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COLOR PRODUCTION FROM ENERGETIC IONS IMPINGING ON METALS

Kenneth W. Ehlers

June 25, 1962

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ABSTRACT

It has been observed that energetic gaseous ions mark their region of impingement on certain metals in color. Under identical conditions of bombardment, multicharged ions of the same gas mark their impact areas with different colors. The colors are sensitive to the type and energy of the ion as well as to the metal bombarded. These colors may result from reflections from thin films formed by reactions at the target. Because the colors indicate that thicker films are produced with increased ion energy, ion penetration depth as predicted by theory is compared with the depth indicated by the colors observed.

COLOR PRODUCTION FROM ENERGETIC IONS IMPINGING ON METALS*

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During analysis of the ionic output of experimental ion sources operated in a mass spectrometer arrangement (Fig. 1), it was observed that energetic ions mark metal surfaces in an interesting manner. This visible indication of the region bombarded by the various ion beams is not always what would be expected from a freshly sputtered metal surface, but is often brilliantly colored. In the region of ion energy of this investigation--approximately 5 to 50 keV--these colors have ranged throughout the visible spectrum from violet to red. The colors, which seem extremely reproducible, depend on the type and energy of the impacting ion as well as the kind of metal bombarded.

The ion source being tested, and subsequently used for this investigation, was designed to operate continuously and to produce beams of tens of milliamperes of multiply charged heavy ions. The Coulomb repelling forces in ion beams with such high current density often spread the beam, in line with the magnetic field, far beyond the limits of the current-monitoring Faraday cup. Therefore, to obtain an indication of the limits of the beam, I placed an aluminum plate at the 180-deg focal plane of a multiply charged nitrogen ion beam. I had previously observed that energetic ions would mark the region of their impingement, but the effect had never been investigated as I had assumed that the marks were caused by sputtering or a breakup of adsorbed molecules on the surface of the metal. Upon removal of the plate I noted that the ion beams had not only marked the surface of the aluminum, but also that the marking from each of the charged states of nitrogen was a different color. In Fig. 2, a photograph of this original plate, the largest line to the right (N^+) is an olive-brown color. The line formed by the N^{2+} ions is blue, and the N^{3+} is yellow. The faint background markings were made by the N^+ ion beam as the extraction potential was raised in several steps to 11.6 keV.

Additional plates were exposed to the ion beams and it was determined that the effect was nicely reproducible. Although the colors are quite distinct, it is possible that this effect could have gone unnoticed except for the presence of the multiply charged ions of the same gas which, under the same operating conditions, had resulted in different colored markings. For each line, nitrogen was the impacting element; however, as $W = neE$, the energy W of the more highly charged ions is a direct multiple of the number of times n each is ionized. The E in this case represents the extraction potential or positive bias applied to the arc structure.

To determine whether the ionic charge variation was in itself a color-determining parameter, aluminum plates were bombarded at half potential and, in this case, the markings left by the N^{2+} assumed the same color as N^+ at full potential. In Fig. 3[†] the colors of marks produced by N^{2+} change from light brown to blue as the energy increases from 14.8 to 26 keV. Markings from N^{3+}, in turn, change from blue to yellow as the energy is increased from about 22 to 39 keV. Thus the indications are that the resulting color is a function of charge only in that it affects the energy of the ion.

Although nitrogen ions mark aluminum extremely well, they are ineffective in marking copper. With a 10-kV extraction potential and the ion source operating on air, the copper plate shown in Fig. 4 was bombarded. It can be seen that the first three charged states of nitrogen succeed only in sputtering away the copper surface. The oxygen ions in turn have marked very clearly with colors ranging from violet to yellow.

In Fig. 5 a combination of metals was bombarded with He^+ as well as with traces of nitrogen and oxygen, which were impurities left within the source structure from a preceding operation. In this case a single aluminum plate was placed normal to the magnetic field and parallel to the upper edge of the extracted ion beam. The purpose of this plate, which defined the upper edge of the ion beam, was to partially neutralize the repelling forces of space charge.[1] As can be seen, under the same operating conditions the metals mark to varying degrees and in various colors, molybdenum being the most difficult to mark with the gases tried. The aluminum neutralizing plate, in this case and in others tried, is not marked by the ion beams that graze its surface.

When an ion in the energy region of 5 to 50 keV strikes a metal surface positioned normal to its trajectory, it has sufficient energy to penetrate the lattice of atoms that comprise the target surface and lodge within. The impacting ion need only overcome barriers of the order of 10 eV to dislodge a target atom from its preferred location to an interstitial site, or to sputter away an atom located near the surface.[2] After a period of bombardment the lattice consists of normal vacancies (Frenkel defects) plus many dislocations caused by the energetic ions. It is in the velocity region of these ions that the cross section for elastic nuclear collision becomes large and dominates the ionization cross section, and since the nuclear collision is effective in producing atomic displacement it is in this energy region that the maximum radiation damage can occur.[3]

The capability exists for the formation of alloys of the target element and the impinging ion. These alloys can be of the nature of stable chemical compounds (such as the oxides and nitrides), which in this case must remain stable in the presence of vacuums of the order of 10^{-6} mm Hg, and at target temperatures which may reach several hundred degrees centigrade. It may well be for this reason that nitrogen was unable to mark copper (Fig. 4), as nitrides of copper, should they form, would be extremely unstable in the environment described.[4] The same is true of the nitrides and oxides of silver,[4] and when an uncooled silver target was bombarded, it too showed only sputtering indications in the region impacted by oxygen and nitrogen ions.

If the impinging particle were an inert gas ion, an alloy could exist in the form of a solid solution which may or may not be stable. Barring diffusion, however, all the possible effects and reactions between the target and the incoming particle should exist in a thin layer extending in from the surface of the target. The thickness of the layer should be a function of the range energy of the particular combination of energetic ion and target. If the thickness of this layer, including its index of refraction is equivalent to a quarter of a wave length (or odd multiples of $\lambda/4$) in the visible spectrum, the layer may appear colored, as in Newton's rings. Making this assumption, and including a unity index of refraction, the first set of colored rings ($\lambda/4$) would indicate a film thickness ranging from 1000 Å (violet) to 1750 Å (red).

