Fine Structure of the K X-Ray Absorption Edge of Germanium*

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A double-crystal spectrometer, with a proportional counting system for intensity measurement, has been employed for the investigation of the x-ray absorption structure on the high-energy side of the Ge K edge, using 38% polarized x rays and a thin single crystal of germanium in the transmission method. The structure was studied up to 185 ev from the main edge and several new absorption structure features, not reported hitherto, were obtained. These features are satisfactorily explained by Hayasi's theory in the close-in region and by Kronig's theory in the extended region. A significant shift, without any intensity variation, was noted in the extended fine structure on changing the orientation of the single-crystal absorber. A quantitative correlation has been made between the observed structures and the theoretically predicted values with a view to testing different theories on absorption fine structure.

INTRODUCTION

VARIOUS theories have been proposed to explain the nature of the fine structure on the high-energy side of x-ray absorption edges. They have been successful in interpreting qualitatively certain aspects of the fine structure in different energy regions depending upon the different assumptions on which they are based.

Hayasi's theory[1] for the K x-ray absorption structure assumes that the final state of the ejected photoelectrons, a "quasi-stationary" state, is still coupled to the inner K vacancy and is the result of the electron in the p-type state being reflected back and forth from a pair of planes near the parent atom and at right angles to the electron's direction of propagation. This condition may be satisfied, in most cases, in the region lying within 75 ev of the main absorption edge. Hence, Hayasi's theory is expected to be more successful in this region. However, it is quite likely that some absorption structures in the close-in region may be the result of characteristic energy losses of the ejected photoelectron, either by multiple interband transitions[2] or by plasma oscillations.[3]

Kronig's theory[4] considers the photoelectron ejected by the x-ray quantum to be essentially free and moving in the crystal lattice as a plane wave. This condition presumes the electron to be relatively energetic and relatively far away from the emitting atom. Hence, Kronig's theory is expected to be more successful in explaining the so-called extended structure in the absorption spectra, which, in most cases, lies in the spectral region beyond 50 or 75 ev from the main edge. According to this theory, maxima in absorption fine structure on the high-energy side of the absorption edge correspond to the ejection of an electron from an inner level to one of the allowed energy zones, determined from the band theory of solids. Minima correspond to forbidden energy regions, for which the photoelectron suffers Bragg reflection from some set of lattice planes in the crystal.

There is also the possibility of an indefinite intermediate energy region in which the fine structure of the absorption spectra may not be accurately predicted by either of these theories.

One verification of Kronig's theory, and the range of its validity, can be obtained by using highly polarized x rays and a single-crystal absorber. In this case, photoelectrons would be emitted predominantly in the direction of the electric vector of the incident x rays. The angle θ_0 between the ejected electrons and the normal to some important plane in the crystal lattice can be changed by rotating the single-crystal absorber about an axis parallel to the direction of incident x rays; this would shift the position of the corresponding absorption discontinuity because of the dependence of E on $\cos^2\theta_0$. It might also slightly affect the intensity of absorption maxima or minima because of their shift, one way or the other, on the monotonically decreasing part of the absorption vs energy curve. If a shift of position and change in intensity are observed on the rotation of the absorber in some spectral region of the absorption curve, there would be provided an excellent verification of Kronig's theory together with an indication of the region in which it is valid.

Earlier measurements[5,6] along these lines were not completely successful because the subject crystal was used both as absorber and analyzer in the double-crystal spectrometer. Recently, El-Hussaini and Stephenson[7] used a thin single crystal of germanium placed between the two cleaved calcites of a double-crystal spectrometer in the transmission method, and observed a significant shift in the extended structure positions for three different orientations of the absorber. Such a result had hardly been expected since the degree of polarization was only 7%. However, they could not

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[3] D. Pines, Revs. Modern Phys. **28**, 184 (1956); I. B. Borovskii and V. V. Schmidt, Soviet Phys.—Doklady **4**, 885 (1960).
[4] R. de L. Kronig, Z. Physik **70**, 317 (1931); **75**, 468 (1932).
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[7] J. M. El-Hussaini and S. T. Stephenson, Phys. Rev. **109**, 51 (1958).

TABLE I. Bragg angles, dispersions in (1, −1) and (1, +1) positions, and the degree of polarization for the Ge K-edge wavelength with the different crystal combinations.

