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# Recoil-ion fractions in collisions of keV $Ar^+$ and $Kr^+$ ions with clean and adsorbate covered GaAs(1 1 0) surfaces

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## Abstract

Ion scattering and recoiling spectroscopy with time of flight analysis is used to study the ion fractions of Ga and As atoms recoiled in collisions of 5 keV $Ar^+$ and $Kr^+$ with clean GaAs(1 1 0) and with GaAs(1 1 0) covered with H, alkali metals (K and Cs) and fluorides ($AlF_3$). For the case of the clean surface, the Ga ion fraction is positive, large (~50%) and independent of the projectile type. The As ion fraction is also positive, low for $Ar^+$ (<10%) and relatively large (25%) for $Kr^+$ projectiles. The adsorption of H produces slight changes in both the As and Ga ion fractions, which is in agreement with the adsorption model where H reacts with both As and Ga atoms. The adsorption of alkalis produces strong changes in the ion fractions. At the beginning of the alkali adsorption the neutralization of Ga recoils increases fast with the coverage and follows approximately the variation of the work function. At coverages above half of the saturation value, where the work function has attained a stable value, the ion fraction in Ga remains low (~10%) and stable while that in As changes, the positive ion fraction decreases and the negative ion fraction increases. This behavior is related to the preferential adsorption sites and the modification of the electronic structure at the surface. The adsorption of $AlF_3$ produces no change in the Ga and As ion fractions for the whole range of coverages investigated (up to full coverage), supporting a non-dissociative and weak reacting model for adsorption.

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## 1. Introduction

Charge exchange processes during interaction of keV ions with clean and adsorbate-covered surfaces have been studied extensively over the past decades. The interest comes from the necessity to understand these processes in detail in order to apply quantitatively ion scattering spectroscopy (ISS) and because a thorough knowledge of these processes could contribute to the understanding of complex problems of reactions of molecules at surfaces. While a large number of experiments has been dedicated to study the charge state of projectiles scattered from metal surfaces [1–4], the case of semiconductors and insulators has been less

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investigated [5–9]. In this work we study the ion fractions in substrate recoiling atoms resulting from collisions of 5 keV $Ar^+$ and $Kr^+$ ions with clean and adsorbate covered (H, Cs, K, $AlF_3$) GaAs(1 1 0) surfaces. We focus the study on the Ga and As direct recoils (DR) because it is expected that they should present a clearer dependence on the local electronic structure and its modifications upon adsorption. The ion fractions in scattered particles and adsorbates will be discussed elsewhere. Some interesting aspects of the GaAs surface are: (1) its clean surface has been characterized by many surface analysis techniques, details of its electronic structure and relaxation are known, however, for the adsorption cases studied here, H, alkalis, and fluorides, there are some controversies not yet clarified. (2) It is an open surface, allowing, at some incident directions, scattering from first layer atoms without major contributions from deeper layers and without shadowing and focusing effects [10] and at other directions it allows strong focusing onto second layer atoms. (3) The similarity of the As and Ga masses should make the analysis of the ion fractions simpler because effects due to the outgoing velocity and detection efficiencies should be similar to both elements. (4) The different electronic structure around Ga and As atoms should be reflected in their ion fractions, as it has been observed before in the sputtering process [11]. Despite these interesting aspects, studies of charge exchange for the GaAs surface are scarce [8,11,12].

