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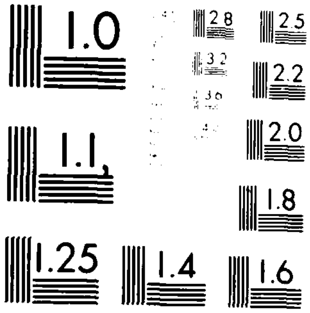
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TECHNICAL REPORT NO. 1

Ionic Conductivity in Lithium Hexaoxometallate Solid Solutions

By

E. Nomura and M. Greenblatt

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IONIC CONDUCTIVITY IN LITHIUM HEXAOXOMETALLATE SOLID SOLUTIONS

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ABSTRACT

Lithium ion conductivity in solid solutions formed between Li_7TaO_6, $Li_7M^VO_6$ (M^V = Nb, Bi) and Li_8ZrO_6 has been measured as a function of temperature and composition using complex impedance method. The samples prepared were $Li_7Ta_{1-x}Nb_xO_6$, $Li_7Ta_{1-x}Bi_xO_6$ and $Li_{7+x}Ta_{1-x}Zr_xO_6$. At 200°C, the conductivities of $Li_7Ta_{0.7}Nb_{0.3}O_6$, $Li_7Ta_{0.6}Bi_{0.4}O_6$ and $Li_{7.4}Ta_{0.6}Zr_{0.4}O_6$ are 4.3×10^{-4} $(\Omega cm)^{-1}$, 3.0×10^{-4} $(\Omega cm)^{-1}$ and 4.0×10^{-4} $(\Omega cm)^{-1}$, respectively.

INTRODUCTION

There is considerable interest in developing solid lithium ion conductors for utilization in high energy density battery systems. This has led to a search for new solid electrolytes exhibiting high lithium ion conductivity and has stimulated interest in developing a fundamental understanding of ionic transport in solids(1-3).

The compounds of lithium hexaoxometallates, formulated as Li_nMO_6; n = 6,7 or 8, M = IV, V or VI group element, have a pseudo two dimensional structure and high lithium ion conductivity(4). The structure of Li_nMO_6 is characterized by octahedral sheets of CdI_2-type, between which 6 Li^+ are inserted in a tetrahedral environment as

$$Li_6^{tetr}\ (Li_aM_b\square_c)^{oct}\ O_6$$

$$a+b+c = 3$$

$\square$: vacancy

Among the lithium hexaoxometallates, Li_7TaO_6 (i.e. $Li_6(LiTa\square)O_6$) is the best lithium conductor with $\sigma_{200} = 5 \times 10^{-4}$ $(\Omega cm)^{-1}$ and $\sigma_{RT} = 4.3 \times 10^{-8}$ $(\Omega cm)^{-1}$(4).

It has been shown that the activation energy for Li^+ diffusion in the tetrahedral sites between the layers is significantly larger than that for the octahedral lithium ion in the layers$^{(5)}$. Thus by altering the lithium content and/or the vacant sites in the layers higher ionic conductivities might be achieved. We have examined the effect of increaed Li^+ content by partial substitution of tantalum ions with Zr^{4+} to yield $Li_6(Li_{1+x}Ta_{1-x}Zr_x \square_{1-x})O_6$. We have also attempted to look at the effect of decreased Li^+ content (or increased vacancies) in the layers by studying the conductivities of samples with substituted divalent cations (e.g. $Li_6(Li_{1-2x}Mg_xTa \square_{1+x})O_6$), however our results are inconclusive on this system due to difficulties in obtaining single phase compounds.

Alternatively we hoped to obtain higher lithium conductivities in this system by partial substitution of the tantalum ions with other group V metal ions of larger ionic radii in order to optimize the channel size of the framework structure for lithium diffusion. In this paper we report the results of the lithium conductivity in: $Li_7Ta_{1-x}Nb_xO_6$, $Li_7Ta_{1-x}Bi_xO_6$, $Li_{7+x}Ta_{1-x}Zr_xO_6$.

EXPERIMENTAL

Li_2O was obtained by thermal decomposition of anhydrous Li_2O_2 in vacuum at 450°C for 6 hours. Other starting materials were reagent grade Nb_2O_5, Ta_2O_5, Bi_2O_3 and ZrO_2. Mixtures of appropriate composition were throughly mixed using agate mortar in a He dry box. For example, $Li_7Ta_{1-x}Nb_xO_6$ and $Li_7Ta_{1-x}Bi_xO_6$ were prepared from Li_2O, Ta_2O_5 and Nb_2O_5, and Li_2O, Ta_2O_5 and Bi_2O_3, respectively, according to the following equation

$$7Li_2O + (1-x)Ta_2O_5 + xNb_2O_5 \rightarrow 2Li_7Ta_{1-x}Nb_xO_6$$

$$7Li_2O + (1-x)Ta_2O_5 + xBi_2O_3 + xO_2 \rightarrow 2Li_7Ta_{1-x}Bi_xO_6{}^{(6)}.$$

The mixtures were pressed into cylindrical pellets 3/8" diameter and about 0.2" thick at 15 kpsi. The pressed pellets were transferred to high purity alumina crucibles, were embedded in excess Li_2O powder in order to prevent Li_2O losses, and were heated at 650°C for 18 hours in air. After cooling, the pellets were crushed and examined by X-ray powder diffraction using a Norelco diffractometer with Ni filtered copper radiation. Lithium content was determined by atomic absorption spectrophotometric methods.