Crystal combination	θ_A	θ_B	$D(1,-1)$ sec/xu	$D(1,+1)$ sec/xu	Degree of polarization %
Calcites (211) and (211)	10° 36′	10° 36′	0	69.3	7.0
Calcites (741) and (211)	51° 11′	10° 36′	195.6	264.9	91.2
Calcites (655) and (211)	32° 44′	10° 36′	84.4	153.7	70.6
Quartz ($20\bar{2}3$) and ($20\bar{2}3$)	24° 0′	24° 0′	0	164.9	38.2
Quartz ($20\bar{2}3$) and calcite (211)	24° 0′	10° 36′	47.8	117.1	38.2

study the structure shifts quantitatively in terms of angles measured with respect to particular crystal planes because, before the angular orientation of some prominent set of planes within the absorber could be determined, the absorber was accidently fractured.

Considering both the present theoretical and practical interest in germanium, it was thought desirable to pursue the study of the extended structure of the K absorption edge of a single crystal of germanium in known different orientations using highly polarized x rays, and to correlate the observed absorption structure quantitatively with the structures predicted by different theories.

EXPERIMENTAL

The double-crystal spectrometer and x-ray supply have been described previously.[6–8] The intensity registration system consisted of a Tracerlab, Inc. end-window type of proportional counter (model TGC-12) filled with 90% argon+10% methane, nearly at atmospheric pressure, and having a mica window of 1.4 mg/cm^2 thickness, a regulated high-voltage power supply (Baird-Atomic model 318), a Baird-Atomic preamplifier (model 219A), a Baird-Atomic linear amplifier (model 218), a Baird-Atomic single-channel pulse-height analyzer (model 510), and a Potter Instrument Company predetermined decade scaler (model 341A). This system of pulse-height discrimination made it possible to increase the intensity of the x-ray beam by increasing the x-ray tube voltage without recording the overlapping of higher order radiations in the observed energy spectrum. It was therefore possible to overcome the major difficulty of the accurate measurement of the very low intensities reflected from the two crystals of the spectrometer while using highly polarized x rays for the study of the Ge K edge.[7]

The germanium crystal used in this study was generously supplied by W. F. Leverton of the Raytheon Manufacturing Company. It was of n-type (40 ohm-cm) with about 20 mm diameter and about 3 mm thickness. For thinning down to the optimum thickness of about 12 μ,[7] the method of successive lapping and polishing followed by chemical etching was employed.[9] The thickness of the crystal, which was reduced to 95 μ after lapping and polishing, was further reduced to 12.5 μ (as determined from x-ray absorption measurements) by the chemical etching of the crystal at room temperature with a solution of glacial acetic acid (8 parts), nitric acid (3 parts), and hydrofluoric acid (1 part). An investigation of the Laue patterns of this thin crystal revealed that its face was parallel to the (111) lattice planes within two degrees, and the [110] direction lying in a (111) plane made an angle of 16°±2° with a fiducial mark on the crystal holder. This crystal holder could be placed in or out of the x-ray beam between the two crystals of the double-crystal spectrometer, and could also be rotated about the direction of the incident x rays which was kept perpendicular to the face of the crystal absorber.

For checking the performance of the proportional counter and its associated electronic equipment, cleavage faces of optically perfect calcite crystals were used in the double-crystal spectrometer. Preliminary measurements were made of the absorption fine structure up to 100 ev from the Ge K edge using the single-crystal absorber described above, and the structures up to 60 ev from the absorption edge were found to be in satisfactory agreement with those obtained by El-Hussaini and Stephenson,[7] and others.[10,11]

For higher polarization work, a calcite crystal with reflecting face cut and polished along the (741) planes (supplied by G. Brogren of the University of Uppsala, Sweden) was used as crystal A in the spectrometer, and one of the calcites with cleavage face was retained as crystal B. The dispersion and the polarization obtained with this crystal combination are given in Table I. Attempts to investigate the Ge K-absorption structure with these crystals in the (1, +1) position were not successful, since the incident intensity I_0 in the neighborhood of the wavelength of the Ge K edge was only 0.6 counts/sec with an equal intensity of the background. A significant side result obtained in these investigations was a rocking curve of half-width 6.6 ev for the W $L_{\gamma 1}$ emission line, which was smaller than the least value obtained by others,[12] using two cleaved calcite crystals.

