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Abstract: A HeHPC (helium high pressure chamber) filled up with pure gaseous helium at initial pressure about 1.1 kbar was irradiated by braking γ -rays of 10 MeV threshold energy during 1.0×10^5 s at the electron beam current (22-24) μ A. After irradiation, the residual pressure inside was equal to 430 bar. Synthesized foils of black color and other multiple objects were found inside the HeHPC mainly at the entrance window for γ -rays made from beryllium bronze as a plug of beryllium bronze HPC, (published earlier) at the inner surfaces of the reaction chamber made of high purity copper and at the copper collector. Firstly, the element analysis, using SEM (scanning electron microscopy) and MPRA (microprobe roentgen analysis), allowed us to establish that the foils consist predominantly of carbon and oxygen and smaller quantities of other elements up to iron. Two years later some physical properties such as (low) density, (high) resistivity, magnetic (high paramagnetic) and dielectric (medium relative dielectric constant) properties were determined. A new carbon reach structure was also postulated basing on obtained diffractometer data. The second used method (see text) for element content determination, in principle, has confirmed the previous one.

Key words: Irradiation, high pressure, helium, PACS: 25.20.Dc, 25.45.De.

1. Introduction

The element compositions of synthesized particles and objects, as well as the surface structure of elements of the DHPC (deuterium high pressure chamber), have been studied using samples of metals such as Al, V, Cu, Pd, Sn, Re, YMn₂ alloy and stainless steel in the shape of rods and wires which were placed in molecular deuterium gas under high pressure and acted on by braking γ -rays of 10 MeV [1-7] and 23 MeV [8-12] energy. Analogous investigations aimed to study the possibilities of nuclear reactions were performed using HHPC (hydrogen high pressure chambers) with Pd-rods inside [13] and in the presence of hydrogen without any metallic samples in the chamber [14, 16] under irradiation by 10MeV braking γ -rays. Possible phenomenological modeling approaches for nuclear fission reactions in the liquid-drop model and for nuclear fusion are discussed in Refs. [18-24].

The goal of this paper is to present some interesting physical properties and possible crystallographic structure of the chosen graphite-like elements which were found in the PC fulfilled before gamma irradiation only by pure gaseous helium under pressure (1-3) kbar observed earlier by authors and first described in [16, 17]. The data presented here, concerning physical properties of graphite-like

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elements, have a preliminary character.

2. Description of the Technique

Fig. 1 shows the scheme of a modified high pressure chamber filled with helium (HeHPC) whose pressure was equal to 1,092 bar at the start of the irradiation with γ -rays. Helium of specific spectral purification had 99.999 at .% purity specified in its data sheet. The filling of the HeHPC chamber with helium was carried out by iterations of the following operation: filling of the chamber of volume with helium up to a pressure of 0.2 kbar, releasing the pressure up to atmospheric one, repeating this procedure and then filling the chamber with helium up to the 2 kbar pressure. The degree of purification from atmospheric gases was approximately of some ppm.

The initial pressure in the HeHPC, measured prior to irradiation using a strain sensor (see Fig. 1, pos. 11) and Model P3 measuring module, was found to be 1,092 bar. At helium pressure of about 1.1 kbar, its atomic density is evaluated as being approximately 1.5×10^{22} at.He/cm³ [27].

Irradiation of the HeHPC was performed during 27 hours and 51 minutes (or 1.02×10^5 s) using the

MT-25 electron accelerator of the G. N. Flerov Laboratory of Nuclear Reactions of JINR, Dubna, Rus. The energy of the electron beam was 10 MeV, and the electron beam current ranged from 21 to 23 µA. The braking γ -rays of continuous spectrums, with threshold energy of about 10 MeV, were obtained using a braking target in the shape of a tungsten foil of 2.5 mm thickness and an aluminum absorber of electrons with 25 mm thickness. During the irradiation, the temperature inside the outer protective steel cylinder of HeHPC increased to about 60 °C at the steady-state regime of the chamber's irradiation. The pressure in the chamber increased at the start of irradiation from 1,092 bar to 1,242 bar, i.e. by 150 bar, which seems to be a pure temperature effect. Before opening of the chamber it was the most objective and precise measurement of its inside pressure which was registered to be 426.0 ± 2.0 bar. Thus, it had dropped down by 666 bar. Upon opening the chamber, a few oily (see explanation below) black foils of round shape were observed in its inside area (see Fig. 1, pos. 5).

