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Phosphate Glass Detectors for Heavy Ion Identification

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Abstract: The problem of the boundaries of the Mendeleev table of chemical elements is closely related to the understanding of the properties of nuclear matter. In this regard, the synthesis of superheavy nuclei on accelerators and the registration of their decay products are of fundamental scientific interest. The Joint Institute of Nuclear Research in Dubna (JINR) conducts research on the synthesis of superheavy nuclei on the new DC-280 cyclotron (the Factory of Superheavy Elements). As part of the development of this experiment, the possibility of using phosphate glass as a material for detectors of heavy and superheavy nuclei is being considered. This issue requires test experiments to study the recording properties of the glass at different irradiation and treatment conditions. The article presents a method for identifying heavy ions in phosphate glass detectors under various conditions by the geometric characteristics of ion tracks. The results obtained indicate the possibility of using the KNFS-3 phosphate glass detectors for registration and identification of accelerated superheavy nuclei.

Keywords: phosphate glass detectors; superheavy elements; ion charge identification

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1. Introduction

The problem of the existence of superheavy transfermium elements is one of the urgent problems of modern physics. The largest experimental laboratories around the world are working on the synthesis and registration of superheavy nuclei [1,2]. Dielectric track detectors, including those made of glass, have high efficiency in detecting heavy ions and low sensitivity to background radiation, and, therefore, are widely used in experimental physics, including heavy-ion physics [3–7]. In this regard, the study of the registration properties of phosphate glasses for the study of synthesized heavy nuclei at different irradiation and treatment temperatures is of great interest.

The principle of the operation of the dielectric detector is based on the effect of destruction of the local crystal structure of the material along the trajectory of a penetrating swift heavy ion. This local destruction is intensified by subsequent selective etching, when the damaged areas of the material react with the etching agent more intensively (with a higher rate) than the undamaged ones, resulting in formation of the visible etched channels of a scale up to several tens of microns. The correlation between etching speed in the area of a latent track and the ionization losses formed a basis for determining a particle's characteristics. Identification of heavy nuclei in dielectric detectors is based on the analysis of geometric parameters of the etched tracks, since the scale of radiation damage to the dielectric material is proportional to the charge of a particle.

Prompt development of the dielectric detector technology occurred in the 1960–1970s, when these detectors found their application for the registration of the synthesized superheavy nuclei [8,9], in particular, for registration of the element 105 in the Flerov Laboratory of Nuclear Reactions (FLNR) of the Joint Institute of Nuclear Research (JINR, Dubna, Russia) [10]. Currently, JINR is conducting experiments on the synthesis of superheavy nuclei on the new DC-280 cyclotron, the Factory of Superheavy Elements [11]. In this regard, the interest in the phosphate glass detectors renewed. Considering that the registration of ions in the experiment on the synthesis of superheavy nuclei at the accelerator occurs at elevated temperatures, up to 500 °C, test experiments are needed to study the registration properties of phosphate glasses, including at different irradiation and treatment temperatures.

The successful application of phosphate glass as a track detector in a thermochromatographic column used in experiments to study the chemical properties of superheavy elements requires that phosphate glass retain the ability to register fragments of nuclear fission in a wide temperature range. In general, thermal oxidation processes can occur in the heated glass, changing its properties. The registration characteristics of phosphate glass may change under the influence of heating. In particular, heating can lead to annealing (shortening or complete disappearance) of tracks. The effect of temperature on the result of etching of latent tracks depends on whether the heating took place before, during or after irradiation. According to available data [12], phosphate glass does not change its registration properties even after melting (and subsequent cooling). The track preservation temperature (annealing temperature) for phosphate glasses, according to various data, ranges from 250 to 500 °C [12,13]. However, the size and shape of the track etching figures can noticeably change. In this regard, it is necessary to determine the annealing temperature of the tracks in the phosphate glass used.

In this paper, we present the results of testing KNFS-3 phosphate glass as a track detector for registering superheavy nuclei. The article describes the works on the choice of the glass irradiation mode with accelerated heavy ions, the etching and processing modes and the method of the track image analyzing. The influence of different temperatures on the characteristics of etched tracks is considered. It is shown that the analysis of the geometric parameters of the etched tracks based on the original technique enables the identification of the charge of the incident ions, which indicates the applicability of the tested glass for the registration of superheavy ions on the DC-280 cyclotron.

