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Stopping power of palladium for protons in the energy range 0.300–3.100 MeV

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ABSTRACT

The stopping power of palladium for protons has been measured using the transmission method with an overall uncertainty of around 5% over the energy range $E_p = (0.300\text{–}3.100)$ MeV. These stopping power data are then compared to stopping power values calculated by the SRIM-2010 code and to those derived from a model based on the dielectric formalism. Subsequently, and within the framework of the modified Bethe–Bloch theory, this stopping power data were used for extracting Pd target mean excitation and ionization potential, ($I = 468 \pm 5$ eV), and Barkas effect parameter, ($b = 1.51 \pm 0.06$). A good agreement is found between the obtained results and values reported in literature.

It is worth mentioning that these are the first reported results for protons on palladium over this energy range, which is often used in IBA applications, such as Rutherford Backscattering Spectrometry (RBS) and Proton Induced X-ray Emission (PIXE).

1. Introduction

It is well known that the stopping power of charged particles in matter S (E) not only is an important quantity in atomic and nuclear physics, but also has important applications in other fields of Science and Technology [1,2]. Moreover, in recent times there has been a renewed interest in increasing the accuracy of these values, particularly for light ions, in order to better determine some key parameters included in the theory of stopping power [3–5]. However, surveying the available literature it turns out that experimental data of stopping power for certain materials are absent. For instance, in the case of palladium the available data for protons is rather scarce [6–11], though this material is no less crucial in a number of important applications. Nowadays, palladium is used as the active ingredient in catalytic converters and it is also found in many electronic devices including multi-layer ceramic capacitors and low voltage electrical contacts. It is in this respect, that within a long-term research project, we propose to measure the stopping power curves for different materials of technological interest over the energy range between 0.3 and 3.1 MeV, which is often used in Ion Beam Analysis.

2. Experiment

The stopping power measurements were performed at the 3.75 MV Van de Graaff accelerator of the Faculty of Sciences of University of Chile [12]. Its absolute energy calibration was carried out over a wide energy range using the 872, 974 and 1370 keV resonances of the $^{19}F(p,(\alpha\gamma))^{16}O$ reaction, the 1735 keV resonance of the ^{12}C (p,p)^{12}C reaction and the 2085 and 3103 keV resonances of ^{28}Si (p,p)^{28}Si reaction.

In order to achieve a high accuracy in the stopping power data a stainless steel 600 series ORTEC scattering chamber was used during the acquisition, which allows a precise positioning of the detector ($150.0^\circ \pm 0.1^\circ$) in relation to the beam direction. This facility also features two collimators (antiscattering) which define a beam spot that can varied from about 3 to 0.5 mm in diameter. A goniometer is used to position and orient the samples with respect to the beam (±0.5 mm) and to select the tilt angle (±0.5°). The data acquisition system includes an ORTEC surface-barrier detector Model BA-014-50-100 with 14 keV nominal resolution FWHM for a ^{241}Am alpha source (5.486 MeV). Pulses were analyzed with proper electronic circuitry (preamplifier ORTEC Model 142E, an ORTEC amplifier Model 572) and collected by an ORTEC PC MCA Model Trump-8K. The acquisition system was periodically calibrated in energy by using ^4He backscattering spectra of a thin Al/Ti/Ta multilayer deposited on a carbon substrate with a well-known concentration. High-vacuum conditions 10^{-6} torr were achieved during the measurements by using two turbo-molecular pumps. The beam current on target was kept relatively constant at around 5.0 nA in order not only to attain sufficient statistics in each RBS spectrum, but also to prevent any further damage of the target.