Pellet samples for ionic conductivity measurements were prepared by pressing to 1/4" diameter and about 0.15" thickness at 30 kpsi followed by sintering at 950°C for 18 hours in air and quenching in air. Again, Li_2O loss was minimized by covering the pellets with Li_2O powder during the sintering process. The X-ray diffraction pattern and lithium content of the sintered samples were checked to confirm the identity and composition of the phases present. Weight loss was not observed after sintering. The density of the sintered pellets were about 85% of theoretical value. Both surfaces of the pellets were polished using silicon carbide (#400) paper and sputtered with about 1μm of gold followed by a coating of silver paint (Engelhard #16).

The device for the conductivity measurement is shown in Fig. 1. Platinum contact leads were made by spot welding pieces of platinum foil of 10 x 10 mm^2 to platinum wires. Two discs of α-alumina of 10 mm diameter and 3 mm thickness were used for insulating the materials. Contact between the sample electrode (sputtered gold and painted silver) and the platinum lead was made

Fig. 1
Device for conductivity measurement

1. sample Pellet 3. platinum lead
2. α-alumina disc 4. stainless steel disc
5. screw

Fig. 2
Circuit diagram for AC conductivity measurement

A. tuned amplifier PS. phase shifter
M. mixer I. integrator
R_x. sample R_{std}. standard resistance

by tightening the screw (Fig. 1). The AC conductivity measurements were made using ionically blocking electrodes. The schematic electrical circuit diagram of the AC conductivity measurement is shown in Fig. 2. Lock-in amplifier (PAR Model 128A) was used as a phase sensitive detector and a Hewlett-Packard Model 200 CDR was used as a signal generator. The frequency range used was between 5 HZ and 100 KHZ. The measurement temperature was from room temperature to 250°C in air.

RESULTS AND DISCUSSIONS

Li_7TaO_6

The ionic conductivity of $\sigma_{200} \equiv 5 \times 10^{-4}$ $(\Omega \text{ cm})^{-1}$ and activation energy of 0.66 eV of Li_7TaO_6 have been measured by C. Delmas et al(4). We have also measured the conductivity of sintered pellets of Li_7TaO_6. Fig. 3 shows the conductivity, σ versus $^1/_T$ of Li_7TaO_6. Different activation energies corresponding to a lower and higher temperature regions are observed. In the higher temperature region, the deviation of ionic conductivity between different pellets is small (which is also a measure of the reliability of the data) and the activation energy is 0.67 eV. In the lower temperature region, the ionic conductivity of different pellets differ more significantly but the activation energies are the same (0.46 eV). The change in the slope of the σ vs $^1/_T$ plot of Li_7TaO_6 probably corresponds to a transition from extrinsic to intrinsic conductivity regions; then the variations in the ionic conductivities of different samples in the lower temperature region can be explained by differences in defect concentrations or particle packing from sample to sample.

Fig. 3

The temperature dependence of
the conductivity of Li_7TaO_6

$Li_7Ta_{1-x}M^V_xO_6$(M^V=Nb,Bi)

The X-ray diffraction patterns of the solid solutions prepared were identical to that of Li_7TaO_6 except for the shifts in 2Θ. The lattice parameters calculated for a hexagonal unit cell increased with increasing values of x for $Li_7Ta_{1-x}Nb_xO_6$ and for $Li_7Ta_{1-x}Bi_xO_6$; a = 5.42Å, c = 15.17Å for $Li_7Ta_{0.6}Nb_{0.4}O_6$ and a = 5.44Å c = 15.23Å for $Li_7Ta_{0.6}Bi_{0.4}O_6$. The lattice parameters of Li_7TaO_6, Li_7NbO_6 and Li_7BiO_6 are a = 5.39Å; c = 15.11Å; a = 5.40Å; c = 15.12Å; and a =5.50Å, c = 15.45Å, respectively(7). It is clear that the lattice parameters of $Li_7Ta_{0.6}Nb_{0.4}O_6$ are larger than those of either of the component compounds, Li_7TaO_6 and Li_7NbO_6, while the lattice parameters of $Li_7Ta_{0.6}Bi_{0.4}O_6$ are intermediate between those of Li_7TaO_6 and Li_7BiO_6. The variation of lattice parameters with x in $Li_7Ta_{1-x}Nb_xO_6$ and $Li_7Ta_{1-x}Bi_xO_6$ is shown in Fig. 4. The bismuth substituted phases appear to obey Vagard's law while the $Li_7Ta_{1-x}Nb_xO_6$ system does not. This suggests that there might be a new phase close to the $Li_7Ta_{0.6}Nb_{0.4}O_6$ composition.

