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TSDC (THERMALLY STIMULATED DEPOLARIZATION CURRENT)
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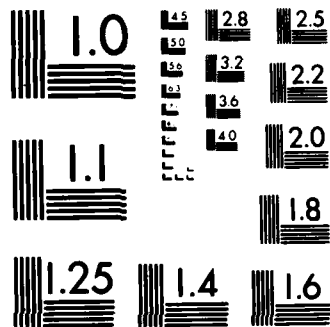
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TSDC STUDIES OF PEO AND PEO COMPLEXED WITH KSCN

by
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TSDC Studies of PEO and PEO Complexed with KSCN

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ABSTRACT

A digitally controlled thermally stimulated depolarization current (TSDC) facility is described and measurements on poly(ethylene oxide) (PEO) and PEO complexed with KSCN are reported. The results complement and extend previous electrical relaxation studies on these materials. In particular, in PEO the presence of a relaxation previously labeled α_c is confirmed. The relaxation is assigned to defects in the crystalline phase. In addition, the γ relaxation region is shown to consist of at least two relaxations. Further, a high temperature family of peaks is obtained which are associated with space charge. For KSCN-complexed PEO three low temperature relaxations are observed in the γ relaxation region. The relationship between these relaxations and previously reported dielectric relaxation results is established and possible origins of the relaxations are discussed. Finally, differential scanning calorimetry studies (DSC) were carried out on all of the materials.

Additional keywords:
poly(ethylene oxide); polymer electrolyte

I. INTRODUCTION

Relaxation phenomena in poly(ethylene oxide) (PEO) have been extensively studied. The early literature has been reviewed by McCrum et al.¹ Of continuing interest is the fact that the α relaxation was observed mechanically but not electrically.² A subsequent report of the observation dielectrically of α by Arisawa et al.³ has been disputed by Porter and Boyd.⁴ The difficulty is that PEO is conductive in the relatively high temperature region where α is expected to be found at audio frequencies. One approach would be to lower the frequency and hence lower the temperature of the relaxation to where the effects of the DC conductivity are minimized. Unfortunately, ultra low frequency AC measurements are difficult to perform at the level required to study relaxation phenomena in polymers. Fortunately, there are alternative, essentially equivalent techniques.

One such technique is thermally stimulated depolarization currents (TSDC). A recent, brief summary including references to reviews on the subject has been given by Gourari et al.⁵ As TSDC utilizes DC voltages, there is no contribution from the mobile charges for blocking electrodes. However, there may be space charge peaks from the mobile charges trapped at the electrodes by the DC bias. Consequently, the combination of these effects makes some relaxations more readily observed via TSDC than by DR. A second advantage of TSDC is the possibility of using "peak cleaning" techniques whereby closely spaced relaxations can be separately observed. In the present paper, the authors' implementation of TSDC is described along with some results for PEO and PEO complexed with KSCN. These results

appear to represent the first TSDC studies of PEO. In addition, differential scanning calorimetry (DSC) studies are reported for the purpose of materials characterization.

II. EXPERIMENT

The samples were prepared by dissolving PEO (Polysciences, MW 5×10^6) in methanol at about 60°C . In the case of the complexed materials, the salts were added in the correct proportions and dissolved simultaneously. The resulting solutions were poured onto a teflon plate and allowed to dry in air. After completing the drying under vacuum at 60°C for 24 hours, aluminum electrodes were evaporated onto the resulting films. These films were then cut to form disks about 8 mm in diameter and 0.7 mm thick. While one electrode covered the full surface, the other electrode was only about 7 mm in diameter.

The TSDC sample chamber was constructed by Dr. G. E. Matthews, Jr. of Wake Forest University and is the chamber used to obtain previously reported results.⁶ The chamber consists of a partially hollowed out copper cylinder surrounded by a heating coil, inside an airtight chamber. A spring loaded brass electrode presses the sample and the other electrode down onto the copper cylinder. A sapphire disk insulates the lower electrode from the copper. This arrangement guarantees good thermal contact and electrical insulation.

The temperature control was achieved using digital techniques. The sample temperature was derived from a copper-constantan thermocouple, buried in the copper cylinder 6mm from the sample, in conjunction with a

liquid nitrogen temperature reference junction. The thermocouple voltage was measured with a Keithley Model 195 digital multimeter and fed into an Apple IIe computer every 0.7 second. After comparing this to a desired thermocouple voltage (for the particular time) by subtraction, the resulting number was converted into a heating signal by a discrete time proportional - integral - differential control algorithm implemented on the computer. The heating coil was powered by a computer controlled power supply constructed by one of the authors (JPC). This temperature control system was generally used to generate a linear heating rate of 6K/min, but other programs such as "reciprocal" heating schemes were easily implemented. The control was generally to within 0.025K from 80K to 300K.

