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QUARTERLY PROGRESS REPORT:

INVESTIGATION OF KILOVOLT ION SPUTTERING

by

HAROLD P. SMITH, JR., F. C. HURLBUT AND T. H. PIGFORD

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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INVESTIGATION OF KILOVOLT ION SPUTTERING

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Harold P. Smith, Jr., F. C. Hurlbut, T. H. Pigford,
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TABLE OF CONTENTS

FIGURES

I. INTRODUCTION AND SUMMARY

Sputtering or ionic erosion of the accel electrode and focusing structure of the ion rocket engine can be the dominant mechanism limiting long term operation of the engine. Although the field of sputtering has been known since the phenomenon of gas discharge was first observed, no reliable theory to predict the yield, angular distribution, and velocity spectrum has been developed. Furthermore, it has only been within the past few years that experiments have been made under suitably defined conditions. In addition, there has been little work with either cesium or mercury beams so that it is difficult to predict the electrode erosion on the basis of previous data. For these reasons, as well as applications to thin film technology and ion implantation, the Lewis Research Center has sponsored detailed investigation of ion bombardment of suitable monocrystalline electrode material where target parameters such as temperature, angle of incidence, etc., are well known and varied over the range of interest.

The University of California (Berkeley) Space Sciences Laboratory began an investigation of this field in 1964. This document is submitted as a report of progress in this investigation for the period of time from May through July 1966.

During the past quarter, proportional counter technique and operation have been improved so that it is now possible to separate completely the oxygen characteristic x-ray peak from the associated low energy noise. Measurement of oxygen surface density is planned for the coming quarter.

Preliminary data is presented in which the electron microprobe has been used to determine the angular distribution of sputtered aluminum. This technique offers improved resolution over the radioactive tracer technique previously developed for cesium-copper sputtering. Preliminary data is also presented in which neutron activation analysis after cesium ion bombardment of aluminum has been successfully incorporated with the standard anodization-strip technique. As expected, the median range of penetration is less than that encountered at low dose rate.

II. SURFACE DENSITY MEASUREMENTS*

The surface density of oxygen atoms on an aluminum substrate may be determined by detecting and counting characteristic oxygen x-rays produced by bombardment with a 100-KeV proton beam using a gas proportional counter, coupled with suitable pulse height amplifying and pulse counting equipment.

The low energy of the characteristic oxygen x-rays (500 eV) places stringent requirements on the x-ray detection system. The proportional counter window must be extremely thin for good transmission and yet be able to withstand sufficiently high counter gas pressure for adequate gas amplification. As previously reported[1] a thin (4,000 Å) alumina window assembly, which allows 60% transmission of characteristic oxygen x-rays and can withstand 100 Torr counter gas pressure, has been constructed. However, with maximum gas amplification, i.e. proportional counter gas pressure of 100 Torr and maximum counter voltage, the pulse heights corresponding to characteristic oxygen x-rays were less than the electronic noise level of standard pulse amplifiers. Consequently, the standard pulse amplifying equipment was replaced by a very low noise commercial field effect transistor preamplifier and mating pulse shaping amplifier.

Characteristic oxygen x-rays were produced by bombardment of an anodized aluminum target with the duoplasmatron ion beam. Using 90% argon, 10% methane proportional counter gas, the pulse height spectrum was obtained with a 400-channel pulse height analyzer. At

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

maximum gas amplification, the characteristic oxygen x-ray peak was resolved, but the peak width (500 eV at half maximum) was so broad that the leading edge of the peak was not completely clear of the electronic noise level. The proportional counter gas was changed to 96% helium, 4% isobutane, which produced a much narrower peak (300 eV at half maximum) apparently due to greater penetration of the characteristic oxygen x-rays into the active volume of the proportional counter. The peak was then clear of the electronic noise level.

A target holder assembly that will permit up to ten targets of varying thicknesses of anodized high purity aluminum to be individually positioned in the proton beam within the ultra high vacuum target chamber has been designed and partially constructed. In comparison to a single target capability, the time required for characteristic oxygen x-ray yield measurements as a function of oxygen atom surface density will be greatly reduced, since only one pump down to ultra high vacuum will be required for each set of ten targets.