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[9] J. W. Faust, Jr. in *Methods of Experimental Physics*, edited by K. Lark-Horovitz and V. A. Johnson (Academic Press, Inc., New York, 1959), Vol. 6, Part A.
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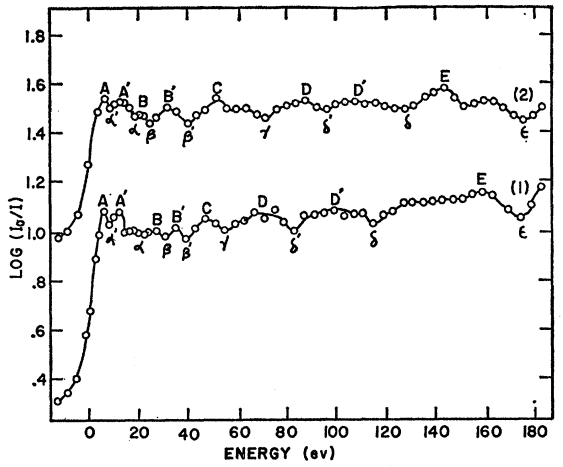

FIG. 1. The x-ray K-absorption spectrum of a single crystal of germanium in two different orientations using a quartz crystal $(20\bar{2}3)$ and a calcite crystal (211) in the double-crystal spectrometer.

A calcite crystal cut and polished along the (655) plane (also supplied by G. Brogren) was next used as crystal A, while crystal B was retained. The resolution of the double-crystal spectrometer with this crystal combination was not so high as had been obtained with the (741) crystal, but it was considered sufficient for the study of the extended structure in the K-absorption spectrum of germanium. However, preliminary experiments on the Ge K edge showed that the calcite (655) crystal was but poorly suited for the problem. The advantage of the high polarization (70.6%) obtained from the crystal was more than offset by its low reflectivity. The signal-to-background ratio was only about 1.5 on the high-energy side of the Ge K edge and no dependable extended structures in the Ge K-absorption curves could be obtained.

Attempts were then made to use a reflecting face of a quartz crystal having a grating space suitable to give high polarization, and at the same time having strong reflectivity. A quartz crystal cut and polished along the $(20\bar{2}3)$ plane was selected for which the available degree of polarization (38.2%) was a compromise with the expected reflectivity. Two quartz plates (2 in.$\times$1 in.$\times$0.25 in. each), cut and polished along $(20\bar{2}3)$ planes, were obtained from the Valpey Crystal Corporation. On mounting these crystals in the double-crystal spectrometer, a $(1, -1)$ rocking curve of half-width 22.8 sec was obtained for W L_{γ_1}. A calcite (211) crystal was then used to replace one of the quartz crystals as the crystal B. A satisfactory $(1, -1)$ curve of 13.4 ev half-width for W L_{γ_1} was obtained with the combination of the quartz $(20\bar{2}3)$ crystal and the calcite (211) crystal. As the dispersion in the spectrometer in the $(1, -1)$ position was quite sufficient for the problem, the final measurements of the absorption structure on the high-energy side of the Ge K edge were made using this crystal combination of the quartz $(20\bar{2}3)$ crystal and the calcite (211) crystal in the $(1, -1)$ position. The incident intensity I_0 in the region investigated was about 15 counts/sec with signal-to-background ratio of about 10, and the transmitted intensity I on the high-energy side of the Ge K edge was about 2 counts/sec with signal-to-background ratio of about 3. Intensities I_0 and I were obtained alternately, with at least 2000 and 1000 counts, respectively, recorded for each point in a run over the absorption structure.

RESULTS

Figure 1 shows the results for two different orientations of the absorber. The curve labeled (1) is for the orientation (1) of the absorber, in which the fiducial mark on the crystal holder was parallel to the electric vector of the polarized component of the incident x rays. It is a composite of three separate runs over the main edge and up to about 35 ev, and a composite of

TABLE II. Absorption fine structure of germanium measured in electron volts from the main edge.

