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COLLISIONS OF FAST CLUSTERS WITH SOLIDS AND RELATED PHENOMENA

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Abstract

Fast ions, impinging on solid targets at a few MeV per atom, deposit energy in the material through electronic excitation processes. The relaxation of this energy induces emissions of photons, electrons, ions and neutral species from the target. The detailed study of these ejecta can give insight on the energy deposition and relaxation steps of the ion-solid interaction.

Fast polyatomic projectiles allow to deposit very high energy densities (at the surface and in the bulk) well beyond that being possible with any single ion impact. As a result, new and unexpected phenomena are observed which will be discussed : large non linear emission of ions and cluster ions, very high sputtering yields, production of giant tracks and craters in various irradiated materials including metals.

When entering the solid, the atomic constituents of the projectile remain in a close proximity for a certain distance, and the energy density deposited in the overlapping trajectories region is high enough to induce collective effects. Secondary ion emission experiments, performed with various carbon cluster beams delivered by the Tandem accelerator in Orsay, aim to determine this depth of spatial correlation by probing these collective effects inside the solid. The influence of the proximity of the cluster constituents on some of their properties such as their charge state distributions inside the solid has been studied and it has been shown that the mean charge state of the constituents is significantly smaller than when the atoms penetrate the target independently.

The collisions of atomic ions with various types of solids at an energy of tens of keV is a broad field of fundamental and applied research that has started long ago. The sputtering mechanisms involved in the emission of neutral atoms and atomic ions from a solid are now well understood. Much less is known on sputtering of complex species as clusters or molecules where collective effects are required for the transport of energy and the coupling to phonons. The formation of dense regions of energy or "spikes" resulting from statistical fluctuations of the energy deposition has been used to explain the cluster sputtering [1]. A very efficient way to increase the spike formation probability is to use polyatomic ions as projectiles. In the keV energy range, it was shown experimentally that these kind of projectiles could enhance considerably the secondary emission of neutrals, ions and especially complex

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species from solids. The first results were obtained on neutral emission [2, 3]. Systematic studies were made more recently using $(Au)_n$ clusters [4, 5], $(CsI)_nCs$ [6] clusters and C_{60} molecules as projectiles. In the keV range of projectile energy, the energy loss processes proceed through atomic collisions. An important step in the field of collisions with solids results from the possibility offered with new projectiles as polyatomic ions at several tens of MeV. These projectiles are accelerated by electrostatic accelerators. Beams of various kinds of clusters are now routinely available at velocities where the dominant modes of energy loss in solids are electronic collisions. So far little experiments have been reported on interactions of fast heavy polyatomic and large molecular ions with solids.

Energy densities much higher than with any other projectiles can be deposited in solids and it is thought that the impact of a fast cluster ion can create extreme transient conditions (temperature and pressure) that cannot be found in the impact of individual atoms. In the exit channels of energy relaxation, the prompt secondary emissions of electrons, atoms, clusters, photons are important probes of the interaction. The solid modifications after full energy relaxation are residual states that give information on the energy transfer and on the response of the solid. This leads to craters, surface deformations, large tracks in material. In this MeV energy regime, the range of the atomic constituents of a cluster is large and can exceed several microns. Therefore the projectile fragment characteristics can be studied inside and after exiting the solid. The spatial and temporal correlations of the atomic constituents travelling through the material are interesting and unique features of these collisions. The correlations are certainly the source of new phenomena. How do they affect the charge state per atom, the energy loss, the secondary emission by transmission, the target properties...? In this short paper we will review some of the most recent results obtained at Orsay with exotic MeV beams of Au_n , C_n and C_{60} .

