Materials Physics and Mechanics 39 (2018) 56-60 Received: November 1, 2017

C60 COPPER FULLERITE: SYNTHESIS AND PROPERTIES

Victor Gerasimov, Ruslan Zarafutdinov*, Olga Proskurina

Department of Mechanics and Control Processes

Peter the Great St. Petersburg Polytechnic University, Polytekhnicheskaya 29

195251, St. Petersburg, Russian Federation

*e-mail: zarafutdinovruslan@mail.ru

Abstract. Chemical method for copper fullerite synthesis is presented. In order to complete reaction, chemical reagents fullerenol $C_{60}OH_{42}$ and copper sulphate II $CuSO_4$ were used. The data obtained allow conclude that chemical method for metal fullerites synthesis is perspective for further study.

Keywords: chemical synthesis, copper, fullerene, fullerite

1. Introduction

One of the most demanded areas of new materials is electrical engineering and electronics [1]. Composite materials based on copper and carbon nanoparticles are promising as structural and functional materials [2, 3]. Today the actual task is the synthesis of new metal-carbon nanocomposites with improved properties.

The main objective of this work is synthesis of bivalent copper fullerite from the water soluble form of fullerene – fullerenol $C_{60}OH_{42}$ and copper sulphate II - $CuSO_4$.

2. Reaction description

A chemical method of copper fullerite synthesis was used in this work. It consists in the possibility of two OH groups replacing by a copper atom, or by two hydrogen atoms replacing with a copper atom. During the reaction a gas is released and a black powder substance insoluble both in water and in a solvent is formed. To accomplish the reaction, fullerenol obtained by peroxide synthesis [4] was used. In order to produce fullerenol, the isomer of fullerene C_{60} with 99.98% purity was employed [5, 6]. Five-water chemically clean copper sulphate II took part in the reaction too.

For the reaction visualization, the Avogadro program was employed [7]. The process is illustrated in Fig. 1.

The reaction consists in merging two solutions: copper sulfate II in distilled water and fullerenol in distilled water. Almost immediately the granularity of the mixture appears, flakes of the reacted material are formed, the solution color is clarified. The formation of a precipitate indicates a chemical reaction. After about 30 minutes the reaction is complete. Depending on the ratio of salt and fullerenol taken, the color of unreacted solution varies from light brown (excess fullerenol) to green and blue (excess salt). The examples are shown in Fig. 2. After the reaction is finished, the solution is decanted. The precipitate is dehydrated in vacuum, weighed and pressed into solid samples.

http://dx.doi.org/10.18720/MPM.3912018_9

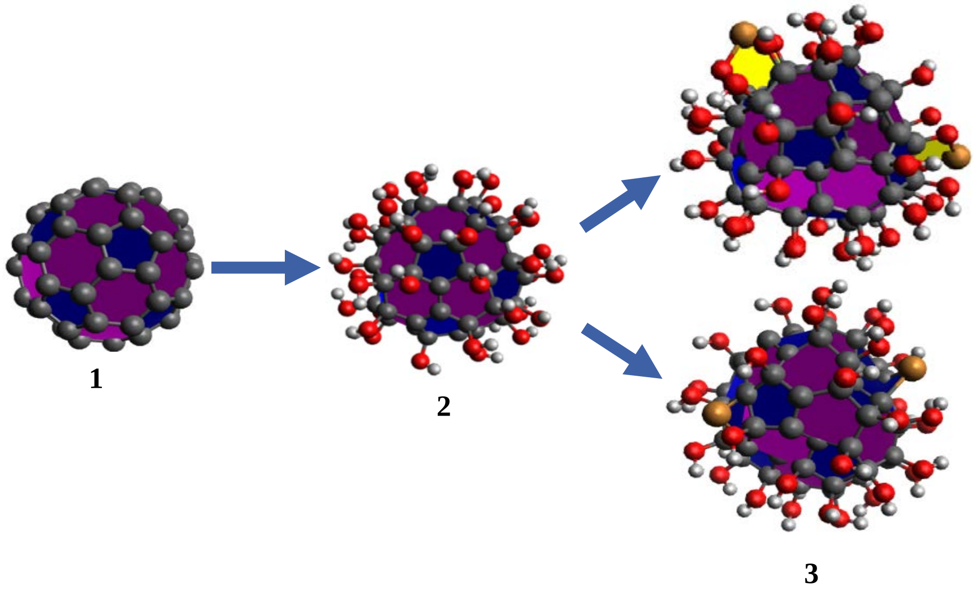
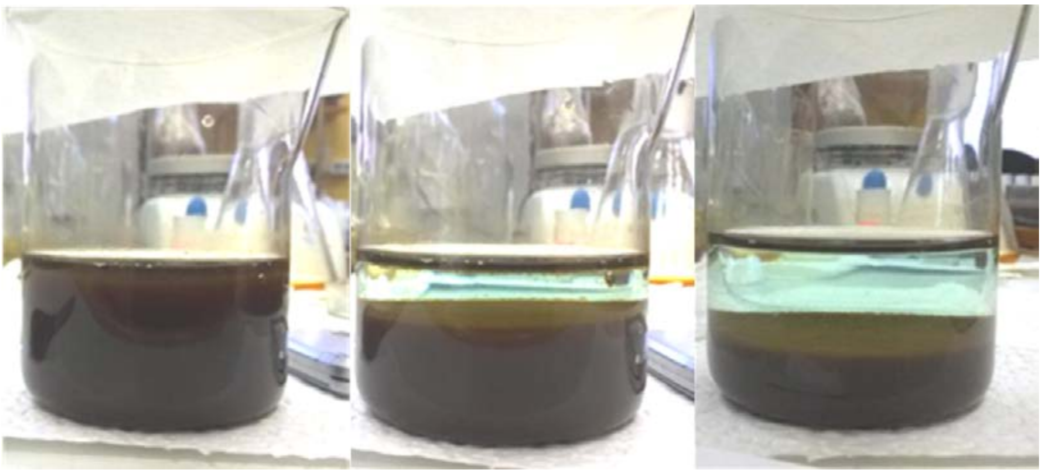