The ionic conductivities of $Li_7Ta_{1-x}Nb_xO_6$ are shown in Fig. 5. The ionic conductivities increase from 1.1 x 10^{-4} $(\Omega\ cm)^{-1}$ for x = 0.0 to 4.3 x 10^{-4} $(\Omega\ cm)^{-1}$ for x = 0.3 at 200°C and decrease as x is increased further (Fig. 5). The activation energy of conduction is 0.63 eV for $Li_7Ta_{0.7}Nb_{0.3}O_6$, somewhat smaller than the 0.67 eV observed for Li_7TaO_6.

The ionic conductivities of $Li_7Ta_{1-x}Bi_xO_6$ phases are shown in Fig. 6. The ionic conductivities increase to 3.0 x 10^{-4} $(\Omega cm)^{-1}$ at 200°C at x = 0.4. The activation energy for conduction is 0.60 eV in $Li_7Ta_{0.7}Bi_{0.3}O_6$.

Fig. 4
Lattice parameters of
$Li_7Ta_{1-x}Nb_xO_6$(O) and
$Li_7Ta_{1-x}Bi_xO_6$ (O)

Fig. 5

The temperature dependence of the conductivity of $Li_7Ta_{1-x}Nb_xO_6$ phases

Fig. 6

The temperature dependence of the conductivity of $Li_7Ta_{1-x}Bi_xO_6$ phases

In the $Li_7Ta_{1-x}Nb_xO_6$ and $Li_7Ta_{1-x}Bi_xO_6$ phases the observed increase in ionic conductivity and decrease in activation energy at $x = 0.3$ and 0.4 respectively compared to Li_7TaO_6 is clearly due to optimizing the channel size for lithium diffusion at these compositions.

$Li_{7+x}Ta_{1-x}Zr_xO_6$

The powder X-ray diffraction pattern of $Li_{7+x}Ta_{1-x}Zr_xO_6$ is similar to that of Li_7TaO_6. The lattice parameters increased with increasing values of x; a = 5.44Å and c = 15.25Å were found for $Li_{7.4}Ta_{0.6}Zr_{0.4}O_6$.

The ionic conductivities of $Li_{7+x}Ta_{1-x}Zr_xO_6$ are shown in Fig. 7. The change in the slope of σ vs $^1/_T$ curves of $Li_{7.1}Ta_{0.9}Zr_{0.1}O_6$ corresponds most likely again to the transition from extrinsic to intrinsic mechanism of conductivity. The activation energies are 0.50 eV and 0.60 eV in the lower and higher temperature regions respectively. A similar change in the slope of the σ vs $^1/_T$ plot of $Li_{7.3}Ta_{0.7}Zr_{0.3}O_6$ is observed at a higher temperature, suggesting that the increased conductivity observed in the $Li_{7+x}Ta_{1-x}Zr_xO_6$ phases is due to the larger concentration of mobile Li^+ ions. $Li_{7.4}Ta_{0.6}Zr_{0.4}O_6$ is in the region of extrinsic conductivity for the entire range of temperature measured.

In Table 1, we summarize conductivity data for the best Li solid electrolytes. It may be concluded that solid solutions of lithium hexaoxometallates are also members of good lithium ion conducting solid electrolytes.

Fig. 7
The temperature dependence of conducitivity of $Li_{7+x}Ta_{1-x}Zr_xO_6$ phases

Table 1 Conductivity data for Li compounds.

Compounds	σ_{RT} $(\Omega cm)^{-1}$	σ_{200} $(\Omega cm)^{-1}$	Activation Energy (eV)	Temp. Range (°C)	Ref.
Li-β-Al_2O_3	1.3×10^{-4}	2.0×10^{-3}	0.19	-100 - 180	8
			0.36	180 - 800	
$Li_{14}Zn(GeO_4)_4$ (LISICON)		1.7×10^{-4}	0.50	50 - 300	9
$Li_4B_7O_{12}Cl_{0.68}Br_{0.32}$	$5.4 \times 10^{-7*}$	6.5×10^{-4}	0.53	50 - 230	1
$Li_{3.75}Si_{0.75}P_{0.25}O_4$	$4.8 \times 10^{-7*}$	1.0×10^{-3}	0.60	30 - 230	1
$Li_{4.4}Si_{0.6}Al_{0.4}O_4$	$2.8 \times 10^{-7*}$	7.6×10^{-4}	0.58	70 - 230	1
$Li_{4.6}Al_{0.6}Si_{0.4}O_4$		7.7×10^{-5}	0.68	85 - 230	1
$Li_7Ta_{0.7}Nb_{0.3}O_6$	$7.3 \times 10^{-8*}$	4.3×10^{-4}	0.63	80 - 230	this work
$Li_7Ta_{0.6}Bi_{0.4}O_6$	$7.3 \times 10^{-8*}$	3.0×10^{-4}	0.60	50 - 230	this work
$Li_{7.4}Ta_{0.6}Zr_{0.4}O_6$	$3.4 \times 10^{-7*}$	4.0×10^{-4}	0.52	50 - 230	this work

