PREPARATION OF HOLLOW NANOPARTICLES

Schur D.V.*, <u>Dubovoy A.G.</u>, Kaverina S.N., Shul'ga Yu.M.¹, Tarasov B.P.¹, Zaginaichenko S.Yu., Shaposhnikova T.I.

Institute for Problems of Materials Science of NAS of Ukraine, lab. # 67, 3, Krzhizhanovsky str., Kiev, 03142 Ukraine

(1) Institute of problem of chemical physics RAS, Chernogolovka, 142432, Russia

Introduction

Investigations into electric sparc metal erosion, that have been performing for the last two decades, allow us to gain sufficient information on mechanism of the ultradispersed particle formation, effect of arc parameters and physical properties of medium on the parameters and properties of the particles prepared [1-2].

Moreover, the work directed to the investigation into the mechanism of the dispersed phase formation suggests that monolithic solid particles will not form in all the cases [3]. Experiments demonstrated that in some experimental conditions the hollow particles formed. They can have various geometric forms. In the work presented the formation of hollow ultradispersed particles is considered. The particles have been prepared by nickel sputtering in the hexane medium.

Experimental

As showed in [3], the main techniques for hollow particle preparation may be:

- a) dispersion of the substance solution using ultrasound or frothing the operation solution before pyrolytic treatment;
- b) interaction between liquid particles (solutions or melts) and gas or vapor in aerosols;
- c) emulsion method;
- d) precipitation of different compounds on the surface of polymer particles followed by treatment with oxygen and hydrogen;
- e) other techniques representing modifications of one or another method or their combination.

Various materials may be produced by these techniques [4-23].

Basically, macro- and microparticles are produced by all the techniques above.

In our previous work we prepared microand nanodispersed particles by two techniques: 1) final reduction in H₂ of the mechanical mixture from highly dispersed powders of required metal oxalates and the base followed by removing the base material by chemical method; 2) cladding the highly dispersed base powders with required metals followed by removing the base material by different methods. In this report we represent the results of the work aimed to prepare the hollow nanoparticles of metal carbides.

The apparatus for electric sparc material dispersion in liquid media was used for this purpose. The apparatus was designed in laboratory 67 in Institute for Problems of Material Science of National Academy of Sciences of Ukraine.

The apparatus allows preparation of particles $10 \text{ nm} - 10 \text{ }\mu\text{m}$ in size. There is possibility to study the effect of temperature, the nature of liquid phase, plasma parameters on properties, geometric parameters and structures of the products produced.

In the experiment nickel was used as the material sputtered. It has been sputtered in C_2H_5OH , toluene, benzene and hexane. The products produced have been studied using transmission electron microscopy (Fig.1-9).

Results and discussion

In the course of the experiment the formation of hollow particles has been observed only in the hexane medium. Moreover, the formation of nanoparticles has been observed in all the media. Nanoparticles are gathered it the fractal clusters which consist of larger or fewer number of initial units (Fig.2). Nanoparticles may present as separated aggregates or they adhere to the large metal particles (Fig.2). Particles may be both hollow with crystals on the surface (Fig.3) and completely monolithic (Fig.4).

Additionally, the particles with the almost regular form (Fig.5) and the noticeably distorted spherical form have been prepared in hexane. It is rather difficult to describe the shape of some hollow particles produced (Fig.7). Among the particles prepared there were particles with faces typical for monocrystals (Fig.8,9) and the particles which have the shape like an acrumpled paper sheet (Fig.1).

We suppose that the hollow particle formation occurs in two stages:

a) the refractory carbide, oxycarbide or carbon (graphite or hydrocarbon) layer forms on the surface of the metal particles, when it is cooled. Quenching the layer occurs in the operation liquid (Fig.5).

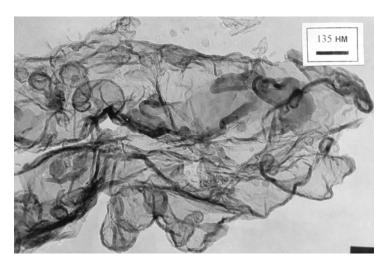
b) The particle returns from the liquid medium (due to the turbulent movement of liquid) into the plasma zone. Heating occurs above the metal melting point. As a result, vapor forms inside the refractory shell. The shell tears, vapor is ejected. This process results in the formation of both metal nanoparticles and hollow structures which, obviously, will be of great practical interest as filters, sorbents and catalysts.