Structure	Present work, single crystal in orientation (1)	Present work, single crystal in orientation (2)	El-Hussaini and Stephenson, single crystal in three different orientations	Doran and Stephenson, single crystal in unknown orientation	Hulubei and Cauchois, polycrystalline powder
A	6.0	6.5	6, ..., 6	6.2	5.7
α'	8.0	9.0	...	...	...
A'	12.0	13.0	...	...	...
α	21.0	19.0	17.7, ..., 17.7	15.7	14.6
B	27.0	22.0	24, ..., 25	25.2	20.0
β	31.0	25.0	34, ..., 36	31.7	28.0
B'	35.0	33.0	...	...	...
β'	39.0	40.0	...	...	...
C	47.0	52.0	45, 48, 47	51.0	39.0
γ	55.0	72.0	59, 60, 61	61.0	47.0
D	70.0	88.0	79, 78, 73	79.0	77.0
δ'	83.0	96.0	...	...	...
D'	99.0	108.0	...	...	...
δ	115.0	126.0	113, 114, 108	114.0	108.0
E	157.0	143.0	151, 138, 138	147.0	157.0
ϵ	175.0	177.0	196, 185, 169	186.0	193.0

TABLE III. Absorption fine structure of a germanium crystal measured in electron volts from the zero-energy level.

Structure	Energy in orientation (1)	Identification of lattice planes possibly causing the structure	Energy in orientation (2)	Identification of lattice planes possibly causing the structure
A	17.5	004 (18.88 ev)	18.0	004 (18.88 ev)
α'	19.5	...	20.5	...
A'	23.5	133 (22.42 ev)	24.5	133 (22.42 ev)
α	32.5	333, 115 (31.86 ev)	30.5	333, 115 (31.86 ev)
B	38.5	044 (37.76 ev)	33.5	...
β	42.5	135 (41.30 ev)	36.5	044 (37.76 ev)
B'	46.5	026 (47.20 ev)	44.5	026 (47.20 ev)
β'	50.5	335 (50.74 ev)	51.5	335 (50.74 ev)
C	58.5	...	63.5	...
γ	66.5	246 (66.08 ev)	83.5	066, 228 (84.96 ev)
L	81.5	...	99.5	...
δ'	94.5	048 (94.40 ev)	107.5	139 (107.38 ev)
D'	110.5	...	119.5	...
δ	126.5	159, 377 (126.26 ev)	137.5	359 (135.70 ev)
E	168.5	0012 (169.92 ev)	154.5	1311, 179, 559 (154.58 ev)

two runs beyond 35 ev. The curve labeled (2) is for the orientation (2) of the absorber, with the fiducial mark on the holder making an angle of 45° with the electric vector of the polarized component of the incident x rays, and is a composite of two different runs. The absorption maxima and minima, indicated by capital letters and small Greek letters, respectively, are listed in Table II with their energies in electron volts relative to the main edge as a zero. For comparison, the values reported by Hulubei and Cauchois, Doran and Stephenson, and El-Hussaini and Stephenson are also tabulated.

It is evident from Table II that several structure features, not reported previously, were observed in the close-in region on the high-energy side of the absorption edge and in the region between the absorption maxima D and E. No significant change was observed in the absorption fine-structure positions up to 40 ev from the main edge on changing the orientation of the absorber, but beyond 40 ev the shifts in the structure were quite pronounced with the absorption maximum E shifting towards the main edge and all other extended structures (except ϵ) shifting away from the edge. The minimum ϵ was presumably under the strong influence of the W $L_{\gamma 1}$ line, which was located in very close proximity to this structure. No change (beyond experimental error) in the intensity magnitude of the extended structure features was observed on changing the orientation of the absorber.

DISCUSSION

Earliest investigations on the K edge structure of germanium (in polycrystalline powder form) were by Beeman and Friedman,[13] and by Hulubei and Cauchois.[11] Recently, work on the K x-ray absorption spectrum of a single crystal of Ge has been done by Doran and Stephenson,[10] El-Hussaini and Stephenson,[7] and Tsutumi and Obashi.[14] Doran and Stephenson obtained a very rich extended structure by using a thin single crystal of germanium in the transmission method in a double-crystal spectrometer. El-Hussaini and Stephenson later used this transmission technique to study the effect of the single crystal orientation on the K-absorption spectrum of Ge and found shifts in positions of the extended structures even with 7% polarized x rays. Tsutumi and Obashi studied radiations reflected from (100), (110), and (111) planes of a germanium single crystal in a tube spectrometer using unpolarized x rays and photographic registration; although no close-in structure was detected, they observed in all three cases almost similar extended structure, which was in agreement with the results reported by Doran and Stephenson in that energy region. A comparison of the energy positions of the structures observed in the present investigation with those obtained by other workers (Table III) shows that (apart from the new fine structure features observed in this study and taking into account the different orientations of the absorber used in different studies) there is a good general agreement between the absorption structure found in the present work and that reported earlier by Doran and Stephenson and by El-Hussaini and Stephenson, and a fair agreement with that reported by Hulubei and Cauchois, whose work was with a completely different condition of the absorber.

