



``Hot" Atomic Halogen Beams from Sputtering of Silver Halides

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resolution problems but our particular system needs dictated otherwise.

R1 [Fig. 2(b)] limits the base current when the transistor is saturated. When SW1 is closed the BFC is virtually off. The voltage drop through R2 assures the capability of base biasing into saturation. R3 is a current sensor read out through a multiposition switch by a digital voltmeter. The binding posts in series with the BFC can be either shorted or jumpered with a suitable resistance to give a fixed coarse control of the BFC current. The transistor type is specially selected for high collector power and low collector-emitter saturation voltage characteristics. The entire control unit is forced air cooled to insure maximum power capability. One 16-channel unit has successfully operated for over a year without a failure and with a long term current stability of about 2%.

It is a pleasure to acknowledge the contributions of Bruce Nelson and Richard Sumner to this work.

[†] Present address: Argonne Cancer Research Hospital, The University of Chicago, Chicago, Illinois 60637. ¹ L. Lavoie, B. Nelson, R. Sumner, J. Watson, R. Winston, D. Wolfe, and H. Hinterberger (unpublished).

² Private communication, suggested by H. Hinterberger.

⁸ The contribution of Professor T. Romanowski's group at Argonne National Laboratory during this phase of the work is gratefully acknowledged.

"Hot" Atomic Halogen Beams from Sputtering of Silver Halides*

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N sputtering of metals by ion bombardment, it is generally found that atoms of the target material are ejected with an energy distribution extending from thermal energies up to 40 or 50 eV and peaking at 0.5 to 2.5 eV.¹⁻⁴ The same sputtering mechanisms should apply as well to nonmetallic targets, but low thermal conductivity can cause thermal spike sputtering, and low electrical conductivity can cause defocusing of the bombarding ion beam due to charge buildup.

Recently, fast atomic beams of potassium were produced by sputtering methods in two laboratories.^{3,4} From the viewpoint of chemical reaction kinetics (hot atom chemistry), it would be particularly desirable to have available fast beams of species which have already been studied by



means of nuclear recoil techniques,⁵ primarily monovalent iodine, bromine, and hydrogen (tritium). To this end we have investigated the sputtering of silver halides (AgI and AgBr).

AgI targets were produced by reacting I₂ with a metallic silver slab. A layer of iodide was formed (~ 0.05 mm thick) in good thermal and electrical contact with the backing. Electron diffraction showed the layer to be AgI, and electron microscopy indicated well developed crystalline orientation.

Figure 1 depicts the apparatus used in this study. The silver halide targets were bombarded with beams of argon ions ranging in energy from 0.5 to 7 keV. Elements of "source 2" can be used to (a) repel ions coming directly from the target, (b) ionize emitted neutral species with a crossed electron beam, (c) repel ionized neutrals after they have passed through the ion source, (d) apply acceleration voltage, and finally (e) focus the ions into the attached 60° mass spectrometer. The mass spectrometer was set to a resolution $\Delta M/M = \Delta E/E$ of 1 in 500. The translational energy of the sputtered particles was measured in two ways. First, the ionized beam was swept through a slowly decreasing acceleration voltage, at fixed magnetic field, for several mass units at a time. At 1 keV the energy resolution was 2 eV, and a detailed trace of the peaks showed high kinetic energy contributions (see Fig. 2, dashed lines). The second method utilized the repeller plate in "source 2" to suppress ionized particles of low kinetic energy, thereby permitting the study of the transmitted beam as a function of the barrier potential. The solid lines in Fig. 2 represent acceleration voltage sweeps with stepwise increased repeller potentials. The virtual disappearance of masses 108, 110, 128, and 129 after the application of only 2 V barrier potential illustrates the effect of the repeller on peaks due to background gas in source 2. The spectrum taken in the AgI region contains the "cleanest" sputtering results since there is no interfering background in this mass region.

The particles of greatest interest in the present study were halogen atoms. For iodine, inspection of the mass 127 mass peak in Fig. 2 shows not only a very large thermal energy contribution but clearly also a high energy tail.

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FIG. 2. Mass spectra obtained by sweeping acceleration voltage at fixed magnetic field. Electron bombardment ionization gives the number density distribution; E^{-1} correction must be applied for flux distribution comparison with Refs. 3 and 4.

The total 127 peak contained, in addition to the iodine atoms ejected directly from the target, a background due to ambient impurities at 127, as well as contributions from dissociative ionization of HI and I₂. Contributions from dissociative ionization of AgI are small because the total AgI peak was only 10% of the corrected I atom peak. I₂ and HI are found to be produced in large quantities during the sputtering process; however, their energy distribution is almost completely thermal. These species are probably formed by secondary reactions of the sputtered atomic iodine with iodine atoms and water molecules adsorbed on the walls of the sputtering chamber. They are permanent gases and remain in the ion source region. The high energy component in the composite mass 127 must originate from sputtered atomic iodine. In order to assess the "pure" sputtered mass 127 peak, we assumed the I energy distribution to be identical with the AgI distribution and underlaid the AgI and I peaks on a semilog plot. Subtracting the sputtered contribution in mass 127 on this basis yielded two results: (a) The residual peak at mass 127 has a purely thermal shape and (b) the net atomic yield of sputtered iodine is, within experimental error, identical to the sputtered atomic silver yield.

The energy distributions of sputtered I, Ag, and AgI resemble closely those observed for sputtered potassium.^{3,4}

For iodine atoms, the sputtering yield is ~ 1 for bombardment with 3 keV argon ions, the neutral/ion ratio was estimated to be $>10^5$, and, at a kinetic energy of 10 eV, >10% of the maximum intensity was observed. Results obtained with AgBr target were very similar to those described for AgI targets. Mechanical velocity selection is necessary with any sputtered beam source and in this case will eliminate interference from HI and I₂. Some fast Ag and AgI will accompany a fast velocity-selected I atom beam, but these can be discriminated by the detector.

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Hydrothermal Pressure Vessel Assembly for the Measurement of Pressure Generated by Corrosive Solutions

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HYDROTHERMAL syntheses and crystal growth investigations necessitate the use of very corrosive solutions, and it is necessary to use noble metal containers to confine these solvents.¹⁻⁴ When the cold seal "test tube" vessels are used, the reactants and solutions are sealed inside noble metal capsules with flexible walls, which transmit the pressure. There is, however, a need for a container for use with autoclaves of 2.5 cm diameter and larger. Monchamp, Puttback, and Nielsen¹ scaled up the sealed tube technique to large flexible-wall cans which filled most of the chamber of the vessel. This arrangement was not entirely satisfactory because deformation of the walls of the can influenced the convection pattern inside the vessel, and this affected the rate and quality of the growth on some seeds.

After a number of trials, two successful and complementary methods for the measurement of saturated vapor pressures inside small autoclaves were developed. In both methods, the high pressure chamber of the autoclave is lined with a noble metal container and covered with a noble metal diaphragm isolating the solution from the