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Mechanism of the monoclinic-to-tetragonal phase transition induced in zirconia and hafnia by swift heavy ions

Abdenacer Benyagoub*

Centre Interdisciplinaire de Recherche Ions Lasers (CIRIL-GANIL), CEA-CNRS-ENSICAEN, BP 5133, F-14070 Caen Cedex 5, France

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Recent results demonstrated that defect formation or amorphization are not the only structural changes induced by swift heavy ions in crystalline materials and that under certain circumstances crystalline-to-crystalline phase transitions can also occur. For instance, it was found that both zirconia and hafnia transform from the monoclinic to the tetragonal phase with a kinetics involving a double ion impact process. In order to understand the origin of this ion-beam induced phase transition, the behavior of these twin oxides was analyzed and compared. In fact, the likeness of these materials offered the unique opportunity to impose drastic constraints on the possible models proposed to explain the creation of atomic displacements in the wake of swift heavy ions. This comparison clearly suggests that the thermal spike is the most appropriate process which governs the transition from the monoclinic to the tetragonal phase in zirconia and hafnia.

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I. INTRODUCTION

It is now well established that the electronic energy loss released by swift heavy ions can cause significant atomic movements in various types of solids. For instance, it was found that these ions can: (i) Produce a giant anisotropic deformation in amorphous solids (Klaumünzer's effect);[1–4] (ii) induce, for moderate electronic energy losses in metals, a recovery of the defects generated by nuclear collisions;[5,6] and (iii) create damage in the irradiated target independently of its original structure (either crystalline[7,8] or amorphous[9,10]). More precisely, in the case of crystalline materials, the huge amount of electronic energy loss can lead to the formation of highly defective or amorphous tracks (the so-called "*latent tracks*"). Presently, two competing models are usually proposed to account for the creation of these ion tracks: The "Coulomb explosion"[11,12] and the "thermal spike"[13–16] models. The first one is based on the electrostatic repulsion between the ions generated in the wake of the incident ion which can disrupt the original crystalline structure. The second one considers the energy transferred ultimately as heat to the lattice atoms which can then reach the melting temperature followed by a rapid quenching with, as a result, the possibility to lead to the frozen-in of an amorphous structure. Since in both cases, the timescales of the processes involved to explain track formation are rather inaccessible to usual characterization techniques, it is not easy for the moment to identify the actual mechanism. In addition, very few results demonstrated that amorphization is not the only phase transformation process which can be induced by swift heavy ions in crystalline solids. As a matter of fact, recent irradiation experiments performed on some oxides[17–24] reported that crystalline-to-crystalline phase transitions can also occur. It was found, for example, that monoclinic zirconia and hafnia transform to the tetragonal phase at room temperature when the deposited electronic energy loss is in excess of an effective threshold around 12 keV nm^{-1} for zirconia[18,19,21,22] and around 20 keV nm^{-1} for hafnia.[23,24]

Therefore, the aim of the present paper is to try to address the question concerning the origin of this phase transition: Is it related to a process based on the Coulomb explosion effect or a mechanism based on the thermal spike phenomenon? For this purpose, the transition from the monoclinic to tetragonal phase in zirconia will be analyzed and compared with that found in hafnia, by performing additional irradiation experiments in these two twin compounds with the aim of confronting the whole obtained results with the existing models. It is worth noting that these oxides share many physical and chemical properties but have also some differences. For example, both materials are hard ceramics with comparable high dielectric constants and wide band gaps. In addition, under the normal conditions of temperature and pressure, both oxides have a monoclinic structure in their pure (i.e., undoped) state. For zirconia, the lattice parameters are: $a=5.150$ Å, $b=5.211$ Å, $c=5.317$ Å, and $\beta=99.23°$; while for hafnia, the structure is very slightly modified: $a=5.117$ Å, $b=5.175$ Å, $c=5.291$ Å, and $\beta=99.21°$.[25] They have thus practically the same atomic density ($\sim 8.3\times 10^{22}$ atoms cm^{-3}). Accordingly, an important difference arises in their specific gravity ($\sim$5.68 for ZrO_2 and $\sim$9.68 for HfO_2) due to the huge difference in the mass of the cations. Moreover, during heat treatment, both zirconium oxide and hafnium oxide are known to transform first to the tetragonal phase and then to the cubic phase. For zirconium oxide, the tetragonal phase appears at $\sim$1400 K and the cubic phase at $\sim$2600 K;[26,27] while for hafnium oxide, the corresponding phase transitions appear at $\sim$2000 and $\sim$2900 K,[28,29] respectively. For all the above-mentioned reasons, the comparison of the behavior of zirconia and hafnia regarding swift heavy ion irradiation will certainly provide the rare opportunity to confer stringent constraints on the models aimed to describe ion-beam induced phase transitions with, as a result, the possibility to select the appropriate one.

