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SURFACE INTERACTION MECHANISMS OF 5eV ATOMIC

Final Report on NAGW-823

OXYGEN: DATA ANALYSIS FROM THE

UAH EXPERIMENT ON STS-8

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Chemistry Department The University of Alabama in Huntsville Huntsville, AL 35899

Dr. J. C. Gregory Principal Investigator

Abstract

The UAH experiment which flew on the STS-8 mission had several objectives which were mostly of a speculative nature since so little was known of the processes of interest. The experiment provided original, if limited, data on: (a) oxidation of metal surfaces, (b) reaction rates of atomic oxygen with carbon and other surfaces and the dependence of these rates on temperature, and (c) the angular distribution of 5eV atoms scattered off a solid surface. This paper provides a review of the results, with references given to fuller published accounts where these are available.

The paper was the basis for an invited presentation at the NASA Workshop on Atomic Oxygen Effects, November 10-11, 1986 at the Jet Propulsion Laboratory, Pasadena and has appeared in the proceedings at that workshop (JPL Publication 87-14; June 1987).

Other work also supported in part by this grant, has appeared in Rarefied Gas Dynamics 15 (1986), a copy of which is included in an appendix to this report.

Introduction

The chemistry of reactions between species with relative kinetic energies in the range of a few eV to a few 10's of eV has only been sparsely studied, principally because of the experimental difficulties and expense of generating beams of usable flux at these energies. The region is of considerable theoretical interest since it overlaps the region of energies of chemical bonds and activation energies. Thus enhancement of reaction rates (over those at ordinary temperatures) is possible and even likely. With this in mind, in 1974 we proposed an experiment for the Long Duration Exposure Facility (LDEF) spacecraft, originally projected to fly in 1978 (Ref. 1). This mission was launched in 1984 and has not yet been recovered from orbit. The early 1980's saw an awakening of interest within NASA in the effect of hyperthermal oxygen atoms on spacecraft materials, and several exposures of materials were made and studied. In 1983 a NASA-wide materials experiment was proposed, for which UAH was invited to provide a modified version of the LDEF hardware. This composite experiment, designated EIOM-2, was flown in September of the same year, which must be one of the shortest completion times for a flight experiment of this size.

The UAH experiment included the following components:

- (a) Seven thin (optically-transmitting) metal films deposited on quartz flats. The metals used were Al, Au, Ir, Nb, Ni, Pt and W.
- (b) Samples of materials known or suspected to exhibit gross erosion or corrosion effects (C, polymers, Os, Ag). These samples were mounted on heated plates at different temperatures to allow measurement of temperature dependence of rate.
- (c) Scatterometers: a passive device of novel design in which the intensity of oxygen atoms reflected from a solid surface was measured.
- (d) In addition to the above, carbon specimens were provided to Johnson Space Center experiments designed to measure (i) synergistic effects of solar UV on reaction rates and (ii) effects of atmospheric oxygen ions on those rates.

A short description of the experimental approach and the hardware is given below for each of these components, together with a summary of the results. Orbital exposure data pertiment to the results described here in given in Table I.

TABLE I: STS-8 ATOMIC OXYGEN EXPOSURE DATA

Payload Bay Forward Facing: Altitude: Velocity: Mean Oxygen Atom Density (Calculated: Surface Impact Frequency: Integral Fluence: t = 41.2 hrs. 120 nautical mi. (225 km) 7.8 km s⁻¹ 2.65 x 10^9 cm⁻³ 2.07 x 10^{15} cm⁻² s⁻¹ 3.5 x 10^{20} atoms cm⁻²

Results

(a) Effects on Optical Surfaces

This investigation was directed to determining some quantitative effects on uncoated optical surfaces. Others have shown that standard optical coatings (where permissible) of metal oxides or metal fluorides are very resistant to atomic oxygen.