Fig. 1. Stages of copper fullerite synthesis: 1) fullerene synthesis, 2) fullerenol synthesis, 3) synthesis of copper fullerite



a) *b)* *c)*

Fig. 2. Process of precipitation: *a)* immediately after mixing; *b)* in 1.5 hours; *c)* in 5 hours from mixing

3. Correct reagent ratio

To optimize the reaction process and minimize the amount of solution decantation, a study of optimum reagent ratio was conducted. The method of successive approximations was used. Initially, the reaction is carried out with an excess of copper salt. After the main reaction the liquid part of the solution is taken away. Then fullerenol is added to it until the reaction and precipitation stops. After that the precipitate is placed on Petri dish for liquid evaporation. In the end, the collected precipitate is weighed.

As a result, the following ratio was found: 589.9 mg fullerenol and 500.5 mg $CuSO_4{*}5H_2O$ have reacted completely. Taking into account the error of 0.5 mg, we can assume that the correct ratio of copper salt and fullerenol solution is 5/5.9.

4. Sample sintering

Sintering was carried out by two methods: cold pressing in the pressure range from 100 MPa to 800 MPa, and by vacuum hot pressing method at a temperature from 900°C to 2100°C at a constant pressure of 800 MPa. The solid samples are shown in Fig. 3.

Fig. 3. Solid samples obtained: *a)* by cold pressing, *b)* by hot vacuum pressing

5. Properties of the obtained material

After drying the precipitate is looks like black fragile flakes. When grinding, a homogeneous black powder insoluble in water and organic solvents was obtained. Solid samples made by cold pressing are extremely fragile, so they have not been tested. The microhardness of the hot vacuum pressed sample, determined by the Vickers method, is 236 kgf/mm^2. The resonance capacitance measurement gave no evidence of a resonance peak on an oscilloscope picture, so it was concluded that the sample is a conductor.

The sample resistance dependence on a temperature in the range from -15 to +140°C is shown in Fig. 4. The resistance increases with the temperature growth tending to saturation. The resistance decrease with a diminishing temperature makes this new material promising for high-temperature superconducting studying.