* extrapolated value

ACKNOWLEDGEMENT

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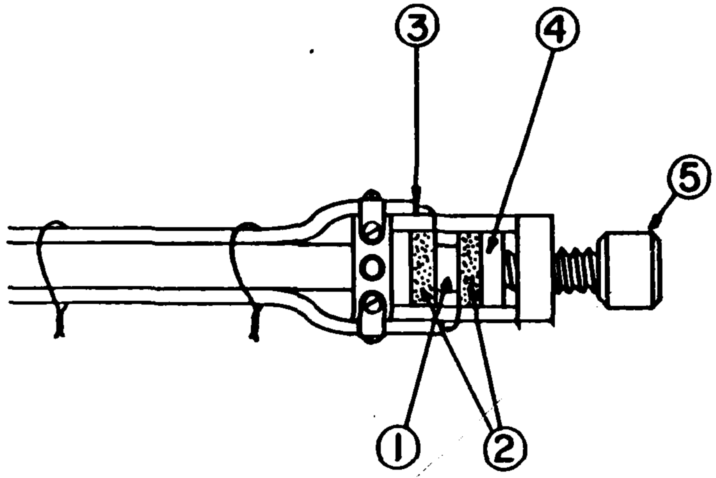
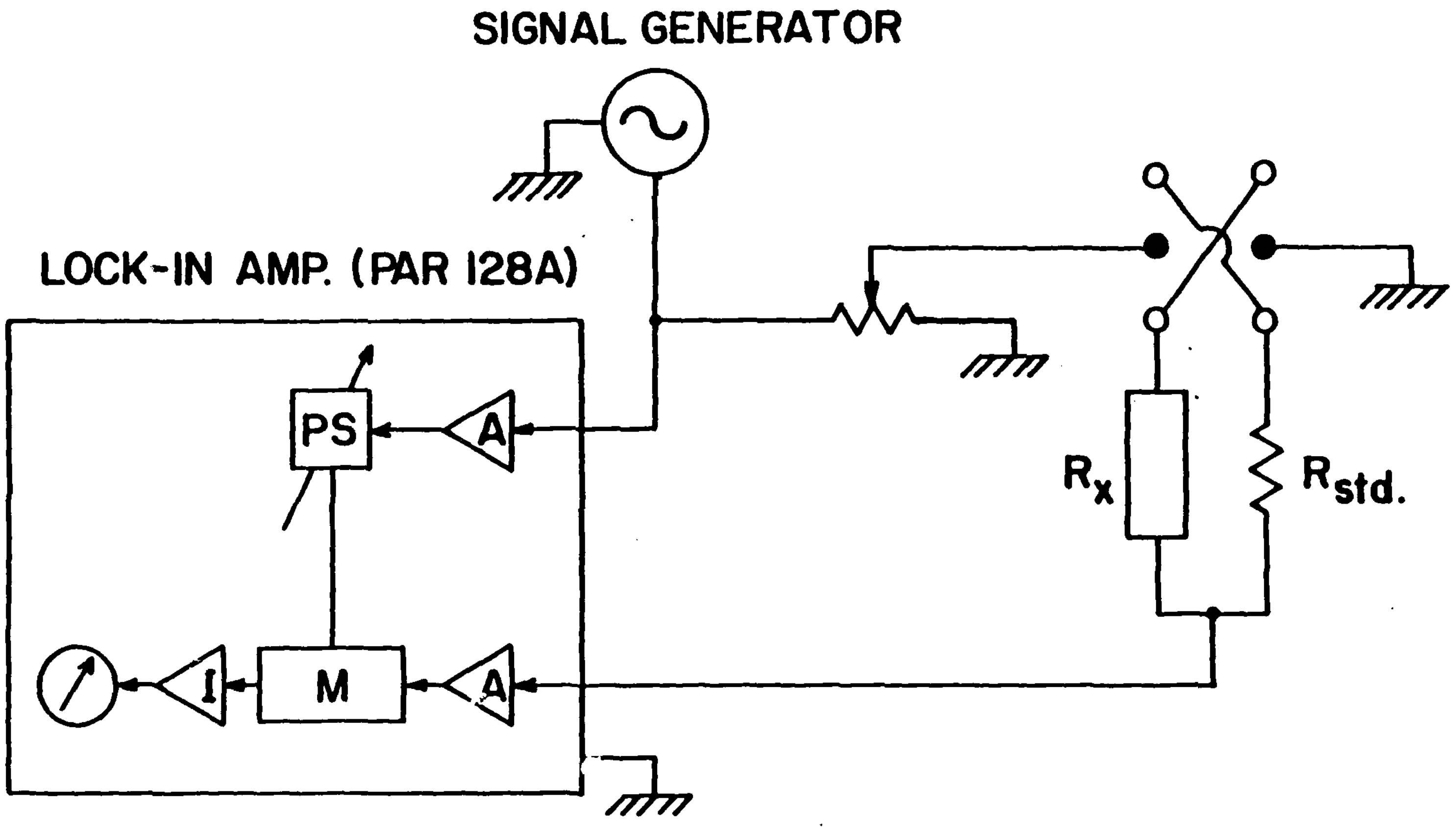


