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PHYSICAL-MECHANICAL PROPERTIES OF γ-IRRADIATED SiC CERAMICS FOR RADIOACTIVE WASTES IMMOBILIZATION

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The interest in silicon carbide (SiC-based) ceramics and composites as matrix material for nuclear waste immobilization is grown up. Long-term chemical durability and radiation resistance of SiC are important factors for radionuclides immobilization. Advantages of SiC-based ceramics as structural materials in nuclear applications are the high-temperature properties, high density and reduced neutron activation. The use of radiation resistant materials is a strong requirement for safe and environmentally beneficial energy system. The SiC ceramics stability under irradiation for temperatures up to 1273 K is also very important for nuclear power applications. The SiC matrices doped by additives of Cr, Si were fabricated using High Speed Hot Pressing Method. Additives content was in the range from 0.5 to 3 wt %. Microstructural characteristics of silicon carbide ceramics were analyzed by X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), and infra-red spectroscopy (IR) methods. The results of microcracking under indentation conditions were revealed the lack of cracks in the SiC ceramics with Cr additives before and after irradiation process. In addition, it was demonstrated that samples of SiC with alloying additives Cr and Si possess high mechanical parameters under γ-irradiation process. The strength of ceramics increases with the uniform and fine-grained structure formation. The modification of phase composition and mechanical properties of the SiC ceramics with Cr and Si additives under γ-irradiation were analyzed for further development of radiation resistant and matrix materials for radioactive wastes immobilization.
KEYWORDS: silicon carbide, irradiation, physical-mechanical properties, nuclear waste immobilization, microstructural characteristics.

ФІЗИКО-МЕХАНІЧНІ ВЛАСТИВОСТІ γ-ОПРОМІНЕННОЇ SiC КЕРАМІКИ ДЛЯ ІММОБІЛІЗАЦІЇ РАДІОАКТИВНИХ ВІДХОДІВ

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Інтерес до карбіду кремнію, кераміки та композитів на основі SiC як матричного матеріалу для іммобілізації ядерних відходів зростає. Довготривала хімічна та радіаційна стійкість SiC є важливими факторами для іммобілізації радіонуклідів. Перевагами кераміки на основі SiC як конструкційних матеріалів для застосування у ядерної енергетиці є високотемпературні властивості, висока щільність та зменшена активація нейтронів. Використання радіаційностійких матеріалів є суворою вимогою до функціонування безпечної та екологічно чистої енергетичної системи. Структурна стабільність кераміки при опроміненні до температури 1273 К також дуже важлива для застосування в ядерної енергетиці. Матриці SiC, леговані добавками Cr та Si, були виготовлені методом високошвидкісного гарячого пресування. Вміст добавок складав від 0,5 до 3 % мас. Мікроструктурні характеристики кераміки карбіду кремнію були проаналізовані методом рентгенівської дифракції (XRD), сканувальної електронної мікроскопії (SEM), енерго-дисперсійної рентгенівської спектроскопії (EDX) та методів інфрачервоної спектроскопії (ІЧ). Результати тріщиностійкості в умовах навантаження виявили незначну кількість тріщин у кераміці SiC з добавками Cr до і після процесу опромінення. Крім того, було продемонстровано, що зразки SiC з легуючими добавками Cr і Si мають високі механічні параметри при γ-опроміненні. Міцність кераміки зростає з утворенням однорідної та дрібнозернистої структури. Проаналізовано модифікацію фазового складу та механічних властивостей кераміки SiC з добавками Cr і Si при γ-опроміненні для подальшого розвитку матеріалів, що стійкі до випромінювання, та матриць для іммобілізації радіоактивних відходів.
КЛЮЧОВІ СЛОВА: карбід кремнію, опромінення, фізико-механічні властивості, іммобілізація ядерних відходів, мікроструктурні характеристики.

