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QUARTERLY PROGRESS REPORT:

INVESTIGATION OF KILOVOLT ION SPUTTERING

by

HAROLD P. SMITH, JR., F. C. HURLBUT, T. H. PIGFORD,
JHAN KHAN AND N. THOMAS OLSON

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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SPACE SCIENCES LABORATORY
UNIVERSITY OF CALIFORNIA, BERKELEY

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I. INTRODUCTION AND SUMMARY

Contained in this report is a summary of the progress made during the period of time from August through October 1966 on work supported by the Lewis Research Center of NASA. This work, begun in 1964, constitutes an area of surface physics involving the interaction of ions and protons with material surfaces. Specifically, heavy ion (cesium and mercury) surface interactions are observed by measuring sputtering yields, angular distribution of sputtered atoms, energy distribution of ejected atoms and implanted ion distributions within the target lattice. The proton-surface interactions studies constitute an investigation of the proton induced characteristic oxygen x-rays as a possible surface analysis device for measuring oxygen contamination of material surfaces.

The following is a summary of the quarter's progress in each area.

AUTHOR

II. SURFACE DENSITY MEASUREMENTS*

The density of oxygen atoms adsorbed on an aluminum substrate will be measured by the oxygen x-ray yield produced under 100 keV proton bombardment. The experimental apparatus, illustrated in Figure 1, consists of a duoplasmatron ion source that produces H_1^+, H_2^+, and H_3^+, an analyzing magnet to pass only protons into the target chamber, the collimator-target assembly, and the low energy photon detector (gas proportional counter).

With the entire system now assembled, the differential pumping afforded by the cryogenic-sublimation pumps permits operation of the target chamber in the 10^{-10} Torr region with the beam on target. In the target chamber, the beam passes through two collimators (see Figure 2), the first 3/16" dia and the second 1/4" dia, and a beam alignment tube before striking the target. In this arrangement, similar to that used by Khan and Potter,[1] the collimator assembly is biased 300 volts above ground to suppress secondary electrons.

The beam alignment tube serves the dual purpose of limiting the maximum beam divergence to $\pm 1\ 1/2^{\circ}$ and also prevents any scattered protons or secondary electrons from reaching the electron shield where they would give an erroneous target current. Secondary electrons produced by the proton beam striking the alignment tube could also give rise to erroneous target currents, but since the intercepted beam amounts to only 0.5% or less of the target current, the error is small.

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* The work reported in this section was performed by R. R. Hart

The target current is measured with a conventional faraday cage in which the electron shield (Figure 2) is biased 300 volts negative with respect to the target to prevent secondary electrons from leaving the target. To allow for vacuum pumping in the immediate region of the target, two 2" x 2" holes have been cut in the faraday cage and then covered with 250 lines/inch, 70% transmission nickel mesh.

The target holder (see Figure 2) is a ten sided copper heat sink, which at full beam power will limit the target temperature rise to 0.1° C per minute. Typical beam-on-target times will be 10 minutes per target, so that the total temperature rise for a 10-target run will be 10° C.

With this collimator-target assembly beam current, measurements of 5 to 10 microamps of 50 to 100 keV protons (corresponding to a current density of 25 to 50 microamps/cm^2) can be focused on the target. With the system essentially completed, oxygen x-ray yield measurements are planned for the coming quarter.

III. VELOCITY SPECTRUM MEASUREMENT*

The objective of the experiment is to measure the energy spectrum of sputtered particles using a time of flight technique. In particular, a pulsed cesium beam sputters a copper target while a quadrapole mass spectrometer (QPMS), a known distance from the target, detects the time rate of arrival of the copper after an ion pulse.

During this quarter additional cesium ion source troubles were encountered but were overcome by the installation of an ion source loaned by the Lewis Research Center. This new source, along with a beam chopping and transport lens structure, has been installed in the vacuum chamber containing the target and the QPMS. This arrangement is illustrated schematically in Figure 3.