The stopping medium was a single palladium foil with nominal thickness of 0.5 μm supplied by Goodfellow [13]. However, the

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precise areal density was determined by the transmission method using a calibrated alpha source which is composed of three radionuclides ^{239}Pu (5.156 MeV), ^{241}Am (5.486 MeV) and ^{244}Cm (5.806 MeV). Assuming that the stopping power for 5.486 MeV alphas on palladium is 55.69 eV/10^{15} at/cm^2 [7] then an areal density of (2.64 ± 0.13)×10^{18} at/cm^2 was obtained which correspond to a thickness of 0.38 ± 0.02 μm. According to Eckardt [8] it is possible that the content of hydrogen in palladium may affect the stopping power measurements up to 10% if the target is considered as Pd_4H_3. In order to address this point, the areal density of a similar 2.5 × 2.5 cm^2 palladium foil from the same supplier was measured by weighing, which gave M = 3.150 ± 0.001 mg (Microbalance Sartorius M5P-000V001). Subsequently, the foil was placed in an oven at 200 °C for 24 h and weighed again. However, no significant difference was observed between both areal density values. Furthermore, to check for repeatability in our stopping power values the measurement at 1.118 MeV was repeated within a time span of one month (see Table 1) but no noticeable difference was observed.

2.1. Transmission method and energy loss measurement

When a material is irradiated with charged particles of a certain energy, such as protons, alpha particles or heavy ions, the incident particle progressively loses its energy in large part due to inelastic collisions with atomic electrons [14]. Theoretically, the energy loss dE due to these interactions is commonly described by the well known Bethe–Bloch formula of the stopping power S (E). which will be examined in further detail in the next section of this article. From the experimental point of view, the energy loss of an incident charged particle with energy E along its trajectory in any material can be determined in terms of the stopping cross section, which is defined by:

$$\varepsilon(E) = \frac{S(E)}{N} = -\frac{dE}{Ndx} \approx -\frac{\Delta E}{N\Delta x}, \quad (1)$$

with N denoting the atomic number density (atoms cm^{-3}) of the material under study. In order to determine the stopping cross section of palladium for protons, the transmission method [15–17] was used by measuring the energy loss ΔE of protons passing through a thin palladium foil of thickness Δx. As shown in Fig. 1, the proton beam, with initial energy E_0, impinges on a scattering center which consists of a 1000 Å thickness gold film deposited on a mica substrate. Given a fixed scattering angle θ = 150°, the backscattered protons will have an energy E_1 which can be measured by a particle detector. Then in a separate measurement, the palladium foil is interposed between the scattering center and the detector which now will show a lower energy E_2. Both energy distributions were fitted by gaussian functions to obtain the mean energy and width (FWHM) of the peaks [18]. From the difference between the peak positions in the spectrum, the total energy loss ΔE in the foil was calculated. However, to properly determine the stopping cross section value, it must be evaluated at an effective average energy [19,20] given by:

$$E_{avg} = E_1 - \frac{\Delta E_{Pd}}{2}, \quad (2)$$

where ΔE_{Pd} is the energy loss in the palladium foil and E_1 is the backscattered energy of protons which should take into account not only the kinematic factor K but also the energy loss in the gold scattering center ΔE_{Au}. Therefore, the last expression can be rewritten as:

$$E_{avg} = \left(KE_0 - \frac{\Delta E_{Au}}{2}\right) - \frac{\Delta E_{Pd}}{2}. \quad (3)$$

Table 1
Stopping power values S_{exp} of palladium for protons measured in this work. Reduced stopping power X_{exp} values corresponding to I = 468 eV and b = 1.51 are also shown.

E_{avg}	S_{exp}	ΔE/E	X_{exp}
keV	eV/(10^{15} at/cm^2)	%	
304.4	29.8 ± 1.5	24.8	5.67 ± 0.04
364.5	27.4 ± 1.4	19.1	5.77 ± 0.05
423.9	25.6 ± 1.3	15.4	5.84 ± 0.05
540.5	22.5 ± 1.1	10.7	5.97 ± 0.06
658.3	20.1 ± 1.0	7.9	6.07 ± 0.06
774.8	18.6 ± 0.9	6.2	6.12 ± 0.07
889.0	17.1 ± 0.9	5.0	6.19 ± 0.07
1005.4	16.4 ± 0.8	4.2	6.19 ± 0.07
1117.8	15.4 ± 0.8	3.6	6.23 ± 0.08
1118.8	15.5 ± 0.8	3.6	6.22 ± 0.08
1406.3	12.9 ± 0.6	2.4	6.38 ± 0.08
1574.3	12.4 ± 0.6	2.1	6.37 ± 0.09
1971.4	11.5 ± 0.6	1.5	6.31 ± 0.10
2146.4	10.7 ± 0.5	1.3	6.37 ± 0.10
2255.4	10.4 ± 0.5	1.2	6.37 ± 0.11
2538.8	9.8 ± 0.5	1.0	6.36 ± 0.11
2827.2	8.9 ± 0.4	0.8	6.44 ± 0.11
3111.9	7. ± 0.4	0.7	6.76 ± 0.10

