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On the effect of oxygen on the creation of colour centres in swift heavy ion-irradiated AlN

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Abstract

We readdress the nature and creation mechanism of the defects absorbing at 4.7 eV in Swift Heavy Ion (SHI)-irradiated AlN semiconductor. Irradiations are performed in different controlled atmospheres (vacuum, oxygen, helium or argon partial pressure) to investigate the coupled effects of (i) the synergy between electronic excitations (S_e) and nuclear collisions (S_n) for point defect creation and (ii) SHI-induced impurity introduction/diffusion into the AlN layer. We found that the synergy, previously reported, between S_e and S_n for defect formation must also be related to the presence of oxygen during irradiation. At the same S_e , the synergy parameter is indeed increased by up to two orders of magnitude during irradiation under O_2 compared to irradiation under vacuum. By SHI irradiation and by a fine-tuning of the irradiation atmosphere, we then control the activation or inhibition of these light-emitted point defects (oxygen-vacancy complexes) which can be used as qubits for applications in quantum computing.

Keywords: Nitride semiconductors, Swift Heavy Ions, color center, electronic excitations, synergy, nuclear collisions

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1. Introduction

The family of nitride semiconductors is considered for many applications in the emission and detection of light in the blue and the ultraviolet [1]–[3] domains. In particular, AlN with its wide bandgap is used as a native substrate for UV LEDs. As such, attention is paid to the formation of light absorbing defects, especially those with a deep UV absorption band at 4.7 eV that can hinder the development of application [4]. Moreover, the corresponding defects are also proposed as qubit centers for application in quantum information technologies [5], [6]. Therefore, the identification and introduction/removal of these defects must be well controlled. The creation of these deep UV-absorbing defects can occur either during the sample growth and depends on due to the presence of impurities [7]–[10] or by irradiation. Collazo et al. [10] established a linear relation between the intensity of the 4.7 eV absorption band and the concentration in carbon impurity. Furthermore, based on density functional theory calculations, they found that substitutional carbon on the nitrogen site (C_N) might explain the observed absorption, as later on supported by the work of Alden et al. [11]. But, very recently, Jin et al. [12] concluded as well experimentally as theoretically that the influence of C_N is in fact limited and nitrogen vacancy would be the dominant defect. Complexes of an aluminum vacancy and oxygen impurity are also proposed to explain the absorption band in AlN containing high levels of oxygen impurities [9], [13]. In fact, the defect nature is still a controversy. Depending on the concentration of element impurities (such as carbon and oxygen, which are the

most common impurities) during the sample growth, the absorbing defects could be related either to an O or a C complex [14], [15].

In UV transparent AlN, the absorption band at 4.7 eV also appears after ion irradiation [16]–[18]. Ion implantation is widely used in semiconductors to achieve doping or to incorporate optically active elements [19], [20]. In this context of low energy ion irradiation, defect creation mostly stems from elastic collisions (S_n). When these materials are used in the outer space, they are subjected to Swift Heavy Ions (SHI) irradiation from cosmic rays and defects mainly stems from electronic excitations (S_e). Synergy between S_e and S_n for defect creation is possible and is being actively studied in different materials. A S_e -induced annealing of defects created by elastic collisions is the most reported synergy mechanism [21]–[23]. There are very few studies where the amount of defects is increased by a combination of S_e and S_n : point defect in iron [24] and more recently in the case of S_e -induced ion track formation in materials pre-damaged by elastic collisions [25], [26]. In irradiated AlN, we demonstrated a positive S_e/S_n synergy for the creation of defects absorbing at 4.7 eV [16], [17]: elastic displacements are required for defect creation but a huge enhancement is induced by the electronic excitations.

The combination the S_e/S_n synergy and oxygen impurities for the creation of the absorbing defects is studied here. ~~In this paper, we study~~ We investigate under ion irradiation both the creation of point defect in AlN and the radiation-induced impurity diffusion of impurities into the AlN layer, as well as their coupled effects for the creation of deep UV absorbing defects.