To check this hypothesis it was necessary to have range-energy information in an energy region for which there is very little experimental evidence. On the theoretical side, however, the penetration into metals of ions with an energy of 50 keV and less has been discussed by K. O. Nielsen.[5] By incorporating the theoretical considerations of N. Bohr,[6] Nielsen has arrived at expressions for predicting the range energy of ions in metals. The expressions relate to the two situations in which the target atom (A_2, Z_2) is either heavier or lighter than the impacting atom (A_1, Z_1). At the velocity of ions in this energy region, the cross section for electron capture is generally of such magnitude that charge can be neglected, and the incoming particle is regarded as a neutral atom throughout its process of slowing down. Nielsen's expressions have been used to calculate the penetration depth ($\mu g/cm^2$) of some of the ion-metal combinations that have been tried. Figure 6 is a plot of the penetration depth (in angstroms) for several combinations in which the impinging ion is heavier than the target atom. The distribution in density of particles trapped at various depths within the target, as shown in the insert in Fig. 6, is that reported by Nielsen. The position t on the abscissa of the distribution curve is the depth plotted.

Cases in which the incoming ion is lighter than the target atom are plotted in similar fashion in Fig. 7. Here the distribution of particle density with penetration depth will be somewhat different, as the impinging particle in almost every collision suffers a large deflection, and diffusion effects dominate the slowing-down process. The value of the width of the distribution curve at half height $(2\tau)^{1/2}$ is the depth plotted in Fig. 7. It will be noted that in both cases the penetration depth is a linear function of the ions' impacting energy.

With the Gaussian type of distributions in both cases there is considerable range straggling, and the penetration depths, as plotted in Figs. 6 and 7, become only a measure of the range. Thus to pinpoint a range experimentally becomes difficult, as the indicated range depends upon how deeply target saturation extends into the tail of the Gaussian. This in turn becomes a probability function which depends upon the number of ions required to saturate the deeper levels of the range, while as the same time the surface is being removed by sputtering action. In this energy range sputtering effects in most cases reach a maximum. After the target surface has been sputtered away for a while, the distributions in both cases will probably appear quite similar.

If the impinging ions remain in the target lattice by forming a thin film alloy, the density of the alloy will certainly be different from that of the parent metal. If the density changes during the bombardment, then the effective range will change as the target becomes saturated. The formation of thin target films will also affect the sputtering ratios, as the films formed may be either more or less sputtering-resistant than the parent metal. It is known, for example, that the oxide film on aluminum is much more resistant to sputtering than aluminum itself.[7] If the sputtering ratio (atoms removed per impinging ion) is high, it is difficult to see how any thin film can be produced.

In this short investigation into the cause of the colored ion markings, nitrogen on aluminum was the ion-metal combination that received the most attention. To minimize effects from impurities within the metal, 1100-series (2S) aluminum was used throughout. Before insertion into the vacuum system the metal received the standard dipping or pickling process, which includes alternately dipping in a hot caustic solution and nitric acid. This process removes the protective oxide film prior to attacking the metal surface.[8] A fresh oxide film re-forms almost immediately, however, and therefore it was important to know whether the observed colors were at all related to the oxide film, despite the fact that in all probability it was less than 100 A thick.[8] A mechanical scraper was constructed which was operated through a vacuum seal in one wall of the vacuum chamber, and the aluminum surface was repeatedly scraped while undergoing bombardment by nitrogen ions. No differences in color could be noted between areas that had an oxide coating or had had the oxide coating physically removed.

There is a minimum exposure time for a colored mark to form, which seems related to the current density of the ion beam as well as to its energy. Regardless of energy, singly ionized nitrogen marks first, N^{2+} next, and so on. An approximate minimum exposure time has been experimentally determined to be equal to the number of charges required to assure one gas ion for each metallic atom to the expected penetration depth. For aluminum this results in a charge of approximately 0.1 millicoulomb per cm^2/A. For the ion source used the extracted beam current of singly ionized nitrogen was greater by about one order of magnitude than that of the triply ionized nitrogen. In terms of actual nitrogen atoms this factor becomes 30. Although the penetration distance of the N^{3+} is greater, reduced space-charge effects do not spread the beam over such a large area, and thus the increase in required exposure time over the time required by the N^+ to mark is somewhat less than one order of magnitude. Although a current analysis has always revealed a small current of N^{4+} extracted from the ion source, a colored mark from this ion has never appeared during the various exposures, which have ranged from 5 minutes to 1 hour. The minimum-exposure-time approximation indicates that from the amount of N^{4+} beam present, exposure time in excess of 10 hours would be required before this mark would appear.

Because the current density of the impacting ion beam is not uniform, the mark it makes on the metallic target is not always uniform in color. In general this is most evident at the edges of the beam, where the current density approaches zero, and the borders of the mark are often of a different color from the area impacted by the bulk of the beam. These border colors indicate a thinner film thickness by being of a color more toward the violet than the principal color of the mark. The color of a mark has also been observed to change during the initial exposure to the beam. Typically, the mark produced by a 30-keV nitrogen ion on aluminum (N^{3+} at 10 kV extraction potential), is violet or blue when first discerned; however, after approximately 10 minutes of exposure time, the mark changes to yellow with a light-blue border. Additional exposure time results in no additional color changes, although the area of the mark grows to include the lower-current-density regions of the beam.

The data in Fig. 8 have been plotted on the assumption that the variation in colors observed is a result of a variation in thin-film thickness as indicated by reflection. In this case $t = \lambda/4\mu$, where t represents the film thickness in angstroms, μ the index of refraction, and λ the wave length (in A) of the color observed. Also, the theoretical range of energetic nitrogen ions in aluminum (from Fig. 7) is compared with the penetration depth as indicated by the marking colors obtained experimentally.

Singly ionized nitrogen ions in the energy region below about 12 keV mark the aluminum surface with a brown color. This also is true of the molecular ions, N_2^+, in which the relative particle energy is equal to one-half the extraction potential. The marking color in this lower energy region is quite consistent, ranging from various shades of brown to near black, and is assumed to be due to the destructive interference that results from thin films whose thickness is less than $\lambda/4$.[9] As the ion energy increases above 12 keV the markings become colored, starting with violet and progressing in order (blue, green, yellow, orange, red) with increased ion energy. Owing to the inability of the source structure to hold high extraction potentials for extended periods of time, no red mark was ever observed until this difficulty was overcome. The red marking was then obtained with nitrogen ions of about 50 keV energy (N^{3+} at 16.5 kV extraction potential).

