

CHAPTER 3

EXPERIMENTAL PROCEDURES

Chapter - 3

Experimental procedures

This chapter describes the experimental procedure used for:

- (a) Processing and synthesis of silver-cadmium oxide and silver-zinc oxide composite powders by different processing routes.
- (b) Characterization of processed powders by techniques such as AAS, XRD, SEM, ESCA, DTA/TGA., Laser diffraction, etc.
- (c) Consolidation of above powders using PM techniques of pressing, sintering, repressing/hot pressing to near theoretical density.
- (d) Evaluation of physical, mechanical and microstructural properties of final compacts.
- (e) Fabrication of Life-testing set-up to evaluate electrical contact properties and Life-testing of Ag-MeO composite contacts.

3.1 Raw materials and system selection :

The starting powders used in this investigation for synthesis by conventional PM route and Mechanical Alloying (MA) were silver, cadmium oxide and zinc oxide. AR grade - 400 mesh argon packed silver powder was obtained from Sisco Research Laboratory (SRL), Bombay. AR grade zinc oxide of E-Merck and cadmium oxide of SD Fine Chemicals, Bombay were used. The characteristics of these powders are summarized in Table 3.1. The mean particle size and particle size distribution for these powders were estimated using CIS-1 Computerized Inspection System of Galai make, based on Laser diffraction principle. Fig. 3.1-3.3 show the probability number distribution graphs for

these powders. The apparent density, tap density and flow-rate were estimated as per the recommended practice of ASTM standard B 417.

Table 3.1: Characteristics of starting powders

Powder	Ag	CdO	ZnO
Purity (wt%)	99.9 min.	99.5	99.6
Mean particle size (μ)	1.41	1.16	0.75
App. density (g / cc)	1.14	1.00	0.43
Tap density (g / cc)	1.72	1.92	0.77
Flow rate (g/min)	NFF*	NFF	NFF

* non free flowing

BDH make AR grade silver nitrate of min. 99.9% purity was used as the source of silver in spray- coprecipitation, electroless coating and freeze-drying routes.

Present investigation deals with Ag-CdO and Ag-ZnO systems. In Ag-CdO group, Ag-10 wt% CdO, Ag-12 wt% CdO and Ag-15 wt% CdO compositions were selected for this study as they are the most frequently used compositions of contact materials in various switchgear devices for low and medium-voltage applications as per the literature. It is customary to compare the performance of contact materials of different material combinations on the basis of volume fraction of dispersed oxide phase and hence the compositions in Ag-ZnO group were worked out accordingly. Table 3.2 gives compositions and theoretical densities for Ag-CdO and Ag-ZnO systems under investigation.