ФИЗИКО-МЕХАНИЧЕСКИЕ СВОЙСТВА γ-ОБЛУЧЕННОЙ SiC КЕРАМИКИ ДЛЯ ИММОБИЛИЗАЦИИ РАДИОАКТИВНЫХ ОТХОДОВ

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Интерес к керамике из карбида кремния (на основе SiC) и композитам в качестве матричного материала для иммобилизации ядерных отходов повышается. Долгосрочная химическая и радиационная стойкость SiC являются важными факторами для

иммобилизации радионуклидов. Преимуществами керамики на основе SiC в качестве конструкционных материалов для применения в ядерной энергетике являются высокотемпературные свойства, высокая плотность и уменьшенная активация нейтронов. Использование радиационностойких материалов является строгим требованием к функционированию безопасной и экологически чистой энергетической системы. Структурная стабильность керамики при облучении до температуры 1273 К, также очень важна для применения в ядерной энергетике. Матрицы SiC, легированные добавками Cr и Si, были изготовлены с использованием метода высокоскоростного горячего прессования. Содержание добавок находилось в диапазоне от 0,5 до 3 мас.%. Микроструктурные характеристики керамики из карбида кремния были проанализированы с помощью рентгеновской дифракции (XRD), сканирующей электронной микроскопии (SEM), энергодисперсной рентгеновской спектроскопии (EDX) и инфракрасной спектроскопии (ИК). Результаты трещиностойкости в условиях индентирования показали небольшое количество трещин в керамике SiC с добавками Cr до и после процесса облучения. Кроме того, было продемонстрировано, что образцы SiC с легирующими добавками Cr и Si имеют высокие механические параметры при γ-облучении. Прочность керамики растет с образованием однородной и мелкозернистой структуры. Проанализирована модификация фазового состава и механических свойств SiC-керамики с добавками Cr и Si при γ-облучении для дальнейшего развития радиационно-стойких материалов и матриц для иммобилизации радиоактивных отходов.

КЛЮЧЕВЫЕ СЛОВА: карбид кремния, облучение, физико-механические свойства, иммобилизация ядерных отходов, микроструктурные характеристики.

SiC-based ceramics and composites possess superior properties, such as high corrosion resistance, excellent chemical and thermal shock stability, high mechanical strength, thermal expansion and small neutron absorption cross-section [1–3]. Such properties are very attractive for further operating under extreme environmental conditions, in particular, as matrix material for water-cooled and fusion power reactors, for the nuclear fuel as a cladding material, and us protective form for radioactive waste immobilization [4-6].

Advantages of SiC-based ceramics and composites as structural materials in fusion applications are the high-temperature properties and stability, density and reduced neutron activation. The use of activation materials (LAMs) is a strong requirement for safe and environmentally beneficial energy system. The SiC ceramics dimensional stability under irradiation for temperatures up to 1273 K [7] is also very important for fusion power applications.

The interest in SiC matrix material for nuclear waste immobilization is grown up. SiC ceramics and composites have been proposed as an inert matrix for the burning or transmutation of long-lived fission products and minor actinides from fuel cycle reprocessing. Nuclear waste is highly radioactive and toxic for hundreds of thousands of years. The basis of light water reactor (LWRs) fuel cycle is storing of processing fuels into geologically stable repositories in specially designed canisters, where the radioactive materials can decay for long times, with minimal release to the environment. [8,9]. In all proposed deep-burn fuel cycle involving tristructural isotropic (TRISO) fuel, where SiC is the primary barrier to fission product release, high burn up reduces the need for reprocessing, and the high-burn up TRISO fuel could be placed directly in a geologic repository. TRISO fuels, incorporated in a dense SiC matrix has been proposed for both transuranic waste destruction and as a potential fuel for light water reactors [9].

The long-term chemical durability and radiation resistance of SiC are important factors for radionuclides immobilization. Due to the low induced activation, SiC ceramics were proposed as structural components for long-term geologic disposal of the low-level or intermediate level waste. In operating conditions, energetic particles may alter SiC retention capability for the fission products, producing atomic displacements. The resulting microstructural changes may eventually modify the properties of the SiC ceramics [10].

The oxidation behaviour of SiC in water vapour containing environments was examined in details [11]. SiC exhibits exceptional oxidation resistance in water vapour up to 1700°C. The oxidation of SiC has been studied in a variety of gaseous environments. Of particular interest for the nuclear industry is oxidation resistance in a water/steam environment [12]. It has been shown, that radiation significantly affects important material properties [13]. Recent studies on the corrosion rates of unirradiated and irradiated SiC in different simulated ground waters over a range of temperatures revealed no significant long-term effect of irradiation on corrosion behaviour [14]. The mechanisms and rates of corrosion for SiC in different aqueous media under conditions relevant to geologic repositories have also been reported [15], and indicated that the radiotoxicity of spent TRISO fuel is at least four orders of magnitude lower than for current spent light water reactor fuel.

