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Regular Article

Experimental and theoretical studies of the energy-loss straggling of H and He ion beams in HfO_2 films

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Abstract. We report an experimental-theoretical study of the energy-loss straggling of protons and alpha particles in HfO_2 films. In the case of H ions the experiments were performed in the energy range 40–1750 keV. For the lower energy interval (40–250 keV) we have used the medium energy ion scattering (MEIS) technique with a resolution of $\Delta E/E \sim 4 \times 10^{-3}$, while for the higher energies the Rutherford backscattering technique (RBS) was employed with an overall resolution of 7 keV. Concerning the He ions the straggling study has covered an energy range between 250 and 3000 keV by using RBS measurements, which in this case had a resolution better than 10 keV. The theoretical calculations were done in the framework of the dielectric formalism using the MELF-GOS model to obtain a proper description of the energy loss function (ELF) of the HfO_2 target. It is shown that for both projectiles the experimental data and the theoretical predictions for the energy-loss straggling display a very good agreement.

PACS. 34.50.Bw Energy loss and stopping power – 77.22.-d Dielectric properties of solids and liquids

1 Introduction

When a fast light ion moves through matter, its initial energy is lost in the target due mainly to interactions with the electrons of the material. As these interactions are of statistical nature, during the motion of the fast projectile inside the target there are fluctuations in the individual energy-transfer values, as well as in the number of interactions taking place. Therefore a spread in the energy-loss distribution appears, which gives rise to the so called energy-loss straggling.

A good control of the energy deposited by an energetic projectile is essential for achieving a reasonable interpretation in ion beam analysis or a satisfactory result in ion beam modification of materials. The main parameters that quantify the energy deposition are the electronic stopping power (which is the mean energy deposited per unit path length) and the energy-loss straggling (which represents the mean square deviation of the energy loss distribution per unit path length) [1]. Both magnitudes are widely studied experimentally as well as theoretically, although results for the latter are much scarcer than for the former, in particular for the case of compound targets.

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Therefore, further work on the energy-loss straggling in elemental and, in particular, compound targets is necessary. These studies are very important in the microelectronics industry, because when dealing with thin films the undesirable broadening of the deposited energy (due to the straggling) may significantly affect the profile of the implanted dopant [2].

At low incident H and He ion velocity, the energy-loss straggling is only affected by the target valence electrons, while at higher projectile velocities the more tightly bound electrons in the target atomic core also contribute [3,4]. Therefore for compound materials the energy-loss straggling at low and intermediate projectile velocities is affected by aggregation (chemical and phase) effects while at higher energies the participation of the target atomic core electrons increases, which behave almost as when they belong to isolated atoms.

Assuming that all the target electrons contribute to the energy loss, Bohr [5] provided a simple expression for the value of the energy-loss straggling in the case of an elemental target:

$$\Omega_B^2 = 4\pi Z_1^2 e^4 Z_2 N, \tag{1}$$

where Z_1 and Z_2 are the projectile and target atomic numbers, respectively, e is the electronic charge and N is the target atomic density. Equation (1) is referred to as the Bohr straggling and is frequently used to estimate the corresponding energy-loss straggling value for the case of high projectile velocities. The value of $\Omega_{\rm B}^2$ can be also calculated straightforwardly for compound materials through Bragg's additivity rule [6] applied to their elemental constituents.

There is some available information in the literature on the straggling for metals or, more generally, conductors. However in the case of insulators the measurements are more complicated and the experimental information is scarce [7]. The major experimental difficulties concern the smoothness and homogeneities of the film under study, as well as target charging effects.

Due to its high dielectric constant, wide energy bandgap energy [8], and good thermal stability [9], HfO_2 films play a strategical role in the microelectronics industry because they already start to replace the SiO_2 films in metal oxide field effect transistors (MOSFET) allowing further miniaturization [10]. In fact, for this compound there is a lack of information about the straggling and even on the stopping power for light ions, which should be of great utility for the industry oriented research, as well as for basic studies [11].