II. EXPERIMENTAL

Similarly to the case of the previous experiments,[18,19,21–24] the samples were prepared from commercial zirconia and hafnia powders having a grain size around 2 and 5 μm, re-

TABLE I. Computed irradiation parameters as deduced from the TRIM code (Ref. 30) in the cases of the irradiation of pure zirconia and hafnia.

Irradiated material	Ion species	Energy (MeV)	Ion range (R_p) (μm)	Electronic energy loss (at sample surface) (keV nm^{-1})	Nuclear energy loss (at sample surface) (eV nm^{-1})
ZrO_2	^{58}Ni	300	25.1	12.3	11
	^{58}Ni	135	11.6	14.2	22
	^{127}I	250	14.4	26.8	87
	^{129}Xe	595	26.4	28.1	44
HfO_2	^{86}Kr	800	42.4	18.3	14
	^{86}Kr	300	17.5	21.9	32
	^{127}I	250	12.9	32	110

spectively. For the purpose of easy handling during ion irradiation and characterization, these powders were compacted in pellets at ambient temperature and under a pressure of 330 MPa. In order to improve their mechanical strength, these pellets were heated up at 800 °C for 5 h in the case of zirconia and at 900 °C for 6 h in the case of hafnia. The obtained specimens were characterized by X-ray diffraction and both types exhibited a pure monoclinic phase. The zirconia samples were irradiated at the Ganil accelerator with 595 MeV Xe ions while the hafnia samples were irradiated with 250 MeV I ions delivered by the Vivitron accelerator in Strasbourg, giving rise to an average electronic energy loss at the sample surface of ~28 and ~32 keV nm^{-1}, respectively. All the irradiations were performed at room temperature with an ion flux limited to 3×10^8 ion cm^{-2} s^{-1} in order to minimize charge effects and target heating. For the same reasons, each sample was encapsulated between a copper cover (with an opening for the ion beam) and a copper block having a cavity with dimensions close to those of the sample. A thermocouple was placed at the backside of each sample and the temperature measured during irradiation never exceeded 50 °C. These experiments supplement the previous ones[18,19,21–24] where zirconia samples were irradiated with 135 MeV Ni, 300 MeV Ni, and 250 MeV I ions and hafnia samples with 300 and 800 MeV Kr ions. Table I collects the computed irradiation parameters deduced from the TRIM code[30] for all these experiments. The samples were characterized by the X-ray diffraction technique either *in situ* at various increasing fluences for the Xe irradiation or *ex situ* (after a few days) for the I irradiation. In both cases, the analyses were performed with a diffractometer equipped with a copper anticathode and a curved position sensitive detector covering an angular domain of 120°. The X-ray incident angle was fixed at 11° in order to have a probed depth limited to 4.4 μm for zirconia and 1.5 μm for hafnia, quite below the ion projected ranges and well within the sample thickness where the electronic energy loss of the irradiating ions remains practically constant.

