J MATER RES TECHNOL. 2016;5(3):213–218

Available online at www.sciencedirect.com

jmr&t
Journal of Materials Research and Technology
www.jmrt.com.br

Original Article

# TEM and SEM study of nano $SiO_2$ particles exposed to influence of neutron flux

*Elchin Huseynov*[a,*], *Adil Garibov*[a], *Ravan Mehdiyeva*[b]

[a] *Department of Nanotechnology and Radiation Material Science, National Nuclear Research Center, Baku, Azerbaijan*
[b] *Institute of Radiation Problems of Azerbaijan National Academy of Sciences, Baku, Azerbaijan*

ARTICLE INFO

*Article history:*
Received 23 April 2015
Accepted 1 November 2015
Available online 11 December 2015

*Keywords:*
Nano $SiO_2$
TEM
SEM
Neutron irradiation
Nanopowder

ABSTRACT

Before and after neutron irradiation, in order to identify the “adhesion” in silica nanoparticles, analyses have been conducted on transmission electron microscope (TEM) at small nano dimensions. Simultaneously, at relatively larger nano dimensions, the surfaces of the samples were observed by the scanning electron microscope (SEM). Moreover, analyses of the samples with SAED (selected area electron diffraction) technology on TEM device used for determining the structure of the nanomaterial. From TEM analyses, it has been found that little “adhesion” is observed at small dimensions (maximum 70 nm) under the influence of neutron irradiation and this “adhesion” directly influences the electrophysical properties of nanomaterials.



**Take home message**

- Silica nanoparticles analyzed with TEM, SAED and SEM.
- Study neutron flux effects on silica nanoparticles.
- After radiation determined “adhesion” of nanoparticles.

## 1. Introduction

Recently, nanomaterials have been widely used in computer, telephone, satellite technology, different types of detectors and other various fields of industry for their unique features [1–4]. Size effects of inorganic nanoparticles increase their application possibilities in nano dimensions [5,6]. Nano $SiO_2$ compound used in the experiment has a wide application

* *Corresponding author*.
E-mail addresses: e.huseynov@mntm.az, elchin.huse@yahoo.com, elchin.huse@gmail.com (E. Huseynov).
http://dx.doi.org/10.1016/j.jmrt.2015.11.001

fields in space electronics and nuclear technology in macro and micro sizes. Recently, the application of these nano-size materials in space and nuclear technologies are very topical, therefore we have studied stability of these materials after neutron irradiation. As mentioned before, the size effect of nanomaterials influence to their application possibilities. Therefore, on the SEM and TEM devices we have studied the "adhesion" process, which can be formed in big and small size nanomaterials after influence of neutron flux. Moreover, we have determined nanoparticles sizes in the local state by TEM device and the nature of nanoparticles by selected area electron diffraction (SAED) technology. The samples used within the experiments have been irradiated by neutron flux ($2 \times 10^{13}$ n cm$^{-2}$ s$^{-1}$) in the central channel (Channel A1) of the TRIGA Mark II light water pool-type research reactor at full power (250 kW) in Jozef Stefan Institute (JSI). TEM analyses (images and SAED) have been carried out on "Jeol JEM-2100" device and SEM analyses on "Jeol JSM-7600F" device for all samples (initial circumstance and after continuously 5, 10, 15 and 20 h irradiated by neutrons), in the "Department of Nanostructured Materials – K7" at JSI in Ljubljana, Slovenia.

TEM equipment has been used for observing nanomaterials with size smaller than 100 nm and obtaining quantitative results [7–9]. The main reason, minimum 100 keV of the accelerated electrons energy on TEM devices and its increases several times the degree of sensitivity in small sizes of the TEM devices relatively to SEM devices. There are some problems for observation far background of the samples operating on the basis of high-energy electrons TEM devices. In this case, it is advisable to use SEM device operating with maximum 30–50 keV energy electron flux. Taking into account these properties for TEM and SEM devices, we have used both of the devices for completely observing nano $SiO_2$ samples. Thus, we have reviewed the images of $SiO_2$ nanoparticles at the relatively far background (SEM capabilities) and at small sizes (TEM capabilities), taking advantage of both devices.