Radiation effects induced by neutrons at high temperature were widely studied. Microstructural evolution over a large range of temperatures and swelling [16] were the main investigated topics. However, the gamma radiation influence on SiC ceramic is of great interest due to an action of gamma radiation as main source of the effect on the protective matrices during wastes immobilization process.

At the process of SiC exposure to high-temperature, and extreme radiation environments, microstructural evolution and micro cracking may occur. During SiC matrix exposure to beta and gamma radiation from fission product decay irradiation-induced amorphization took place. The critical temperature for amorphization from high flux electron beams was about 300-340 K depending on SiC structure modification [17-19].

The retention capability of SiC for the typical fission product, particularly when it is submitted to irradiation-induced structural modifications was studied. The evolution of a disorder with the ion fluence and the cumulative dose in dpa (‘displacements per atom’) was calculated [20]. It appears that total disorder (i.e., amorphization) is achieved at 0.25 dpa. In previous studies, the structural modifications induced by the implantation of a typical fission product iodine

(I) into silicon carbide were examined. This result is in good agreement with previous investigations using other ion species [21] and demonstrate that SiC is a very easily amorphizable material by low energy ion implantation at room temperature while it can hardly be disordered by ion bombardment at elevated temperatures. For inert matrix fuels, SiC ceramics demonstrate phase compounds with reasonably high radiation stability to high temperatures for different fissile species [22].

The good corrosion and radiation resistances exhibited by the carbides are due to the unique combination of high hardness and fracture toughness. The bulk mechanical properties and stability of carbides are strongly affected by their composition and microstructural parameters [23, 24]. The different additives, such as oxides, Si, B, and transitional metals of IV-VI subgroups, as sintering activators, were added to improve the mechanical properties of SiC ceramics. The effect of additives on the ceramic sintering mechanism relates to its content and uniformity. The uniform distribution of additives is beneficial for improving the densification and microstructure formation during the sintering procedure [25-28].

The aim of the present study was the studying the influence of γ-irradiation on surface and physic-mechanical properties of the SiC ceramics modified by additives of Cr and Si.

MATERIALS AND METHODS

The highly dispersed powders were used as alloying additives for producing ceramics based on silicon carbide (SiC): SiC powders of the grade 440 NDP (Superior Graphite Co) with a predominant particle size 0.44 μm were chosen as primary material, and powders of Si, and Cr, with a predominant particle size <3 μm as additives. Mixing of the initial powders was carried out in a planetary mono-mill "Pulverisette 6" (Germany) in isopropyl alcohol medium with a rotation speed of 300 rpm during 3 hours.

Forming and sintering of the samples were made by the method of high-speed hot pressing in vacuum in a graphite die. The equipment was developed in NSC KIPT in the framework of joint collaboration project (STCU P-154) with Argon National Laboratory and Superior Graphite Co (USA). The optimum process parameters were previously determined and described [29]: sintering temperature 2050 °C, pressure 40 MPa and holding time 30 minutes. The samples size was 25×25×4 mm. Finishing surface treatment and polishing of SiC ceramics were made by series of diamond pastes graded from 28/20, 7/5, 5/3, 3/2. As a result of surface polishing with low rates diamond pastes treatment, 25-75 μm of defect surface layers were removed to a high degree of mechanical tests accuracy. The open porosity and density of the samples were determined by hydrostatic weighing method.

The diffractometry was carried out using a DRON-4-07 X-ray diffractometer in copper Cu-Kα radiation using a Ni selectively absorbing β-filter. The diffracted radiation was detected by a scintillation detector.

The absorption spectra in the IR range were recorded by IR spectrophotometer IRS-29 (LOMO) in the KIPT NSU. The spectra detection was made in the spectral range 4000 – 400 cm^{-1} (mean infra red area).

SiC samples were irradiated by bremsstrahlung obtained in Electron Linear Accelerator (ElA) with beam energy up to 10 Mev, current 800 mA, dose rate 1.1 kGy / h up to dose 10 kGy. To realize the conditions of external γ-irradiation effect of the SiC specimens, the bremsstrahlung of the ELA was used. Irradiation scheme is shown on Fig.1.