In a typical run the sample chamber was evacuated for 30 minutes after a sample had been loaded to eliminate surface water. The chamber was then filled with helium at about 0.5 psi and maintained there for the duration of the experiment. After polarizing the sample at a selected temperature and voltage for a suitable time (usually 300-500V, 10 min.) , the assembly was cooled with liquid nitrogen or helium to the starting temperature of the heating program. At this point the polarizing voltage was removed and an Applied Physics Corporation Model 30 vibrating reed electrometer was connected accross the sample. Once electrometer drift had subsided (about 5 min.), the heating program was begun and data was recorded on a Varian G-2000 chart recorder.

In several cases a peak cleaning technique had to be used to isolate a particular peak. While this is not generally appropriate for processes with a distribution of relaxation parameters, it was done as a compromise to allow a reasonably simple data analysis. Care was taken to

ensure that the temperature of maximum current and the curve symmetry were preserved.

The DSC studies were carried out using a DuPont 990 Differential Scanning Calorimeter which is interfaced with an Apple IIe computer. Data are transferred to the microcomputer via an Applied Engineering 12 bit A/D converter.

III. DATA ANALYSIS

A Gaussian-broadened TSDC equation was fitted to the data. The equation used was developed as follows. First, the monoenergetic TSDC equation for the current density, $J(T,E)$, was taken to be:⁷

$$J(T,E) = \frac{P_0}{\tau_0} \exp \left[-\frac{E}{kT} - \frac{1}{\beta \tau_0} \int_{T_0}^T \exp \left(-\frac{E}{kT'} \right) dT' \right] \quad (1)$$

where P_0 is the dipole moment per unit volume and β is the heating rate. τ_0 is the so-called reciprocal frequency factor (preexponential) and E is the activation enthalpy in the Arrhenius expression for the relaxation time which is assumed. T_0 is the starting temperature of the data run and the integral was calculated using Squire's approximation where $x = E/kT'$:⁸

$$\int_{T_0}^T \exp \left(-\frac{E}{kT'} \right) dT' = \frac{T' \exp(-x)(x+3.0396)}{(x^2+5.0364x+4.1916)} \bigg|_{T'=T_0}^{T'=T} \quad (2)$$

Next, the monoenergetic equation was modified to include the effects of distributed parameters by assuming a distribution function in E :

$$dJ = f(E)J(T,E)dE \quad (3)$$

and defining the distribution function to be a Gaussian:

$$f(E) = \exp \left(-\frac{(E-E_0)^2}{2\sigma^2} \right) \quad (4)$$

where E_0 is the mean activation energy of the reorientation.

Finally, the limits of integration were set at 0 and $3E_0$ so that the

working equation becomes:

$$J(T) = \int_0^{3E_0} f(E)J(T,E)dE \quad . \quad (5)$$

These integrations were performed using 3000 values of E from 0 to $3E_0$.

The fitting was done by manual adjustment of E_0 , τ_0 , σ , and a peak height factor in an interactive process via a graphics terminal.

Initially, a reasonable τ_0 was chosen from DR measurements,⁵ and σ and E_0 were chosen to produce a good match in the lower temperature part of the curve. Subsequently, adjustments were made in all four parameters to achieve a good fit. Typically 15 to 20 iterations were required.

The "best fit" criterion was visual rather than mathematical. The reason for this was that this procedure was the most efficient and accurate way of excluding the spurious contributions from space charge or peak-cleaning residuals which were always present. The presence of the spurious contributions and the "goodness" of the fits will be obvious from the appropriate plots (figs. 2, 6, 7).

IV. DISCUSSION

A. PEO

A typical data run for PEO is shown in Fig. 1. In addition to the dominant features labeled γ and α_c , there is a shoulder on the low temperature side of the γ relaxation at the position tentatively labeled γ_2 . The γ_2 relaxation will be discussed further in the following section. In order to obtain quantitative information on α_c the lower temperature peaks were "burned away." The resulting data and best fit to Eq. (5) are shown in Fig. 2. The activation parameters obtained from the best fit are listed in Table I.

The relaxation labeled α_c in the present work corresponds to the relaxation labeled α_c in previous DR studies⁹ for the following reason. The relaxation parameters for α_c from Table I predict that a DR peak measured at 100 Hz would be observed at about 258K and that is very close to the position of the peak labeled α_c in Fig. 1d of Ref. 2.

Also listed in Table I are the activation parameters for a peak in $\text{PEO}_{6.5}:\text{Ba}(\text{SCN})_2:3\text{H}_2\text{O}$ which was tentatively identified as α_c .¹⁰ α_c was observable in that material because the conductivity in the vicinity of room temperature was very low. The agreement is excellent considering that peak cleaning will decrease some of the lower energy components of the distribution that makes up α_c . The end result will be not only to reduce the apparent width of the distribution of activation energies but also to shift the apparent mean activation energy to higher values. Apparently, these effects were very small in the present application of the technique.

