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Zero-bias anomalies in tunnel junctions made with a polymerized benzene barrier*

R. Magno and J. G. Adler
Department of Physics, University of Alberta, Edmonton, Alberta, Canada T6G 2J1
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Zero-bias anomalies (ZBA's) observed in tunnel junctions, whose barriers were formed in a benzene glow discharge, are found to be associated with the formation of organic radicals. The temperature and magnetic field dependence of these anomalies is that predicted by the Appelbaum model for tunneling via an exchange interaction with magnetic moments in the barrier. Comparison with junctions prepared in the same system in an oxygen glow discharge show the anomaly to be due to organic radicals (known to form in a benzene glow discharge) rather than to metallic magnetic impurities. The g values and other parameters deduced from the ZBA's depend on the junction electrodes indicating interaction of the radicals with the metals comprising the junction. These results extend the usefulness of electron tunneling as a tool to study organic materials: by using inelastic tunneling to study the molecular vibrations, and elastic tunneling near zero bias to study the presence and nature of radicals.

I. INTRODUCTION

The interaction of a tunneling electron with organic molecules located in the barrier region of a junction has been the subject of many papers[1] over the past few years. Most of these have examined the excitation of the vibrational modes of the molecules, and one has dealt with the excitation of an electronic transition.[2] This paper reports the observation of a temperature and magnetic-field-dependent zero-bias anomaly (ZBA) in junctions whose barriers were formed in a benzene glow discharge. The molecular vibrations seen in these junctions are the subject of a second paper.[3] The ZBA consists of a conductance peak which increases in amplitude as the temperature is lowered, and decreases in amplitude when a magnetic field is applied until in a large field a conductance minimum is seen. Typically the ZBA is about 2 meV wide at 1 K and ranges in amplitude from 0.1% to 3% of the background conductance.

Similar ZBA's have been reported for metal-insulator-metal,[4-8] metal-semiconductor,[9-12] and *pn* junctions.[13,14] The results of several of these experiments[8,11] have been used in detailed examinations of Appelbaum's[15-19] model of tunneling via a spin-exchange interaction with magnetic moments in the barriers. These tests have shown that in the absence of a magnetic field the theory gives a good description of the temperature and voltage dependence of the conductance peak. In the presence of a field the agreement is not as good and it is necessary to extend the model to include magnetic-field-induced lifetime broadening.

It is not the aim of this paper to carry out another detailed study of Appelbaum's theory, but rather to use it to show that exchange scattering is the source of the observed ZBA's, and to show how to extract some of the parameters which characterize these barriers.

Appelbaum used perturbation theory to calculate the conductance of a junction with a group of non-interacting localized paramagnetic states located in the barrier on a plane near one of the metal-barrier interfaces. In this picture, there are three contributions to the total conductance $\sigma = \sigma^{(1)} + \sigma^{(2)} + \sigma^{(3)}$. $\sigma^{(1)}$ describes the usual elastic tunneling and electron scattering from the magnetic impurities by mechanisms other than the spin exchange. $\sigma^{(2)}$ describes tunneling from one electrode to the other with spin exchange. In the absence of a magnetic field, no energy is required to flip the local moment's spin, so $\sigma^{(2)}$ is independent of voltage. When a magnetic field is applied, this tunneling channel is not available until the tunneling electron can supply energy equal to the local moment's Zeeman splitting $\Delta = g\mu_B H$. Therefore, $\sigma^{(2)}$ has a threshold of $|eV| \simeq \Delta$. By measuring the width of the minimum the g value of the local moment may be found.

The calculation of $\sigma^{(3)}$ involves summing terms which correspond to the various paths through an intermediate state k with energy ϵ_k. Because the Pauli principle governs the intermediate as well as initial and final states the Fermi functions do not cancel. This, together with the perturbation-theory energy denominator $1/(\epsilon_i - \epsilon_k - \Delta)$, gives the temperature and voltage dependence of the zero-bias conductance peak. Physically, the peak corresponds to interference between reflected and transmitted exchange-scattered currents. When a magnetic field is applied, $\sigma^{(3)}$ splits into a small peak at zero bias and two larger ones at $eV = \pm\Delta$. Thus, $\sigma^{(3)}$ determines the shape of the conductance curves in the absence of a magnetic field, and both $\sigma^{(2)}$ and $\sigma^{(3)}$ contribute when a field is applied.

