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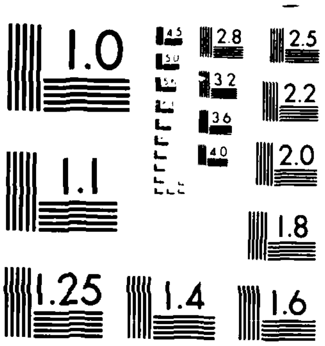

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ION AND POLYMER CHAIN MOTION IN A SUPERIONIC SODIUM-POLY(ETHYLENE OXIDE) COMPLEX

by

S.G. Greenbaum and A.N. Shetty

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Magnetic resonance measurements have been performed on the ion conducting complex poly(ethylene oxide)$_{4.5}$NaClO$_4$. Low temperature ^{23}Na NMR spectra suggest a highly symmetric environment for the Na-ions as evidenced by the absence of quadrupole broadening. Proton spin-lattice relaxation measurements provide an estimate of 4×10^{-10} sec for the polymer chain motional correlation time at T=69C. Correlation times of tumbling paramagnetic probe molecule have been extracted from EPR spectra of ^{15}N-enriched TANOL-doped complex. Changes in polymer chain mobility above T=120C are inferred from the results and may be consistent with previous scanning calorimetry measurements.

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Magnetic resonance measurements have been performed on the ion conducting complex poly(ethylene oxide)$_{4.5}$NaClO . Low temperature ^{23}Na NMR spectra suggest a highly symmetric environment for the Na-ions as evidenced by the absence of quadrupole broadening. Proton spin-lattice relaxation measurements provide an estimate of $\sim 4 \times 10^{-10}$ sec for the polymer chain motional correlation time at T = 69C. Correlation times of tumbling paramagnetic probe molecule have been extracted from EPR spectra of ^{15}N-enriched TANOL-doped complex. Changes in polymer chain mobility above T = 120C are inferred from the results and may be consistent with previous scanning calorimetry measurements.

The discovery of ion conducting complexes formed between alkali salts and poly(ethylene oxide) (PEO) by Wright and co-workers,(1-3) and subsequent investigations by Armand and co-workers (4,5) have generated a great deal of interest in this new class of materials. Because PEO and related complexes offer considerable promise as electrolytes in potentially superior solid state batteries, much of the research to date has focused on their ion transport properties (6-10).

In this paper we present magnetic resonance results for $PEO_{4.5}$ $NaClO_4$ with particular emphasis on addressing the natures of both ionic and polymer chain motion in complex. Previous nuclear magnetic resonance (NMR) studies of lithium-salt association complexes with PEO (11) and related polymers (12,13) demonstrate a feature common to superionic solids - motional narrowing of ^7Li NMR lines in the temperature region of enhanced conductivity. As will be discussed, similar results are obtained for ^{23}Na in the present study. An important comment to be made regarding this observation is that the NMR measurements are also sensitive to local motional processes which do not necessarily contribute to the long range ion transport mechanisms that determine the material's conductivity.

Proton NMR can shed light on the dynamics of the polyether chains just as ^7Li or ^{23}Na NMR can for alkali ion motion. Another technique for probing molecular chain dynamics involves the use of paramagnetic impurity molecules such as 4-hydroxy-2,2,6,6-tetramethylpiperidine-1- N-1-oxyl(TANOL), in which tumbling correlation times of the impurities are extracted from electron paramagnetic resonance (EPR) spectra at various temperatures.(14) ^1H NMR and ^{15}N-enriched TANOL EPR data have also been obtained for $PEO_{4.5}NaClO_4$.

The samples were prepared by dissolving the appropriate amounts of PEO (MW-5x10^6; Aldrich) and $NaClO_4$ (Aldrich) in methanol. The resulting solution was cast onto a Teflon plate and slowly evaporated at 40C. The films were then packed into sealed evacuated tubes for the resonance measurements, after being treated in a vacuum oven at 80C for 24 hours to drive off residual solvent or water. For the EPR measurements 0.05 weight percent perdeuterated ^{15}N-enriched (99.8%) TANOL (obtained from MSD Isotopes) was added to the solution prior to evaporation. NMR spectra and relaxation times were obtained with a JEOL GX-400 spectrometer, and an IBM ER200 X-band spectrometer was utilized in conjunction with a Varian 4540 temperature controller for the EPR measurements.

← Fig 1

The ^{23}Na NMR lineshape for $PEO_{4.5}NaClO_4$ at a resonance frequency of 105.8 MHz and T=-85C is shown in Fig. 1. From linewidth and $\pi/2$ pulse-width considerations it can be concluded that there is no appreciable quadrupole broadening present. The absence of an observable quadrupole interaction indicates a highly symmetric environment for the rigid Na ions. Arguments based on conductometric studies of PEO ·KSCN complex in methanol (15) and ^{23}Na NMR chemical shifts in Na-containing complexes (in solution) (16) suggest a tetrahedral configuration in which the cation is fourfold coordinated by the ether oxygens. (17) Although this model is consistent with the solid state measurements reported in this work, it is important to note that Berthier and co-workers have observed a quadrupole broadened ^{23}Na resonance line in $PEO_{10}NaI$. (18) These findings suggest a sensitivity of cation coordination to the stoichiometry of the complex.

