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To Study the Effect of Cryogenic Treatment on Cathode Electrode of Lithium Cobalt Oxide (C-$LiCoO_2$) Material

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Abstract: The automotive lithium ion battery (LIB) packs is an escalating concern as the world-wide sale of electric vehicles (EVs) continues to rise. These batteries are more popular now days to get the maximum efficient utilization in electric vehicles. The aim of the study is to find out the effect of cryogenic treatment on the conductivity, resistivity and grain size of lithium cobalt oxide. To get the results on these parameters the lithium cobalt oxide layer is recovered from the dry rechargeable lithium battery. Cryogenic treatment done on battery at – 192 degree Celsius and test the parameters and its effect on conductivity, resistivity ad grain size of battery. This paper studies the review on these materials research effect & cryogenic treatment effects studied by other researchers.
Keywords: Conductivity, resistivity, cryogenic treatment, Lithium Cobalt Oxide, etc.

I. INTRODUCTION

The automotive industry's pursuit to actively reduce its impact on the environment by shifting its dependence from the internal combustion engine (ICE) vehicle to alternative sustainable technologies continues to gain momentum. This shift is occurring amidst an ever increasing framework of legislation to reduce carbon emissions, such as the EU 2020 targets and growing concerns over local air pollution. Fuel combustion arising from transport (including international aviation) has increased significantly since 1990 to comprise 23% of all greenhouse gas emissions across the EU in 2015. The adoption of hybrid electric vehicles (HEVs), plug-in hybrid electric vehicles (PHEV) and battery electric vehicles (BEVs) have the potential to yield considerable greenhouse gas emission reductions. These electric vehicles (Evs) typically contain lithium-ion batteries (LIBs) as the dominant technology due to their relatively high energy density, long life cycles, lack of memory effect and slower self-discharge rates. Market adoption of LIBs continues to grow; for example the Boston Consulting Group predicts 26% of new cars sold in 2020 will have electric or hybrid power trains and in total 11 million of these vehicles will be equipped with LIBs. Furthermore, Bloomberg New Energy Finance annual long-term forecast estimates that 54% of new cars sold in 2040 will be Evs, underpinned by impending reductions in LIB prices.

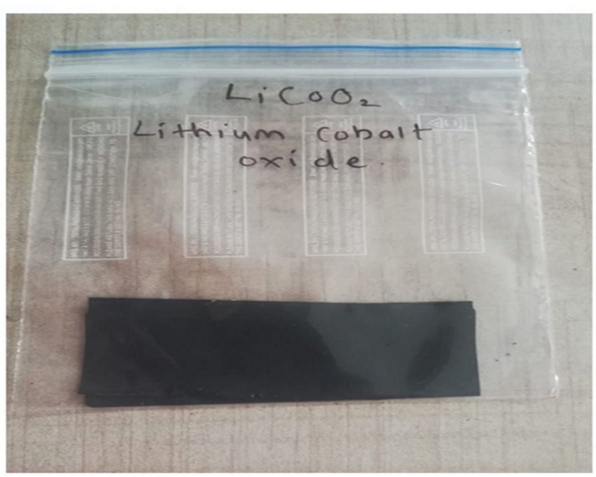

Fig. 1.1 Lithium Cobalt Oxide Material ($LiCoO_2$)

A. *Objective of Project*

Objectives of the project are as follows,

1) To conduct the cryogenic treatment on lithium cobalt oxide
2) To test the conductivity of material before and after cryogenic treatment.
3) To test the resistivity of material before and after cryogenic treatment.
4) To test the grain size of material before and after cryogenic treatment.

II. LITERATURE REVIEW

Thomas R.B. Grandjean, 2019, The invert calculated test of shipping waste car lithium particle battery (LIB) packs is a raising worry as the overall closeout of electric vehicles (Evs) keeps on rising. Under the European Union (EU) Battery Directive, EV makers are delegated battery makers and are in charge of the accumulation, treatment and reusing of waste or harmed vehicle batteries. The European understanding concerning the International Carriage of Dangerous Goods by Road (ADR) stipulates that harmed or faulty LIB packs must be shipped in endorsed blast confirmation steel compartments. This requires expensive testing so as to meet ADR necessities. Besides, the additional size and weight of this bundling adds further restrictive cost to the transportation of harmed or imperfect LIB. In this examination, cryogenically solidified cells are demonstrated to be not able discharge any vitality even in outrageous maltreatment conditions [1].

