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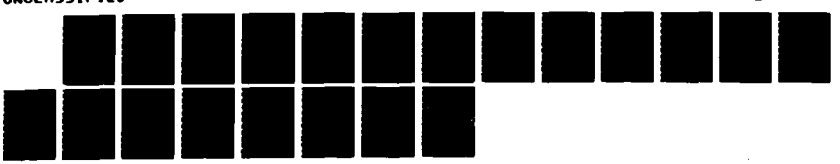

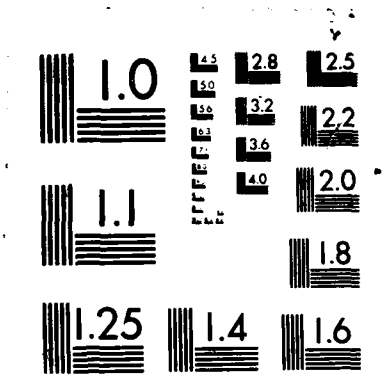

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Inelastic Ion-Surface Collisions

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15 May 1987

Scientific Report No. 1

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Ion scattering and direct recoil spectrometries were used to investigate electronic excitation and charge transfer processes ocurring during rare gas ion bombardment of CuLi alloy. Excitations in the close encounter appear to be governed by the degree of inner shell mixing between the colliding pair. Excitations in the outgoing trajectory are governed by the probabilities of Auger and resonant charge exchange between the scattered or recoiled particle and the surface. Measurements of the vacuum ultraviolet photon emission of the scattered and recoiled particles proved to be unsuccessful due to low

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20. photon yields.

The ion induced surface damage to transition metal fluoroanions was investigated by XPS and UPS. Ion bombardment reduces the transition metal atom to lower oxidation states, in some cases all the way to the metal. These results are consistant with the thermal spike model of ion damage.

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Objectives:

The objectives of this contract are to characterize the fundamental physical and chemical processes occurring in ion-surface reactions and to apply these to the fundamental problems pertinent to spacecraft orbiting the earth. The determination of these elementary processes will enable the elucidation of the reaction mechanisms involved in such hostile environment phenomenon as vehicle glow and surface corrosion. The observation of vehicle glow from the space shuttle is one example of such a reaction in a hostile environment (1-5). The origin of the emission is not understood, however, it may arise from reactions of reactive species with the spacecraft surfaces or with implanted or absorbed species on these surfaces. It has been proposed (6) that N and O can react on the spacecraft surface to produce excited NO which decays by photon emission yielding a glow phenomenon. The laboratory investigation of ion-surface reactions will provide insight into the fundamental physical and chemical processes giving rise to the glow phenomenon as well as such processes as surface degradation and erosion encountered by spacecraft. In order for a complete understanding of ion surface processes, it is necessary to characterize the chemical and physical state of the scattered and sputtered particles as well as the chemical state of the resulting damaged surface. With these goals in mind, research was planned in the following areas.

i. The characterization of the scattered and sputtered particles and their internal energies resulting from medium energy (1-10 KeV) ion bombardment by direct recoil spectrometry and VUV emission spectroscopy.

ii. The characterization of surface damage to multicomponent targets from medium energy ion bombardment.

The progress in each of these areas is discussed below.

I. Vacuum Ultraviolet Emission Spectroscopy Of Sputtered Ions And Particles.

Initial studies of vacuum ultraviolet(VUV) photon emission of sputtered

particles have used very high primary beam energies and fluxes (300 KeV, 200 A)(7-10). Little work has been done in the medium energy (1-10 KeV) range. Preliminary studies in this laboratory (11) have verified the possibility of measuring the VUV emission of scattered projectile atoms following medium energy ion bombardment of metal and graphite surfaces. These experiments, however, suffered from low signal levels due to poor photon collection efficiency. These low signal levels precluded the observation of emission of sputtered particles which constitute only a small fraction of the total VUV emission intensity. The entrance slit of the monochromator, in this initial system, was over 1 meter from the target surface. Photon collection efficiency decreases as $1/r^2$, therefore, to maximize collection efficiency the distance from the surface to the monochromator should be minimized. It was decided to build a new UHV chamber specifically dedicated to measuring bombardment induced photon emission. This new chamber would minimize the distance from the target to the monochromator, maximizing photon collection efficiency.