2. Material and Methods

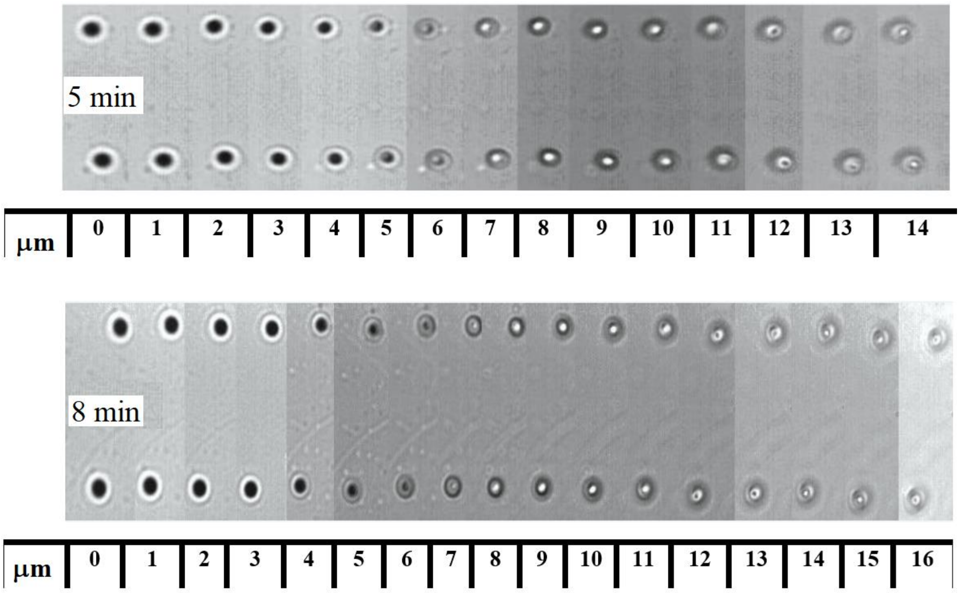
2.1. Glass Characteristics and Irradiation Mode

Phosphate glass is an inorganic dielectric with a wide range of composition, with P_2O_5 as the main glass-forming component. The technology of manufacturing optical phosphate glass of the KNFS-3 brand on LZOS provides high uniformity, temperature resistance and thermal conductivity, and low coefficient of linear thermal expansion. These properties are of fundamental importance for operation in high radiation conditions, as on accelerators. Since the composition of phosphate glass can vary, the study of the properties of a particular detector implies calibration experiments on accelerators with accurate knowledge of the ion charge and energy. In the presented tests, about 100 samples with dimensions of $10 \times 5 \times 4$ mm, providing an optimal combination of optical transparency and mechanical strength, were irradiated with $^{40}Ar^{8+}$, $^{84}Kr^{17+}$ and $^{132}Xe^{26+}$ ion beams with an energy of 1.16 MeV/nucleon at different inclination angles of the beam to the glass surface.

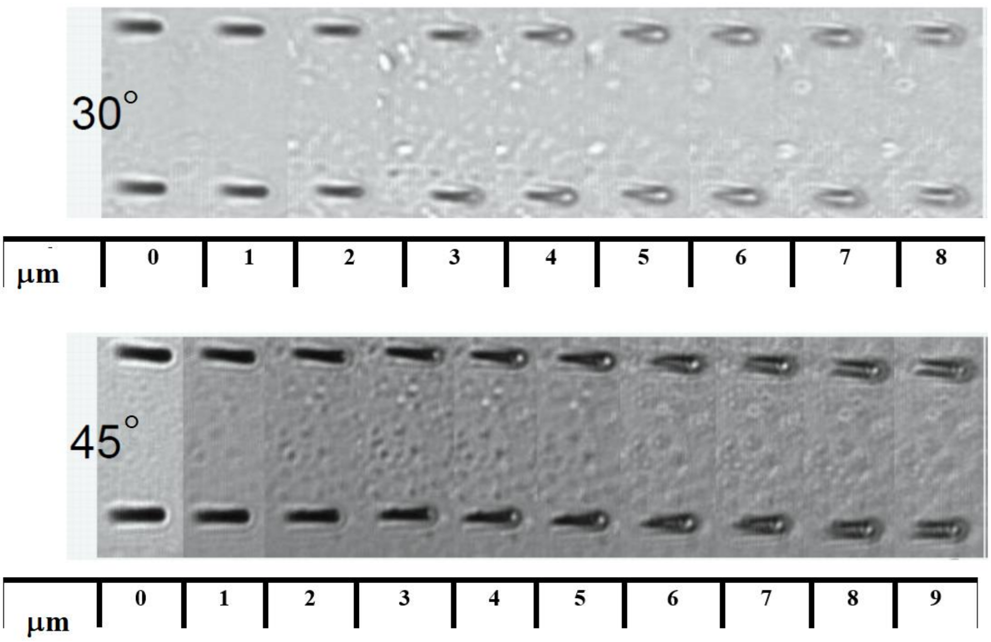
A factor significantly affecting the efficiency of the ion registration by a solid detector is the density of the ion beam: high density leads to the overlap of the tracks and loss of accuracy in measuring their characteristics. Modeling performed for various beam densities demonstrated that the best efficiency of the ion track registration can be achieved at beam densities of 10^4–10^5 particles per cm^2, depending on the possible track size [14]. The prepared phosphate glass samples were exposed to radiation in the transport channel of the withdrawn ion beam of the resonant cyclic accelerator IC100 (FLNR JINR). The ion flux controlled by PMT (Hamamatsu H-10721-110, Japan) made 1300 particles/cm^2 during the irradiation session of about 1 s.

