

## Measurements of energy loss and straggling for fast $H^+$ in metals and their compounds by means of a nuclear resonant reaction

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The narrow resonances of the  $^{27}\text{Al}(p,\gamma)^{28}\text{Si}$  reaction were utilized to determine the energy loss and straggling of fast  $H^+$  in metals and their compounds precisely. The energy-loss data obtained for  $H^+$  in single elements are in good agreement with the semiempirical data given by Andersen and Ziegler, whereas the experimental results of the energy straggling for high- $Z$ -number materials deviate from the theoretical predictions by Bohr and Vavilov. With regard to material dependence of the energy straggling, the results obtained are relatively consistent with the theoretical values given by Chu. Validity of the additivity rule for both the energy loss and straggling of fast  $H^+$  in  $\text{In}_2\text{O}_3$ ,  $\text{TiO}_2$ , and Al-Cu was verified within experimental error.

### I. INTRODUCTION

When a fast ion beam penetrates matter, its average energy decreases and its energy distribution is broadened via a number of successive collisions. The latter phenomenon is called energy straggling. The energy-loss and straggling data provide important information on beam-solid interactions.

In surface-layer analysis by fast ion beams, accurate data of energy loss and straggling for the projectiles passing through solid materials are needed to determine the depth scales and to estimate both mass and depth resolution. Up to the present, much data<sup>1-3</sup> of energy loss for light ions in single elements have been reported, but the data for compound materials are still insufficient. Therefore, to determine the depth scales for compounds, a linear combination of energy-loss cross sections for constituent elements (additivity rule) is frequently postulated. The energy straggling data are required to derive an accurate depth distribution of impurity atoms from the raw data measured by Rutherford backscattering or nuclear-reaction techniques. Several theories<sup>4-6</sup> concerning the straggling phenomenon have been developed, but there are very few precise experimental measurements<sup>7-12</sup> to verify the theories.

Several investigations<sup>13-15</sup> on the additivity rule for energy loss or fast H and  $^4\text{He}$  ions have been performed. For  $H^+$  at high velocity [ $v \gg v_0$  (Bohr velocity)], the validity of the rule was verified within a few percent,<sup>16</sup> but at energies lower than 1 MeV/amu significant deviations from this additivity

rule are expected. On the relationship to the energy straggling of compounds to that of the elemental components, no reliable data have been published.

To measure the energy loss and straggling, the backscattering method is often utilized. In this method, the energies of backscattered ions are measured with a Si surface-barrier detector, whose energy resolution is about 15 keV and the energy-loss and straggling values must be derived from the backscattering spectrum which includes the contributions from the incoming and the outgoing paths in the thin film. Recently, Möller and Nocken<sup>17</sup> measured the energy straggling of fast  $H^+$  in Ag and Au by using nuclear resonant reactions of  $^{19}\text{F}(p,\alpha\gamma)^{16}\text{O}$  at 340 keV,  $^{27}\text{Al}(p,\gamma)^{28}\text{Si}$  at 992 keV, and  $^{13}\text{C}(p,\gamma)^{14}\text{N}$  at 1747 keV. They used thin Ag and Au films evaporated onto thin carbon foils.

In the present work, we propose a simple nuclear resonance method with thick targets and measure the energy loss and straggling of fast  $H^+$  in Al, Ti, Cu, Se, In, Sb,  $\text{TiO}_2$ ,  $\text{In}_2\text{O}_3$ , and AlCu by using narrow resonances of the  $^{27}\text{Al}(p,\gamma)^{28}\text{Si}$  reaction. The stopping materials were deposited on Si wafers covered with a thin Al layer. If one employs this method, both the energy-loss and straggling values of  $H^+$  at the resonance energies can be directly obtained from the  $\gamma$ -ray yield curve as a function of incident  $H^+$  energies. The results obtained were compared with semiempirical data given by Andersen and Ziegler<sup>1</sup> and theoretical predictions by Bohr,<sup>4</sup> Vavilov,<sup>5</sup> and Chu.<sup>6</sup> Applicability of the additivity rule to both energy loss and straggling was also tested.