The chamber consisted of a Perkin Elmer 20-115 ion gun (12) and an ARC model UHVM-502 monochromator (13). The ion gun was placed 5 cm in front of the target surface which was oriented at 45° to the beam direction. The monochromator was placed at 90° to the beam direction. The entrance slit was approximately 20 cm from the sample. A Surface Science Laboratories Model 3390B (14) Open Face Sensor with resolution of 100 μm over a 25 mm active area was used as the detector. With this system, it was possible to measure the VUV photon emission of 3 KeV Ne^+ and Ar^+ scattered from Al and C surfaces with better signal to noise levels than with the original system. However, no emission was observed from sputtered target atoms or clusters. This could be due to poor collection efficiency or to low photon yields in the medium energy range. Poor collection efficiency is doubtful as this system was designed

specifically to maximize collection efficiency. This system should have equal or higher collection efficiency than the system of Schatner et. al. (7) with which emission of target species was observed. The most likely reason for the failure of this experiment was due to low photon yields of the sputtered particles at the primary beam energies used. All of the previous work in this area had used high primary beam energies (>100 KeV). The emission of sputtered particles in the VUV originates from highly excited levels in the species. The population of these highly excited levels would be expected to be more efficient with increasing primary ion energy. Therefore at the lower energies used in this work, a smaller fraction of the sputtered particles will be in these highly excited levels, resulting in a low photon yield. Also, the sputtering yield is lower at these lower energies, resulting in a lower density of sputtered particles. This is compounded by the fact that the ion currents used in this work are an order of magnitude lower than in the work of Schartner et. al. (7). In order to obtain reasonable signal levels, it would be necessary to work at higher primary beam energies and currents. This is beyond the range of our equipment and also out of the scope of this contract. It was therefore determined to terminate work in this area so our resources could be more productively employed in other areas covered by this contract.

II. Investigation Of Inelastic Ion-Surface Collisions. Direct Recoil And Scattered Ion Fractions Of Ne^+ On CuLi.

Atoms on a surface which are directly recoiled (DR) into a forward scattering angle as a result of a direct collision from an energetic primary ion have a well defined origin and energy distribution which can be described by the binary elastic collision model (11). This is in contrast to the broad energy distributions of secondary particles which result from multiple collisions. DR events, therefore, provide an excellent means for investigating atom-surface

electronic transitions and the final charge state of the direct recoiled atoms. Such studies are important to the understanding of inelastic processes in ion surface collisions. A model has recently been developed (11) for describing electronic transitions occurring during scattering of KeV ions from surfaces. This model has also been extended to describing DR particles (15). This model divides the ion trajectory near the surface into three segments: i. the incoming trajectory, ii. the close encounter and iii. the outgoing trajectory. In segments i and iii, electron promotion, neutralization and ionization can occur by Auger and resonant charge exchange with the surface as in the treatment of Hagstrum (16). In segment ii, the ionization and neutralization probabilities are governed by the distance of closest approach according to the Fano-Lichten mechanism (17,18). Therefore by measuring the energy dependence of the scattered and recoiled ion fractions, insight may be gained into the mechanism of charge exchange, neutralization and ionization of scattered and sputtered particles during ion-surface collisions.