In order to separate the proton beam component from the total duoplasmatron ion beam, an analyzing magnet has been installed.

It is expected that characteristic oxygen x-ray yield measurements as a function of oxygen atom surface density will be performed next quarter.

III. VELOCITY SPECTRUM MEASUREMENT*

In performing an analysis of the velocity spectrum of sputtered particles, the parent ion beam is first modulated by electrostatic techniques and then allowed to impinge on a target as a series of discrete pulses. Since the time lapse in sputtering is of negligible magnitude in comparison with flight times of the sputtered particles and since the ion flight time from modulator to target is known, spectra of arrival times of sputtered particles on a detector can be translated into spectra of velocities for these particles. Analysis proceeds straightforwardly if the parent ion beam is monoenergetic. The elements of an arrival time analysis system are modulated ion source, mass spectrometer, multiplier detector, signal amplifier train, and data acquisition system. Progress in the last quarter in the development of these system elements is indicated below.

Following breakdown of the previous ion source, a new source has recently been obtained. Although the new source is of different design no prolonged difficulties in the redesign of the lens and beam transport system are anticipated. Modulation of the ion beam will be accomplished by the extractor pulse method. A generator to produce the required square pulse has been obtained and the amplifier previously built for this project will be used to provide the final pulse to the extractor electrode.

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*The work reported in this section was performed by G. Cowell, M. Kosaki, and N. T. Olson

The high vacuum system in which the experiment will be performed has been maintained at 6×10^{-9} Torr. The system has been reoriented so that the newly acquired source may be operated in a vertical position (necessitated by source design).

Although the quadrupole mass spectrometer had broken down several weeks ago, it is now operating properly.

The detection and counting system is in its final stage of development. A scaler gating circuit to be triggered by the ion beam pulser is currently under construction. Except for a power supply bin that is undergoing repair, the rest of the system — electron multiplier, charge-sensitive preamplifier, pulse-shaping amplifier, timing discriminator, two scalers and a variable time-delay gate-pulse generator — is assembled and in operable order.

IV. MERCURY ION SPUTTERING*

The apparatus for mercury ion sputtering has been completed and successfully tested. The system can now provide a magnetically analyzed mercury ion beam of about 10 - 20 $\mu a/cm^2$ at the target with a target chamber pressure below 2×10^{-7} Torr. Measurements at Hg^+ ion energies of 5.0, 7.5, and 10 KeV can be made without any further changes; while measurements for 1.0 and 2.5 KeV must await additional refinements in focusing. The preparation of the copper crystals for the higher energy measurements has been initiated.

Insertion of a circular aperature at the exit of the magnet chamber has decreased the energy spread in the ion beam. A retarding potential technique has been used to determine the energy spread of the Hg^+ ion beam at the target assembly. The energy spread in the 7.5 KeV Hg^+ ion beam was found to be less than 50 eV. Thus, in the least favorable case (ion energy of 1 KeV), $\Delta E/E_0$ will be less than five percent. It should be recalled that the ion beam is transported at a high energy (7.5 KeV), with the target assembly biased to provide the desired ion energy at the target.

A schematic diagram of the mercury ion apparatus is given in Figure 1. Since the ion beam moves in a horizontal plane, the schematic represents a top view with a horizontal cut through the beam center line. Although not shown, the source chamber has a cryogenically baffled six inch oil diffusion pump and a cylindrical cryogenic pump as vacuum.

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

pumping elements. It should be noted that the total ion beam path distance is about 120 cm. All chambers are constructed of 304 stainless steel while the isolation valve is made of brass. The isolation valve allows the pressures in the ends of the system to be completely independent of one another. After a sputtering measurement has been completed, the valve is closed and the target chamber brought to atmospheric pressure so that the collector assembly may be changed while the source remains at vacuum. After the collector is changed, the target end is pumped down, and when proper vacuum conditions are obtained the valve may be opened for another measurement. The source end employs O-rings for vacuum seals, while it is possible to use only metal seals in the target end. The auxiliary port of the target end may serve a variety of purposes but was specifically designed to accomodate a quadrupole mass spectrometer. Alignment requirements dictated that all chambers through which the ion beam passes be rigidly connected. One flexible section has been used to join the target chamber to the vac-ion pump.