## 2. Theoretical frameworks

In this section, we will review briefly and simply the dependence on external potential of the wave property of electron flux used on SEM and TEM technologies. For this reason, first of all, it should be mentioned the influence of wave properties on growing and resolution. The shortest distance between two points, which can be distinguished from each other, is called resolution. For example, the average resolution for the human eye is approximately 0.1–0.2 mm and the resolution in optic microscopes is much smaller than this value. In the general case, we can calculate the resolution of the microscope with the following equation: $\delta = (0.61\lambda/\mu \sin\beta)$ (here $\lambda$ – wavelength, $\mu$ – refraction index and $\beta$ – observation angle in zoom lens) [10].

We can say that, resolution of microscope dependence on the wavelength which directly used beam if we consider the refraction index and sinus of observation angle change very little in most cases. So, with an increase of wavelength, the numerical value of microscope resolution increases and thus its zoom decreases. For example, using a wavelength of 550 nm for optic microscopes in the best case, we will get at least 300 nm for its resolution and it does not allow us to see the distance of 0.2 nm (or small nanoparticles of which size is in nano order) between two atoms. During the SEM and TEM analyses, electron flux is used and this time wave properties of electron directly influence the device resolution. Thus, according to dual property of the particles we can write $\lambda = (h/p) = (hc/E)$ for de-Broglie wavelength of the electron. This equation indicates the wavelength of the $E$ energy (or impulse is $p$) electron. It should be mentioned that, it is obtained $\lambda = 1.22/E^{1/2}$ expression in accordance with the last relation, without consideration of relativistic effects in well-known de-Broglie's expression for wave property of the electron [10]. Here $E$ – energy of the electron, which is expressed with eV, $\lambda$ – de-Broglie wavelength expressed with nm. In the SEM and TEM devices, the electron moves in $V$ potential field and in this case, its kinetic energy ($eV$) is defined as $eV = (m_0 v^2/2)$. Making some simplicities in this equation, we can define the electron impulse as $p = m_0 v = (2m_0 eV)^{1/2}$. We will get the expression $\lambda = h/\sqrt{2m_0 eV}$ in simple case for its wavelength while moving in $V$ potential inside electron microscope. From the expression, it is seen that the potential of the field is inversely proportional to wavelength and by changing the potential at SEM or TEM device, we can control the wavelength. It should be mentioned that in previous expressions we have not considered the relativistic movement of electron but the electron moves relativistic in reality. The relativistic effect is usually observed on TEM devices, so on TEM devices the speed of electron at potentials more than 100 kV is higher than the half of light speed. Consequently, we can write the equation $\lambda = h/(\sqrt{2m_0 eV(1 + (eV/2m_0 c^2))})$ for relativistic state [10]. A bit more potential is required which is calculated with the initial equation at the relativistic state. If we consider the potential is 100 keV in the last equation, then we will find that the resolution for TEM is 0.0037 nm, and it allows us to observe nano and angstrom sizes at atomic level. It should be mentioned that on TEM devices it can be used more potential than SEM devices and that is why magnification capacity of TEM devices is larger than SEM devices.

## 3. Materials and methods

At the presented work, it has been investigated the transmission electron microscope (TEM) analyses and scanning electron microscope (SEM) images of $SiO_2$ nanoparticles before and after neutron irradiation. In this research we have chosen $SiO_2$ nanoparticles with 160 m$^2$ g$^{-1}$ specific surface area (SSA), 20 nm particle size and some parameters of the used sample has been studied [11–15]. Simultaneously, it has been carried out "selected area electron diffraction" (SAED) analyses of $SiO_2$ nanoparticles before and after neutron irradiation on TEM device. The samples have been irradiated by neutron flux ($2 \times 10^{13}$ n cm$^{-2}$ s$^{-1}$) in the central channel of the TRIGA Mark II research reactor at full power.

It is important to note that the JSI TRIGA reactor has been thoroughly characterized [16,17] and the computational model used for computational characterization has been thoroughly verified and validated against several experiments [18–21]. In addition, the model has been used to support