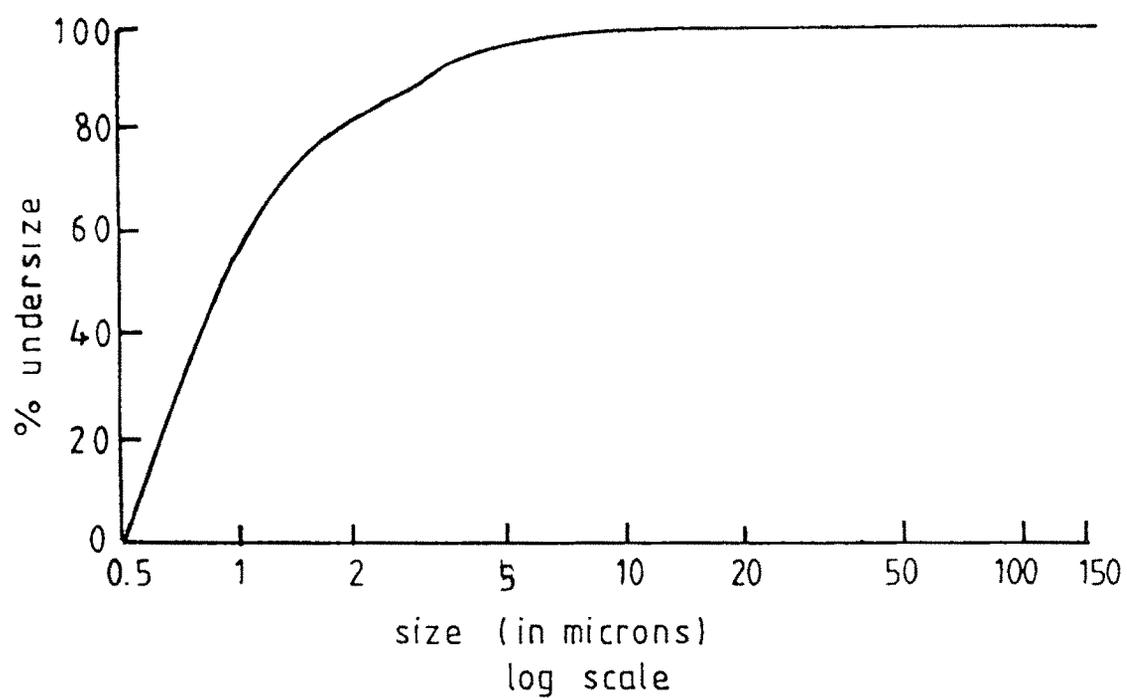


Fig. 3.1: Probability number distribution plot for Ag powder.

