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## ORIGINAL STUDY

# Electrical Conductivity of GMZ Grade Graphite as a Function of Fast Neutron Fluence

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## Abstract

The electrical conductivity of GMZ grade graphite irradiated by fast neutrons with fluence of  $2.22 \cdot 10^{19}$  n/cm<sup>2</sup> in the temperature range of 298–448 K was studied. It is shown that the more neutrons fluence, than the more the electrical conductivity of the material decreases.

**Keywords:** Electrical conductivity, GMZ grade graphite, Fast neutrons, Fluence, Dose

## 1. Introduction

In 1942, Enrico Fermi and his colleagues used graphite as a neutron moderator for the world's first self-sustaining nuclear chain reaction. Since graphite has proven itself as a neutron moderator, it has become widely used in the nuclear industry. Also, graphite was used in all industrial reactors for producing weapons-grade plutonium, in the first industrial reactor for producing tritium, as well as in the world's first nuclear power plant in Obninsk. Since 1973, large power plants with a reactor of the RBMK-1000 type, using graphite as a moderator, began to be built. Due to such widespread use of graphite in reactor construction, it was necessary to study the properties of graphite under long-term neutron irradiation. Such work was carried out at the Graphite Research Institute by scientists Yu.S. Virgiliev, G.M. Butyrin, I.I. Kalyagina. The Institute of Atomic Energy named after I.V. Kurchatov, as a leading center for reactor materials science, studied irradiated graphite samples. Significant contributions to these studies were made by P.A. Platonov, O.K. Chugunov, N.S. Burdakov, V.I. Karpukhin and others. In addition, work was carried out on calculation substantiation of the strength of parts and

assemblies made of graphite, in which Gurovich B.A., Tutnov A.A., Sergeeva L.V., Tkachev V.V., Manevsky V.N. participated. etc. Since the general designer of the RBMK reactors was NIKIET named after N.A. Dollezhal', then A.V. made a great contribution to the study of graphite. Subbotin, E.N. Sinitsyn.

A wide base of experimental knowledge was developed by specialists from the State Scientific Center RIAR (Dimitrovgrad): Pokrovsky A.S. Kharkova D.V.

Studies of the radiation resistance of various structural and carbon-containing composite materials for promising high-temperature nuclear reactors, as well as the effect of irradiation on the characteristics of nuclear fuel elements, were carried out by leading scientists from research centers around the world, including American (Y.S. Kim, A. Yacout), Russian (K. A. Konoplev, S.R. Friedman, V.M. Lebedov, S.N. Votinov), Kazakh (O.P. Maksimkin, A.V. Yarovchuk, L.G. Turubarova), Polish (W. Steke, T. Wagner, E. Khaevska), Israeli (A. Minitz, A. Shtechman), Ukrainian (V.N. Voevodin and others), Uzbek (R. Khanbekov, M. Sulaimonov, M. Zhamatov, T.B. Ashrapov, Kh. Khalilov, I. Abdukadyrova) and other specialists.

The development of nuclear technology is moving in the direction of increasing the energy and heat

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intensity, the intensity of radiation impact on individual structural elements of nuclear reactors, which puts forward increased requirements for the structural materials used. Among others, carbon-graphite materials are increasingly used in nuclear engineering, not only as masonry material, but also for structural elements, for example, for cladding and matrices of ball fuel elements, coatings of micro-fuel elements in high-temperature gas-cooled reactors (HTGR), screens in fusion reactors (TNR), etc. Depending on the operating conditions (neutron fluence, irradiation intensity and temperature), the requirements for materials for these installations differ significantly from the usual requirements for nuclear graphite [1,2].

Structural defects affect the electronic properties of graphite-like materials in a complex way. Thus, defects in the layer, on the one hand, are electron traps and create an excessive concentration of hole carriers, and on the other hand, they become effective centers for the dispersion of charge carriers and significantly reduce their mobility. If the effect of reduced mobility prevails over the effect of increasing the concentration of charge carriers (as, for example, in irradiated graphite), then the resistance increases. In graphite samples, in addition to structural defects and microheterogeneity, the electrical conductivity is significantly affected by macroscopic factors such as texture, porosity, and three-dimensional defects, as well as changes in the concentration of impurities.

The works [3,4] consider the possibility of using carbon-carbon compositions as cladding of fuel elements of materials for advanced HTGR (taking into account the limited capabilities of conventional nuclear graphites and matrix materials based on them). Of interest is the possibility of using carbon composite materials for the first wall of a fusion reactor, diaphragms, and intermediate shields separating the first wall from the plasma [5].