## II. EXPERIMENTAL METHOD

The principle way to determine the energy loss and straggling is illustrated in Fig. 1. The utilized resonance energies of  $^{27}\text{Al}(p,\gamma)^{28}\text{Si}$  are 632, 774, 936, and 992 keV and their natural widths ( $\Gamma$ ) are 100, 15, 340, and 100 eV, respectively. In order to determine the incident energy and energy spreading precisely, the  $\gamma$ -ray yield for a thin Al film evaporated onto a Cu plate was plotted as a function of the incident  $\text{H}^+$  energies around the resonance energy  $E_R$  (excitation yield curve). The energy distribution of the incident  $\text{H}^+$  beam is approximately Gaussian,

$$g(E_0, E) = \frac{2\sqrt{\ln 2}}{\sqrt{\pi}\Gamma_b} \exp\left[-\frac{4\ln 2(E - E_0)^2}{\Gamma_b^2}\right], \quad (1)$$

where  $E_0$  is the average incident energy corresponding to the midpoint of the excitation yield curve and  $\Gamma_b$  is the full width at half maximum (FWHM) of the distribution. The typical values of  $\Gamma_b$  were 600–800 eV. The total FWHM of the incident beam energy near the resonance  $\Gamma_0$  corresponds to the difference in energy between the 0.12 and 0.88 points of the step height for the excitation yield curve. The  $\Gamma_0$  is approximated by

$$\Gamma_0 \simeq (\Gamma^2 + \Gamma_b^2)^{1/2}. \quad (2)$$

At relatively long penetration depth  $x$ , the straggling distribution  $f(x, W)$  is given approximately by

$$f(x, W) = \frac{2\sqrt{\ln 2}}{\sqrt{\pi}\Gamma_s(x)} \exp\left[-\frac{4\ln 2}{\Gamma_s(x)^2} [W - \Delta E(x)]^2\right], \quad (3)$$

where  $W$  and  $\Delta E(x)$  and  $\Gamma_s(x)$  are the energy loss, the average energy loss, and the FWHM of the straggling distribution at depth  $x$ , respectively. If the thin film of interest deposited onto a thin Al substrate is used as a target, the energy loss and straggling after passing through this film can be easily derived from the shift of the half-height point and slope of the excitation yield curve by comparison with the curve obtained for the exposed Al film. The straggling width  $\Gamma_s$  is given by

$$\Gamma_s \simeq (\Gamma_t^2 - \Gamma_0^2)^{1/2}, \quad (4)$$

where  $\Gamma_t$  is the total FWHM of the energy distribution at the interface of the thin film of interest and the aluminum-substrate film. The  $\Gamma_t$  is determined by measuring the excitation curve in the same procedure as the one utilized to obtain  $\Gamma_0$ .

Figures 2(a) and 2(b) show the excitation yield curves around the 992- and 936-keV resonances for the Al film sputtered onto the Cu plate, the Cu film deposited onto the Al-covered Si wafer, and the  $\text{AlCu}_{0.747}$  film deposited onto the Si wafer. For Al and  $\text{AlCu}_{0.747}$  films, the FWHM's of the excitation yield curves give the energy-loss values and the energy straggling values can be derived from the energy difference between 0.12 and 0.88 points of the step height on the rear edge of the excitation curve. From the rise of the excitation curves for the Al-covered Cu plate, the initial energy spreading ( $\Gamma_b$ ) is calculated to be 680 and 870 eV at the resonances of 992 and 936 keV, respectively.