In this paper we present energy-loss straggling measurements and calculations for H and He ion beams in HfO_2 films. For the H beams the experiment was done in the energy range from 40 keV up to 1750 keV, while for the He ions, the investigated energy range was between 250 and 3000 keV. The experimental results are compared with theoretical calculations based on a proper description of the dielectric properties of the target [12,13] adapted to the present case of a HfO_2 target.

The rest of this work is organized as follows. In Section 2 the experimental procedure and details are described, while the theoretical model is outlined in Section 3. The comparison of experimental and theoretical results is shown in Section 4, and finally a summary is drawn in Section 5.

2 Experimental procedure

2.1 Sample preparation

The HfO_2 films were grown on a Si (100) substrate by radio frequency (rf) magnetron sputtering (150 W) using a HfO_2 target with a nominal purity of 99.95% and O_2/Ar gas mixture as sputtering gas. The sputtering system was evacuated to 8.0×10^{-8} torr by a turbo molecular pump backed by a mechanical pump before the deposition. The total work pressure was 5.7 mtorr during the deposition with an Ar gas flow of 19.6 sccm and an O_2/Ar ratio flow of 0.35 was used. The deposition rate (3.3 nm/min) was checked by the analysis of low-angle X-ray reflectivity scan on one of the HfO_2 films, and the thicknesses ($t = 17, 32, 63, 72, 91$ and 1450 nm) of the HfO_2 films were controlled using the deposition time and after check by the X-ray reflectivity technique. A Phillips X-Pert θ–2θ diffractometer employing Cu $K\alpha$ radiation was used to obtain the low and high angle diffraction scans. The surface was observed by atomic force microscopy (AFM) using a NanoScope-IIIA from Digital Instruments. The mean square roughness of the films was on average less than 5% of the total film thickness and that of the Si substrate was 0.3 nm. The stoichiometry of the films was checked using the Rutherford backscattering technique (RBS) and the fitting on the experimental results has confirmed that in fact we are in presence of stoichiometric HfO_2 films. Also we have used a very thin film ($t = 5$ nm, with mean roughness less than 0.5 nm) provided by IBM, Yorktown, USA, in order to perform the measurements with very low energy H beams.

2.2 Experiments and measurements

For the case of H^+ projectiles we have used the medium energy ion scattering (MEIS) technique for the lower energies (from 40 up to 250 keV), with a resolution $\Delta E/E \sim 4 \times 10^{-3}$. For higher energies we have used the standard Rutherford backscattering (RBS) technique. With this aim we had two detectors with an overall resolution of 7 keV situated symmetrically at $\pm 165°$ with respect to the beam direction. For each analyzed energy, a suitable set of samples with different thicknesses was used. In each case RBS spectra were taken at $\theta_1 = 0°$, 30° and 45° with respect to the normal of the sample. Since at non-normal incidence (e.g. under a target tilt different from $\theta_1 = 0°$) a non-symmetrical detector geometry is mandatory, then two different spectra are obtained for each energy, each one corresponding to each detector situated at different angle with respect to the beam direction. In order to obtain the energy-loss straggling we have analyzed only the Hf component of the film, since the O part laid on top of the Si spectra corresponding to the Si wafer and the straggling analysis would be very hard. Therefore, for each energy we had at least six spectra. From them, as will be described in the next sub-section, the corresponding straggling was extracted and an average value was obtained.

The determination of the energy-loss straggling for He^+ projectiles in the 250–3000 keV energy interval was done using only the RBS technique with an overall resolution better than 10 keV. The experimental set-up and procedure was the same as described for the H^+ case and consequently again for each energy we had at least six spectra from where the individual straggling was extracted and an average value was obtained.

