

## Sublimation and Heterogeneous Reactions of Fullerene (C<sub>60</sub>) in Different Gases

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### ABSTRACT

The sublimation process and heterogeneous reactions of C<sub>60</sub> Fullerene under a flow of argon, air, or hydrogen have been investigated at 25-1000 C. The results are compared against those obtained with other forms of well characterized carbons; Saran char (a highly reactive disordered carbon) and SP-1 graphite (less reactive, well ordered carbon). In Ar, 83% of the C<sub>60</sub> aggregates (examined here) sublime, leaving behind a carbonaceous non-volatile residue insoluble in toluene. In air, the C<sub>60</sub> first chemisorbs (reacts with) oxygen to a limit corresponding to 1.5 oxygen atoms/C<sub>60</sub> molecule. The carbon then loses weight (gasifies) at a rapid rate; higher than the rate of Saran gasification. In hydrogen, while the rates of SP-1 and Saran gasification at 1000 C are insignificant, the volatile part of C<sub>60</sub> aggregates completely sublimates or gasifies at 500-800 C, leaving behind the 17% residue left after the sublimation process. Some possible future applications of C<sub>60</sub>, particularly in the areas of carbon composites and molecular sieve materials, are addressed.

Keywords: C<sub>60</sub>, Buckyballs, Fullerene.

### INTRODUCTION

Fullerene C<sub>60</sub>, a newly established form of carbon, is a molecule containing 60 carbon atoms arranged in a closed hollow cage with 32 faces; 12 pentagons and 20 hexagons [1]. The molecule is a stable cluster of carbon atoms often known as "buckyball". The buckyballs are prepared by laser vaporization of graphite discs [1-3], and by carbon arc vaporization of graphite rods [4-6]. The evaporation is performed under subambient pressures (100-200 Torr) at 1000-1300 C in an inert atmosphere of helium or argon. The graphite vapor nucleates in the gas phase forming particles which are collected on a cold substrate, yielding soot and aggregates of C<sub>60</sub> with a small fraction of other larger cages. To isolate the buckyballs the particles are first dispersed in a solvent; benzene [4], toluene or carbon disulfide [5,6]. The buckyballs dissolve but the other forms of carbon (soot) remain suspended. After filtration, the solvent is evaporated, leaving behind aggregates of C<sub>60</sub>.

The structure of buckyballs has been supported by infrared and ultraviolet spectra [1,4], by NMR [7], and by theoretical calculations [8,9]. The literature is full of C<sub>60</sub> physical properties; including electrical and magnetic properties [10,11], electron energies [12-14] and optical spectrum [14]. Scanning Tunneling Microscopy has indicated that the C<sub>60</sub> molecules are arranged in hexagonal arrays with an intercluster spacing of 1.0-1.1 nm [15,16]. The spacing agrees with the sum of the theoretical molecular diameter of C<sub>60</sub> (0.701 nm) plus the interlayer spacing of graphite (0.335 nm). These values are also in agreement with those calculated from X-ray and electron diffraction studies [17]. With the assumption that the C<sub>60</sub> molecules are arranged in a hexagonal close-packed lattice, the estimated nearest neighbor distance is 1.002 nm, and the X-ray density is 1.678 g/cc. It is consistent with the experimental value of  $1.65 \pm 0.05$  g/cc obtained by density gradient columns [17].

The structure of C<sub>60</sub> and its different methods of preparation are well established. The buckyballs can now be prepared at a rate of 10 g/h or higher [6]. Yet, little is known about their properties and performance as a truly carbonaceous material. Because of their unique spherical structure, the C<sub>60</sub> molecule does not have dangling carbon atoms or active sites as the other carbonaceous materials. The C<sub>60</sub> molecules should be less reactive to air, hydrogen, or oxidizing agents than the other carbons. The treatment of soot and fullerene with perchloric acid at 100-160 C has verified this point [18]. The treatment indicated that fullerene is more oxidation resistant than soot [18]. On the other hand, C<sub>60</sub> sublimates at considerably lower temperatures than the other carbons. Therefore, when exposed to air at

high temperatures, the homogeneous reaction between air and  $C_{60}$  in the gas phase (if any) could be faster than the "normal" heterogeneous reaction between air and active sites of the other carbons. Thus, it is not clear how the behavior of  $C_{60}$  in different gases compares to the other forms of carbon.

