

Interaction of Small Hydrocarbons with Fusion Relevant Beryllium Thin Films

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ABSTRACT

Ion-surface collision studies are carried out with small deuterated hydrocarbon cations *i.e.* CD_x^+ with $x = 2-4$ colliding with fusion relevant Beryllium (Be) thin films with ions incident energy as low as 0 eV and as high as $E_{in} = 100$ eV. Be films are coated on stainless steel surface by the technique of Thermionic Vacuum Arc (TVA); a novel thin film deposition method with primary as well distinguished characteristic of control of ion flux and respective dose towards the substrate. Prior to scattering, methane-d₄ 99 atom % D is ionized by electron impact and ions are mass and energy analyzed. Ionization and collisions are performed in ultra high vacuum conditions. In these kinds of collision experiments, we have recorded secondary ion mass spectra and plotted respective incident energy resolved abundances of secondary product ions. Relative abundances in percentage of total secondary ions are plotted and it is observed that such beryllium films can accumulate charged hydrocarbon layers as surface adsorbates. These self assembled layers play a primary role in surface-scattering of primary ions. Moreover, it is seen that bond dissociation energy in lighter hydrocarbons is higher than that for heavier species and shows primarily that the deuterium atoms are loosely bounded to carbon atoms in heavier hydrocarbons than in lighter ones.

Keywords: Fusion Reactor Walls, Ion-Surface Collisions, Surface Induced Reactions.

1. INTRODUCTION

Study of reactive collisions of ions with surfaces is the area accelerated in last few years towards characterizing gaseous molecular ions and also nature of the surfaces. These studies are evidenced by most recent research work on properties of multiply-charged ions, on their spectroscopy and on their gas-phase reactivity [1-5]. Considerable interests are taken to study physical sputtering as well chemical sputtering caused by colliding slow ions with hyper thermal energy range (up to 100 eV) and the processes like Surface Induced Dissociation (SID), Charge Exchange Reactions (CER) and reflection properties have been identified and investigated [6-8]. In addition to being of fundamental importance, reactions of polyatomic ions with surfaces are relevant in applications like modifying surfaces to prepare

advanced electronic materials and plasma-wall interactions in fusion plasma [9-12]. A transfer of translational kinetic energy of projectiles into internal modes of energy has been observed during surface collisions of polyatomic ions, causing their dissociation. This energy transfer may help in characterizing the structures of projectile ions as well nature of surfaces [13, 14].

In our earlier papers, we have reported SID and CER of hydrocarbon cations and NH_3^+ with a variety of materials including stainless steel and fusion relevant materials like tungsten and others [15-19]. Reflections of hydrocarbons with fusion relevant tungsten (plasma sprayed tungsten) and Carbon Fiber Composite (CFC) surfaces have been studied experimentally [20-22]. An urgent demand for data on collisions of low-energy (0-100 eV) small hydrocarbon ions C_1-C_3 group, with

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International Thermonuclear Experimental Reactor (ITER) proposed materials *i.e.* beryllium, carbon and tungsten, motivated us to investigate the said materials through ion-surface collision experiments.

2. EXPERIMENTAL SETUPS

2.1 Thermionic Vacuum Arc (TVA) setup

Adhesive and dense layers of metals vapors like tungsten (W), beryllium (Be), nickel (Ni) or chromium (Cr) are deposited in this technique, in high vacuum conditions ($<10^{-3}$ Pa). An externally heated tungsten filament with 100 mA of electron current accelerates electrons towards an anode biased at 1-6 kV *i.e.* Be inside a W holder. A Wehnelt cylinder focuses the said beam and it ignites an electrical discharge at the anode and creates a strong local heating resulting in metal evaporation. These metal vapors are ionized and may cause the expansion of the plasma in the whole available volume. The parameters like filament current, anode voltage, cathode-anode distance and orientation of the anode are the keys to control the discharge conditions, ultimately whole deposition process. Using the optimized parameters like in [23], a deposition rate of about 5 ± 0.5 nm s^{-1} is achieved.