III. X-RAY ANALYSES

Figure 1 presents some X-ray spectra recorded on zirconia and hafnia samples before and after irradiation with different fluences of 595 MeV Xe and 250 MeV I ions, respectively. In both cases, the spectra exhibit a noticeable change with irradiation indicated by the growth of a new peak at $2\theta\sim30°$ associated with a reduction of the intensity of the $(11\bar{1})$- and (111)-lines belonging to the initial monoclinic phase. However, even at very high fluences, the latter lines do not vanish completely, revealing that a small fraction of the monoclinic phase still remains in the irradiated samples. Moreover, the X-ray diffraction patterns do not exhibit any indication for amorphization. It was already mentioned[18,19,21–24] that the new peak, which appears at $2\theta\sim30°$, can be attributed either to the line (101) of the tetragonal phase or to the line (111) of the cubic structure[25] or even to a possible mixture of both phases because X-ray analysis can hardly allow one to discriminate these two phases. However, additional Raman spectroscopy analyses[18] performed on zirconia clearly proved that the new formed phase is mainly tetragonal with a small remainder of the original monoclinic phase. An interesting feature of this phase transition is that the obtained tetragonal phase is stable at room temperature for more than several months. This is in contrast with the fact that the tetragonal phase is thermodynamically stable only at temperatures of ~1400–2600 K for pure zirconia and at temperatures of ~2000–2900 K for pure hafnia.

IV. KINETICS OF THE PHASE TRANSFORMATION

In a similar fashion as in our previous studies, the analyses of the X-ray spectra were performed using the procedure proposed by Garvie and Nicholson[31] to quantify the composition of a zirconia sample containing a mixture of the monoclinic and tetragonal phases. According to this method, the concentration C of the tetragonal phase is just given by:

$$C = \frac{I(101)}{I(11\bar{1}) + I(111) + I(101)} \tag{1}$$

where $I(11\bar{1})$, I(111), and I(101) are the peak integrals of the corresponding lines $(11\bar{1})$, (111), and (101) of the monoclinic and tetragonal phases. The results of these analyses are collected in Fig. 2 for both zirconia and hafnia. It clearly ap-