The objective of the present work is to address several subjects. First, how does  $C_{60}$  respond when heated in inert or reactive medium. Initially, we discuss the sublimation reaction of  $C_{60}$  in Ar. Then we move to the reaction between  $C_{60}$  and air or  $H_2$ . Second, a comparison is made between the reactivity of  $C_{60}$  and other forms of carbon. We finally outline some potential future applications of this form of carbon; especially in the areas of carbon composites and molecular sieve carbons.

## EXPERIMENTAL

### Materials

Two batches (A and B) of  $C_{60}$  crystals were graciously supplied by Professor D. R. Huffman (University of Arizona - Tucson). They were used as-received, without further purification. To understand their relative reactivity in air or  $H_2$ , two additional carbons were included in the study; a reactive Saran char and a considerably less reactive SP-1 graphite. Saran is a highly disordered non-graphitic char with a large micropore surface area of  $850 \text{ m}^2/\text{g}$  [19], an average micropore width of  $1.05 \text{ nm}$  [20], and an active surface area (ASA) of  $37 \text{ m}^2/\text{g}$  [19]. The sample has a low level of impurities [19], so we can rule out the catalytic effects which enhance the rates of  $C/O_2$  and  $C/H_2$  reactions. SP-1, on the other hand, is composed of well ordered natural flakes which have been purified by the manufacturer to a minimum level of impurities. It is non-porous graphite with an external surface area of  $2.00 \text{ m}^2/\text{g}$  [21] and an ASA of  $0.05 \text{ m}^2/\text{g}$  [22].

### Measurements of Reaction Rates

The experiments were performed on a Cahn Thermogravimetric Analyzer (TGA), system 113, connected to a vacuum system. The samples (about 5 mg) were evacuated at room temperature until a pressure of  $10^{-5}$  Torr was reached. The sample was flushed with ultrahigh purity Ar flowing at 50 cc/min to ambient pressure. Then, starting from room temperature, all samples were heated at a constant heating rate (HR) in, Ar, air, or  $H_2$  flowing at 50 cc/min. The change in sample weight was monitored as a function of temperature and time every 15 sec.

## RESULTS AND DISCUSSION

### The Sublimation Process in Argon

Figure 1 illustrates the drop in  $C_{60}$  weight, due to sublimation, as a function of temperature (HR = 5 C/min). The sublimation in Ar begins at 550 C and ends at 900 C; the two temperature limits are dependent on HR and sample size. At higher temperatures, 900-1000 C, there was no further drop in sample weight. The apparent remaining weight of residue was  $21.6 \pm 0.7 \%$  of the original sample. To get the true weight, a correction had to be made which takes into consideration the thermal and buoyancy effects imposed by reactor geometry, HR, geometry of sample container, and type of gas injected as well as its flow rate. When the TGA reactor is heated, the density of the flowing gas drops, and as a result, there is an apparent mass-gain which increases with temperature. By performing a blank run, with Ar flowing at 50 cc/min over empty sample container, the proper correction factors at different temperatures have been obtained. At 900 C, the correction factor is 4.6%. Therefore, the true weight of the  $C_{60}$  residue, left after the sublimation reaction, is  $21.6 - 4.6 = 17.0\%$ .

When the samples reached 1000 C in Ar, with no indication of further weight change, they were held isothermally at this limit. Air was then introduced to replace Ar at the same flow rate. The residue gasified with the liberation of only CO which is normally the main product for the carbon/ $O_2$  reaction at 1000 C. After gasification, the apparent final weight dropped from 21.6 to 4.9%, the latter value is equivalent to the new correction factor at 1000 C, 4.9%. It means that the "non- $C_{60}$ " residue is just

another stable form of carbon or soot. This trend has been confirmed with all batch A and B samples not only after the sublimation process, but also after the hydrogenation reaction (Section 3.4).

These results imply that the C<sub>60</sub> aggregates, at least the ones examined in this study, are composed of 83% volatile C<sub>60</sub> carbon contaminated with 17% of "non-C<sub>60</sub>" carbon. Since the X-ray and experimental densities are very close [17], the density of both type of carbons should be essentially the same. The 83 to 17 ratio suggests that each five molecules of C<sub>60</sub> are approximately associated with one particle of the "non-C<sub>60</sub>" carbon. If one assumes that the particle volume is, on the average, equivalent to the size of one C<sub>60</sub> molecule; 0.180 nm<sup>3</sup>, the X-ray dimensions of the particle can be estimated. Knowing that the area of one graphitic hexagon is 0.052 nm<sup>2</sup>, and assuming the particle has an average interlayer spacing of 0.35 nm, the number of hexagons in the particle can be estimated. If the particle has three graphitic layers, as a first reasonable guess, this number is given by: 0.180/(0.052 x 0.7) = 4.9; about 5 hexagons. A graphitic crystal with such a small size (0.57 x 0.7 nm) is hard to exist. Therefore, the "non-C<sub>60</sub>" carbon component in the aggregates is probably present as larger particles of soot randomly imbedded within the C<sub>60</sub> crystal.