In this study the magnetic moments are associ-

ated with the unpaired electrons found on organic radicals. The precise nature of the radicals is not understood. They may be associated with benzene rings which have lost or gained an electron during the glow discharge. The discharge is also breaking the benzene molecules into small fragments which then recombine on the metal surface to form a solid insulating barrier. If the recombination process is not complete the radicals may be associated with the fragments rather than a benzene ring.

The technique of forming an organic insulator by running a discharge in an organic vapor is usually referred to as glow discharge polymerization. Infrared[20-23] and electron-spin-resonance[24] experiments have been done on a variety of materials formed by glow-discharge polymerization. The infrared data show that the starting molecules do break down and that they recombine in many ways. As an example, $-CH_2-CH_2-CH_2-$, $-C=C-$, and $-C\equiv C-$ along with aromatic rings have been identified when benzene is glow-discharge polymerized. Electron-spin-resonance[24] studies have reported the presence of organic radicals in glow-discharge polymerized naphthalene and anthracene which are very similar to benzene. Typically values of $g \simeq 2$, linewidths $\simeq 5$ to 20 Oe, and spin concentrations $\simeq 10^{19}$/g have been reported. There have been numerous other studies on glow-discharge polymerized materials and they have been summarized in several reviews.[25-27]

II. EXPERIMENTAL

Sample preparation consisted of evaporating a base metal of either aluminum or magnesium, growing an organic barrier on it, and then evaporating lead or silver counter electrodes. The organic barrier was formed by breaking a benzene filled capillary in the bell jar and then running a dc glow discharge in the vapor. Typically the benzene pressures ranged from 300 to 400 μm, and the discharge was run for 30 min at 500 V. The discharge electrodes also became covered with organic material, and as a result they required cleaning between runs. This usually involved running the discharge in a few hundred microns of room air for several hours. On many occasions, junctions were produced with an oxygen discharge after carrying out a benzene discharge and the cleaning procedure. These oxygen-discharge junctions showed neither the ZBA reported here, nor inelastic tunneling structure associated with the presence of hydrocarbon contamination in the barrier.[28]

The measurements were made in a cryostat providing temperature down to 0.9 K and fields up to 70 kOe. The junctions were always mounted with the tunneling current flowing parallel to the magnetic field. Because of the random distribution of moments in the barrier no angular magnetic-field dependence is expected.

Absolute measurements of the dynamic conductance $\sigma = dI/dV$ and $d\sigma/dV = d^2I/dV^2$ were made using a bridge technique discussed elsewhere.[29] The software had been updated since the last report to increase the bias voltage resolution by a factor of 5. Values of

$$\sigma_e/\sigma(V_n) = [1/2\sigma(V_n)]\,[\sigma(+V) + \sigma(-V)]$$

and

$$\frac{1}{\sigma(V_n)}\frac{d\sigma_e}{dV} = \frac{1}{2\sigma(V_n)}\left(\frac{d\sigma(+V)}{dV} - \frac{d\sigma(-V)}{dV}\right)$$

were calculated for comparison with the theory. $V_n = 9$ meV was used with the aluminum data while $V_n = 2.5$ meV was used for the magnesium data.

III. RESULTS

The presence of a ZBA in junctions prepared in a benzene glow discharge was confirmed by examining more than 30 junctions over a period of several months. The temperature and magnetic-field dependences of the ZBA were studied in detail for several of these to determine the nature of the ZBA. The results for two junctions sharing a common magnesium base film are shown in Fig. 1 to illustrate the general features. The series of curves shown in Fig. 1(a) demonstrate how the conductance changes from a parabolic shape at 10 K to parabolic background with a sharp peak at 0.96 K. For this Mg-Ag junction the peak reached an amplitude of $\simeq 0.25\%$ of the background conductance. This same peak is shown, in Fig. 1(b), to decrease in size, and then to become a conductance minimum as a magnetic field is applied. As the field is increased the minimum becomes wider and deeper. At 68 kOe the depth is 2.5% of the background conductance, about ten times the size of the peak in zero field. A lead counter electrode was used to form the second junction on this magnesium base. The magnetic-field dependence for this junction is shown in Fig. 1(c) with the same scale used in Fig. 1(b). Comparison shows that the data in Figs. 1(b) and 1(c) are very similar except in size. More will be said later about the dependence of the ZBA on the electrode metals used and on the sample-preparation techniques.