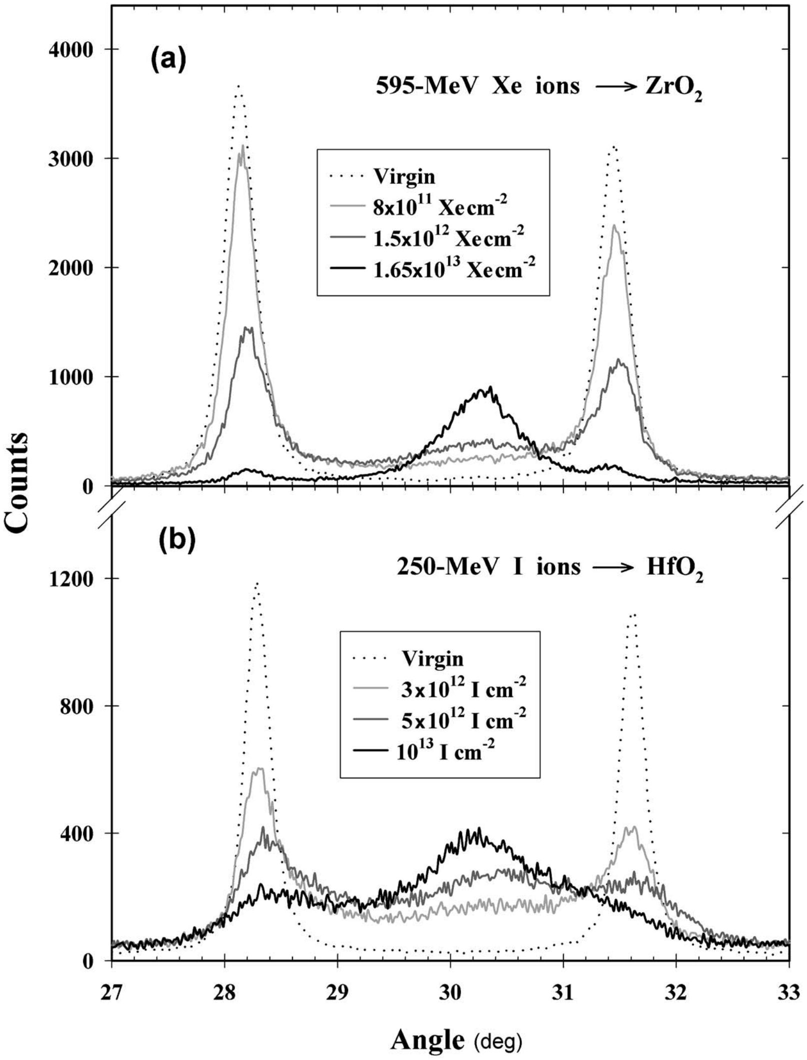

FIG. 1. Evolution with the ion fluence of the X-ray diffraction patterns recorded on monoclinic zirconia irradiated with 595 MeV Xe ions (a) and monoclinic hafnia irradiated with 250 MeV I ions (b).

pears that the evolution of the fraction of the tetragonal phase with the ion fluence exhibits a sigmoidal behavior as already found in the previous experiments.[19,21–24] This result is a strong indication that with swift heavy ions track overlapping is necessary to produce phase transition from the monoclinic to the tetragonal structure in both zirconia and hafnia. If one assumes that the spatial distribution of the ion impacts obeys to a Poisson statistics and additionally supposes the necessity of track overlapping to ensure phase transition, one can then reproduce the experimental data with the following expression:[32–34]

$$\Delta C(\Phi) = \Delta C(\infty)\left(1 - \sum_{k=0}^{n-1} \frac{(\sigma\Phi)^k}{k!}\exp(-\sigma\Phi)\right) \qquad (2)$$

where $\Delta C(\Phi)$ is the increase of the concentration of the tetragonal phase with the ion fluence Φ, $\Delta C(\infty)$ the total increase of this concentration measured at saturation, σ the cross section of the cylinder around the ion path where matter undergoes transformation, and n the minimum number of ion impacts necessary to transform this cylinder to the final state. The fit of this equation to the experimental data is represented by a solid line in Fig. 2 for both experiments and provides the following values: $\Delta C(\infty)=0.89\pm0.02$, $\sigma=(7.1\pm0.3)\ 10^{-13}$ cm^2, and $n=2$ for the irradiation of zirconia with 595 MeV Xe ions, and $\Delta C(\infty)=0.73\pm0.02$, $\sigma=(4.8\pm0.3)\ 10^{-13}$ cm^2, and $n=2$ for the irradiation of hafnia with 250 MeV I ions. One can notice in the latter case that the transformation is much more complete than in the experiment where hafnia was irradiated with 300 MeV Kr ions.[23,24] Furthermore, it arises from the fit of the experimental data that in both oxides the kinetics of phase transition is based on the same mechanism: necessity of two ion impacts within the same region in order to ensure phase transition. Therefore, this ion impact number ($n=2$) appears to be a constant feature of this phase transition. This peculiarity can be understood by the fact that the presence of oxygen vacancies is frequently invoked as a prerequisite[35,36] for the thermally induced phase transition from the monoclinic to the tetragonal structure obtained usually by heating. Accordingly, the main role of the first ion impact is probably to create enough radiation defects including a sufficient amount of oxygen vacancies. However, the structure of these once-irradiated regions even though disordered certainly remains still monoclinic otherwise the kinetics of the phase transition would be linear and not sigmoidal at low fluences. On the other hand,