Jakobus Groenewald, 2019, The International Carriage of Dangerous Goods by Road prerequisite to move harmed or flawed LIB packs in endorsed blast confirmation steel compartments forces costly accreditation. Further, the physical weight and volume of LIB bundling builds transport expenses of harmed or damaged packs as a major aspect of a total reusing or repurposing methodology. Cryogenic glimmer solidifying (CFF) expels the likelihood of warm out of control in LIBs even in outrageous maltreatment conditions [2].

Naoki Nitta, 2015,This audit spreads key innovative improvements and logical difficulties for an expansive scope of Li-particle battery cathodes. Occasional table and potential/limit plots are utilized to look at numerous groups of appropriate materials. Execution attributes, current impediments, and ongoing leaps forward in the advancement of business intercalation materials, for example, lithium cobalt oxide (LCO), lithium nickel cobalt manganese oxide (NCM), lithium nickel cobalt 916luminium oxide (NCA), lithium iron phosphate (LFP), lithium titanium oxide (LTO) and others are appeared differently in relation to that of transformation materials, for example, alloying anodes (Si, Ge, Sn, and so on.), chalcogenides (S, Se, Te), and metal halides (F, Cl, Br, I). New polyamine cathode materials are additionally talked about.

The cost, bounty, wellbeing, Li and electron transport, volumetric development, material disintegration, and surface responses for each sort of cathode materials are portrayed. Both general and explicit methodologies to conquer the present difficulties are secured and arranged [3].

Anna Boyden, 2016,The Environmental Impacts of Recycling Portable Lithium-Ion Batteries, This paper will examine the various procedures that are at present utilized for reusing versatile lithium-ion batteries, for example, hydrometallurgy, hydrometallurgy, and blends of procedures. Reviews are done to comprehend the materials recovered from each procedure, and are gotten from a few reusing organizations around the globe.

A relative life cycle evaluation will be performed for two diverse reusing forms (hydrometallurgy and pyro-metallurgy), so as to comprehend the related environmental impacts. This investigation demonstrates that the biggest supporters of the natural effects are power age, cremation of plastics, and land filling of build-up. As far as ecological impacts, it is proposed that the most advantageous procedures are those that use low [4].

Linmin Wu, 2014,Thin film battery-powered battery has turned into an examination hotspot due to its little measure and high vitality thickness. Lithium cobalt oxide as a run of the mill cathode material in traditional lithium particle batteries is likewise generally utilized in meagre film battery-powered batteries.

In this work, the electrochemical, mechanical and warm properties of $LiCoO_2$ were efficiently researched utilizing the first principles strategy. Versatile constants under hydrostatic weights between 0 to 40 Gpa were processed. Explicit warmth and Debye temperature at low temperature were talked about.

Warm conductivity was acquired utilizing the forced motion strategy. The results show great concurrences with exploratory information and computational outcomes in writing [5].

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III.EXPERIMENTAL PLAN, SETUP AND PROCEDURE

A. Block Diagram

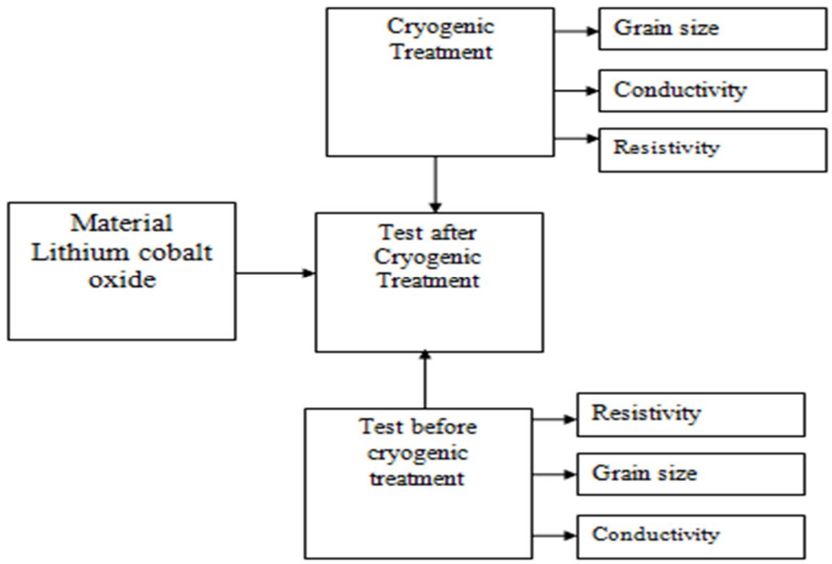

Fig. 3.1 Cryogenic Treatment

The basic aim of the project is to find out the effect of cryogenic treatment on conductivity, resistivity and grain size befire and after cryogenic treatment.