The conductance curves shown in Fig. 1 are considerably different from those found when an oxygen glow discharge is used. This difference is illustrated by the conductance curve for a Mg–oxygen-discharge–Ag junction which is shown in the

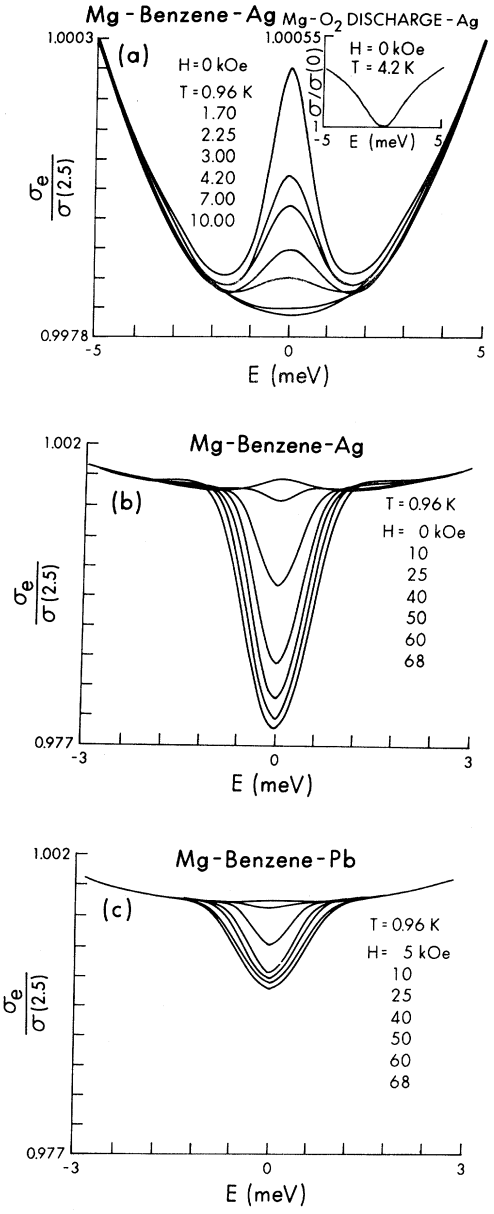

FIG. 1. Even part of the conductance for (a) temperature dependence of Mg-Ag, (b) field dependence of Mg-Ag, and (c) field dependence Mg-Pb.

insert in Fig. 1(a). The conductance minimum shown in this inset has been studied in detail, and it has been shown that it is due to nonequilibrium tunneling effects.[30] Several other junctions were made with an oxygen discharge during the course of this study and they all showed this nonequilibrium type of conductance minimum. Tunneling experiments have also shown that the nonequilibrium conductance minimum is independent of magnetic field when the tunneling current flows parallel to the field.[31] This is the orientation which was used in all these experiments. Further tests on some of the oxide junctions prepared during this work showed their conductance minima were field independent up to 70 kOe as they should be if the minima is due to nonequilibrium effects. The fact that the oxide barrier junctions exhibit standard nonequilibrium ZBA's indicates that the ZBA's found in the benzene glow discharge junctions are associated with the organic barrier and *not metallic magnetic impurities*. If magnetic impurities were present in the vacuum system (say from transition-metal impurities) then the oxide junctions would have shown ZBA's such as those in Fig. 1 (main) rather than the standard nonequilibrium ZBA's as in the inset of Fig. 1. It should be emphasized that the same vacuum system, metal boats and metals were used in preparing junctions whether an oxygen discharge or a benzene discharge was used. That is, the essential difference in the two types of junctions was the use of either benzene vapor or oxygen.