2.2. Measurements

Figure 1 illustrates results of etching. The photos of $^{132}Xe^{26+}$ ion tracks on the glass surface obtained in the test experiment for different beam inclination angles with various etching duration are presented.



(**a**)



(**b**)

Figure 1. The shape of the $^{132}Xe^{26+}$ ion tracks after etching in 40% HF for different etching depths (indicated under the images): (**a**) vertical entry, ion energy E = 65–68 MeV (the etching duration is given); (**b**) entry at 30° and 45° to the normal to the glass surface, ion energy E = 160 MeV; the etching duration is 5 min (No color).

The geometric parameters of the etched tracks were measured on the automated complex PAVICOM using the original author's programs [15,16]. One of the programs enables the search for tracks and determines their geometric shape on the glass surface. Since the track of a heavy ion on the glass surface, in general, is of elliptical shape, the program sets the procedure for its parameterization with an ellipse. Figure 2 illustrates the procedure.

Figure 2. Algorithm of the image processing for ion tracks with inclination of 30° to the glass surface. The ion track shape on the surface is parameterized by an ellipse with the PAVICOM software. The image was simulated for ellipse with large axis of 18 microns and small ellipse axis of 11 microns corresponding to xenon tracks (Color).

2.3. The Etching Mode

The irradiated pieces of glass were processed in the chemical laboratory of the Lebedev Physical Institute (LPI). Depending on the characteristics of the glass, different means and modes of chemical etching are used with variation of reagents, concentration, temperature, and duration of etching. Testing and selection of the optimal etching agent was held at room temperature. In this work, hydrofluoric acid HF and sodium hydroxide NaOH were tested as etching solutions. The time of action of etchants to achieve the same result varies in a large range. The optimal etching duration in terms of track contrast was less than 1 h for hydrofluoric acid solution and from 5 to 10 h for sodium hydroxide solution. This result determined the choice in favor of hydrofluoric acid solution due to the significantly shorter etching duration and higher chemical stability of the solution.

In the first tests, the expected dependence of the track dimensions on the ion charge was not observed. The reason for the lack of the expected result could be a high concentration of the etchant (40% HF), at which the phase of optimal track development granting the best resolution (for the given glass and etchant) could be skipped at the taken etching intervals.

In the subsequent series of the etching tests, the optimal concentration of hydrofluoric acid was determined to obtain the maximum track contrast. The etching sessions were carried out in hydrofluoric acid of various concentrations, from 3% to 40%. According to [17], etching in a weaker HF solution gives a higher efficiency of track registration (i.e., a greater difference between the etching rate of the latent track area and of the undamaged material). This may be due, in particular, to better convection of the etchant in the track area while slow etching than while fast etching in a concentrated solution. At the same time, too low concentration turns insufficient for the appearance of visible tracks. In our studies, as a result of multiple tests, a 20% concentration of the HF solution was taken, which made it possible to "separate" tracks of the different accelerated ions ($^{40}Ar^{8+}$, $^{84}Kr^{17+}$, $^{132}Xe^{26+}$)

(see Section 3). At the same time, the etching duration was increased to 30 min for the first etching with consequent 10 min steps.