Figure 1 shows a typical RBS spectrum of the Hf component of the HfO_2 film for the case of 600 keV He^+ incident with $\theta_1 = 45°$ with respect to the normal of the 72 nm sample; the red continuous line represents the fitting to the experimental data, which are depicted by symbols. As it may be noted, by comparing the front and backside of the spectrum a straggling produced in the sample is clearly observed. The inset in Figure 1 illustrates the whole corresponding RBS spectrum.

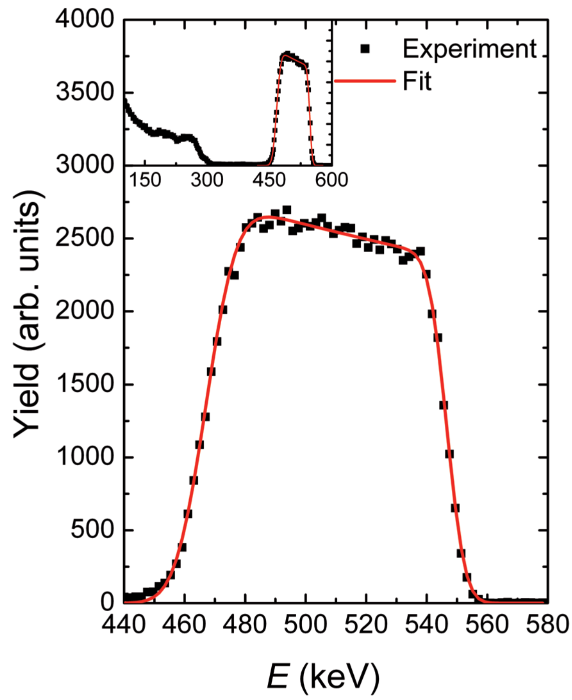

Fig. 1. (Color online) The inset displays a typical spectrum of a 165° Rutherford backscattering measurement from HfO_2 on Si tilted 45° with respect to a 600 keV He^+ beam (symbols), as well as the fitting (red continuous curve). The main panel depicts only the Hf signal of the RBS spectrum (symbols) and the corresponding fitting line, which shows the straggling effect. The sample thickness is 72 nm.

2.3 Data analysis and results

In order to determine the straggling values from the experimental spectra, we carried out a fitting procedure based in the following expression by [14]

$$H(E_1) = A \int_0^t dx \int_0^{KE_0} \frac{dE'}{E'^2} f\left(E_0, \frac{x}{\cos\theta_1}, E'\right) \times f\left(KE', \frac{x}{\cos\theta_2}, E_1\right), \quad (2)$$

where E_0 is the beam energy at the target surface, $H(E_1)$ is the number of particles scattered to the detector with energy E_1, A is a normalization fitting parameter, E_1^2 describes the energy dependence of the Rutherford backscattering cross section, K is the kinematic factor, θ_1 is the angle between the incident beam direction and the sample normal, θ_2 is the angle between the detection direction and the sample normal, and x is the depth in the film, of thickness t.

The function $f(E_0, x, E)$ can be approximated by the following Gaussian form:

$$f(E_0, x, E) = \frac{1}{\sqrt{2\pi\Omega^2 x}} \exp\left(-\frac{\left[E - \left(E_0 - \frac{dE}{dx}x\right)\right]^2}{2\Omega^2 x}\right), \quad (3)$$

where Ω^2, a fitting parameter, is the energy-loss straggling (in eV^2/Å) and dE/dx is the stopping power (in eV/Å). This Gaussian form is a reasonable approximation provided that the sample thickness t is above 10 nm [1]. Finally, equation (2) is convoluted with the system resolution. As it may be observed, from expressions (2) and (3) one can extract the value of the energy-loss straggling Ω^2 corresponding to each energy E_0 of the incident projectile.

3 Theoretical model

3.1 Dielectric formalism of the energy loss of swift projectiles

The energy loss of swift H and He ion beams through a HfO_2 target is evaluated within the dielectric formalism [15], which describes the passage of projectiles through matter to first order in perturbation theory. An important input data in this formalism is the energy loss function (ELF) of the target, which accounts for its electronic excitation spectrum in response to external electromagnetic perturbations. The combined use of the dielectric formalism together with a proper description of the target electronic properties is especially well suited for swift light-ion beams, which corresponds to the experimental situation discussed in this paper.