The curves in Fig. 8 are sufficiently similar to suggest that the colored indications obtained experimentally are truly indicators of range energy. Since it would be difficult to assign an index of refraction to the thin-film medium, a unity index of refraction is assumed in Fig. 8. Should the effective refractive index by greater than unity, the penetration as indicated by color would be less. This would bring the two curves closer together; however, it would change the slope of the experimentally obtained curve. That the experimentally obtained data do not appear to return to zero is interesting, and may actually suggest that the target density decreases during the bombardment.

Other ion-on-metal combinations with $A_1/A_2 < 1$, such as oxygen on copper, chromium, and aluminum, and nitrogen on magnesium, produced similar characteristics. Reproducible results with nitrogen on magnesium were difficult to obtain, apparently because of the high vapor pressure of magnesium, which tends to evaporate when exposed to moderate amounts of beam power.

Nitrogen on beryllium, a combination with $A_1/A_2 > 1$, was tried, and it proved very interesting. As the density of beryllium is small (1.82 g/cc), energetic nitrogen ions can penetrate to greater depths (Fig. 6). Figure 9 shows the beryllium bar mounted on an aluminum plate. Close observation of the marks made by nitrogen ions with energies in excess of approximately 20 keV reveals that the edges are composed of a full spectrum of colors. The border colors of the mark made by N^{2+} at 26 keV in Fig. 9 change in very narrow bands from yellow through red, and back into the violet. Thus to plot the penetration depth shown in Fig. 10 the colors were recognized in order through the first set of colored bands ($\lambda/4$) and on into the next set of colored bands ($3\lambda/4$). The actual color of the main area of ion impact was difficult to define and of no use in attempting to determine the film thickness. A considerably longer exposure time (approximately 1 hour) was needed to fully develop the marks made at higher energy, and as visual observation of the target during bombardment was not adequate it is not known for certain that the marks were fully developed. Once again, however, the penetration depth as indicated by color (Fig. 10) was in excess of that predicted by theory.

A number of metal targets have been bombarded with helium, since with this inert gas it is not very probable that true chemical combinations of the gas with the target would occur. As noted in Fig. 5, helium does mark the metals to varying degrees. Perhaps the most interesting combination, and the only one thus far investigated to any degree, is He^{+} on copper. When first observed (Fig. 11),[†] the mark left by the 10-keV He^{+} ions was a bright red, and, unlike the markings thus far discussed, the area of impingement appeared to be covered by a thin granular coating which obliterated the copper surface. It was later observed that when the target was adequately cooled this effect disappeared, and although the copper was marked, the red granular coating was gone. Thus the color of the marking of helium on copper appears temperature-sensitive, a condition which was not true for the films previously discussed and which could be chemical in origin. It is estimated that the average uncooled target temperature did not exceed 300°C; however, the effective temperature in the region bombarded is difficult to determine, and because of this temperature dependence no attempt has been made to correlate ion penetration depth with color.

An uncooled copper plate was bombarded with 10-keV He^{+} ions, and the marked area of the target removed for helium analysis. The total helium content of the copper sample was 1.78 micromoles, which represented slightly less than 2% of the total amount of helium that had impacted the copper. During the analysis the evolution of helium from the sample was the greatest in the temperature range of 700 to 1000°C, and more than 99% of the total helium evolved was obtained before the copper sample melted. If this amount of helium had been contained in the copper sample of known area to a depth no greater than that predicted by theory (Fig. 7), the volume of copper involved would have contained more than 2000 equal volumes (STP) of helium. The copper would thus occlude 258 cm^{3} (STP) of helium per gram of metal, which is quite similar to the amount of hydrogen that can be occluded by titanium.[4] The helium would be contained at a density which would be greater than liquid helium by a factor of 3, a condition which also exists with hydrogen in the case of titanium hydride. As the helium was most probably in solution,[10] it is possible that some diffusion to a greater depth could have occurred during the short period of bombardment, but it is more probable that considerable amounts of helium evolved from the target surface before the sample could be analyzed.[11]

Regardless of what may be the true cause of the colored indications of ion impact, the effect has already proven an extremely useful research tool. It allows a permanent photograph to be made showing a cross section of various ion beams at various positions. Thus it can be used to indicate the effect of changes in ion optics or the effects on the beam shape of the repelling forces of space charge. Once familiar with the color scheme produced by a certain energetic ion-metal combination, one can even analyze the atomic component of an ion beam. Figure 12, for example, shows a spectrum that was made to determine the inherent contaminants within the test ion source structure. In this case, in the presence of a vacuum of 2.7×10^{-7} mm Hg, the source filament was heated, arc voltage applied, and a weak ionizing current which was space-charge-limited by the physical spacings within the source was made to flow. With a positive bias of 16 kV applied to the source structure, a weak ion beam of less than 30 microamp could be extracted. The aluminum plate, located at the 18-deg focal plane, was exposed for 1/2 hour, which was sufficient to show that of the contaminants present, nitrogen was by far the largest. This spectrum was made primarily to determine if carbon was present to any large degree, and as can be seen, carbon is present (mass 44 = CO_2^+ and mass 12 = C), but not in large amounts. Although mass 28 could contain some CO, it is believed to be molecular nitrogen, as it has assumed the characteristic color of N^+ at 8 keV. It can also be seen in Fig. 12 that the ion beam is not remaining normal to the magnetic field but is tilted down. This is a characteristic of weak arcs that are not electrically symmetric.[12]

An additional observation, which seems worthy of mention, was made while dipping a nitrogen-marked aluminum plate in warm water to determine if the marked areas would dissolve (AlN is soluble in H_2O). Figure 13, a photograph of the plate taken after the lower half had been immersed in water for 30 minutes, shows that the marked areas were affected, possibly by the formation of an additional thin film of aluminum oxide. But the more interesting observation is the development of the O^{2+} line. This line had initially be invisible; it cannot be seen on the upper area of the plate not exposed to water.

We have not used electron diffraction or other methods for analyzing the structure and composition of the marked target surfaces; however, that the surface is visible altered means that this effect must be considered in connection with studies in basic physics in this energy region. Several of the studies, such as sputtering or secondary electron production by impinging ions, are sure to be affected. Thus far, no color production by ions grazing metal surfaces has been observed, and this effect therefore must be carefully considered in connection with studies related to the angle of incidence of impacting ions.

ACKNOWLEDGMENTS

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FOOTNOTES

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[*] Work performed under the auspices of the U. S. Atomic Energy Commission.

[†] This figure appears in color in UCRL-9845.