The ion source and the electrostatic lens, illustrated in Figure 4, have been operated DC in the 1 to 10 keV energy range with 100 microamps of cesium ion current focused to a 1/8'' diameter on the target. Typical bias potentials for the pulsed mode at 5 keV ion energy are illustrated in Figure 4. To suppress the ion beam the extractor electrode is biased at 5012 bolts and by coupling the pulser to the extractor through the high voltage isolation capacitor, it can be pulsed down 250 volts to 4762 volts. The extraction potential of 238 volts draws the beam out, passing it on to the focusing lens and the target. To date, in this mode of operation, the pulsed beam when pulsed with 5 μsec square wave at 4K Hz has resulted in a 8 μa square current pulse.

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* The work reported in this section was performed by G. Cowell

For the coming quarter, this performance, as measured by the target current, will hopefully be improved by increasing the pulse voltage. Also, now that the ion source-target-QPMS are assembled the detection system can be tested at the coper mass setting.

IV. MERCURY ION SPUTTERING*

The mercury ion sputtering apparatus described in the previous quarterly progress report (NASA CR-54908) has been used to complete the room temperature sputtering yield and angular distribution measurements at normal, 18^{o}, 45^{o}, and 63^{o} incidence to the (100) face of copper. These results and associated analysis will be presented in the forthcoming final report of the contract.

Work is now progressing on preparation of the nickel and molybdenum targets, and it is expected that similar measurements on these metals will be completed during the coming quarter.

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* The work reported in this section was performed by R. G. Musket

V. ALUMINUM – ALUMINUM OXIDE SPUTTERING*

The angular distribution omeasurements of cesium ion sputtered aluminum have been completed. The technique employed here has been to detect the sputtered aluminum by characteristic x-ray excitation in an electron microprobe. This process is more tedious than the radioactive tracer technique used for the copper so the data, rather than being taken as a function of both the polar and azimuthal position, has been taken in the (110) or (100) planes as a function or polar angle. These results will be included in the final report.

To make sputtering yields on the dielectric aluminum oxide, it is necessary to neutralize the surface charge buildup. This will be accomplished using the arrangement illustrated in Figure 5. The electron emission filament will flood the target surface with electrons such that the posi^tive ion charge will be neutralized and additional electron induced secondary emission conpensates for the excess electron current. The target ion current will be measured periodically by deflection of the ion beam into the faraday cage at the bottom of the collector. Previous experience has shown the source to be very stable so that periodic checks are sufficient.

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* The work report in this section was performed by E. H. Hasseltine

This charge neutralization method is now being developed and it is expected that sputtering yield measurements using the neutron activation method[2] will be performed during the next quarter.

VI. ION IMPLANTATION*

The distribution of implanted cesium ions within the target is being measured by neutron activation of the implanted cesium. Stripping successive surface layers (approximately 40 Å thick) from the target and measuring the cesium in each layer by conventional radioactivety-counting techniques gives the distribution of implanted ions.

Following the preliminary results reported last quarter additional modifications were required for the experimental apparatus. Heat conduction from the source caused an undesirable temperature rise in the target so that forced convection gas cooling was added to maintain the target at 20° C. To facilitate computer data reduction, a program has been written to calculate by the Covell method the amount of cesium present in each thickness of stripped material.

With the experimental equipment complete and the data reduction computerized, several aluminum targets have been prepared for cesium ion bombardment and surface stripping. They include seven each of polycrystalline, (100) surface and (110) surface targets which will be bombarded at 20 ke V for integrated currents varying from 10^{16} to 10^{19} ions per square centimeter.

It is expected that these aluminum targets along with targets of copper and silicon, will be completed this coming quarter.

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* The work reported in this section was performed by W. Siekhaus B. Southworth, and T. H. Pigford

Work is continuing on the development of a slit focusing system for the krypton-argon ion source.

REFERENCES

1. Kahn, J. M., and D. L. Potter, Characteristic K-Shell X-Ray Production in Magnesium, Aluminum and Copper by 60 to 500 keV Protons, Phys. Rev. 133, A890 (1964).

2. Andrews, A. E., E. H. Hasseltine, N. T. Olson, H. P. Smith, Jr., Cesium Ion Sputtering of Aluminum, J. Appl. Phys. 37, 3344-3347 (1966).