1 Beam of clusters

The beams of Au_n , C_n , C_{60} were accelerated by the Orsay tandem accelerators [7, 8, 9]. As in the normal tandem operation mode, a Cs ion gun is used to bombard targets of gold, carbon or pure C_{60} in order to produce negative cluster ions accelerated at a few keV of energy. These large complex ions are then accelerated to energy qV , V being the high voltage of the terminal that can be varied from 1.5 to 14 MeV, q being the cluster charge. Collisions with N_2 gas in a very low pressure cell at the terminal give positive cluster ions which are then accelerated in the second part of the tandem. C_n^{+1} ($n = 2$ to 20), Au_n^{1+} ($n = 2$ to 5), C_{60}^{p+} ($p = 1$ to 3) can be obtained. The intact clusters are identified at the target site by time-of-flight measurements combined with magnetic deflection and energy measurements. A cluster beam line with a reaction chamber is set up at 1.2° from the zero degree direction of the beam.

2 Energy loss by cluster ions in a solid

Implanted Si detectors were used to measure the energy loss of cluster projectiles passing through carbon foils. The detector response signals for direct cluster impact on the Si detector were first investigated and rather strong phenomena were observed with C_n clusters ($n = 2-20$) and C_{60} at an energy per atom from 0.3 to 3 MeV/atom. The results show a large pulse height deficit of the detector in contrast to the case of n spatially separated atoms at the same velocity. The latter were obtained by breaking the cluster C_n in a thin foil (30 nm) placed a few mm upstream of the detector.

Fig. 1 shows the two spectra recorded for direct impacts of a C_{60} molecule at 18.3 MeV onto a Si detector and for simultaneous impacts of 60 carbons fragments after passage through a 300 Å carbon foil. Similar results were obtained for C_n clusters. Although there is a certain amount of energy loss in the foil, the pulse height signal that is recorded by the 60 carbon atoms spatially separated is much larger than the pulse height signal obtained by direct C_{60} impacts. The reason for the large pulse height deficit is not yet elucidated. Due to the zones of high energy density created along the passage of the correlated fragments, electron-hole recombinations can occur before separation by the electric field which may be reduced in the zone of large ionization and excitation. An extended zone of defects could also be produced along the correlated paths of the carbon atoms that would modify the material structure and therefore reduce the electron mobility and collections. More experiments are needed for understanding these phenomena. It is however important, for energy loss measurements of cluster in solids, to be aware of these perturbing effects. The procedure adopted for energy measurement with a silicon detector was therefore to place systematically thin foils in front of the silicon detector. The detectors were carefully calibrated with α particles (5.443 MeV and 5.486 MeV) from an ^{241}Am source and with carbon atoms of well defined energy. The dead layer was determined by several energy measurements at given tilted detector angles. If E_0 is the total energy to be measured, the remaining energy after passage through the dead layer is $E_0 - E_{dl}$ and the energy which is available for e^- -hole pair creation is $E_p = E_0 - E_{dl} - \Delta E_n$, ΔE_n being the part of energy associated with elastic collisions. The pulse height deficit per atom of C_{60} and C_p ($p = 2-10$) varies linearly with the available energy per atom $(E_0 - E_{dl} - \Delta E_n)/p$.

Comparison of the detector responses for a carbon atom from a cluster and for a single atom at the same velocity were made with various C_p and C_{60} projectiles. Different foil thicknesses were used. If $\Delta E(C_p)$ is the energy loss in a given foil by a cluster at energy E_i and $\Delta E(C_1)$ the energy loss in the same foil by a single atom at $\frac{E_i}{p}$ the difference between the energy loss per atom of a cluster and the energy loss of single atom is $DE = \frac{\Delta E(C_p)}{p} - \Delta E(C_1)$. Fig. 2 shows this energy difference as a function of the number of constituents p at different values of the energy per atom. The thickness of the foil was in this case 250 Å. The energy loss per atom of the cluster is similar to that of a single atom at the same velocity. There is a slight enhancement of energy loss for incident energy per atom above 2 MeV/atom while for 1 MeV/atom the results indicate a decrease of energy loss. For C_{60} projectiles, no difference was found (within the experimental error of $\pm 5\%$) between a carbon