The question is then raised regarding the residue origin. The residue could be a biproduct formed during the sublimation of C<sub>60</sub>. In other words, a polymerization reaction could be taking place in Ar at elevated temperatures to form larger stable graphitic crystals. Alternatively, the residue could be composed of soot or particulates that were originally present with the buckyballs but trapped inside the aggregates. The solvent extraction process was not able to completely remove the residue because the size of these particles is very small. During the filtration process, they may have passed through the filtering device and stayed with the C<sub>60</sub> extract solution. At present, the possibility of a polymerization reaction cannot be ruled out, however, the last explanation is probably more valid. It is noteworthy to add that the residue obtained after sublimation is insoluble in toluene.

#### Oxidation in Air

Exposure of C<sub>60</sub> to air, Figure 2, is associated with two main processes; a weight increase at lower temperatures, followed by a weight decrease at higher temperatures. While the increase in weight is attributed to oxygen chemisorption or an addition reaction, the weight loss is due to carbon gasification. Unlike the sublimation process, the oxidation reaction shows some difference between batch A and B. The reason for this discrepancy may be due to the way by which the C<sub>60</sub> molecules aggregate in each batch. It could also be dependent on the average particle size or distribution. Apparently either the internal structure and porosity of batch B is more open than A, or the average particle size of B is smaller than A. With either explanation, batch B chemisorbs (or reacts with) more oxygen than A as shown in Figure 2. In spite of this difference, the general trend with both C<sub>60</sub> samples is essentially the same. The weight increases to reach a maximum value, then the rate gradually decreases. With batches B and A, the maximum "apparent" increase in weights at 360-380 C are 5.4 and 4.8%, respectively. After considering the proper correction factor of 2.4% at 360-380 C, the corresponding true maximum weight gains of oxygen are reduced to 5.4-2.4 = 3.0% and 4.8-2.4 = 2.4%, or 30 and 24 mg O<sub>2</sub>/g C<sub>60</sub>, respectively. The average value of the two is 27 mg O<sub>2</sub>/g C<sub>60</sub> which yields 0.73 mole O<sub>2</sub>/mole C<sub>60</sub>. This calculation assumes that the molecular weight of fullerene is 720, and that oxygen molecules are only consumed by the C<sub>60</sub> component in the crystal which constitutes 83% of the total carbon.

It is interesting to note that just before the beginning of C<sub>60</sub> weight loss (gasification), each molecule acquires, on the average, 0.73 x 2 = 1.46 (say 1.5) oxygen atoms. There are several possible explanations for this. First, the "attack" with oxygen starts by opening, at least, one C=C bond to form an ether-type linkage on each C<sub>60</sub> molecule. Then, on every other molecule, another C=C bond opens up in the same way. Second, one C<sub>60</sub> molecule forms the ether-type bond while the next molecule converts a C=C to either C-O-O-C (peroxide) or two carbonyl groups. Third, some (accessible) C<sub>60</sub> molecules are attacked by more than 1.5 oxygen atoms while others (hidden inside the crystal) are not attacked at all because oxygen cannot reach them. Fourth, several complicated organic reactions between C<sub>60</sub> and O<sub>2</sub> could be taking place. Whatever the oxidation mechanism is, it is clear

that the gasification of the  $C_{60}$  crystals starts by formation of carbon-oxygen functional groups. This is exactly the mechanism of gasifying the other forms of carbon in air. It is well known that chemisorption of oxygen on carbons is the first step of the overall gasification reaction [23,24].