3. Analysis of the results and discussion

As can be seen in Table 1, an overall relative uncertainty of around 5% was achieved for the stopping power values, which is mainly due to the uncertainty in the palladium foil thickness. A good agreement in the whole energy range is found between our measurements and those derived from Moreno-Marín theoretical approach [21,22], as can be observed in Fig. 2. On the other hand, values calculated by the SRIM-2010 computer code [7] agree well only for higher energy protons, while at lower energies calculated values are systematically above our measurements and in some cases beyond the error bars. This is probably due to a larger energy loss ($\Delta E/E_{avg}$) in the foil for our lowest energy points, which is clearly seen in Table 1. Though there are no other experimental data in the energy range covered in this work, those from Eckardt et al. [8] show a tendency that agrees very well with our stopping power measurements at lower energies.

It should be pointed out that Eq. (3) is valid if the condition $\Delta E/E_{avg} \leqslant 20\%$ is fulfilled [15,19]. Table 1 shows that only our first measurement does not meet this condition. However, this datum will be included in the next analysis where the modified Bethe–Bloch expression for the stopping power will be used to determine both the mean excitation potential and the Barkas parameter.

3.1. Modified Bethe–Bloch expression for stopping power

The theory of the energy loss of charged particles in matter has always been of continuing interest. Particularly, a lot of effort has been given to find corrections to the well known Bethe–Bloch formula [23–25] which being based on first-order perturbation theory depends on the square of the charge of the incident particle $q_1 = Z_1^2 e$. Thus, the stopping power expression can be written as a sum of contributions that directly depend on the normalized speed of the incident charged particle $\beta = v/c$ as follows:

$$S(E) = \frac{kZ_1^2 Z_2}{\beta^2} L_0(\beta) \quad (4)$$

$$L_0(\beta) = \left[\ln\left[\frac{2m_e c^2 \beta^2}{(1-\beta^2)I}\right] - \beta^2 - \frac{C}{Z_2} + \frac{\delta}{2}\right], \quad (5)$$

where k is a constant value that depends on the chosen units, I is the mean excitation energy, (C/Z_2) is the shell correction, and $\delta/2$