The behavior shown in Fig. 1(a) is that predicted by the Appelbaum theory for the conductance in the absence of a magnetic field. The shape of the conductance peak and its temperature dependence have been compared with

$$\sigma^{(3)}(V,T) = -A \ln\{[(eV)^2 + W^2]/E_0^2\}, \qquad (1)$$

which is an approximation to the integral form given by Appelbaum.[19] A gives the amplitude of the conductance peak while W gives its width. Normally, $W = \alpha k_B T$ is expected since the width should be determined by thermal smearing. E_0 is a cutoff parameter used in the perturbation-theory calculation of $\sigma^{(3)}$. The assumption used is that the exchange-coupling parameters are constant within $\pm E_0$ of the Fermi surface and zero elsewhere. A comparison of Eq. (1) and the data presented in Fig. 1 is shown in Fig. 2 where values of $\sigma^{(3)}(V, T = 0.96\ \mathrm{K})$ and $\sigma^{(3)}(V = 0, T)$, are plotted versus $\ln[(eV)^2 + W^2]$. The conductance curve at 10 K was chosen as the background curve in extracting $\sigma^{(3)}$ from the data, thus, $\sigma^{(3)}(V,T) = \sigma_e(V,T) - \sigma_e(V, T = 10\ \mathrm{K})$ was used in preparing Fig. 2. A value of W was found for each temperature using the $d\sigma/dV$ data, and by noting that the functional form of $d\sigma^{(3)}/dV$ has a minimum at $eV = W$. $W = \alpha k_B T + \beta$, was fit to the data with $\alpha = 1.1 \pm 0.2$ and $\beta = 0.15 \pm 0.03$. These values along with those from other junctions are listed in Table I. Contrary to the expectation that thermal smearing would give $W = \alpha k_B T$, the data indicate a temperature independent broadening is also present. The results of an analysis of Table I are similar to those of Wolf and Losee for metal-semiconductor junctions.[12] A value of E_0 has not been ex-

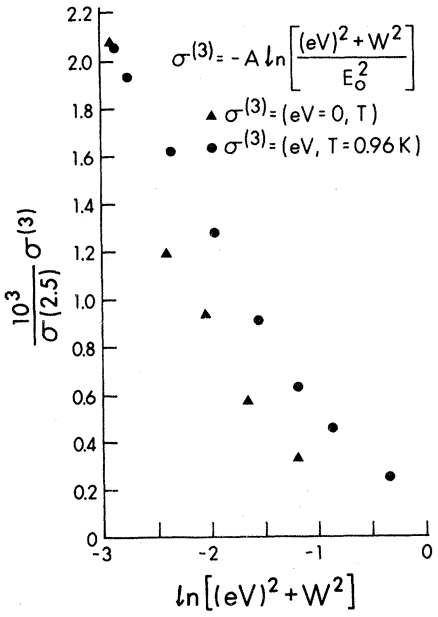

FIG. 2. Plot of $\sigma^{(3)}(eV=0,T)$ and $\sigma^{(3)}(eV,T=0.96\ \mathrm{K})$ vs $\ln[(eV)^2+W^2]$ for Mg-Ag.

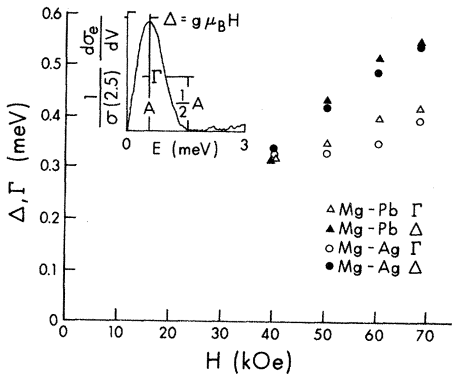

FIG. 3. Plot of the Zeeman energy Δ(meV) and the linewidth Γ(meV) vs H(kOe) for Mg-Ag and Mg-Pb.

tracted from the data, as this corresponds to adding a constant to the background conductance. This is easily understood by examination of the form of Eq. (1). Also E_0 is arbitrary since it enters the perturbation theory as a cutoff parameter. Considering the size of the ZBA, $\simeq 0.25\%$ of the background conductance at the lowest temperature, and thus the experimental resolution necessary, the data are in reasonable agreement with the linear plot predicted by Eq. (1).