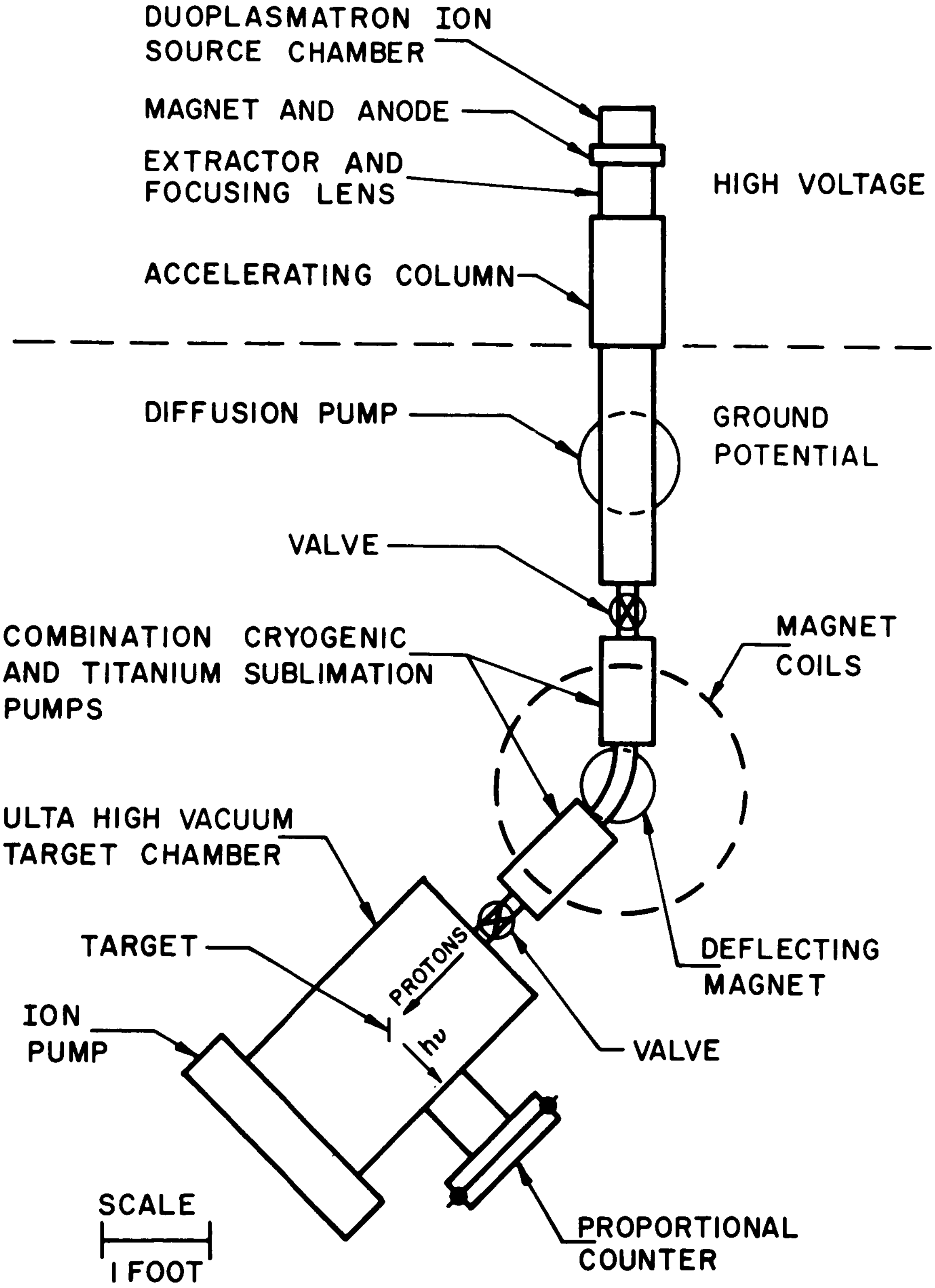

FIGURE 1: Schematic of surface density measurement apparatus.