References

- Schur DV, Dubovoi AG, Anikina NS, Zaginaichenko S Yu, Dobrovol'skij VD, Pishuk VK, Tarasov BP, Shul'ga Yu M, Meleshevich KA, Pomytkin AP; The production of utrafine powders of fullerites by the salting out method, Proceedings of VII International Conference "Hydrogen Material Science and Chemistry of Metal Hydrides", Alushta-Cremia-Ukraine, September, 16-22, 2001,
- Kharlamov AI, Loytchenko SV, Kirillova NV, Kaverina SN, Vasilev AD, Fomenko VV, Zolotarenko AD, Kazimirov VP; Tubular and filamentous nanostructures of hexagonal silicon carbide, Proceedings of VII International Conference "Hydrogen Material Science and Chemistry of Metal Hydrides", Ukraine, 572-574,2001,
- Slys IG, Berezanskaya VI, Schur DV, Zaginaychenko SYu, Rogozinskaya AA, Adejev VM, Zolotarenko AD; Making the point metal coatings on the particles of hydride-forming intermetallides, Proceedings of VII International Conference "Hydrogen Material Science and Chemistry of Metal Hydrides", Ukraine,404-405,2001.
- Matysina ZA, Schur DV; Hydrogen and solid phase transformations in metals, alloys and fullerites, Dnepropetrovsk: Nauka i obrazovanie, 420p (in Russian),2002,
- 5. Matysina ZA, Pogorelova OS, Zaginaichenko S Yu, Schur DV; The surface energy of crystalline CuZn and FeAl alloys, Journal of Physics and Chemistry of Solids, 56, 1,9-14, 1995, Elsevier
- Schur DV, Lavrenko VA, Adejev VM, Kirjakova IE; Studies of the hydride formation mechanism in metals, International journal of hydrogen energy, 19,3,265-268,1994, Elsevier
- 7. Schur DV, Matysina ZA, Zaginaichenko S Yu; Theoretical study of interstitial atoms distribution in the bulk and at the surface of crystal. Surface segregation, Journal of alloys and compounds, 330,81-84,2002, Elsevier
- 8. Shul ga Yu M, Martynenko VM, Tarasov BP, Fokin VN, Rubtsov VI, Shul ga N Yu, Krasochka GA, Chapysheva NV, Shevchenko VV, Schur DV; On the thermal decomposition of the C60D19 deuterium fullerite, Physics of the Solid State, 44, 3,545-547, 2002, Nauka/Interperiodica
- 9. Schur DV, Matysina ZA, Zaginaichenko S Yu; Study of physico-chemical processes on catalyst in

- the course of synthesis of carbon nanomaterials, Hydrogen Materials Science and Chemistry of Metal Hydrides: Proceedings of the NATO Advanced Research Workshop on. Alushta Crimea, Ukraine, 16-22 September 2001,235,2002, Kluwer Academic Pub
- Tarasov BP, Shul'ga Yu M, Fokin VN, Vasilets VN, Shul'ga N Yu, Schur DV, Yartys VA; Deuterofullerene C 60 D 24 studied by XRD, IR and XPS, Journal of alloys and compounds, 314,1,296-300,2001, Elsevier
- 11. Tarasov BP, Fokin VN, Moravsky AP, Shul'ga Yu M, Yartys VA, Schur DV; Promotion of fullerene hydride synthesis by intermetallic compounds, Hydrogen energy progress, 2, 1221-1230,1998,
- 12. Schur DV, Zaginaichenko S Yu, Matysina ZA, Smityukh I, Pishuk VK; Hydrogen in lanthannickel storage alloys, Journal of alloys and compounds, 330,70-75,2002. Elsevier
- Schur DV, Tarasov BP, Shul'ga Yu M, Zaginaichenko S Yu, Matysina ZA; Research of Fullerites Hydrogen Capacity, Hydrogen Materials Science and Chemistry of Metal Hydrides: Proceedings of the NATO Advanced Research Workshop on. Alushta Crimea, Ukraine, 16-22 September 2001, 1,2002, Kluwer Academic Pub
- 14. Matysina ZA, Zaginaichenko S Yu, Schur DV, Pishuk VK; Theoretical investigation of isopleths of hydrogen solubility in transition metals, Journal of alloys and compounds, 330,85-88,2002, Elsevier
- Trefilov VI, Schur DV, Pishuk VK, Zaginaichenko S Yu, Choba AV, Nagornaya NR; The solar furnaces for scientific and technological investigation, Renewable energy, 16,1,757-760, 1999, Elsevier
- 16. Трефилов ВИ, Щур ДВ, Загинайченко СЮ; Фуллерены-основа материалов будущего, 2001, Laboratory 67
- 17. Schur Dmitry V, Zaginaichenko Svetlana Yu, Veziroğlu T Nejat, Javadov NF; The Peculiarities of Hydrogenation of Fullerene Molecules C60 and Their Transformation, Black Sea Energy Resource Development and Hydrogen Energy Problems,191-204,2013, Springer Netherlands
- 18. Muratov VB, Meleshevich KA, Bolgar AS, Zolotarenko AD; Application of dynamic ccalorimetry method for investigation of enthalpy at hydride dissociation, Proceedings of VII International Conference "Hydrogen Material Science and Chemistry of Metal Hydrides", Ukraine, 342-343, 2001,
- Anikina NS, Schur DV, Simanovskiy AP, Zolotarenko AD, Dubovoy AG, Ivanchenko NV; Problem on fullerene production by electric arc method, Proceedings of VII International Conference "Hydrogen Material Science and Chemistry of Metal Hydrides", Ukraine,590-591, 2001,
- Pishuk VK, Schur DV, Bogolepov VA, Savenko AF, Zaginaichenko SYu, Zolotarenko AD, Mar'yanchuk IV, Prikhod'ko AB; Problem on production of highly dispersed extra pure powders,