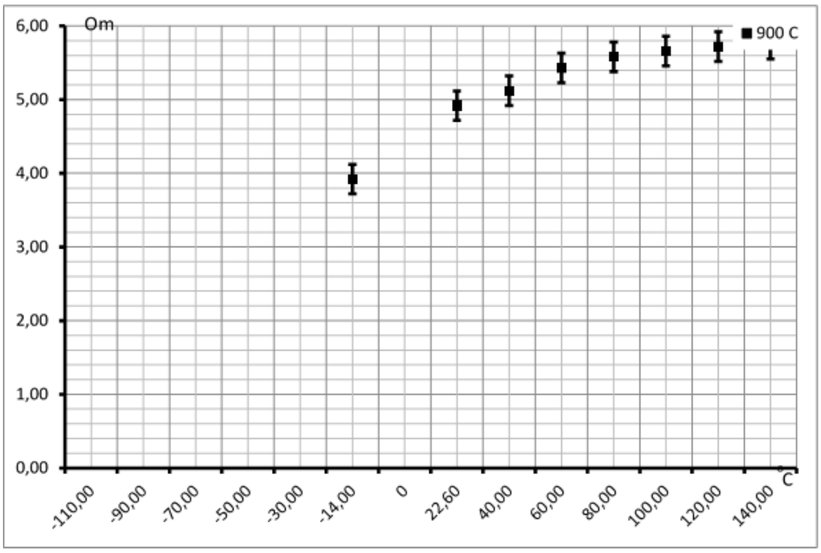


Fig. 4. Temperature dependence of sample resistance

The calorimetric method for measuring specific thermal conductivity was implemented in this work. For this purpose, a differential bicalometer was used. The coefficient of sample thermal conductivity determined is 14 W/m·K, while the thermal conductivity of fullerene and fullerite does not exceed 1-2 W/m·K.

The material obtained was also subjected to chemical analysis. The results obtained with the Oxford instruments installation are presented in Table 1. The average number of atoms in the final product converted to one fullerene molecule (60 carbon atoms) is: carbon - 60.12, oxygen - 37.82, copper - 2.13. The presence of other components is a consequence of contamination of the sample or reagents.

Table 1. Chemical composition

Element	Conditional concentration	Weight percent	Mean square deviation %	Reference
C		49,9		
O	21,85	37,8	0,14	SiO_2
Al	0,09	0,24	0,01	Al_2O_3
Si	0,51	0,79	0,02	SiO_2
Ca	0,57	0,19	0,01	Wollastonite
Ni	0,61	0,18	0,02	Ni
Cu	6,92	10,8	0,07	Cu
Sum		100		

6. Conclusion

A chemical method of copper fullerite synthesis is considered. It is found that during the interaction of fullerenol $C_{60}OH_{42}$ with cupric sulfate a chemical reaction with precipitate formation and gas evolution occurs. The optimal conditions for the reaction are determined, as well as the methods of producing technologically valuable powder materials obtained during the reaction. Chemical analysis showed that final product consists of two copper atoms per molecule of fullerene. Solid samples of the new material are investigated and their mechanical and electrical properties are found. The investigations show the prospects of chemical methods for the synthesis of metal fullerites.

References

[1] A. Polishchuk, Semiconductor devices based on silicon carbide // *Components and Technologies* **8** (2004) 67.

[2] D.V. Konarev, I.S. Neretin, Y.L. Slovokhotov, E.I. Yudanova, N.V. Drichko, Y.M. Shulga, B.P. Tarasov, L.L. Gumanov, A.S. Batsanov, J.A.K. Howard, R.N. Lyubovskaya, New molecular complexes of fullerenes C60 and C70 with tetraphenylporphyrins [M(tpp)], in which M=H2, Mn, Co, Cu, Zn, and FeCl // *Chemistry – A European Journal* **7** (2001) 2605.

[3] E.S. Vasilyeva, S.V. Kidalov, V.V. Sokolov, G.G. Klimov, Ji Puguang, Properties of copper-detonation nanodiamond composites obtained by spraying solutions // *Applied Physics Letters* **39** (2013) 81.

[4] A. Hashim, *The Delivery of Nanoparticles* (InTech, Japan, 2012), p.142.

[5] V.I. Gerasimov, A. Trofimov, O.V. Proskurina, Isomers of fullerene C60 // *Materials Physics and Mechanics* **20(1)** (2014) 25.

[6] V.I. Gerasimov, M.Y. Matuzenko, O.V. Proskurina, Purity analysis of trade produced C60 fullerene // *Materials Physics and Mechanics* **13(2)** (2012) 181.

[7] Marcus D. Hanwell, Donald E. Curtis, David C. Lonie, Tim Vandermeersch, Eva Zurek, Geoffrey R. Hutchison, Avogadro: an advanced semantic chemical editor, visualization, and analysis platform // *Journal of Cheminformatics* **4** (2012) 17.