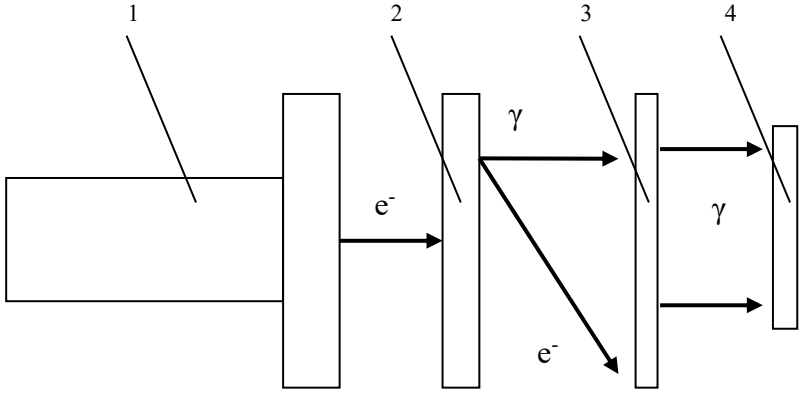


Fig. 1. Scheme of γ-irradiation in electron accelerator:
e^- – electrons, 1 – electron accelerator, 2 – Ta-converter,
3 – filter for electrons and neutrons, 4 – SiC specimen

The hardness tests of SiC ceramic samples before and after γ-irradiation process were made on a AMH-3 microhardometer by indentation of a four-sided 136-degree Vickers diamond pyramid. The optimal load value was chosen: 9.8 N. The hardness calculations were carried out according to the standard procedure with the measurement of the diagonals of the print:

$$H_V = P/d^2 \quad (1)$$

where H is hardness, P is the load on the indenter, d^2 is the square of the diagonals of the indentor's print.

The measurements of fracture toughness of ceramic carbide are complicated because of high brittleness of these materials. Estimation of fracture toughness values was made at 9.8 N load conditions. The equations of fracture toughness of ceramic brittle materials, which are in a good agreement with experimental data were formulated by Evans, Charles and Wilshaw [30] and by Niihara [31]. Equations for fracture toughness coefficient (K_{1C}) calculation were obtained by Niihara semi empiric dependence, commonly used for brittle ceramics [31]:

$$K_{1c}=(0{,}035H_v\, a)\,(E\phi/H_v)^{2/5} / \phi\, L^{0.5} \quad (2)$$

where a is the half-diagonal of the indentor's print, Hv is the hardness of the material, and L is the length of the radial crack, E is Young modulus, ϕ – is the constraint factor (≈ 3)

The microstructure and morphology of the samples were studied by JEM-700F scanning electron microscope. The Energy-dispersive X-ray spectroscopy (EDS) method with a high-energy electron beam was used for determination of the elements and distribution in the SiC-Si and SiC-Cr samples.

RESULTS AND DISCUSSION

The physical properties and phase composition of SiC ceramic samples with/without alloying additives obtained at the same technological parameters as previously reported [28] were presented in Table 1.

Table 1.

Properties and crystalline phases of the SiC samples

Ceramic composition	SiC	SiC + 0.5%Cr + 0.15%C	SiC + 1.0%Si + 0.3%C
Open porosity, %	0	0-1	0-1
Density g/cm^3	3.19	3.16	3.18
Relative density, ρ, %	99.4± 0.75	97.8 ± 0.75	99.7 ± 0.75
Phase composition	SiC-6H 99% C-1%	SiC-6H-95.5% SiC-4H-2.8% C – 1.7 %	SiC-6H-83.8% SiC-4H-16.2%

Analysis of the data given in Table 1 indicates that a slight decrease in the relative density is observed in SiC ceramic samples with alloying additives: up to 97.8% (Cr).

X-ray diffraction analysis of the samples revealed the presence of one phase α-SiC of polytype – SiC-6H with hexagonal lattice, whereas the introduction of the alloying additives leads to structural changes and the formation of a second α-SiC polytype of silicon carbide – SiC-4H in different weight contents (Table 1, Fig. 2).