originating from C_{60} and a single atom. As a first approximation the energy loss of a cluster C_n is equal to the sum of the energy losses of the constituents [10]. The close proximity of the multiply charged atomic constituents may play a role (energy loss enhancement) in the first layers of the solid material. The orientation of C_n clusters when entering the target may also be another important parameter for the amount of energy loss in the first layers of material. Experiments on stopping powers of molecules were reported previously, showing that both the velocity and the orientation of the molecule are important parameters. For small hydrogen clusters with ~ 100 keV/nucleon, an enhancement of stopping power was observed [11, 12]. For small oriented molecules of N_2 and O_2 (1 MeV to 2.6 MeV total), a diminution of stopping power was found. These molecules had their internuclear axis parallel to the beam direction [13]. The diminished stopping powers were observed only in very thin carbon foils ($\sim \mu\text{g}/\text{cm}^2$) and vanished with increasing thicknesses.

The present results are the first ones with large molecules. Further experiments are planned in the future with C_n clusters passing through very thin foils. Theoretical models for explaining energy loss with clusters are based on oscillating electric fields created by the electron density fluctuations during the passage of the correlated atoms. Interferences take place which depend on the internuclear distance and may give rise to "braking" forces or "pushing" forces [14, 15]. The electronic stopping power $\frac{dE}{dx}$ is often used to characterize a swift ion passing through a certain material. This quantity is also often used to parameterize physical phenomena occurring in the solid (formation of tracks, secondary emission yield of electrons, ions...).

In the case of cluster impact, the density of deposited energy, E/v , seems to be a much more relevant parameter than dE/dx . If one considers, for example, a MeV cluster ion with the same dE/dx as a single swift ion, the situation is very different in both cases. The secondary electrons (δ rays) - produced by, say a ~ 10 MeV/u heavy ion - have a large range and will transport energy far away from the ion tracks. In the cases of a cluster C_n at less than 0.1 MeV/u, the lower collision velocity with electrons generates smaller range δ electrons. The energy loss is first deposited in a small track core volume. The fast dissipation of this high energy density induces transient phenomena in the solid which are specific of cluster impacts with solids.

3 Solid modification, secondary emission

Spectacular effects were observed in metals bombarded with C_{60} at 20 MeV [16]. A continuous zone of damage was visualized by electron scanning microscope inside a Ti target. The diameter of the track was 25 nm on a distance of more than 100 nm. With a Uranium beam of 1 GeV (having the same $\frac{dE}{dx}$ as C_{60} , i.e $\sim 4\text{-}5$ keV/ $\text{\AA}$), much smaller diameters of damages were observed. The difference between C_{60} and U was even more pronounced with a Zr target since no damage was observed with fast U ions while large damage zones were still observable in the collision C_{60} - Zr target. This is a typical example where it is obvious that the $\frac{dE}{dx}$ is not the relevant parameter. At the entrance and exit sides of a target, energy dissipation could easily occur through surface deformation and crater formation as well as large emission of

matter. The release of matter can be measured in terms of total quantity of mass but some more important information as momentum, angular distribution could also be measured. In this electronic regime of collisions with clusters, the experimental program has just started but the measurements of total "erosion" and "backward total momentum" per impact are certainly important objectives to be achieved.

Different kinds of material have been irradiated with cluster beams. As an example, Fig. 3 shows the results obtained with a single organic crystal made of valine molecules. Individual molecules have a molecular weight of 117 u. Such crystals were bombarded with ^{127}I ions at 78 MeV and C_{60} at 23 MeV [17]. The surface was examined with an atomic force microscope at Uppsala (ion physic group). Small craters with a diameter of a few nanometers are produced with single ^{127}I ions while giant craters 50 nm wide and 15 nm deep are observed after impact of 23 MeV C_{60} projectiles. Targets of Langmuir-Blodgett films were also irradiated with 23 MeV fullerene ions [18]. Atomic force microscope images show crater sizes equivalent to the valine craters. Here again the energy density is a "key parameter". Such a crater with C_{60} corresponds to a total emission of mass of about 10^7 mass units and we can ask the questions: which are the species which are emitted? To what extent are they characteristic of the material composition? The secondary ions emitted from this solid were identified by time-of-flight mass spectrometry. Not only valine molecules were ejected intact but large size "clusters" of molecules were also observed with a high emission yield. In the case of bombardment with fission fragments (similar to MeV ^{127}I impact), the emission yield of valine clusters drops down very quickly with the size of the emitted clusters. Fig. 4 shows the variation of yield with the number of molecules in the valine cluster. With large molecular weight samples (mw = 5760) the emission of intact molecular structure is also observed with a yield more than hundred times higher than with single ions at 100 MeV. It is believed that rather large chunk of matter can be ejected intact with eventual subsequent "in gas phase decomposition", according to the types of binding energies in the chunks.