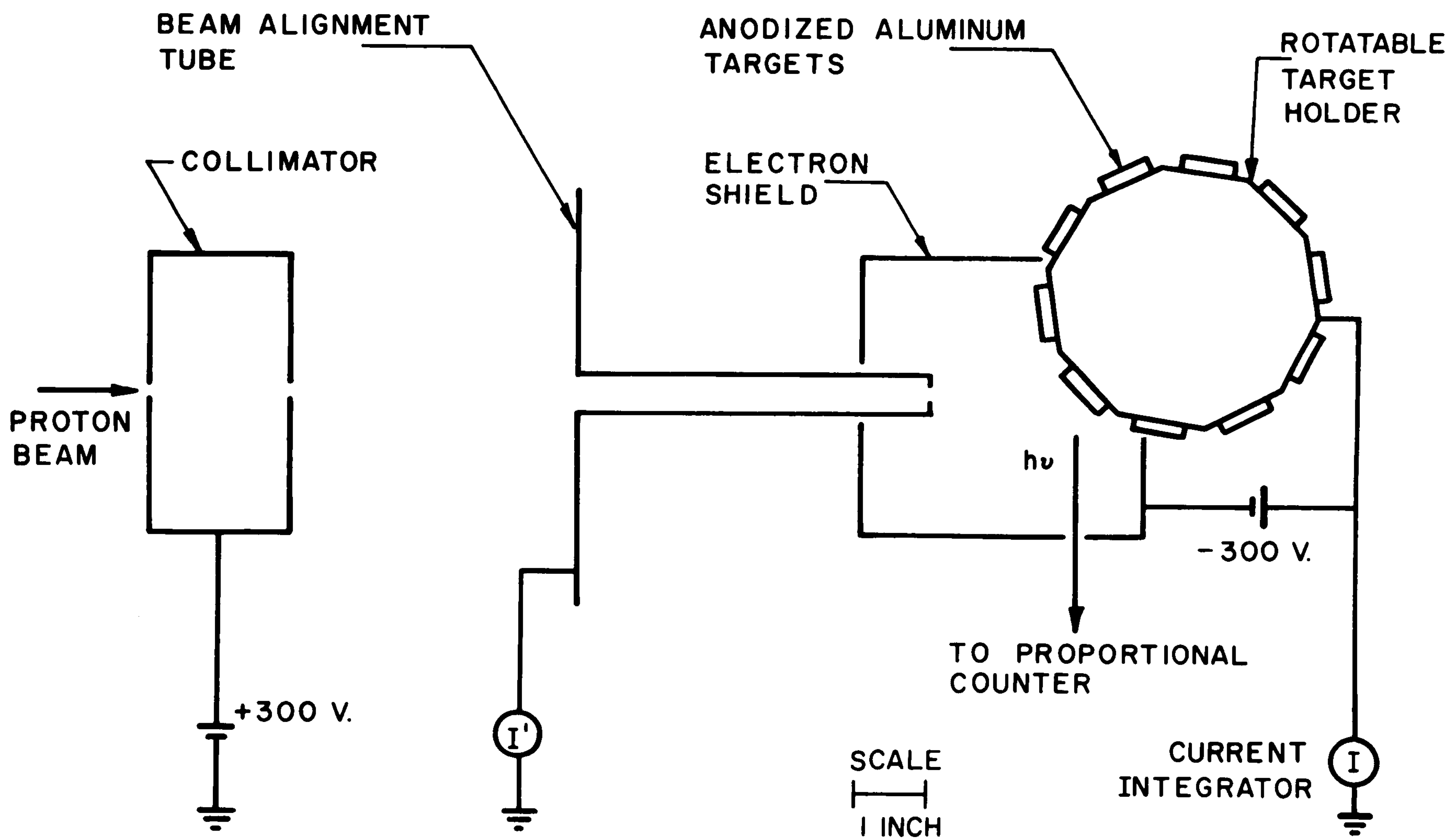

FIGURE 2: Proton beam collimator and target holder assembly.

