Monte Carlo modeling of cavity imaging in pure iron using back-scatter electron scanning microscopy

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ABSTRACT

Backscattered electrons (BSE) in a scanning electron microscope (SEM) can produce images of subsurface cavity distributions as a nondestructive characterization technique. Monte Carlo simulations were performed to understand the mechanism of void imaging and to identify key parameters in optimizing void resolution. The modeling explores an iron target of different thicknesses, electron beams of different energies, beam sizes, and scan pitch, evaluated for voids of different sizes and depths below the surface. The results show that the void image contrast is primarily caused by discontinuity of energy spectra of backscattered electrons, due to increased outward path lengths for those electrons which penetrate voids and are backscattered at deeper depths. Size resolution of voids at specific depths, and maximum detection depth of specific voids sizes are derived as a function of electron beam energy. The results are important for image optimization and data extraction.

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1. Introduction

Microstructural changes and mechanical property degradation of reactor structural alloys under neutron damage in reactors present large challenges for reactor performance and safety [1-3]. Since its first observation in 1967 [4], neutron-induced void swelling in stainless steels especially has been a subject of intensive research, as swelling leads to significant dimensional instabilities via swelling and irradiation creep, with concurrent changes in mechanical and physical properties and also in new modes of embrittlement [2-8]. Swelling and bubble formation (both hereafter referred to as cavities) induced by charged particle irradiation is also being increasingly studied as a surrogate for neutron irradiation [9-18]. Void swelling is caused by incomplete interstitial-vacancy recombination and biased defect-sink interactions during displacive irradiation by neutrons or charged particles, and its general dependence on damage dose defined in displacements per atom (dpa), dpa rate, temperature, and stress are relatively well known [1-3].

Characterization of cavities is primarily accomplished using transmission electron microscopy (TEM) which requires electron-transparent specimens of 50 nm to 200 nm thickness, prepared using either electrochemical polishing, ion milling, or focused-ion-beam (FIB) lift out techniques. For neutron-irradiated materials, TEM specimen preparation involves creation of radioactive waste, posing safety, disposal and cost concerns, especially for smaller research entities such as universities.

An alternative non-destructive examination approach to cavity characterization can be realized using backscatter electrons (BSE) in scanning electron microscopy (SEM). This technique has the advantage that a very large area of a specimen can be characterized, significantly increasing the measured volume compared to techniques involving transmission microscopy. The SEM-BSE technique has been used previously to detect and quantify surface-connected cavities in nuclear fuel, shale, ceramic coating on dental implant, integrated circuits, but also to measure near-surface cavities covered with metallic films [19-24]. Most recently, the technique has been used to detect and quantify subsurface cavities in neutron-irradiated steels [25]. The potential for such quantification is shown in Fig. 1, where a complex Russian alloy is exhibiting very heterogeneous void swelling imaged with BSE that would be difficult to characterize by transmission microscopy alone.

Fig. 1. A SEM-BSE image obtained by using 30 keV electron analysis beam and from Russian EK-164 fuel cladding irradiated to 82 dpa at 515°C. These micrographs were supplied by Vladimir Pashtukov of the Institute of Nuclear Materials in advance of publication.

While the study that produced the micrographs above has demonstrated the feasibility of the SEM-BSE technique and has begun to explore experimentally the combination of materials, specimen preparation procedures, detector type and configuration, and beam characteristics that influence cavity imaging, additional studies are required to better define the physical fundamentals of image generation, and to guide future experimental efforts to better define choices of parameters that determine resolution, sensitivity, and limitations of the SEM-BSE technique.

In this study, Monte Carlo modeling is used to understand the mechanism of image formation, and to identify key factors that influence cavity imaging. The derived knowledge can be applied to better optimize the conditions for best imaging.

2. Modeling procedure

This study used the CASINO Monte Carlo simulation code [26-30]. to obtain backscattered-electron (BSE) images of cavities under different configurations and beam conditions. The code has been widely used to simulate electron trajectories in solids, often needed for SEM imaging or electron lithography [30]. The code is based on binary collision approach with electron-atom scattering described by tabulated Mott cross sections. The electron energy loss is given by [31]

$$\frac{dE}{ds} = -7.85 \times 10^4 \frac{\rho Z}{EM} \ln\left(\frac{1.166E}{J^*}\right) \quad (\text{KeV/cm}) \quad (1)$$

where J^* is the modified ionization potential, given by

$$J^* = \frac{J}{1 + k \frac{J}{E}}, \text{ where } J = \begin{cases} 11.5Z (Z < 13) \\ 9.76Z + 58.5Z^{-0.19} (Z \geq 13) \end{cases} \quad (2)$$

Z is the element number of target atom. Eqs. (1) and (2) are known to be valid to describe electron-solid interactions to energies as low as 100 eV.