As stopping materials, Ti, Cu, Se, In, Sb,  $\text{TiO}_2$ , and  $\text{In}_2\text{O}_3$  films deposited onto the Al-covered Si wafers and Al-Cu deposited onto Si wafers were used. Further, Al-covered Cu plates were prepared also to determine the initial beam profiles. Deposition of Ti,  $\text{TiO}_2$ , and  $\text{In}_2\text{O}_3$  was performed by usual sputtering methods and other materials were evaporated onto the substrates in a high vacuum. The composition and thickness of the stopping films were measured by Rutherford backscattering of 2.0-MeV  $^4\text{He}$  ions. As composition of the compound materials,  $\text{TiO}_{1.90}$ ,  $\text{In}_2\text{O}_{2.93}$ , and  $\text{AlCu}_{0.747}$  were obtained. The thickness of the stopping films ranged from a minimum of  $4 \times 10^{17}$  to a maximum of  $2 \times 10^{18}$  atoms or  $\text{mol}/\text{cm}^2$ . The maximum error possible in this method was estimated to be about 3% from the reproducibility of the measurements.

The  $\text{H}^+$  beam from a 3-MV Van de Graaff accelerator passed through a  $60^\circ$  deflection magnet

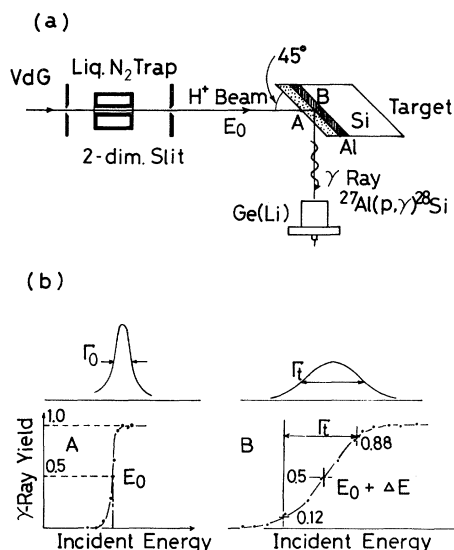


FIG. 1. (a) Experimental setup. (b) Schematic excitation yield curves for an Al-covered Cu plate (A) and for a stopping film deposited onto an Al-covered Si wafer (B).

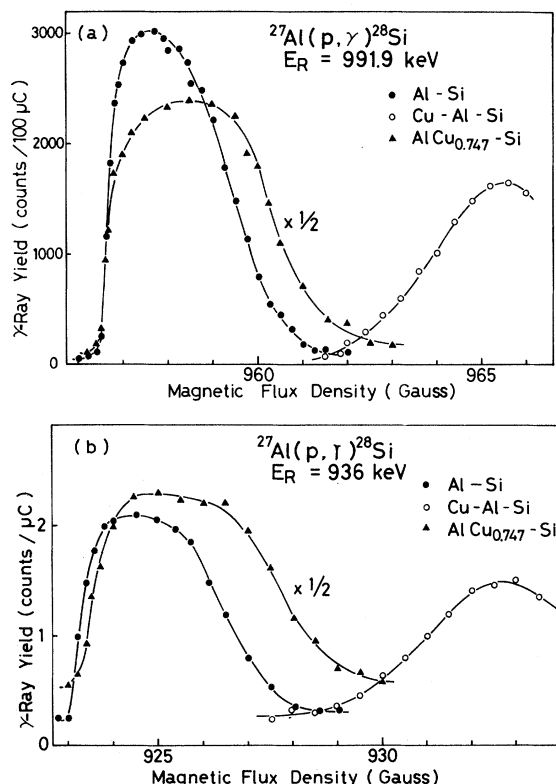


FIG. 2. (a) Excitation yield curves around the 991.9-keV resonance for an Al film sputtered onto a Cu plate, a Cu film deposited onto an Al-covered Si wafer, and an  $\text{AlCu}_{0.747}$  film deposited onto a Si wafer. (b) Excitation yield curves around the 936-keV resonance.

with a radius of 1.50 m. Energy calibration of the magnet was performed through observation of the narrow resonances of  $^{27}\text{Al}(p,\gamma)^{28}\text{Si}$  at 991.9 keV (Ref. 18) and  $^{31}\text{P}(p,\gamma)^{32}\text{S}$  at 811.3 keV.<sup>19</sup> The magnetic flux density was measured with proton nuclear magnetic resonance. The energy stabilization obtained was better than  $\pm 100$  eV.