The magnetic-field-dependent data shown in Figs. 1(b) and 1(c) are also in agreement with the Appelbaum theory. On applying a magnetic field the conductance peak decreases in size and eventually a conductance minimum is found. Then, as the field is increased the minimum becomes wider and deeper. Other experiments on metal-insulator-metal[8] and metal-semiconductor junctions[11] have shown that a detailed comparison of the shape and field dependence requires considering both $\sigma^{(2)}(V,T,H)$ and $\sigma^{(3)}(V,T,H)$. They have also shown that it is necessary to modify the theory to include magnetic field induced lifetime broadening of the paramagnetic states. The approach taken here is to show that when $\sigma^{(2)} > \sigma^{(3)}$ and when $\Delta/k_BT > 1$ the width of the conduction minimum increases linearly with field. The width of the transition from low to high conductance at $eV \simeq \Delta$ has also been measured since it too is expected to increase linearly with the applied field. The reason for this is that the number of conduction electrons in the neighboring electrode capable of relaxing the local moment increases with field.[11] The exchange interaction provides the coupling between the local moment and the conduction electrons. The inset shown in Fig. 3 shows how these widths were measured using $d\sigma_e/dV$.[10,11] The data plotted in Fig. 3 are for the Mg-Ag and Mg-Pb junctions already shown in Fig. 1. In carrying out this analysis the field dependence of $\sigma^{(3)}$ has been neglected. The justification for this is Appelbaum's prediction[17] that in a field the $\sigma^{(3)}$ terms should be about the same size or smaller than the peak found in zero field. Figure 1 shows that $\sigma^{(3)}(H=0)$ is about ten times smaller than the dip in σ_e at large fields for both the Mg-Ag and Mg-Pb data. The g values found from the slope of Δ vs H and the coefficients of $\Gamma = \Gamma' H + \Gamma_0$ are listed in Table II. It should be noted that the values of Γ_0 are $\simeq 5k_BT$ the amount of broadening expected from smearing of the Fermi surface.

The g values measured in these experiments are

TABLE I. Summary of fits to Eq. (1).

Junction	A	α	β (meV)
Mg-Ag	-0.8×10^{-3}	1.1 ±0.2	0.14±0.03
Al-Ag	-8.2×10^{-3}	2.1 ±0.2	0.26±0.08
Al-Pb	-0.9×10^{-3}	0.55±0.2	0.48±0.08

TABLE II. g values and linewidths.

	g	Γ' (meV/kOe)	Γ_0 (meV)
Mg-Ag	1.4±0.1	$(2.4\pm0.3)\times10^{-3}$	0.22±0.02
Mg-Pb	1.5±0.1	$(3.7\pm0.3)\times10^{-3}$	0.17±0.02
Al-Ag	1.3±0.2		
Al-Pb	1.7±0.2		

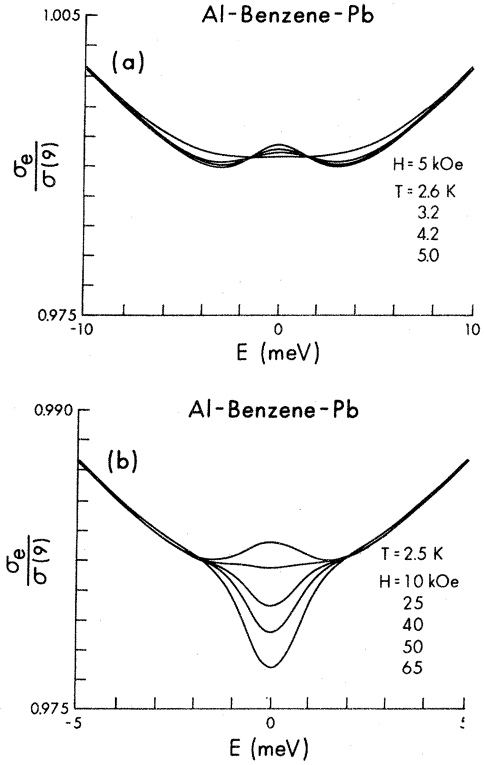

FIG. 4. Plot of the even part of the conductance for (a) temperature dependence of Al-Pb and (b) field dependence of Al-Pb.