The MELF-GOS model, described elsewhere [12,13], has proven to be useful in modelling in a realistic way the ELF of targets with quite different electronic properties (metals, insulators and semiconductors), either elementals or compounds. As the details and methodology of the MELF-GOS model has been already published, in the following we only summarize the main results.

The dielectric formalism provides the following expression for the energy-loss straggling Ω_q^2 of a projectile with atomic number Z_1 and charge state q moving with velocity v through a target represented by the dielectric function $\varepsilon(k,\omega)$

$$\Omega_q^2 = \frac{2e^2\hbar}{\pi v^2} \int_0^\infty \frac{dk}{k} \left[Z_1 - \rho_q(k)\right]^2 \int_0^{kv} d\omega\, \omega^2 \mathrm{Im}\left[\frac{-1}{\varepsilon(k,\omega)}\right], \quad (4)$$

where $\rho_q(k)$ is the Fourier transform of the projectile electronic density, described here by the Brandt-Kitagawa statistical model [13,16], and $\mathrm{Im}[-1/\varepsilon(k,\omega)]$ is the energy loss function (ELF) of the target, which contains information about the probability of producing an electronic excitation of momentum ($\hbar k$) and energy ($\hbar\omega$) in the target.

Taking into account that the projectiles can have different charge states q while moving through the target, the total energy-loss straggling Ω^2 is obtained by properly weighting the respective contributions of each charge state, namely

$$\Omega^2 = \sum_{q=0}^{Z_1} \phi_q \Omega_q^2, \quad (5)$$

where ϕ_q is the probability for each charge state fraction, which depends on projectile type and velocity as well as

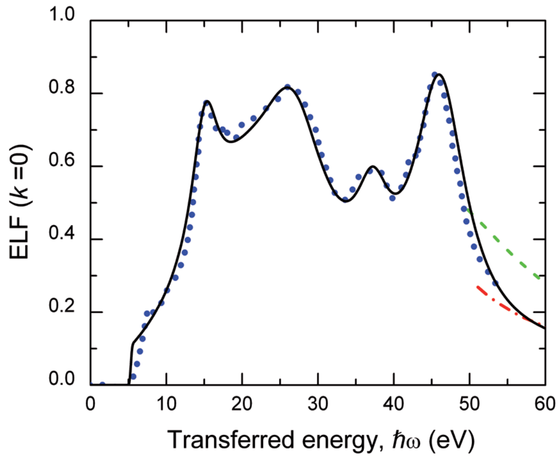

Fig. 2. (Color online) Energy loss function of HfO_2 as a function of the transferred energy, $\hbar\omega$, in the optical limit ($k = 0$); the black solid line is the ELF obtained through the MELF-GOS model, the blue dotted line represents experimental data from Frandon et al. [19], the green dashed line and the red dash-dotted line represent the data derived from X-rays scattering factors from Henke et al. [23] and NIST [24], respectively.

on the target nature. We use in this work the values of ϕ_q obtained from the CasP code [17]. For compound targets this code applies Bragg's additivity rule according to the charge state fractions of each component of the target.

3.2 Energy loss function of HfO_2

The ELF of the HfO_2 target is obtained by using the MELF-GOS model [12,13], which separates the contribution of the target electrons to the excitation spectrum into two parts, one due to the outer electrons (which accounts for aggregation effects, such as chemical bonds and phase state) and the other due to the inner-shell electrons (which preserve the atomic character of the target atomic constituents).