The surfaces studied were of two types: high-purity thin films sputtered or evaporated onto 2.54-cm diam $\lambda/20$ fused silica optical flats, and highly polished bulk samples. Films were prepared with optical densities of ~2.5 or less. Measurement of optical densities using a Perkin-Elmer PDS scanning microdensitometer allowed sensitive determination of any changes to these thin films produced by the exposure. By masking one-half of each sample during flight as a control it was possible to measure changes in optical density of ~0.01, corresponding to a few percent change in the thickness of ~10-nm thick films. (No increase in scattering was assumed, since most films became smoother; changes in reflection due to very small oxide thickness increases were

also neglected.) In several cases the sensitivity of the method was ± 1 monolayer of surface atoms. Total film thicknesses were accurately measured by step height changes in stylus traces of the film/substrate surfaces using a Tencor Alpha-Step 200 stylus profilometer with a nominal 2-µm radius diamond stylus. Decreases in both the optical density and total film thickness were attributed to metal film removal. A decrease in optical density but an increase in total film thickness was attributed to film expansion due to formation of a nonvolatile oxide of lower density than the original film. Topographic measurements were also made on a number of surfaces with an optical heterodyne instrument. These measurements provided an optical determination of surface roughnesses for comparison to stylus results as well as several correlation functions from the same measurement. A fuller account of these measurements is given in Ref. 2.

Estimates of metallic film thicknesses in exposed and unexposed areas were calculated from the optical density measurements using equations for transmission and reflection.

A summary of the results is shown in Table II. Al, Ni, Nb and Cu showed small but measurable increases in oxide thickness over that obtained by normal exposure to the atmosphere. Au, Pt and Si showed no measurable effects by these techniques. Os showed massive erosion, and Ag massive corrosion. Ir showed evidence of slight erosion.

(b) <u>Kinetic Studies on Carbon and Other Highly Erodible</u> Surfaces

The experimental approach used was necessarily very simple as no electromechanical devices such as lids, shutters, etc. were available, and no intermediate measurements could be made, i.e., only a single integral effect could be measured for each sample. Samples were nominally one inch discs, with the hotplate discs only being one quarter-inch diameter. For the case of the erodible materials discussed here, a bar-pattern of small rectangles of niobium was deposited on the surface using a photo-resist technique. The niobium was sputtered

TABLE II

Material	Nominal Thickness (nm)	Thickness of Metal Converted to Oxide (nm)	Thickness of Metal Lost (nm)
Al film	3.4 .	0.8	none
Au film	35.5	none	none
Ir film	32.3	none	2.5
Ni film	54.2	0.7	none
Nb film	16.8	1.3	none
Pt film	10	none	none
Os bulk	bulk	none	1100n m
Ag bulk	bulk	>100nm, variable	none
Cu bulk	bulk	~3.5	none
Si bulk	bulk	no measurable e	ffect

on as a uniform film ~200-nm thick. Although it oxidized, it still served to protect the underlying carbonaceous material. The bar-pattern allowed multiple measurements of the step-height to be made using the Tencor stylus profilometer. Amplitudes were checked using SEM micrographs. Half of each sample was covered at all times before and during the flight, and served as a control.

Erodible surfaces studied included single crystal graphite (basal and prismatic planes, vitreous (or glassy) carbon from various manufacturers, polymethyl methacrylate (lucite), bisallyl diglycol carbonate (CR-39, an optical plastic), and diamond.

Erosion observed by this experiment ranged from 75-nm for diamond (which appears to be particularly resistant to oxidation under these conditions), to about 10,000-nm for the poly-carbonate resin, CR-39, which was the most heavily eroded sample reported on any flight exposure.

The temperature dependence of the oxidative effects was measured by conducting the erosion measurements at three temperatures spanning about 120° C. The Arrhenius activation energy, ΔE , was estimated, assuming:

 $r = A e^{-\Delta E}/RT$

-4-

where r is the rate of the reaction and A is a constant assumed independent of temperature T. These studies were performed for 6 materials, vitreous carbon, 2 graphites, CR-39, silver, and osmium. All activation energies were small and positive.

The conclusions from the measurements on various forms of carbon exposed in the STS-8 mission appear applicable to organic solids in general. They may be summarized as follows:

- 1. Measured erosion was linear with total fluence (Fig. 1).
- 2. No induction time was observed before onset of erosion (Fig. 1).
- 3. Erosion rate linear with oxygen flux (i.e., reaction probability independent of flux) measured over a small range 1.5 to 2.5 x 10^{15} atoms cm⁻² s⁻¹.
- 4. Arrhenius activation energies for the reactions were measured (Fig. 2) as follows:

vitreous	carbon		1200	(cal	mole ⁻¹)
graphite	(basal	plane)	1400		
CR-39			1050		

- Reaction probabilities depend on temperature as shown in (4) above. Reaction probabilities for carbons exposed at ~300^oK ranged from .1 to .15 where reaction probability equals the number of carbon atoms lost divided by the number of incident oxygen atoms.
- 6. No effect was observed on the measured erosion rates (at the 5% level) which could be ascribed to the presence for absence either of solar UV or of charged oxygen species.