Another example of large secondary ion and cluster emission has been obtained with amorphous carbon targets [19]. Several carbon foils of various thicknesses were prepared. The secondary ions emitted from the foils were observed from the entrance surface (reflection mode) and the exit surface (transmission), after passage of the cluster projectile through the solid foil. As mentioned before the cluster structure is broken but the atomic constituents may travel in close proximity through the carbon foil. Fig. 5 shows the emission yield of C_n clusters emitted from the carbon foil under impact of C_{60} projectiles. Let us consider the reflection emission yield curve. Several measurements performed with several carbon foil thickness have shown that the emission was not dependent on the thickness. By comparison with other types of projectiles (keV and MeV atomic ions) the emission yield is several orders of magnitude larger. For cluster size corresponding to $n = 20$ for instance, the enhancement factor is larger than $\sim 10^4$ with respect to single carbon impact at the same velocity. Small bumps are seen in the curves fitting the experimental points. They correspond to cluster structure being more stable for even number of atoms than for odd numbers. Surprisingly, the emission yield from the exit side of the thin foil of 32.5 nm reveals that the "cluster effect" is still very important since a large emission yield is measured. The transmission and reflection yield curves are

parallel. The even larger yield observed in transmission may be explained by the increase of the projectile constituent charge state that would increase the amount of energy loss close to the exit surface. Therefore after such a thickness, the atomic constituents are still very close to each other and the resulting effects are similar to direct impacts on the foil (reflection mode). It can be concluded that Coulomb explosions and multiple scattering phenomena that tend to separate the atoms in the solid are minor processes on this distance of penetration. For a larger distance (results with 140 nm in the figure) there is still a pronounced effect of collective processes occurring at the exit surface, although it is reduced by almost a factor of 10, by comparison with the reflection results. This is again a strong indication that some of the atoms are close to each other after a rather long distance. Experiments with small C_n projectiles in the reflection mode would give information on the number of atoms necessary to obtain the same quantitative effects on secondary emission. Dynamic simulations with 60 atoms at this velocity are not yet possible over such a distance of penetration.

The secondary emission of ions and clusters in the transmission and reflection mode after impact of cluster projectile is therefore a way to estimate the distance of spatial correlation of the travelling atoms in the solids. The memory of the incident projectile cluster size is conserved over a surprisingly long distance. Another important question related to the two aspects previously evoked in this short paper, namely the stopping power $\frac{dE}{dx}$ and the atom spatial correlations, is the charge per atom of a cluster and its variation with the distance of penetration in a solid.

3.1 Charge state per atom of energetic clusters

When fast single ions pass through solid matter, an equilibrium charge state is reached after a certain number of electronic collisions and loss or gain of electrons by the projectiles over a certain distance. The variation of the atomic charge state with the distance x from the entrance surface can be theoretically estimated as well as the statistical fluctuations giving rise to a charge state distribution. For large Z atomic projectiles with a great number of atomic excited levels, the situation is somewhat complex and some semi-empirical formulas have been derived. One can easily imagine that large size clusters add a new dimension to the problem since interactions could take place between the atoms of the clusters and with the atoms of the target.

Up to now, the question was investigated with diatomic projectiles as N_2 , CH_4^+ , OH after dissociating in thin foils [20-22]. Lower charge states were obtained by comparison to single atoms (N, C or O) at the same velocity.