The former contribution to the target ELF is provided by a linear combination of Mermin-type ELFs [18] that fits the experimental ELF in the optical limit ($k = 0$). The ELF of HfO_2 in the optical limit ($k = 0$) obtained through the MELF-GOS method is represented in Figure 2 by a black solid line, which fits the ELF measured by Frandon et al. [19] using transmission electron energy loss spectroscopy at low transferred energy, $\hbar\omega$, depicted by a blue dotted line. A similar energy loss spectrum was recently obtained by Agustin et al. [20]. An interpretation of the ELF structure of HfO_2 in terms of collective excitations and electronic transitions can be found in references [20] and [21].

The contribution of the inner-shell electrons to the ELF, at high transferred energy, is analytically obtained from the generalized oscillator strengths (GOS) of the target atomic constituents; adopting hydrogenic GOSs instead of more embarrassing numerically computed GOSs has proven to be an acceptable and useful approach [22]. For the HfO_2 target we consider as inner-shells the K-, L- and M-shells of Hf and the K-shell of O, because their ELF display an atomic character in the excitation spectrum, as can be derived applying Bragg's rule to the X-rays scattering factors of Hf and O, from Henke et al. [23] and NIST [24] data.

The resulting ELF must fulfill the f-sum rule [25,26], which provides the effective number of electrons per atom (or molecule in a compound target) participating in the excitation for a given transferred energy $\hbar\omega$,

$$N_{eff}(\hbar\omega) = \frac{m}{2\pi^2 e^2 N} \int_0^{\omega} d\omega' \omega' \mathrm{Im}\left[\frac{-1}{\varepsilon(k=0,\omega')}\right], \quad (6)$$

where m is the electron mass and N is the atomic (or molecular) density of the material. In the limit when $\hbar\omega \to \infty$, N_{eff} must tend to the total number of electrons per atom (or molecule) of the target for all transferred momentum.

Besides the good overall, as well as detailed, agreement of our fitting procedure, the resulting ELF satisfies the previously mentioned f-sum rule, where the effective number of electrons excited up to an energy $\hbar\omega$, N_{eff}, displays the corresponding shell structure of the HfO_2 atomic constituents, and at large enough values of $\hbar\omega$, N_{eff} tends to 88, which is the total number of electrons in the HfO_2 molecule.

4 Results

The Bohr straggling Ω_B^2 for compounds can be obtained, in a first-order approximation, by adding the Ω_j^2 values corresponding to each one of the j-atomic constituents of the compound target, according to their stoichiometric proportions, similarly to the so-called Bragg's additivity rule for the stopping power of compound targets [6]. Therefore for a compound A_xB_y, the Bohr straggling $\Omega_B^2(A_xB_y)$ will be,

$$\Omega_B^2(A_xB_y) = N(A_xB_y)\left[x\frac{\Omega_B^2(A)}{N(A)} + y\frac{\Omega_B^2(B)}{N(B)}\right] = 4\pi Z_1^2 e^4 \left[xZ_2(A) + yZ_2(B)\right] N(A_xB_y) \quad (7)$$

where $N(A_xB_y)$ is the molecular density of the compound target, $N(A)$ and $N(B)$ are the atomic densities of each element of the compound. The Bohr straggling for each element, $\Omega_B^2(A)$ and $\Omega_B^2(B)$, is given by equation (1). The Bohr straggling deduced from equation (7) for HfO_2 is represented by a horizontal blue dashed line in Figures 3 and 4. In this calculation we assume a mass density for HfO_2 of 9.68 g/cm^3, resulting in a molecular density $N(HfO_2) = 2.77 \times 10^{-2}$ molec/Å^3. Since the Bohr straggling is deduced on the assumption that all target electrons can be considered as free, it is expected to be valid only at large projectile energies.

