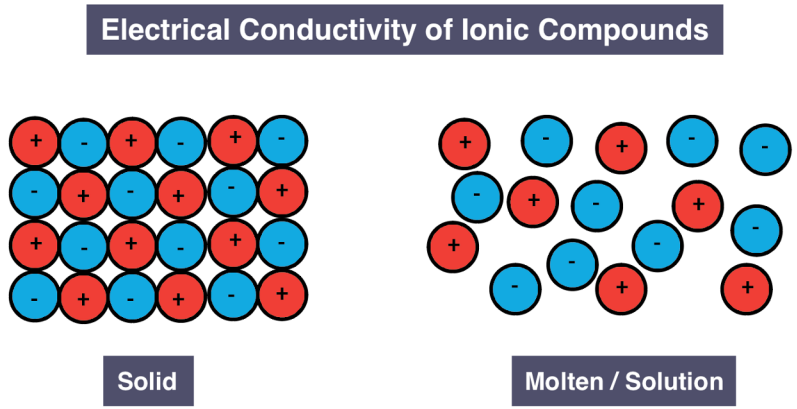

Experiment-9 Conductivity of ionic solid

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ESO205

Objective

- To understand the electrical conduction in ionic solid
- To measure the resistivity variation of NaCl, an ionic material, with temperature and estimate the energy of vacancy migration and vacancy formation (in eV)
- To measure room temperature resistivity of zirconia sample and report resistivity variation with temperature.

Apparatus

- Alumina Crucible , Nacl , vertical Furnace, Electrode Connector, K-type Thermocouple (Chromel-Alumel), Mild Steel plate , Fireclay bricks Glass Wool,
- Zirconia Sample, Probe Arrangement, PID controlled oven, PID controller High voltage power supply, Digital Pico ammeter,

Theory

The electrical conductivity in ionic solids usually arises from the migration of ion under influences on an electric field. In this experiment Alkali halides, the positive ions are much mobile than negative one. the mobility of ion in a perfect lattice is extremely low because of high energy barrier preventing interchange of atom. due to presence of some lattice defects (like vacancy or interstitials is essential for the occurrence of ionic conductivity. the defect concentration is dependent on temperature
Thus, expression for the concentration of pair positive and negative ion vacancies is given

$$n = c \exp\left(-\frac{\phi}{2kT}\right)$$

Where,
$n = no.of\ vacancies\ pair$
$\phi = formation\ energy\ of\ such\ pairs$
$C = a\ constant$
$k = Boltzmann\ constant$
$T = Absolute\ temperature$

The expression of conductivity σ is given by

$$\sigma = A \exp\left(\frac{-E_m + \frac{\phi}{2}}{kT}\right)$$

Where,

$A = a\ constant\ for\ particular\ crystal$

$E_m = Activation\ energy\ of\ vacancies\ pair$

Resistivity of Zirconia

Zirconia is very important ceramic having wide application as refectories material like as insulator, abrasive enamel jeweler, electronics sensor. It has high melting point (2715 $°C$), low thermal conductivity high fracture toughness high hardness high wear resistance, and it is chemically very inert . the electrical conductivity in zirconia is predominantly an ionic conduction process that occurs above 600 $°C$.

Zirconia is usually used Calcia stabilized zirconia or Yttria stabilized Zirconia. The addition of either Calcia or yttria to it result in defect represent by following equation respectively:

$$CaO\ (s) \leftrightarrow Ca''_{Zr} + O_o + V_{\ddot{o}}$$

$$Y_2O_3(s) \leftrightarrow 2Y'_{Zr} + 3O_o + V_{\ddot{o}}$$

The Excess electron may not remain localized at the vacant site($V_{\ddot{o}}$) and become separated giving rise to electron conduction represented as

$$V_{\ddot{o}} = V_o + e''$$

Methodology

In this experiment, two experiment will be performed

a) The resistivity variation of NaCl with temperature and estimate the energy of vacancy migration and vacancy formation (in eV)
   i. Take Alumina Crucible, two mild steel plate and NaCl salt.
   ii. Pour the salt into the crucible and mount the mild steel electrodes in the crucible.
   iii. Insert the whole assembly in vertical furnace
   iv. Fused (NaCl) salt in the crucible is heated to $700 - 750°C$ (below it's melting point $805°C$).
   v. Cool the molten salt slowly to 450°C
   vi. Measure the resistance of fused salt (NaCl) in kΩ with the LCR meter at the interval of $5°C$ fall of the temperature.