The code uses tabulated values from ELSEPA (Dirac partial-wave calculation of elastic scattering of electrons and positrons by atoms, positive ions and molecules) cross-section to calculate elastic collisions, which involves the calculation of the relativistic partial-wave for scattering [32]. Modeling obtained backscattering efficiency—the ratio of backscattered electron number to incident electron number—agree with experimental results [30].

The present study simulated BSE images arising from pure iron, which was selected as a model substrate to represent stainless steels currently being investigated as fuel cladding materials for fast reactors. The modeling considered different electron-target configurations in order to evaluate the resolution and limitation of cavity imaging by BSE-SEM technique. The electron beam energies range from 0.5 keV to 40 keV. The beam spot sizes range from 1nm to 10

nm. Cavities are introduced at different depths, with their sizes ranging from 5 nm to 60 nm. Furthermore, a comparison is made among bulk Fe and Fe membranes of 100 to 200 nm thickness, since the latter represent typical electron-transparent TEM specimens.

3. Results and discussion

3.1 Mechanisms

Fig. 2a shows the simulated electron trajectories of 10 keV electrons in Fe. The blue lines represent trajectories of incident electrons, while red lines refer to trajectories of backscattered electrons. Most backscattering events occur at depths much shallower than the projected range of incident electrons. For backscattered electrons, a fraction of them lose energy along their outward paths and stop within the iron target.

Fig. 2. (a) Trajectories of incident electrons (blue) and backscattered electron (red) arising from 10 keV electrons bombarding pure iron, (b) schematics of electron backscattering in cavity-free iron and (c) schematics of electron scattering in iron containing a cavity.

If cavities exist at a depth shallower than the depth where the majority of backscattering events occur, incident electrons hitting a cavity will fly as free electrons without energy loss within the cavity,. After penetrating through the cavity, these electrons will either stop or be backscattered at a depth deeper than those electrons that do not intersect the cavity. Figs. 2b and 2c schematically compare the difference between inward and outward paths for electrons with or

without the presence of a cavity. For backscattering events occurring at deeper depths, their outward path towards the surface will be longer. Consequently, two effects are operating: (1) some backscattered electrons will never reach the surface due to increased outward lengths, and (2) for those energetic electrons reaching the surface, their backscattering energy spectrum is shifted. Both effects will change the BSE imaging intensity and make it possible to image cavities.

Fig. 3a shows the depth distributions of 10 keV electrons in Fe. The electron projected range is about 180 nm (black curve) in cavity-free Fe. In comparison, for a cubic cavity with dimensions of $30 \text{ nm} \times 30 \text{ nm} \times 30 \text{ nm}$ introduced at depth of 10 nm (the position of the cavity's upper surface), the electron depth distribution curve is shifted deeper, with projected range slightly increased to $\sim 200 \text{ nm}$ (red curve).

Fig. 3. (a) Depth distributions of electrons in 10 keV electron-irradiated Fe without (black) and with a $30 \text{ nm} \times 30 \text{ nm} \times 30 \text{ nm}$ cubic cavity introduced with its top surface at 10 nm depth, and (b) the probability of backscattering depths for BSE electrons, without (black) and with the cavity (red).

Fig. 3b shows the probability distribution of backscattering events as a function of their occurring depths in 10 keV electron irradiated Fe. The plot considers only the electrons which are able to reach the surface. In other words, Fig. 3b shows those backscattering events

producing the electrons received by the BSE detector. For cavity-free Fe, the most-likely-backscattering depth is about 60 nm (black curve). With the cavity, the probability peak shifts to ~80 nm (red curve), with a dip occurring in the near surface region, corresponding to the cavity embedded region.