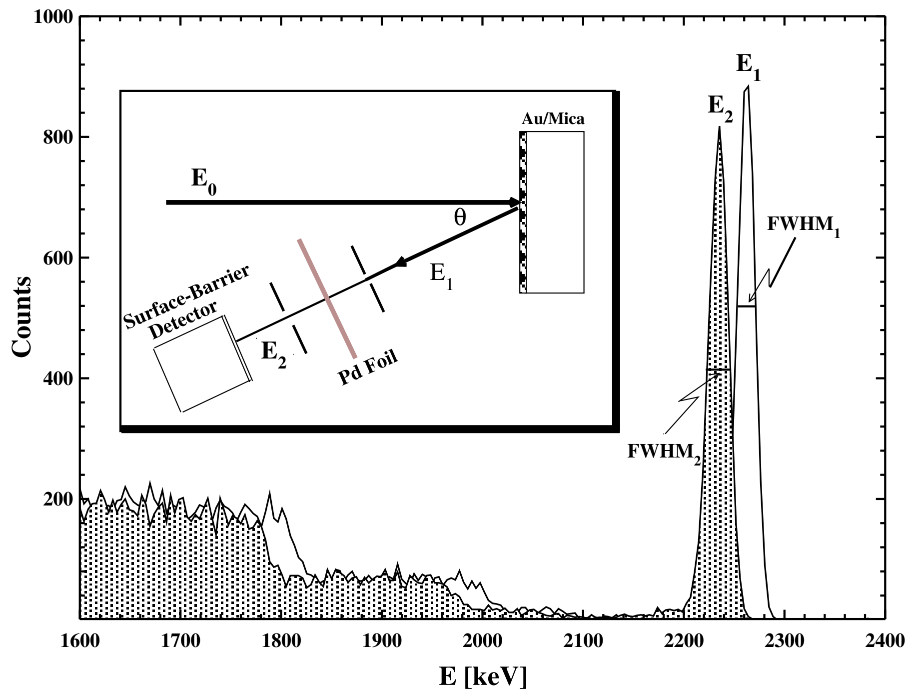

Fig. 1. RBS spectrum for $E_{avg} = 2255.4$ keV protons on palladium sample which is subsequently used to determine the energy loss in the foil. In the inset, the outline of the transmission method used in this work is shown.

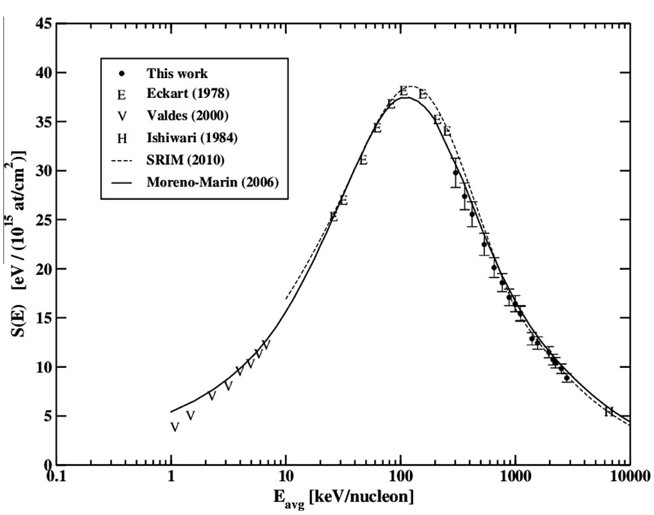

Fig. 2. Experimental stopping power cross section data of palladium for protons. Present values are compared to previous stopping power data and also to calculated values from SRIM-2010 and Ref.[21]

corresponds to target density effects. Since this last term is only important for highly-relativistic particles, it can be safely neglected for protons with energies lower than 20 MeV. However, the study of Barkas [26] demonstrated that at the same energy, the energy loss process for positive and negative charged particles showed small but notorious differences. In this way, a correction term proportional to $Z_1^3 L_1(\beta)$ had to be added to the Eq. (4) in order to take into account this contribution. Based on a harmonic oscillator model Ashley et al. [27,28] derived the so-called Barkas term which is given by:

$$Z_1 L_1(\beta) = \frac{Z_1 F_{arb}(b/\sqrt{x})}{\sqrt{Z_2 x^3}} \tag{6}$$

$$x = \frac{v^2}{v_0^2 Z_2} = \frac{40.2}{Z_2 M_p} E_p, \tag{7}$$

where x is a variable proportional to the energy per unit mass E_p of the particle, $v_0 = 2.19 \times 10^6$ m/s corresponds to the Bohr velocity and function F_{arb} can be found in Ref.[27]. It is worth noting that in Eq. (6), the *Barkas parameter b* is the only free parameter which can be determined by a fit of experimental stopping power values, a procedure that will be explained later. In the same reference a semi-empirical expression for the Barkas parameter is given, namely:

$$b = 0.8 Z_2^{1/6}\left(1 + 6.02 Z_2^{-1.19}\right). \tag{8}$$

Thus, for protons on a palladium sample a value of $b_e = 1.61$ can be considered as an expected value which will be later compared to that extracted from our stopping power data.