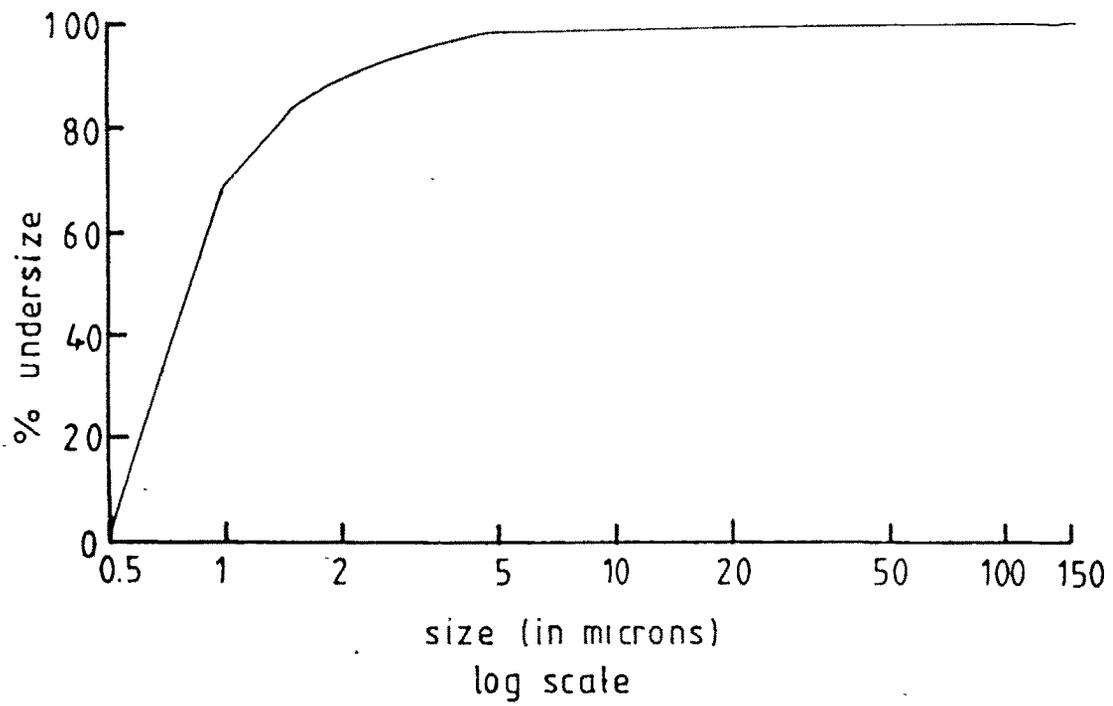


Fig.3.2.: Probability number distribution plot for CdO powder

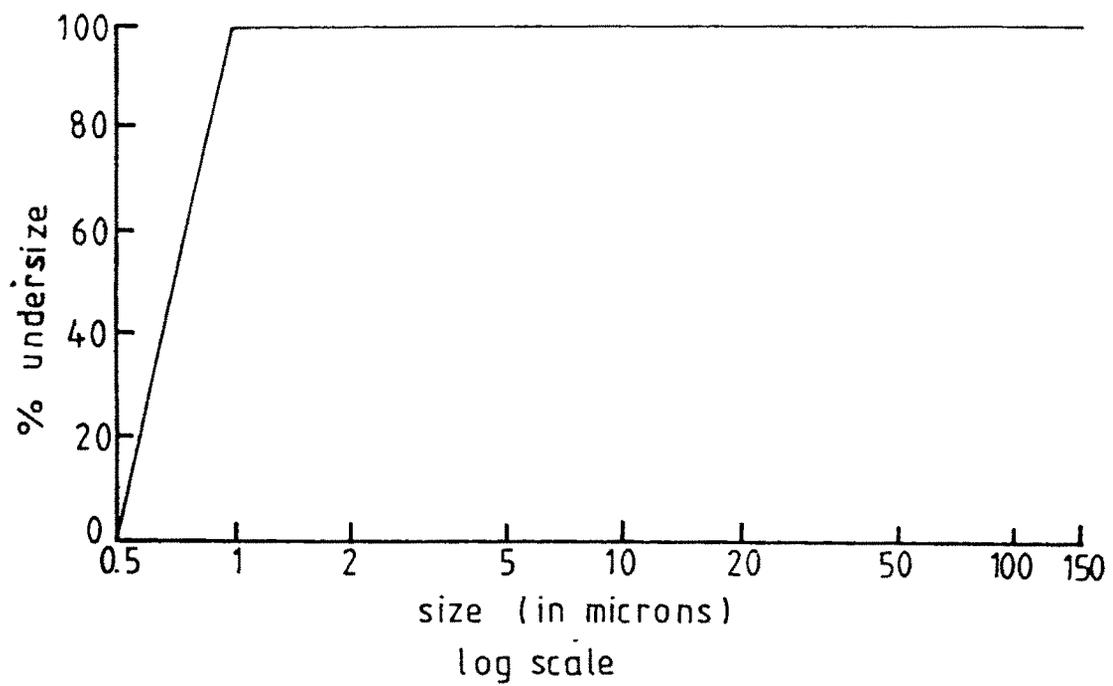


Fig.3.3 : Probability number distribution plot for ZnO powder

Table 3.2: Equivalent Ag-CdO and Ag-ZnO Systems at a glance

System	Theoretical density (g / cc)	System	Theoretical density (g / cc)
Ag-10 wt % CdO	10.20	Ag-7.1 wt % ZnO	9.90
Ag-12 wt % CdO	10.15	Ag-8.6 wt % ZnO	9.77
Ag-15 wt % CdO	10.06	Ag-10.8 wt% ZnO	9.60

Thus Ag-10 wt % CdO and Ag-7.1 wt% ZnO are identical on equal volume fraction (of oxide phase) basis.

3.2. Powder processing :

Silver-cadmium oxide (Ag-10 wt % CdO, Ag-12 wt % CdO and Ag-15 wt % CdO) and silver-zinc oxide (Ag-7.1 wt % ZnO, Ag-8.6 wt % ZnO and Ag-10.8 wt % ZnO) composite powders were prepared by following processing routes :

- i) Conventional PM route (blending)
- ii) Spray-coprecipitation process
- iii) Electroless coating process
- iv) Freeze-drying route
- v) Mechanical-alloying method