b) To measure room temperature resistivity of Zirconia sample and report resistivity variation with temperature
   i. Measure dimension of sample diameter & thickness and placed under the probe. There are two probe one is for the sample and another for Reference Temperature detector (RTD), placed it into the PID oven, and PID oven is connected from picometer, high voltage supply, and PID controller.
   ii. Before starting the experiment make sure current ,voltage should be adjusted to Zero.
   iii. Take reading at constant voltage (100), at temperature of $40°C, 60°C, 80°C, 100°C\ and\ 100°C$

Experimental setup : Furnace

Electrode Connectors

Thermocouple

Ceramic Bricks

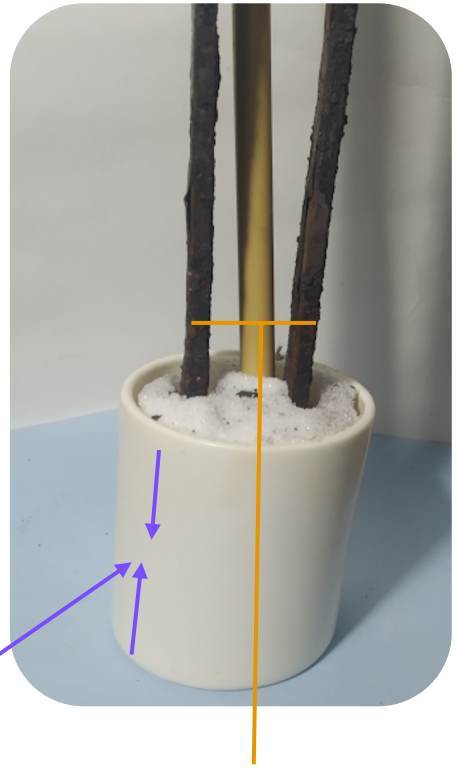
Pid Controller

Vertical furnace

LCR meter

Experimental setup : Assembly

Plate immersed in fused Nacl
length of plate(L)=70mm
width of plate W) = 20
thickness of plate (t)= 5mm



Distance between two electrodes (l)=18 mm

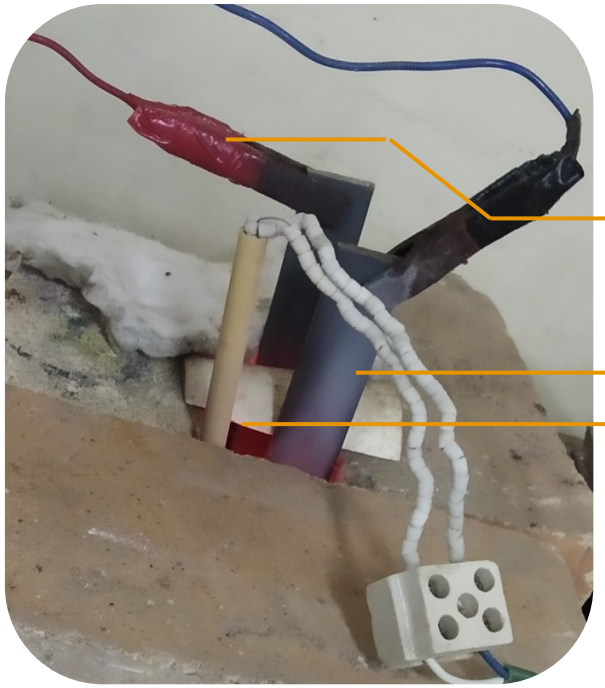
Assembled set up placed in to the vertical furnace

LCR Meter

An LCR meter, is an electrical test instrument used to Measure the inductance (L),Capacitance (C), Resistance (R)

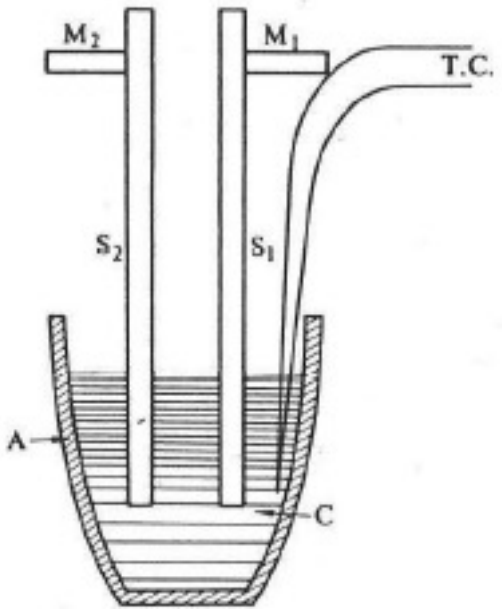
PID Controller

There are two temperature devices, one is the PID controller another is temperature indicator, temperature indicator measure the Fused Nacl temp. and PID Measure the furnace temperature