FIG 1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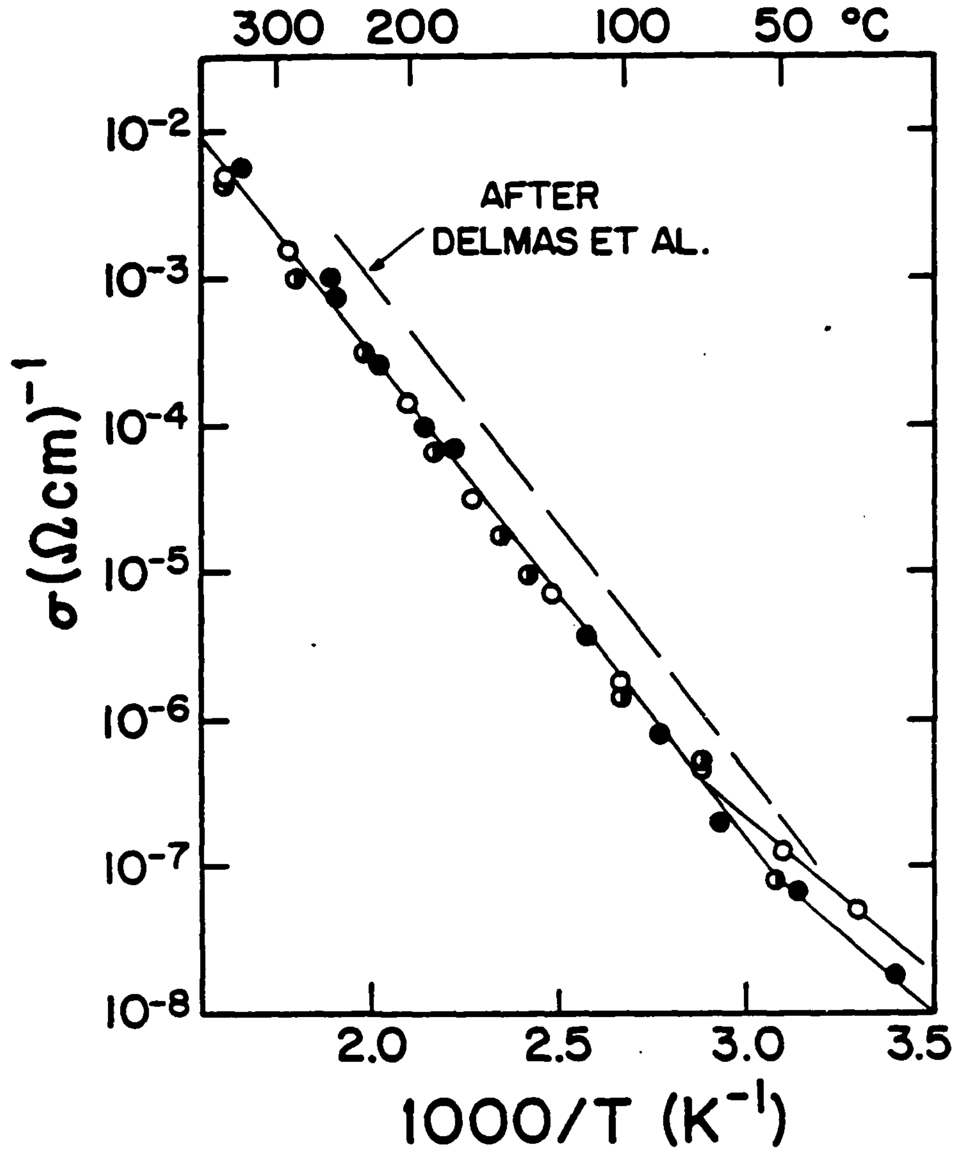
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FIG. 3