In works [6–13], the electronic structure and properties of composites of different grades of graphite for a high-temperature gas-cooled reactor, as well as the durability of reactor graphites, the structure of graphites and the electrical conductivity of graphite were studied. Reactor graphite is widely used as a core masonry material in uranium-graphite reactors, in particular, in RBMK-type power reactors.

It has been shown that reactor irradiation leads to a strong degradation of graphite's electrical conductivity, and a particularly rapid decrease in electrical conductivity is observed when neutrons are irradiated with small fluences.

In this paper, the experimental dependencies of the electrical conductivity of GMZ grade graphite on the neutron fluence of  $2.22 \cdot 10^{19}$  n/cm<sup>2</sup> are presented.

## 2. Materials and methods of the experiment

Samples of the GMZ grade graphite were prepared with a length of  $L = 5.94 \cdot 10^{-2}$  m, a width of  $a = 8.21 \cdot 10^{-3}$  m, a thickness of  $b = 8.25 \cdot 10^{-3}$  m, a mass of  $m = 6.3 \cdot 10^{-3}$  kg and were irradiated in the vertical standard channel 1 in the WWR-SM reactor to a neutron fluence of  $2.22 \cdot 10^{19}$  n/cm<sup>2</sup> ( $E > 0.8$  MeV) at temperatures of 323 K.

The neutrons spectrum emitted during fission is contained in a wide energy range from low energies to 25 MeV, which was calculated according to the method described in Ref. [14]. The average energy of fission neutrons is about 2 MeV, and the most likely is 0.8 MeV.

Above 3 MeV, as the neutron energy increases, the neutron flux decreases almost exponentially. The fast neutrons flux density ( $E_n > 0.8$  MeV) in standard channel 1 was  $3.73 \cdot 10^{12}$  n/(cm<sup>2</sup>\*s). The irradiation time of the samples in the channel was 1182 h.

The samples were placed in the vertical channel of the WWR-SM research reactor of the INP AS RU for fast neutron irradiation according to a loading scheme at a power of 8 MW.

Fig. 1 shows the WWR-SM reactor core and the location of the vertical channel in which the samples were irradiated.

The temperature in the channel was maintained at about 318 K. The heat released due to radiation heating was removed by circulating the primary circuit water at a volumetric rate of 1250 m<sup>3</sup>/h.

Fig. 2 shows the spectrum of neutron flux density in 1 channel for neutron energies from 1 eV to 9 MeV.

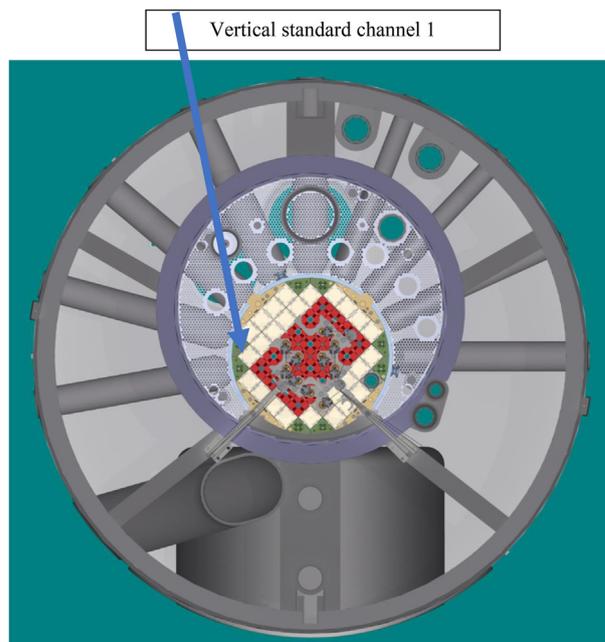


Fig. 1. The WWR-SM reactor core.