In the Orsay experiments, results were first obtained with C_5 at 2 MeV/atom and compared with C_1 at the same velocity ($E_{C_1} = 2$ MeV). Experimentally the beam was collimated by two pairs of slits to a dimension of 0.1 x 0.1 mm and centered in front of two parallel deflector plates as shown in Fig. 6. Carbon foils can be placed at the entrance side of the plates. In Fig. 6, the two different projectiles are represented, for convenience, off axis. The charged fragments are deflected - with respect to the 0° beam direction - at a certain angle, according to their charge value and the voltage applied on the plates. They are detected by a new multianode

position sensitive detector that can record several simultaneous impacts [23]. The deflection distance distribution corresponds to the charge state distribution. With large counting statistics, a precise determination of the centroid of the distribution is obtained that gives the average charge state. Measurements were performed for several voltage values applied on the deflection plates. A marked shift toward lower states (- 15 %) was observed for the cluster constituents by comparison with the charge state of C_1 at the same velocity. It is known that the equilibration of charge for single carbon atom is already achieved in a carbon foil thickness of 32 nm.

A calculation by G. Maynard [24] demonstrates that carbon atoms with low incident charge states (0,1,2) reach a mean charge state $\langle q \rangle = 2.5$ within a travelling distance ~ 1 nm in a carbon foil. At the considered velocity, an equilibrium charge state (of ~ 3) is obtained for single atoms, within less than 10 nm of penetration distance.

Another experiment on charge state measurement of carbon atoms after dissociation of C_5 clusters was performed at the same energy with a 25 nm carbon foil [25]. An electrostatic deflection technique was also used, with a long distance between the deflector and the detector (~ 1.8 m). In this experiment a row of silicon detectors was used for detection. Similar results were obtained with C_5 at 2 MeV/atom.

When using single atomic ions with relatively large equilibrium charge states (above 20), it has been shown that the charge state at the exit side of a foil could be different from the charge measured some nanoseconds later [26]. The explanation was a postionization effect due to Auger decay of the excited projectiles. The measurements of the hydrogen ion emission rate from the surface of the solid was used as a probe of the charge state q of the projectiles at the exit and entrance surface of a foil [27]. The H^+ emission rate varies as q^n with $n \sim 3$. Hydrogen ions originate from surface contaminant molecules of water and/or hydrocarbons. This sensitive method, which is well adapted to ion charge state measurements, has been applied to the determination of the average charge state per atom of a carbon cluster. Fig. 7 illustrates the principle of the experiments. Two thin foils were used. In the first one which has the same thickness as the one used in the previous experiment, the cluster C_5 breaks into 5 atoms which hit a second foil placed at a few mm.

To ensure that all the cluster fragments hit the second foil an energy signal is taken in coincidence by a Si detector placed behind foil 2. The H^+ ions emitted from foil 2 are accelerated by a voltage applied between an annular extractor and the foil. They are identified by time-of-flight (TOF) mass spectrometry and their rate of emission (number of H^+ /number of projectiles) is deduced from the TOF spectra. For a single carbon at 2 MeV/atom, the emission yield $Y(H^+)$ was found to vary at $q^{2.7}$, q being the average charge state in the few layers underneath the surface [28]. In the present case let us call p the average charge state of a carbon atom issued from C_5 after breaking in foil 1. The emission yield from foil 2, $YH^+(5C)$, corresponding to the simultaneous impact of 5 carbon atoms spatially separated is $YH^+(5C) \propto 5p^{2.7}$. The measured ratio $R = \frac{Y(H^+)5(C)}{Y(H^+)1(C)} = \frac{5p^{2.7}}{q^{2.7}} = 3.31 \pm 0.16$ gives $\frac{p}{q} = 0.86 \pm 0.06$. This result obtained with the H^+ method is in total agreement with the values measured by the electrostatic deflection techniques.