2.4. Thermal Testing

Since different temperature regimes may occur in the experiment, the development of a heavy ion registration technique requires the study of the possible effect of high temperatures on the detector material and its registration properties. In the tests presented, the dependence of the etched track parameters on the temperature and duration of heating of the glass sample at different stages of the irradiation and processing was studied.

As a result of heating, thermal oxidation process occurs in glass, which can change its properties. In particular, the heating of glass can lead to thermal annealing of latent tracks, up to their complete disappearance. The temperature effect on the latent track etchability depends on the stage at which the heating occurred—before, during, or after irradiation. According to classical studies, phosphate glass, even after melting (and subsequent cooling), does not change its registration properties up to temperatures of about 500 °C [18,19]. An indicator of the resistance of the tracks to temperature changes is thermal stability, that is, the temperature at which 100% of the tracks in the material retain the ability to etch after annealing for 1 h. This value depends on the chemical composition and etching conditions of the irradiated material.

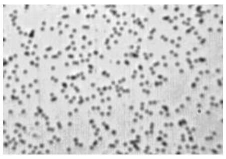
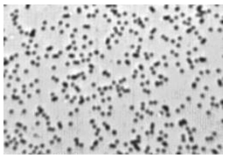
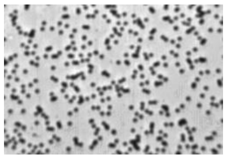
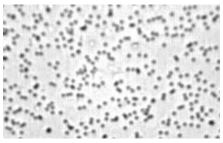
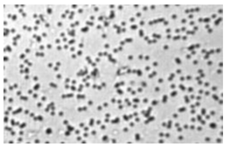
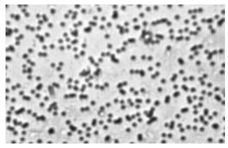
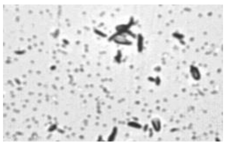
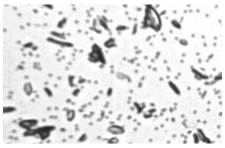
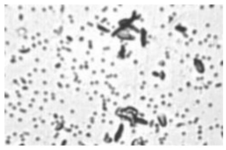
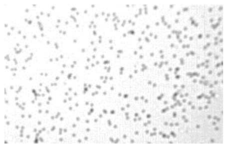
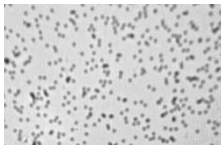
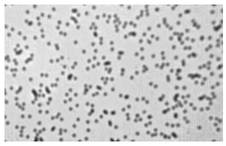
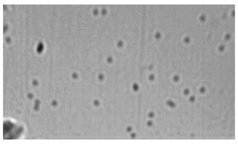
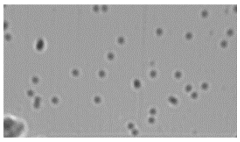
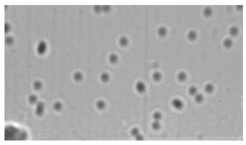
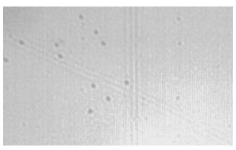
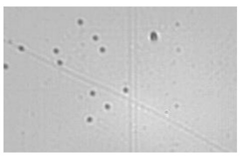
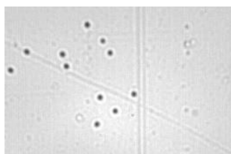
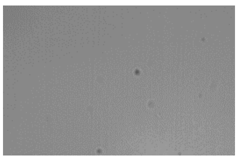
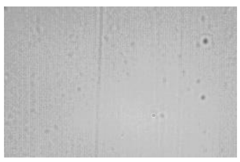
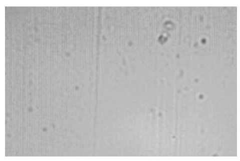
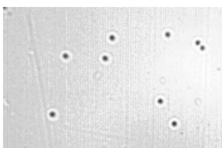
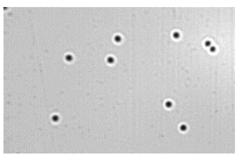
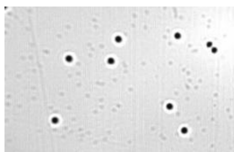
We have analyzed the changes in the properties of the tracks of heavy nuclei in the KNFS-3 phosphate glass exposed to irradiation in a heated state with subsequent cooling in two modes: immediate cooling after irradiation and cooling after several hours of holding at a given temperature. The thermal studies were carried out for $^{132}Xe^{26+}$ with a beam energy of 167 MeV at normal incidence. The samples were irradiated at temperatures of 100, 200, 300, 400, and 500 °C.