It is hoped that most of the angular distribution measurements on copper, nickel, and molybdenum can be completed during the coming quarter.

V. ALUMINUM SPUTTERING*

The apparatus previously described[1] was assembled and tested at operating conditions. Optimum parameters for ion beam focusing were determined at various energies between 1 and 10 KeV. Using these parameters, data was taken to determine the angular distribution of aluminum atoms sputtered by cesium ions of 2.5, 5.0, 7.5 and 10.0 KeV. These were made at room temperature under conditions such that the poisoning ratio[2] was 0.1 or less. A further run at 7.5 KeV and 77° K was also made. The distribution data were obtained using the electron microprobe technique and are presently being analyzed. Data for the run at 7.5 KeV and room temperature are shown in Fig. 2.

Preliminary arrangements for the determination of the sputtering yield and angular distribution of Al_2O_3 have been made. Methods of monitoring the beam current and neutralizing the surface charge buildup on the dielectric material have been devised. The current will be determined by periodically swinging a beam flag into the beam with a solenoid. The relative constancy of the ion current in this apparatus has been verified during the above runs. The surface charge will be neutralized by electron bombardment using a heated tantalum wire. Angular distributions will be determined using the electron microprobe whereas the yield will be determined using the neutron activation technique, both of which have been fully described in previous progress reports.

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

VI. ION IMPLANTATION*

The earlier problems of surface irregularities resulting from extensive ion bombardment appear to have been solved through proper preparation of target surfaces and by sweeping the ion beam far beyond the apertures defined by the focusing electrodes. The beam sweeping system has been improved by the construction of a linear sweeping circuit, which moves the beam at 1000 cycles/sec in one dimension and at 60 cycles/sec in the other dimension of the target surface. Aluminum targets have been irradiated to 10^{17} ions/cm^2 without visible alteration of the microscopically clean surfaces.

Several targets of polycrystalline and monocrystalline aluminum have now been bombarded with 15 and 20 KeV cesium ions and have been neutron irradiated for activation analysis of the implanted cesium. These samples are now undergoing electrochemical surface oxidation, followed by chemical dissolution of the oxidized layer, to determine the location and relative amounts of implanted cesium. At present the amount of aluminum removed in each such surface treatment is determined from the calibration reported by Davies.[3] A new calibration using bulk impurity activation in the aluminum will soon be made in this laboratory for the high ion bombardments characteristic of the present experiments.

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

Preliminary results for polycrystalline cesium bombarded to 10^{17} ions/cm^2 by 20 KeV cesium, obtained by measuring radioactivity levels of Cs^{134} in the anodizing and stripping solutions, are shown in Figure 3. The indicated depth of penetration and shape of the penetration curve appear reasonable for 20 KeV cesium onto aluminum. The comparison with penetration in lightly bombarded aluminum of the same initial preparation is yet to be made.

Focusing electrodes in slit geometry are now being constructed for the radioactive-ion source. This source has been successfully tested with krypton and argon.

REFERENCES

1. H. P. Smith, Jr., F. C. Hurlbut, and T. H. Pigford, "Quarterly Progress Report: Investigation of Kilovolt Ion Sputtering", NASA CR-54907, April 30, 1966.

2. A. E. Andrews, E. H. Hasseltine, N. Thomas Olsen, and Harold P. Smith, Jr., Cesium Ion Sputtering of Aluminum, J. Appl. Phys. (in press).