The silver and osmium data has not been fully analyzed, but it is clear that the apparent activation energy of the rate-controlling step in both the osmiumloss process and for the production of bulk silver oxide under these conditions is positive.

(c) Atom Scattering at Orbital Velocities

When an atom or molecule strikes a surface a number of processes may take place. Some of these are:

- 1. Accommodation of momentum and energy.
- 2. Chemical reaction with a surface atom or adsorbed molecule.
- 3. Recombination, dissociation or excitation of projectile species.
- 4. Sputtering.

Measurements of the spacial and energy distribution of scattered species provides a probe of the interaction potential existing between the projectile and target species. Understanding of the processes taking place during such an interaction requires some knowledge of this potential.

While the UAH experiment was passive and very simple, it has provided a unique measurement of the angular distribution of oxygen atoms scattered from a polished carbon surface in orbit. It was not capable of measuring the velocity or energy profile of scattered species. The apparatus has been described elsewhere (Ref. 3), and the data has been used to estimate satellite lift and drag parameters (Ref. 4).

The scatterometer, shown in Figure 3, consisted of an aluminum enclosure, a polished vitreous carbon disc mounted so that the stream of fast atoms passing through the entry slit impinged on its center at 55° . Detection of reflected atoms was accomplished by a silvered strip of clear CR-39 plastic mounted in the can like the x-ray film in a diffraction camera.

Silver absorbs oxygen atoms with efficiency ~100% and is converted to clear oxide. Increase in optical transmission measured with a scanning optical densitometer yields reflected 0 intensity as a function of angle.

The resultant angular distribution of 5eV 0 atoms is shown in Figure 4. Such a distribution is described as wide lobular with a maximum intensity in the reflection hemisphere 15° from the surface normal.

A calculation of the mass balance for atomic 0 showed that the silver converted to oxide accounted for at least 80% of atoms incident on the carbon (after deducting those which reacted with carbon).

Conclusions, discussed more fully in Ref. 3, are that the incident atoms are almost, but not quite, fully accommodated at the carbon surface, and that recombination efficiency (to form 0_2 molecules is low (<20%). We intend to pursue further studies of scattering in this energy regime both in space and in simulation facilities on the ground.

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Figure 1. Vitreous Carbon Dosimeter, STS-8 Exposure



Figure 2. Arrhenius Plots for Carbon Oxidation by 5eV Oxygen Atoms (STS-8 data)





APPENDIX

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A MEASUREMENT OF THE ANGULAR DISTRIBUTION OF 5 eV ATOMIC OXYGEN

SCATTERED OFF A SOLID SURFACE IN EARTH ORBIT

J. C. Gregory and P. N. Peters, Huntsville, Alabama USA

Summary

The angular distribution of 5 eV atomic oxygen scattered off a polished vitreous carbon surface was measured on a recent Space Shuttle flight. The experimental apparatus was of novel design, completely passive and used thin silver films as the recording device for oxygen atoms.

We observed that most of incident oxygen was contained in the reflected beam, remained in an active form, and was probably still atoms. We allowed for 12% loss of incident atoms which are converted to CO at the carbon surface.

The scattered distribution, which was wide lobular peaking 15° in the forward direction, showed almost but not quite full accommodation. The method of Nocilla (1) was used to fit the shape of the lobe. The implications of this measurement for satellite lift and drag calculations are discussed in a companion paper by Karr, Peters and Gregory (2).

1. Introduction

In the scattering of beams of gas particles from solid surfaces, the angular dependence of the scattered particles has been found to be dependent on the incident angle of the beam, the temperatures of the gas and the surface, the masses of the gas and surface atoms, and the form of the surface (including roughness and contamination). For a full description of the scattering process the velocity distribution of the scattered particles must also be known. Clearly, the present single measurement does not permit such a comprehensive description.