These black foils of considerable size (see Fig. 2 below) were composed mainly of carbon. As the foils were laid out on a special clean sheet of roll paper



Fig. 1 The schematic drawing of high pressure apparatus. 1—gamma quanta, 2—closing screw (CuBe2), 3—reinforcing SS high pressure chamber body, 4—CuBe2 "window—plug" (58°/60° cones), 5—places where mainly C elements were observed, 6—CuBe2 high pressure chamber, 7—helium under high pressure, 8—pure Cu sleeve, 9—expected reaction product on pure Cu plug, 10—temperature measurement channels, 11—high pressure connecting capillary to high pressure valve, strain gauge pressure sensor and inlet-outlet system of gaseous being investigated (not shown in the picture).



Fig. 2 Images of group of black foils observed at position 5, shown in Fig. 1.

(tracing one), it appeared that the latter (its surface) looked to be soaked with oil in the area of such foils.

After opening the HeHPC chamber, foils of black color with reinforcing needles were observed at the juncture of the entrance window and reaction chamber (see Fig. 1, pos. 5), part of such needles protruding from the foils as lengthy rods of constant thickness and white color. Fig. 2 shows images of multiple black foils of round form that acquired such shape due to cylindrical symmetry of the entrance to the reaction chamber. These images were obtained using a special photo-micrographic device.

For density measurements the analytical microgram weight MYA 53Y Radwag, located in Microanalysis Laboratory of WUT and typical instrumentation of length and thickness measurements were used. Resistivity was measured using typical Mega ohm meter, relative dielectric constant using universal capacitance meter, and for diffraction investigations a higher standard instrumentation was used.

3. SEM and MPRA Studies of Black Thin Foils Synthesized after Irradiation γ-rays

The structure and element composition of all the elements of the HeHPC chamber which were in

contact with dense helium, such as the cut along the cylinder symmetry axis a) two inner and outer surfaces of the beryllium bronze entrance window, b) two inside surfaces of the copper sleeve in which nuclear and chemical reactions predominantly took place, c) copper collector of reaction product were studied at two independent analytical centers (Skobielcyn Laboratory of Moscow Łomonosov State University, Russian Federation and FGBNU NII PMT, Moscow, Russian Federation).

As was noted in the previous section, upon opening the HeHPC, oily foils of black colour with reinforcing rods of small diameter and uniform thickness, either protruding or scattered over the surfaces of the black foils, i.e. inside them, were found at the juncture of the entrance window and reaction chamber (see Fig. 1, pos. 5).

Here, results of the second Laboratory (where full elements analyses were done), will be taken under consideration. The set of foils being considered here, found after opening of the HeHPC is shown in Fig. 2.

Similar to the results described by the first center, the patches of studied foil exhibit thin lengthy sticks, either observed on the foil surface or protruding in random directions. Figs. 4a and 4b in Ref. [16] show SEM images of two regions of this large foil, where element content was measured at points 18 and 19, respectively. Data shown in Table 1 give element concentrations at these two points.

Almost two and half years later, another determination of element content of investigated elements was done. Some differences were noted, such as the absence of nitrogen, smaller relative amount of oxygen and confirmation of quite large content of magnesium. See below Table 2.

4. Density Measurements

For this purpose two almost flat elements and regular shapes were taken under consideration. Using proper instruments the thickness was defined as 0.22 ± 0.02 mm, values of surfaces 13.3 and 20.1 mm² with

relative uncertainty \pm 6% and masses 4.157 and 4.316 mg. Since two values of density 1.42 and 0.98 g/cm³ and means value as a result d = (1.20 \pm 0.24) g/cm³

(relative uncertainty \pm 20%). Our results are related to other data for different coal phases as below see Table 3. One can see its small value.