2.2 Double focusing mass spectrometer, BESTOF

Ion-surface collisions experiments were carried out at double focusing sector field mass spectrometer, designed in reverse geometry (*i.e.* BE) and combined with collision chamber containing Surface (S) and linear Time-Of-Flight (TOF) mass spectrometer, namely BESTOF [24-25] at the Institute of Ionphysics and Applied Physics, University of Innsbruck, Austria. CD_x^+ [$x = 2-4$] ions are produced by a 75 eV electron impact ionization of gaseous CD_4 molecules, in a Nier-type ion source. Cations produced are accelerated and extracted to about 3 keV of translational kinetic energy and mass and energy analysed. Ion beam current is measured using a Faraday cup installed at exit slit of E-sector. The beam is refocused by a combination of Einzel lens and primary part of the lens stack, held inside the collision chamber. Projectile ions impact at 45° while the scattering angle was fixed at 91° . The energy spread of the projectile ion beam *i.e.* CD_x^+ [$x = 2-4$] was about 0.50 eV and the corresponding ion currents were measured as 100-200 pA, focused on a

spot of 1×1.5 mm². In another study, we have simulated ion beam trajectories using SIMION 8.0, and found that about 10 % of the ion current may reach the surface to strike.

Normally, background pressure of the collision chamber is kept less than 10^{-9} Torr if the valve between the collision chamber and the mass spectrometer remains closed. However, during our experiments, opening this beam-line valve increases the pressure in this chamber to about 2.0×10^{-8} Torr. Under these conditions, the number of collisions between background molecules and the surface is of the order of 10^{12} mm⁻²s⁻¹. When the ion current was kept constant of the order of 10^{11} particles mm⁻²s⁻¹, it is worth noticing that the surface would always be covered with a few layers of hydrocarbons.

After reflection in front of the charged surface or collision with adsorbed layers, majority of the product ions resulting from a variety of reaction channels leave surface. A secondary lens stack guides them towards a pulsed deflection and acceleration field, a starting point for the TOF. These secondary ions are detected by a double stage Multi-Channel Plate (MCP), connected to a multi-channel scaler with time resolution of 2 ns per channel.

3. RESULTS AND DISCUSSION

3.1 Beryllium film structure

A variety of surface diagnostic techniques are applied to study the structure and composition of Be films at the National Institute of Lasers, Plasma and Radiation Physics, Bucharest, Romania. Roughness of about 300 ± 50 nm is observed in Atomic Force Microscopy (AFM) images (Fig. 1) while the Environmental

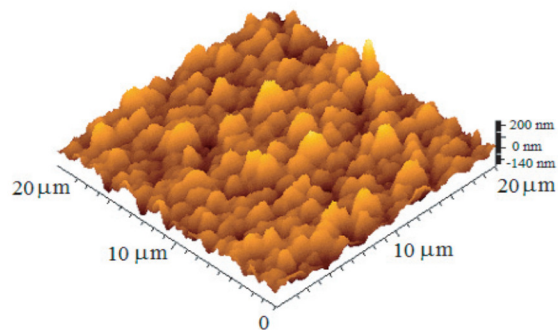


Fig. 1: An AFM image of Be film on stainless steel

Scanning Electron Microscopy (ESEM) showed a smooth and pin-hole free Be coating (Fig. 2). The X-Ray Diffraction (XRD) analysis proved the crystalline structure of the deposited film while some contaminations in the outermost layer (3 nm) are found when studied by Auger Electron Spectroscopy (AES).

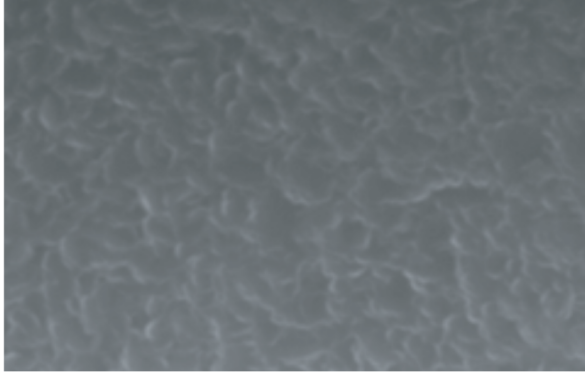


Fig. 2: An SEM image of Be film

3.2 Ion-surface collisions

3.2.1 Surface induced dissociation

Secondary ion mass spectra are taken by TOF MS explained in experimental section of this paper, after the collisions of CD_2^+ , CD_3^+ and CD_4^+ ions with beryllium film deposited on stainless steel by TVA.