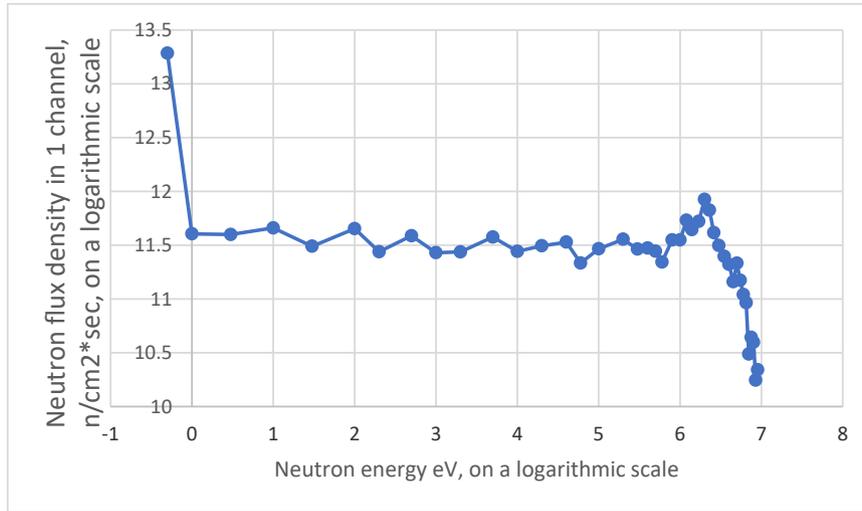


Fig. 2. Neutron spectrum in vertical channel 1.

Neutron flux density in 1 channel,  $n/cm^2 \cdot sec$ , on a logarithmic scale.

From the figure it can be seen that in vertical channel 1 there are more thermal neutrons, but during the fission of uranium –235 fast neutrons with an average energy of 2 MeV are formed. More burned-out nuclear fuels are located on the periphery. Neutron moderation occurs due to reactions of elastic scattering of neutrons with water atoms.

When graphite is irradiated with fast neutrons ( $E > 0.1$  MeV), the main contribution to the change in the parameters of the graphite crystal lattice comes from the formation of interstitial atoms and vacancies, double, triple and larger, up to extraplanes.

Thermal neutrons form radioactive nuclides in graphite, long-lived radionuclides with high migration ability in natural systems. Such radionuclides include  $^{14}C$  (half-life - 5730 years).

Fig. 3 [15] shows the spectrum of neutrons in a research thermal nuclear reactor.

As can be seen from Figs. 2 and 3, the maximum of thermal neutrons is located in the reflector where the vertical irradiation channels are located, and the maximum of fast neutrons is in the nuclear fuel, where fission of uranium 235 occurs.

### 3. Results of the experiment and calculations

Solid (piece) and powdered GMZ grade graphite was prepared to determine the impurity composition of the sample.

Table 1 shows the percentage of impurities of GMZ grade graphite obtained using the EVO MA10 scanning electron microscope.

Fig. 4 shows an electronic image of the surface of GMZ grade graphite and X-ray fluorescence spectral

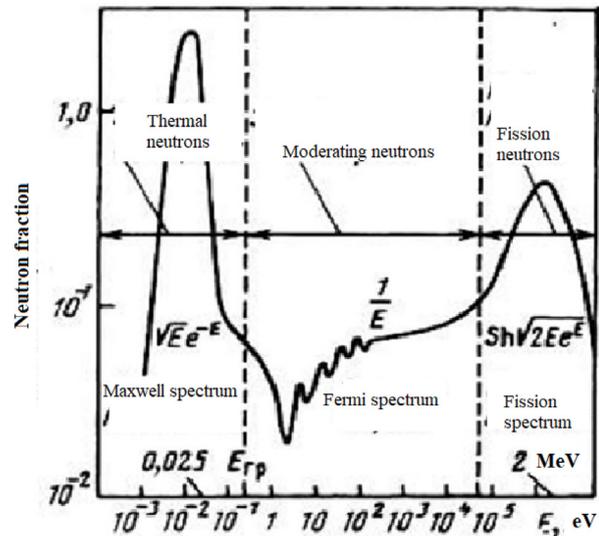


Fig. 3. Neutron spectrum in a research thermal nuclear reactor.

Table 1. Content of impurities of GMZ grade graphite.

Element	Type of line	Weight, %	Error Weight, %	Reference Name
C	K Series	98,39	0,25	C Vit
O	K Series	1,51	0,24	SiO2
Si	K Series	0,11	0,05	SiO2
Sum:		100,00		

analysis using the EVO MA10 scanning electron microscope.