The question of the identification of this relaxation remains. In

previous papers,^{9,10} the authors tentatively associated the relaxation with the mechanical α relaxation discussed by McCrum et al.¹ However, the present analysis shows that the peak positions are significantly lower than those of the mechanical α relaxation which occurs at 263-273K for 0.5 Hz.² This has recently been confirmed by Kalfoglou¹¹ who reports a mechanical α relaxation at 303K for 110 Hz. Further, the usual interpretations of α as due to "the motion at the surface of the crystallites"¹² or "a crystal-disordering mechanism"⁴ do not seem to be consistent with a relaxation which occurs at a temperature as low as 170K, as is the case for α_c in the present work. Since, as is confirmed below, the glass transition occurs at about 210K, the α_c relaxation is independent of whether the amorphous phase is a glass or a rubber. Consequently, the former mechanism is unlikely though it would remain a possibility if the surface motion were independent of the state of the amorphous phase. The latter mechanism is almost certainly ruled out as crystal-disordering is unlikely to extend to such low temperatures. However, the independence of the glass transition strongly suggests that the relaxation is, indeed, associated with the crystalline phase. Specifically, the relaxation may be attributable to defects within the crystallites. The defects may be due to impurities, for example.

That the middle peak in Fig. 1 is the γ relaxation is confirmed by using the DR parameters in Table I for PEO¹⁰ to predict the position of the TSDC peak which is approximately equivalent to a 1.8 mHz DR peak. The predicted peak position is about 110K which is in good agreement with the results shown in Fig. 1.

Finally, some data for higher temperature phenomena are shown in

Fig. 3. A family of peaks is shown which increase in both peak position and intensity as the polarization temperature increases. Such peaks were only noted for polarization temperatures above the glass transition and thus are attributed to space charge. In this case the space charge is thought to be composed of charges within the sample which become mobile above the glass transition temperature.

In order to confirm the quoted glass transition temperature, DSC studies were carried out and are shown in Fig. 4. A weak glass transition is observed at about 210K which is in good agreement with the literature. Fig. 4 also shows that the material melts at about 60°C.

B. $\text{PEO}_{4.5}:\text{KSCN}$

A typical data run for $\text{PEO}_{4.5}:\text{KSCN}$ is shown in Fig. 5. Three distinct features are seen. The complexity of this relaxation region has been reported previously in DR work on this material.⁹ In contrast to the situation for DR, quantitative results were obtained for two of the three features as peak cleaning techniques were used to isolate γ_1 and γ_3 .

The data and best fit curve for the lowest temperature peak, γ_1 , are shown in Fig. 6 and the activation parameters are also listed in Table I. In this case higher temperature relaxations were removed via selective polarization and thus the actual activation enthalpy will be slightly higher than the value listed in Table I. Also, the actual width will be slightly greater. The activation parameters predict that a 100 Hz DR peak should occur at about 104K and thus, as is obvious from Fig. 4c of Ref. 9, γ_1 corresponds to the lowest temperature relaxation observed dielectrically⁹ in the γ relaxation region for PEO complexed with KSCN.

Next, the data and best fit curve for the highest temperature peak, γ_3 , are shown in Fig. 7 and the activation parameters are listed in Table I. In this case, the peak cleaning eliminated the lower temperature relaxations and thus the actual activation energy will be slightly lower and the width slightly greater than the values listed in Table I. The activation parameters predict that a 100 Hz DR peak should be observed at about 211K. That temperature is significantly higher than the position of the highest temperature peak observed dielectrically in the γ relaxation region for PEO complexed with KSCN⁹ and thus the two peaks probably do not correspond. However, there is residual loss at 100 Hz in the vicinity of 211K for the DR data and it is suggested that γ_{33} is responsible for that loss even though the associated DR peak is not distinguishable.

Finally, quantitative results have not been obtained for γ_2 because both the high and low temperature sides of the peak require cleaning. Since no part of the relaxation would have been preserved in its entirety, it was decided not to carry out the procedure. However, even though quantitative results have not been obtained, it is clear that γ_2 corresponds to the middle peak observed dielectrically⁹ in the γ relaxation region for PEO complexed with KSCN. It may also be that this peak corresponds to the weak shoulder tentatively labeled γ_2 for uncomplexed PEO. Further work concerning this point is also necessary.

The greater complexity of the relaxation spectrum for PEO complexed with KSCN compared to that for PEO is attributed to distortions in the polymer chains due to the ions. In the DR paper,⁹ the authors discussed the origin of those relaxations in terms of the structural model where the ions were thought to reside within a helical tunnel.¹⁴⁻¹⁶ More recently,

Hibma has suggested that the ions actually reside outside the chains.¹⁷ In either case, however, severe distortions in the polymer chains exist and thus the origin of the complex relaxation spectrum can be understood since significant modification of potential wells of the responsible polar groups is expected. These comments hold whether the responsible polar groups are segments of the chain¹⁸ or chain end hydroxyl groups.¹²

Attempts were made to study the high temperature relaxation region in PEO complexed with KSCN. As expected, extremely large currents were observed. However, as the results were not reproducible, they are not included in the present paper.