The incident energy E_{in} range chosen for the projectile ions is from about 0 eV to a maximum of 100 eV which includes the energy range of hydrocarbons expected in next step fusion devices [26]. In Fig. 3, we show some of the mass spectra recorded at an incident energy of $E_{in} = 30$ eV. A very few ionic species appear at this low energy, as mentioned there in.

We plot incident Energy Resolved Mass Spectra (ERMS) for surface induced dissociation products after the impact of CD_2^+ , CD_3^+ and CD_4^+ ion beams with the beryllium film in the incident energy range from about $E_{in} = 0$ eV-100 eV. These are the plots of normalized abundance of the product ions $\frac{I}{\sum I}$ versus incident energy. Fig. 4 summarizes these graphs. Here and onwards, by Appearance Threshold Energy (ATE) of the product ions, we mean the incident energy of the primary ions corresponding to relative abundance of 1 % of a specific product ion. We see that the overall range of the ATE, to separate one D atom from the projectile ion, is lower than to separate two and respectively three deuterium atoms. Energy range of appearance threshold at the loss of one D atom from CD_x^+ lies between 10-20 eV where as this range for the loss of two D atoms is around 18-68 eV and for three D atoms this energy range is about 32-80 eV.

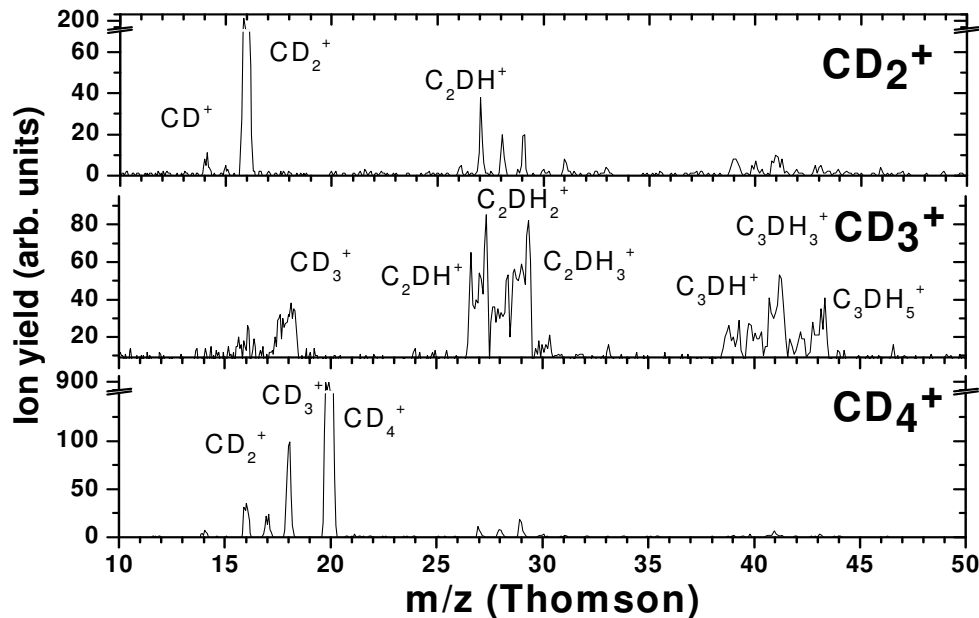


Fig. 3: Secondary ion mass spectra after collisions of CD_x^+ [$x=2-4$] with Be film deposited on stainless steel surface. These spectra are taken at incident energy of 30 eV.