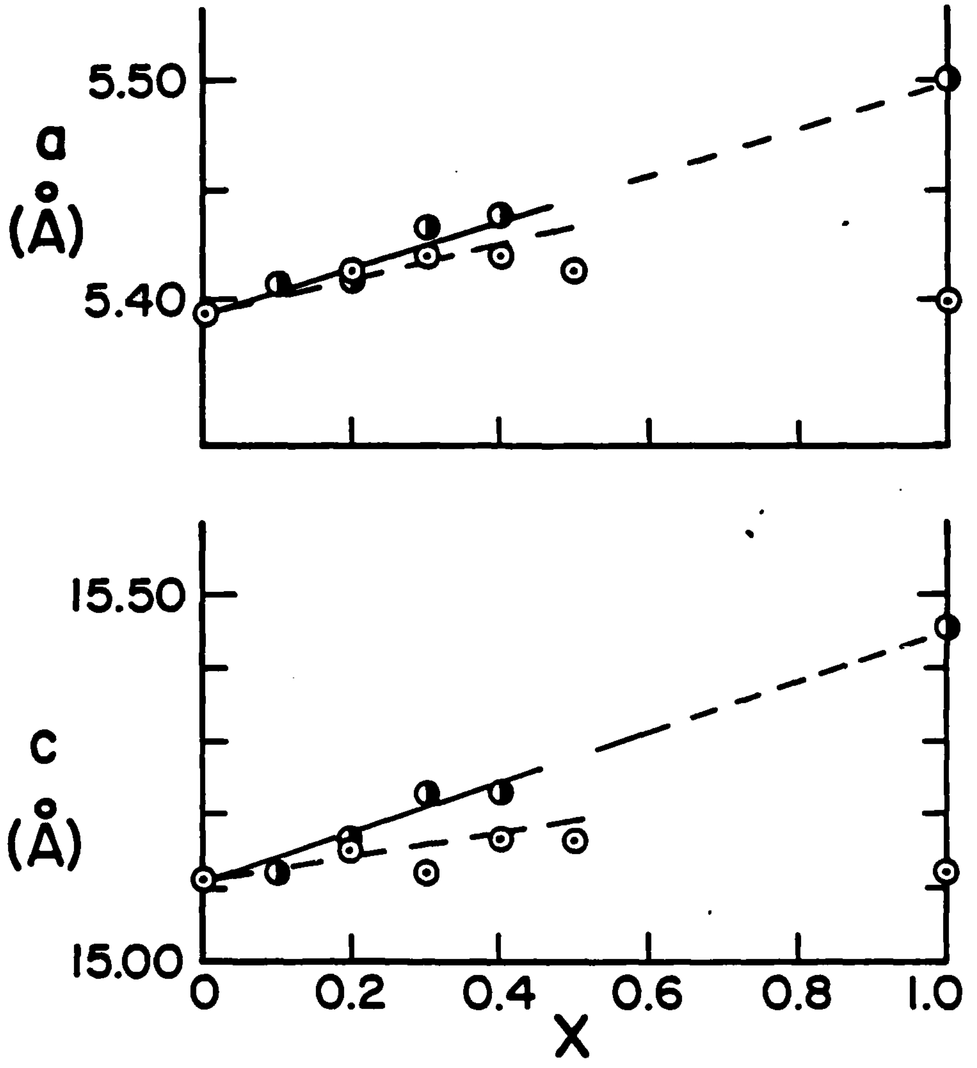
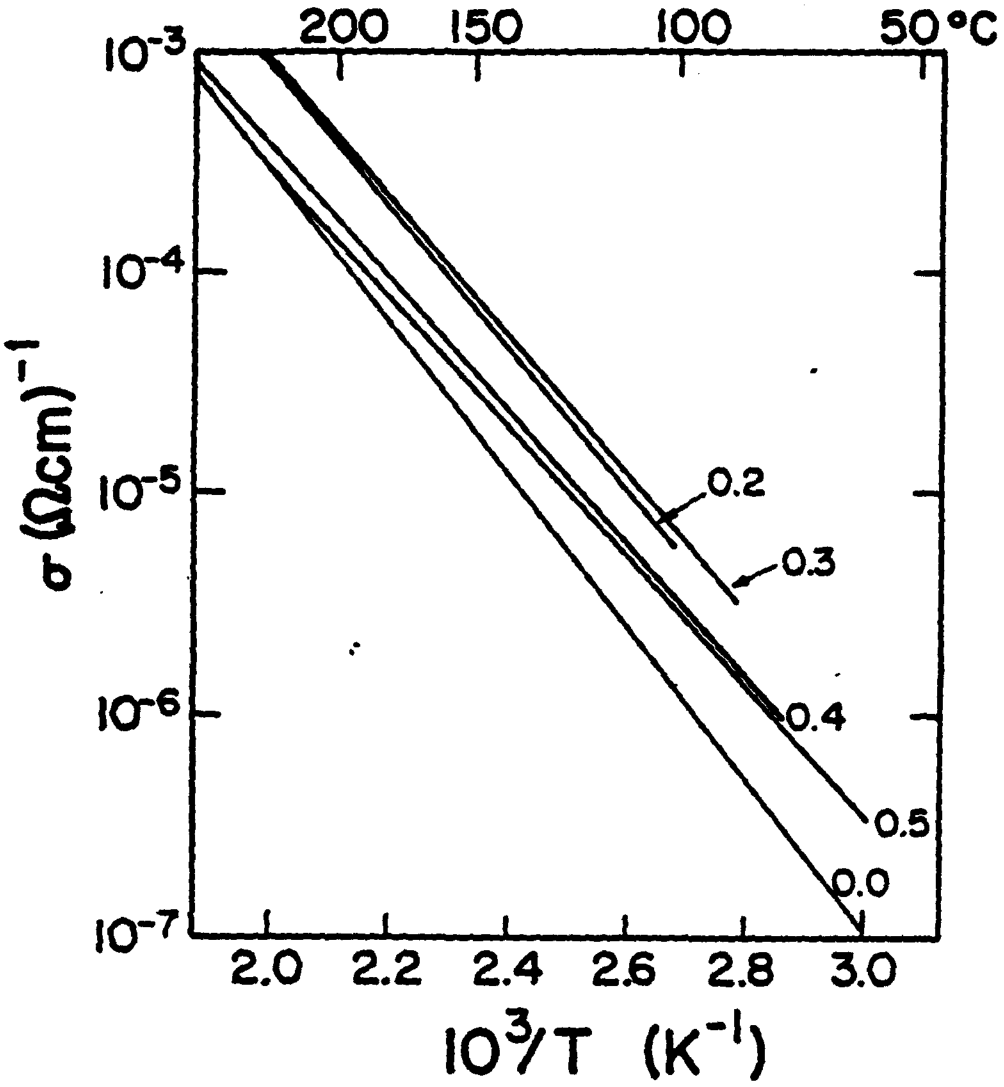


