

The Resonance Integral of Niobium

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Abstract

The resonance integral of niobium has been studied by both pile oscillator and activation techniques. A value of $8.15 \text{ b} \pm 0.65 \text{ b}$ was obtained for the infinitely dilute integral. In addition, the variation of the resonance integral with foil thickness has been measured for thicknesses in the range 0.06 mm to 1.36 mm.

A separate study of the half-life of the isomeric state in Nb^{94} yielded a value $T_{1/2} = 6.30 \pm 0.03 \text{ m}$ which is about 5 % lower than the value given in literature.

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THE RESONANCE INTEGRAL OF NIOBIUM

E. Hellstrand, G. Lundgren

• Introduction

There is a growing interest for using niobium in reactor materials. It is for instance a constituent in zircaloy, and recently a number of uranium-niobium alloys have been studied as promising fuel element material. Especially for the latter use the amount of niobium will be large enough to be of importance for the neutron balance in a reactor system. The thermal cross section is known within about 2 %, but the resonance integral and its variation with sample thickness are less well determined. We have therefore found it worth while to make an experimental study of the resonance absorption in niobium.

• Method of measurement

The investigation was performed in two steps. A measurement of the resonance integral, RI, for thin foils was first made using both pile oscillator and activation techniques. Only the former gave an absolute value, for reasons outlined below. However, the agreement between the results confirmed the possibility of using the activation technique for the second study, that of the variation of RI with foil thickness.

The niobium samples consisted of metal foils, the thinnest being 0.06 mm thick. The metal was of high purity; no detailed chemical analysis could be obtained from the manufacturer, but a spectrographic check of the sample material revealed only traces of impurities.

• Dilute integral

• 1 Pile oscillator measurements

The technique for this kind of pile oscillator measurements has been described in an earlier report (1), and only a few details will be given here. Boron was used as the cross section standard. The samples were not surrounded with cadmium, so the reactor response was proportional to the total absorption cross section in the reactor RI spectrum. The maximum available sample size for the 0.06 mm thick

foils was $24 \times 200 \text{ mm}^2$. This amount of niobium corresponds to an effective absorption cross section of about 2.5 mm^2 , which is quite small for the type of measurement under consideration. In addition, the resonance absorption contributes only about 25 % to this value. To obtain more reliable results not only one foil but also two and three (on top of each other) were oscillated at a time.

The resonance integral was obtained in the way described in (1) by using the known thermal cross section, σ_o^{Nb} , and a measured value of the factor $a = r \sqrt{\frac{4T}{\pi T_o}}$, the latter being obtained from the cadmium ratio of gold.

For σ_o^{Nb} a value of 1.16 ± 0.02 was used (2), and a was determined as 0.0478 ± 0.0015 (compare section 2). After correction for scattering and moderation effects the following results were obtained

Table 1

Number of foils	Thickness, mm	RI, b
1	0.06	7.34 ± 0.30
2	0.12	7.64 ± 0.20
3	0.18	7.22 ± 0.16

The uncertainties given in the table are only the experimental ones and do not include the errors in the cross sections used. As may be seen the uncertainties are large enough to partly conceal the expected variation of RI with foil thickness.

3.2 Activation measurements

Neutron capture in niobium yields Nb^{94} which decays to Mo^{94} with a half-life of 10^4 years. This half-life is inconveniently long for the type of measurements under consideration because it requires large neutron dosages and very pure samples. However, Nb^{94} has an isomeric state 40 keV above the ground level with a half-life of about 6 m. Most of the decay from this state occurs to the ground state but a minor part, 0.1 %, is a combined β - γ decay to Mo^{94} . The predominant γ -line in the latter decay has an energy of about 0.9 MeV and is well