3.2.1. Conventional PM route (blending) :

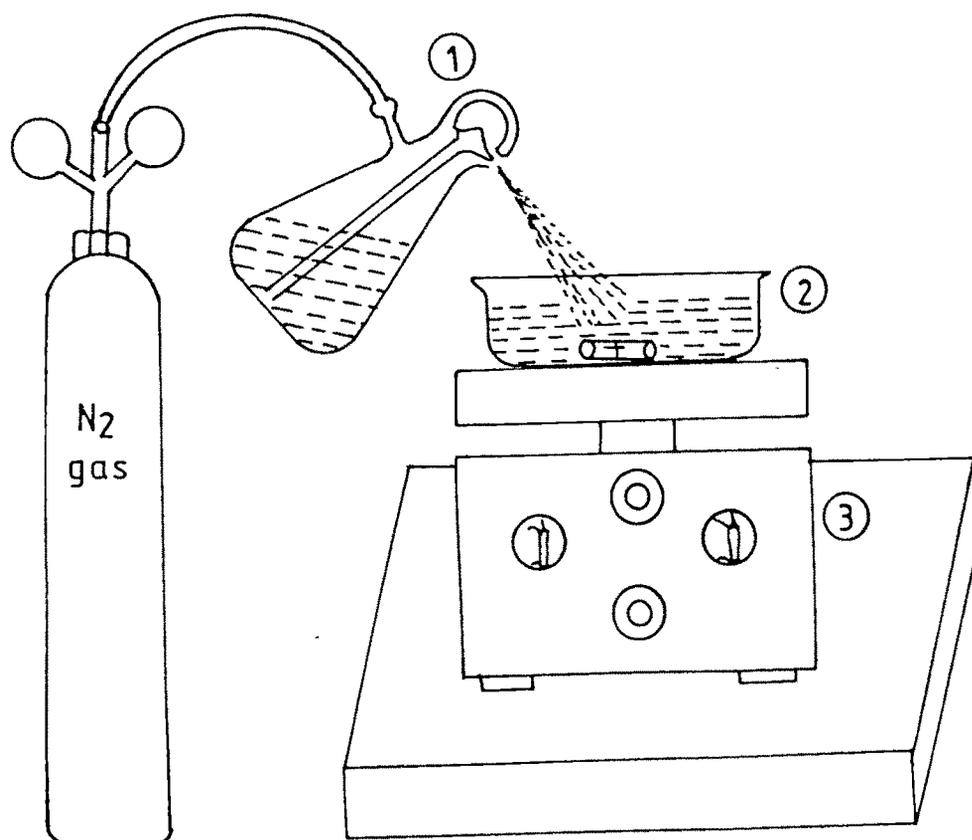
Powders of aforesaid compositions were prepared by blending-400 mesh AR grade silver powder with stoichiometric amounts of cadmium oxide and zinc oxide, respectively, in a cylindrical blender of 56 mm dia. x 70 mm ht. fitted with four rectangular baffle plates of 10 mm x 70 mm size. The blending was carried out using

laboratory roller mill at a speed of 130 rpm for 20 min. time in each case. The blended powders were finally sieved through 100 mesh sieve.

3.2.2. Spray-coprecipitation process :

Stoichiometric amount of metal oxide (CdO or ZnO as the case may be) was dissolved in AR grade dilute nitric acid solution to form respective metal nitrate solution. Sameway, stoichiometric quantity of silver nitrate was dissolved in distilled water to form Ag NO₃ solution. 1M solution of mixed nitrates of Ag and Cd or Ag and Zn was formed by mixing the two solutions in required proportions. The pH of the mixed nitrate solution was raised to ~ 5 by adding to it 1:1 ammonia solution. The pH was measured and monitored by a portable digital pH meter of Hanna make. The final volume of mixed nitrate solution was raised to 100 ml.

The precipitant solution was prepared by dissolving stoichiometric amount of sodium carbonate and potassium carbonate in distilled water to make 1M mixed carbonate solution. The 100 ml. of mixed nitrate solution was then sprayed through glass capillary spray nozzle at a N₂ gas pressure of 1.0 Kg/cm² into 750 ml. of 1M mixed carbonate solution of 11 pH maintained at 20° C under constant stirring. Fig. 3.4 shows schematically the spray-coprecipitation set up. The canary-yellow coloured precipitates of mixed carbonates of Ag and Cd or Ag and Zn so obtained were allowed to stand for some time and were filtered through a Buckner funnel using Whatman Gr. 41 filter paper. The precipitates were then washed several times with copious amount of distilled water to remove the impurities of alkali metals like Na and K. The precipitates were dried in air in an oven at 110°C and were sieved through 100mesh sieve. Sieved powder was fired at 550°C for 90 min. in air in a muffle furnace to ensure decomposition of mixed carbonates to respective silver-metal oxide composite powders. The fired powder was once again added to distilled water and the same cycle of filtration, washing, drying,



- ① spray- nozzle
- ② reacting solutions
- ③ magnetic stirrer-cum - heater

Fig.3.4: Schematic of spray-coprecipitation set-up

firing and sieving was repeated to get the final powder with Na and K impurities within the permissible limits of a few ppm.