← Fig 2

The temperature dependence of the ^{23}Na linewidth (full-width at half-maximum, υ = 105.8 MHz) is shown in Fig. 2. It is clear from that data that the "rigid-line" region corresponds to temperatures below -50C. At higher temperatures motional narrowing occurs, the linewidth decreasing monotonically with increasing temperature up to the highest temperature measured (95C). Motional narrowing of ^{7}Li NMR spectra has been reported for several Li salt-polymer complexes, (11-13) although the onset of the narrowing generally does not occur until -15C or higher. For spin I=1/2 nuclei the narrowing process arises from motional averaging of the local field at the nuclear site due to the magnetic dipole-dipole interaction. Motional narrowing of quadrupole broadened spectra (for I>1) results principally from averaging of the electric field gradient (efg) at the nuclear site. The absence of a static efg deduced from the low temperature ^{23}Na spectrum in Fig.1 suggests that efg fluctuations do not contribute to line-narrowing, thus yielding essentially the same result as for I=1/2 nuclei. It is possible, however, that additional insight into the ion hopping mechanism could be gained through ^{23}Na spin-lattice relaxation (T_1) measurements, due to the sensitivity of T_1 to efg fluctuations.(19)

Proton NMR spectra of PEO-alkali complexes
ıre generally composed of two distinct features:
ı relatively narrow lorentzian component
superimposed on a broad gaussian component.
(11,18,20) The broad component is identified
vith the relatively rigid CH_2 segments in the
:rystalline phase of the complex while the
ıarrow line arises from mobile CH_2 segments in
:he elastometric phase. Proton T_1 data over the
:emperature range 0-100C is displayed in Fig.3.
'he bandwidth of the spectrometer employed in
he measurements was limited to ∿30 kHz by a
ombination of available rf power and digitizer
peed designed for a nominally high-resolution
nstrument. Consequently ^1H spectra whose
inewidths approached or exceeded this limit
below 0°C) were not utilized. The T_1 recovery
rofiles were found to be single-valued
xponential with no resolvable differences
etween the broad and narrow components. From
he local T_1 mininum occurring at T∿69C a
olymer-chain motional correlation time τ =
x10^{-10}s (at 69C) can be inferred (assuming that
$\tau = 1$ at the T_1 minimum, where ω is the NMR
requency).

← Fig 3

In order to obtain additional information
egarding the chain dynamics, EPR linewidth
easurements of TANOL-doped $PEO_{4.5}NaClO_4$ were
mployed. It is important to note that because
he probe molecules are not covalently bonded
o the polymer chains, their rotational behavior
rovides only a qualitative measure of motion
f the host polymer matrix in which the probes
re imbedded. An interesting feature of the
PR spectra is that they do not reflect the
oexistence of both crystalline (expected to
ield "slow-tumbling") and elastometric
fast-tumbling) phases. A possible explanation
or this may be related to different
olubilities of TANOL in each phase that would
end to favor the segregation of the probe
olecules in the elastomeric phase.

In the "fast-tumbling" region ($\lesssim 10^{-9}$ s) the
^{5}N EPR hyperfine component linewidths are
elated to the rotational correlation time τ of
ıe probe molecule in a simple manner (14,21).
ıe EPR first-derivative spectra are symmetric
ith respect to the baseline in this region.
ɔr $PEO_{4.5}NaClO_4$, the onset of fast-tumbling
:curs at 72C, above which Kivelson's forma-
lsm (22) for computing τ is applicable.
ıe temperature dependence of the nitroxide
ımbling correlation time is shown in Fig.4.

← Fig 4

The probe tumbling behavior appears to be characterized by two activation energies, 0.46 ± .05 eV for $T \leq 120$ C, and 0.13 ± .08 eV above this temperature. Differential scanning calorimetry (DSC) results for $PEO_{4.5}NaClO_4$ reveal the presence of a sharp thermal event in the vicinity of T = 150C (23). The sharpness of the DSC feature suggests melting of the crystalline complex at $T \sim 150$ C, although progressive dissolution of the crystalline phase in the elastometric phase cannot be ruled out, as indicated by similar results obtained for $PEO_8LiCF_3SO_3$ (18). In either case it appears as though the abrupt drop in nitroxide probe rotational activation energy should be related to increased chain mobility above ~120C which is also consistent with the DSC result. It is interesting to note that the probe tumbling correlation time at $T \sim 69$C is about $7X10^{-10}$ s, which is close to the NMR-determined τ for the polymer chains ($\sim 4X10^{-10}$ s) at that temperature.

Additional measurements (including ^{23}Na T_1) as a function of Na concentration are currently in progress.

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Fig. 1. ^{23}Na NMR absorption lineshape at $T = -85C$, $\nu_0 = 105.8$ MHz in $PEO_{4.5}NaClO_4$.

Fig. 2. ^{23}Na full-width at half-maximum vs. temperature (C) in $PEO_{4.5}NaClO_4$.

Fig. 3. ^{1}H spin-lattice relaxation time (T_1) vs. reciprocal temperature in $PEO_{4.5}NaClO_4$.