If incident particles are captured on a surface long enough to reach equilibrium (a period of at least several vibrations) the particles are accommodated atom-lattice energetically and their desorption typically exhibits a cosine angular distribution. Such behavior is often seen with low, or particles If the were not energy particles. thermal, accommodated, their reflection would be close to specular, if

the surface is considered to be an ideal flat plane. This is sometimes observed with particles at higher speeds, but the increase in particle energy also causes a deeper penetration of the surface potential and a transition to a different scattering model, called 'structure scattering', is indicated.

Transition from thermal to structure scattering has been postulated by Oman (3) to occur at some value of incident energy E_i at which the scattered lobe properties show a systematic change. This has generally been shown to be so in the cases of rare gases scattering from single crystal surfaces under UHV conditions (see for example Liu, Rodgers and Knuth, 4). At the energy of the present measurement, 5 eV, we might assume according to this theory that the cube model no longer holds and that the 'structure regime' is penetrated (Goodman and Wachman, 5).

In low earth orbits most of the atmosphere is atomic oxygen and the orbital motion of the spacecraft results in an interaction energy with the forward facing surfaces of approximately 5 eV per atom. Most reported laboratory studies of oxygen atom/surface interactions have been carried out at energies either considerably below, or in excess of this energy; lower or higher energies having been usually obtained by thermal or electrical techniques, respectively. Satisfactory, high density, 5 eV oxygen atom beams have been lacking for ground based studies due to technical difficulties. There is good reason to believe that this situation is now changing (Cross, 6, Bareiss and Sjolander, 7).

2. Experimental Description

All gas-beam scattering experiments include three components: the gas beam, the target surface and the detector. In this experiment the beam was provided by the relative velocity of the atmosphere past an orbiting spacecraft, and the detector was a passive silver-film device (described below) which is sensitive only to oxygen atoms. Our target surface was polished vitreous carbon and in some ways was the least well defined component because of our lack of knowledge of its surface condition during the experiment. A diagram of the reflectometer hardware is shown in Figure 1. This was originally designed for the Long Duration Experiment Facility (LDEF), Gregory and Peters (8), which is currently in orbit.

2.1 Beam Characteristics

While low Earth orbit does not provide the perfect beam for many experimental purposes (for example, the composition and energy can only be varied over a small range and then not independently) it does provide intense beams of atomic oxygen and molecular nitrogen at energies which have proven difficult to generate in the laboratory. The characteristic energy of around 5 eV, depending upon the orbital altitude, is roughly equal to chemical bond energies so that reactive scattering processes may be studied. Enhanced reactivities have, in fact, been observed with 5 eV oxygen on carbon and other surfaces (Gregory and Peters, 9, 10).

The density, composition and temperature of the atmosphere are highly variable, but have been extensively studied and modelled. The beam was formed by aligning the entrance slit and solid target with the velocity vector of the spacecraft. This was obtained in this case by controlling the attitude of the entire Shuttle Orbiter. The flight, designated STS-8, took place in September 1983, and was particularly well suited to the experiment discussed here since during the entire mission 95% of the 3.5×10^{20} atoms cm⁻² fluence was accumulated with the experiments oriented within 1° of the orbital direction. Beam divergence (14° FWHM) was accurately known both from knowledge of the spacecraft velocity and ambient gas temperature, and from a direct measurement of the etch-profile of a shadowed carbon surface, which we have described elsewhere (Peters, Sisk and Gregory, 12).

2.2 Atomic Oxygen Detector

The sensor was a thin silver film deposited on a strip of optically clear CR-39 plastic which fitted within the cylindrical reflectometer cavity in the same manner as x-ray film in a Debye-Scherrer camera. The Ag film formed a circle with the carbon target at its center. The use of Ag films as sensors was reported by Henderson and Schiff (13) and by Thomas and Baker (17). Unlike these workers, we used the change in optical density of silver as it was converted to Ag_2^0 .

The film thickness chosen was designed to approach, but not exceed, saturation in the most heavily exposed area, thus providing maximum sensitivity for the measurement. The interior of the aluminum reflectometer housing was also silvered to act as a sink for any stray oxygen atoms. Most of the flight was flown with the reflectometer accurately aligned in the forward direction; a preflight decision was made not to further collimate the beam with tandem slits, for fear that orientational inaccuracies would limit knowledge of the actual flux reaching the surface or, worse, completely block the beam. Thus, a distribution of tangential components of velocity associated with thermal energies of the oxygen atoms are present. Since we know the gas temperature, as discussed above, this does not introduce serious error.

