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EDP STUDY OF NANOCRYSTALLINE SILICON CARBIDE (3C-SiC) UNDER THE NEUTRON IRRADIATION

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Abstract: In this present work nanocrystalline silicon carbide (3C-SiC) has been irradiated with neutron flux (~$2x10^{13}n\cdot cm^{-2}s^{-1}$) up to 20 hour at different periods. Electron diffraction patterns (EDP) investigation of nanocrystalline 3C-SiC particles (~18nm) is comparatively analyzed before and after neutron irradiation. The effect of irradiation on the crystal structure of the nanomaterial was studied by selected area electron diffraction (SAED) and EDP analysis. Amorphous transformation effects of neutron irradiation on the nanocrystalline silicon carbide (3C-SiC) has been studied by EDP method.

***Keywords*:** nanocrystalline 3C-SiC, nanoparticles, neutron irradiation, TEM, FE-SEM

1. Introduction

Over the past few years, obtaining new materials or reprocessing current materials in the nuclear and cosmic technologies have been at the focus of various studies worldwide. SiC is an attractive material that can be applied in nuclear technologies due to the physical and chemical properties [1-5]. High temperature resistance, high-perfection structure, mechanical stability, high oxidation resistance increase the application potential of SiC as a nuclear material [2-4]. The combination of perfect mechanical and functional properties is the basis of application of SiC as a semiconductor in modern electronics. It’s known that, SiC has more than 200 polytypes. Cubic (3C - SiC) and hexagonal (4H and 6H) phase polytypes are most widely used between them in electronics system. Cubic modification nano SiC has more application potential in microelectronics due to the wide band gap (2.26eV), thermal and electrical properties [6-11]. Therefore, nano 3C-SiC (also known as β-SiC) particles with cubic modification have been used in all experiments in the given work.

The study of neutron irradiation effects to the physical and chemical properties of SiC, which is widely applied in fission - fusion reactor and electronics systems, is very important issue. The neutron flux influence on the lattice structureof 3C-SiC has been studied in the present work. Up to the present, in the large number of papers, it has been studied the effects of ionizing irradiation on SiC [12-19]. But the radiation effects on the lattice structureof these type materials have not been fully studied. Neutron irradiation formed defects and its lead to important changes in electro-physical properties of the samples [12-19]. First of all, *let* us *briefly consider* the defects formed in nanoparticles after neutron irradiation. 3C-SiC nanoparticles consist of Si and C atoms with cubic modification and high-energy recoils can be observed in primary knock-on atoms (PKA) within the neutron flux influence on lattice atoms [20-28]. Effects of ionizing radiation on nanocrystalline 3C-SiC particlesare spacious studied [24-28]. Energy exchange between PKA and other neighbor atoms, point defects or clusters are generated and they are the basis of fundamental defects. The defects formed under the influence of neutron flux, it can be migrating and they are a real place for storage any charge [20-28].

2. Experimental

Cubic modification silicon carbide (3C-SiC) nanoparticles have 120 $m^2 \cdot g^{-1}$ specific surface area (SSA), 18nm particles size, 99+% purity and 0.03 $g \cdot cm^{-3}$(real density 3.216 $g \cdot cm^{-3}$) density is used at the present experiments (US Research Nanomaterials, Inc., TX, USA). The samples have been irradiated at full power mode (250 kW) by neutron flux ($2x10^{13} n \cdot cm^{-2} s^{-1}$) in central channel (channel A1) of TRIGA Mark II light water pool type research reactor in "Reactor Centre" of Institute Jozef Stefan (IJS) in the Ljubljana, Slovenia. The parameters of neutron flux at full power mode in the central channel are follows: $5.107x10^{12} n \cdot cm^{-2} s^{-1}$ (1±0.0008, E_n< 625eV) for thermal neutrons, $6.502x10^{12} n \cdot cm^{-2} s^{-1}$ (1±0.0008, E_n ~ 625eV ÷ 0.1MeV) for epithermal neutrons, $7.585x10^{12} n \cdot cm^{-2} s^{-1}$ (1±0.0007, E_n> 0.1 MeV) for fast neutrons and finally for all neutrons in central channel the flux density is $1.920x10^{13} n \cdot cm^{-2} s^{-1}$ (1±0.0005) [29-35].

Nano SiC powder filled to aluminium containers which have high purity in a special condition have been made appropriate to the channels of the reactor. At first this experimental sample has been irradiated for 5 minutes and activity analysis have been conducted. Then the other sample has been continuously irradiated for 20 hours by neutron flux ($2x10^{13} n \cdot cm^{-2} s^{-1}$) at full power (250kW) in channel A1. The EDP analysis of non-irradiated and neutron irradiated nanocrystalline SiC powder were observed by transmission electron microscope (TEM, Jeol JEM-2010F, analytical transmission electron microscope is equipped with a scanning unit (ADF, BD STEM detectors), EDXS and EELS). To prepare the TEM specimen, the powdered samples named "SiC (0h)" and "SiC (20h)" were dispersed in acetone solution by ultrasonication. The solution was dropped on carbon coated Cu-carbon mesh, dried in vacuum dryer and transferred to TEM for further EDP analyses. SAED and EDP analysis of the samples were carried out with up to 200 kV accelerating voltage before and after neutron irradiation.