3. J. A. Davies, J. Friesen, and J. D. McIntyre, Can. J. Chem., 38, 1526 (1960).

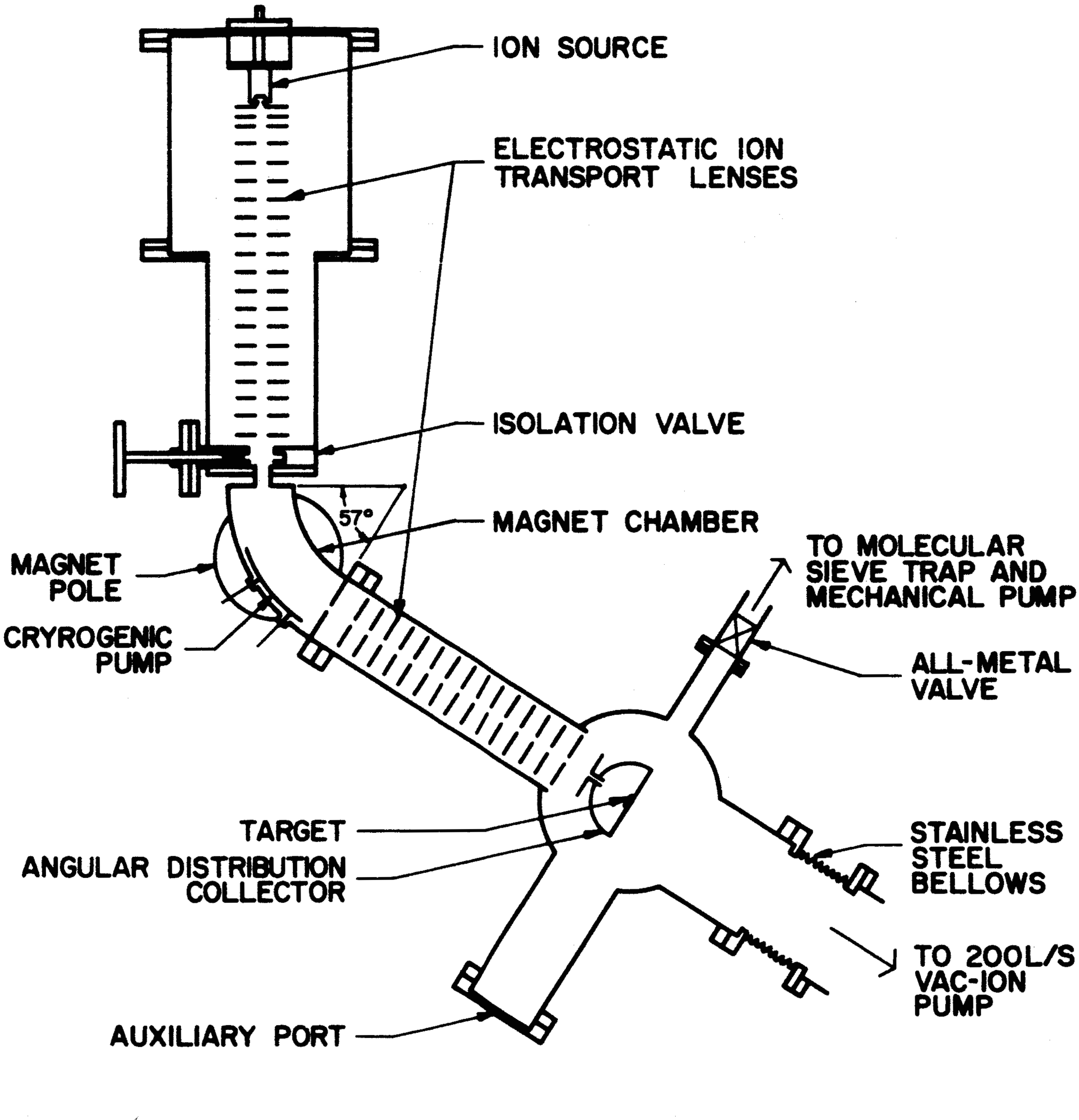

FIGURE 1: Schematic of mercury ion sputtering apparatus.