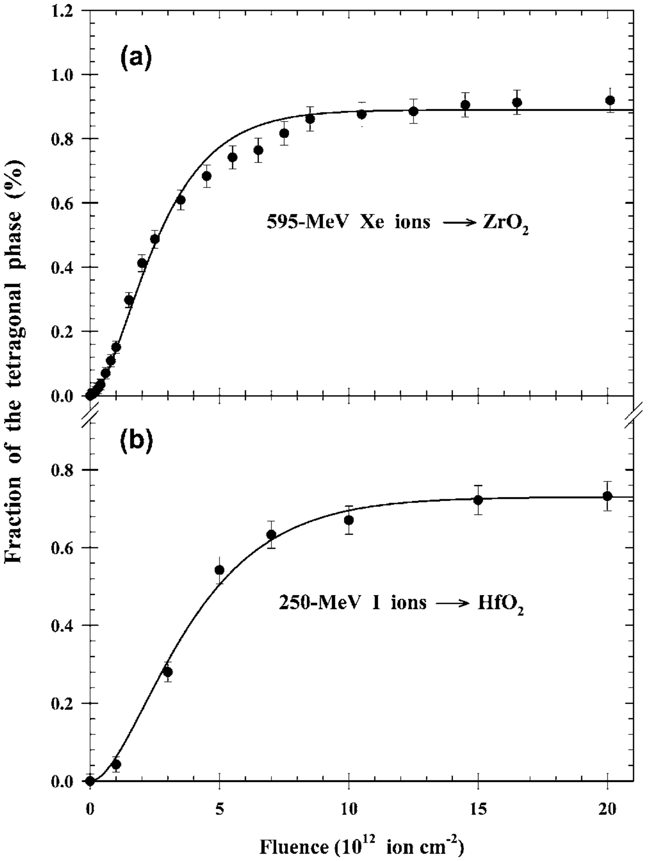

FIG. 2. Evolution with the ion fluence of the fraction of the tetragonal phase in the case of the irradiation of pure zirconia with 595 MeV Xe ions (a) and the irradiation of pure hafnia with 250 MeV I ions (b). The solid lines are the best fits of the experimental data with the expression given by Eq. (2) of the text.

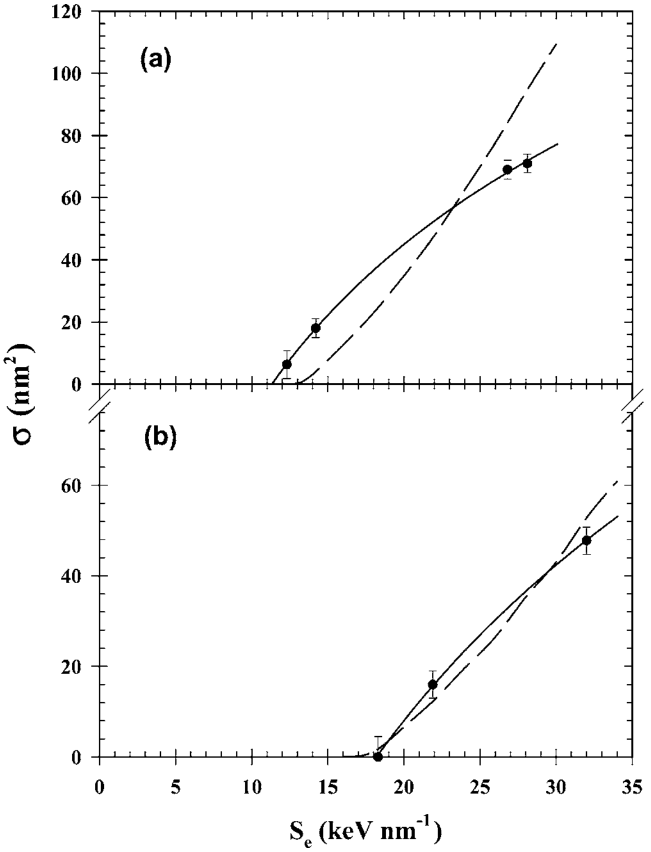

FIG. 3. Evolution with the electronic energy loss of the cross-section for phase transformation in the case of pure zirconia (a) and pure hafnia (b). The dashed and solid lines are the best fits to the experimental data according, respectively, to the numerical solution (Refs. 14 and 15) and the analytical approximation (Ref. 16) of the thermal spike model.

since at the moment these regions contain enough oxygen vacancies they become ready to transform to the tetragonal phase after any subsequent ion impact.