for the paramagnetic moment as it interacts with the electrons in the neighboring electrode. Wolf and Losee have suggested[11] that $g = g_0 + 2J\rho$ and $\Gamma' = \pi (J\rho)^2 \Delta$ may be used to relate the measured g value to the free-electron value g_0 and to relate the field-dependent linewidth to the Zeeman energy. $J\rho$ is the product of the exchange constant J and the metal density of states ρ. Values of $|J\rho| = 0.3$ for Mg-Ag and $|J\rho| = 0.4$ for Mg-Pb are found using the slope Γ'. These values are consistent with the measured g values if $g_0 = 2$ is used with $J < 0$. $J < 0$ is consistent with finding as conductance peak in the zero magnetic field and a conductance minimum in a large field.

The effect of the electrode metals was further examined by making Al-Pb and Al-Ag junctions. The results for two junctions sharing the same aluminum base film are shown in Figs. 4 and 5. 2.5 K was the lowest temperature used to avoid superconductivity in the thin aluminum film. Analysis of the conductance peak gives graphs similar to that shown in Fig. 2 for the Mg-Ag data, and qualitatively the field dependences are the same as shown in Figs. 1(b) and 1(c). The point to be noted is that the ZBA is larger for the silver electrode than for the lead one for both Mg and Al base films. When Mg and Al base films are prepared together the Al base always gives the larger ZBA. These results indicate that the ZBA depends upon the metals chosen for the electrodes, and it suggests that the magnetic scattering may be occurring near both top and bottom electrodes.

To find more accurate values and linewidths it would be necessary to fit the line shapes noting that $\Delta/k_B T \simeq 1$ and that $\sigma^{(3)}$ is about the same size as $\sigma^{(2)}$. At the present time the apparatus used to carry out these experiments is limited to 1 K as the lowest temperature and 70 kOe as the highest field. Since the purpose of this paper is to show that the organic radicals found in glow-discharge polymerized benzene are the source of a ZBA similar to that discussed by Appelbaum these experimental limits are not a problem.

The dependence upon preparation technique is illustrated by the data shown in Fig. 6. These curves represent the peaks found at 4.2 K for junctions prepared on seven different occasions. Generally the benzene pressure used in these preparations was 350 ± 30 mTorr while the voltage was 460 ± 10 V dc and the current was 9 ± 1 mA. These results indicate that the size of ZBA and the shape of the background conductance are sensitive to the

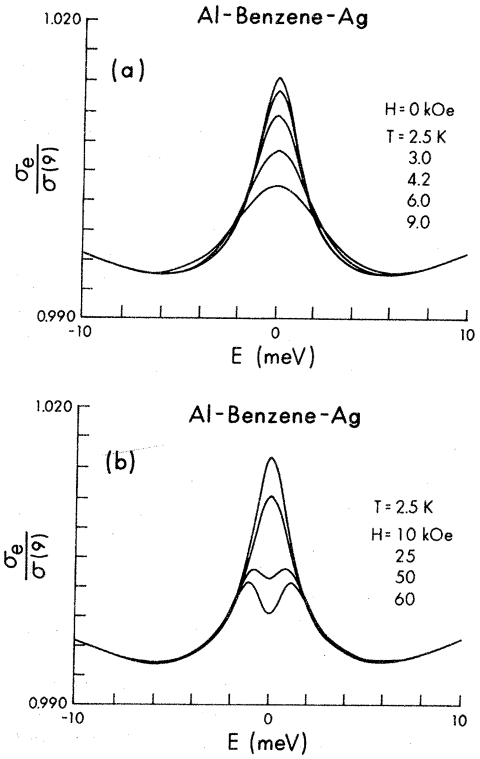

FIG. 5. Plot of the even part of the conductance fo (a) temperature dependence of Al-Ag and (b) field dependence of Al-Ag.