FIG. 4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150
100
50°C
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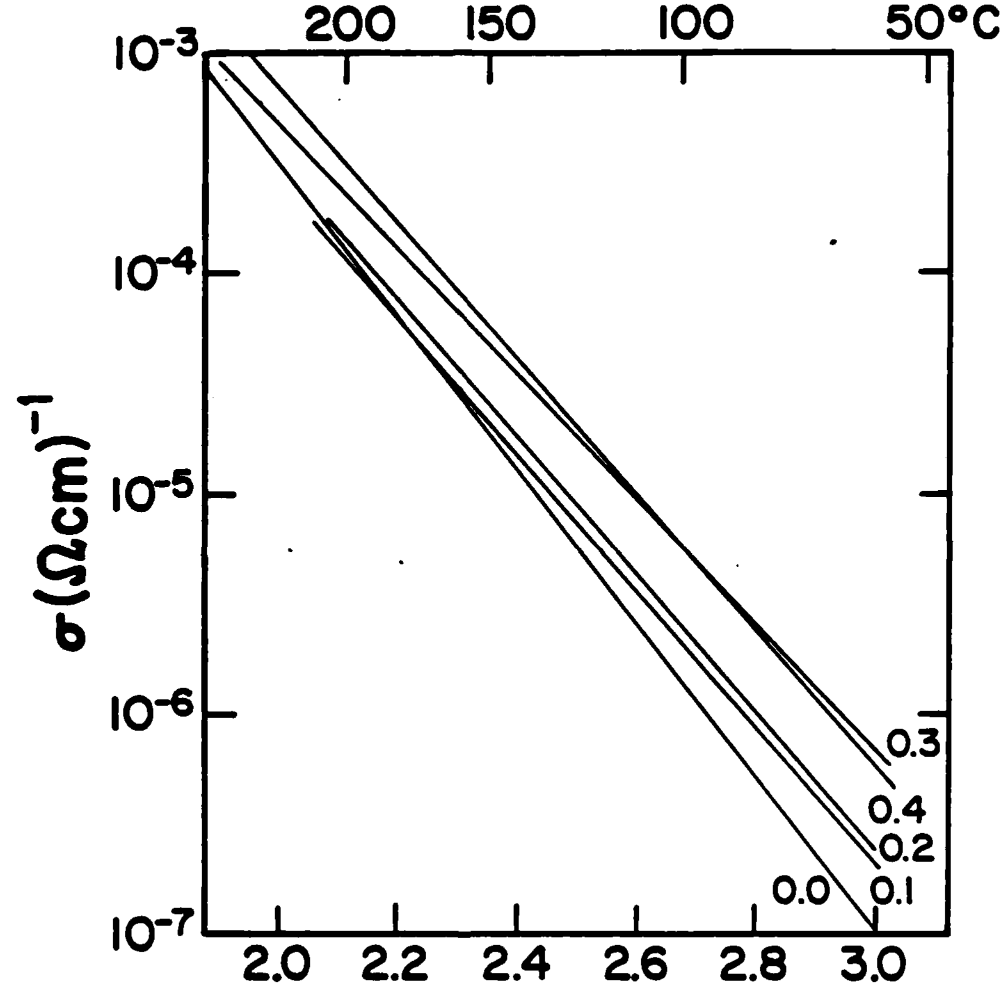
10^{-4}
10^{-5}
10^{-6}
10^{-7}
σ $(\Omega cm)^{-1}$
0.2
0.3
0.4
0.5
0.0
2.0
2.2
2.4
2.6
2.8
3.0
$10^3/T$ (K^{-1})