The optical density of the silver-coated sensor strip was measured at 1.0 mm intervals with a Perkin-Elmer Micro-10 scanning microdensitometer. Calibrations of silver thickness versus optical density were obtained by measuring 16 coatings of silver thicknesses on 1/20th-wave fused silica flats with a stylus profilometer. This data, which was corrected for different substrate absorptions, was used to determine the initial silver film thickness as a function of position on the sensor strip. This varied from approximately 630 Å to 770 Å over the region that received the reflected beam, with approximately 700 A thickness where the maximum exposure occurred later. Postflight measurements of optical densities were used to determine changes due to exposure. The average optical properties of an Ag_20 film produced on an earlier flight were used to correct for overlying silver oxide on the postflight sensor strip. Since the silver film used in the reflectometer was approximately one-third as thick as the film totally converted on an earlier flight, and since we obtained a good mass-balance as discussed in section 3, the assumption of linearity between optical density changes and oxygen atom exposure seemed reasonable.

While the atmosphere at the flight altitude contained some 10% N₂ molecules, our detector did not respond to them and we assumed their presence did not affect any measured process. Our detector gave angular intensities of scattered oxygen atoms with good sensitivity and linearity but, so far as is known, responded equally efficiently to oxygen atoms of all velocities. Consequently, the velocity distribution of the scattered beams was unknown.

2.3 The Scattering Surface

The structure and condition of the target surface, a vitreous, or glassy, carbon disc, cannot be described with any great certainty (as is frequently the case in some of the older beam-surface work). Vitreous carbon is formed by heating thermo-setting polymers to high temperatures (1000 - 2000° C). A refractory, non-porous, isotropic form of carbon is obtained which is much less reactive to thermal oxidation than graphite. The target was in the form of a one-inch diameter disc, the surface of which was polished, finishing with 1 μ m diamond. Scanning electron micrography at 100,000X showed a smooth featureless surface. The surface was slightly smoother after exposure than before. We discuss contamination of the surface in section 4.

3. <u>Results</u>

The variation in optical density of the exposed silver film is shown on a polar diagram in Figure 2. The bulk of the reflected atoms lie in a wide lobe whose maximum intensity lies at $\theta_r = 15^\circ$. A small supraspecular peak (~2% of total) may be seen at $\theta_r = 60^\circ$. It is not clear whether this is real or an absorption artifact. The figure also shows a coseine distribution for comparison, calculated for an equal number of oxygen atoms as the data contain.

No changes in optical density were observed on portions of the silver film which were shadowed from line of sight with the slit, providing good evidence that there were no stray oxygen atoms inside the reflectometer. The film holder cut-off may be seen at $\theta_i \sim 20^\circ$. This prevented proper measurement of the backward region, and has been corrected in the latest design for future flights.

Optical densities were corrected for absorption by CR-39 and Ag_2^0 and it was estimated that at lobe maximum, only 200 Å of Ag remained out of an initial thickness of 700 Å. Since we were given the atom fluence from the MSIS-83 model (Ref. 11) and we know the slit size and the measured lobe shape, we can calculate the amount of Ag converted to Ag_2^0 . The best estimate for the total number of oxygen atoms striking unit area for the flight was $3.5 \times 10^{20} \text{ cm}^{-2}$. Since the slit area was 0.024 cm^2 , 8.4×10^{18} atoms should have struck the reflectometer carbon surface. Based on the 11.5% reaction efficiencies observed on etched external samples, 88.5% of the incident oxygen should have left the carbon surface as unreacted atoms or molecules. The result obtained was that approximately 600 Å of silver should have reacted at the maximum exposure location for the measured distribution.

This agreement within 20% between the measured and calculated values shows that the sensor is linear over the range used and that the recombination of 0 to 0_2 on silver has a low probability. We note also that the errors in particle densities used in the MSIS model may not be better than 15%.