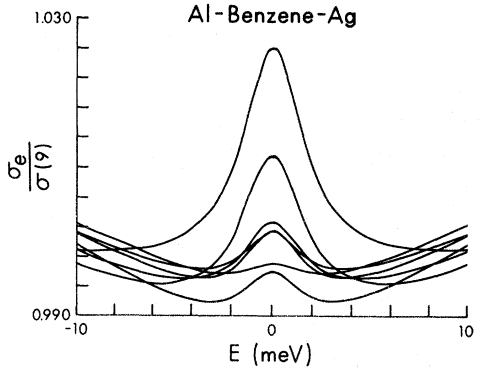

FIG. 6. Plot of the even part of the conductance at 4.2 K for seven Al-Ag junctions described in text.

preparation technique. A systematic study of the nature of the barrier as a function of the preparation techniques is beyond the scope of the present paper.

IV. DISCUSSION

The ZBA illustrated here has been found to be a common feature of junctions prepared in a benzene glow discharge. The temperature and magnetic-field dependences are the same as those predicted by Appelbaum's theory for tunneling via exchange scattering from local magnetic states in the barrier. A comparison of these junctions with ones prepared in an oxygen glow discharge indicates that the magnetic states are associated with organic radicals. It was also observed that the specific size of the ZBA depends upon the metals used for both the top and bottom electrodes, and upon the benzene barrier preparation techniques.

The data have been examined to determine what parameters may be used to characterize the junctions. Equation (1) gives a reasonable description of the conductance peak in the absence of a magnetic field. When a field is applied values of g and Γ may easily be extracted if $\sigma^{(2)} \gg \sigma^{(3)}$ and $\Delta/k_B T \gg 1$. There are some problems in making detailed comparisons with Appelbaum's theory. The choice of the background conductance $\sigma^{(1)}$ is a significant experimental problem, particularly when $\sigma^{(2)}$ and $\sigma^{(3)}$ appear to be only 0.1% to 3% of the conductance. It has already been mentioned that in other experiments it was found necessary to consider lifetime broadening of the local-moment states in order to use parameters found at low fields to calculate conductance curves at high fields. A third problem is that the Appelbaum model considers the local moments to be located on a plane near one of the electrodes. The data presented here suggests that magnetic effects are happening near both electrodes. Thus, in general it should be assumed that there is a nonuniform distribution of moments throughout the barrier. Appelbaum has considered the problem of averaging over the barrier[18] when calculating $\sigma^{(3)}$ and finds the functional form of Eq. (1) is still valid. The situation for the $\sigma^{(2)}$ terms may be different. Still it is expected that if $\Delta/k_B T > 1$ and $\sigma^{(2)} > \sigma^{(3)}$, the width of the conductance minimum still measures some average g value.

It should be noted that the g value measured in this experiment is different from that measured for a bulk sample. The shift occurs because of the overlap of the conduction electrons from the neighboring electrode with the local magnetic state. Since the electrode wave functions decay exponentially going into the barrier the amount of overlap changes too. Thus the exchange constant J, and the g values should be spatially varying.

The results of this study show that moments on organic radicals cause magnetic and temperature-dependent ZBA's of the type discussed by Appelbaum. This helps to explain the conductance peaks occasionally found in junctions prepared in oil contaminated vacuum systems. It is highly probable that the glow discharge is breaking the oil down and forming organic radicals. These experiments also show that tunneling may be used to study the organic insulators formed by glow-discharge polymerization. Currently there is a lot of interest in correlating the electrical and physical properties of these materials with their organic structures. There is also an interest in determining how the organic vapors polymerize in a glow discharge. The radicals formed in the glow discharge are expected to play an important roll in such polymerization. Generally people interested in studying such materials have had to do one experiment to study a material's conductivity then prepare a second sample for a magnetic resonance experiment and then a third to carry out an infrared experiment. A single tunneling experiment enables these studies to be carried out simultaneously on a single sample. Tunneling is also particularly well suited to studying effects occurring in the metal-barrier interface region.

At this point further study is needed to determine the exact nature of the radicals formed in the benzene glow discharge. It is not known whether the radical is a benzene ring which has gained or lost an electron or whether it is a fragment such as $H_3C-CH_2-$with an unpaired electron.

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