3. Results and Discussion

SAED analysis were conducted on small areas. The corresponding electron diffraction patterns (EDP), recorded over larger area containing particles, show well-defined diffraction rings composed from diffraction spots, indicating nanocrystalline nature of the particles. (Fig.1). EDP ring patterns were analyzed in Gatan's Digital Micrograph software package.

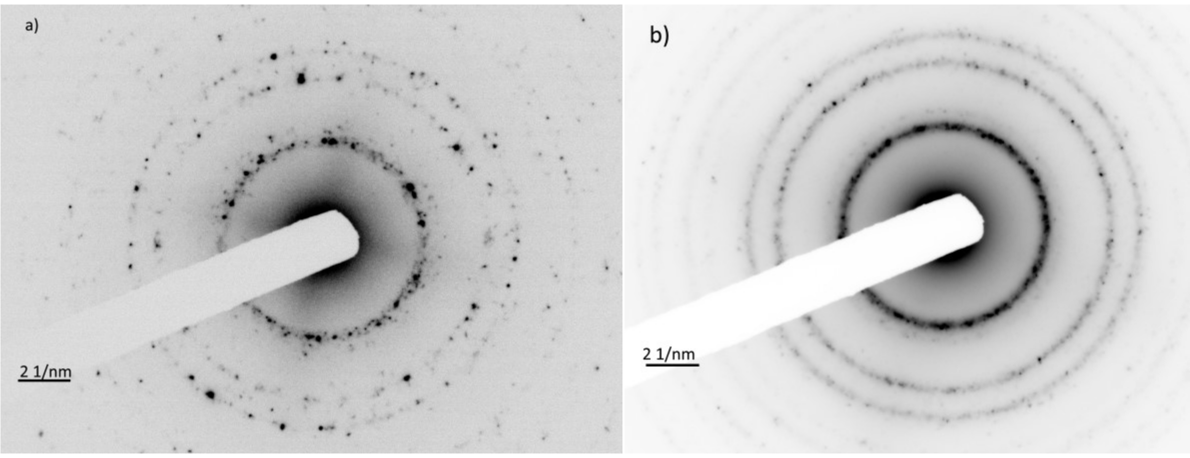

Fig. 1. EDP of nanocrystalline 3C-SiC particles before (a) and after (b) neutron radiation (recorded on agglomerated particles).

In both diffraction patterns (Fig.1) we performed rotational average (RA) analysis, which improves the resolution of the intensity profile. Such obtained patterns were merged together (Fig.2) and detailed comparison (Fig.3) show no significant difference in the position of the diffraction peaks, implying there was no influence of the neutron bombardment on the 3C-SiC crystal structure. Simultaneously, it can be concluded that some of the particles of irradiated sample may exhibit somewhat thicker amorphous layer surrounding the nanoparticle, however more analytical work (larger statistics) is needed to confirm this. It is assumed that this amorphous layer can be either the result of displacement of some lattice atoms or oxidation of some atoms on the surface as a result of neutron irradiation.

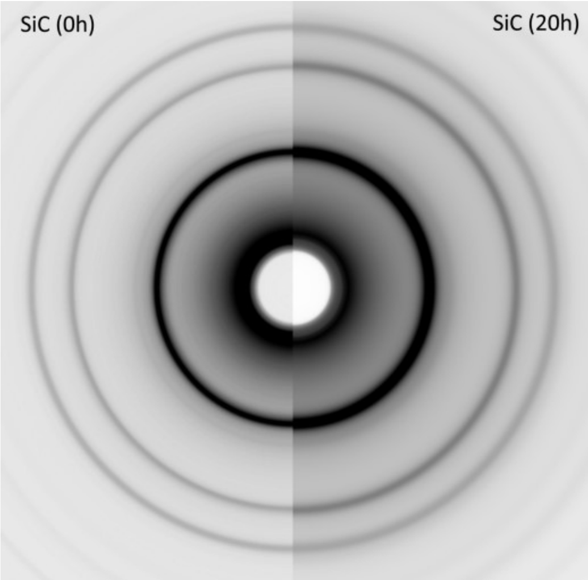

Fig. 2. Comparison of rotation average (RA) of EDPs between 3C-SiC (0h) and 3C-SiC (20h).

There were not any changes in comparative EDP analysis. And it allows us to say that radiation with neutron flux affects crystal structure of samples little or it doesn't affect. Moreover, comparative analysis of EDP d-values were conducted (Fig. 3).