Let us look at the same data in a different way. The amount of oxygen uptake can be converted to estimate the active surface areas (ASA). ASA calculations assume the area occupied by one chemisorbed oxygen atom as  $0.083 \text{ nm}^2/\text{site}$ , and that each active site chemisorbs one oxygen atom [25]. Based on the two assumptions, the ASA of batches B and A, with maximum values of 30 and 24  $\text{mg O}_2/\text{g C}_{60}$ , are 93.8 and 75  $\text{m}^2/\text{g}$ , respectively. If, as just mentioned, each oxygen atom is shared by two carbon atoms, the ASA values are reduced by half to yield 46.9 and 37.5  $\text{m}^2/\text{g}$ , respectively. Compared to other forms of carbon, the  $C_{60}$  ASA values are exceptionally high. With the other carbons, typical ASA values are 0.2 - 5.3  $\text{m}^2/\text{g}$  for graphitized carbons [25,26], 2.2 - 11.3  $\text{m}^2/\text{g}$  for ungraphitized carbons [26], and 37  $\text{m}^2/\text{g}$  for the highly reactive microporous Saran char [19]. In fact, the  $C_{60}$  ASA values computed here represent a low estimate. The more realistic values should be even higher because the oxygen uptake at the maxima, shown in Figure 2, represents the net of two processes. Oxygen chemisorption which increases the weight and carbon gasification which lowers the weight. If gasification were not occurring concurrently with chemisorption at the maxima, the maximum weight gain and, consequently, the ASA would have been even higher.

Now we examine how the gasification reactions of  $C_{60}$  in air compare to other forms of carbons. With the non-isothermal oxidation procedure, which has been utilized in the current investigation, the results can be presented in different ways. The most useful ones are the oxidation rate profiles (ORP) and Arrhenius plots [27]. The ORP is a relation between the rate of weight loss (gasification rate) and sample temperature. Figure 3 compares the ORP of  $C_{60}$ , Saran (a highly reactive carbon) and SP-1 Graphite (a less reactive carbon). Both rates were based on starting sample weight. Once again, there is some difference between the ORP of batches A and B. Batch A is more reactive and starts gasifying at a slightly lower temperature (360 C) than B (380 C). The maximum rate of oxidation of batch A is higher, but occurs at a lower temperature than the maximum of B (430 vs 470 C). Nevertheless, both batches oxidize at considerably lower temperatures than Saran. In fact, it was noted that most of the  $C_{60}$  completely gasified when the highly reactive Saran char was just beginning to lose some weight. The main reason for the remarkable reactivity of  $C_{60}$  in air is its highly developed ASA. It is attributed to the early attack of oxygen on  $C_{60}$  molecules. If each  $C_{60}$  molecule is, on the average, attacked by at least one oxygen atom before the start of gasification, the buckyballs begin breaking down. This process continues at a faster rate during the course of gasification. Because of the increasingly large number of nascent carbon active [25] or reactive [28] sites generated, and the small size of carbon fragments generated during oxidation, the  $C_{60}$  gasifies faster than the rest of carbons.

#### Behavior of $C_{60}$ in Different Gases

A comparison is now made between the sublimation process in Ar and gasification of  $C_{60}$  in air and hydrogen. The results, illustrated in Figure 4, reveal several features. First, the oxidation reaction is the fastest, the sublimation process is the slowest, and the hydrogenation reaction is in the middle. Second, the oxidation reaction starts and ends at a region far below the sublimation temperature range. The  $C_{60}$  carbon oxidizes in the solid form before it sublimates. Third, the shape of the profiles in Ar and  $H_2$  are essentially the same. In air, the profiles look different, indicating that the  $C_{60}/O_2$  reaction is more complicated than the other two reactions. The profile indicates that different type of sites (or two different forms of carbon:  $C_{60}$  and soot) are gasifying at different temperature ranges. Fourth, in spite of the low temperature range at which the  $C_{60}$  gasifies, the maximum rates of oxidation are 40-50% higher than the hydrogenation or sublimation rates measured at higher temperatures. Fifth, the similarity between the profiles of the  $C_{60}/H_2$  reaction and  $C_{60}/Ar$  sublimation process is somewhat puzzling. If the  $C_{60}/H_2$  reaction was a typical heterogeneous reaction, the profile shown in Figure 4 should have acquired, to some extent, the shape of the  $C_{60}/O_2$  profiles. That is, the profile in  $H_2$  should not have been symmetrical. Since this is not the case, other possible reasons have to be considered, and this subject is addressed next.