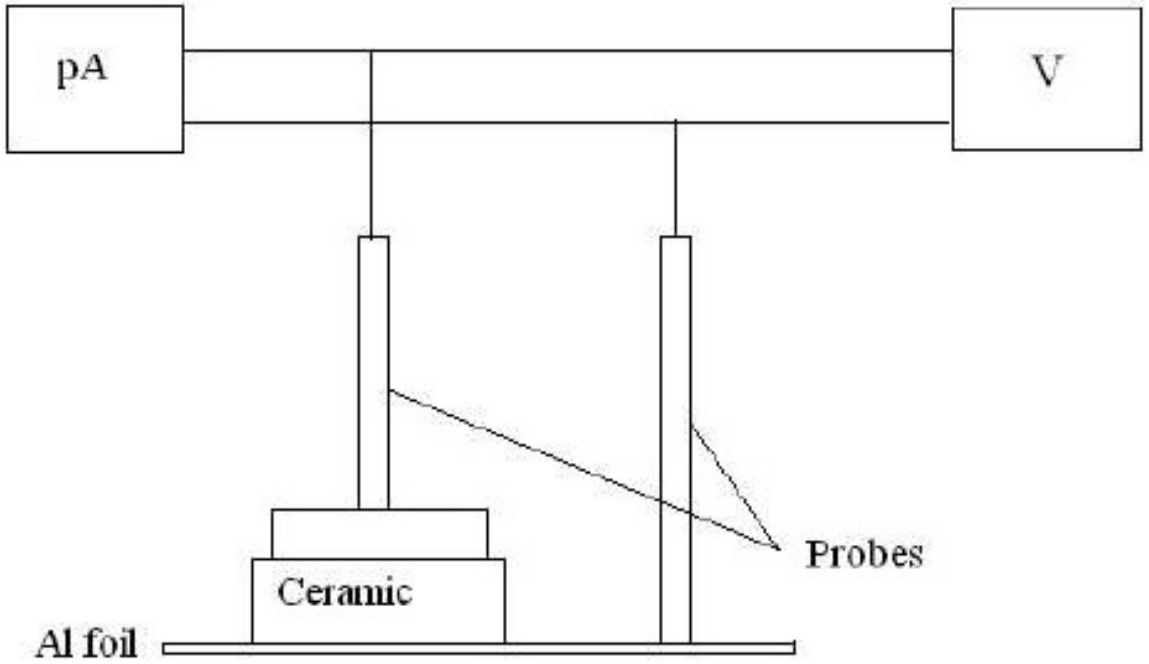
Schematic Diagrams

Ionic Conductivity of ionic solid



A alumina Crucible, C is polycrystalline Nacl,
S_1 and S_2 are two mild steel plate for electrical conductivity act as electrode
T.C represent the thermocouple wires.

Two probe method



Conductivity variation of Zirconia by using Two probe method
ρA =is the Picometer,
V =is the High voltage power Supply