Using the technique of instrumental neutron activation analysis of elements, the content of impurity elements in the samples was determined. A gamma-ray spectrometer with a semiconductor detector GC-1820 and a multichannel analyzer InSpector-2000 with

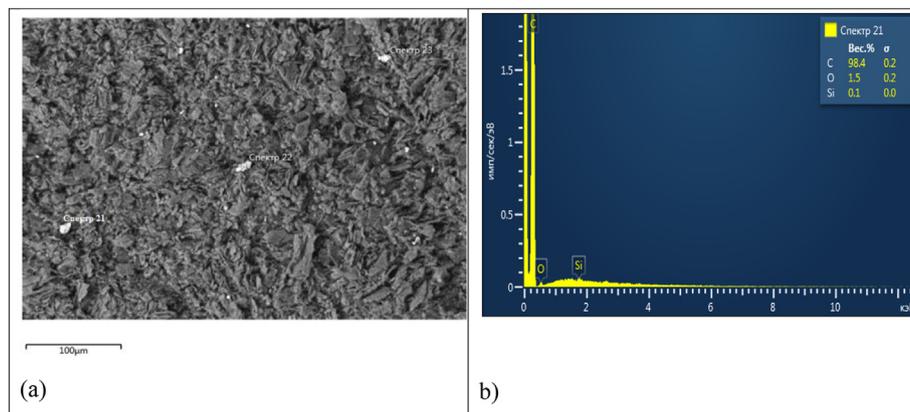


Fig. 4. Electronic image of the surface of GMZ grade graphite (a) and elemental composition (b).

Genie-2000 software (CANBERRA, USA) was used to measure the samples. IAEA standards with certified content of elements in soil and bottom sediments were used to calculate the concentration of impurity elements.

Table 2 shows the results of determining the content of impurity elements in the GMZ grade graphite, obtained by the instrumental neutron activation analysis method.

The content of other impurity elements (for example, Boron) is less than 0.0001 µg/g, so we did not detect it.

#### 4. Electric conductivity measurement results

The method for measuring the electrical conductivity of samples is based on the use of a four-probe compensation method (type 3207 microohmmeter) [16].

Type 3207 microohmmeter [16] is a digital microohmmeter with a 4½-digit LED display, capable of measuring low resistance with a resolution of 0.1 µΩ, is ISO 90001 internationally certified. This method is

used to eliminate the effect of pin resistance on the accuracy of measurements.

The specific electrical resistivity  $\rho$  was determined from the relation:

$$\rho = \frac{R \cdot S}{L}$$

where  $R$  is the resistance of the sample,  $S$  and  $L$  are the cross-section and length of the sample, respectively.

The dependence of the electrical resistance of graphite samples on temperature was studied using an electric resistance furnace in the range of 298–448 K. The temperature in the furnace changed at a rate of 2–5 K/min and was controlled by a chrome-aluminum thermocouple. Measurements were carried out in heating and cooling mode at 25 K, with a holding time of 30 min at each measurement point.

Fig. 5 shows the results of measurements of the resistivity of non-irradiated GMZ grade graphite depending on the temperature of the sample.

Table 2. The content of impurity elements in GMZ grade graphite.

Element	Concentration, C ± ΔC,	Concentration, C ± ΔC,	Element	Concentration, C ± ΔC,	Concentration, C ± ΔC,
	ppm (µg/g)	ppm (µg/g)		ppm (µg/g)	ppm (µg/g)
	Graphite Chunk	Graphite Powder		Graphite Chunk	Graphite Powder
As	(2,96 ± 0,09) E–02	(1,24 ± 0,07) E–01	Nd	(7,52 ± 0,83) E–01	(1,26 ± 0,23)
Ba	(7,48 ± 0,60)	(1,50 ± 0,27) E+01	Ni	(8,60 ± 1,03) E–01	(2,73 ± 0,63)
Br	(4,16 ± 0,40) E–02	(3,65 ± 0,13) E–01	Rb	(5,33 ± 0,69) E–02	(3,88 ± 0,39) E–01
Ce	(1,59 ± 0,13)	(2,33 ± 0,19)	Sb	(8,90 ± 0,71) E–03	(1,02 ± 0,09) E–01
Co	(1,79 ± 0,20) E–02	(1,69 ± 0,19) E–01	Sc	(2,83 ± 0,11) E–01	(4,12 ± 0,16) E–01
Cr	(8,28 ± 0,75) E–02	(5,40 ± 0,38)	Se	(3,96 ± 0,59) E–03	(2,10 ± 0,76) E–02
Cs	(8,25 ± 1,32) E–03	(2,25 ± 0,36) E–02	Sm	(7,53 ± 0,45) E–02	(1,48 ± 0,10) E–01
Eu	(1,42 ± 0,12) E–02	(2,01 ± 0,17) E–02	Sr	(3,10 ± 0,43)	(5,10 ± 0,72)
Fe	(5,30 ± 0,27) E–04	(4,51 ± 0,17) E–02	Ta	(2,14 ± 0,30) E–03	(5,76 ± 0,98) E–03
Hf	(8,23 ± 1,25) E–03	(2,35 ± 0,53) E–02	Tb	(2,60 ± 0,26) E–02	(4,03 ± 0,13) E–02
K	(9,69 ± 1,98)	(6,51 ± 1,33) E+01	Th	(2,92 ± 0,23) E–02	(5,55 ± 0,78) E–02
La	(7,79 ± 0,62) E–01	(1,23 ± 0,10)	U	(5,71 ± 0,79) E–02	(1,22 ± 0,11) E–01
Lu	(1,31 ± 0,09) E–02	(2,36 ± 0,33) E–02	W	(3,60 ± 0,40) E–02	(9,17 ± 0,73) E–01
Mn	(1,66 ± 0,22) E–01	(5,14 ± 0,41)	Yb	(7,92 ± 0,46) E–03	(1,78 ± 0,24) E–02
Mo	(1,56 ± 0,14) E–01	(4,18 ± 0,67) E–01	Zn	(5,79 ± 0,87) E–01	(1,02 ± 0,15) E+01
Na	(2,39 ± 0,25)	(3,19 ± 0,21) E+01			