## 2. Experimental methods

The measurements were performed using the Bariloche setup [10,13], which consists of an ion accelerator connected to three collision chambers equipped with facilities for electron spectroscopy induced by ions and electrons and time of flight ion scattering and recoiling spectroscopy (TOF–SARS). The base pressure in the collision chamber used for these measurements is $\sim 2 \times 10^{-10}$ Torr and remains in the low ten range when the ion beam line is open. The ions are generated in a radio frequency source, accelerated in the range of 3–100 keV and then mass selected and collimated to 0.1° of angular divergence. For TOF measurements the ion beam (5 keV $Ar^+$ and 5 keV $Kr^+$ in this case) is pulsed at frequencies around 30 kHz. The sample is mounted on a manipulator that allows variation of the ion incident direction with respect to the surface plane ($\theta$) or to a main crystallographic axis ($\phi$) (inset of Fig. 1). Ions and neutral atoms scattered from the sample are detected with a channeltron electron multiplier located at a scattering angle of 45° (at the relatively large energies (~2500 eV) of the Ga and As recoils measured in this work the channeltron efficiency for ions and neutrals should be similar). A deflector plate located in front of the channeltron, which can be polarized either positive or negative, is used to deflect the scattered ions. The geometry of the apertures located in front of the channeltron can be arranged in such a way that ions having the same polarity than the deflector plate do not enter the detection region, while those having the opposite polarity do enter and are detected together with the non deflected neutrals. This geometry allows us to differentiate positive from negative ions with one channeltron. For the measurements discussed here most of the ions are positive, the only case where a fraction of negative ions was detected corresponds to As direct recoils coming from the

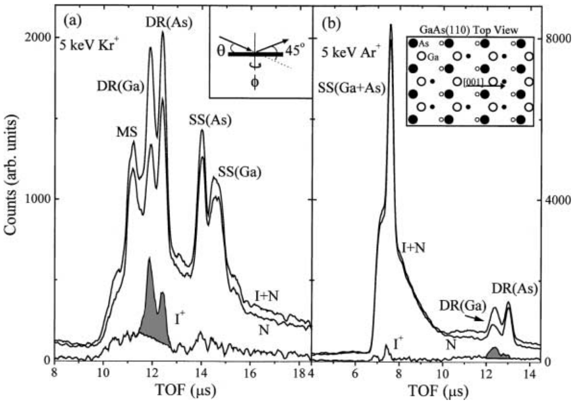

Fig. 1. Time of flight spectra for neutral plus ions (N + I), neutrals (N) and positive ions (I) obtained for 5 keV $Kr^+$ (a) and 5 keV $Ar^+$ (b) scattering from a clean GaAs(1 1 0) surface. The insets show the angle definition and a top view of the GaAs(1 1 0) crystal. The labels indicate SS, MS and DR processes. The straight line on the (I) spectrum shows the background used to separate the DR peaks.

GaAs surface covered with large amounts of alkalis (close to the saturation value).

It is known that semiconductor surfaces can be strongly modified by ion bombardment. In our case this effect was reduced by: (1) the use of very low incident currents, with a typical dose required to measure an ion fraction of $\sim 10^{12}$ ions/cm$^2$, i.e. at least 2 orders of magnitude smaller than the amorphization dose and (2) preparing the surface before each measurement with cycles of grazing ($\theta \sim 2°$) $Ar^+$ bombardment at 20 keV and annealing at 450 °C. We have shown before [10] that this method produces a clean and very flat surface, crystallographically ordered with the well-known relaxation where the first As layer is ~0.7 Å above the first Ga layer.

The alkalis (K and Cs) depositions were performed at room temperature from a K (Cs) dispenser (SAES-Getters, Italy), carefully degassed and placed at 3.5 cm from the sample [14]. Aluminum fluoride depositions were performed in situ from a Knudsen cell carefully degassed. Details of the evaporation are described in [15]. Throughout this work we define one monolayer as $8.8 \times 10^{14}$ atoms/cm$^2$, which is the atomic density of the clean GaAs(1 1 0) surface.