suitable for activity measurements. We therefore chose this line for our measurements. As not all of the γ -cascades from the original excited states pass to the ground level via the isomeric level the cross section for activation to this state does not coincide with the absorption cross section. With the method of measurement used this is in principle of no importance. However, the fractional population of the isomeric level corresponding to absorption at a given resonance will depend on the spin of the original excited state. There are two possible spin states for s-wave capture and this number is further increased by the circumstance that some of the resonances are p-wave levels. Therefore, with the present inadequate knowledge of the decay schemes for the different levels our measurement could not yield an absolute value of the resonance integral. However, in actual fact the discrepancy between our activation cross section and the absorption cross section need not be large, since the effect of the spin difference between the excited levels of the compound nucleus is obscured by the complex decay scheme. In addition, the resonance integral is in principle obtained by comparing the resonance and thermal capture (compare below), and the latter contains contributions from the same spin states as the former. That the above discrepancy is made small by these various factors is borne out by the fact that the results from the activation measurements agree well with those from the pile oscillator.

The method chosen for the cross section measurements was simply to compare the cadmium ratio for niobium and gold foils irradiated in the central channel of the reactor. Assuming the thermal cross section for niobium and gold and the resonance integral for gold as known, the resonance integral for niobium may be obtained from the following relation

$$RI = \frac{g \sigma_o}{R - 1} \left[\frac{1}{a} - R \sqrt{\frac{4E_o}{E_{Cd}}} \right] \quad (1)$$

- σ_0 is the 2200 m/s absorption cross section for niobium
 R is the cadmium ratio for niobium
 g is the non- $1/v$ correction factor, which is assumed to be unity for niobium
 a is a measure of the epithermal flux relative to the thermal flux. It is obtained from the cadmium ratio for gold. With Westcott's nomenclature a is equal to

$$r \sqrt{\frac{4T}{\pi T_0}}$$
 E_0 is 0.025 eV
 E_{Cd} is the cadmium cut-off energy. $E_{Cd} = 0.60 \pm 0.05$ eV

The gold foils were actually made from an alloy of lead and gold containing 0.1 weight per cent of gold. They were 0.1 mm thick. Both gold and niobium foils were circular, with a diameter of 20 mm. The niobium foils were 0.06 mm thick. The foils were mounted back to back, the bare samples being wrapped in aluminium and the cadmium-covered ones in 1 mm thick cadmium. A bare and a cadmium-covered package were rigidly mounted at a distance of 24 cm from each other and irradiated symmetrically about the point of maximum flux in the central channel of the reactor. The irradiations were repeated several times with alternatively the cadmium-covered or the bare package on top. By suitable averaging, small differences in flux level at the two positions became of negligible importance. The results of earlier experiments (unpublished) have shown that the disturbance of the neutron flux by the small amount of cadmium used in the experiment is small. In addition, its influence on the niobium cross section deduced from the experiment becomes even smaller by the fact that it affects the gold and niobium activities in the same way.

The following cadmium ratios, R , were obtained

$$R^{Nb} = 3.74 \pm 0.025$$

$$R^{Au} = 2.33 \pm 0.018$$

From the R -value for gold a value of 0.0478 ± 0.0015 was obtained for a . However, the use of this value in Eq. 1 requires that the epithermal flux is constant in lethargy units. This is not quite the fact for the $R1$ central flux, as has been demonstrated experimentally (3).

Normalizing the flux, $\phi(u)$, to 1 at 5 eV (the main gold resonance) $\phi(u)$ increases gradually to about 1.09 at 1000 eV. For higher energies it starts to decrease. A correction factor to transform the α -value obtained with gold to one more correct for the niobium resonances has been obtained by calculating the quantity

$$\gamma = \frac{\sum \phi(u_i) RI_i}{\sum RI_i}$$

where RI_i is the resonance integral for a resonance at an energy corresponding to u_i . RI_i for energies below 1.3 keV has been calculated from the parameters given in (2). Inserted in the expression for γ they yield a value of 1.07 ± 0.02 for γ . This value may also be used for the whole resonance region as the inclusion of the contribution to RI from energies above 1.3 keV has a very small effect on γ . $\phi(u)$ slopes downwards quite slowly and as much as 80 % of the unresolved part of RI comes from energies below 10 keV.