The targets were mounted on a water-cooled target holder which allowed a vertical translation of 40 mm and a  $360^\circ$  rotation in the horizontal plane. The targets were tilted  $45^\circ$  with respect to the incident beam axis. The emitted  $\gamma$  rays corresponding to the transition from the first excited state to the ground state of  $^{28}\text{Si}$  (1.778 MeV) were detected with a  $115\text{-cm}^3$  Ge (Li) detector placed 2.5 cm from the irradiation area at an angle of  $90^\circ$  with the incident beam axis. The target chamber which was isolated electrically was evacuated with a 750 l/sec cryopump and a normal operating pressure of less than  $5 \times 10^{-7}$  Torr was maintained. In order to avoid surface contamination, especially carbon buildup, two coaxial cylindrical traps at liquid- $\text{N}_2$  temperatures were installed in front of the target chamber.

### III. RESULTS AND DISCUSSION

#### A. Energy loss

Figure 3 shows the material dependence of the stopping powers for 1.0 MeV  $\text{H}^+$ . The data obtained agree well with the semiempirical data compiled by Andersen and Ziegler.<sup>1</sup> The stopping powers of In,  $\text{In}_2\text{O}_{2.93}$ , Ti, and  $\text{TiO}_{1.90}$  for 632, 774, and 992 keV  $\text{H}^+$  are shown in Tables I and II. In Table III, the stopping powers of Al, Cu, and  $\text{AlCu}_{0.747}$  for 632, 774, 936, and 992 keV  $\text{H}^+$  are given. The semiempirical values for O, Al, Ti, Cu, and In are also included in these tables. The results obtained for Ti, In, Al, and Cu are in good agreement with the semiempirical values. Except for a few data points, the discrepancies between our data and the semiempirical ones are less than about 6%, which corresponds to the value expected from the experimental uncertainties in the film thickness and geometrical factors.

The additivity rule for stopping cross sections (Bragg's rule) is given as follows:

$$S_{A_m B_n}(E) = mS_A(E) + nS_B(E), \quad (5)$$

where  $S_A(E)$  and  $S_B(E)$  are the atomic stopping cross sections in elements  $A$  and  $B$ , respectively, and  $S_{A_m B_n}(E)$  is the molecular stopping cross section in a compound with a chemical formula  $A_m B_n$ . The stopping powers of compound materials agree well with those derived from the additivity rule within about 5%, except for  $\text{In}_2\text{O}_{2.93}$  at 632 keV (Tables I–III). In applying the additivity rule for  $\text{In}_2\text{O}_{2.93}$  and  $\text{TiO}_{1.90}$ , we used the stopping-power values for oxygen given by Andersen and Ziegler.

#### B. Energy straggling

The energy straggling of fast ions in matter is caused by two factors; one is the electronic collision process and another is the nuclear collision process.

TABLE I. Stopping powers of O, In, and  $\text{In}_2\text{O}_{2.93}$  for 632, 774, and 992 keV  $\text{H}^+$  [ $\text{eV}/(10^{15}$  atoms or  $\text{mol}/\text{cm}^2$ )]. The data for O and In given by Andersen and Ziegler (Ref. 1) are also included.