### Reduction/Gasification of C<sub>60</sub> in Hydrogen

Figure 5 compares the Arrhenius plots of the C<sub>60</sub> sublimation and hydrogenation reactions. The plots were computed from the corresponding ascending branches of Figure 4. The Arrhenius plots are practically linear with activation energies of 39-42 Kcal/mole for the sublimation process, and 34 (batch A) or 42 (batch B) Kcal/mole for the hydrogenation reaction. Once again, batch A is slightly more reactive to H<sub>2</sub> than B. Thus, the activation energy in Ar and H<sub>2</sub> is practically the same, suggesting that there is some similarity between the nature of the two processes. On the other hand, the activation energy for the C<sub>60</sub>/H<sub>2</sub> reaction is in line with those values reported in the literature for other carbon/hydrogen reactions;  $36 \pm 6$  Kcal/mole at atmospheric pressure [29], and 35.8 - 50.9 Kcal/mole at 27 atm [30]. So far, the C<sub>60</sub> and the other "old" carbons appear to take the same activation energy path when exposed to H<sub>2</sub>. However, the hydrogenation reaction of C<sub>60</sub> may be different than the normal gasification reactions of other carbons which yield CH<sub>4</sub>.

The C<sub>60</sub> hydrogenation reaction takes place at a considerably lower temperature range than the rest of "older" carbons. Figure 6 illustrates this point. Under similar experimental conditions, the extent of gasifying the highly reactive Saran, and the less reactive SP-1 graphite, is small. The change in apparent sample weights between 500 and 1000 C is also small; less than 5% (no correction factor is included here). By contrast, the reaction between C<sub>60</sub> and hydrogen starts at 500 C and ends at 900 C. Once again, the carbon leaves behind the "non-C<sub>60</sub>" residue (soot) which has low reactivity in H<sub>2</sub> at 1000 C, resembling Saran and SP-1 graphite.

Although the hydrogenation reaction of C<sub>60</sub> has an activation energy similar to the "old" carbon/H<sub>2</sub> reaction, one may conclude that fullerene is just gasifying in H<sub>2</sub> like a typical carbonaceous material. This may be true to some extent. Yet, at the present time, we cannot rule out other possible explanations until further conclusive experiments are performed. That is, the observed hydrogenation reaction may be taking place through an entirely different route than the "normal" C/H<sub>2</sub> gasification reaction. It is possible that H<sub>2</sub> molecules are opening the C=C bonds and saturating the surface with C-H bond, yielding C<sub>60</sub>H<sub>36</sub> and other smaller hydrocarbons. In other words, the reaction could merely be starting with hydrogen addition rather than carbon gasification. The resulting products, with different amounts of hydrogen, could simply be a wide variety of different organic molecules that have different sizes, properties, and boiling points. If this is the major reaction, then the TGA experiments were merely measuring the weight loss due to evaporation of organic compounds with minor contribution of gasification of the carbonaceous matrix.

### CONCLUSIONS

1. The C<sub>60</sub> carbon crystals examined here sublime in inert atmospheres, leaving behind a non-volatile carbon residue weighing 17% of the original sample. The residue, possibly soot is another form of carbon which gasifies in air yielding CO at 1000 C and is insoluble in toluene.
2. Oxidation of C<sub>60</sub> in air starts by a weight gain process during which, each C<sub>60</sub> molecule acquires, on the average, 1.5 oxygen atoms. At this point, a portion of the C<sub>60</sub> molecules begin opening up. With increasing temperature, the buckyballs further oxidize with a concurrent gasification reaction of the carbonaceous matrix.
3. Under otherwise similar conditions, C<sub>60</sub> is considerably more reactive in air or hydrogen than the other forms of carbon. This is attributed to the fast rate of generating active sites when the C<sub>60</sub> molecules start breaking.
4. The hydrogenation reaction of C<sub>60</sub> may, or may not, resemble the "old" carbon/H<sub>2</sub> gasification reaction to yield CH<sub>4</sub>. The current data indicate some similarity between the two carbons. However, at the present time, the possibility of other hydrogenation reactions, that may convert fullerene to other forms of hydrocarbons, cannot be ruled out.