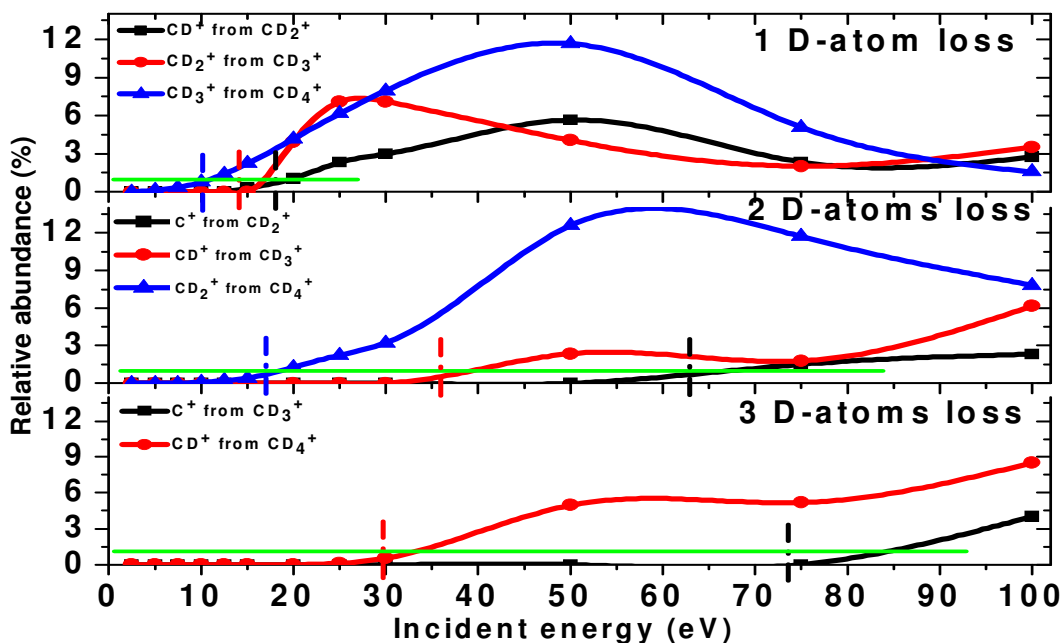


Fig. 4: Energy resolved mass spectra (ERMS) for surface induced dissociation (SID) products after the impact of CD_x^+ [$x=2-4$] with Be film. Lines are drawn to guide the eye.

Moreover, we see that the value of ATE for one D atom loss, is lower for heavier projectile ions than in the case of lighter projectiles e.g. ATE for one D-atom loss from CD_4^+ (product ion CD_3^+ m/z 18) lies at 10 eV whereas this value is 15 eV for the projectile ion CD_3^+ (product ion CD_2^+ m/z 16) and in the case of CD_2^+ , this energy threshold lies at 20 eV. Such sequential variation in threshold energy is also seen for the separation of two D-atoms i.e. ATE for two D-atoms loss from CD_4^+ (product ion CD_2^+ m/z 16) lies at 20 eV and for projectile ion CD_3^+ (product ion CD^+ m/z 14), this value is 35 eV whereas in the case of CD_2^+ (product ion C^+ m/z 12), this energy threshold lies at 70 eV. Similar behavior was observed for the dissociative reaction channels which result in the formation of products at the loss of three D atoms from the projectile ions. We see that the product ion CD^+ m/z 14 at the loss of three D atoms from CD_4^+ , appears with 1 % of relative abundance at 32 eV where this energy threshold lies at about 80 eV for the projectile ion CD_3^+ (product ion C^+ m/z 12). With these observations we can say that the *bond dissociation energies always remain at lower values for the heavier hydrocarbon species than those for lighter ones*. In other words, *the deuterium atoms in smaller hydrocarbons are strongly bonded with the central*

carbon as compared to their binding in heavier hydrocarbons.

4. CONCLUSIONS

Small deuterated hydrocarbon cations CD_2^+ , CD_3^+ and CD_4^+ are collided with fusion relevant beryllium thin films in the incident energy range from about 0 eV to 100 eV. Be is coated on polished stainless steel surfaces by TVA and a variety of experimental techniques reveal that smooth and crack free crystalline coatings are obtained. Secondary ion mass spectra are recorded by linear TOF mass analyzer and respective incident energy resolved mass spectra are presented in this article. We observe that bond dissociation energies are higher for lighter hydrocarbons than for heavier species respectively, C-D bonds seem stronger in lighter hydrocarbons than in heavier ones.

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