Fig. 4 compares the energy spectra of backscattered electrons when they leave the surface. Both electron beam energy and cavity location are the same as Fig. 3. The highest energy of ~10 keV corresponds to the backscattering which occurs at the surface. While the energy spectrum of cavity-free Fe appears similar to a Gaussian curve with the peak at about 8 keV, a dip in the energy spectrum of cavity-embedded Fe is observed at energy of ~9 keV. For backscattering events which occur immediately beneath the cavity, their outward paths towards the surface is suddenly increased, which contributes to a “discontinuity” of the energy spectrum. For the same reason, the energy spectrum of deeper backscattering events is slightly shifted to the low energy end due to additional energy losses along their outward paths.

Fig. 4. Probability distribution of BSE energies from 10 keV electron-irradiated Fe, without (black) and with a 30 nm × 30nm × 30nm cubic cavity (red) introduced at 10 nm depth.

The energy spectral difference is critical to understand the response of the BSE detector. The larger the cavity, the wider the dip region, and the less of backscattered electrons available for BSE imaging. Furthermore, cavity position is important as well. A better cavity image contrast is expected when the cavity is located at the depth which corresponds to the probability peak in Fig. 4. If the cavity is located too deep, the corresponding spectrum dip is shifted to the lower energy end which is too far away from the yield peak, and the impact on BSE detector yields become negligible. Hence cavity imaging become more difficult. Fig. 4 further suggests that the cavity resolution (minimum size that can be imaged) depends on both the electron beam energy and the cavity depth location.

Fig. 5 compares the probability distributions of BSEs as a function of backscattering angles at the surface, with or without presence of the cavity. The angle zero corresponds to the sample normal direction. Without a cavity, the angle distribution is symmetric around 45°. With the cavity, the distribution is slightly skewed with the peak shifted to about 50°. Fig. 5 also shows that at angles less than ~50°, BSE yields become slightly lower if a cavity is introduced. The difference is, again, caused by increased path length for the outward backscattered electrons, which increases lateral straggling toward the surface. Since the BSE detector is usually positioned to collect BSEs backscattered at small angles, the reduced backscattering yields contribute to additional cavity image contrast when cavities are present.

Fig. 5. Angular distributions of BSE at the surface, arising from 10 keV electron-irradiated Fe, without (black) and with a 30 nm × 30nm ×30 nm cubic cavity (red) introduced at 10 nm depth.

Fig. 6 summarizes the most-likely-backscattering depth as a function of electron incident energy from 0.5 keV to 40 keV. The solid line is the best fitting line described by $\ln R(\text{nm}) = 0.2 + 1.6 \ln E(\text{keV})$, where R is the depth and E is the beam energy. Roughly, R can be used to represent the maximum depth at which a cavity can be imaged. For cavities located deeper than R , the elastic cross section for BSE creation drops significantly. For cavities located shallower than R , their cavity imaging quality, however, is determined by many other parameters, as will be further discussed below.

3.2 Optimization

Cavity imaging for different electron beam energies is compared in Fig. 7. For all images, electron beams have the same diameter of 3 nm, and the same scan pitch of 2.5 nm. As schematically shown in the top portion of Fig. 7, a total of 7 cavities were introduced in the Fe matrix. Their sizes are 5, 10, 20, 30, 40, 50, and 60 nm, respectively. All cavities are located at the same depth (10 nm or 50 nm) defined by the distance of the upper surface of the cavity beneath the surface.

Fig. 6. Most-likely-backscattering-depth for BSE creation as a function of electron incident energies in Fe.

Fig. 7. BSE images of cubic cavities of different sizes (edge length, from 5 nm to 60 nm), and of different depths (10 nm and 50 nm) and in Fe of different thicknesses (100 nm, 200 nm, and infinite), obtained by using different electron beam energies (1 keV to 40 keV). The electron beam spot size is 3 nm and the scan pitch is 2.5 nm for all conditions.

First, there is an optimized electron beam energy range for the best imaging of cavities at a specific depth. Figs. 7 a-1 to a-8 (the first column) compare the results for cavities located at 10 nm depth. The lowest beam energy, 1 keV, cannot image cavities (Fig. 7 a-1). Starting from 2.5 keV, cavities appear, and the best image contrasts are obtained using beam energies of 5 keV to 10 keV (Figs. 7 a-3 to a-5). At energies of 20 keV and higher (Figs. 7 a-6 to a-8), cavity contrast becomes weaker. Under the best imaging contrast conditions using beam energies of 7.5 -10 keV, the minimum size of cavities which can be detected is about 10-20 nm. No cavities smaller than 10 nm can be imaged.