## FUTURE APPLICATIONS

There are several potential applications for this new form of carbon, especially in the areas of carbon composites and molecular sieve carbons. Carbon composites have been used extensively in several aerospace applications. When stored, especially in an environment with high humidity, the composite adsorbs (absorbs?) moisture, some of the mechanical properties may deteriorate, and the component weight increases. To avoid this, a layer (blanket) or two of  $C_{60}$  could be spread to cover the entire external surface and pore entrances of the component. The assumption is made here that  $C_{60}$  has a hydrophobic surface that does not adsorb moisture. This assumption is reasonable because the  $C_{60}$  molecule does not have functional groups which normally enhance the moisture adsorption on carbons. To "spread the blanket" on a composite, the process has to be performed in an oxygen-free atmosphere, perhaps under vacuum. While a  $C_{60}$  reservoir is heated, the composite surface is cooled. Alternatively, a very dilute solution of  $C_{60}$  in benzene or toluene can be sprayed on the substrate. The solvent is then evaporated leaving behind the  $C_{60}$  blanket. This process does not require large quantities of fullerene. Based on the 1.0 nm diameter of  $C_{60}$  molecules, the computed surface area is  $675 \text{ m}^2/\text{g}$ . This means that 6.2 g of  $C_{60}$  can make a "blanket" for a substrate whose area is equivalent to an acre!

The second potential application is the preparation of molecular sieve carbons, which are highly microporous carbons with large adsorption capacity. Yet the size of their pore entrances, the "gate", has to be tailored to the specific application. The  $C_{60}$  comes as good and bad news for preparing this class of carbons. The starting substrate for molecular sieve carbons is normally a microporous (activated) carbon with a wide micropore size distribution. If one layer of  $C_{60}$  is deposited on the micropore entrance, the "gate" is narrowed by approximately 1.0 nm. Deposition of additional layers will narrow the "gate" further by multiples of nanometers (2,3,4,5,...nm). If the average pore size is 1.3 or 2.3 nm, then deposition of one or two layers of  $C_{60}$  will reduce the "gate" (of both pores) to 0.3 nm. The product is an excellent sieve to separate argon from oxygen whose minimum dimensions are 0.38 and 0.28 nm, respectively [31]. The sieve will preferentially adsorb oxygen, leaving argon passing through the separation tower. This is the good news. Now, if the starting average pore size is 1.0 or 2.0 nm, the deposition of  $C_{60}$  will just close the pores; and with it, come the bad news.

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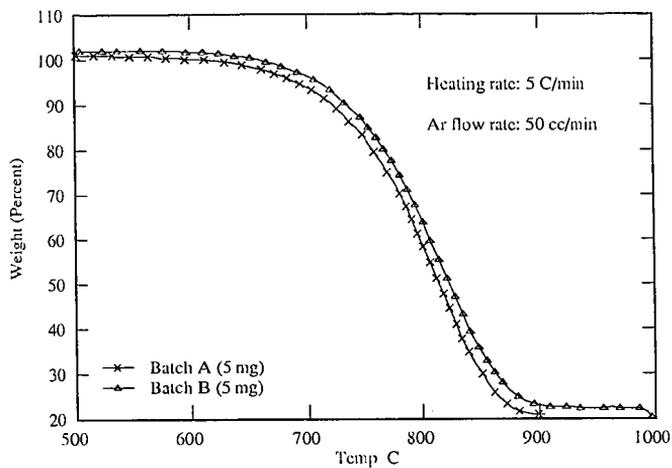


Figure 1: Sublimation of Fullerene (C<sub>60</sub>) in Argon.

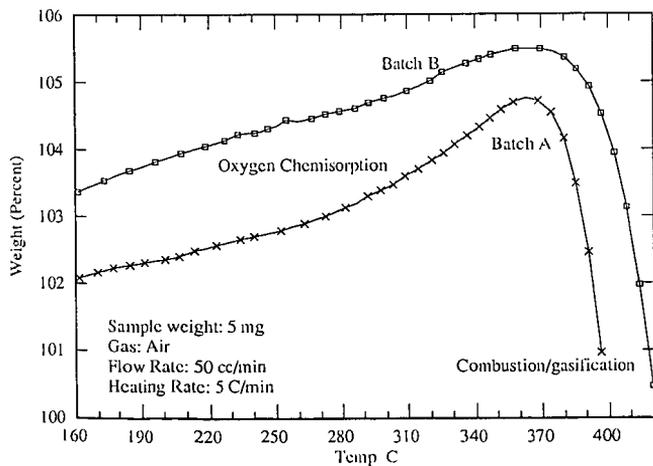


Figure 2: Reaction between Fullerene and Air at Low Temperatures.