A cryogenic treatment is the process of treating work pieces to cryogenic temperatures (i.e. below −190 °C (−310 °F)) in order to conductivity. In addition to seeking enhanced resistance relief and stabilization, or wear resistance, cryogenic treatment is also sought for its ability to improve conductivity by precipitating micro-fine eta carbides, which can be measured before and after in a part using a quantised.

Fig. 3.2 Cryogenic Process Setup

B. Electrical Resistivity Measurement

Fig. 3.3 Resistivity measuring instrument (Kelvin double bridge)

Electrical conductivity is reciprocal of resistivity and Resistivity can be calculated by using the following equation 3.3

$$Electrical\ Resistivity = \frac{RA}{L} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(3.3)$$

Where,
R = Resistance of sample material
A = Cross Sectional Area
L = Length of Sample

C. Calculation

The sample of dimensions 10 * 10 * 0.5 mm is prepared for measurement of electrical resistance.

1) Untreated Sample

YOKOGAWA instrument meter reading = 7.9
Multiply Factor = 10
Resistance = 7.9 * 10 = 79 Ω

$Electrical\ Resistivity = \frac{RA}{L} = \frac{79 * 0.5 * 10^{-3} * 10^{-2}}{10 \text{ x } 10^{-2}}$ = 0.0395 Ωm = 3.95 Ωcm

Electrical Conductivity = $\frac{1}{3.95}$ = 2.5 * 10^{-1} S/cm

2) Treated Sample

YOKOGAWA instrument meter reading = 6.2
Multiply Factor = 10
Resistance = 6.2 * 10 = 62 Ω

$Electrical\ Resistivity = \frac{RA}{L} = \frac{62 * 0.5 * 10^{-3} * 10^{-2}}{10 \text{ x } 10^{-2}}$ = 0.031 Ωm = 3.1 Ωcm

Electrical Conductivity = $\frac{1}{3.1}$ = 3.225 * 10^{-1} S/cm

D. X-Ray Diffraction Analysis (XRD)

X-ray diffractometer is utilized to create X-ray diffraction pattern. XRD peaks analysis like percentage change in crystanility,grain size etc. was done by using software called origin software.

Fig 3.4.XRD Machine

E. Grain size Determination:

1) *Before Cryogenic Treatment:* Crystalline size determination Before cryogenic treatment

Before cryogenic treatment the grain size determination is as follows,

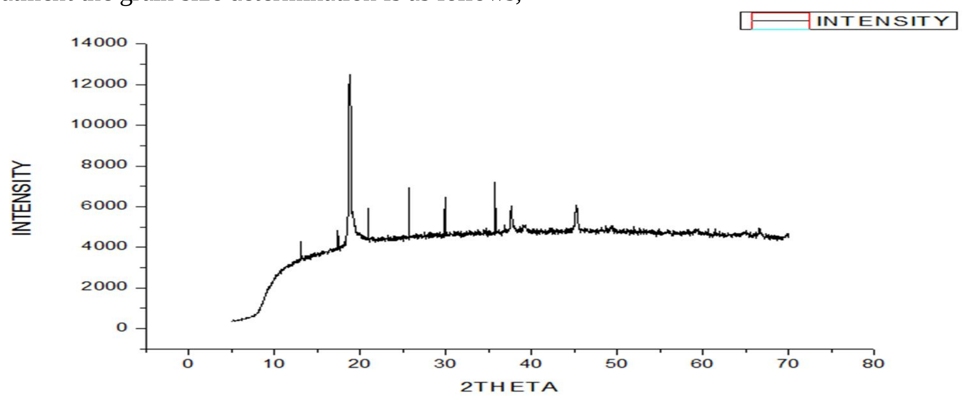

Fig. 3.5 XRD graph of untreated $LiCoO_2$

Above graph shows the intensity of the readings got from the uxd file.

The grain size is calculated from the following formula,

Dp = (0.94 X λ) / (β X Cosθ)

Where,

Dp = Average Crystallite size,

β = Line broadening in radians,

θ = Bragg angle,

λ = X-Ray wavelength

2) *Crystalline Size Determination After Cryogenic Treatment:* After cryogenic treatment the grain size determination is as follows,

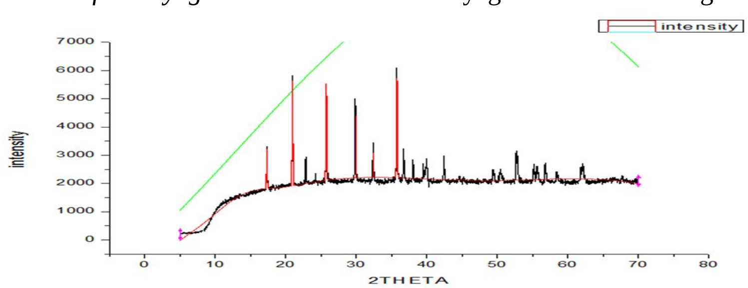

Fig. 3.6 XRD graph of treated $LiCoO_2$

Above graph shows the intensity of the readings got from the uxd file.