FIGURE CAPTIONS

Fig. 1 Ion sources and mass spectrometer equipment. Vacuum equipment (not shown) embodies 16-inch diffusion pump and baffles.

Fig. 2 Aluminum target plate marked by nitrogen ions. Extraction potential, 11.6 kV; magnetic field, 8 kG; total beam current, 13 ma; exposure, 10 min.

Fig. 3 Nitrogen ions on aluminum target.

A, C, D --- N^{3+} @ 22.2, 30, and 39 kV, respectively;

B, E, F --- N^{2+} @ 14.8, 20, and 26 kV, respectively;

G, H, I --- N^{+} @ 7.4, 10, and 13 kV, respectively.

The magnetic field is 7 kG; total beam, 10 to 14 ma; exposure, 12 min. for each voltage setting.

Fig. 4 Nitrogen and oxygen ions on copper. 12 kV; 7 kG; 20 ma; 1 hr.

Fig. 5 Oxygen, nitrogen, and helium ions on several metals. 10 kV; 5 kG; 25 ma; approx 5 min.

Fig. 6 Theoretical penetration depth vs ion energy and ion distribution for $A_1/A_2 > 1$.

$$t = 0.6 \frac{(Z_1^{2/3} + Z_2^{2/3})^{1/2}}{Z_1 \times Z_2} \times \frac{A_2(A_1 + A_2)}{A_1} \times E \quad \mu g/cm^2.$$

Fig. 7 Theoretical penetration depth vs ion energy and ion distribution for $A_1/A_2 < 1$.

$$(2\tau)^{1/2} = \frac{0.7}{\xi(1 - \cos \varphi)^{1/2}} \times \frac{(Z_1^{2/3} + Z_2^{2/3})^{1/2}}{Z_1 Z_2} \times \frac{A_2^{\,2} E}{A_1 + A_2} \quad \mu g/cm^2.$$

Fig. 8 Comparison of the penetration depth of energetic nitrogen ions in aluminum as indicated by theory (---) and by color of marking (——).

Fig. 9 Nitrogen ions on beryllium bar mounted on aluminum plate. 11.5 kV; 7.5 kG; 18 ma; 1 hr.

Fig. 10 Comparison of the penetration depth of energetic nitrogen ions in beryllium as indicated by theory and by color of marking.

Fig. 11 Helium ions on copper. Also visible: colored oxygen marks and sputter marks from nitrogen. 10 kV; 5 kG; 26 ma; 30 min.

Fig. 12 Ion-marked vacuum spectrograph. 16 kV; 8 kG; 30 μa; 30 min.

Fig. 13 Nitrogen-marked aluminum plate immersed halfway in warm water for 30 min.