Finally, DSC data for KSCN complexed PEO is shown in Fig. 4 both for as-prepared material and for the polymer after quench from 200°C. It is clear that there is very little resemblance between the traces for PEO_{4.5}:KSCN and that for PEO. In the as-prepared complexed material the dominant feature is a high temperature melting at about 80°C which is attributed to melting of the crystalline complexed phase. There is no evidence of the 60°C melting of pure crystalline PEO in the complexed material. There are small endothermic events at about 150°C and 180°C which are due to trace amounts of salt. The quenched material exhibits a glass transition at about -10°C with an exothermic recrystallization of the complexed crystalline phase followed by its melting again at 80°C. It is interesting that the recrystallization of the KSCN complexed PEO occurs at about the melting point of PEO. While this could indicate a strong interaction between PEO and KSCN, it may be that the result is fortuitous. The small features at 150°C and 180°C are also observed.

V. SUMMARY

In summary, then, a digitally controlled TSDC facility is described and data are presented for PEO and PEO complexed with KSCN. For the PEO, the existence of a previously reported relaxation is confirmed and the relaxation is attributed to defects in the crystalline phase. Two relaxations are found in the γ relaxation region. In addition, space charge relaxations are reported. For the complexed material, three relaxations are observed and the relationship between these relaxations and previously reported DR results is established and possible origins of the relaxations are discussed. Finally, DSC studies are reported for all of the materials.

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TABLE I. Activation parameters for the relaxations observed in "pure" PEO and PEO complexed with various salts.

Relaxation	Material	$\tau_0(10^{-14}s)$	E(eV)	σ (eV)
α_c	PEO	41	0.49	0.023
(α_c)	PEO _{6.5} Ba(SCN) ₂ :3H ₂ O	36 ^a	0.48 ^a	
γ_1	PEO _{4.5} :KSCN	0.6	0.235	0.039
γ	PEO	6.0 ^a	0.33 ^a	
γ_3	PEO _{4.5} :KSCN	20	0.415	0.025

^aDR studies of reference 10.

FIGURE CAPTIONS

Figure 1. Low temperature TSDC spectrum for PEO. The polarization temperature was 163K and the applied potential was 300 V for 10 minutes. The sweep rate was 6.13 K/min.

Figure 2. Data after peak-cleaning and best fit curve for the α_c relaxation in PEO. The polarization temperature was 163K and the applied potential was 300 V for 10 minutes. The sweep rate was 6K/min.

Figure 3. High temperature TSDC spectrum for PEO. 300 V were applied for 10 minutes in all cases. The sweep rate was 6.13 K/min. The polarization temperatures are indicated on the drawing.

Figure 4. DSC traces for PEO (chain link line) and PEO complexed with KSCN before (solid line) and after (dashed line) quench from 200°C. The sweep rate was 10 K/min. The inset shows the PEO data with the ordinate expanded by a factor of 5.

Figure 5. TSDC spectrum for $\text{PEO}_{4.5}:\text{KSCN}$. The polarization temperature was 133.2K and the applied potential was 300 V for 10 minutes. The sweep rate was 6 K/min.

Figure 6. Data after peak-cleaning and best-fit curve for the γ_1 relaxation in $\text{PEO}_{4.5}:\text{KSCN}$. The polarization temperature was 78K and the

applied potential was 400 V for 8 minutes. The sweep rate was 6 K/min.

Figure 7. Data after peak-cleaning and best-fit curve for the γ_3 relaxation in $\text{PEO}_{4.5}:\text{KSCN}$. The polarization temperature was 133K and the applied potential was 300 V for 10 minutes. The sweep rate was 6 K/min.