Experimental Setup : Resistivity of Zirconia

Digital Picometer

PID Controller

High Voltage Power Supply

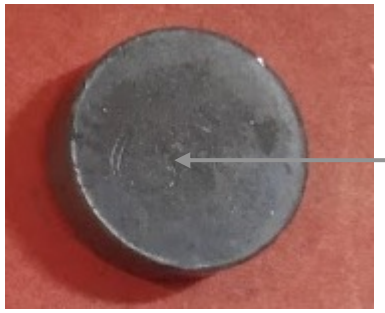
PID Oven

Oven

Probe Arrangement

Sample

Two probe
There are two probe one is sample probe, another is RTD probe



Zirconia Sample

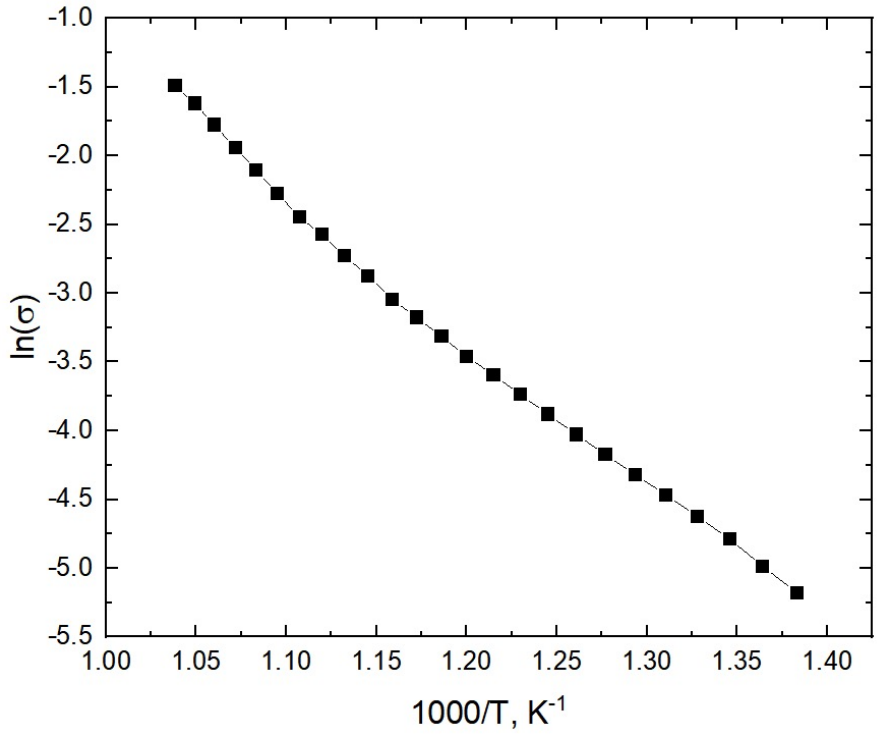
This is very versatile and general purpose instrument . The unit is specially for the measurement of current to1p.
Multiplier : X1, X102, X103, X104, X105.
Accuracy : 0.2% for all ranges.
Resolution :1pA, 10pA, 100pA, 1nA, 100nA,
Input Resistance :2.5kW, 0.25kw, 25W, 2.5W, 0.25W, 0.025W.
Display 3.5digit 7 segment LED (12.5 height) with auto polarity and decimal indication

This is high quality temperature-controlled oven suitable for the Two probe Set –Up. The oven has been designed for the fast heating and cooling rates. Platinum RTD sensor is use for sensing the Temperature. A Wheatstone bridge and instrument amplifier are used for signal conditioning. Temperature Ranges ambient to 200°C,and power rating 150W, resolution 0.1°C
Measurement Accuracy is +/- 0.5 °C.
Range Switch at X1 side display reading would actually reading, when At X10 side range is to be multiplied by 10.
WAIT-RUN Switch- in wait mode the power to oven is OFF and RUN mode power to oven is ON. Temperature would be in Degree centigrade.
SET-MEASURE Switch –in set mode display the set Temp. and in MEASURE mode it display the Current Temperature

This is a fully solid-state power supply designed to meet the power requirement of broad range of Application . such as Radiation detector, photo-multiplier tubes ionization chamber, and where a high voltage source with high degree of regulation and stability is required
Out put : 1-1500 V continuously adjustable.
Current : 1mA max.
Polarity : Negative.
Power Requirement 220Vand 50HZ

Test data obtained at lab

Details of plate immersed in fused NaCl
Length of plate(L)=80 mm
Width of plate (W) = 25 mm
Thickness of plate (t)= 5 mm
Distance between two electrodes (l)=20 mm



Temp.°c	Resistance Ω	Resistivity ρ	Conductivity б	ln(б)	1000/T(K-1)
690	57.162	4.4459	0.225	-1.4916	1.0384
680	65.011	5.056	0.1977	-1.621	1.0493
670	75.915	5.9045	0.16936	-1.775	1.06
660	89.836	6.9872	0.14311	-1.944	1.0718
650	105.72	8.222	0.1216	-2.106	1.834
640	125.46	9.758	0.1025	-2.277	1.0953
630	148.76	11.57	0.0864	-2.448	1.1074
620	168.64	13.116	0.0762	-2.574	1.1198
610	197.65	15.372	0.065	-2.73	1.1325
600	228.85	17.733	0.0564	-2.875	1.1454
590	270.03	21	0.0476	-3.045	1.1587
580	308.52	23.996	0.04167	-3.177	1.1723
570	353.83	27.52	0.0363	-3.316	1.1862
560	409.12	31.82	0.03142	-3.46	1.2
550	468.34	36.426	0.02745	-3.595	1.215
540	540.7	42.054	0.02377	-3.739	1.23
530	622.97	48.453	0.02064	-3.88	1.245
520	721.85	56.144	0.01781	-4.028	1.261
510	836.69	65.076	0.01536	-4.176	1.277
500	967.67	75.257	0.0133	-4.319	1.2936
490	1120.11	87.119	0.01148	-4.4671	1.3106
480	1309.3	101.834	0.00982	-4.6233	1.328
470	1542.7	119.987	0.00833	-4.7878	1.3459
460	1873.7	145.732	0.00686	-4.985	13642
450	2281.4	177.442	0.00563	-5.1786	1.3831