## 3. Experimental results and discussion

### 3.1. Clean surface

Fig. 1 shows TOF spectra for neutrals plus ions (N + I), only neutrals (N) and only positive ions (I) acquired with 5 keV $Kr^+$ (Fig. 1(a)) and 5 keV $Ar^+$ (Fig. 1(b)) hitting a clean GaAs(1 1 0) surface at an incidence angle $\theta = 20°$ and along the [0 0 1] channels. We first describe the main features of the (N + I) spectrum induced by Ar bombardment. This spectrum is a fairly typical one in TOF–ISS; we can see that it is dominated by a strong quasi-single scattering (SS) peak coming from collisions with both As and Ga atoms. At the forward scattering angle used, 45°, it is not possible to separate the As from the Ga contribution. In contrast, the Ga and As DR peaks are clearly separated at larger TOF. The Ga DR peak appears broader because there are two isotopes ($^{69}$Ga and $^{71}$Ga) with similar abundance (40% and 60%, respectively). Possible contaminants (mainly H and C) would appear at the left side of the spectrum, within the range shown in the figure. We have shown before [13] that at this incident angle the observed recoils come mainly from the first layer, without major shadowing or focusing effects. This allows us to use the area of these peaks as a measure of the first layer composition; their variation upon adsorption, together with the variation of the DR adsorbate peaks, can be used to characterize the adsorption processes [13–16]. The SS and recoiling peaks are superimposed on large backgrounds coming from different sequences of multiple collisions (MS).

The spectrum taken for $Kr^+$ at the same conditions than for $Ar^+$ is less typical of TOF-SARS because here the projectile is heavier than all the target atoms. As a consequence, the scattering peaks are less intense than for Ar, scattering from As and Ga is separated, the multiple scattering (MS) peak is more clearly distinguished and appears far from the SS peaks and the Ga and As DR peaks appear dominant. These features enhance the visibility of the recoil peaks, while on the other hand, shadowing effects by light adsorbates are less strong.

Observation of the (N + I), (N) and (I) spectra of Fig. 1 confirms a basic feature of ISS: particles coming from quasi-single collisions have a larger probability to escape as ions [17]. As previously mentioned, in this work we focus on the study of the ion fractions in the Ga and As DR peaks (shadowed region in Fig. 1) and how they change upon adsorption. Comparison of the (N) and (I) DR peaks shows that neutralization of the ejected Ga and As atoms proceeds with very different probabilities. Although first layer Ga atoms are located ~0.7 Å deeper than As atoms, the neutralization probability for the Ga recoils is considerably smaller than for the As recoils. Integration of the DR peaks yields ion fractions of $\Gamma^{Ar}(Ga) \sim 0.5$ and $\Gamma^{Ar}(As) \sim 0.08$ for Ar projectiles and $\Gamma^{Kr}(Ga) \sim 0.5$ and $\Gamma^{Kr}(As) \sim 0.25$ for Kr projectiles. These values are weakly dependent on both the polar and the azimuthal angles (except at grazing outgoing trajectories). Since the masses and outgoing velocities of the direct recoils are similar, the large difference in the Ga and As ion fractions must be the result of electronic effects. In this system, two different electronic effects are easily identified: the different energy position of the 4p levels in Ga and As free (outgoing) atoms and the different local electronic structures at the surface. The first ionization energy is 6 eV for free Ga and 9.8 eV for free As, i.e. both levels are below the Fermi level and are resonant with the valence band. Both levels could then be populated by resonant charge transfer and by Auger capture during the outgoing trajectory. However, in bulk GaAs, the electron density around Ga atoms is lower than around As atoms and presents some ionic character [18]. This difference in the electron density is enhanced for top layer atoms by the surface relaxation process [19]. In the clean (relaxed) surface, the Ga atoms adopt almost planar $sp^2$ bonds, while the As atoms tend to a pyramidal $AsGa_3$ configuration and having more p-like character. This dehybridization can be related to a charge transfer from the Ga to the As surface atom. The Ga dangling bonds become emptied and the As states occupied [19,20]. This process of charge transfer between Ga and As surface atoms is consistent with the higher ion fraction of the Ga DR shown in Fig. 1. The *empty* $C_3$ surface state (due to Ga dangling bonds) evolves into the 4p Ga. This evolution takes place in front of the main gap, making neutralization not very efficient. On the other hand, the *occupied* $A_5$ states (due to As) evolve from just below the top of the valence band into the 4p As level, where resonant ionization should not be efficient. Due to the presence of the gap, one could also expect to observe $As^-$, as it has been observed in sputtering [11]. This is not the case for the clean surface, where, within the sensitivity of the DR technique, the As ions are positive. This point will be discussed later for the alkali-covered surface.