V. MECHANISM FOR THE ION-BEAM INDUCED PHASE TRANSITION

It was previously[18,19,21–24] pointed out that with swift heavy ions the phase transformation in zirconia and in hafnia is solely driven by the electronic energy loss at least within the outmost region probed by the X-rays. Therefore, Fig. 3 presents the evolution of the cross section σ with the deposited electronic energy loss for the irradiations performed either in this study or in previous experiments.[19,21–24] For zirconia, it appears that σ starts to rise up gradually within a transitional region around 12 keV nm^{-1} and then increases steadily with the electronic energy loss. The same behavior also occurs for hafnia but with a shift of the transitional region to the neighborhood of 18 keV nm^{-1}. Nevertheless, an important question arises on how the electronic energy loss can induce a transformation from the monoclinic to the tetragonal phase in these materials. Two competing models are generally proposed to account for atomic displacements in solids driven by the huge amount of electronic energy loss released in the wake of swift heavy ions: The "Coulomb explosion" model and the "thermal spike" mechanism.

The "Coulomb explosion" model is primarily based on the charge separation between the ejected electrons and the transiently ionized atoms and focuses on the repulsion between the latter. This ionic repulsion can then lead either directly to atom displacements[11] or to the propagation of a shock wave[12] which can ultimately induce a phase transition. For instance, in their Coulomb explosion model, Fleisher, Price, and Walker[11] established a criterion for atomic displacements leading to track formation based on the assumption that the Coulomb repulsive forces must overcome the mechanical strength of the irradiated material. They define the sensibility for atomic displacements by the following quantity: $Y\varepsilon a_0^4/10e^2$ where Y is the Young's modulus, ε the dielectric constant of the material, a_0 the average atomic spacing, and e the electron charge. Therefore, owing to the fact that the Young's modulus,[37,38] the dielectric constant[39,40] and the average atomic spacing[25] of zirconia are quite similar to those of hafnia, this model predicts the same sensibility for both materials. This is in contradiction with the data shown in Fig. 3 where it appears that zirconia is quite more sensitive to the electronic energy loss than hafnia: The threshold in the electronic energy loss is about 60% higher in hafnia than in zirconia. Such a difference can hardly be explained solely by the uncertainties in the experimental values of the material parameters entering in the definition of the sensibility parameter. On the other hand, according to the model proposed by Lesueur *et al.*[12] the cross section σ must be proportional to $(dE/dx)^4$. This is visibly not the case if one refers to the data concerning zirconia in Fig. 3 where one can easily notice a rather linear increase above the transitional region. In addition, this model also predicts that the radial recoil energy transmitted by the incident ion to the lattice atoms surrounding the ion path is proportional to $(dE/dx)^4/(M_2\omega_p{}^2)$ where M_2 is the target atomic mass and ω_p the electron plasma frequency.[41] Therefore, if one tries to apply this model to predict the threshold for inducing the phase transition in hafnia by using the experimental value obtained for zirconia ($\sim$12 keV nm^{-1}) one will find nearly 14 keV nm^{-1} which is well below the actual experimental value ($\sim$18 keV nm^{-1}). Consequently, it appears that the transformation from the monoclinic to the tetragonal phase induced by swift heavy ions in both zirconia and hafnia cannot be explained by these Coulomb explosion models.