2. Experiment

Experiments were performed on either 2- μm or 0.5- μm thick AlN layers. Samples were cut from wafers grown by HVPE on a c-plane sapphire substrate, provided by TDI Oxford Instruments (Silver Spring, USA) or by Nitride Solutions Inc. (Wichita, USA). Most of the irradiations were carried out under either vacuum or controlled atmosphere (oxygen, helium or argon partial pressure) with a specific setup that allows irradiating under various gas pressures and *on-line* analyses. In some cases, sequential irradiations with different beams were done on the same sample. All irradiations were performed at 300 K, using the GANIL accelerator (Caen, France) with beams indicated in Table 1. During irradiation, the ion-beam flux was kept low enough to prevent sample heating. Irradiations and measurements done in previous work [16], [17] were mostly performed at 15 K. However, we had checked, by performing a few room temperature irradiation, that, under vacuum, this change in irradiation temperature do not influence the point defect creation. This is consistent with the very weak irradiation-temperature effect on damage buildup in AlN for temperatures from 25 K to 300 K [27], [28].

Before and after each irradiation, optical absorption spectra were collected in the 190-900 nm range with a resolution of 2 nm using a UV-visible Varian Cary 300 spectrometer. Isochronal annealing (15 min) up to 1500 K was performed under vacuum on an irradiated AlN sample.

Table 1: Irradiation conditions: ion and energy, σ_d is the mean elastic displacement cross-section over the AlN sample thickness calculated using SRIM-2008 [29] in full cascade calculation mode with threshold displacement energies of 25 eV and 28 eV for Al and N, respectively. S_e is the mean electronic stopping power over the AlN thin layer, ~~ENSP is the S_e/S_n ratio at the sample surface with S_n the nuclear stopping power.~~ The asterisk and double asterisk indicate that, for these high energy ^{129}Xe irradiations, in addition to under vacuum irradiations, extra irradiations were performed under Ar or O₂ gas atmosphere (), and under He atmosphere (**). In those cases, the beam has crossed a 15 μm -thick steel window or a 5 μm -thick havar window, respectively. The indicated energies are calculated at the entrance of the AlN sample.*

Ion - Energy	σ_d (10^{-16} ion/cm ²)	S_e (keV/nm)	ENSP
^{129}Xe – 14 MeV	6.5	4	10
^{129}Xe – 92 MeV	1.1	16.5	130

^{238}U – 106 MeV	3.6	17.1	40
^{208}Pb – 930 MeV	0.5	33	540
^{129}Xe – 370 MeV*	0.2	23	550
^{129}Xe – 800 MeV**	0.2	23	1030

3. Results and discussion

3.1. Point defect annealing

Fig. 1 (a) shows the absorption band at 4.7 eV in AlN samples irradiated with ^{238}U 106 MeV at room temperature at a fluence of 5×10^{12} ions/cm² after isochronal annealing at different temperatures. On Fig. 1 (b), we compare the temperature-dependent evolution of the normalized area of this absorption band area with that of Al_{0.6}Ga_{0.4}N irradiated with multi-energetic protons reported in literature by Zhou et al. [18]. The defects in the two materials can be compared as we demonstrated in our previous works [16], [17] that they are created both in AlN and in Al_xGa_{1-x}N by the same mechanism for the different irradiation particles.