Although the correlation between ion fractions and local electronic structure is generally more easily established for target recoiling particles [21] than for scattered projectiles, where the atomic levels in the projectile play a critical role, dependences on projectile type may also be evidenced for recoiling particles [22]. The above description of the neutralization processes neglects the effect of the projectile on the local electronic structure of the surface. For the particular case of GaAs we have observed that the ion fractions in Ga recoiled by $Ne^+$, $Ar^+$ and $Kr^+$ ions at energies around 5 keV are always high and around 50%, while those for As atoms recoiled by $Ne^+$ or $Ar^+$ ions are below 10%. As discussed above, this is in agreement with the specific features of the electronic structure around Ga and As atoms and reveals little dependence with the projectile type. In contrast, As atoms recoiled by $Kr^+$ ions present an ion fraction value around 25%. Since the outgoing trajectory of the As DR produced by Ar and Kr projectiles is the same (same experimental conditions) and even their recoiling energies are similar, this effect should be related to differences in the close encounter. A calculation of the hybridization of projectile and surface atom levels and of the occupation of the respective atomic levels during the outgoing trajectory would be necessary to interpret this result. However, one could speculate that due to the local character of the occupied As dangling bonds an interpretation based on a free atomic collision could yield some hints. In this case, the Kr–As collision is quasi-symmetric [23] and because of the relatively short As–Kr distance of closest approach (∼1 au) strong promotion of the outer levels in the lighter atom (As) should take place. A similar effect in the ion-surface collision may reduce the population of the As dangling bond by electron transfer to either the conduction band or to Kr projectiles.

### 3.2. Adsorption of H

The study of H adsorption on GaAs(1 1 0) [13,16] has some experimental difficulties. First, since $H_2$ does not adsorb on the GaAs(1 1 0) surface, the molecule has to be first dissociated by using, for example, a hot filament close to the sample [24]. Due to the low efficiency of this process [16,24] large amounts of $H_2$ are required. TOF–SARS with Ne or Ar projectiles is an adequate technique to monitor the adsorption of H on GaAs because H, the possible contaminants and the substrate DR can be identified directly (inset of Fig. 2). Quantification of the H coverage is more difficult than for other heavier adsorbates

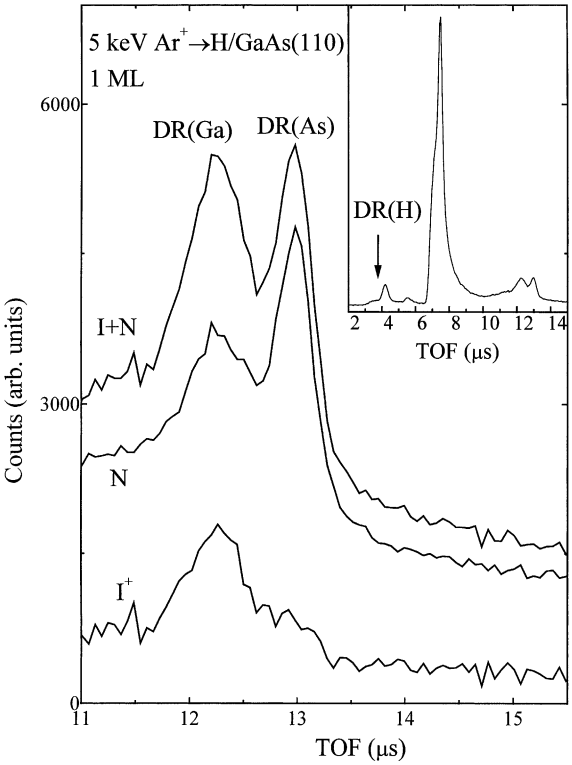

Fig. 2. Time of flight spectra for neutral plus ions (N + I), neutrals (N) and positive ions (I) obtained for 5 keV Ar$^+$ scattering from the hydrogenated GaAs(1 1 0) surface. Only the region corresponding to the Ga and As direct recoils is shown. The straight lines indicate the estimated backgrounds. The spectrum in the inset shows the total (N + I) contribution in the full range of TOF.