Energy (keV)	632	774	992
In	19.65	18.72	17.80
$\text{In}_2\text{O}_{2.93}$	71.58	58.57	55.12
In (Andersen and Ziegler)	21.0	19.5	17.2
O			
(Andersen and Ziegler)	7.3	6.8	6.0
Additivity rule	60.7	57.4	53.2

TABLE II. Stopping powers of O, Ti, and TiO<sub>1.90</sub> for 632, 774, and 992 keV H<sup>+</sup> [eV/(10<sup>15</sup> atoms or mol/cm<sup>2</sup>)]. The data for O and Ti given by Andersen and Ziegler (Ref. 1) are also included.

Energy (keV)	632	774	992
Ti	13.95	12.70	10.39
TiO <sub>1.90</sub>	26.63	24.10	22.52
Ti (Andersen and Ziegler)	14.4	13.0	11.3
O (Andersen and Ziegler)	7.3	6.8	6.0
Additivity rule	27.8	25.5	21.8

At high velocity ( $v > v_0$ ), the electronic factor dominates the collision processes. The first theoretical work on the energy straggling was carried out by Bohr,<sup>4</sup> who described the process by successive binary collisions between the projectile ion and the target electrons and got the following expression:

$$\Gamma_B^2 = (8 \ln 2) 4\pi Z_1^2 Z_2 e^4 N \Delta t, \quad (6)$$

where  $Z_1$  and  $Z_2$  are the atomic numbers of the projectile and the target,  $N$  is the atomic density,  $e$  is the electronic charge, and  $\Delta t$  is the thickness of the target. In Bohr's theory, the target electrons were regarded as equivalent free electrons and it was assumed that the number of the collisions have a Poisson distribution. Equation (6) is hence valid only at high velocities and for relatively thick targets. For short path lengths, Vavilov<sup>5</sup> derived a straggling distribution, which is asymmetric with a pronounced tail for large energy losses. The Bohr theory has been refined by Chu,<sup>6</sup> who has calculated the straggling widths by using atomic charge-density distributions based on the Hartree-Fock-Slater model and Bonderup and Hvelplund straggling theory.<sup>20</sup> In the Chu theory, a remarkable energy dependence and a

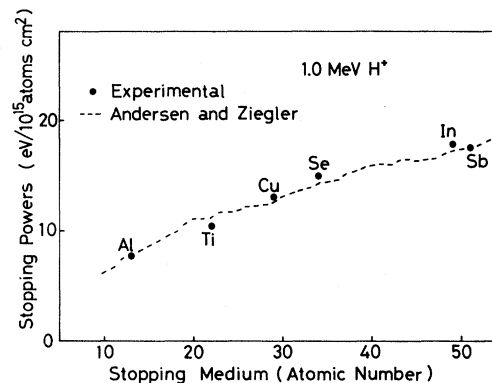


FIG. 3. Material dependence of stopping powers for 1.0 MeV H<sup>+</sup>. The dotted line denotes the data compiled by Andersen and Ziegler.

Z<sub>2</sub>-oscillatory structure at low energy is predicted. In fact, Chu's results agree quite well with experimental data<sup>21</sup> for Al targets at ion energies less than 0.5 MeV/amu, while for gaseous targets his theory predicts a stronger reduction of the straggling width than found experimentally<sup>20,22</sup> for the same energy range. Besenbacher *et al.*<sup>21</sup> explained the deviations from Chu's estimates by additional straggling contributions from spatial atomic correlation effects and charge-state fluctuations. Furthermore, Besenbacher *et al.* and Sigmund<sup>23</sup> showed that for solid targets the spatial correlation effects are much smaller than for gaseous targets. Recently, Friedland and co-workers<sup>7,8</sup> (Pretoria group) have been investigating the energy straggling phenomenon for H<sup>+</sup>, D<sup>+</sup>, and  $\alpha$  particles in various solid materials using a transmission technique. Their results agree well with Bohr's estimates at energies higher than 0.5 MeV/amu but deviate remarkably from Chu's prediction for relatively high-Z<sub>2</sub> materials at energies less than 1 MeV.