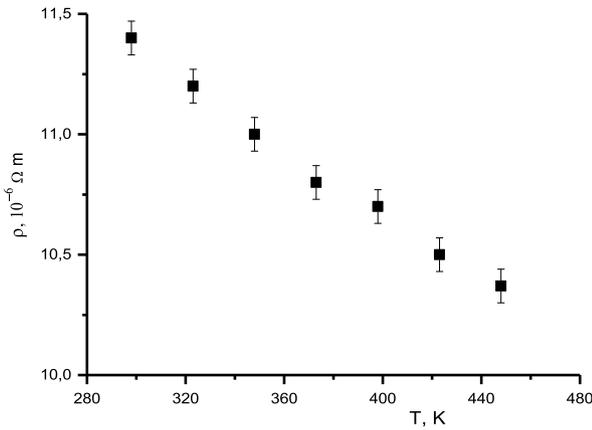


Fig. 5. Results of resistivity measurements of non-irradiated GMZ grade graphite depending on the temperature of the sample.

Fig. 6 shows the results of measurements of GMZ grade graphite resistivity irradiated with  $1.59 \cdot 10^{19}$  n/cm<sup>2</sup> fluence depending on the temperature of the sample.

## 5. Analysis of experimental data

The value of GMZ grade graphite resistivity at room temperature prior to irradiation was  $1.14 \cdot 10^{-7}$  Ohm\*m. The value of GMZ grade graphite resistivity as a result of irradiation increased by 2.78 times and amounted to  $3.17 \cdot 10^{-7}$  Ohm\*m. The increase in resistivity is due to the formation of defects as a result of irradiation of samples with fast neutrons in the core of the WWR-SM reactor.

At the same time, a decrease in resistivity was observed depending on the increase in the temperature of the sample in the range from 298 K to 423 K. This is due to an increase in the mobility of the charge carriers with an increase in temperature.

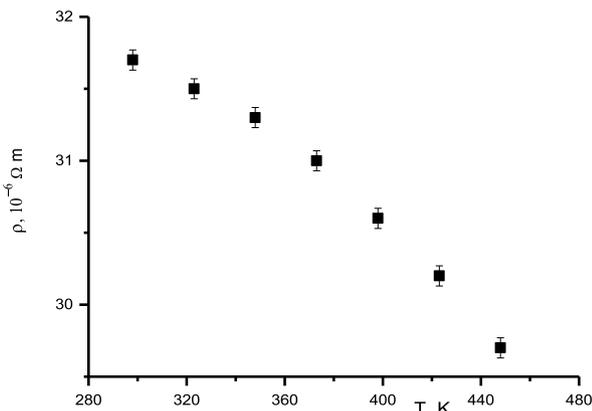


Fig. 6. Results of measurements of GMZ grade graphite resistivity irradiated with  $2.22 \times 10^{19}$  n/cm<sup>2</sup> fluence depending on the temperature of the sample.

An increase in the resistivity of G347A graphite samples at high fluence was also noted in Ref. [17].

## 6. Conclusion

Thus, the analysis of the experimental results obtained made it possible to establish that the resistivity of graphite changes non-linearly with temperature. At the same time, with an increase in neutron fluence, the resistivity of GMZ grade graphite increases.

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## Conflict of interest

The authors declare no conflict of interest regarding the publication of this article.

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