Second, the specimen thickness weakly influences the contrast. The first, second and third columns of Fig. 7 compare bulk Fe, and Fe membranes of 200 nm thickness and 100 nm thickness, respectively. A thinner thickness means that fewer electrons will be backscattered. However, image contrast may be enhanced if the cavity region has even higher electron loss ratios. As shown in Fig. 7, using electron energies of 20 to 40 keV, a thinner specimen shows slightly enhanced contrast. Fig. 7 a-8 shows that cavities smaller than 50 nm cannot be imaged for bulk Fe by using 40 keV electron. In comparison, Fig. 7 c-8 shows that under the same energy, cavities of 30 nm size can be imaged for a specimen of 100 nm thickness. Overall, the specimen thickness has no noticeable effect on resolution for low energy electron beams of <20 keV. Slightly enhanced resolution is observed by using electron beams of higher energies.

Third, cavity images are sensitive to depth locations. The first and the forth columns of Fig. 7 compare the cavity images for bulk Fe. The cavities in the first column are all located at depth of 10 nm, while the cavities in the forth column are located at a depth of 50 nm. As shown in Fig. 7 d-4, cavities at 50 nm are barely observable by using beam energies of 7.5 keV. In comparison, Fig. 7 a-4 shows clear cavity images under the same beam energy. In general, deeper cavities require higher electron beam energies, but cavity resolution becomes poorer.

The effects of beam size and scan pitch were systematically investigated. Fig. 8 compares the cavity images obtained by using a scan pitch of 2.5 nm, 5 nm, and 10 nm and by using beam sizes of 1 nm, 3 nm, 5 nm, and 10 nm, respectively. The electron beam energy is fixed to be 5 keV for all cases. Within the same column (having the same scan pitch), there is no obvious difference in image contrast by changing beam size from 1 nm to 10 nm. Hence, it is concluded that using a fine beam size, smaller than the resolution limit ~10 nm, does not benefit resolution

enhancement. On the other hand, a comparison within the same row (having the same beam size) shows that the scan pitch influences the image contrast. The smaller the scan pitch, the sharper the image contrast and smaller the cavities being differentiated. As shown in Fig. 8 a-1, a cavity of 10 nm size can be imaged for scan pitch of 2.5 nm. By increasing the scan pitch to 10 nm, all cavity edges are dim and only cavities larger than 20 nm can be imaged. Overall, the image quality is sensitive to scan pitch, and a small scan pitch is always favored.

Fig. 8. Cavity images obtained by using scan pitch of 2.5 nm, 5 nm, and 10 nm and by using beam sizes of 1 nm, 3 nm, 5 nm, and 10 nm, respectively. All voids (sizes from 5nm to 60 nm) are located at the depth of 10 nm.

Due to limited electron penetration depth at a given energy, the cavity characterization is restricted to a certain depth. Fig. 9 compares the images of series of cavities located at different depths, obtained by using different electron beam energies. A total of 16 cavities of equal size (40 nm) are buried at depths ranging from 5 nm to 280 nm. The top of Fig. 9 schematically shows the top view of all cavities. The numbers refer to the depths of cavities, defined as the location of the cavities' top surface. For the lowest electron energy of 1 keV, no cavities can be imaged (Fig. 9a). Increasing the energy to 2.5 keV, cavities at 5 nm and 10 nm depths become visible (Fig. 9b). At 7.5 keV, the third cavity at 30 nm begins to appear (Fig. 9d). At 20 keV, the fourth cavity at 50 nm begins to appear (Fig. 9f). At 30 keV and higher energies, cavity image qualities are further degraded (Fig. 9h). Note this is the conclusion for cavities of 40 nm size only. Cavities of different sizes have different maximum detectable depths, requiring testing of a large sample matrix.

Significant efforts were made in this study to obtain systematic information on both size resolution and maximum detectable depths under various electron beam energies. Both parameters are useful in realistic applications, in order to select appropriate beam conditions for best imaging effects and also to estimate swelling levels. These observations are summarized in Table I and Table II.

Table I summarizes the cavity resolutions at different depths. The column corresponds to size resolutions obtained using different beam energies. This is particularly useful if the position of cavities are known already. For example, helium ion irradiation induces bubble cavity formation that often starts at the helium peak concentration region and then gradually extends toward the surface with increasing helium dose. Table I helps to select the beam energies for the best size resolution.