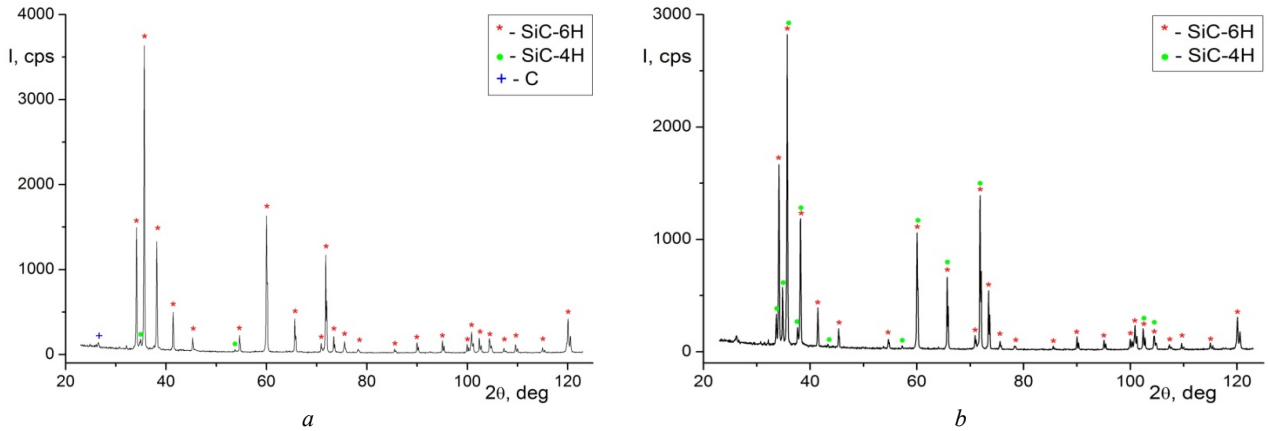
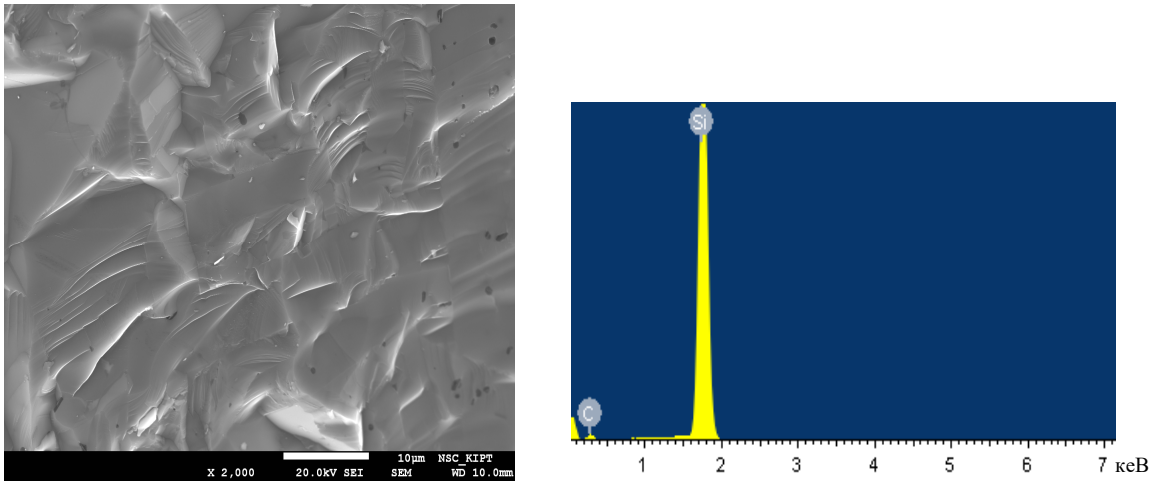


Fig.2. XRD patterns of the samples:
a – SiC + 0.5%Cr + 0.15%C, b – SiC + 1.0%Si + 0.3%C