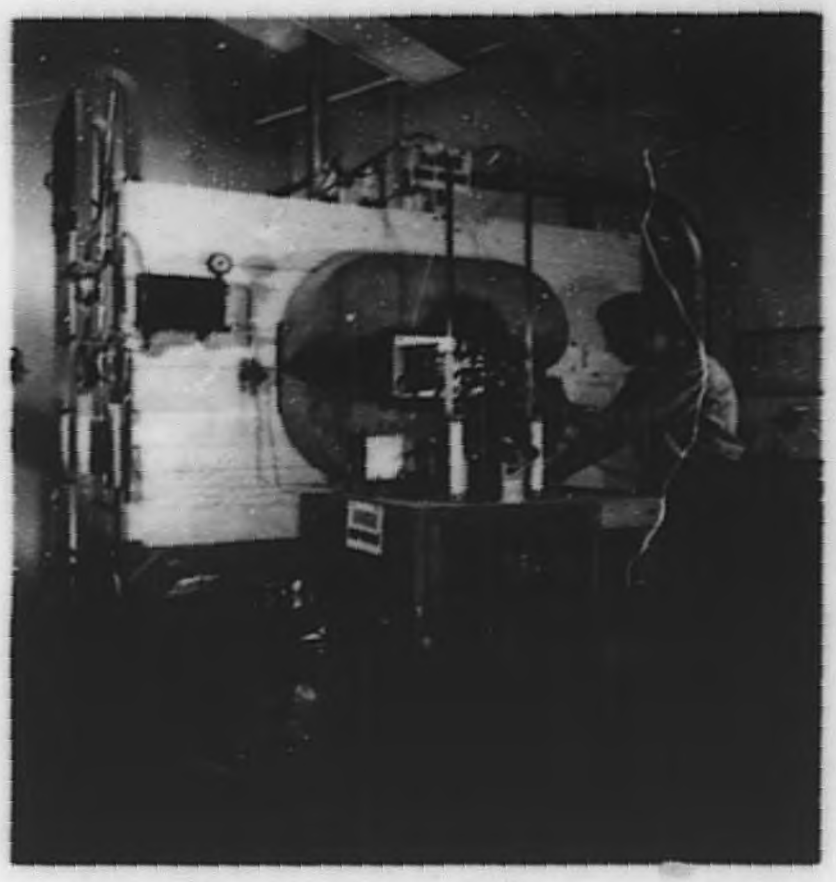

Fig. 1

Fig. 2

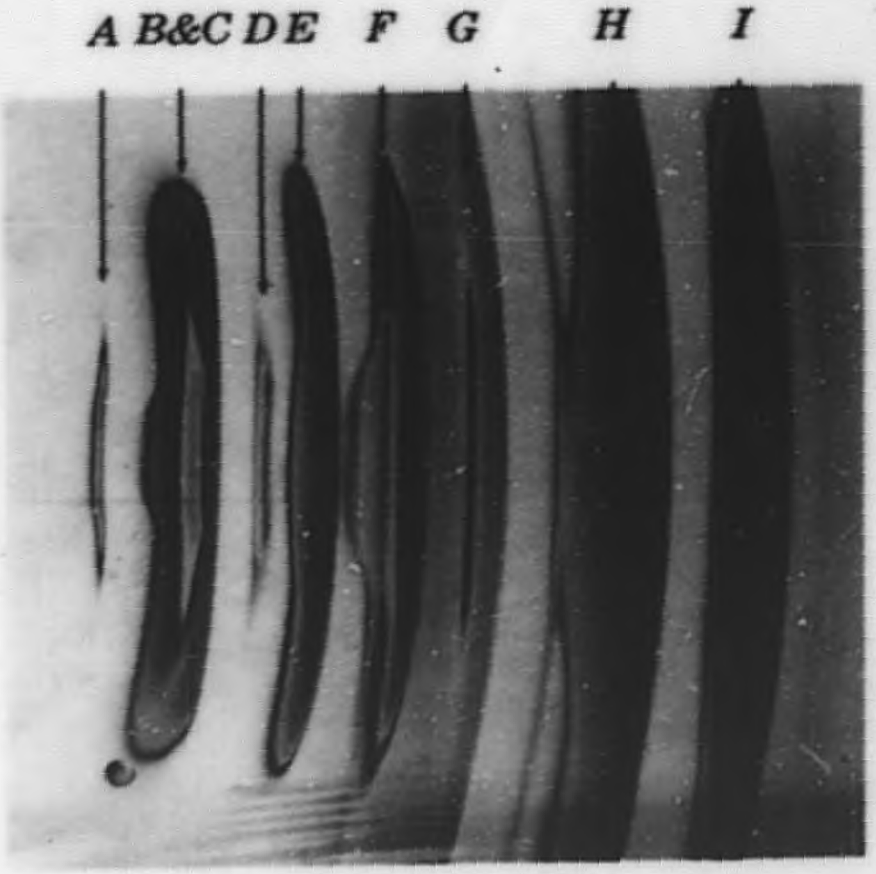

Fig. 3

Fig. 4

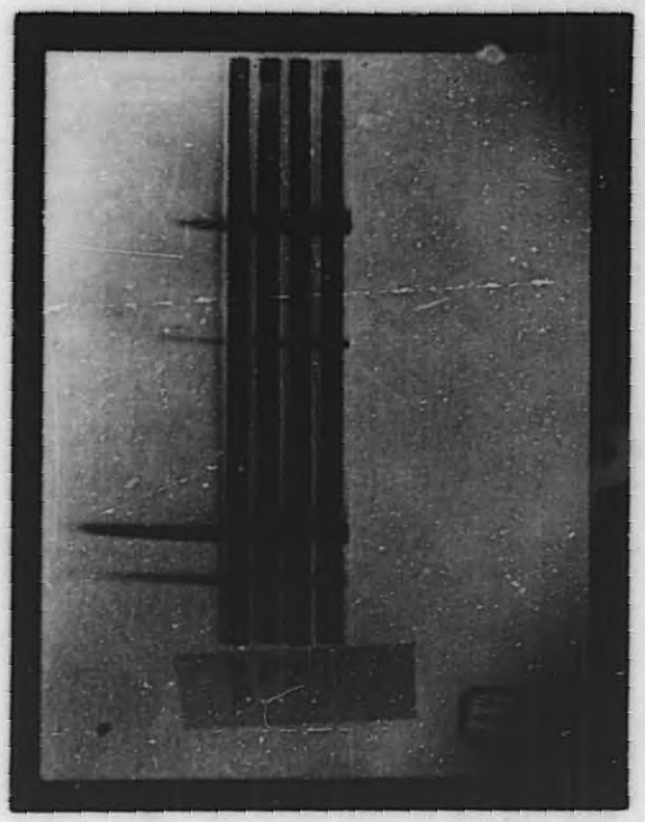

Fig. 5

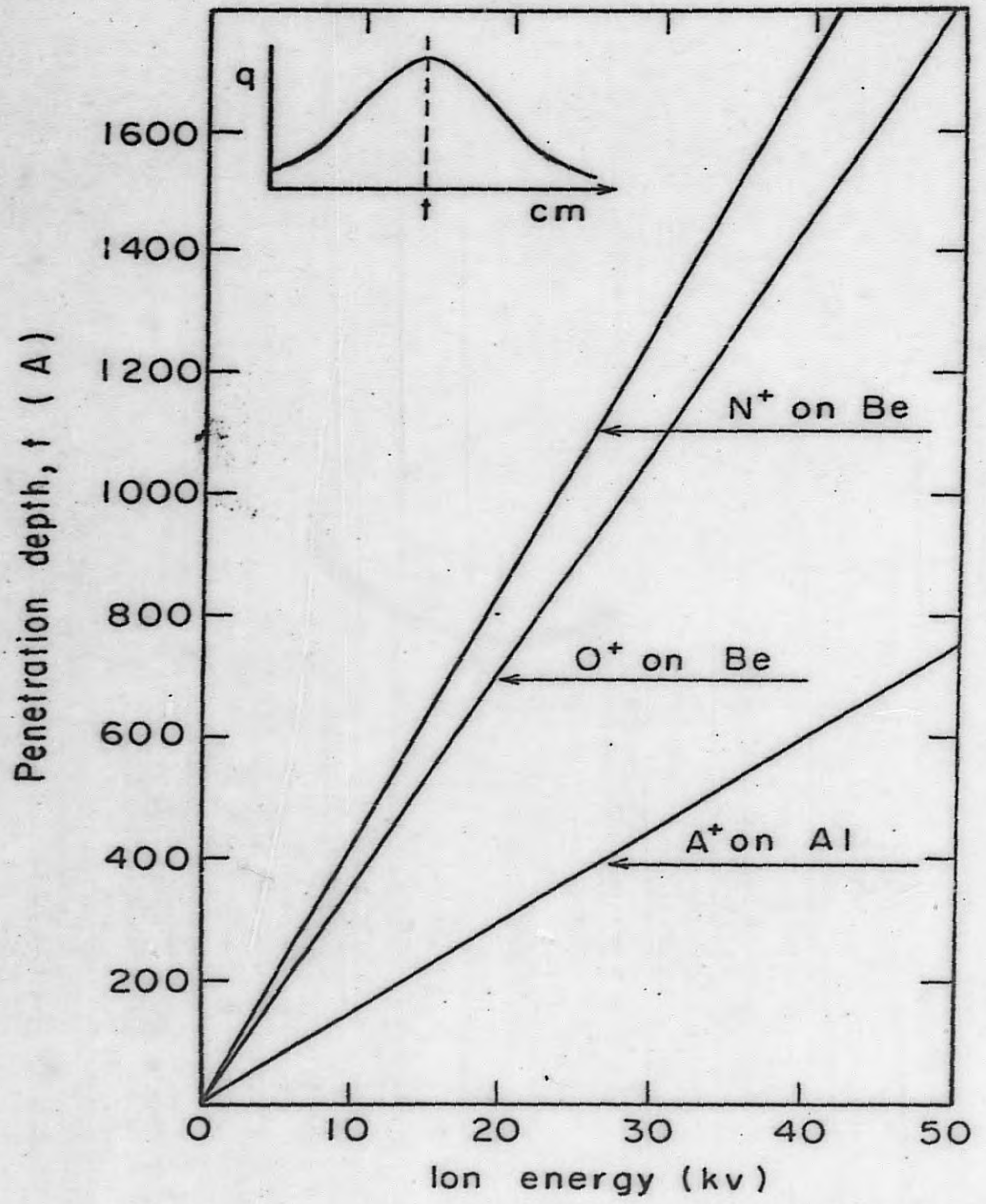

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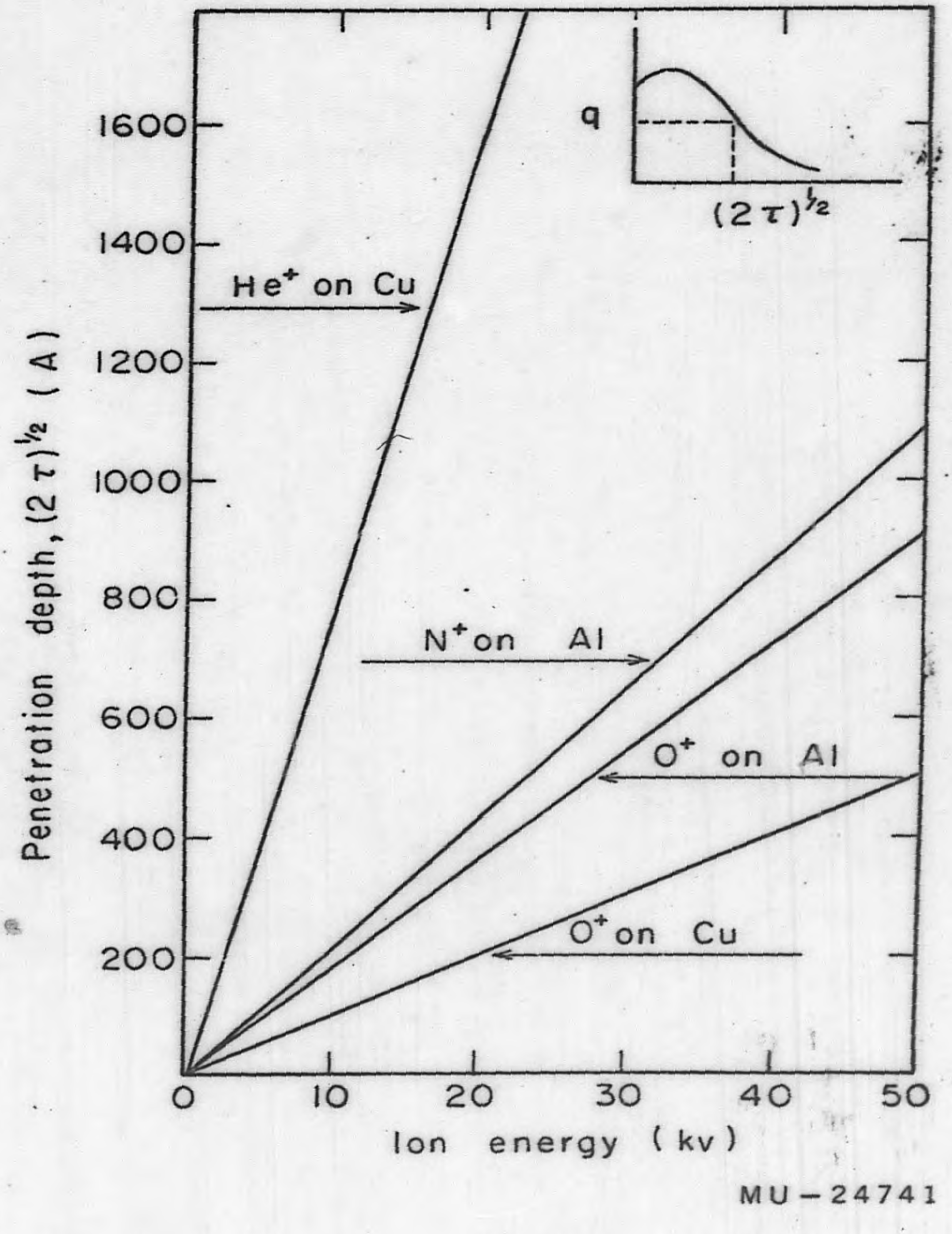
Fig. 6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$(2\tau)^{1/2}$
1600
1400
1200
1000
800
600
400
200
Penetration depth, $(2\tau)^{1/2}$ (A)
He^+ on Cu
N^+ on Al
O^+ on Al
O^+ on Cu
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10
20
30
40
50
Ion energy (kv)
MU-24741