Kronig's theory predicts the absorption minima at the forbidden energy regions for the ejected photoelectron in a solid. The mean energy of such a region for a cubic crystal, as measured from the average inner potential of the crystal, is given by

$$E=h^2(\alpha^2+\beta^2+\gamma^2)/8md^2\cos^2\theta_0, \quad (1)$$

at which energy the de Broglie waves of the photo-

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[14] K. Tsutumi and M. Obashi, J. Phys. Soc. Japan **13**, 591 (1958).

electron are Bragg-reflected from a set of planes (α,β,γ), d being the grating space, m the mass of the electron, and θ_0 the angle of incidence of the electron on the plane (α,β,γ).

On the other hand, Hayasi's theory predicts absorption maxima when the photoelectron is ejected into a "quasi-stationary" state, which corresponds to the 90° Bragg reflections of the de Broglie waves of the photoelectron from a set of parallel lattice planes. The energy of this quasi-stationary state for a cubic crystal, with the average inner potential of the crystal taken as zero energy level, is given by

$$E=(150/4d^2)(\alpha^2+\beta^2+\gamma^2) \quad \text{ev}, \tag{2}$$

when the lattice constant d is expressed in angstroms. This is essentially the same expression as Eq. (1) with θ_0 put equal to 0°.

Since germanium has a diamond-type lattice, which may be considered as consisting of two interpenetrating face-centered cubic lattices, all those reflections from the crystal planes will be extinguished for which the indices α, β, γ are mixed (i.e., both odd and even), and extinction will also occur for some planes (α,β,γ) due to the diamond glide. Moreover, the average inner potential, given by

$$\bar{W}=(h^2/8md^2)(3N/\pi)^{\frac{2}{3}}, \tag{3}$$

where N is the total number of free electrons for a grating cell, is 11.5 ev for the germanium crystal. This value gives the energy of the K-absorption edge of germanium measured from the zero level, and hence it must be added to the observed energies of the absorption maxima and minima given in Table II, so that a comparison can be made between the observed structure and the structure predicted theoretically. Table III gives the energies of the observed absorption structures of Table II, measured from the bottom of the potential well as the zero-energy level, and gives also the lattice planes which would normally reflect the electron waves with energies close to these observed values.

None of the observed absorption maxima can be ascribed to the characteristic energy losses of electrons passing through the Ge crystal, since the values of these losses in a Ge crystal are 16.4 ev and 33.8 ev as obtained by Watanabe,[15] and are 16.0 ev and 30.1 ev as obtained by Marton and Leder.[16]

It is apparent from Table III that the absorption maxima A, A', B, and B' in the close-in region, i.e., up to about 50 ev from the zero-energy level, are in good agreement with the energies predicted by Hayasi's theory, except that the maximum B in the orientation (2) of the Ge crystal cannot be identified by any lattice plane. This particular structure B seems to have been formed due to superposition of two adjacent absorption minima, α and β, on the monotonic part of absorption curve.

The absorption minimum α does not have any identifying lattice plane, but all the succeeding minima (lying beyond 30 ev) show very good agreement with the energies predicted by Kronig's theory, the agreement being excellent for minima in the extended region (i.e., beyond 50 ev). Further, the absorption maxima C, D, and D' in the extended energy region do not correspond to any quasi-stationary states predicted by Hayasi's theory, although the maximum E seems to be fairly satisfactorily explained by that theory. This latter fact seems to support the view that, provided suitable planes for the normal reflection of the ejected photoelectrons at those energies, Hayasi's theory might hold good even up to 170 ev, i.e., even in the extended energy region.

The appearance of the new absorption structure features, which were not observed before, seems to be chiefly due to the orientation of the single-crystal absorber and the high degree of polarization employed in the present study, which made it possible for certain lattice planes (as indicated in Table III) to be effective in producing these significant features.