A $Cu_{.12}Li_{.88}$ alloy was chosen to test this model for two reasons. First, heating the sample is known to induce thermal diffusion of the Li to the surface. Therefore by controlling the annealing time and temperature of the alloy, a surface varying in Li concentration from 12-100% can be prepared from a single sample, allowing studies of the Li coverage dependence of the scattered and recoiled particles. Also, CuLi alloys have been suggested for use as first wall and limiter materials in fusion devices (19). These alloys are of potential value in such devices because of the segregation of Li at the surface at elevated temperatures. This segregation forms a protective Li coating on the surface, preventing sputtering of Cu. The Li is sputtered primarily as a positive ion which can be reimplanted in the surface in a suitable magnetic or electric field, yielding a self sustaining surface. More information on the

inelastic ion-surface processes occurring for this material is needed if it is to be used in fusion devises.

The experiments were performed on a DR-ion scattering TOF spectrometer which has been described previously (11,15). The instrument allows the measurement of both scattered and recoiled ions and neutrals or neutrals only, allowing the determination of accurate ion fractions.

The ion fractions determined for Ne scattering off this surface at several Li coverages are given in Table 1. As can be seen the Ne and Li ion fractions both increase with increasing kinetic energy of the primary ion. The Ne ion fractions increase with increasing Li coverage, while Li ion fractions decrease with increasing Li coverage. More data needs to be obtained before a detailed application of the Hagstrom/Fano-Lichten model can be made, however some qualitative conclusions can be reached.

The decrease in Li ion fraction with increasing Li coverage may be explained by the fact that an electropositive atom on the surface decreases the work function of the alloy, thus increasing the probability of Auger neutralization of the DR Li in the outgoing trajectory. Therefore, as the Li coverage increases, Auger neutralization of the recoiled Li increases, lowering the ion fraction. The increase in the Cu ion fraction with increasing Li coverage may be explained by considering electron promotions during the close encounter. Barat and Lichten (17,18) have predicted that electron excitation cross sections should rise to a maximum for collision partners with similar atomic number and then decrease with increasing atomic number. This is due to the degree of inner shell penetration of the colliding pair. Ne has a much different atomic number than both Li and Ne. However the results indicate that there is more efficient inner shell mixing in a Ne/Li collision than in a Ne/Cu collision. This inner shell mixing results in electron promotion to excited and auto ionizing states yielding high ion fractions. Therefore, the Ne ion

fraction increases with increasing Li coverage.

Additional data is currently being collected on the scattered and recoiled ion fractions for this system. This additional data will allow a more detailed description of the roles that electron promotion in the close encounter and Auger and resonant transitions in the incoming and outgoing trajectories play in the resulting charge state of the scattered and recoiled particles. This data will be ready for publication shortly and a copy of the manuscript will be forwarded to your office as soon as possible. Future work will involve the scattering of neutral rare gas atoms off metal surfaces. This will allow the measurement of only those ions formed during the close encounter, free from any residual primary ions.

III. Investigation of ion induced surface damage of multicomponent targets. XPS and UPS of ion induced decomposition of transition metal fluoroanions.

Ion bombardment of a surface deposits large quantities of energy into localized regions near the surface. This energy can induce a variety of processes such as preferential sputtering, dissociation, atomization and recombination resulting in an altered surface layer (20-22). An understanding of the mechanism of ion induced surface damage is necessary for a complete understanding of ion surface collisions. The momentum transfer collision cascade theory (23-25) of sputtering provides a good model of ion damage to single component targets. The mechanism of ion damage in multicomponent targets is not well understood, however.

In this laboratory, salts of complex oxyanions have been used as model systems for the study of ion damage to multicomponent systems. Oxyanions, such as CO_3^{2-}, NO_3^{2-} and SO_4^{2-}, whose central atoms (C,N,S) form volatile compounds

with oxygen were found (26,27) to become deficient in this central atom upon bombardment. In contrast, ion bombardment of oxyanions with metal central atoms, (such as MO_y^{n-} where M = Cr, Mo, W, V, Nb, and Ta) which form involatile compounds with oxygen, resulted in preferential loss of oxygen and reduction of the central metal atom to lower oxidation states (28-30). In some cases, reduction of the central metal atom to oxidation state (0) was observed but not in others. These results were explained using the thermal spike model of Kelly (31). This model states that when an ion strikes a surface, a localized high temperature region is produced, resulting in atomization and fragmentation of the species within this region. As the system relaxes, recombination occurs. Of the numerous decomposition reactions possible in such a process, the model predicts that those with the highest free enthalpy of reaction occur with the highest probability. This model successfully predicts the decomposition behavior of the oxyanions studied.