3.2.3. Electroless coating process :

Similar batches of powders were prepared by electroless coating process. Stoichiometric amount of CdO or ZnO was first suspended in distilled water and a few drops of concentrated liquor ammonia were added to form colloidal suspension, ultrasonically. Liquor ammonia acts as a hydroxyl ion donor for metal oxide particle surfaces. The pH of the solution was maintained at 9. The colloidal suspension of CdO or ZnO was then transferred to a magnetic stirrer-cum-heater and vigorously stirred for 30 min. The particles of cadmium oxide or zinc oxide were coated with silver by simultaneously adding 0.8 M AgNO₃ and 1.0 M Na-formate solutions under controlled conditions of pH and feed rate. During the entire reduction stage, the pH was maintained at 7 by addition of 1:10, NaOH solution. Sodium formate leads to in situ reduction of silver nitrate to silver ions, which in turn get attracted to negatively charged surfaces of metal oxide (CdO or ZnO) particles in the suspension leading to silver coating. During coating, bath was continuously stirred and the bath temperature was maintained at 50° C. The coated powder was washed repeatedly with distilled water to prevent contamination mainly from sodium ions, dried and fired in air at 110° C in an oven and finally sieved through 100 mesh sieve.

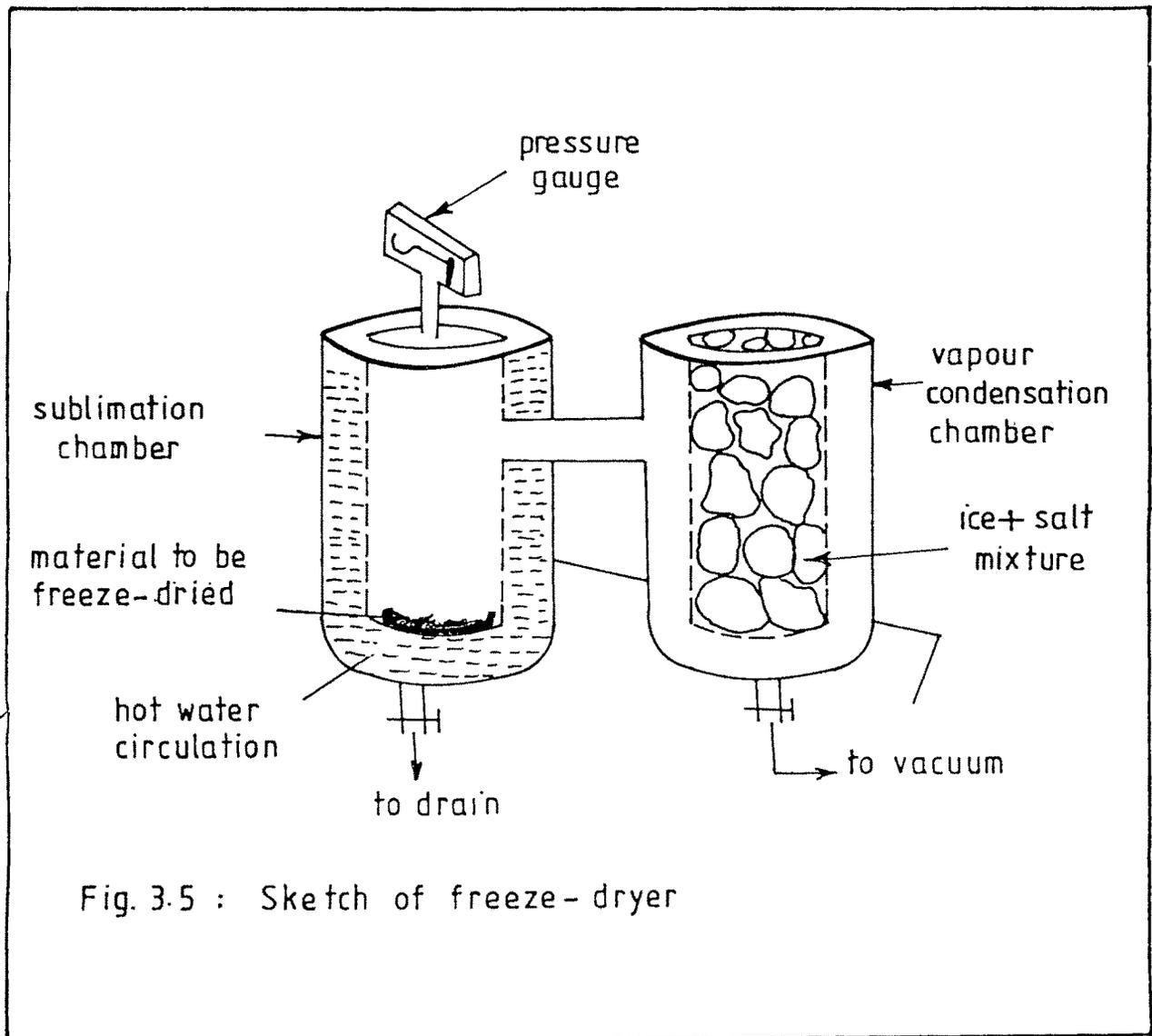
3.2.4. Freeze-drying route :