With γ equal to 1.07 ± 0.02 the α -value for niobium should be $\gamma \cdot \alpha_{Au} = 0.0511 \pm 0.0020$. Using this value one obtains from Eq. 1

$$RI = 7.65 \pm 0.32$$

for the 0.06 mm thick foils.

Self-screening effects are not negligible for the foils used. To make an approximate extrapolation to zero foil thickness, packages of one, two or three foils were irradiated together with gold foils (for normalizing purposes). The activities of the foils were counted and the relative intensities per unit mass calculated. After correction for the $1/v$ contribution, the results are as in Table 2.

Table 2

Number of foils	Activity
1	1.000
2	0.958 ± 0.010
3	0.928 ± 0.010

These values have been used to obtain the relative intensity at zero foil thickness by simply assuming a constant second difference for the first four points. This yields a value of 1.054 for the point at zero thickness. The uncertainty in this value is difficult to estimate but is likely to be of the order of 2 %. The self-screening factor for the 0.06 mm foil may also be calculated from Wilkins's formula (4). Calculations were made for the 12 strongest resonances and a weighted mean value was formed assuming self-screening for the rest of the resonances to be negligible. A value of 0.95 was obtained in this way, which is in agreement with the value $1/1.054$ estimated from the experimental points. A correction factor of 1.050 ± 0.015 was therefore used for transforming the single foil resonance integral to that for an infinitely thin sample.

The factors in Table 2 were also used to correct the pile oscillator results for two and three foils to correspond to a single foil.

3.3 Results

Applying the self-screening correction to the pile oscillator and activation results yields the following sets of values

Table 3

Method	Number of foils	RI _{corr.} b
Pile oscillator	1	7.70 ± 0.70
	2	8.37 ± 0.65
	3	8.16 ± 0.65
	Mean:	$8.15^{*\pm} 0.65$
Activation	1	8.05 ± 0.35

* The weighting of the individual values from the pile oscillator runs was made with considerations to the experimental errors given in Table 1 as well as to the uncertainties in the correction factors given in Table 2.

Although the pile oscillator and activation results agree we only quote the former, i. e. 8.15 ± 0.65 b, as an absolute value of RI. Earlier measurements have yielded 8.3 b (5) and 13 ± 5 b (6) from pile oscillator measurements and $4.0 \pm 15\%$ (7) from an activation measurement (the latter has been corrected to correspond to the new value of 1500 b for the gold resonance integral used as the cross section standard). The spread of the oscillator results is large but the limits of error overlap. The activation result is only half of that obtained in the present work.

Calculations of the resonance integral from resonance data below 750 eV have been made by Saplakoglu et al. (8) in connection with their time-of-flight measurements of the niobium resonances. The s and p-wave contributions in the unresolved region were obtained from the average level parameters deduced from the data below 750 eV. A value of RI of 7.8 b was obtained. The extension of the resolved resonance region up to about 1.3 keV (2) has, however, revealed a series of quite strong resonances which change the computed value considerably. The sum of the resonance integrals for the resolved resonances now amounts to 7.45 b. Above 1.3 keV a table of the (n, γ) cross section as a function of energy has been used. The table has been compiled by J. Schmidt, Karlsruhe, (private communication) and the values are mostly based on work at Oak Ridge Nat. Lab. (compare also (9) and (10)). Weighted over a $1/E$ flux these data yield a value of 4.85 b for the unresolved energy region. The total calculated resonance integral thus becomes 12.3 b. The limits of error should be 20-30 % as estimated from the uncertainties of the cross section data. We have not found any cause for the large discrepancy between the calculated value and our measured one.

Variation of RI with foil thickness

We had some difficulty in obtaining pure niobium metal foils at the time of our measurement. Therefore the activation method, which requires less material, was chosen for the study of the dependence of RI on foil thickness. Although this method had the drawbacks mentioned under section 3.2 the agreement between the pile oscillator

and activation results for the dilute integral justified the use of this technique for the present study. The scarcity of the material also limited the range of sample thicknesses to 0.06 mm - 1.35 mm.