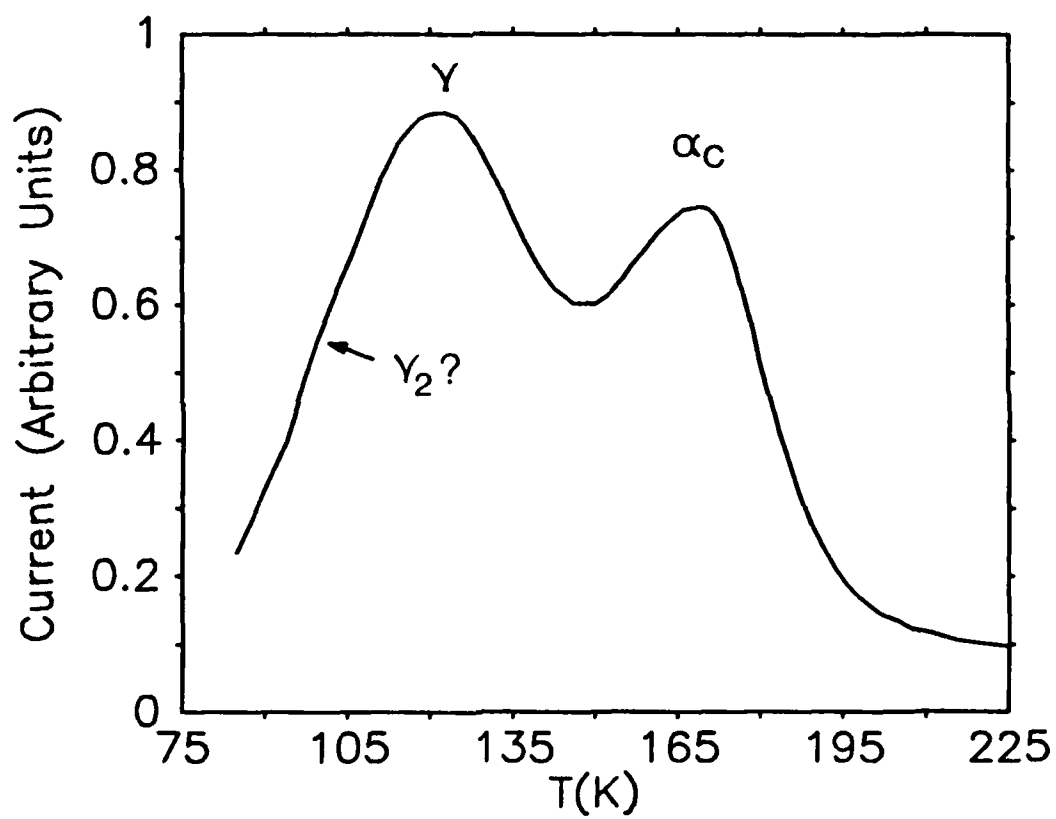
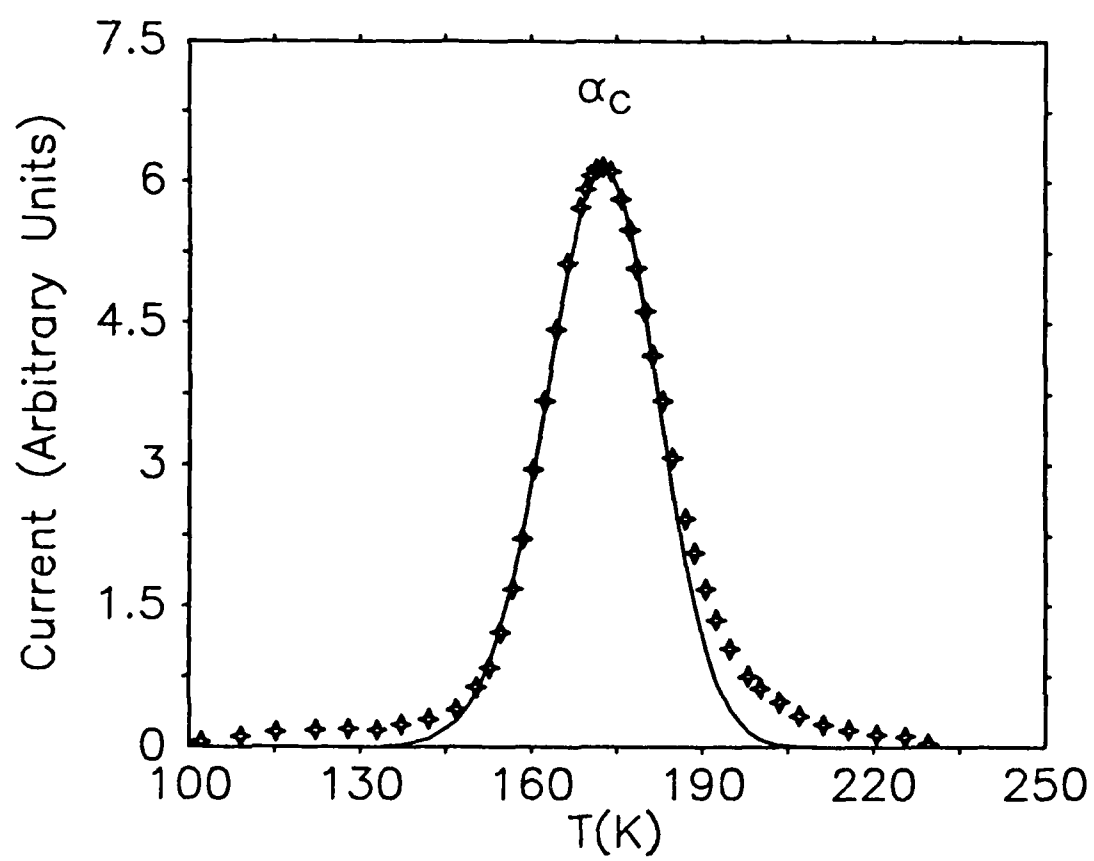