In the present study, the straggling widths for fast H<sup>+</sup> in various metals and their compounds were

TABLE III. Stopping powers of Al, Cu, and AlCu<sub>0.747</sub> for 632, 774, 936, and 992 keV H<sup>+</sup> [eV/(10<sup>15</sup> atoms or mol/cm<sup>2</sup>)]. The data for Al and Cu given by Andersen and Ziegler (Ref. 1) are also included.

Energy (keV)	632	774	936	992
Al	11.45±0.34	8.70±0.37	8.75±0.14	7.75±0.14
Al (Andersen and Ziegler)	10.3	9.2	8.2	7.8
Cu	17.18±0.15	15.93±0.25	13.35±0.18	13.04±0.09
Cu (Andersen and Ziegler)	16.1	14.7	13.2	12.6
AlCu <sub>0.747</sub>	25.22±0.41	21.52±0.67	20.28±0.35	18.71±0.22
Additivity rule	24.27	20.59	18.74	17.54

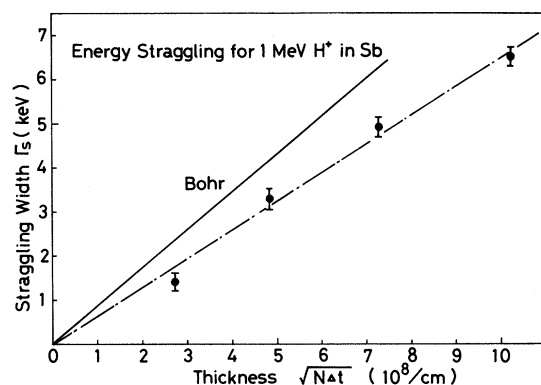


FIG. 4. Stragglng widths of 1.0 MeV  $H^+$  in Sb as a function of film thickness compared with the theoretical prediction by Bohr. The dot-dashed line is drawn by the least-squares method.

determined assuming Gaussian distributions, because for relatively thick targets  $\geq 5 \times 10^{17}$  atoms/cm<sup>2</sup>, deviation from a Gaussian is expected to be small. Figure 4 shows the stragglng widths for 1.0 MeV  $H^+$  in Sb as a function of the film thickness. The stragglng widths obtained are proportional to the square root of the film thickness, but the values are considerably lower than those of Bohr. In Fig. 5, stragglng widths divided by the square root of the target thickness (normalized stragglng width) for Al, Ti, Cu, In, and Sb are plotted as a function of  $H^+$  energies. The present results show a small energy dependence of the normalized stragglng widths except for the data point at 632 keV for In. The data for Cu films are compared with those by Friedland and Kotze<sup>8</sup> and theoretical values by Bohr, Vavilov, and Chu. Actually, the experimental results as well as the theoretical ones

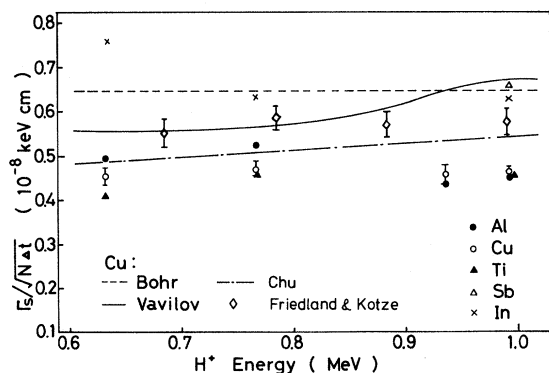


FIG. 5. Normalized stragglng widths vs  $H^+$  energy. For Cu targets the experimental data by Friedland and Kotze are given as open diamonds ( $\diamond$ ) and theoretical predictions are shown by the solid (Vavilov), dashed (Bohr), and dot-dashed (Chu) curves.