The "thermal spike" model[13–16] considers the transfer of the energy initially shared by the excited electrons. The cooling of these electrons occurs as a result of energy transport by electronic heat conduction to a larger volume and also as a consequence of electron-phonons interactions leading ultimately to a rapid heating of the target atoms surrounding the ion path. Amorphization can then occur in these heated regions if the lattice temperature exceeds the melting temperature. Presently, two versions of the thermal spike model are proposed to explain track formation in solids. The first one[14,15] is based on a numerical solution of the coupled equations governing heat transfer between electrons and atoms through a computer code. If one assumes that such a model which is originally dedicated to explain track formation in solids can also be used to describe crystalline-to-crystalline phase transition, one has just to replace the melting temperature T_m by the phase transition temperature T_c. Besides the thermal data of the irradiated materials, this model needs the knowledge of the electron-phonon mean free path λ, which is frequently unknown and then considered as a free parameter. In the case of zirconia, a rather moderate fit of the experimental data is obtained with $\lambda = 10.4$ nm and the corresponding theoretical curve is indicated by a dashed line in Fig. 3(a). Similarly, in the case of hafnia, a good fit of the experimental data is achieved with $\lambda = 9.2$ nm and is represented by a dashed line in Fig. 3(b). Within this model, the threshold for zirconia is predicted at $\sim$13 keV nm^{-1} while that for hafnia is expected at $\sim$18 keV nm^{-1}. Both values are in reasonable agreement with the experiment. The other version of the thermal spike model is based on an analytical approach.[16] The main assumptions of this model is that soon after the passage of the incident ion, a fraction g of the electronic energy loss S_e is converted to heat for the atomic lattice and that the lattice temperature has a Gaussian distribution with an initial radial extension a_0 which evolves with time. According to this model, the cross section of the cylinder where matter reaches the melting temperature T_m is given by:

$$\sigma(S_e) = \sigma_0 \ln(S_e/S_{et}) = \pi a_0^2 \ln(S_e/S_{et}) \quad \text{for } S_{et} \leqslant S_e \leqslant 2.7 S_{et} \tag{3a}$$

$$\sigma(S_e) = \sigma_0 S_e/(2.7 S_{et}) = \pi a_0^2 S_e/(2.7 S_{et}) \quad \text{for } S_e \geqslant 2.7 S_{et} \tag{3b}$$

with the threshold in the electronic energy loss $S_{et} = \pi a_0^2 \rho c (T_m - T_0)/g$, where c and ρ are the specific heat and the density of the irradiated material and T_0 is the irradiation temperature. If here again one supposes that this model, initially devoted to discuss track formation in solids, can also be applied to the description of crystalline-to-crystalline phase transition, one has just to replace the melting temperature T_m by the phase transition temperature T_c in the expression of the threshold in the electronic energy S_{et}. Such an analysis is made for the experimental data exposed in Fig. 3 where the best fit is represented by a solid line and provides the following values $\sigma_0 = (79 \pm 2)$ nm^2 and $S_{et} = (11.3 \pm 0.2)$ keV nm^{-1} for zirconia and $\sigma_0 = (84 \pm 2)$ nm^2 and $S_{et} = (18.2 \pm 0.2)$ keV nm^{-1} for hafnia. It appears that this analytical description of the thermal spike model fits the experimental data better than the numerical version. This is probably due to the fact that the former uses two free parameters while the latter needs only one. From the values of the parameter σ_0, one finds $a_0 = 5.01$ nm for zirconia and $a_0 = 5.17$ nm for hafnia. In addition, if for zirconia one takes $\rho = 5680$ kg m^{-3}, $c = 456$ J kg^{-1} K^{-1}, and $T_c - T_0 = (1400 - 300)$ K $= 1100$ K, one can deduce from the expression of S_{et} that $g = 0.124$. Similarly, if one takes for hafnia $\rho = 9680$ kg m^{-3}, $c = 272$ J kg^{-1} K^{-1}, and $T_c - T_0 = (2000 - 300)$ K $= 1700$ K, one will find $g = 0.129$. It appears then that the values of a_0 and g are nearly the same for both oxides, this finding is in agreement with similar results obtained within other classes of materials.[16,42] Therefore, it is a quite reasonable hypothesis to suppose that a_0 and g are the same for these twin oxides. If, in addition, one takes into account the Dulong-Petit law (which implies that the product ρc is the same for ZrO_2 and HfO_2), one then expects the following relation:

$$\frac{S_{et}^{\mathrm{HfO}_2}}{S_{et}^{\mathrm{ZrO}_2}} \cong \frac{(T_c - T_0)^{\mathrm{HfO}_2}}{(T_c - T_0)^{\mathrm{ZrO}_2}} \tag{4}$$

indicating that according to the thermal spike model the thresholds for zirconia and hafnia must scale with the corresponding transition temperature. This relation can readily be checked by referring to the experimental data. Hence, the l.h.s. of Eq. (4) is given by $18.2/11.3 = 1.61$ while the r.h.s. is $(2000 - 300)/(1400 - 300) = 1.55$. The agreement is quite good and, therefore, clearly suggests that the thermal spike is the right process which governs the transition from the monoclinic to the tetragonal phase in zirconia and hafnia.

VI. CONCLUSION

There is now growing evidence that the structural changes induced by the irradiation with swift heavy ions are not confined to the generally accepted view of damage creation (either defect production or amorphization) but can also comprise crystalline-to-crystalline phase transitions. For example, the irradiation experiments described in this paper again verified by using other irradiation species that both zirconia and hafnia transform from the monoclinic to the tetragonal phase. However, in both materials the kinetics of the phase transition is related to a mechanism involving a double ion impact. Owing to the fact that this behavior occurs for various ions spanning over a wide range of the deposited electronic energy loss, one can conclude that this mechanism is a constant feature of this phase transition for both oxides. Nevertheless, it is worth noting that such a process is quite singular for structural changes induced by swift heavy ions because it differs from the well-known case of amorphization where a single ion impact mechanism is generally invoked. The role of the first ion impact is probably to create a sufficient amount of radiation defects, very likely oxygen vacancies which seem to be a prerequisite for the phase transition. Consequently, this double ion impact mechanism has to be approached with that occurring with the Klaumünzer's effect[1–4] where giant plastic deformation takes place in amorphous solids after an incubation fluence where "shear sites" are supposed to be created by the first ion impact.

Another important result of this paper is related to the use of the monoclinic to tetragonal phase transition in the twin oxides, zirconia and hafnia, as a probe for seeking for the appropriate model aimed to describe structural changes induced by swift heavy ions. These models are either based on the Coulomb explosion concept or on the thermal spike mechanism. Even though these models were originally designed to explain latent track formation, they can readily be extended to the case of crystalline-to-crystalline phase transformations. However, since zirconia and hafnia can be considered as differing mainly by the mass of their cations, the comparison of the features of the monoclinic to tetragonal phase transition in these materials imposed severe constraints on these competing models. For instance, the fact that the thresholds in the electronic energy loss for inducing this phase transformation scale reasonably well with the corresponding transition temperature at thermal equilibrium is quite in line with the thermal spike model. In addition, the evolution of the cross section for phase transformation with the electronic energy loss is consistent with this model. On the other hand, it is worth noting that both results were not satisfactorily explained by the Coulomb explosion models. Therefore, this comparative study led to the conclusion that the thermal spike mechanism is the appropriate interpretation describing the crystalline-to-crystalline phase transition in zirconia and hafnia rather than the Coulomb explosion process. This finding can probably be extended to the case of other swift heavy ion induced structural transformations like amorphization.

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*Fax: + 33 2 31 45 47 14. Email: benyagoub@ganil.fr

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