The method of Nocilla (1) was used to fit the observed scattered lobe. Nocilla's model treats particles emitted from (or impinging upon) a surface as the combination of normal random molecular motions associated with a particular temperature, and a drift or ensemble velocity. The concept is called 'drifting Maxwellian'. We studied two cases, one in which there is assumed to be only one lobe, wide enough to include the small peaks at high θ_r , and the case with a slightly narrower main lobe with a small (2%) supraspecular lobe. The results of the fits are shown in Figures 3 and 4.

We have used Nocilla's notation. Note that the speed ratio s is the ratio of the ensemble velocity to the most probably velocity at the characteristic temperature, $C = (2kT/m)^{\frac{1}{2}}$.

θi S_i T_i U_i θ_i km/s deg U. km7s Sr °₩ 7.8 40° 0.2 8.8 750 Case 1 55° (Single lobe) (see text) 7.8 19° 55° 750 0.6 8.8 Case 2 (excluding specular lobe)

We may not solve further for T_r and U_r unless we either measure the scattered velocities or make some simplifying assumption. For example, if we assume the collision process is adiabatic, i.e., all the primary kinetic energy of the incident beam is converted into heating the scattered gas, we obtain a very high value of $T_r \sim 30,000$ °K, close to the stagnation temperature of the primary beam. Since we know nothing about energy transfer at the surface this merely represents a limit.

4. Discussion

The Nocilla fits gave speed ratios of 0.2 and 0.6 compared with the incident value of 8.8, and wide lobes of at least 80° FWHM. Generally, results of others with rare gases at similar energies but on clean single crystal surfaces show narrow peaks close to the specular angle. We are apparently seeing a high degree of accommodation, characterized by distributions approaching the cosine. It should be noted that our lobe width is affected by the beam divergence (14° FWHM), and by roughness and contamination effects. While the residual pressure in the reflectometer may have been of the order of 10⁻⁵ torr, this gas would have been chiefly 0₂, CO, and N₂, none of which are considered to be strongly bound at carbon surfaces. Contamination by heavier organic molecules, while likely at first, was not expected to last long under the oxidative bombardment of energetic 0 atoms.

Calculations by Chiang and Knuth (15) have shown that physisorbed molecules are sputtered off by hyperthermal gas particles directed at the surface. This does not, however, preclude absorption from the beam itself and, though the oxidation mechanism of carbon is not understood in this regime, it is likely that the surface is covered with chemisorbed oxygen. However, since the masses of 0 and C are quite similar and the force constants of the C-0 bond may be comparable to C-C bonds, it is not clear why a layer of chemisorbed oxygen pattern as observed by others in well defined beam-surface experiments at lower energies.

Further experiments with hyperthermal atomic oxygen beams will be made with well characterized surfaces and scattered velocity measurements in the near future.

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7. Authors Addresses

John C. Gregory, Chemistry Department The University of Alabama in Huntsville Huntsville, Alabama 35899, U.S.A.

Palmer N. Peters, Space Science Laboratory ES64, NASA Marshall Space Flight Center, AL 35812, U.S.A.



FIGURE 2. POLAR DIAGRAM OF ANGULAR DISTRIBUTION OF 5eV OXYGEN ATOMS SCATTERED FROM POLISHED VITREOUS CARBON (ORBITAL FLIGHT DATA). A PURE COSINE LAW RE-EMISSION IS SHOWN FOR COMPARISON. THE CUT-OFF IN THE BACKWARD HEMISPHERE WAS CAUSED BY THE DETECTOR FILM HOLDER.



FIGURE 3. FIT USING NOCILLA MODEL TO THE OUTSIDE ENVELOPE OF THE FLIGHT DATA. DISCREPANCIES ASSUMED DUE TO ABSORPTION ARTEFACTS IN THE SILVER FILM 8, (NOCILLA) = 40°: Sr = 0.2



FIGURE 4. FIT USING NOCILLA MODEL TO THE FLIGHT DATA ASSUMING A BI MODAL DISTRIBUTION. PARAMETERS WERE 0, (NOCILLA): MAIN LOBE 19°, SUPRASPECULAR LOBE 62°; S, (MAIN LOBE) = 0.6, SUPRASPECULAR LOBE, 8.8. ONLY ABOUT 2% OF THE REFLECTED ATOMS WERE IN THE SUPRASPECULAR LOBE.