FIG. 5



200
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50°C
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10^{-4}
10^{-5}
10^{-6}
10^{-7}
$\sigma\,(\Omega cm)^{-1}$
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0.4
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0.1
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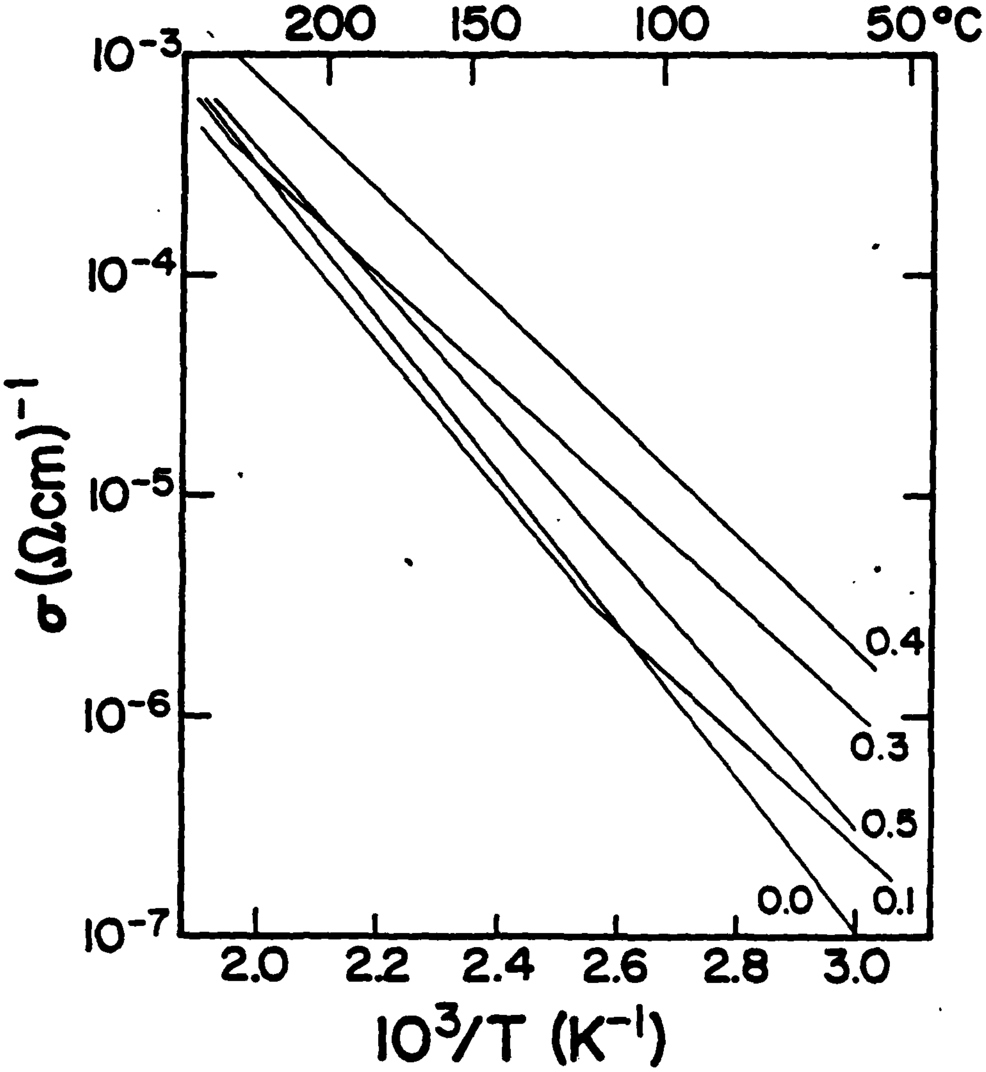
2.2
2.4
2.6
2.8
3.0
$10^3/T\,(K^{-1})$

FIG 6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2.8
3.0
$10^3/T\ (K^{-1})$

FIG. 7

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