Fig. 7

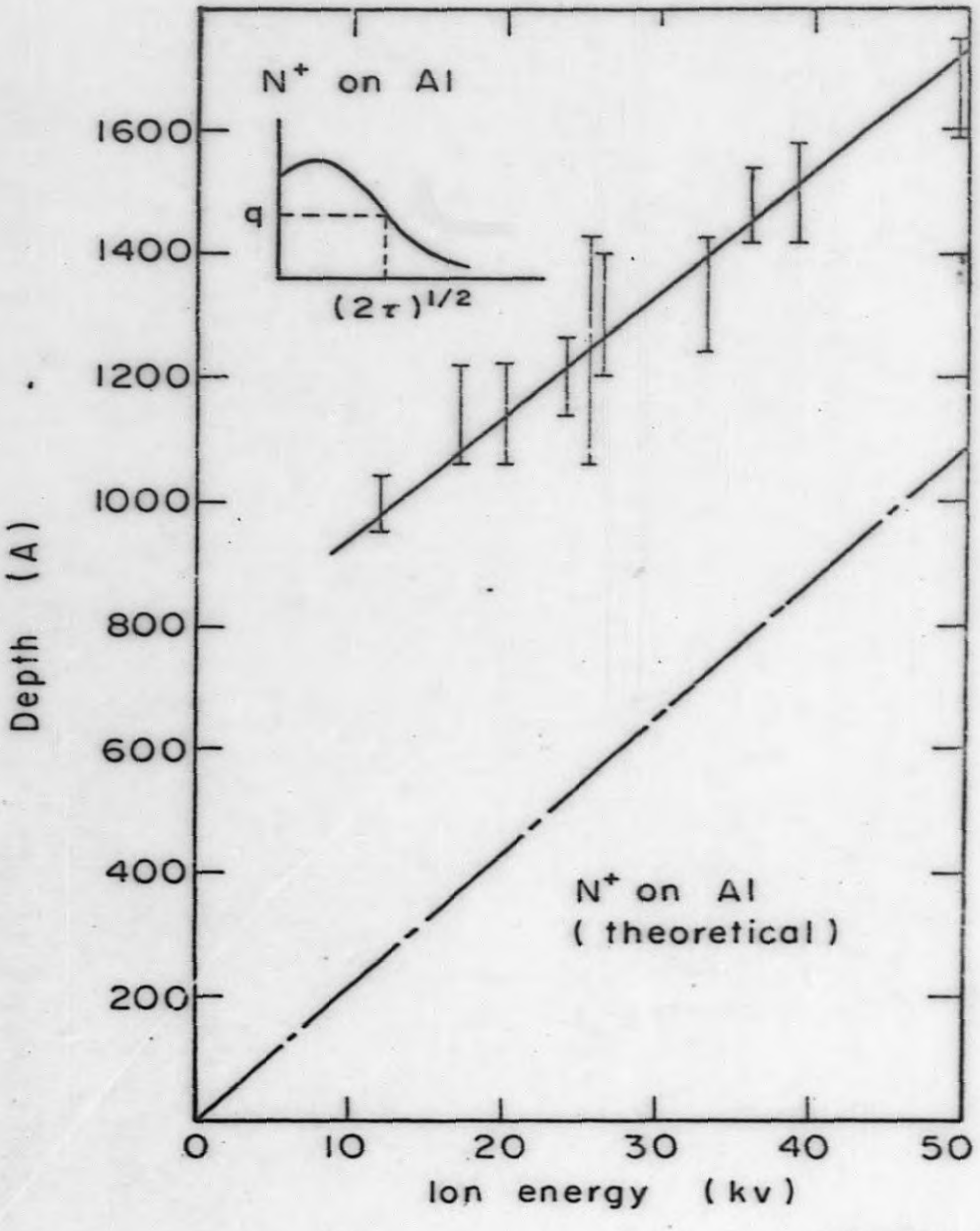

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Fig. 8

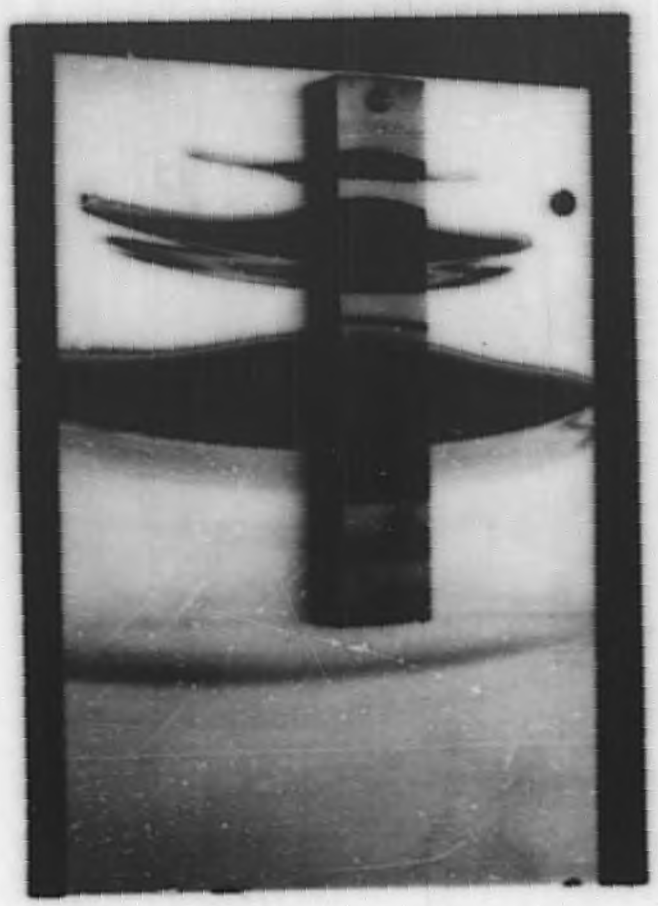
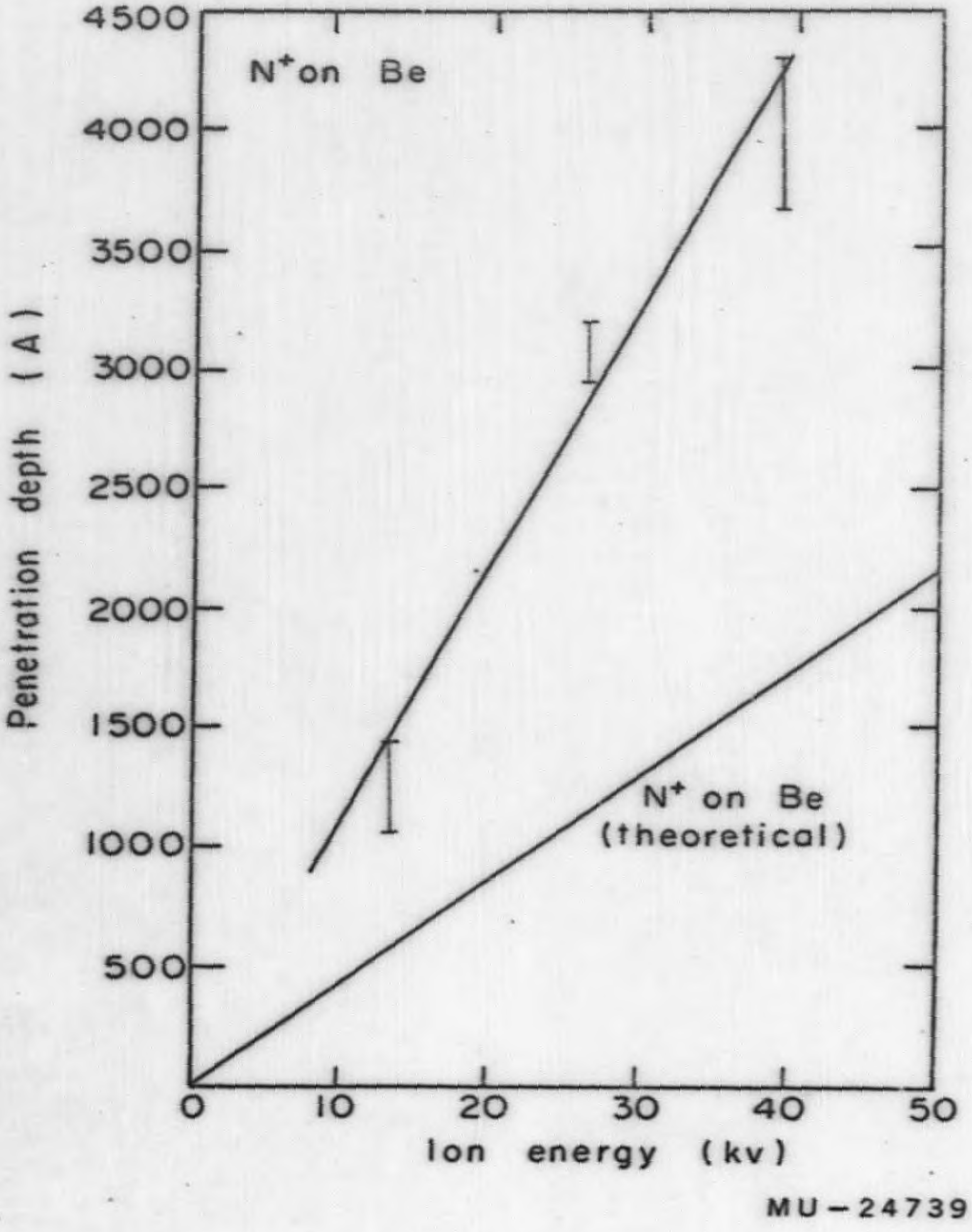