In order to further test the generality of the proposed mechanism, these studies were extended to the ion induced decomposition of the fluoroanion compounds K_2TaF_7, K_2NbF_7 and K_2TiF_6. A manuscript covering this work is in preparation but was not in its final form at the time of this writing. A copy will be forwarded shortly.

XPS and UPS measurements were made of the unbombarded and bombarded transition metal fluoroanions. The measurements were made on a Perkin Elmer PHI Model 550 ESCA/SAM system. The samples were bombarded until no further changes were seen in the XPS spectrum. This saturation condition occurred at an ion dose of 1.5-1.8 x 10^{17} ions/cm^2. The initial samples were all found to contain a small amount of hydrolysis products such as $K_2MO_2F_2$ (M= Ta, Nb, Ti), however, the bombarded samples where found to contain no oxygen impurity. The core level XPS lines of the bombarded samples consisted of a number of overlapping spin orbit doublets due to the various oxidation states present, resulting in a

rather complex line shape. The individual components of this line shape were determined using a deconvolution procedure which has been described previously (32). The binding energies and atomic concentrations before and after ion bombardment are given in Table 2.

The results indicate that all of the fluoroanions decompose under ion bombardment and that the composition of the decomposed layer reaches a steady state after which additional ion bombardment causes no further changes in the system detectable by XPS or UPS. While the specific decomposition product distributions vary widely between compounds, there are several changes which are common to all of the samples: i) The relative concentration of the central transition metal increases while the concentrations of the potassium and fluorine decrease and ii) lower oxidation states of the central metal atom are produced. This behavior is similar to the decomposition behavior of transition metal oxyanions (29,30). The relative abundances of the various oxidation states of the metal in the bombarded sample are given in Table 2.

The most outstanding feature observed in the decomposition of the fluoroanion compounds is the fact that the central metal atom is reduced to the metallic state in K_2TaF_2 and K_2NbF_7 but not in K_2TiF_6. This behavior also correlates well with decomposition of the oxyanion compounds. Oxyanions of 1st row transition metals were not reduced to oxidation state (0) upon ion bombardment, while those of the 2nd and 3rd row transition metals were reduced to (0). This close correlation of the decomposition of the fluoroanion and oxyanion compounds strongly suggests a similar mechanism for these decompositions.

Decomposition of the oxyanion compounds has been successfully described by the thermal spike model. Ion beams can transfer both translational and electronic energy into localized regions near a crystalline surface. Seitz and

Koehler (32) give an expression for a particle of energy, E, striking a surface of thermal conductivity K, heat capacity C, and density, ρ. The temperature, T, at a distance r from the site after a time t is given by

$$T=E(c\rho)^{1/2}[8(\pi Kt)^{3/2}\exp[-c\rho r^2/4kt]. \quad (1)$$

For a 4 keV Ar^+ ion striking a salt, typical of these studied herein, a region of radius 20 Å can be elevated to a temperature of greater than 3000 K. This condition persists for 10^{-11} sec., which is orders of magnitude longer than the vibrational periods of the surface species. The high energy density in the thermal spike region causes ionization, dissociation, and atomization of the surface species. Translational energy acquired by the constituents through collision cascades and attractive or repulsive electrostatic potentials results in ejection of some particles and random collisions between others. As the system relaxes, recombination reactions between atoms and molecular fragments occur. The resulting decomposition products remaining on the surface are nonvolatile species with high negative free energies of formation. Products with high vapor pressures or low free energies of formation are typically lost to vacuum. This is not to imply that a quasi-equilibrium situation is attained during sputtering but that thermodynamics only governs the probability of a compound forming upon relaxation.

