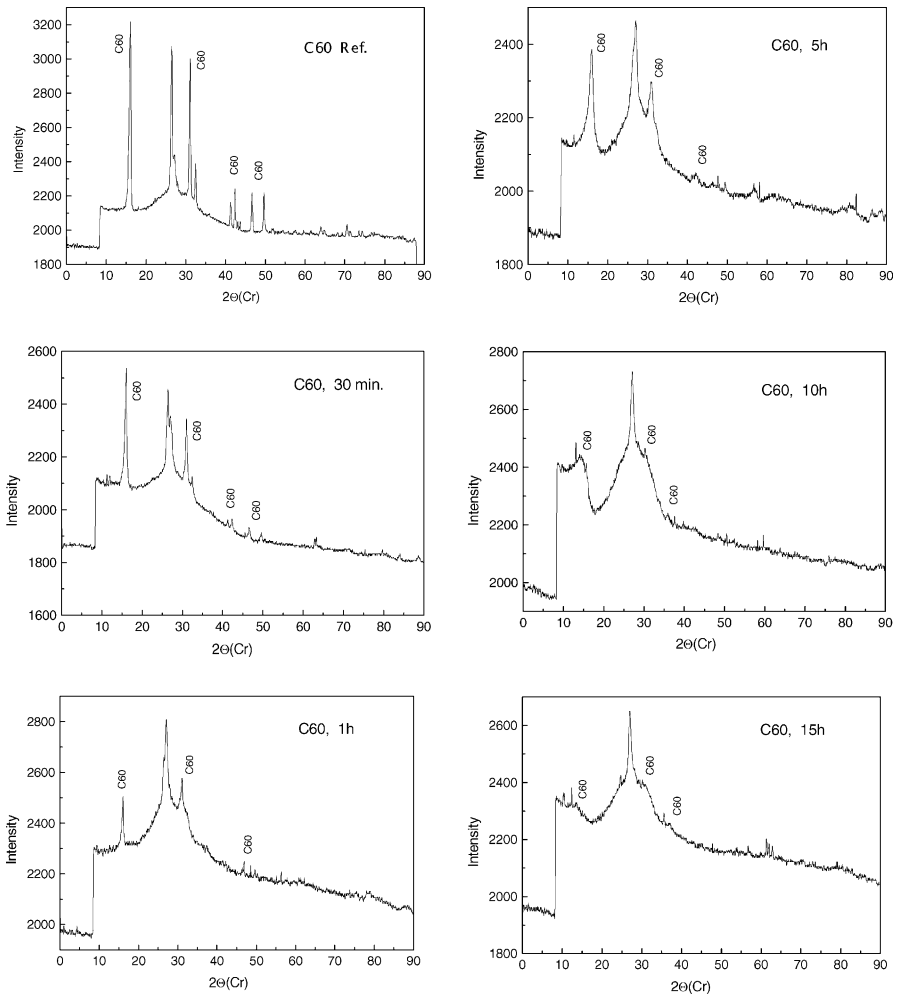

Fig. 1. X-Ray diffraction spectra of C_{60} fullerene samples measured after increasing milling periods.

Mill offers a vigorous motion along three axes with a frequency of 6000/min. The samples have been mechanically treated with milling periods of 30 min, 1, 5, 10 and 15 h.

b. *X-Ray diffraction (XRD)*: XRD measurements were carried out in Phillips Guiner XDC-700 camera with Cr-$K_{\alpha 1}$ radiation. The diffraction patterns were taken by a Zeiss MD100 photometer.

c. *HPLC*: The analytical measurements were carried out on a Dionex Summit HPLC system, consisting of a P 580 NDG precision pump with low-pressure gradient mixing, Rheodyne 8125 sample injector, 340S diode array detector and a Chromeleon chromatography data manager software system. Separations were carried out on a 250×4.6 mm 'Chromspher 5 Fullerenes' column packed with 5 μm particles.

Chromatographic conditions: eluent: isopropanol–isooctane: 10–90; flow rate: 1.2 cm^3 min^{-1}; detector: 254 nm; retention of C_{60} sample $k = 1.8 \pm 0.1$. Temperature: ambient.

3. Results and discussion

We have previously studied also the effect of nuclear reactor irradiation on C_{60} and have

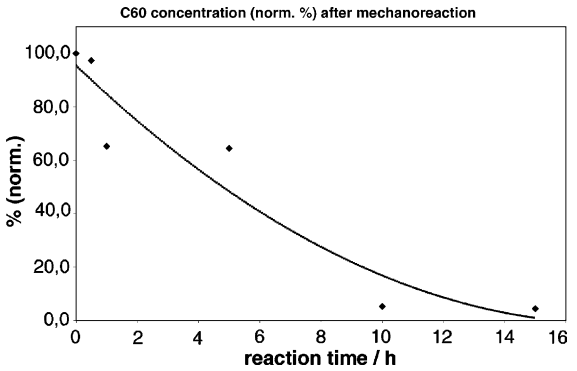

Fig. 2. Percentual survival of C_{60} (by HPLC) after different HSVM time periods.

detected that the irradiation smears out the 260 K solid–solid phase transition in the polycrystals [3]. The same effect has been indicated after mechanical treatment of C_{60} monocrystals and polycrystals by some previous authors [4,5].

The results of our XRD measurements for the polycrystalline C_{60} samples milled for different time periods are collected in Fig. 1. As visible the typical C_{60} peak begins slowly to decrease even after a milling time of 30 min but most of the C_{60} survives up to a milling time of even 5 h. At longer milling times the C_{60} XRD reflections disappear from the spectra. This disappearance is also indicated by the HPLC measurements in Fig. 2. As visible the analytically measured survival of C_{60} during the mechanochemical treatment shows a quasi linear tendency, confirming the XRD measurements. No other decomposition products were detected by HPLC after milling.

We presume that the mechanism of the damaging process leads successively to an amorphous carbon phase.

The important conclusion from our experiments is that the mechanochemical synthesis of new C_{60} compounds can be done by HSVM for even around 5 h. It is to be noted that in most of our syntheses [5] a HSVM time of 10 min has been used which assured a survival of quasi 100% of the C_{60} to be involved in the mechanochemical reaction. The high efficiency of HSVM has been revealed also by Japanese researchers in 1997 [6].

Amount of C_{60} (as determined by HPLC) still surviving after different HSVM times.

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