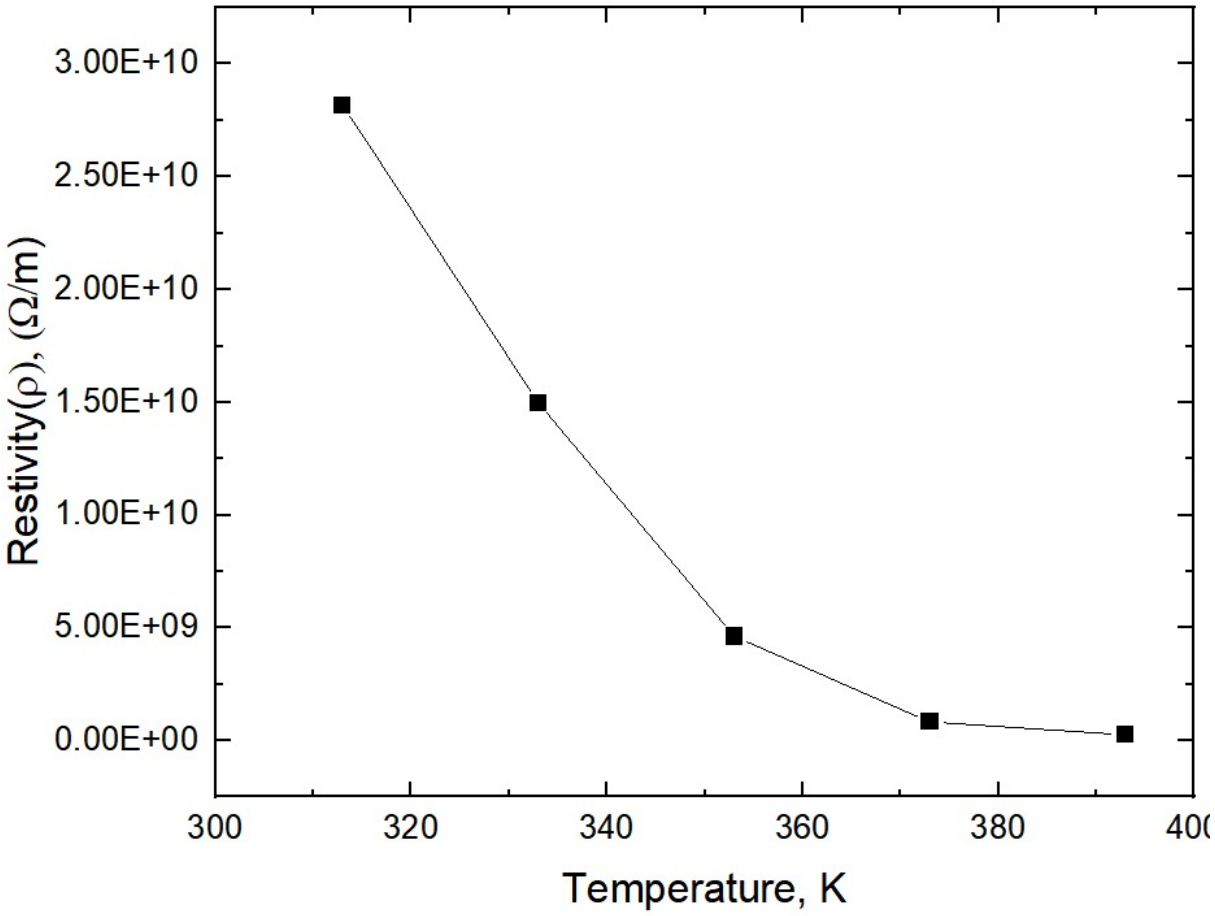
Die mention of Zirconia sample:

Thickness :2.07mm.

Diameter :7.85mm

Temperature °C	Current (Pico ampere)	temperature in (k)	Resistivity ρ (Ω/m)
40	83	313	$2.817*10^{10}$
60	156	333	$1.498*10^{10}$
80	506	353	$4.6183*10^{9}$
100	2830	373	$8.257*10^{8}$
120	9080	393	$2.5735*10^{8}$

At constant voltage =100V



Observation

a. The slope of the graph decreases as the temperature decrease.

b. There is point when the slope get changed slightly this point is known as **break point**.

c. For Zirconia it is like a parabola, because it arise accordingly to a parabola Equation with Temperature.

Thank You