show only a weak energy dependence or no energy dependence at all. Our data are relatively consistent with Chu's estimates for the low-energy range, whereas discrepancies between our results and results based on the free-electron model are larger than 30%. This is probably due to the fact that both the Bohr and Vavilov theories overestimate the contribution from the core electrons. Figure 6 shows the square of the normalized stragglng widths for 1.0 MeV  $H^+$  as a function of the atomic number of the target materials. The stragglng widths increase with an increase in atomic number. Our results agree relatively well with the calculated curve by Chu and are consistent with the data for Ag given by Möller and Nocken.<sup>17</sup> Deviations from Bohr's prediction are large for high- $Z_2$  materials, but for Al a good agreement between the observed and calculated data is obtained. Discrepancies between our data and those by the Pretoria group are large for relatively high- $Z_2$  materials. As mentioned above, the spatial correlation effect is expected to be small for solid targets. Furthermore, contributions from charge-state fluctuations are considered to be negligibly small because the charge state  $H^+$  comprises more than 99.5% of the total beam at  $H^+$  energies greater than 0.5 MeV.<sup>24</sup> Further, according to the Brandt-Sizmann theory,<sup>25,26</sup> the electron cannot be bound inside the solids due to collision broadening and collective screening by the valence electrons. From the above discussions it is reasonable that our results are relatively consistent with Chu's estimates based on the electron gas model taking account of the shell structure.

Finally, we tried to check the applicability of the additivity rule for the energy stragglng for  $In_2O_{2,93}$ ,

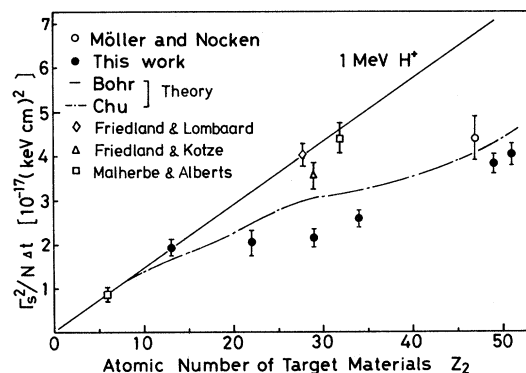


FIG. 6. Square of the normalized stragglng widths for 1.0 MeV  $H^+$  vs target atomic number  $Z_2$ . The present results and other experimental data by Möller and Nocken (<sup>47</sup>Ag), Friedland and Lombaard (<sup>28</sup>Ni), Friedland and Kotze (<sup>29</sup>Cu), and Malherbe and Alberts (<sup>6</sup>C, <sup>32</sup>Ge) are compared with the theoretical predictions by Bohr (solid curve) and Chu (dot-dashed curve).

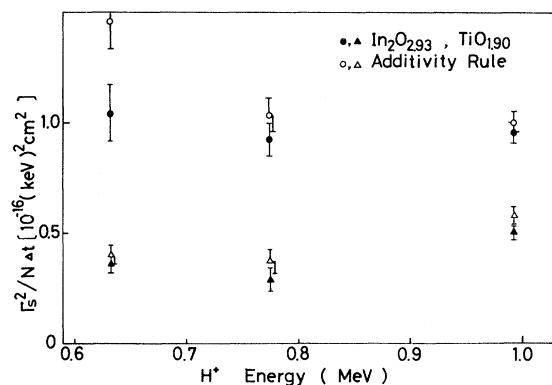


FIG. 7. Square of the normalized straggling widths for  $\text{In}_2\text{O}_{2.93}$  (solid circles) and  $\text{TiO}_{1.90}$  (solid triangles) vs  $H^+$  energy. Open symbols denote the values derived from the additivity rule.