Additionally, a higher-order correction proportional to $Z_1^4 L_2(\beta)$ to the stopping power formula has also been predicted, which connects the classical stopping power theory with the quantum–mechanical approach. This correction, known as the Bloch term [29], can be calculated using the next equation:

$$L_2 = \psi(1) - \mathrm{Re}[\psi(1 + iy)] = -\left(\frac{1}{y^2}\right)\sum_{i=1}^{\infty} \frac{1}{i(i^2 + y^2)} \tag{9}$$

$$y = \frac{Z_1 \alpha}{\beta}, \tag{10}$$

where ψ is the logarithmic derivative of the gamma function [30] and α corresponds to the well-known *fine structure constant*.

Electronic shell-effect corrections C_s to stopping power values can be determined by using the work of Khandelwal [31,32], which take into account the contribution of each target electronic shell ($s = K, L, M, N$). The corresponding values for both C_K and C_L are determined as a function of both the *screening parameter* θ_s, and another parameter η_s which can be considered as a normalized incident energy. Once each pair (θ_s, η_s) is well determined, asymptotic expressions for C_K and C_L can be calculated. Additionally, subshell L_I, L_{II} and L_{III} contributions to C_L can be taking into account following Khandelwal assumption $\theta_{L_{III}} \approx \theta_{L_{II}} = 0.50$. Upper-shell contributions are determined using a scale procedure [33–36]. This approach assumes that the dependency of upper-shells C_M, C_N, C_{O--P} to β^2 is similar to C_L, except for scale factors. In the particular case of protons on a palladium sample, using this procedure we found the following expression:

$$C = C_K(\beta^2) + V_L C_L(H_L\beta^2) + \cdots + V_M C_L(H_M\beta^2) + V_N C_L(H_N\beta^2), \quad (11)$$

where C_K and C_L are the K and L-shell correction terms, while V_i and H_i parameters correspond to scale factors which can be extracted from Ref.[37].

Therefore, a modified Bethe–Bloch expression for the stopping power is derived by adding the above mentioned corrections:

$$S(E) = \frac{kZ_1^2 Z_2}{\beta^2}\left[L_0(\beta) + Z_1 L_1(\beta) + Z_1^2 L_2(\beta)\right], \quad (12)$$

where $L_0(\beta)$, which contain the shell-effect correction, was already defined in Eq. (5).

By a thoughtful analysis of the experimental stopping power measurements $S(E)$, a suitable procedure [37–43] is commonly used to extract values of the key parameters of the above formulation, i.e., the Barkas parameter b and the mean excitation energy I. Firstly, an experimental reduced cross section X_{exp} is derived from the expression (12):

$$X_{exp} = \ln\left(\frac{2m_e v^2}{1-\beta^2}\right) - \beta^2 - \frac{\beta^2}{0.3071 Z_1^2 (Z_2/M)} \times S_{exp}, \quad (13)$$

which can be compared by a fitting procedure to its equivalent theoretical expression:

$$X_{theo} = \ln I + \frac{C}{Z_2} - Z_1 L_1 - Z_1^2 L_2 \quad (14)$$

Assuming that both the shell-effect term and the $Z_1^2 L_2$ correction can be calculated accurately enough, then the target mean excitation energy I and the Barkas parameter b can be derived from the fit.