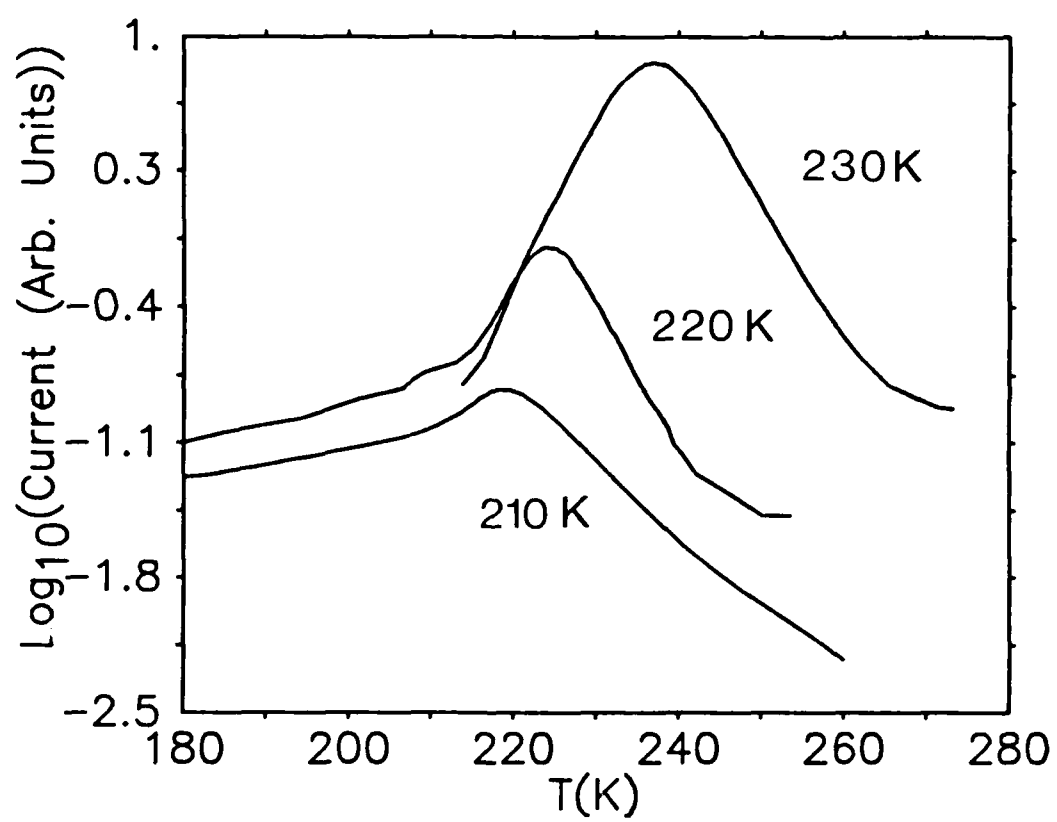
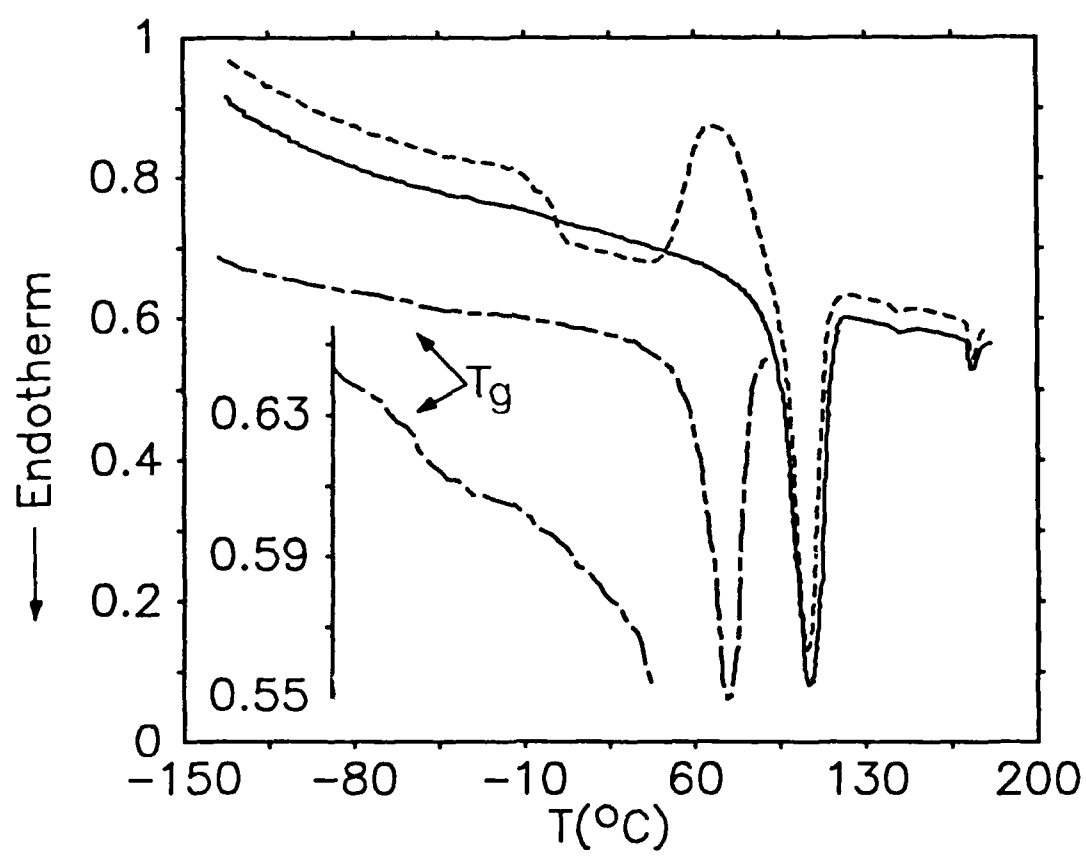