$\text{TiO}_{1.90}$ , and  $\text{AlCu}_{0.747}$  films. By extending Bohr's model to compound targets, the additivity rule for the energy straggling is given as follows:

$$\frac{\Gamma_s(A_m B_n)^2}{N(A_m B_n)\Delta t(A_m B_n)} = \frac{m\Gamma_s(A)^2}{N(A)\Delta t(A)} + \frac{n\Gamma_s(B)^2}{N(B)\Delta t(B)}. \quad (7)$$

The notation is the same as those used in Eqs. (5) and (6). Figure 7 indicates the square of the normalized straggling widths of 632, 774, and 992 keV  $H^+$  for  $\text{In}_2\text{O}_{2.93}$  and  $\text{TiO}_{1.90}$  films. In applying the additivity rule, theoretical values for oxygen given by Vavilov were used. Figure 8 indicates the square of the normalized straggling widths of 632, 774, 936, and 992 keV  $H^+$  for  $\text{AlCu}_{0.747}$  films. The present data for the compounds of  $\text{In}_2\text{O}_{2.93}$ ,  $\text{TiO}_{1.90}$ , and  $\text{AlCu}_{0.747}$  agree with those derived from the additivity rule within experimental error, except for the data point for  $\text{In}_2\text{O}_{2.93}$  at 632 keV. This deviation could probably be ascribed to nonuniformity of our In foil on a microscopic scale. In fact, Besenbacher *et al.*<sup>21</sup> and Malherbe and Alberts<sup>9</sup> pointed out that foil inhomogeneity and texture effects would lead to an additional straggling contribution. In our experiment, homogeneity in the effective foil thickness was observed to be better than 5% by Rutherford backscattering but the absence of foil nonuniformity on a microscopic scale such as island and hillock formation could not be confirmed.

#### IV. CONCLUSIONS

The energy loss and straggling of fast  $H^+$  in various metals and their compounds were measured by a simple nuclear resonance method with thick targets. The stopping-power values obtained are in good

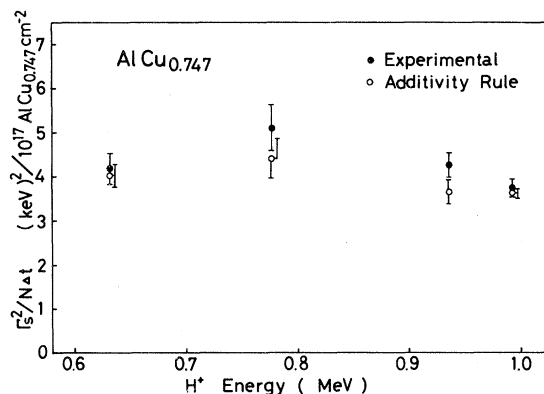


FIG. 8. Square of the normalized straggling widths for  $\text{AlCu}_{0.747}$  (solid circles) vs  $H^+$  energy. Open circles denote the values derived from the additivity rule.

agreement with the semiempirical data given by Andersen and Ziegler. The experimental error for the stopping-power measurement is estimated to be less than about 5%, mainly due to the inaccuracies in film thickness and geometrical factors. Concerning the material dependence of the energy straggling, relatively good agreement between our results and Chu's calculation based on Hartree-Fock-Slater electron densities was obtained, whereas especially for high- $Z_2$  materials, the values obtained were considerably lower than those predicted by Bohr and Vavilov theories based on a free-electron model. The above results indicate that the free-electron model overestimates the contribution from the core electrons of the target. Furthermore, the straggling widths increased with an increase in atomic number of the target material and a relatively small energy dependence of the normalized straggling widths was observed. The straggling widths for 1.0 MeV  $H^+$  in Sb were found to be proportional to the square root of the film thickness. This result is consistent with Bohr's expression. The additivity rule for both the energy loss and straggling was tested for fast  $H^+$  in  $\text{In}_2\text{O}_{2.93}$ ,  $\text{TiO}_{1.90}$ , and  $\text{AlCu}_{0.747}$ , and was verified within experimental error except for one data point. In order to confirm the above conclusions, data covering a wider range of  $H^+$  energies for many kinds of material foils with homogeneity and uniformity on a microscopic scale are required.

#### ACKNOWLEDGMENTS

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