[13] because: (1) the channeltron efficiency decreases strongly for neutral H at a few hundred eV [25,26] and (2) H surface recoils are more difficult to separate from true H direct recoils [27]. The spectra of Fig. 2 were acquired with approximately one monolayer of H coverage. Since H is very light the reflected Ar and the Ga and As recoils are measured with almost no attenuation. The shape of the (N + I), (N) and (I) spectra of Fig. 2 is similar to that for the spectra of the clean surface. Integration of the Ga and As peaks, after a smooth background subtraction gives $\Gamma^{\mathrm{Ar}}(\mathrm{Ga}) \sim 0.45$ and $\Gamma^{\mathrm{Ar}}(\mathrm{As}) \sim 0.12$, which are slightly different from the values for the clean surface. Although these changes in the ion fractions are small and close to the experimental uncertainties (±5%), measurements performed at different H coverages, or with Ne projectiles, give the same trends, i.e. an increase of the As ion fraction and a decrease of the corresponding one for Ga.

Results from high-resolution electron energy loss spectroscopy have shown that H atoms bind to both As and Ga atoms from the beginning of the adsorption [28]. TOF–SARS experiments [13,16] indicate that part of the surface unrelaxes towards the ideal atomic structure and that even for relatively large amounts of H (of the order of one monolayer) there are important regions of the surface (~40%) that maintain the relaxation of the clean surface. Experiments of photoelectron spectroscopy [20] also suggest that at relatively high coverages (when surface disruption starts) not all the dangling bonds are saturated. Based on these results, one could expect that substrate atoms recoiling from clean (relaxed) surface regions will retain their ion fraction values, while those coming from unrelaxed regions (regions with adsorbed H) should be affected by the surface states induced by H [29] and by the surface charge rearrangement associated to the unrelaxation [30]. In particular, Santoni et al. [20] showed that there is an increase of the valence band photoemission around 5.2 and 7.2 eV that is attributed to combinations of H orbitals with Ga derived states and H induced localized states. Together with these structures the authors have observed a decrease in the photoemission intensity around 1–3 eV coming from As surface states ($A_5$). Our present experimental results indicate that the ion fractions in both recoils change from the beginning of the H adsorption, in agreement with the picture of H reacting with both substrate atoms. Even more, the experimental trends (higher ion fraction for As and lower for Ga) would be in agreement with the photoemission experiments, however, a careful evaluation of the contributions from surface states introduced by H would be necessary to confirm this hypothesis.

### 3.3. Adsorption of alkalis

Alkali metal/GaAs interfaces have been investigated extensively with many techniques over the last years mainly because of the behavior of alkalis as promoters of the oxidation process and because it was believed that these interfaces might be ideal systems to study Schottky barrier formation. However, these, systems still present several controversies regarding basic features, such as adsorption sites and the nature of the bonding. In a recent paper we have combined AES and TOF–SARS to study the adsorption of K on GaAs(1 1 0) [14]. The results for low coverages were consistent with a preferential adsorption of K along the [0 0 1] Ga rows, in a region close to the sites of a new As layer.