Figures 3 and 4 display, as a function of the projectile energy, the results of the present energy-loss straggling

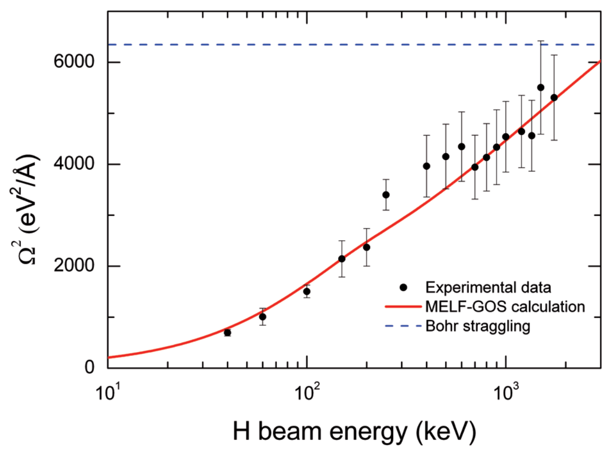

Fig. 3. (Color online) Energy-loss straggling of a H ion beam in HfO_2 as a function of the beam energy. The symbols stand for the experimental results, the red continuous line represents our theoretical calculation and the blue dashed line corresponds to Bohr straggling.

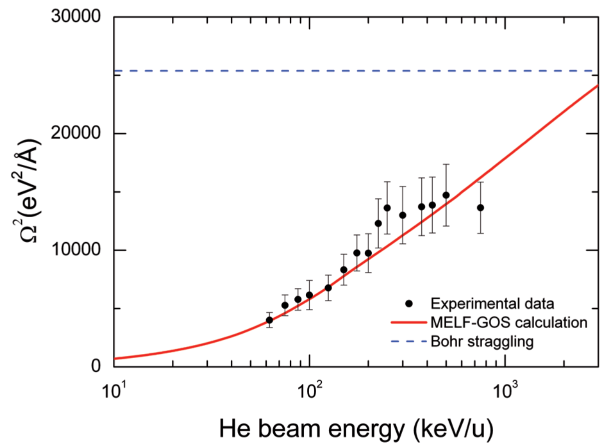

Fig. 4. (Color online) Energy-loss straggling of a He ion beam in HfO_2 as a function of the beam energy. The symbols stand for the experimental results, the red continuous line represents our theoretical calculation and the blue dashed line corresponds to Bohr straggling.

measurements of HfO_2 for H^+ and He^+ beams respectively. Each data point corresponds to an average over at least six independent measurements (as described in Sect. 2) and the error bars were calculated following a statistical treatment of the individual values. In addition we have taken into account the errors in the determination of the film thickness which are of the order of 5% of each determined value.

The solid red curves represent the calculations of the MELF-GOS model discussed in Section 3. Most of the contribution to the energy-loss straggling comes from the outer electrons. The contribution of the inner shell electrons increases with energy and represent, for proton and alpha projectiles, approximately a 7% and 11% of the total straggling at 1 MeV/u and at 5 MeV/u, respectively.

As can be observed for both projectiles, the Ω^2 values increase with energy in the studied interval range, and the Bethe-Livingston shoulder [27] or overshooting at intermediate energies does not appear in the theoretical results and it seems not to be present in the experimental data. For both H and He projectiles an overall good theoretical-experimental agreement is observed. This is particularly true for He where, despite the large energy interval, only few experimental points do not fall on the theoretical curve.

This good agreement can be understood by the realistic description of the dielectric properties of HfO_2 that take into account its complex excitation spectrum including collective excitations and interband transitions. In this case a simple electron gas model is not acceptable.

5 Summary

We have determined for the first time the experimental energy-loss straggling of proton and alpha particle beams in HfO_2 films as a function of the incident energy, for a broad energy range. The experimental data were obtained using both techniques, MEIS and RBS. A theoretical calculation of the energy-loss straggling Ω^2 was done in the dielectric framework, using as an input the energy loss function of the HfO_2 target in the whole momentum-energy excitation space, obtained by a suitable fitting to experimental data and constrained to fulfil physical restrictions. The calculated and the experimental energy-loss straggling of HfO_2 for H and He projectiles agree quite well in the projectile energy range analysed here.

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