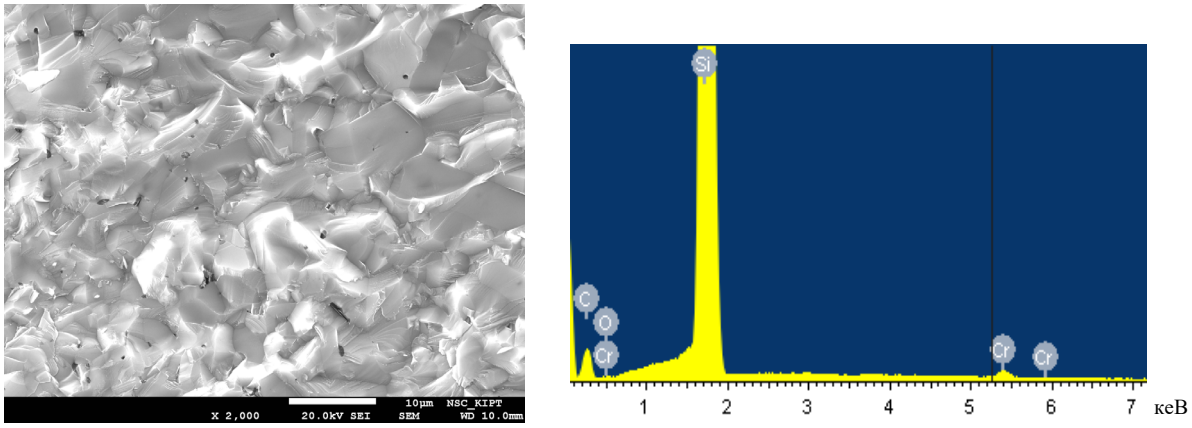
Electron microscope images of the fractured cross-sections of the samples SiC + 1.0% Si + 0.3% C and SiC + 0.5% Cr + 0.15% C were presented (Fig. 3,4). The structure of SiC sample with the Si additives demonstrates a very dense and uniformly fine-grained structure (99.7% of the theoretical values). The grain sizes were in the range 0.3-2.2 μm with a predominant grain size of 1.5 μm.



a b

Fig. 3. SEM images (a) and EDX spectra (b) of SiC + 1.0%Si + 0.3%C sample

Analysis of SEM/EDX results for SiC + 0.5%Cr + 0.15%C sample demonstrate that Cr adding leads to inclusions formation, probably of Cr_nC_x chromium carbide types. According to small amount of Cr, the presence of chromium carbide phase formation was not detected on XRD patterns of the ceramic samples (Fig. 4a).



a b

Fig. 4. SEM images (a) and EDX spectra (b) of SiC + 0.5%Cr + 0.15%C sample

The main approach to enhance the mechanical properties and fracture toughness is sintering of materials with dense and uniform grain microstructure. The strength of ceramics increases with the decrease of grain size and porosity. The evolution of phase composition and mechanical properties of SiC ceramic under γ-irradiation and irradiation-induced structural modifications was studied.

The hardness tests of SiC ceramic samples were made by indentation under load condition 9.8 N before and after irradiation process. Cracks appear on all samples, except the sample with the Cr additives before and after dose γ-irradiation (Fig.5 a, b, e). This fact indicates that the crack resistance and fracture toughness coefficient were not principally changed before and after irradiation process, which is also confirmed by the calculations carried out according to formula (2).

Table 2 shows the values for hardness and fracture toughness calculations before and after γ-irradiation process. As can be seen from the obtained data, hardness and fracture toughness values are not principally changed for both samples with additive Cr and Si. SiC samples without additives produced by High-Speed Hot Pressing Method show high density and microhardness parameters. The fracture toughness parameters demonstrate the values from K_{1c} =4.5 MPa·$m^{1/2}$ – 4.2 MPa·$m^{1/2}$ for SiC ceramic samples to K_{1c} = 5.6 MPa·$m^{1/2}$ – 5.4 MPa·$m^{1/2}$ for SiC samples with Cr additives, and K_{1c} = 4.4 MPa·$m^{1/2}$ – 4.2 MPa·$m^{1/2}$ for SiC samples with Si additives, before and after γ-irradiation process correspondently.

Previously was reported, that dense and uniform SiC ceramics demonstrate high mechanical parameters: micro hardness 24 GPa [32, 33] and fracture toughness coefficient K_{1c} = 5 MPa·$m^{1/2}$·[32]. At present study, SiC ceramic samples with Si, Cr and without additives also show high mechanical parameters: micro hardness 29.8 GPa and fracture toughness coefficient K_{1c} =4.5 MPa·$m^{1/2}$. Furthermore, a dose γ-irradiation process did not significantly change the

mechanical properties of SiC ceramic with both Si and Cr additives, only slight decrease of fracture toughness coefficient up to 5.4 MPa·m$^{1/2}$ and 4.2 MPa·m$^{1/2}$ was detected.