Precipitates of bicarbonates of silver and cadmium as well as bicarbonates of silver and zinc were first obtained by method described in section 3.2.2. by using 1 molar sodium bicarbonate solution as precipitant instead of solution of mixed carbonates of Na and K. This is because the resultant precipitates of silver bicarbonate have greater thermal

stability at ambient temperature and above and hence preferred to silver carbonate precipitates. The precipitates of mixed bicarbonates of Ag and Cd or Ag and Zn were filtered through Whatman Gr. 41 filter paper and washed repeatedly with water to remove alkali metal impurities. The damp cake so obtained on air-drying was washed & dehydrated with extra pure methyl alcohol and finally air dried. This was the starting material for freeze-drying. Methanol dehydrated powder was uniformly dispersed in AR grade tertiary butyl alcohol for 10 min., ultrasonically. The resulting suspension was rapidly frozen by cooling to a temperature of about 5°C by pouring it into the precooled trays maintained at subzero temperature by means of dry ice & acetone mixture.

Freeze-drying of powders was carried out in an industrial freeze-dryer of M/s Alembic Chemical Works Co.Ltd., Baroda. Fig. 3.5 shows the sketch of the freeze-dryer used.

Before transferring the frozen-powders to freeze-drying unit, the entire system was subjected to low temperature. The ice + salt mixture was used in the outer jacket of sublimation chamber as well as inside the condenser chamber to maintain the temperature between -5°C and 0°C . After attainment of this temperature, the system was first evacuated to 1000 microns vacuum and the vacuum was then gradually improved to 500 microns to remove entrapped air and the condensed moisture. The frozen trays containing the powder samples were then stacked in the sublimation chamber after breaking the vacuum and the vacuum was restored quickly. For 4 to 5 hours from the beginning of freeze-drying, the temperature in the outer jacket of sublimation chamber and the inside of condensation chamber was maintained at -5°C to 0°C . Thereafter, chilled water of $+5^{\circ}\text{C}$ temperature and luke-warm water of $35-40^{\circ}\text{C}$ were circulated through the outer jacket of condenser chamber to provide heat of sublimation to frozen mass. The vacuum in freeze-drying system was gradually improved to 30-50 microns by means of rotary and oil-diffusion pumps. Total sublimation time was of the order of 24 hrs. during which tertiary butyl alcohol was



completely sublimed as vapours and was condensed in outer jacket of condensation chamber attached to sublimation chamber. On completion of sublimation, vacuum was released and the freeze-dried samples were withdrawn from the freeze-dryer. The powders were finally sieved through 100 mesh sieve.

3.2.5. Mechanical Alloying method :

Mechanical alloying (MA) of Ag-15 wt% CdO and Ag-10.8 wt% ZnO powders was done in an attritor mill fabricated indigenously as shown in Fig. 3.6 (a) and (b). The design aspects of attritor and the operating parameters are listed in Table 3.3.

The starting powders of Ag and CdO or Ag and ZnO were blended and sieved as per the process steps discussed in section 3.2.1. The sieved powders were then annealed at 300° C for 4 hrs in air in an oven. The annealed powders were subjected to MA with 2% methanol as PCA. Ag-ZnO powders were subjected to MA under two different conditions of attrition viz. 180 rpm speed and 11:1 ball to charge ratio ; and 300 rpm speed and 17:1 ball to charge ratio whereas Ag-CdO powder was subjected to MA at 300 rpm and 17:1 ball to charge ratio. After MA, powders in each case were sieved through 100 mesh sieve, annealed at 550° C for 90 min. in air in muffle furnace and once again sieved through 100 mesh sieve. Samples were drawn at predetermined time intervals during attrition milling to monitor the process of mechanical alloying by subjecting them to XRD.