Table 1	The main element com	position of foils, show	wn in Fig. 4a and 4t	at two points in Ref. [16].
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Element	Z	Weight%	at.%	Weight%	at.%	
		Fig. 5, p.18 [16]		Fig. 5, p.19 [16]		
С	6	59 ± 7	66.38	60.7 ± 7	67.04	
Ν	7	5.9 ± 1.2	5.69	5.5 ± 1.3	5.22	
0	8	30.1 ± 3.8	25.42	32.6 ± 4.4	27.05	
F	9	-	-	0.37 ± 0.17	0.26	
Na	11	0.42 ± 0.06	0.25	$0.03\pm0,03$	0.02	
Mg	12	3.02 ± 0.20	1.68	$0.44\pm0,\!05$	0.24	
Al	13	0.18 ± 0.04	0.09	0.11 ± 0.03	0.05	
Si	14	0.25 ± 0.04	0.12	0.07 ± 0.03	0.04	

Table 2EDX analysis of two graphite-like foils. Processing option: all elements analyzed (normalized). Number of iterations= 6.

Element	Weight%	at.%	Weight%	at.%	
	Point A		Point B		
С	64.31	72.00	71.43	78.36	
0	30.34	25.50	23.27	19.16	
Na	0.61	0.36	-	-	
Mg	2.32	1.28	3.06	1.66	
Al	0.08	0.04	0.08	0.04	
Si	0.27	0.13	0.33	0.15	
Р	0.53	0.23	0.75	0.32	
Cl	0.39	0.15	0.10	0.04	
Κ	0.09	0.03	-	-	
Ca	0.43	0.15	0.57	0.19	
Fe	0.06	0.01	0.12	0.03	
Cu	0.35	0.07	0.15	0.03	
Zn	0.23	0.05	0.15	0.03	

 Table 3
 Some physical properties of different states of coal, including data for investigated probe at room temperature.

Carbon phase	Density, g/cm ³	Resistivity, μΩm	Magnetic properties
Diamond	3.47-3.57	10^{16} - 10^{19}	Weak Diamagnetic
Graphite	2.10-2.23	$//(2.5-5.0)$ and $\pm 3.0 \times 10^3$	Strong Diamagnetic
Amorphous state	1.8-2.1	$(5-8) \times 10^2$	Weak paramagnetism
Soot	1.6-2.0 (1.7-1.9)	?	Weak paramagnetism
Fuleren C ₆₀	1.65-2.6	like diamond	Weak Diamagnetic
Graphen (0.77 mg/m ² /0.5nm)	1.5	1.0×10^{-2}	Diamagnetic
Nanotube, multiwall	0.6-2.0	- 10	Strong diamagnetic
Investigated probes	1.20 ± 0.20	$> 10^5$	Strong paramagnetic
C aerogels	$(0.16-0.18) \times 10^{-3}$	-	-
Cu (for comparison)	8.920	1.68×10^{-2}	Diamagnetic

5. Resistivity Measurements

Resistivity was measured in big approximation because of non regular form of specimen and not the best used electrical contacts. Using universal-high accuracy class—V, I, R meter we have obtained resistances of measured specimens higher than 2 M Ω . Taking under consideration specimen dimensions it was possible to determine the value of resistivity as shown in Table 3. It appeared to be very high. Temperature dependence of resistivity is in progress now.

6. Paramagnetic Properties

The magnetic properties investigation appeared to be very interesting. Comparing behavior of diamond and investigated specimens hanging on the long diamagnetic thread during slowly coming up of the strong constant magnetic fields to the specimens, it was possible to definite magnetic properties of graphite-like object. It was clear about diamagnetism of the diamond probe (no movement was observed) and paramagnetism of investigated specimen (strong attraction force of investigated foils to static magnet was noted). Also a similar effect to the diamond probe was observed in pure graphite probes, its diamagnetism was observed (we had some troubles in obtaining sufficiently pure graphite materials).

7. Dielectric Properties

Two flat probes were covered with gold and such elements as small parallel-plate capacitors were investigated. The results are as follows—relative dielectric constant ϵ_r was equal to 3-4, which means that investigated material can belong to classical, not polar dielectric. Literature data for graphite $\epsilon_r = 10-15$, for diamond $\epsilon_r = 5.5-10$ pointed out its high values. Exemplary for: teflon 2.1, paper 3.5, polyethylene 2.25, polystyrene 2.4-2.7.

The observed difference of presented here graphite-like material relative dielectric constant

relative to graphite or diamond dielectric constant is understandable because we have different materials and consequently different mechanisms of polarization.