Two series of thermal tests were held. During the synthesis of superheavy elements at the JINR accelerator, the detectors are at elevated temperatures. Therefore, it is necessary to study changes in the characteristics of heavy ion tracks under different heating conditions. In the first one, phosphate glass samples (hereinafter indicated as type I), preheated before irradiation to temperatures from 200 to 500 °C in increments of 100 °C, cooled naturally to room temperature immediately after irradiation. When heated, the time to reach the required temperature was less than 60 min, and the cooling time ranged from 30 min for a temperature of 200 °C to several hours for 500 °C. The ion beam density was 10^6 cm^{-2}. In the second series, the samples (designated as type II ones) were heated before irradiation from 100 to 500 °C in no more than 60 min, and, after irradiation, were kept at a heating temperature for 10 h. The subsequent cooling took from 30 min for 100 °C to several hours for 500 °C. The irradiation density of the samples, heated to a temperature of 300 °C, was 10^6 cm^{-2}, and for those irradiated at temperatures of 100, 200, 400, and 500 °C the irradiation density was (5–8)·10^5 cm^{-2}. The etching was carried out at intervals of 5 or 10 min until the total etching period of 90 min was reached.

Table 1 presents photographs of one field of view (75 × 45 microns) of I and II type samples' etched surfaces at 40× magnification. The table shows that the density of the tracks in samples of I and II types noticeably differs, which indicate the dependence of the formation efficiency of the latent tracks on the residence time of the sample in the heated state.

Comparison of these results with the previously obtained ones (without heating [14]) shows that the heating of the detectors before irradiation does not have a noticeable effect on the formation and size of the etched tracks. At the same time, exposure of samples after irradiation in a heated state for 10 h affects the track development. The track diameters in type II samples heated up to 200 °C reach the values obtained in type I samples after a relatively short etching. However, when the heating temperature of Type II samples exceeds 200 °C, visible tracks appear only after 20 min of etching. As the etching duration increases, the diameters reach the sizes obtained without 10-h exposure at high temperature only after 70–80 min of etching. After heating to 400 °C and above, the visible tracks appear after about 40 min of etching. Then they slowly increase, but do not reach the same size as when heated to ≤300 °C.

Table 1. The glass surface at different test conditions.

Type I			
Temperature, °C	**Etching 30 min**	**Etching 40 min**	**Etching 50 min**
200			
300			
400			
500			
Type II			
Temperature, °C	Etching 30 min	Etching 40 min	Etching 50 min
200			
300			
400			
500			

It can be assumed that prolonged exposure of irradiated samples at a temperature of 300 °C and above leads to the restoration of atomic defects and broken interatomic bonds that arose when an ion passed through the glass. The “healing” of tracks, apparently, begins from the surface, and the higher the sample temperature and the longer the exposure time, the deeper this process penetrates. Nevertheless, even with prolonged exposure at higher temperatures, the latent tracks still remain in the depth of a sample. When the etching of samples exposed at 300 °C and above reaches the remaining of the hidden track, a relatively shallow visible track is formed (as evidenced by the less-black color of the etched track). With further etching at 300 °C, the diameter and depth of the tracks increase, and at temperatures of 400 °C and above, the tracks remain barely distinguishable even after an hour and a half of etching.

Thereby, the thermal tests showed that irradiation of the heated phosphate glasses, as well as their subsequent exposure at temperatures up to 200 °C, slightly affect the characteristics of the tracks of the accelerated heavy ions etched in them. However, even when the samples are heated to higher temperatures (up to 500 °C) and maintained in a heated state for a long time, the latent ion tracks remain, but with significant modification of their characteristics. A possible reason for the change in the characteristics of the tracks may be a decrease in the viscosity of the glass or greater mobility of atoms at high temperature, leading to the filling of vacancies resulting from the passage of the ion. The influence of different temperature regimes on the formation of the etchable defect zone requires further experimental study.