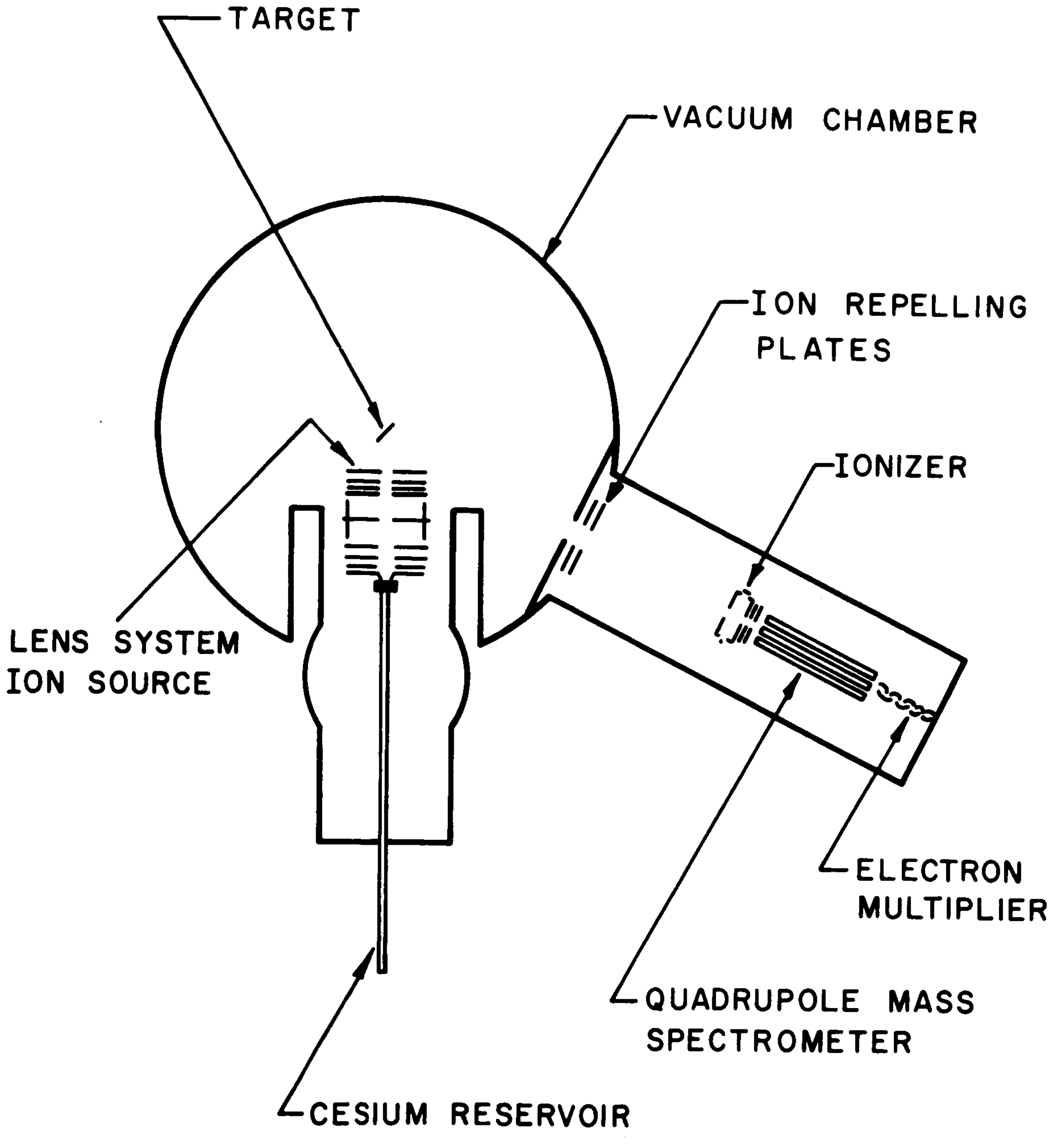

FIGURE 3: Schematic of velocity measurement apparatus.