8. Mechanical Properties

Preliminary studies have shown that graphite-like elements does not show homogeneous structure. The phenomenon of porosity however has not been noticed. They exhibit the fragility, shown in the photos, between areas of strict structure. In areas with a strict structure they also exhibit finite mechanical compression strength. In the compression probe we have noted its micro elasto-brittle state with strength 10MPa. The data, however, should be considered as indicative. No phenomenon of plasticity was observed. It also could not be established whether the investigated objects are able to loosely associate into small grains.

9. Structure of New Elements of Graphite-Like Elements XRD Characterization

Small part of the material called "graphite-like foils" was designed to X-ray diffraction analysis.

We tried to pulverize this material in an agate crucible. The process of pulverizing has demonstrated that the material can be ground, but does not crumble as a typical inorganic material do. During shredding and grinding, parts of the material show elastic properties and cling to each other, typically as polymeric materials.

The material after this procedure was deposited on a substrate of mono crystalline silicon and analyzed with Cu K α radiation using Siemens D500 powder diffractometer, equipped with high-resolution, Si semiconductor detector. Registered spectrum (Fig. 3) indicates a crystalline-like arrangement. The strongest line in the diffraction pattern corresponds to the distance d = 4.75 Å. We are unable to identify a chemical compound on the basis of the registered diffraction pattern, as well on the basis of the largest



Fig. 3 X-ray diffraction pattern of "graphite-like foil" sample.

C - C	$\mathbf{C} - \mathbf{C}$	- C	- C	- C	- C	-c	- C ·	– C
		Т				Т	1	Т
0	0		0		0		Mg	
	1	1	1	1	1	1	1	1
$\dot{\mathbf{C}} - \mathbf{C}$	- C -	- C -	- C -	- C -	- C –	- C -	- C –	· C
1 1		Т		Т		Т	1	I.
N - O	Ó		Ó		0		0	
1 1		Т		Т		Т		Т
$\mathbf{C} - \mathbf{C}$	- Ċ -	- C -	- Ċ -	- C -	- Ċ -	- C -	- <u>C</u> -	- C

Fig. 4 The proposal of the structural form for founded structure graphite-like product of gamma irradiation of compressed helium. Position of smaller content of N (5at%) and Mg (1at%) atoms are presented in not proportional manner. Those could be located in the place of O atom, see table 1 – (The main element composition of foils). It can be problem with Mg localization. One known chemical component magnesium with carbon called "Allilec magnesium" Mg₂C₃, another notationMg₂ (C= C= C), can makes some perturbation in proposed simple structure.

diffraction reference data base ICDD PDF4 + 2015, but the diffraction pattern is similar to patterns of polymeric organic compounds which contain in addition to carbon and hydrogen also nitrogen, oxygen and sulphur.

We have supposition for compose following atomic structure. We have typical graphical layers and in spaces between those hexagonally ordered carbon atoms are fulfilled by—in principle—oxygen atoms and other detected by MXAP as nitrogen, magnesium and other atoms of marginal amounts. Atomic radius, valence and Van der Waals radiuses of C and O and N are similar. Atomic and valence radiuses of Mg are two time larger but Van der Waals radius is almost the same. Localization of O atoms between C layers reduces almost all free electron of carbon sp3 hybrid making element as not conducting composition.

9.1 The structure of the graphite-like objects

We propose the following, in the first step—see Fig. 5, spatial phase structure graphite-like model. In this model, the position of the oxygen can be optional and have the location of the interstitial side. The position of the Mg is difficult to determine at this stage of research, even if it may be located in places like oxygen. Distance C in layers is 0.142 nm, the distance between the layers of 0.475 nm (0.335 nm in graphite), it means of 42% larger.

In Fig. 6 the overview figure illustrating the existence of different forms of carbon with different densities is presented. Only the point of the diamond was set arbitrarily on the horizontal axis.



Fig. 5 The first proposal of crystallographic structure for graphite-like object.

•—possible locations for O, Mg, N atoms and for others elements.

10. Thermal Properties

The thermal properties of the graphite-like object were also an interesting problem. When we have tried to deposit on the surface of the samples the gold thin layer, in the spray chamber the temperature of investigated specimen has increased to level of 200-300 °C. In these conditions the phenomenon of the appearance of large grains was observed, supposing melting phenomenon of the sample. In this situation, it authors made attempts to melting phenomenon, which showed that temperature of melting of graphite-like object obtained in the process of "burning helium" is not greater than 400 °C. It differs significantly from the temperatures of the fusibility of all currently known forms of carbon. The sample melted down, however, it remained non-conductive. The authors have predicted the re measurement of its chemical composition. The appearance of the sample on permanent metal (SS) ground shows photo 13. It should be noted that the sample before spraying on the background was free.