Fig. 1







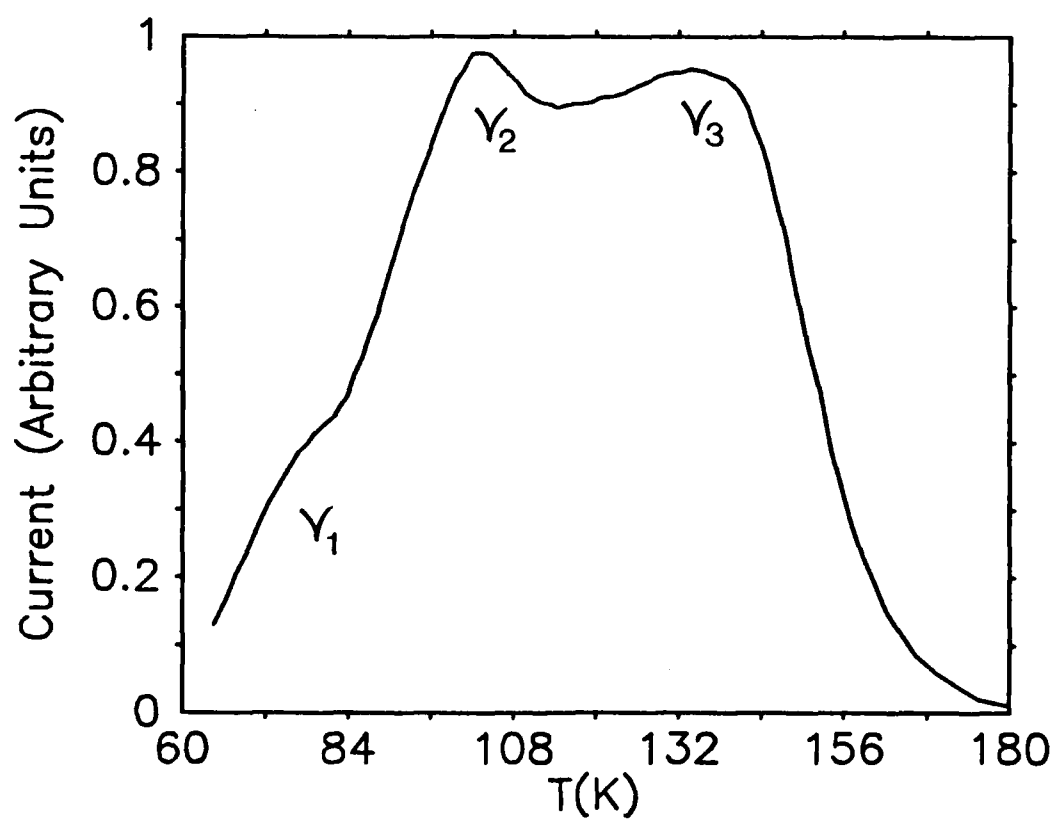
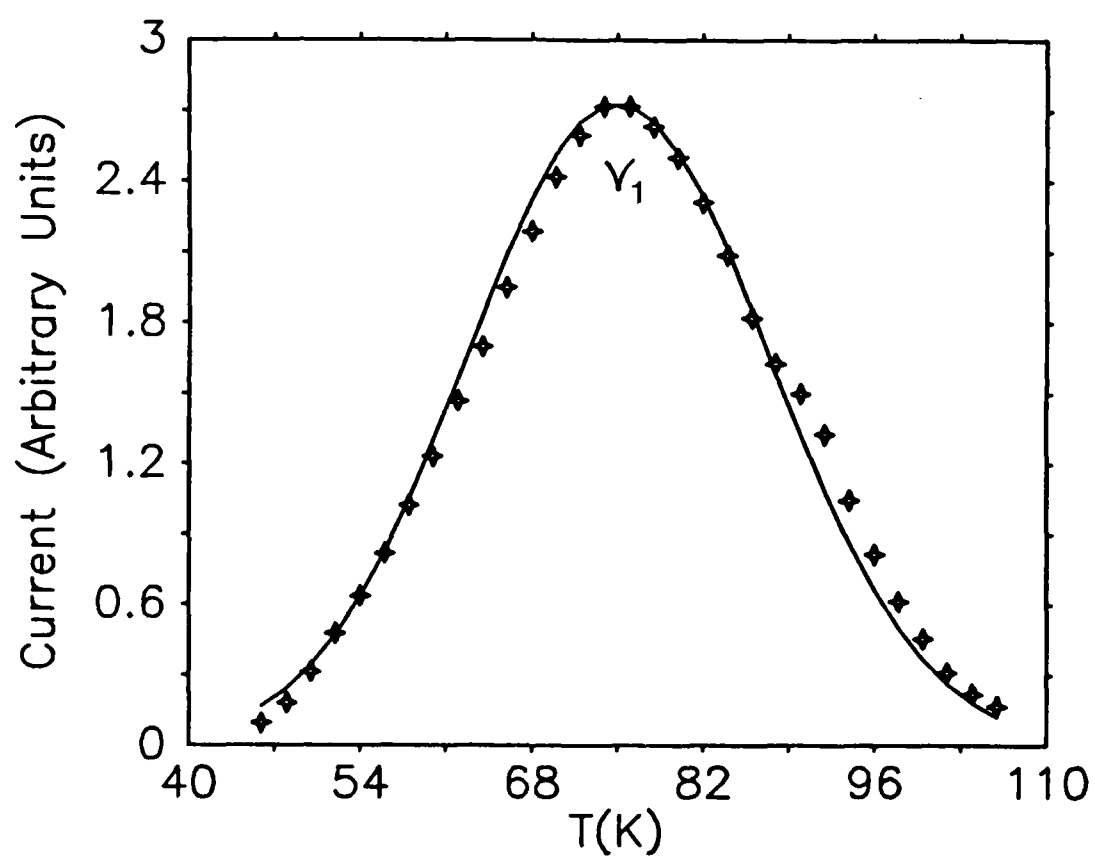