3.3. Powder characterization:

3.3.1. Chemical analysis :

Both Ag-CdO and Ag-ZnO composite powders synthesized by different processing routes were subjected to chemical analysis for estimation of Cd and Zn , respectively.

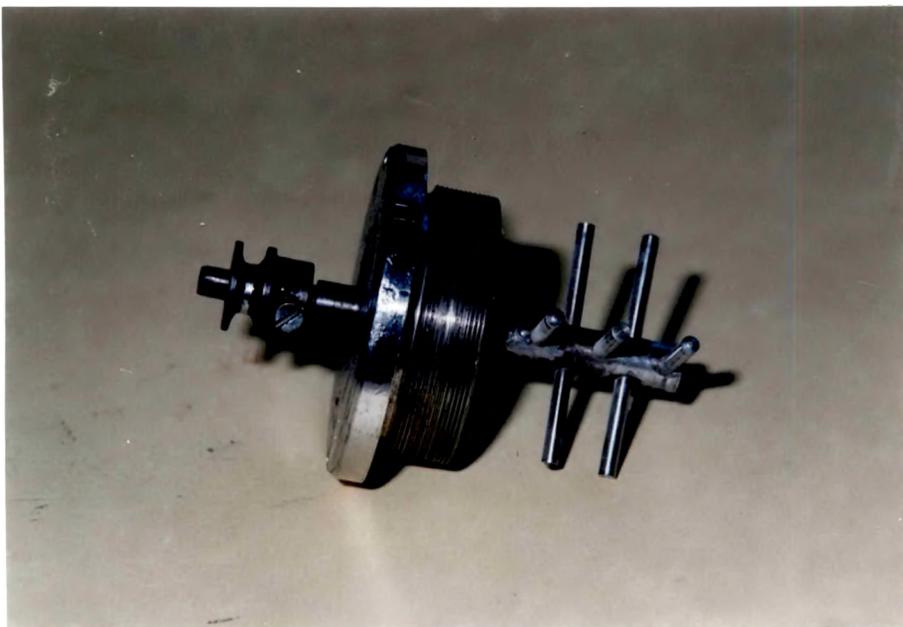
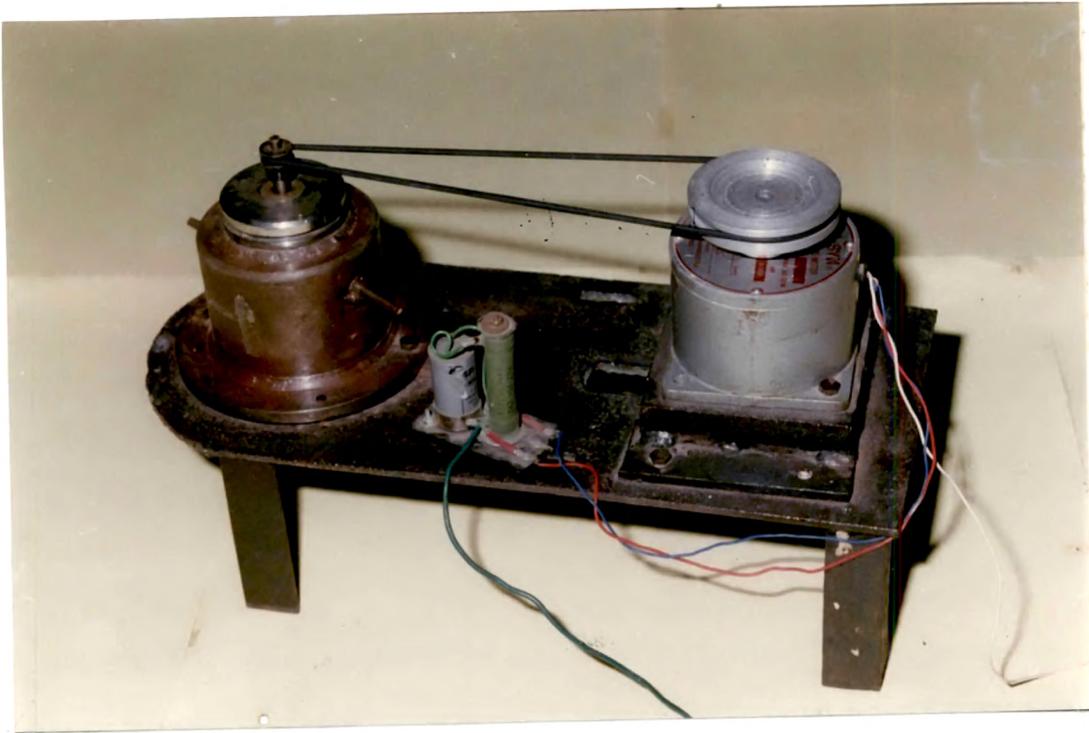