3. Results and Discussion

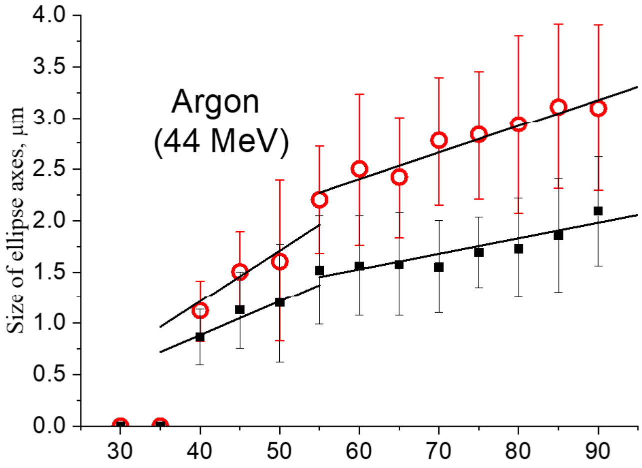
We use an original technique for determining the ion charge by the etched track size. The charge value is estimated on measurements of the major and minor axes of the ellipse of the entrance hole of the etched track on the glass surface.

The results of etching in 20% HF tracks of nuclei of different charges with an entry angle of 30° to the glass surface are presented in Figure 3. The large (red dots) and small (black dots) ellipse axes from various ions on the glass surface are given as a function of the etching duration. The data was obtained without heating the samples.

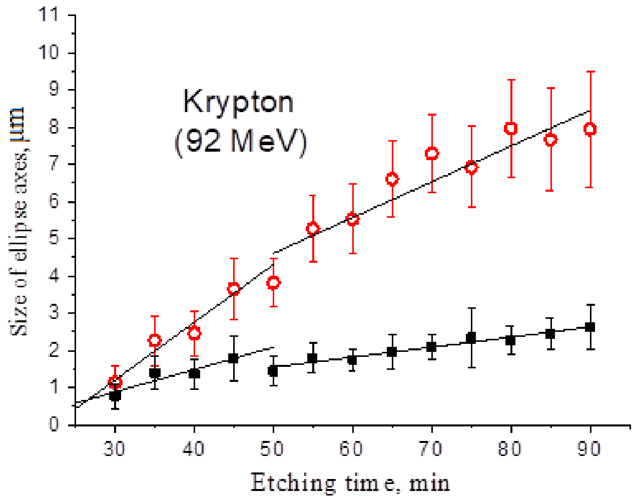
The nature of the dependence obtained testifies that the etching process splits in two stages. At the first one, with shorter etching intervals (before the “knee”), a track is etched to the ion stopping point. In the segment with longer etching intervals (after the “knee”), isotropic etching of the material occurs without further development of the track. The segments in Figure 3 are highlighted by the approximation of the probability density function by the least-squares method. In the area of the second segment, the etching rate determined as an angle coefficient of the corresponding segment is noticeably lower than the first one.

Figure 4 presents the slope factors of the segments (rates of change in the ellipse axes) as a function of the ion charge. The comparison shows that, for short etching intervals (Figure 4a), this dependence is more pronounced; while, with increasing etching duration, the sensitivity to charge decreases (Figure 4b). It follows that, under the given conditions, the etching intervals exceeding 50 min are less informative for determining the ion characteristics.

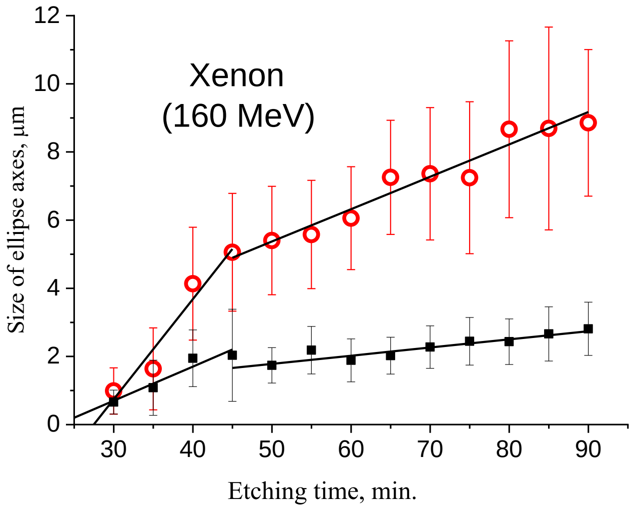
The presented analysis of the dependence of the geometric parameters of the track shape on the glass surface on the etching duration, and the rate of this variation makes an algorithm for identifying ions in phosphate glass. The ion charge can be estimated on the base of dependences similar to those obtained in our calibration experiments (Figure 4 by interpolating the inverse function. Etching at minor intervals and monitoring the characteristics of the etched tracks increases the reliability of the ion identification.