The samples were made up of different numbers of foils of a diameter of 17 mm and a thickness of about 0.13 mm. Foil packages of 2, 4, 6 and 10 foils were used. The packages were covered with cadmium and irradiated in pairs, such as 2 and 4, 4 and 6 etc. The samples were placed at a distance of 8 cm from each other. The flux was monitored with a gold foil in each package.

As the half-life is short for the measured decay each foil in a package could not be measured individually. To ensure that two packages were counted with the same efficiency independent of the number of irradiated foils in the package, unirradiated foils were added on each side of the thinner package to make up for the difference. Comparisons were in the same way made between thin foils (0.06 mm) and a package of 2 foils. Generally, pairs of thin packages were irradiated at higher power levels than thick ones. In addition, to obtain good counting statistics for all packages without too large dead time corrections, packages with a large difference in number of foils could not be irradiated at the same time. The relation between the resonance integral for the thinnest foil and those for the thickest packages was therefore less well determined than the ratio of the integrals for neighbouring packages.

The result of the measurement in relative units is given in Fig. 1 and in Table 4.

Table 4

Effective foil thickness, d, mm	$\frac{RI(d)}{RI(o)}$
Inf. thin	1
0.060	0.950 [*] \pm 0.014
0.110	0.907 \pm 0.017
0.165	0.877 \pm 0.017
0.260	0.836 \pm 0.017
0.510	0.768 \pm 0.018
0.745	0.733 \pm 0.019
0.175	0.694 \pm 0.021

* The relation between the dilute resonance integral $RI(o)$ and that for the 0.060 mm thick foil is discussed at the end of section 3.2.