Quite pronounced shifts in the structure observed beyond 50 ev upon rotating the absorber about the normal to its face indicate that Kronig's theory is valid beyond 50 ev, which is nearly the same result as that obtained by El-Hussaini and Stephenson. The lack of any significant variation in the amplitude of the structure in the present investigation can be ascribed to the partial polarization of the incident x rays which resulted in some integration over all the angles θ_0 for the crystal planes involved. A very small change in amplitude is expected even with 100% polarized x rays and with infinite resolving power of the spectrometer.

As mentioned earlier, the [110] direction in the Ge crystal kept in the orientation (1) was determined as making an angle of $16°\pm2°$ with the direction of the electric vector in the polarized component of the incident x rays. Hence the angle θ_0 of Eq. (1) would be 16° for the set of planes (110) in the orientation (1) and would be 29° for the same planes in the orientation (2). Considering the fourth-order and the sixth-order reflections from the planes (110), we obtain the corresponding absorption minima at 40.9 ev and 92 ev, respectively, in the orientation (1), and 49.3 ev and 111 ev, respectively, in the orientation (2) of the absorber. The observed minima β (42.5 ev) and δ' (94.5 ev) in the orientation (1) and β' (51.5 ev) and δ' (107.5 ev) in the orientation (2) agree quite well with these calculated values, thus satisfying the necessary condition for the validity of the $\cos^2\theta_0$ dependence predicted by Kronig's theory. But this agreement is not necessarily unique in view of the fact that some other lattice planes might also contribute to the formation of these absorption minima (see Table III)

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due to the partially polarized radiation used in this investigation, and also due to all the photoelectrons not being ejected solely in the direction of the electric vector of the incident radiation.

However, this close correspondence between the observed minima in the two known orientations of the absorber, and their expected energies from Kronig's theory, offers for the first time possible quantitative evidence for the $\cos^2\theta_0$ dependence predicted by that theory.

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Intermediary Effects in Nuclear Beta Decay*

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Two intermediate meson theories, the vector meson theory and the scalar meson theory, of weak interactions are analyzed for nonlocal effects in nuclear beta-decay processes. The principal effects are (1) the introduction of a nonlinearity in the Kurie plot in both meson theories and (2) the alteration of the electron-neutrino angular correlation in the vector meson theory only. These effects are shown to be quite small, of the order of $\frac{1}{10}\%$ in the most favorable cases, for the lower mass limits imposed on the mesons by the requirement of compatibility with present experimental data. The magnitude of these effects is considered to be on the threshold, at least, of measurability. Both meson theories produce effective nuclear beta-decay coupling constants that differ in the order of a percent from the effective constants in muon decay.

I. INTRODUCTION

THE growing interest in intermediate meson theories of the weak interactions raises two important questions: (1) What is the present evidence for an intermediary? (2) What are the prospects for more evidence on the intermediary question in the near future? "Evidence" here is taken to mean some experimental finding explainable only, or at least most credibly, by a meson intermediary. The invariance properties and selections rules obtained in the intermediary theories hardly seem admissible as evidence since these results could as well be assumed themselves, there being essentially a one-to-one correspondence between these results and the meson properties assumed in the present theories. On the other hand, a nonlocality of the weak interaction can be considered as evidence, locality being inherent in the Fermi theory and nonlocality being characteristic of intermediate meson theories.

To date, the only evidence for a meson intermediary is found in muon decay. The Michel parameter appears, at least from the recent data,[1] to be greater than $\frac{3}{4}$, the value predicted by the Fermi theory with $V-A$ coupling. A nonlocal weak-interaction theory consistent with the present Fermi theory leads to

$$\rho \approx \tfrac{3}{4}+\tfrac{1}{4}(m_\mu^2/M^2), \tag{1}$$

for the Michel parameter and

$$|\xi| \approx 1+(1/15)(m_\mu^2/M^2) \tag{2}$$

for the asymmetry parameter where M is the mass of the intermediate meson. These results are the same for a vector meson intermediary[2] and a scalar meson intermediary,[3] provided only that the coupling between mesons and fermions is direct and the fermion currents are written in a form such that the Fermi theory is obtained in the local limit. As already noted, $\rho>\frac{3}{4}$ has been reported; however, only $|\xi|<1$ has been obtained so far but with confidence limits insufficient to exclude $|\xi|$ equal to or slightly greater than one. Further, the value of the Michel parameter evidence is considerably reduced by (1) the Plano value $\rho=0.780\pm0.025$ being too large, i.e., requiring M too small (although the broadness of the confidence limits admits the possibility of a reasonable M) and (2) the existence of older data[4]

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