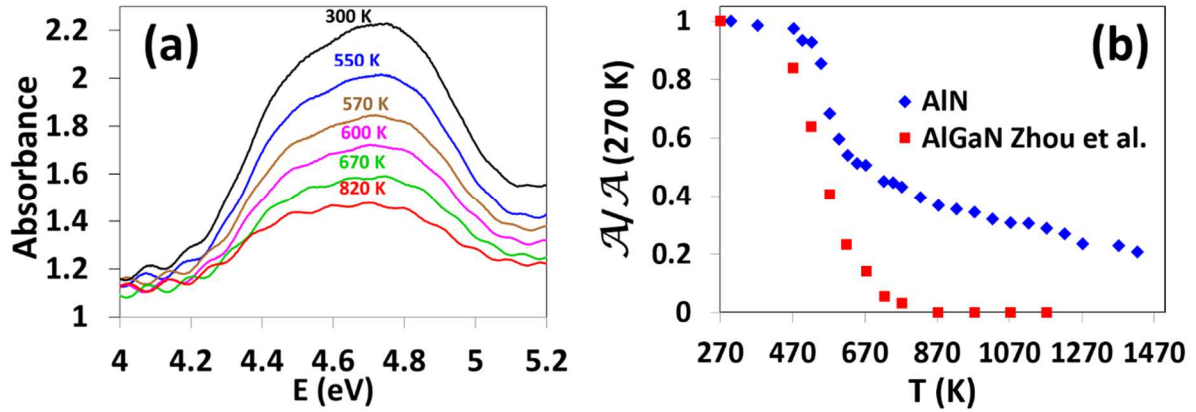


Fig. 1. a) Absorption band at 4.7 eV in ^{238}U 106 MeV irradiated AlN/Sapphire after 15 min-annealing at different temperature. The shoulder near 4.8 eV that disappears between 300 K and 600 K, is due to the contribution of F^+ centers in the sapphire substrate [30]. b) Isochronal (15 min) annealing of the absorption band at 4.7 eV in AlN (this work) and Al_{0.6}Ga_{0.4}N [18].

The single annealing stage in Al_{0.6}Ga_{0.4}N (400 K – 700 K) was attributed to nitrogen vacancies (V_N). In AlN, the curve presenting the normalized area (i.e. defect density) of the absorption band as a function of the annealing temperature presents a two-stage evolution. The first stage, up to 700 K, is congruent in shape with the one observed in the same temperature domain in Al_{0.6}Ga_{0.4}N except the normalized area at 700 K is as high as 40 %, the low contribution of the F^+ centers from the irradiated sapphire substrate also disappears in this temperature range [31], [32]. The second stage is linear and the total defect density does not fall to zero at temperatures as high as 1470 K, even when the sample is kept 96 hours at this temperature. This stage may be related to another type of defects created by irradiation or to a defect resulting from the annealing of the V_N (400 K – 700 K) and absorbing at a similar energy. In any case, the whole absorption band cannot be attributed solely to nitrogen vacancies. The oxygen and/or carbon complexes suggested for explaining the absorption band in non-irradiated AlN could also be at the origin of this absorption band in irradiated AlN.

3.2. S_e and S_n synergy: role of oxygen atoms

The synergy between S_e and S_n reported for the creation of the defects absorbing at 4.7 eV under SHI was demonstrated in [17] and modeled using equation (1):

$$A = A_\infty (1 - e^{-k\beta\sigma_d\Phi}), \quad (1)$$

where A is the area under the absorption band (i.e. defect concentration), A_∞ the area at saturation, $\sigma_d\Phi$ the number of displacements per atom (dpa), and $k\beta$ is the synergy parameter dependent on S_e .

The evolution of the defect concentration as function of the dpa number for irradiations at different S_e either under oxygen atmosphere or under vacuum are shown in Fig. 2 (a). Irradiation under oxygen atmosphere hugely improves the creation of optical defects. Under vacuum, at a given dpa number, the defect concentration, at a given dpa number, increases when S_e increases and the synergy parameter (at 300 K) follows the exponential-like S_e -dependence already observed under vacuum at 15 K [17]. Under 1-bar oxygen atmosphere in the irradiation chamber, the synergy parameter is shifted from this exponential curve and increases by two orders of magnitude. Therefore, the synergy parameter, so far considered as only dependent on S_e , must be closely linked to the presence of oxygen during irradiation (Fig. 2 (b)). The effect of oxygen atmosphere in the creation of the absorbing defect under irradiation is a strong indication that the defect at the origin of the 4.7 eV band is an oxygen complex. This implies that a sufficient amount of oxygen atoms is present in the sample and that they are able to recombine with the radiation induced vacancies.