Kelly (31) has shown that the decomposition of metal oxides upon ion bombardment can be explained in terms of the thermal spike model. Ho, et al. (21) have extended this treatment to three component oxyanion compounds. They observed that those decomposition reactions with the largest negative free energy of reaction tended to have the highest probability of occurring. The similarity of this oxyanion work to the decomposition of the fluoroanion compounds indicates that a similar mechanism is involved. Unfortunately, since the required thermodynamic data is not available for the fluoroanion compounds a rigorous treatment is not possible. However, the stability of the possible

products can be examined.

Compounds of the first row transition metals with oxidation states of (II) and (III) are well known and are stable in the solid state (33). However, lower oxidation states in 2nd and 3rd row transition metals tend to be unstable and do not play a major role in their chemistry. As the thermal spike relaxes, stable nonvolatile products with large free energies of formation are formed and remain on the surface. Thermodynamics governs the probability that a species will be formed upon relaxation. Thermodynamically favorable reaction channels resulting in transition metal compounds with oxidation states of (II) and (III) are more likely for 1st row transition metals such as Ti than for 2nd and 3rd row metals such as Nb or Ta. Therefore K_2TiF_6 should have thermodynamically favorable decomposition channels which do not result in the reduction of the Ti all the way to the metal. K_2NbF_7 and K_2TaF_7, on the contrary, are less likely to have favorable decomposition channels resulting in oxidation states (II) or (III), therefore reduction to the metal is favored. Therefore, while there is insufficient data to prove that the decomposition of the fluoroanion compounds is governed by the thermal spike model, the data is certainly consistent with this model.

Conclusions:

Significant progress has been made in the areas of research currently under investigation. The charge exchange processes of scattered and recoiled atoms appear to be governed by inner shell penetration in the close encounter and Auger and resonant transitions in the outgoing trajectories. Ion damage to multicomponent targets is consistant with the thermal spike model.

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TABLE 1

Ne and Li Ion Fractions
Resulting From Ne^{+} Bombardment of CuLi

Ne Ion Fractions

% Li on Surface	KE(KeV) 2	4	6	8	10
12	1.0	2.5	3.0	4.0	5.0
<90	2.6	7.0	8.5	---	---
100	3.5	12.0	17.0	18.0	18.0

Li Ion Fractions

12	9.0	20.0	28.5	33.4	37.0
100	8.0	11.0	13.0	17.0	22.0

Table II: Binding Energies (in eV) and Atomic Concentrations for K_2TaF_7, K_2NbF_7, and K_2TiF_6 Before and After 4 keV Ar^+ Bombardment to a Steady-State Condition.

		Initial		Bombarded		
		BE	AC	BE	AC	
			K_2TaF_7			
K $2p_{3/2}$		292	.21	292	.18	
F 1s		687	.68	687	.62	
Ta $4f_{7/2}$	(V)	30.0	.07	---	---	
	(IV)	---	0	26.7	.02	0.20
	(II)	---	0	23.7	.02	
	(0)	---	0	21.7	.16	
	impurity	28.4	.04	---	---	
			K_2NbF_7			
K $2p_{3/2}$		293	.22	293	.18	
F 1s		686	.69	686	.64	
Nb $4f_{7/2}$	(V)	208.9	.06	208.8	.09	
	(IV)	---	---	206.4	.03	0.16
	(II)	---	---	204.3	.01	
	(0)	---	---	202.4	.03	
	impurity	207.2	.04	---	---	
			K_2TiF_6			
K $2p_{3/2}$		292	.23	292	.19	
F 1s		686	.68	686	.63	
Ti $3p_{3/2}$	(IV)	461.8	.07	461.2	.01	
	(III)	---	---	459.4	.07	0.17
	(II)	---	---	458.1	.09	
	impurity	459.4	.02	---	---	

END

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