Fig. 4. Temperature dependence of correlation time for nitroxide radical tumbling in $PEO_{4.5}NaClO_4$ doped with 0.05% ^{15}N-enriched TANOL.

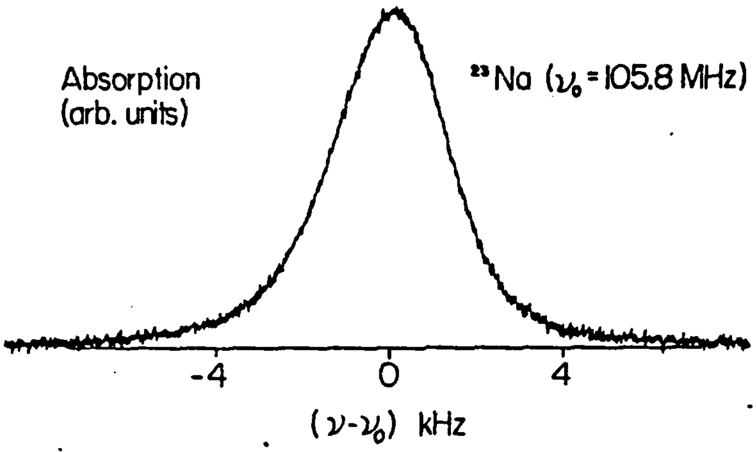

Figure 1

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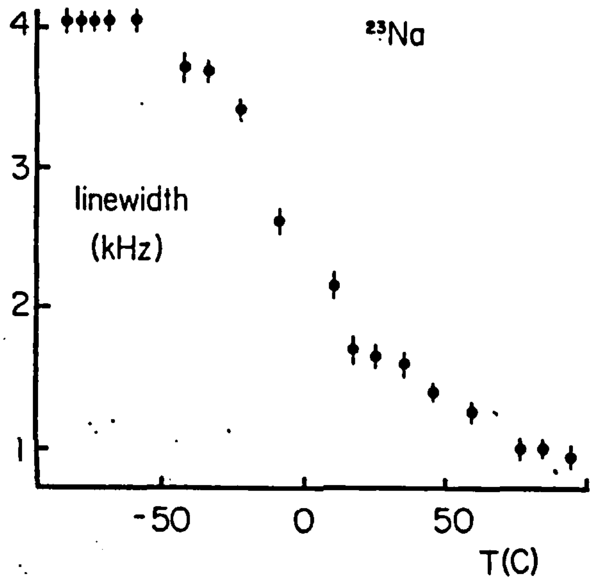

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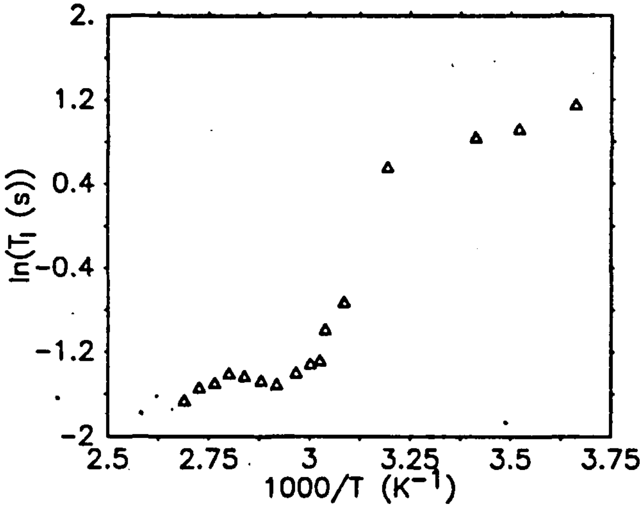

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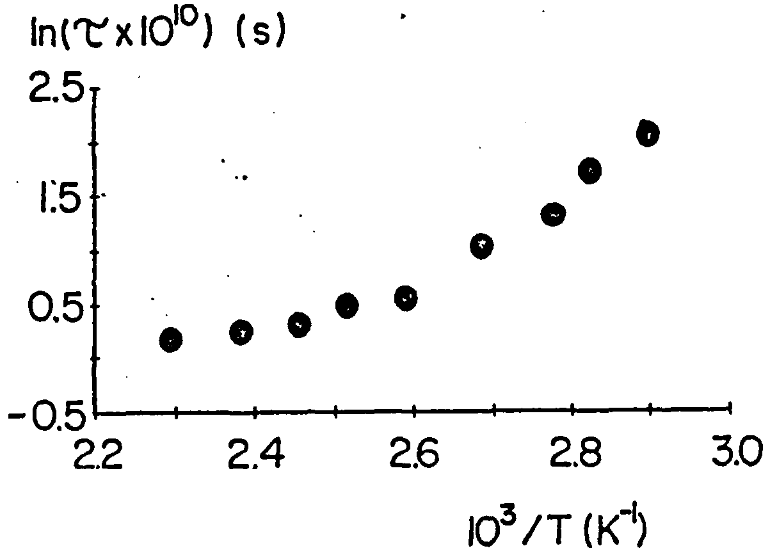

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