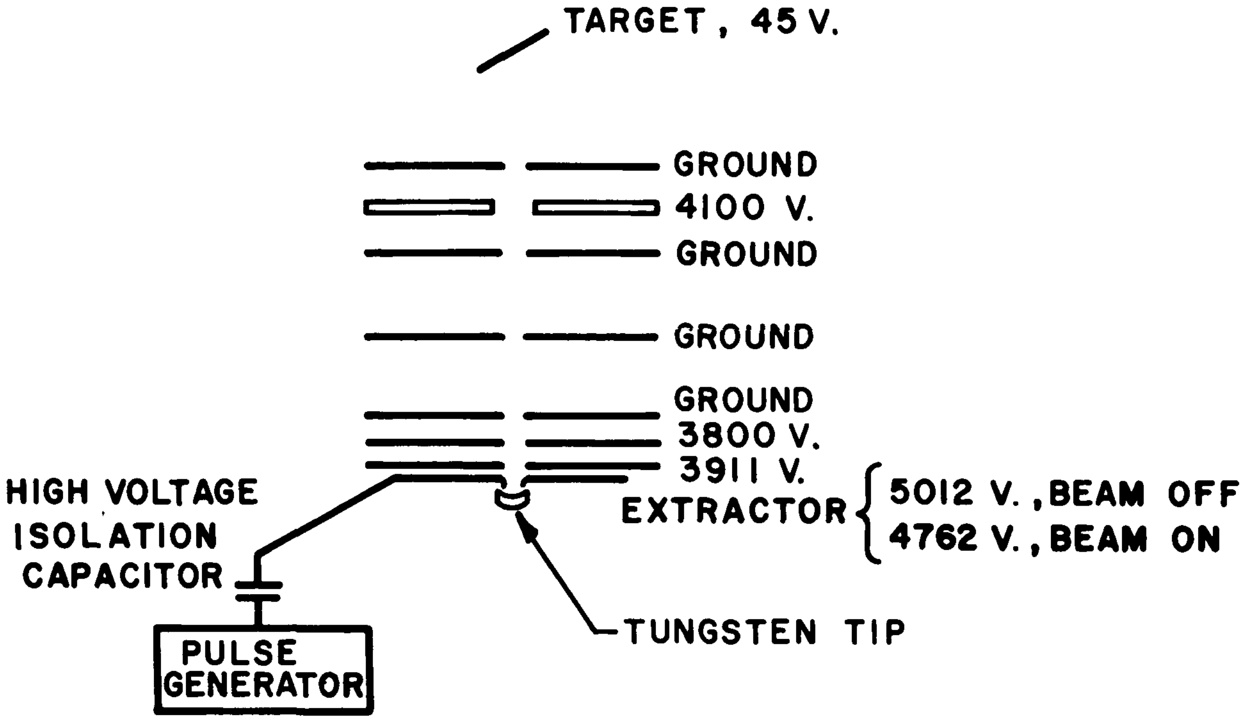

FIGURE 4: Ion beam extractor and electrostatic focusing lens.