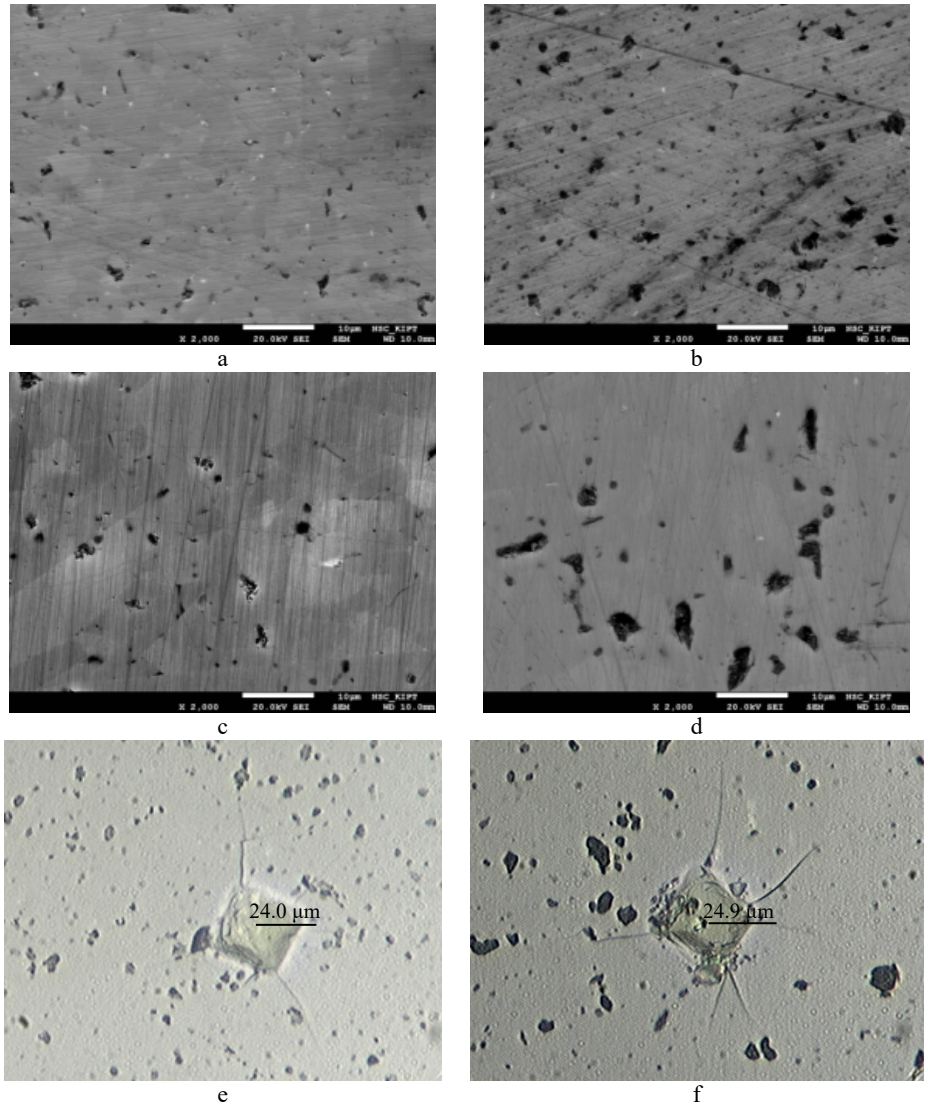


Fig. 5. Images of the ceramic surface before(a, c) and (b, d) after γ-irradiation process and indentor's print (e, f) after irradiation:
a, b, e – SiC + 0.5 % Cr + 0.15 % C; c, d, f – SiC + 1.0%Si + 0.3% C

Table 2.

Hardness and fracture toughness

Ceramic material	Load P, N	Hardness HV, GPa *before / after γ-irradiation process*	Fracture toughness K_{1c}, MPa·m $^{1/2}$ *before / after γ-irradiation process*
SiC	9.81	29.8 / 29.8	4.5 / 4.2
SiC + 0.5 % Cr + 0.15 % C	9.81	33.4 / 32.2	5.6 / 5.4
SiC + 1.0 % Si + 0.3 % C	9.81	30.9 / 29.9	4.4 / 4.2

IR spectrum of SiC samples without additives produced by High-Speed Hot Pressing Method demonstrates peaks of 580, 650, 820, 935 and 990 cm^{-1}, related to the Si-C vibrations at the silicon carbide structure with hexagonal crystal structure. In addition, the intensive absorption bands were detected correlated to stretching and deformation vibrations of

Si-O in tetrahedral SiO_4 quartz structure: 460, 510, 690, 780, 800, 1080 and 1160 cm^{-1} (Fig. 6, curve 1) [34, 35]. The SiO_2 formation can occur as a result of sample oxidation during the heat treatment process. The absorbed water band of 3450 cm^{-1} also was observed.