11. Pictures of Objects Being Investigated

Below few images of the described graphite-like objects, with multiplication factor about 50x, are presented. All surfaces of objects are natural. View of photographed objects is representative for all objects found in different places in HeHPC.

Investigated objects were stored in typical boxes in atmosphere typical for Laboratory Nuclear Reaction of JINR airy, without special deactivation. In such conditions our objects were stabilized in chemical and physical senses. The color of object—typically dark does not change in any degree. Such situation is observed since 2013 so w can speak about stabilized solid state.

With physical properties testing creations graphite-like objects shows that we are dealing with a solid and crystalline object. Construction of the largest objects is like a patchwork of larger blocks. As to the formation of such macroscopic objects after several



Fig. 6 Illustration of known carbon phases related to the increasing density.



Fig. 7 Photograph of graphite-like object with relative homogenous structure.



Fig. 8 Flat graphite-like object with of not known origin of the white place.



Fig. 9 The view of side surface (probably after accidentally breaking) and frontal surface naturally smooth.



Fig. 10 The photo showing natural cracks for one of investigated specimens.



Fig. 11 The photo of graphite-like object showing some colored places of not known origin.



Fig. 12 The photo of the surface of chosen specimen. One can see almost constant thickness of specimen. Specimen after cathode sputtering was mechanically corrected in its sides in order to avoid short circuits phenomena.



Fig. 13 The photo of one of two specimens after gold sputtering. Specimens have changed their look, its surface appeared a grainy structure, remaining not conductive of electrical current and closely bonded to the base material (stainless steel).

hours of the formation of specific atoms needed for their creation in the process of unknown closer to nuclear transformations is an open question. New elements are formed at the same time (different nuclear processes occurring at the same time of irradiation). It also an interesting fact the emergence of major elements in gamma in the inlet area elements made of CuBe2 where involved in the reactions associated with kernels 9Be or short lived 8Be is possible.

Following these photographs taken on the microscope of advanced type, it shows more precisely the construction elements of the graphite-like objects. Common objects of dot-matrix printers are most likely dirt coming from the ground "desiccant" that come with the graphite-like object have put just after demounting of high pressure apparatus (see Fig. 2).

12. Conclusion

It seems that the observed product of "helium burning" in our experiment is not a new carbon phase. We can speak about graphite-like phase or about a quite new solid object which has been just synthesized (discovered). There is another one problem if purity of introduced helium gas was destroyed by other gases being adsorbed on the inner walls of high pressure apparatus as for example on inner surface of high pressure capillaries and so on. The answer to this question and others will be found through the planned control experiment which will be conducted in not far future.

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Appendix

During preparation of the probe to be investigated by Sherer's powder method it was necessary to obtain its proper powder. At the beginning powdering process seems to be normal, but at the end of the process some problem seems to occur-polymer character of the rest material was observed. Maybe some amount of coal phase named polyynes can exist in the material (see systematization of coal phases).

The view of possible existing coal phases

- 1. Graphite a hexagonal β rhombohedron whiskers Graphen
- Diamond 3.

2.

Diamond standard

lonsdaleite (hexagonal diamond and graphite whiskers)

Fullerens (buckminsterfullerene) 4.

> Fullerens C 44, 50, 58, 60, 70...540. nano-tubes nano-onions) Fuleryte

- 5. Nano foam
- 6. Carbines (carbine) chaoit (polyynes) cumulen cyclocarbon
- 7. n-Carbon
- Carbon IV 8
- 9. Amorphous carbon
- 10. Soot
- 11. Investigated objects
- Ryc. 14. Structure of a polyynes



Other possibilities

Structures such as polyynes described above may cause difficulties in obtaining appropriate powder. Also the possibility of manganese compounds in industrial designs coal-like object described here could have an impact on the difficulties in the process of their powder spraying. For example, Allilek (sexsal carbide of magnesium) (solid), having crystalline structure, simplest chemical formula Mg_2C_3 , structural formula: $Mg_2(C = C = C)$ or more accurately Mg = C = C = C = Mg, as well as magnesium Acetylide (solid) - MgC₂, (another structural record Mg[C \equiv C])