Fig. 9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Penetration depth (A)
Ion energy (kv)
MU-24739

Fig. 10

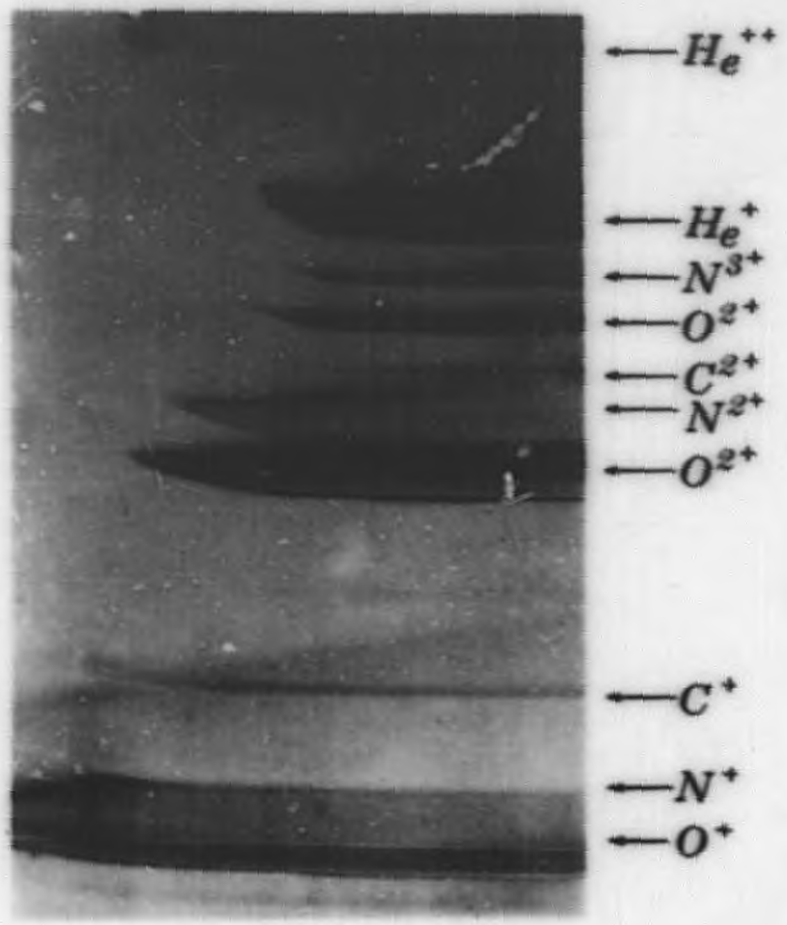

Fig. 11

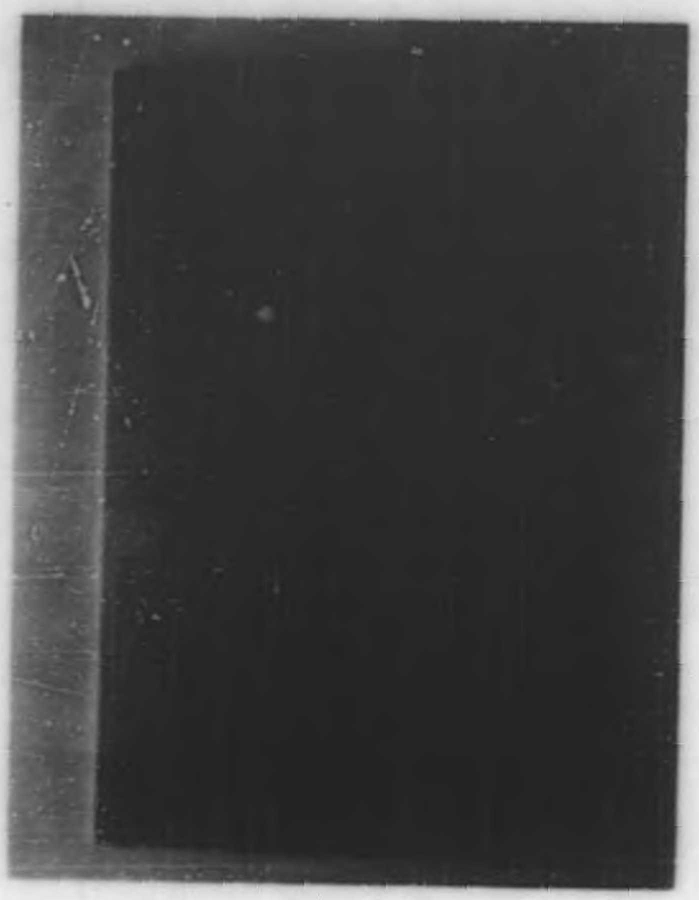

Fig. 12

Fig. 13