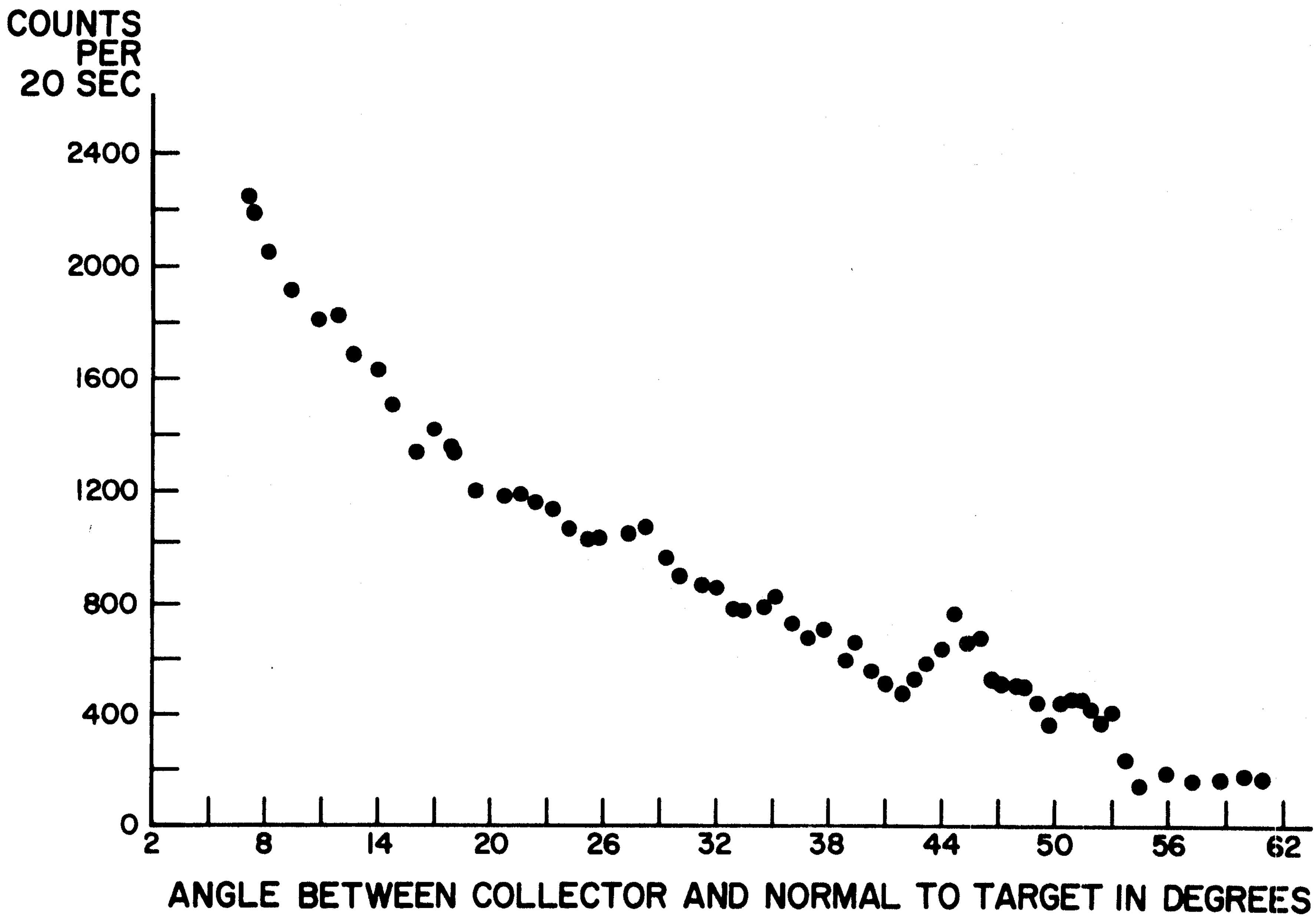

FIGURE 2: Relative angular distribution of aluminum sputtered in the (010) from the (001) face by cesium ion bombardment at 7.5 KeV and at normal incidence. The count rate is proportional to the surface density of aluminum collected at the indicated polar angle on a flat plate parallel to the (001) face.

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