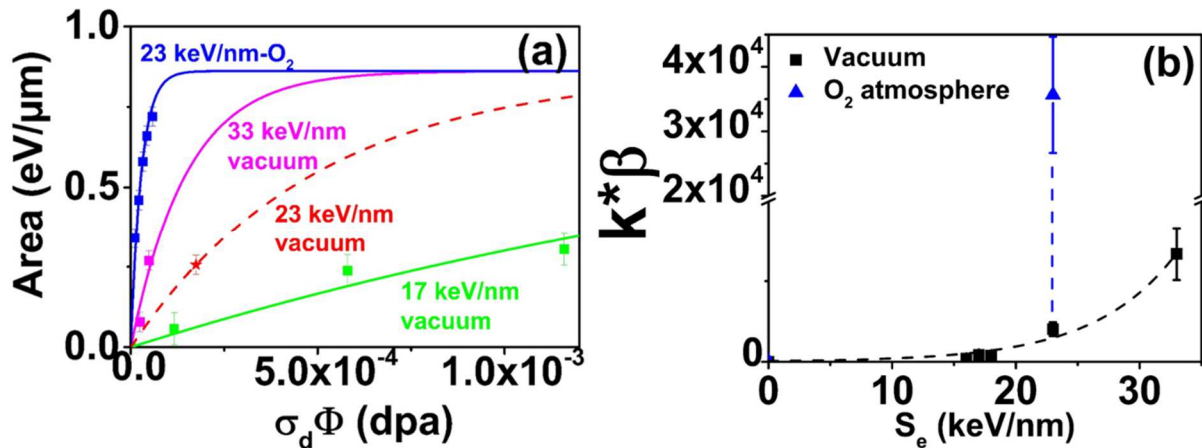


Fig. 2. (a) Evolutions of the absorption band area as function of the dpa number in the AlN layer for irradiations under vacuum or under O₂ atmosphere. The continuous lines represent the results of the fit using the model in equation (1). (b) Evolution of the calculated synergy parameter ($k\beta$) as a function of the electronic stopping power (S_e). The dashed lines are used to guide the eye.

We postulate that, under dioxygen atmosphere, irradiation promotes both the incorporation of oxygen atoms in the sample and their recombination with vacancies by a radiation-enhanced diffusion. Such a radiation-enhanced diffusion has been observed in irradiated Fe [33], [34] and is principally due to electronic excitations. Furthermore, SHI irradiation could promote higher solubility of oxygen into AlN in a non-equilibrium process by a transient local heating. According to the thermal spike model, with 330 MeV ¹²⁹Xe ion (S_e at 23 keV.nm⁻¹) for instance, due to electronic excitations and subsequent electron-phonon coupling, the temperature along the ion path could reach 10⁴ K [35]. In this process for oxygen-vacancy complex formation, the diffused oxygen stabilizes point defects created by irradiation that tend to recombine due to the important dynamic annealing observed in AlN during irradiation with the formation of transient point defects [36].

Even if the concentration of vacancy-oxygen complexes is much smaller under vacuum than under oxygen irradiation (Fig. 2), the amount of point defects is however very significant under vacuum.

This is surprising when considering the potential sources of oxygen. Oxygen could come from the residual vacuum (10^{-6} mbar, mainly of H_2O molecules), surface adsorbates and oxygen contaminants initially present within the AlN matrix. Irradiation probably induces incorporation, by dissociation, of residual-gas molecules and/or adsorbates and enhances diffusion in AlN of incorporated or already present oxygen atoms. The relative contribution of these different potential oxygen sources is still unclear. Besides, the creation of the absorption band is not dependent on whether the irradiation is done under ultra-high vacuum (10^{-8} mbar) at low temperature (15 K) [17] or, as in the present work, under secondary vacuum at room temperature (10^{-6} mbar). At low temperature, the residual-gas source of oxygen is probably negligible. However, a thin layer of water ice mostly likely covers the samples.