The insets of Fig. 3(a) and (b) show (N + I) spectra for a Cs and a K covered GaAs(1 1 0) surface, respectively. Calibration of the recoiling intensities yields coverages close to 0.25 of the saturation value for Cs and K (we will consider this as the low coverage case). The structures associated to the adsorbates are clearly seen when these spectra are compared to those of Fig. 1: the Cs DR peak appears at large TOF (Fig. 3(a)), while the SS from Cs appears overlapped with the MS peak, but sufficiently far from the Ga and As recoil peaks. The K DR and the SS from K (Fig. 3(b)) appear at similar TOF and are difficult to separate. The adsorption of Cs behaves similarly to the adsorption of K described above. At the beginning of the adsorption Cs atoms tend to form long chains along the [0 0 1] Ga rows [31] and then, at larger coverages, also adsorb (with a lower probability) close to first layer As atoms [14]. At large coverages the interface becomes disordered (long range disorder) [32]. At the low coverages indicated in Fig. 3, most of the Cs and K atoms are on the Ga rows and few on the As rows, which agrees with the stronger reduction of the Ga DR peaks seen in the (N + I) spectra of Fig. 3.

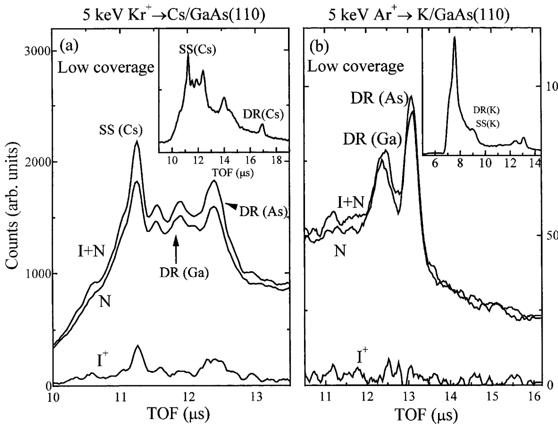

Fig. 3. Idem Fig. 2 for Kr$^+$scattering from GaAs(1 1 0) partially covered with Cs (a) and for Ar$^+$ scattering from GaAs(1 1 0) partially covered with K (b). Note the strong decrease of the Ga$^+$ DR peak.

The ion spectra (I) of Fig. 3 are quite different from those of Fig. 1, the As ion fraction is essentially the same than for the clean surface (this is more clearly seen for Kr projectiles where the As ion fraction is significative), while that of Ga has decreased from 0.5 to ~0.1. These results agree with the preferential modification of the electronic structure around Ga atoms up to medium coverages, leaving that around As atoms almost unchanged [33]. The effect of the alkali adatoms is to donate their valence s-electrons into the Ga sp$^3$-like orbital. Calculations indicate that the probability of finding the electron around the Ga core can reach values of ~90% [33] at very low coverages. This probability decreases with increasing coverage because the states become less localized and the interface more metallic. The electronic structure for Na adsorption [33] show the presence of surface bands (Ga $C_3$-like) that, in contrast to what is seen for the clean relaxed surface, appear inside the gap and are partially occupied at low coverages and fully occupied at high coverages. The Fermi level moves to the center of the gap and there is a strong reduction of the work function. These changes in the electronic structure should favour neutral initial states for the Ga recoils and should prevent the ionization of them during the outgoing trajectory. In case that a positive Ga ion is emitted as a consequence of the close encounter, its neutralization should be very efficient.

A comparison of the results of the ion fractions measured here with results of Auger electron spectroscopy [14] is possible. The energy shifts in the substrate Auger peaks [14] are consistent with a preferential reaction with Ga atoms at the beginning of the adsorption, in agreement with the fact that only the Ga ion fractions are modified at low exposures. At high coverages the As $M_4VV$ Auger line presents a considerable energy shift and

TOF–ISS indicates that the alkali atoms also adsorb near As atoms [14]. These measurements suggest that the electronic structure around As atoms is also modified at high coverages, and consistently, the As ion fractions should change. At high coverages we observe that the positive ion fraction in As recoils decreases and at the same time negative As recoils appear. Fig. 4 shows this effect for Ar on a high K covered GaAs (1 1 0) surface: the $As^-$ ion fraction reaches a value of 25% (the effect for Cs coverage is similar). If after the collision an $As^-$ atom is generated, its survival probability might be enhanced by the presence of the band gap. The fact that no $As^-$ is observed for the clean surface suggests that this probability is low. The high work function (5.5 eV for p-GaAs) and the position of the As affinity level (0.8 eV) should give a low survival probability. Nevertheless, in SIMS experiments [11,34], where a much higher sensitivity can be obtained, it is shown for the clean surface that there is indeed a fraction of $As^-$. The decrease in the work function by ~2.5 eV upon alkali adsorption, together with the presence of occupied states in the region of the gap (which tends to disappear at high coverages changing the character of the interface to more metallic [32]), may enhance the $As^-$ survival probability, as it is seen in the experiment.