There is not a simple trend. It is not always true that better size resolution can be achieved by using a lower energy beam. For example, at the depth of 40 nm, best resolution is achieved by using a beam energy of 15 keV. A higher or lower energy will lead to poorer resolution. For frequently used beam energies of 30 keV, no cavity smaller than 30 nm can be imaged at any depth. If beam energy is reduced down to 5 keV, cavity resolution at 10 nm can be obtained but such cavities are limited to a depth of 10 nm or shallower.

Top view
(number refers to depth in nm)

5	10	30	50
70	100	130	160
190	220	250	280

a 1keV

b 2.5keV

c 5keV

d 7.5keV

e 10keV

f 20keV

g 30keV

h 40keV

Fig. 9. BSE images of 40 nm voids located at depths from 5 nm to 280 nm. The electron beam energies range from 1 keV to 40 keV.

Table II summarizes the maximum detectable depth for a cavity of given size. In general, for a given beam energy, the larger the cavities, the deeper the detectable depths. For example, for 30 keV beam energy, a cavity of 40 nm can be imaged if it is located shallower than 10 nm. If a cavity is 150 nm in size, the maximum detectable depth increases to 100 nm.

Table II is useful to estimate void swelling in samples where cavities are randomly distributed, such as those formed in neutron irradiated samples. For a given void size, the total number of voids from SEM images can be assumed to spread over the maximum detectable depth. Thus the swelling contribution from a particular void size can be calculated. First, under a given electron beam energy, void densities as a function of void sizes are measured. Second, for each size, the corresponding maximum depth can be obtained from Table II. Hence swelling contribution for this particular void size can be estimated. Third, all swelling contributions are summed up. In other words, the total swelling, s , is calculated by

$$S = \sum_i \frac{N_i V_i}{A x_{\max}}$$

where i represent a particular void size, N_i is void number counted, V_i is individual void volume, A is characterization area, and $x_{\max}$ is the maximum detectable depth of size i obtained from Table II.

Although the present modeling is limited to BSE imaging only, a major conclusion can extend to secondary electron (SE) imaging as well. SEs come from two sources: one is induced directly by incident electrons and the other is induced by BSEs in the surface region. Both the changes of BSE numbers and BSE backscattering electron energy spectra will influence SE creation on the surface, hence similar imaging contrast can be obtained if the detection mode is switched from BSE to SE. The present conclusion, however, does not apply to cavity imaging in insulators such as SiO_2 . Different from metals, the presence of cavities at shallow depths may cause charging, i.e. inducing positive charges on the top of cavities and negative charges at the bottom of cavities. Such charging will dramatically distort BSE and SE yields for the detector [9].

Table1 Cavity resolution (in nm) for a given depth and beam energy

		The depth(nm)															
		5	10	20	30	40	50	60	70	80	90	100	120	140	160	180	200
Energy (keV)	2.5	10	10	⊗	⊗	⊗	⊗	⊗	⊗	⊗	⊗	⊗	⊗	⊗	⊗	⊗	⊗
	5	10	10	20	30	80	⊗	⊗	⊗	⊗	⊗	⊗	⊗	⊗	⊗	⊗	⊗
	7.5	10	20	20	30	40	50	100	⊗	⊗	⊗	⊗	⊗	⊗	⊗	⊗	⊗
	10	20	20	20	30	40	40	80	150	200	⊗	⊗	⊗	⊗	⊗	⊗	⊗
	15	30	20	20	30	30	40	50	80	100	150	150	200	⊗	⊗	⊗	⊗
	20	30	30	30	40	40	50	60	80	100	150	150	200	⊗	⊗	⊗	⊗
	30	40	30	40	50	60	80	100	100	100	100	150	150	150	200	200	200
	40	40	40	50	50	80	100	150	150	150	150	150	150	150	200	200	200
Note: ⊗ means invisible																	

Table 2 The maximal depths (in nm) of observable cavities for a given cavity size and beam energy

		The size(nm)										
		5	10	20	30	40	50	60	80	100	150	200
Energy(keV)	2.5	⊗	5	10	10	10	10	10	10	10	10	10
	5	⊗	5	20	20	30	40	40	50	50	50	50
	7.5	⊗	5	30	40	40	40	50	50	60	60	60
	10	⊗	⊗	20	30	50	50	50	50	50	60	60
	15	⊗	⊗	10	20	20	30	40	60	70	100	100
	20	⊗	⊗	⊗	40	50	50	60	90	90	120	120
	30	⊗	⊗	⊗	⊗	10	40	50	80	100	100	140
	40	⊗	⊗	⊗	⊗	10	30	30	70	100	120	160
Note: ⊗ means invisible												

Although BSE imaging cannot achieve nanometer resolution as achieved in TEM, the technique is unique as a complementary characterization method. Without the necessity of FIB specimen preparation, cavity characterization can be performed over large areas and obtain very good statistics, significantly better than when using FIB specimens.