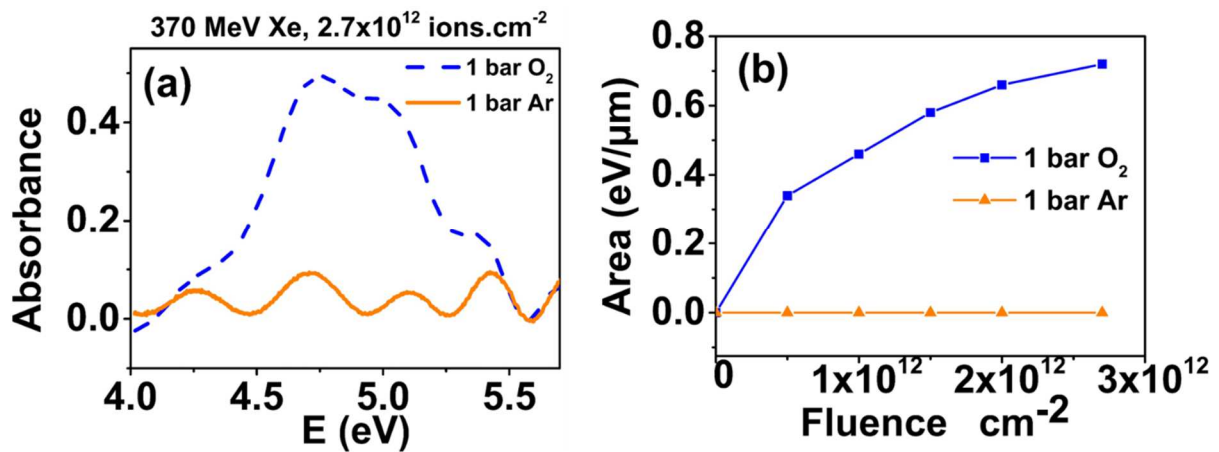


Fig. 3. (a) Absorption band at 4.7 eV in AlN irradiated with 370 MeV ^{129}Xe ion at 2.7×10^{12} ions. cm^{-2} either under O_2 or under Ar atmosphere. (b) Evolution of the area of the absorption band as a function of the fluence.

Fig. 3 compares the creation of the optical defects with irradiation under 1 bar O_2 atmosphere and under 1 bar Ar atmosphere. Clearly, irradiation-induced Ar diffusion into AlN inhibits the optical defect creation. Though the absorption band area (i.e. defect concentration) increases with the fluence in the case of irradiation under O_2 atmosphere, no absorbing defect is created with irradiation under Ar. Similar effect is observed with irradiation under He gas atmosphere, where the synergy parameter for defect creation is near zero (Fig. 4). In the case of He, the small value of the synergy parameter could be due to possible velocity effect as irradiation for the same S_e was done with much higher ion energy.

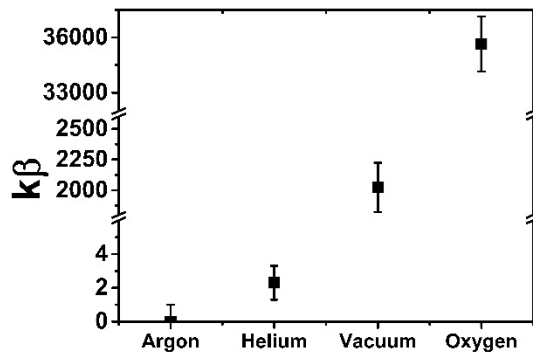


Fig. 4. Calculated synergy parameter for irradiation with $S_e = 23$ keV. nm^{-1} in noble gas atmospheres (He and Ar), O_2 atmosphere and in vacuum.

The effect of the different gases on the formation of the color centers is discussed by an analogy to the Chapman cycle [37]. O₂ molecules in the irradiation chamber would be split by electronic excitations brought by the ion beam and subsequently generating ozone [38], [39] according to the following reactions:



The lifetime of the atomic oxygen (in the range of second) is 500 times shorter than that of O₃ [40]. The oxygen atoms diffusing into the AlN layer probably comes from the dissociation of O₃ after collision with the AlN surface. The diffusion rate depends on the amount of O₃ which is also controlled by the radiolytic dissociation:



Under high concentration of O₂, the resulting oxygen atoms (equation 2) quickly react with O₂ molecule (equation 3), thus maintaining the ozone production cycle, and a steady-state ozone concentration. With irradiation under 1 bar Ar, He or 10⁻⁶ mbar vacuum atmosphere, the amount of residual contaminants O₂ and/or H₂O (<0.1 ppm and <10⁻³ ppm for O₂, respectively) is too low to maintain the ozone production cycle. This would explain, in comparison with irradiation under 1 bar O₂ atmosphere, a lower diffused oxygen atoms into the AlN layer and lower concentration of oxygen-complex optical defects. In the case of Ar, the production of optical defects is negligible despite a higher level in residual contaminant molecules compared to the vacuum. This would be assigned to a shorter lifetime of radiation-induced ozone molecules due to multiple collisions with Ar molecules. Under lighter noble gas atmosphere (i.e. He), the effect is lesser. Indeed, we observe the formation by irradiation of the absorption band, but the calculated synergy parameter is still far below the one obtained during irradiation under vacuum (Fig. 4).