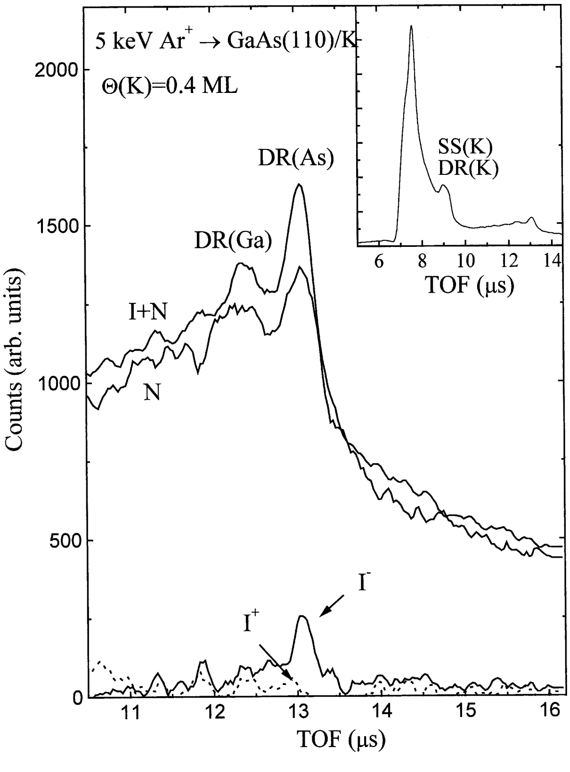

Fig. 4. Idem Fig. 3(b) for high K coverage. Note the increase of the $As^-$ DR peak.

### 3.4. Adsorption of $AlF_3$

The growth of thin insulator layers on semiconductors have attracted wide interest for their possible applications in MIS (metal-insulator-semiconductor) devices and inorganic resists for nanometer-scale patterning in electron beam lithography. In this part of the work, having as a reference a previous characterization of the growth of $AlF_3$ on GaAs(1 1 0) by TOF–SARS and AES [15], we discuss the effects of an adsorbed submonolayer of insulator on the ion fractions of Ga and As direct recoils. The inset in Fig. 5 shows a (N + I) spectrum for the GaAs(1 1 0) surface partially covered with $AlF_3$. The adsorption was carried on at room temperature [15]. This spectrum was taken under the same conditions than the (N + I) spectrum of Fig. 1(a). The main features are the decrease of substrate recoiling peaks and scattering peaks to about one half of its initial values and the appearance of the recoil peaks associated to F and Al. The previous study [15] indicates that, in contrast to the case of alkalis, the $AlF_3$ layer has no crystallographic order, even at the lowest coverages. For this reason the Ga and As DR intensities are similarly attenuated as a function of coverage [15]. This lack of order in the adsorbed layer produced a broadening of the associated recoils (F and Al) which are therefore not well resolved. The corresponding (N + I), (N) and (I) spectra in the region of the Ga and As DR peaks are shown in Fig. 5 for the surface partially covered with $AlF_3$. Integration of the Ga and As DR peaks yields values for the ion fractions of $\Gamma^{Kr}(Ga) \sim 0.5$ and $\Gamma^{Kr}(As) \sim 0.25$, which, within the errors of the integration process, are the values obtained for the clean surface. This trend in the ion fractions is maintained for the whole range of