After γ-irradiation process up to 10^4 Gy dose any visible changes at the absorbed spectra were not detected. An intensity of band at the vibration range of O-H (3430 cm^{-1}) was slightly decreased. Thus, γ-irradiation process up to 10^4 Gy dose had no effect on the phase composition and crystal structure of SiC ceramic samples.

An analysis of IR absorption spectra of SiC samples before and after irradiation process demonstrated no principal distinctions between samples with/without 0.5 % Cr и Si additives.

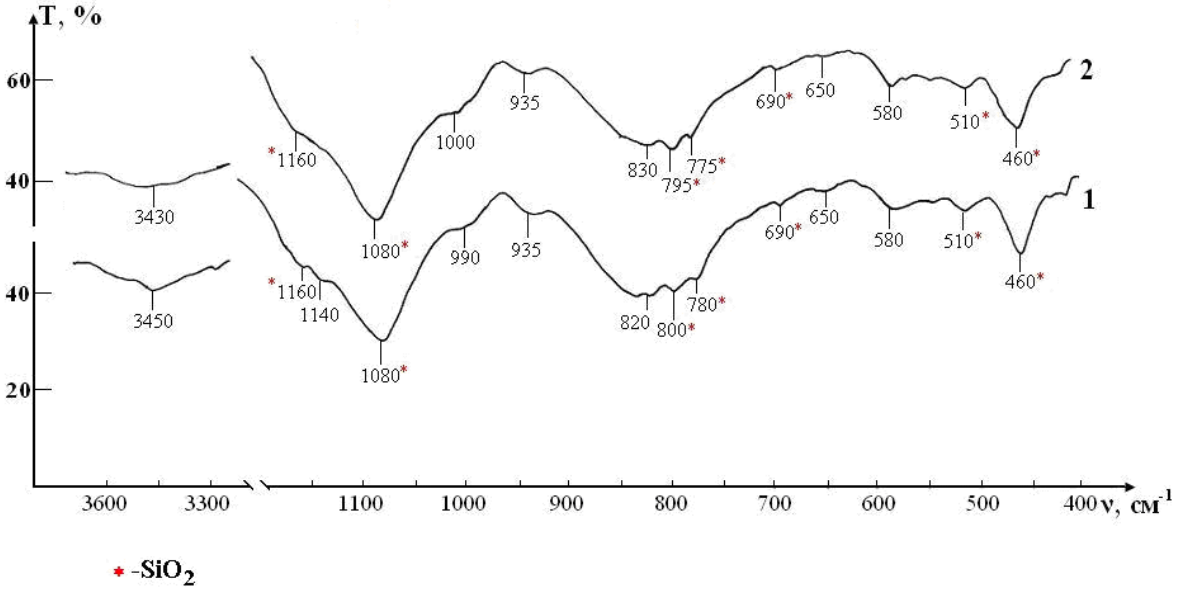


Fig. 6. IR absorption spectra of SiC samples after sintering:
curve 1 – initial SiC, curve 2 – SiC after γ-irradiation process up to 10^4 Gy dose

CONCLUSIONS

Sintering process by High-Speed Hot Pressing Method leads to the fine-grained structure formation and increase of the fracture toughness of ceramics. The hardness and crack resistance of SiC ceramics with Cr and Si additives were not principally changed under γ-irradiation process. The results of microcracking under indentation conditions were revealed the lack of cracks in the SiC ceramics with Cr additives before and after irradiation process. In addition, it was demonstrated that samples of SiC with alloying additives Cr and Si possess high mechanical parameters under γ-irradiation process: microhardness – 32.2 – 29.9 GPa, fracture toughness coefficient K_{1C} = 5.4 – 4.2 MPa·$m^{1/2}$, respectively. The strength of ceramics increases with the uniform and fine-grained structure formation. The analysis of IR absorption spectra demonstrates that any phase composition and crystal structure changes were not observed after γ-irradiation process. The samples sintered with Cr and Si additives demonstrate good radiance resistance and high mechanical properties under γ-irradiation up to absorption dose of 10^4 Gy, which confirms the advantages of alloying additives using. SiC ceramics with Cr and Si additives are promising candidates for further nuclear applications as radiation resistant materials to manufacture protective matrices for radioactive wastes immobilization.

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