When using this method to determine the charge state of cluster atoms, the

H^+ yield measurements can also be made (as with fast ions) directly at the exit side of a thin foil when all constituents are still spatially and temporally correlated. An “apparent global” charge state can therefore be deduced from the H^+ yield at the exit surface. When the thickness increases, the atoms tend to separate and the consequence on the total H^+ emission should result in a linear dependence of the rate of emission per atom. With C_5 at 2 MeV/atom, the H^+ ions emitted from the exit side of the 32 nm foil were accelerated and measured also by TOF mass spectrometry. From the previous results on H^+ yield measured when the atoms are completely separated, one can deduce the experimental ratio $R \left(\frac{C_5}{5C} \right) = \frac{Y(H^+)C_5}{Y(H^+)5C} = 16.5 \pm 0.45$. If Q is the apparent global charge state at the exit side, we can write $R = \frac{Q^{2.7}}{5p^{2.7}}$ that gives $\frac{Q}{p} = 5.12$, p being as above, the average charge state per atom after exit of the foil.

Therefore, if one neglects post-ionization effects which are very unlikely with small Z atom, the apparent charge state Q is equivalent to the sum of the average charge state per carbon. This means that the atoms are very close from each other after a distance of travelling of 32 nm. With respect to H^+ emission it has been estimated that a depth of less than 1 nm is involved in the H^+ emission process [26]. Similarly a characteristic separation distance of less than 1 nm between atomic constituents at the exit surface may be necessary in order to generate the collective charge effect on the emission of Hydrogen.

When increasing the foil thickness, the radial separation distance between atoms increases in the solid. The measurements of the H^+ rate, at the exit side of solid foils, which decreases with thicknesses, is a way to estimate the spatial correlation. As an example, Fig. 8 shows the variation of the H^+ yield as a function of the carbon foil thickness. The measurements were made for C_{10} clusters at 1 MeV per atom.

Above 300 nm the atoms of the cluster are completely separated. Simulations of trajectories for cluster constituents on such a large distance are not easy to perform, and quantitative values on radial separation are not yet available. Experiments combining various cluster sizes and energies as well as various carbon foil thicknesses are needed to understand the correlation effects.

4 Conclusion

The use of large and fast polyatomic ions as projectiles to bombard solids has considerably broadened the field of ion-solid collisions. Some recent experimental results have been reviewed in this paper. A particular emphasis is given to the diminished charge state per atom of the C_5 cluster constituents inside the solid by comparison to single atom and to their spatial correlation. These first results are part of a program which is in progress. Exotic projectiles as C_{60} at high energy are very unique. However heavier clusters at high velocity with large constituent atomic numbers would further enhance the effects which are already observed with the present available beams. Power density at solid surfaces could become extremely high.

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Figure 1: Energy spectra recorded with a Silicon detector. a) direct impact of C_{60} projectiles, b) impact of the projectiles after passing through a thin carbon foil of $300 \text{ \AA}$ [10].

Figure 2: Energy loss difference between the energy loss per atom of a carbon atom in a cluster and of a single carbon atom as a function of the number of carbon atoms in the cluster.

Figure 3: View of craters observed in the surface of a single crystal of L-valine bombarded by 78.2 MeV ^{127}I and by 23.2 MeV C_{60} [17].

Figure 4: Measured yield of molecular cluster ions $(M_n + H)^+$ ejected from an organic target of valine: o: C_{60} at 20 MeV, x: fission fragments from a ^{252}Cf source [19].

Figure 5: Yield values of C_n^- cluster ions emitted per impact of C_{60} projectiles at 0.336 MeV/atom, measured in the transmission mode with carbon foils of 32.5 and 140 nm, and in the reflection mode (backward emission) with various thicknesses (from 32 to 350 nm) [19].

Figure 6: Schematic view of the electrostatic deflection system for comparing the average charge state per atom of a C_5 cluster passing through a thin foil, with the average charge of a single carbon atom.

Figure 7: Schematic arrangement of H^+ ion yield measurements after simultaneous impacts of n carbon atoms from a broken carbon cluster and after impact of single carbon atoms. The emission yield of Hydrogen ions is a probe of the projectile charge state at the surface.

HC1C10NATB

Figure 8: Yield of Hydrogen ions emitted from the exit side of carbon foils after impact of C_{10} and C_1 projectiles at ~ 1 MeV/carbon. Carbon foil thicknesses from 30 to 900 nm were used.