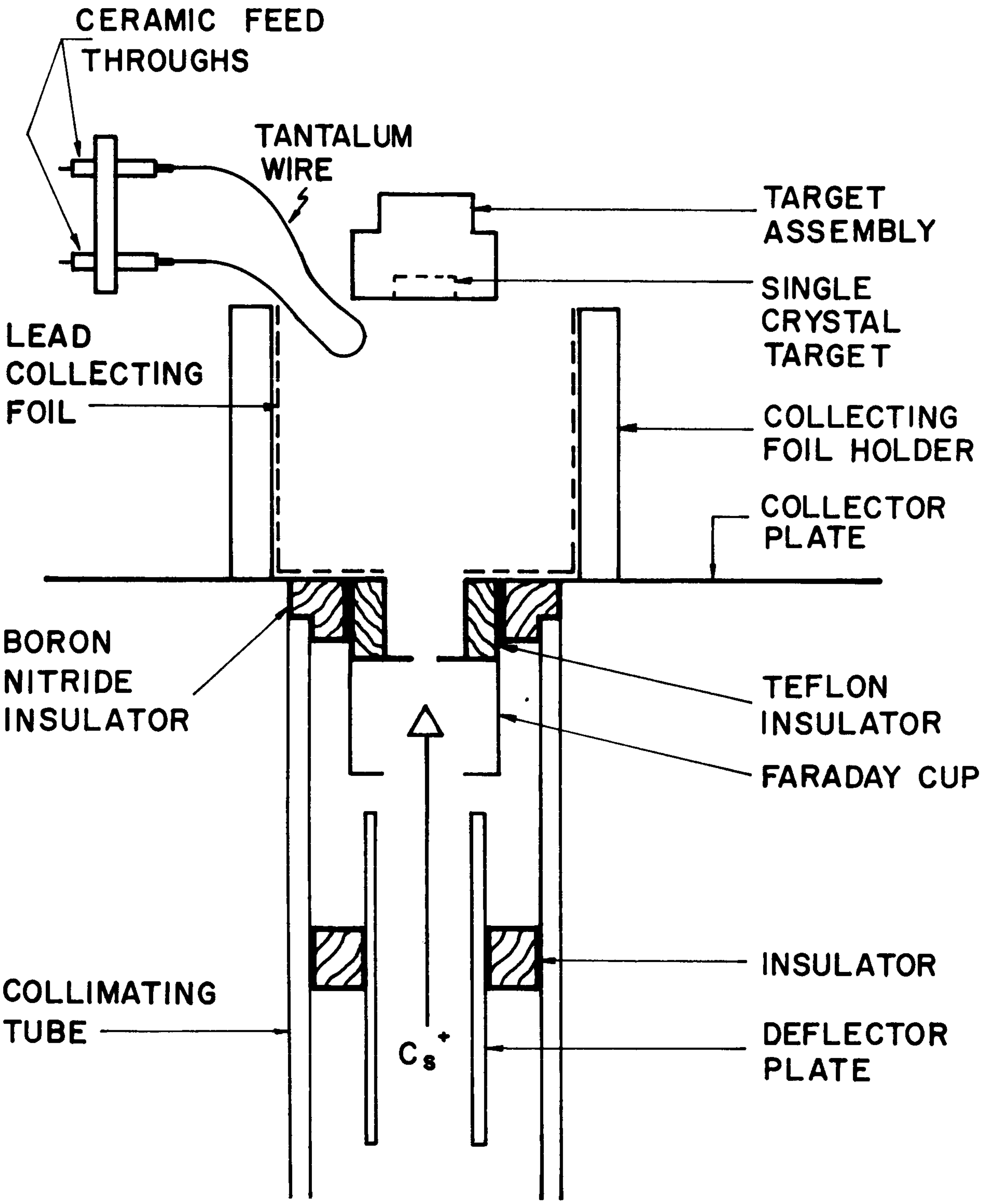

FIGURE 5: Electron neutralization of surface charge build-up and current measurement in Al_2O_3.

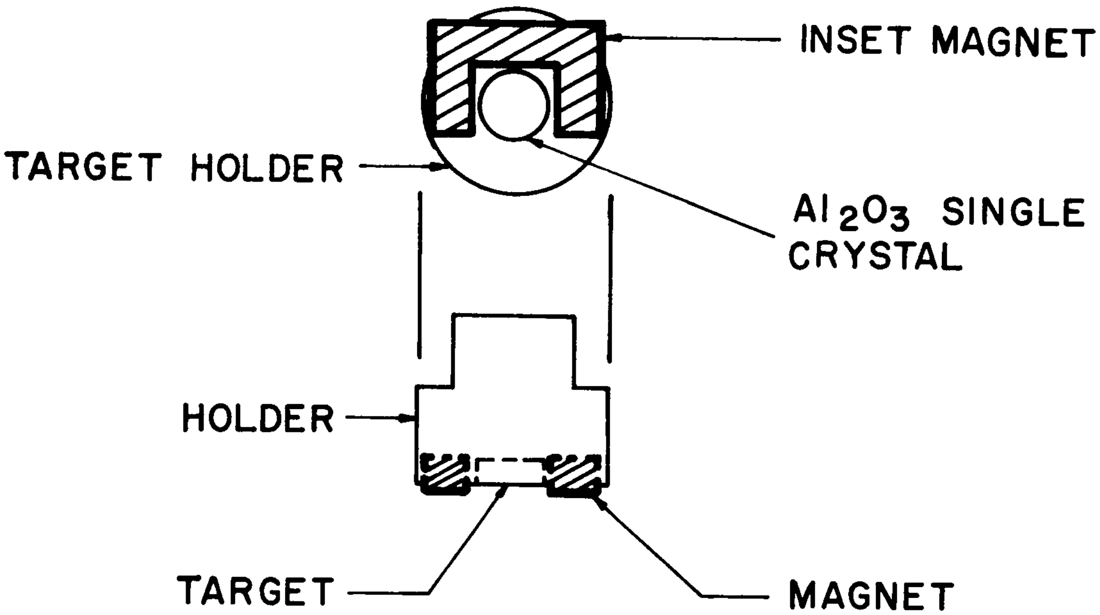

FIGURE 6: Target detail.

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