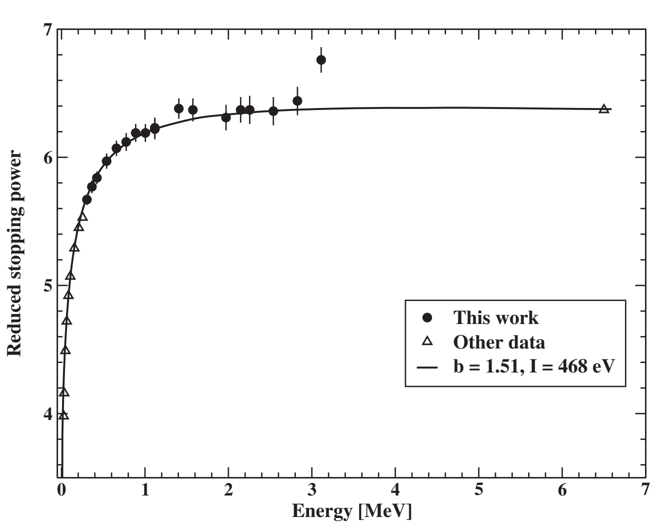

Fig. 3. Reduced cross section X_{exp} as a function of incident energy. The solid line represents the best fit of the theoretical X_{theo} curve, which is obtained with $I = 468$ eV and $b = 1.51$, to experimental reduced cross section data. Our X_{exp} value at the highest energy (3111.9 keV) was not taken into account in this analysis since only values with $\sigma \leqslant 1$, corresponding to an acceptable agreement between theory and experiment, were included.

In Fig. 3 the reduced stopping power data obtained in this work are displayed as a function of the incident energy, along with the currently available experimental data ranging from 100 to 6500 keV/amu [8,9]. At lower energies, there is an excellent match between our data expressed in the form X_{exp} with previous values from reference [8]. However, over the energy range $E > 1.4$ MeV the X_{exp} values begin to show some oscillation. Also, clearly seen in this plot is the fact that our datum at the highest energy (3111.9 keV) departs from the trend shown by the other data. To perform a fitting procedure by using Eq. (14) and the X_{exp} measured data points, a figure of merit is usually defined:

$$\sigma = \left(\frac{1}{N}\sum_{i=1}^{N}\left\{\frac{(X_{exp})_i - (X_{theo})_i}{(\Delta X_{exp})_i}\right\}^2\right)^{1/2}, \quad (15)$$

where $(\Delta X_{exp})_i$ corresponds to the uncertainty in each of the X_{exp} values measured so far, including data from this study as well as the point at 6.5 MeV found in reference [9]. Thus, only $\sigma \leqslant 1$ values were considered in order to achieve an adequate agreement between calculated and experimental data values. In this way, the solid line in Fig. 3 represents the best fit ($\sigma = 0.473$) without considering the last of our experimental data. The deduced parameters of palladium for protons obtained from this procedure are $b = 1.51 \pm 0.06$ and $I = 468 \pm 5$ eV. In the case of the Barkas parameter there is a relative difference of 6.2% from the expected value ($b_e = 1.61$). It should be noted that the measured value agrees fairly well with the accepted value ($b = 1.35$) for silver [38], a medium with an electronic structure (Z = 47) very similar to palladium (Z = 46). On the other hand, our measured mean excitation energy agrees very well with the accepted value of 470 eV [38], since there is a relative difference of only 0.43% between them. This measurement is also in reasonable agreement with the value (459 ± 14) eV reported by Ishiwari et al. at 6.5 MeV [10].

The influence of the three corrections mentioned above can be studied in terms of the "stopping number", a function defined in reference [37] as $L(\beta) = L_0(\beta) + Z_1 L_1 + Z_1^2 L_2$. Over the high energy regime ($E > 1$ MeV), the shell-effect yields the largest contribution to $S(E)$ while at lower energies the Barkas term becomes the predominant correction. For instance, at $E = 1$ MeV, the Barkas term is the most important component reaching up to 12.5% of calculated stopping power $S(E)$ whereas the shell and Bloch corrections contribute 10% and 2%, respectively.

4. Conclusion

Stopping power cross sections of palladium for protons have been measured over an energy range of 0.3 to 3.1 MeV using the transmission method. To our knowledge, this is the first time stopping power data of palladium for protons at this relevant energy regime are reported. Stopping power cross section data measured in this work were then compared to calculated values showing a good agreement particularly at higher energies. In addition, our data show a trend that matches very well with experimental values reported in an energy range below 0.3 MeV. Subsequently, from the reduced stopping power data both the target mean excitation energy I and the Barkas parameter b of palladium for protons were extracted showing a good agreement with expected values. Forthcoming new experimental data and corresponding theoretical analyses for the same target are being planned not only within a broader energy regime, but also for heavier ions like alpha-particles. The latter is in order to study the importance of the projectile electronic structure [43,44] that should certainly also influence the stopping process.

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