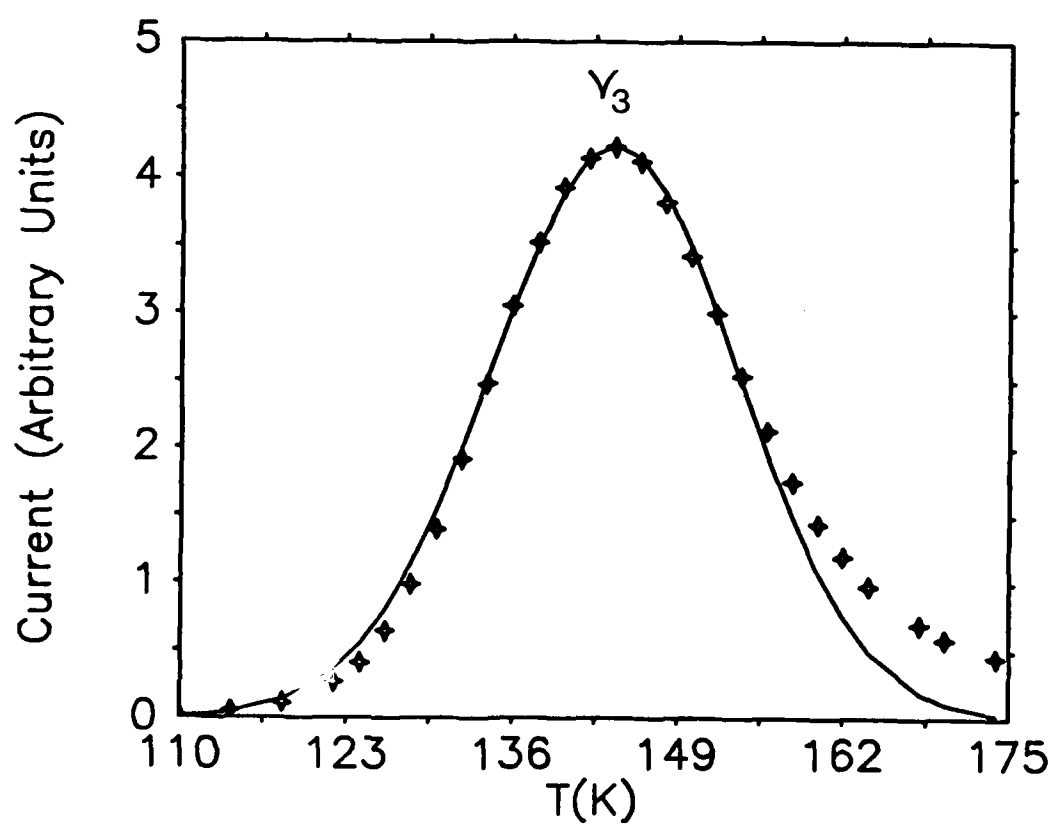


Fig. 5





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