Dp = (0.94 X λ) / (β X Cosθ)

Where,

Dp = Average Crystallite size,

β = Line broadening in radians,

θ = Bragg angle,

λ = X-Ray wavelength

Cryogenic process	Crystenallity (%)	Grain size (nm)
Before cryogenic treatment	18.31	21.91
After cryogenic treatment	21.46	15.72

Table 1: Crystelline & Grain size of material

Above table shows the readings of the grain size in nm.

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IV. RESULT

Sample	Untreated C-$LiCoO_2$	Treated C-$LiCoO_2$
Resistivity	3.95 Ωcm	3.1 Ωcm
Conductivity	$2.5 * 10^{-1}$ S/cm	$3.225 * 10^{-1}$ S/cm

Table 2: Electrical Properties

A. Scanning Electron Microscopy

The SEM analysis of the material before and after cryogenic treatment is given as below;

B. Before Cryogenic Treatment

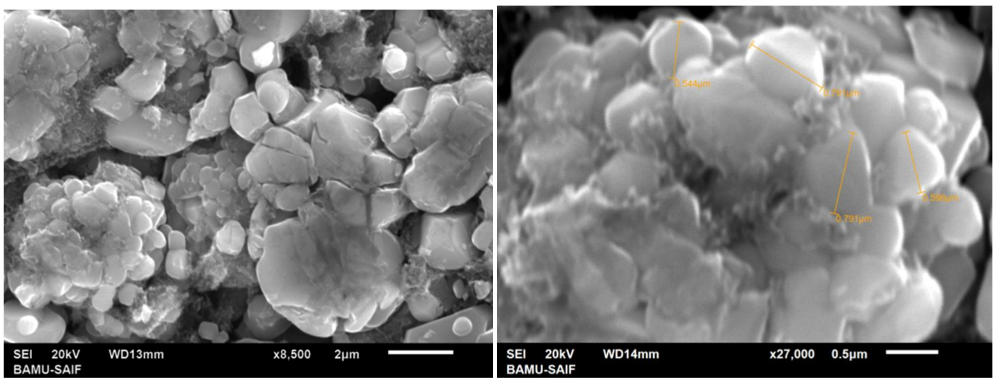

Fig. 3.7 SEM micrograph taken for 2 micro meters & 0.5 micro meters

C. After Cryogenic Treatment

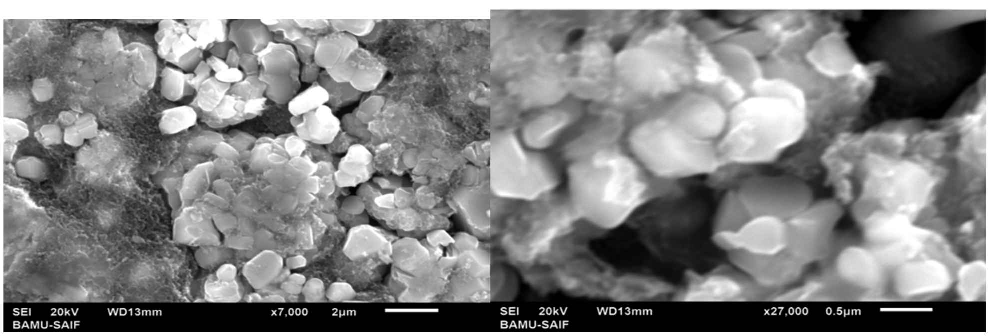

Fig. 3.8 SEM micrograph taken for 2 micro meters & 0.5 micro meters

SEM image of the cryogenic treated sample shows that, the structure gets more compact as well as more uniform and the particle size is reduced.