Besides the decomposition of O₃ molecules due to collisions with the dense population of noble gas, the occupation of oxygen site by Ar or He to form non-absorbing Ar or He-complexes instead of oxygen related complexes would limit the appearance of the absorption band. The formation of noble gas-vacancy complexes (for instance He-vacancy) has been used to explain the damage build-up in irradiated AlN [41].

3.3. Sequential irradiations

Sequential irradiations were performed under vacuum on AlN with a low energy ion resulting principally in the creation of point defects by elastic collisions, followed by a high-energy ion irradiation with a high S_e. The defect concentration (i.e. absorption band area) after high-energy ion irradiation favoring oxygen diffusion is not enhanced by the pre-existence of point defects (Fig. 5). The contribution of the high-energy ion irradiation on the band area in pre-irradiated sample is equal or even smaller (due to the tendency to saturation¹) than the one in virgin sample. For this new type

¹ The damage kinetics is governed by a zero-order creation and first-order annihilation. The efficiency for defect creation decreases when the irradiation dose increases.

of synergy implying the diffusion of impurity element, the effects of S_e/S_n sub-synergy² for vacancy creation and S_e -induced oxygen diffusion should be simultaneous.

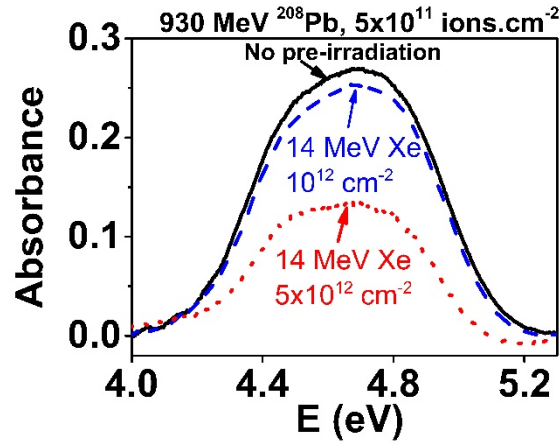


Fig. 5. Contribution of 930 MeV ^{208}Pb , 5×10^{11} ions. cm^{-2} irradiation on the absorption band in as-grown AlN and in AlN pre-irradiated with 14 MeV ^{129}Xe at different fluences.

4. Conclusion

SHI irradiations under different controlled atmospheres (vacuum, oxygen, argon and helium) were performed in AlN to investigate the influence of both S_e/S_n synergy for vacancy creation and S_e -induced impurity introduction/diffusion into the AlN layer for the creation of optical point defects absorbing at 4.7 eV. We found a new effect of the irradiation atmosphere on the color center production. The diffusion of oxygen by S_e coupled to point defect (vacancy) creation by irradiation hugely improves the formation of optical defects, supporting the idea that the absorbing defect nature is likely an oxygen-vacancy complex. The previously reported S_e/S_n synergy should also be linked to the presence of oxygen. The enhancement by oxygen diffusion of the color center production is up to two order of magnitude. Such an effect of oxygen that stabilizes vacancies created by irradiation in AlN by the formation of optically active oxygen related complexes was never reported before. This work gives the opportunity to control the activation or inhibition of light-emitted point defects that can be envisaged as qubits for quantum computing applications [6].

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² Considering that the transition responsible for the absorption band is completely allowed, i.e. oscillator strength equal to unity, we have calculated, by using the Smakula formula [42], the minimum concentration of vacancy in AlN irradiated with 106 MeV ^{238}U at a fluence of 5×10^{12} cm^{-2} . The number of optical defects is ten times higher than the number of dpa. This means that vacancy creation by elastic collisions is supported by electronic excitations for example by a S_e -induced weakening of the atomic bonds or by a transient heating in the ion wake.

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