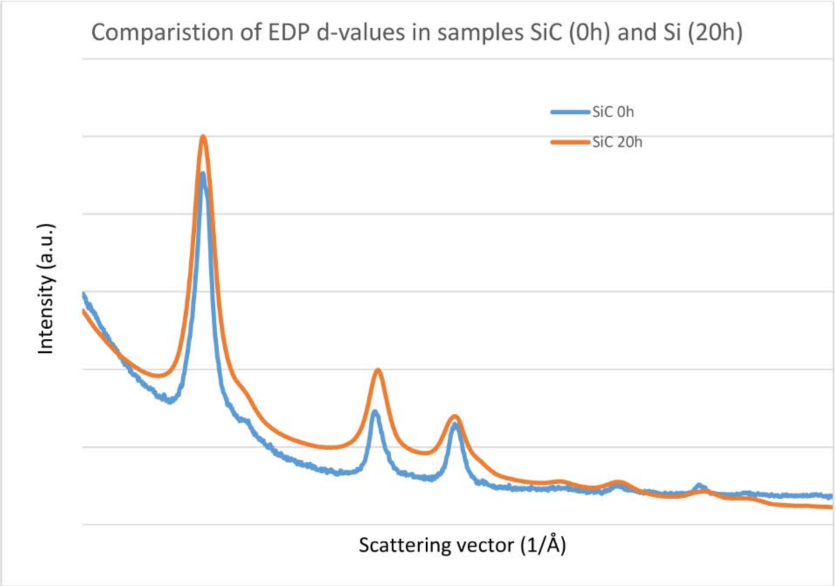

Fig. 3. Scattering vector (1/Å) of samples 3C-SiC (0h) and 3C-SiC (20h).

In the present work no difference at the peak positions between irradiated (orange) and nonirradiated samples (blue) (Fig 3), which implies that neutron bombardment did not influenced the crystal structure of the 3C-SiC nanoparticles. And this once again approves the results obtained from comparative EDP analysis. However, one should consider that the measurements of d values using electron diffraction is inferior to the X-ray powder diffractions.

4. Conclusions

It is clear from EDP analysis that nanocrystalline 3C-SiC particleshas littleamorphous transformation after neutron irradiation. Probably, after neutron irradiation there is thicker amorphous layer surrounding the 3C-SiC nanoparticles. Amorphous layer of the surfaces of nanoparticles can cause a greater or lesser agglomeration degree. As a result of EDP analysis it has been found out that, by a majority, neutron irradiation doesn't affectcrystal structure of 3C-SiC nanoparticles(excluding small amorphous transformation).

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ИССЛЕДОВАНИЕ EDP КАРБИДА НАНОКРИСТАЛЛИЧЕСКОГО КРЕМНИЯ (3C-SiC) ПРИ НЕЙТРОННОМ ОБЛУЧЕНИИ

Э.М. Гусейнов

Резюме: В настоящей работе нанокристаллический карбид кремния (3C-SiC) облучен нейтронным потоком ($\sim 2\mathrm{x}10^{13}\mathrm{n}\cdot\mathrm{cm}^{-2}\mathrm{s}^{-1}$) до 20 часов в разные периоды. Сравнительно проанализировано электронно-дифрактограммовое исследование (EDP) нанокристаллических частиц 3C-SiC (~ 18 нм) до и после нейтронного облучения. Влияние облучения на кристаллическую структуру наноматериала изучалось методом электронной дифракции области (SAED) и EDP. Аморфные трансформационные эффекты облучения нейтронами на карбиде нанокристаллического кремния (3C-SiC) изучены методом EDP.

Ключевые слова: нанокристаллический карбид кремния (3C-SiC), наночастицы, нейтронное облучение, ТЭМ

NEYTRONLARLA ŞÜALANMANIN TƏSİRİ ALTINDA NANOKRİSTALLİK SİLİSİUM KARBİDİN (3C-SiC) EDP TƏDQİQİ

E.M. Hüseynov

Xülasə: Təqdim olunan işdə nanokristallik silisium karbid (3C-SiC) neytron seli ilə ($\sim 2\mathrm{x}10^{13}\mathrm{n}\cdot\mathrm{cm}^{-2}\mathrm{s}^{-1}$) fərqli müddətlərdə 20 saata qədər şüalandırılmışdır. Nanokristallik 3C-SiC hissəcikləri (~18nm) elektron difraksiyası ilə şüalanmadan öncə və sonra müqaisəli analiz edilmişdir. Nanomaterialın kristal strukturda şüalanma effektləri seçilmiş sahədən elektron difraksiyası və EDP üsulları ilə öyrənilmişdir. Neytronlarla şüalanma nəticəsində nanokristallik silisium karbidin (3C-SiC) amorflaşma çevrilmələri EDP metodu ilə tədqiq edilmişdir.

Açar sözlər: nanokristallik silisium karbidin (3C-SiC), nanohissəciklər, neytron irradiasiyası, TEM.