1) *Before Cryogenic Treatment*
a) Area Of The Crystalline Peaks: 326.36
b) Area Of All Peaks: 5978

$$\%\,Crystallinity = \frac{5978}{326.36}$$
$$\%\,Crystallinity = 18.31$$

2) *After Cryogenic Treatment*
a) Area Of The Crystalline Peaks: 272.57
b) Area Of All Peaks: 5852

$$Crystallinity = \frac{5852}{272.57}$$
$$\%\,Crystallinity = 21.46$$

The crystenallity and grain size of the material before and after cryogenic treatment is given as below,

Cryogenic Process	Crystenallity (%)	Grain size (nm)
Before cryogenic treatment	18.31	21.91
After cryogenic treatment	21.46	15.72

Table 3: Crystelline & Grain size of material

V. CONCLUSION

From this paper, it can be concluded that, there is a very less amount of research occurs in the lithium cobalt oxide materials of how we can provide the efficient batteries parameters like conductivity, resistivity and effect of cryogenic treatment on grain size. In this research paper, it has been proved that, after cryogenic treatment the grain size is reduced and conductivity of the material is increased. The lithium cobalt oxide materials compared with before cryogenic treated material. It can be shows that, the parameter or performance of the material of rechargeable dry battery is improve after the cryogenic treatment.

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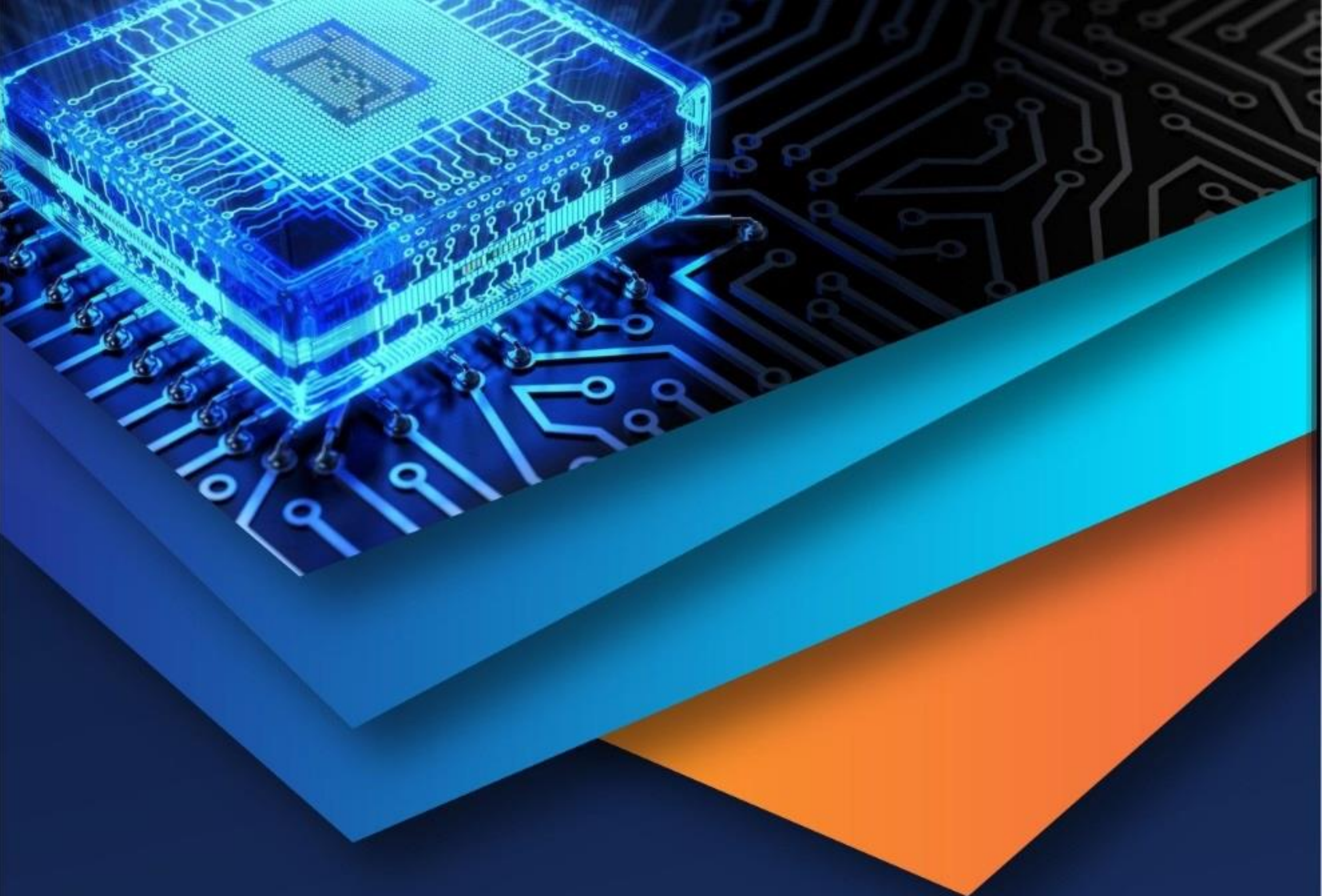

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