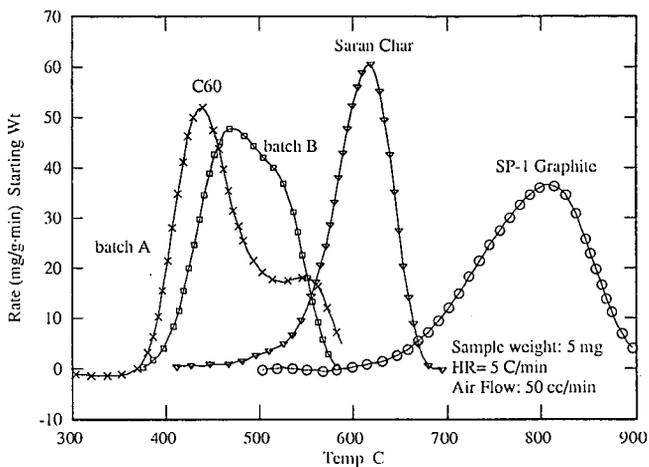


Figure 3: Comparison between Oxidation Rates of C60/Saran/SP-1 in air.

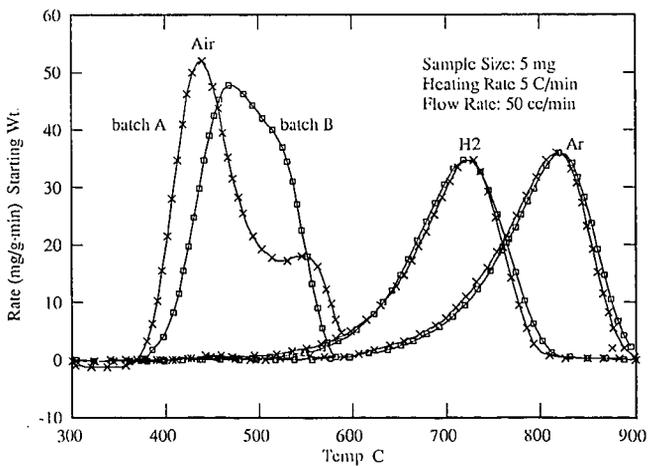


Figure 4: Reactions of Fullerene in Argon - Air and Hydrogen.

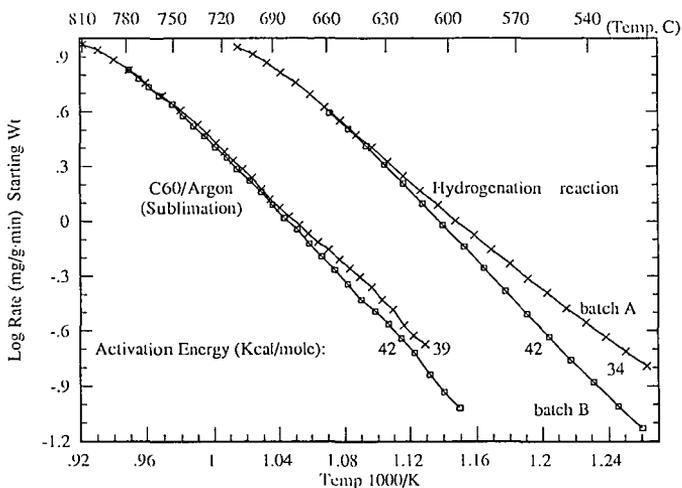


Figure 5: Arrhenius Plots for C60/Argon and C60/Hydrogen Reactions.

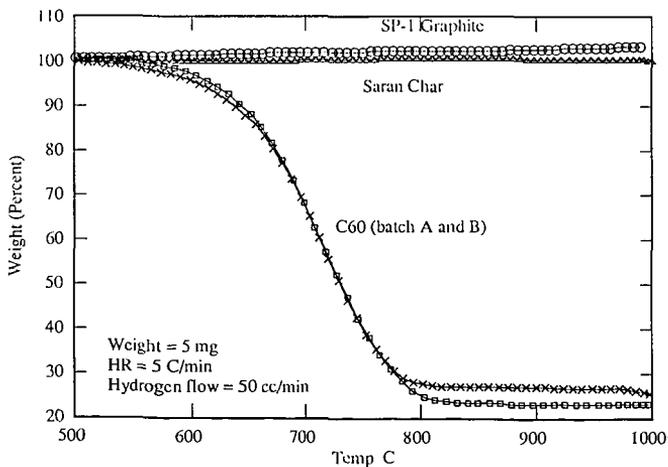


Figure 6: Comparison between C60 and other Forms of Carbon in Hydrogen.