- Proceedings of VII International Conference "Hydrogen Material Science and Chemistry of Metal Hydrides", Ukraine, 586-587, 2001,
- Lavriv LV, Anikina NS, Simanovskij AP, Zolotarenko AD, Schur DV; Features of fullerene extraction wth toluene, Proceedings of VII International Conference "Hydrogen Material Science and Chemistry of Metal Hydrides", Ukraine,596,2001
- 22. Schur DV, Zaginaichenko S Yu, Adejev VM, Voitovich VB, Lyashenko AA, Trefilov VI; Phase
- transformations in titanium hydrides, International journal of hydrogen energy, 21,11,1121-1124,1996, Pergamon
- 23. Schur DV, Tarasov BP, Zaginaichenko S Yu, Pishuk VK, Veziroglu TN, Shul'ga Yu M, Dubovoi AG, Anikina NS, Pomytkin AP, Zolotarenko AD; The prospects for using of carbon nanomaterials as hydrogen storage systems, International journal of hydrogen energy, 27, 10, 1063-1069, 2002, Pergamon



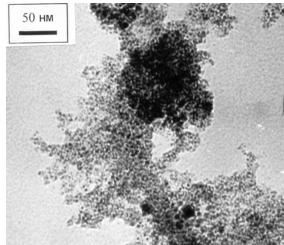
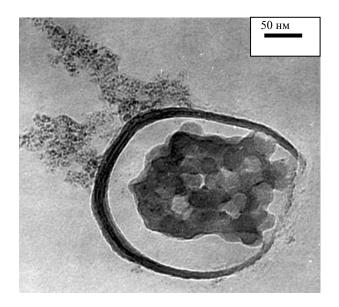


Fig. 1 Fig. 2



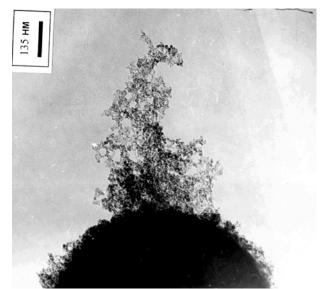


Fig. 3 Fig. 4

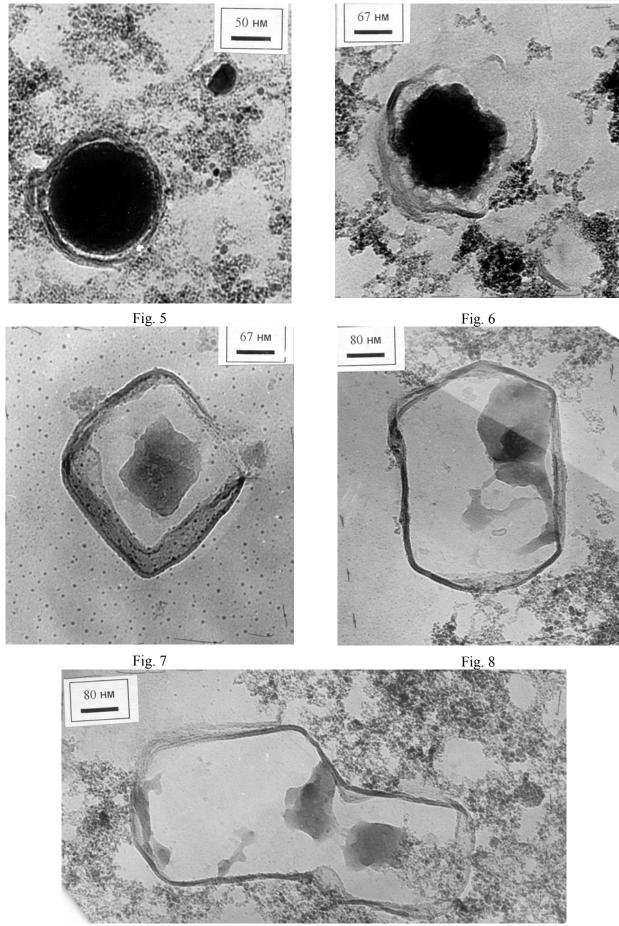


Fig. 9