(**a**)



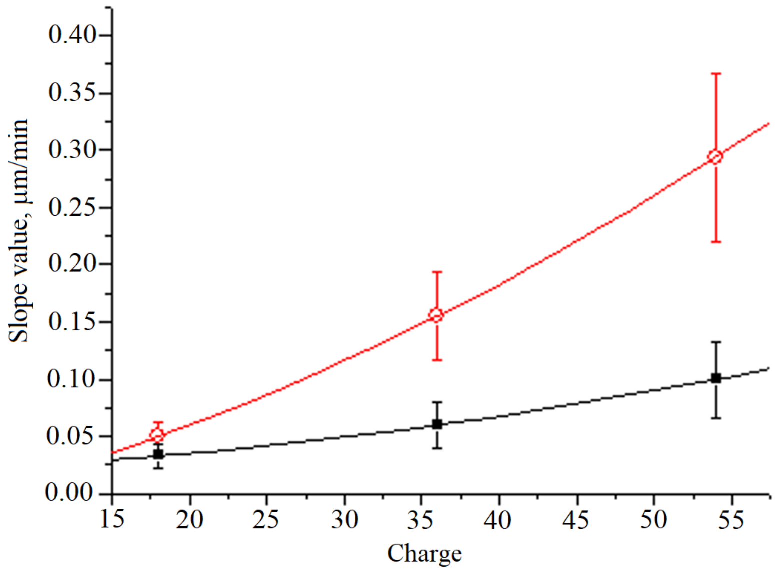
(**b**)



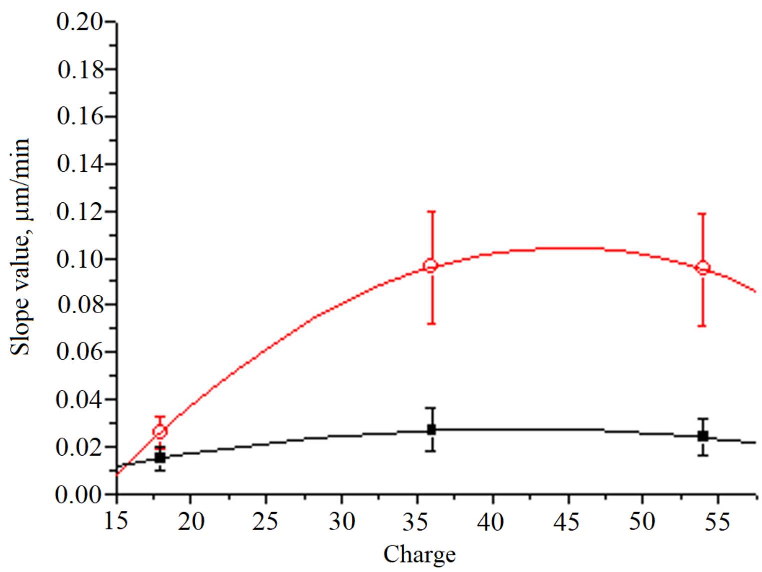
(**c**)

Figure 3. Dependence of the large (red points) and small (black points) ellipse axes on the glass surface on the etching duration for different ions ((**a**)—Argon, (**b**)—Krypton and (**c**)—Xenon). The beam inclination angle is 30° to the glass surface (Color).

Dependence of the slope factors of the segments on the ion charge, as those shown in the Figure 4, enables the estimation of the charge of the other nuclei for which the etching procedure with an etching time of up to 45–50 min was carried out and the slope factor was determined. In Figure 5, the arrows indicate the procedure for determining the nucleus charge for a slope value of 20 microns per minute, determined as Z = 45. To evaluate the accuracy of the charge determining, one can use the magnitude of the slope measurement error for krypton (Z = 36), determining which charges correspond to the upper and lower limits of error. Auxiliary lines on the graph show that these boundaries correspond to charges Z = 33 and Z = 39. Thus, the slope of 20 microns per minute corresponds to the charge Z = 45 ± 3. This algorithm allows the estimation of the accuracy of determining the ion charge by the proposed method.