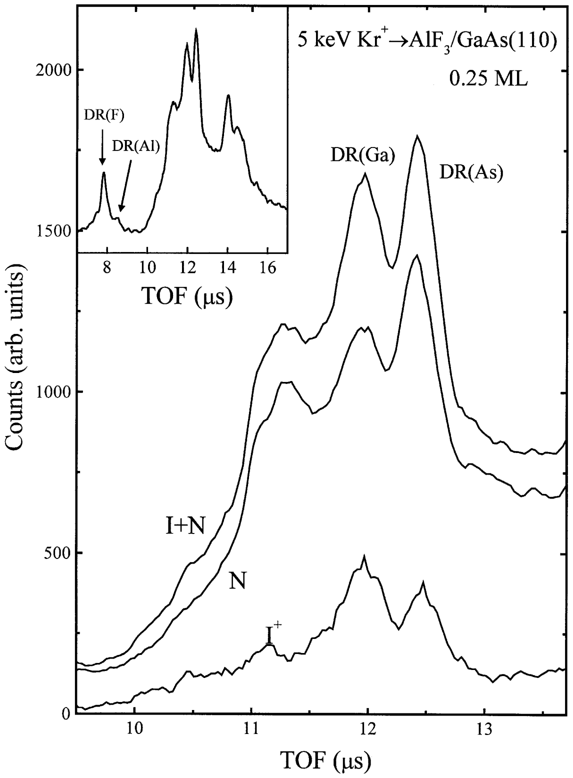

Fig. 5. Idem Fig. 2 for $Kr^+$ scattering from GaAs(1 1 0) partially covered with $AlF_3$.

coverages investigated, from a few percent of a monolayer to full coverage. It is known that metallic Al interacts strongly with GaAs, by replacing Ga atoms and it could also be expected that electronegative F atoms adsorbed on the surface have a strong effect on the ion fractions as it has been observed for O and Cl adsorption on Mg [35] and Ag [36], respectively. Therefore, the fact that the ion fractions in Ga and As DR are not modified by the presence of $AlF_3$ suggests that the molecule is not dissociated and that it reacts very weakly with the substrate.

## 4. Summary and conclusions

In order to compare the results we show in Table 1 the values of the ion fractions in Ga and As direct recoils obtained for the different cases investigated. As a summary of the results we can say: (1) the ion fractions for the clean surface are characterized by a large value of the $Ga^+$ ion fraction and by a stronger neutralization of As DR. (2) Adsorption of H produces slight changes in both ion fractions, a small decrease in the $Ga^+$ and a small increase in the $As^+$ fractions. (3) The strong reactivity of the alkalis is reflected in the changes produced in the ion fractions, which can be correlated to the preferential adsorption sites and the modification of the electronic structure around Ga. It increases the neutralization of Ga DR at low coverages and generates an important fraction of $As^-$ DR at high coverages. (4) The nonvariation of the As and Ga ion fractions upon adsorption of $AlF_3$ would be consistent with a model of adsorption where the molecule remains in the surface without dissociating and with little reaction with substrate atoms. We hope that these results will encourage theoretical studies of recoil ion fractions resulting from collisions of heavy ions with adsorbate covered semiconductors surfaces.

Table 1
Ion fractions in Ga and As direct recoils

| | Projectile | $\Gamma(Ga^+)$ | $\Gamma(As^+)$ | $\Gamma(As^-)$ |
|---|---|---|---|---|
| Clean GaAs | $Ar^+$ | 0.5 | <0.1 | <0.05 |
| Clean GaAs | $Kr^+$ | 0.5 | 0.25 | <0.05 |
| H:GaAs (1 ML) | $Ar^+$ | 0.45 | 0.12 | – |
| Cs:GaAs (low coverage) | $Kr^+$ | 0.1 | 0.25 | <0.05 |
| K:GaAs (low coverage) | $Ar^+$ | 0.1 | <0.1 | <0.05 |
| K:GaAs (high coverage) | $Ar^+$ | 0.1 | <0.1 | 0.25 |
| $AlF_3$:GaAs (0.2–1 ML) | $Kr^+$ | 0.5 | 0.25 | <0.1 |

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