Since SEM can image grain boundaries, void-boundary interactions such as formation of void depleted zones along the boundary, can be studied. The technique can be integrated with *in situ* stress tests or annealing to study void evolution under deformation or Ostwald ripening. For ion-irradiated steels, void characterization will be a little more challenging due to the presence of void-denuded zone at the ion-incident surface. However, after cutting and polishing cross sections of ion-irradiated samples, SEM images can be performed to measure the void distribution as a function of depth.

The present study is limited to modeling only. It is difficult to validate all calculations by using experiments but some relevant experimental results are available and they are published separately [25]. Modeling results are in a good agreement with experimental observations. For electron beam energies of 10 to 30 keV, the smallest observable void size is about 20 nm, according to Table I. In comparison, experimental studies are able to observe voids of 10 to 15 nm [25].

The present study shows that the BES imaging technique is limited to voids located at very shallow depths (<200 nm). Hence this technique is unable to detect voids in many ion-irradiated materials under planar view imaging, since a void denuded zone often exists due to free surface's defect sink property. The technique is more suitable for neutron treated samples which are cut from a bulk material. For ion irradiated samples, it is still possible to image voids under cross sectional view with appropriate etching.

4. Conclusions

Monte Carlo simulations of BSE imaging of cavities in pure iron were performed to understand the imaging mechanism and to optimize the imaging quality. The studies show that the cavity resolution is about 10 to 20 nm under the best beam conditions. In general, deeper cavities require higher electron beam energy but imaging resolution drops significantly. Reducing electron beam spot size from 10 nm to 1 nm does not enhance resolution. On the other

hand, reducing the scan pitch helps to enhance image contrast. In general, these conclusions agree with experimental observations but supply additional insight on how to better optimize image quality and quantification of void ensembles.

Two tables were provided to assist experimental applications: one is the cavity resolution at various combinations of cavity depth and beam energy; the other is the maximum detectable depths at various combinations of cavity size and beam energy.

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References:

- [1] G. S. Was, Fundamentals of radiation materials science: metals and alloys, Springer, New York, 2007
- [2] F.A. Garner, "Radiation Damage in Austenitic Steels", in Konings, R.J.M., (ed.) Comprehensive Nuclear Materials, Volume 4, pp. 33-95, 2012, Elsevier.
- [3] F. A. Garner, Chapter 10, "Void swelling and irradiation creep in light water reactor (LWR) environments", in "Understanding and mitigating ageing in nuclear power plants, Ed. P. G. Tipping, pp. 308-356, 2010, Woodhouse Publishing,
- [4] C. Cawthorne and E.J. Fulton, Nature 216 (1967) 575-576.
- [5] G.R. Odette, M.J. Alinger, B.D. Wirth, Annual Rev. Mat. Res. 38 (2008) 471-503.
- [6] L.K. Mansur, Nucl. Tech. 40 (1978) 5-34.
- [7] F.A. Garner, M.B. Toloczko, B.H. Sencer, J. Nucl. Mat. 276 (2000) 123-142.
- [8] S.J. Zinkle, B.N. Singh, J. Nucl. Mat. 206 (1993) 212-229.
- [9] J.G. Gigax, E. Aydogan, T. Chen, D. Chen, Y. Wu, M.Y. Lo, Y. Yang, F.A. Garner, J. Nucl. Mat. 465 (2015) 343-348.