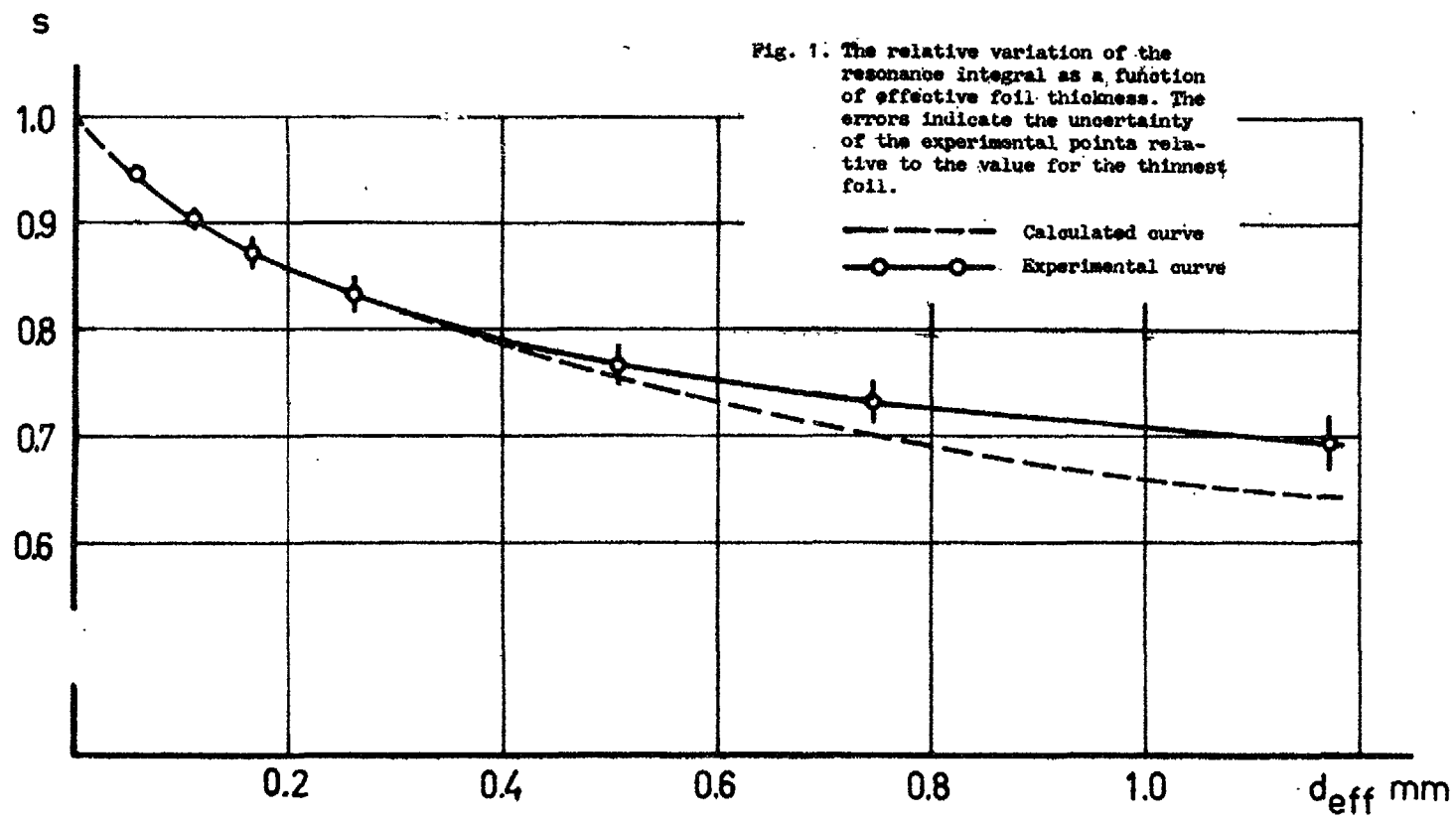


Fig. 1. The relative variation of the resonance integral as a function of effective foil thickness. The errors indicate the uncertainty of the experimental points relative to the value for the thinnest foil.

The values are given as a function of effective foil thickness, i. e. the thickness of an infinite foil having the same surface to mass ratio as the actual package. A calculated curve is also given in Fig. 1. This curve has been obtained from Roe's calculation of self-screening as a function of foil thickness and resonance parameters (11) by forming the expression $s = \frac{\sum s_i RI_i}{\sum RI_i}$ where s_i is the self-screening factor belonging to the resonance i with the strength RI_i . s varies of course with foil thickness, d , and has been calculated for four values of d . For small and medium thicknesses the agreement is good between the experimental and calculated curves. For thicker samples the experimental curve lies higher than the calculated one. The opposite effect would be expected, as resonance scattering is neglected in Roe's work. However, the uncertainty of the experimental points for thick samples relative to the extrapolated value for zero thickness is large, as mentioned earlier (about 3 %). The parameters of the niobium resonances are also quite uncertain and the disagreement may very well be a result of the large limits of error.

5. The half-life of Nb^{94m}

The experiments described in the last section sometimes required quite large time corrections of the measured activities. Thus the half-life of the Nb^{94m} decay had to be known quite accurately. A preliminary study showed that the value given in the literature, 6.6 m (12) was somewhat high. A separate determination was therefore made using the same γ -spectrometer as for the rest of the measurements. The decay of the 0.9 MeV γ -activity from irradiated foils and, for control purposes, from samples of Nb₂O₅ of high purity, was followed over about five half-lives, points being taken about every third minute. No differences in half-lives exceeding the limits of error were found for the two different niobium samples. To ensure that no common short-lived activity affected the measurements, samples were irradiated for 2, 6 and 12 minutes, the aim being to change the proportions of the wanted and possible unwanted activities (the same effect could in principle be obtained by following the decay over a long period of time, but this requires very high starting intensities with subsequent large dead time corrections). No change in the measured half-life was found. The following mean value as determined with the method of least squares was found:

$$T_{1/2} = 6.30 \text{ m} \pm 0.03 \text{ m}$$

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