(**a**)



(**b**)

Figure 4. Dependence of the slope factors (rates of change in the track dimensions) on the ion charge for large (red line) and small (black line) axis of the ellipses: (**a**) the etching duration from 30 to 45 min; (**b**) the etching duration more than 50 min. The experimental points are approximated with parabolas (Color).

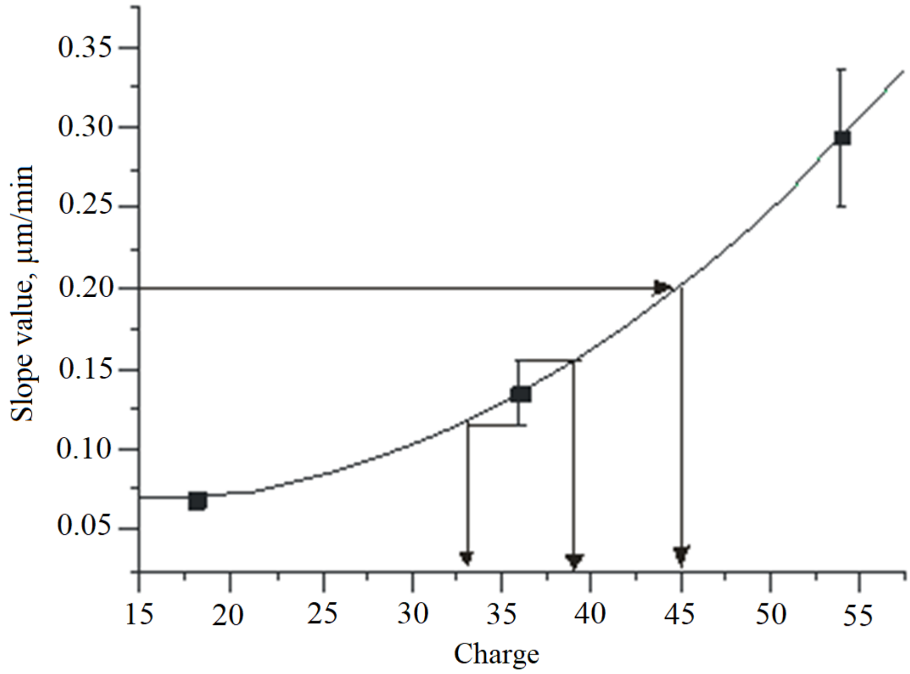


Figure 5. Algorithm for the ion charge determining (No color).

The presented data was obtained for ions with a fixed energy (about 1.16 MeV/nucleon). In general, the etching track modification should also depend on the ion energy and its entry angle, and the function for determining its characteristics will be set by a parametrical surface.

4. Conclusions

The key technological requirement in determining the Mendeleev table limits, which boils down to the f the problem of the superheavy transfermium elements, consists in the possibility of registration and identification of synthesized superheavy nuclei.

This paper discusses the possibility of using KNFS-3 optical phosphate glass to identify accelerated heavy ions with charges $Z > 20$ and energies above 20 MeV for use on the DC-280 cyclotron (the Factory of Superheavy Elements) in the Flerov Laboratory of Nuclear Reactions (FLNR) of the Joint Institute of Nuclear Research (JINR, Dubna, Russia). For the successful application of phosphate glass as track detector in a thermochromatographic column used in experiments to study the chemical properties of superheavy elements, it is necessary for the glass to retain the ability to register fragments of nuclear fission in a wide temperature range. Analysis of the registration properties of the glass detectors required calibration tests on a heavy ion accelerator, determination of optimal irradiation and etching modes, as well as thermal tests. We suggest that a possible reason for the change in the characteristics of the tracks during irradiation of heated samples of phosphate glass is a decrease in its viscosity or greater mobility of atoms at high temperature leading to the filling of vacancies resulting from the ion passage. The original method, based on the study of the rate of change in the size of etched tracks, allows the identification of the ion charge. In subsequent experiments, we intend to ascertain possible correlations between the track parameters and the ion energy, which will expand the potentials of the phosphate glass detectors for registering superheavy nuclei obtained at the JINR Factory of Superheavy Elements.

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