- [10] L. Shao, C.C. Wei, J. Gigax, A. Aitkaliyeva, D. Chen, B.H. Sencer, F.A. Garner, J. Nucl. Mat. 453 (2014) 176-181.
- [11] G.S. Was, Z. Jiao E. Getto, K. Sun, A.M. Monterrosa, S.A. Maloy, O. Anderoglu, B.H. Sencer, M. Hackett, Scripta Materialia 88 (2014) 33–36.
- [12] O. V. Borodin, V. V. Bryk, A. S. Kalchenko, A. A. Parkhomenko, V. N. Voyevodin and F. A. Garner, “Irradiation-induced microstructure, swelling and post-irradiation deformation of stainless steel 18Cr-10Ni-Ti Irradiated with chromium ions at 1-100 dpa at 360-635°C”, in proceedings of 12th International Conference on Environmental Degradation of Materials in Nuclear Power Systems - Water Reactors, 2005, pp. 395-401.
- [13] A. S. Kalchenko, V. V. Bryk, N. P. Lazarev, I. M. Neklyudov, V. N. Voyevodin and F. A. Garner, J. Nucl. Mater. 399 (2010) 114-121.
- [14] O. V. Borodin, V. V. Bryk, A. S. Kalchenko, V. V. Melnichenko, V. N. Voyevodin, F. A. Garner, J. Nucl. Mater. 442 (2013) S817-S820.
- [15] V. Bryk, O. Borodin, A. Kalchenko, V. Voyevodin, V. Ageev, A. Nikitina, V. Novikov, V. Inozemtsev, F. A. Garner, "Ion issues on irradiation behavior of structural materials at high doses and gas concentrations", Proceedings of Accelerator Applications, August 5-8, 2013, Bruges, Belgium.
- [16] Y. E. Kupriyanova, V. V. Bryk, O. V. Borodin, A. S. Kalchenko, V. N. Voyevodin, G. D. Tolstolutsкая, F. A. Garner, J. Nucl. Mater. 468 (2016) 264-273.
- [17] M. Sagisaka, T. Fukuda, Y. Isobe, F. A. Garner, B. H. Sencer, G. M. Bond, G. S. Was, T. Kamada and K. Matsueda, “Evaluation of potential void swelling behavior in PWR core internals using light ion bombardment”, in Proceedings of the 10th International Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, NACE International, Houston, TX, 2002.
- [18] B. H. Sencer, G. S. Was, M. Sagisaka, Y. Isobe, G. M. Bond and Frank A. Garner, J. Nucl. Mater. 323 (2003) 18-28.
- [19] M. Suzuki, Y. Ominami, T. Sekiguchi, G.Y. Yang, Appl. Phys. Lett. 93, 263110 (2008).
- [20] N.S. Rajput, H.C. Verma, Scanning 36 (2014) 430-436.
- [21] Lucille A. Giannuzzi, Daniel Phifer, Nicholas J. Giannuzzi, and Mario J. Capuano, J. Oral and Maxillofacial Surgery 65, 737-747.

- [22] Ludwig Reimer, *Scanning Electron Microscopy Physics of Image Formation and Microanalysis*, 2nd edition, pp235-236, 1998, Berlin, Springer.
- [23] M.E. Curtis, R.J. Abrose, C.H. Sondergeld, C.S. Rai, "Structural Characterization of Gas Shales on the Micro- and Nano-Scales, Society of Petroleum Engineers, SPE-137693-MS. Canadian Unconventional Resources and International Petroleum Conference, 19-21 October, 2010, Alberta, Canada.
- [24] L. M. Gignac, M. Kawasaki, S. H. Boettcher, and O. C. Wells, "Imaging and analysis of subsurface Cu interconnects by detecting backscattered electrons in the scanning electron microscope," *Journal of Applied Physics* 97 (2005) 114506.
- [25] V.I. Pastoukhov, S.A. Averin, V.L. Panchenko, I.A. Portnykh, P.D. Freyer, L.A. Giannuzzi, F.A. Garner, *J. Nucl. Mat.*, in press and to be published in the same issue.
- [26] P. Hovington, D. Drouin, R. Gauvin, *Scanning* 19 (1997) 1-14.
- [27] P. Hovington, D. Drouin, R. Gauvin. *Scanning* 19 (1997) 20–28.
- [28] P. Hovington, D. Drouin, R. Gauvin. *Scanning* 19 (1997) 29–35.
- [29] D. Drouin, A.R. Couture, D. Joly, X. Tastet, V. Aimez, R. Gauvin, *Scanning* 29 (2007) 92–101.
- [30] H. Demers, N. Poirier-Demers, A.R. Couture, D. Joly, M. Guilmain, N. de Jonge, D. Drouin, *Scanning* 33 (2011) 135-146.
- [31] D.C. Joy, S. Luo, *Scanning* 11 (1989) 176-189.
- [32] F. Salvat, A. Jablonski, C.J. Powell, *Comput Phys Commun* 165 (2005) 157-190.