Fig. 3.6 : Photograph showing design aspects of attritor

Conventional methods of EDTA titration were adopted for this purpose as they are accurate and quick [137].

Table 3.3 : Design and operating parameters of attritor

Parameter	Dimensions
Attritor vessel capacity (cc)	130
I.D.X. useful height of attritor vessel (mm)	59.5 dia. x 47.0 ht.
Attritor shaft dia. (mm)	10.3
Attritor shaft useful height (mm)	45.0
Impeller dia. (mm)	4.0
Number and size of impellers (mm)	10, 22.5
Grinding bodies dia. (mm)	6.3
Ball to charge ratio	11:1 and 17:1
Attrition speed (rpm)	180 and 300
Material of attritor vessel	S.S. 304
Material of grinding balls	AISI 52100 steel
Material of impellers	AISI 4340 steel
Groove dia. of drive pulley (mm)	53.7 and 75.7
Groove dia. of driven pulley (mm)	12.0
Drive specifications	0.008 HP, 230V A.C. 60 rpm synchronous motor with 10 kg-cm torque
Batch weight (g)	15

3.3.2. Na and K Estimation by AAS :

The trace impurities of alkali metals like Na and K play a vital role in governing the contact properties and hence they are estimated by atomic absorption spectrometry. The powder samples of Ag-CdO and Ag-ZnO were dissolved in dilute nitric acid solution and were used for estimation of Na and K in ppm level by using Perkin Elmer's model 2380 Atomic Absorption Spectrometer (AAS). During the course of measurement on test samples, standard samples of known concentrations of Na and K were also run periodically for the purpose of verification.

3.3.3. X-ray diffraction (XRD) analysis :

For the purpose of qualitative analysis and the identification of phases present, the composite powder samples were subjected to x-ray diffraction (XRD) on Rigaku Giegerflex D-max (Japan) model of x-ray diffractometer. The diffraction profiles were taken using Cu target and $\text{CuK}\alpha$ radiation of 1.54 \AA wavelength. The powder samples were by and large scanned within the 2θ range of 30° and 70° at a scan speed of 3° per min. and a power rating of 40KV and 20 mA.

3.3.4. Apparent density and tap density of powder :

The apparent density of silver-metal oxide composite powders was estimated using Carney funnel as per the recommendations of ASTM standard B 417.

For tap density measurement, 25 g of Ag-MeO powder sample was taken to $\pm 0.01\text{g}$ accuracy. The powder was then poured into a clean, dry graduated cylinder, taking care that a leveled surface of the powder was obtained. The powder was then settled in the cylinder by mechanical tapping on the base-plate of Ro-tap machine. The tapping was

continued until no further reduction in the volume of the powder was observed. The volume of the fully densified powder in the graduated cylinder was read and was used to calculate tap density ρ as :

$$\text{Tap density } \rho = \frac{M}{V}$$

where M = mass of the powder in grams

V = volume of tapped powder in cubic centimeters.

3.3.5. Particle size analysis :

Particle size and size distribution of final powders of Ag-CdO and Ag-ZnO processed by different processing routes were measured with Galai CIS-1 Computerized Inspection System. Glycerol in distilled water was used as dispersing agent. Proper external ultrasonic stirring of powders was done to obtain a uniform colloidal dispersion of powder particles in liquid media. Particle size analysis was obtained as probability number distribution with particle size in microns on log scale on X-axis and the cumulative percentage undersize on Y-axis.

3.3.6. Powder morphology by SEM :

SEM observations on powder samples were carried out using JEOL-JSM-T300 Scanning Electron Microscope at 20 KV. Morphological investigation of powder samples was done by dusting the powder samples on double-sided adhesive tape and mounting on the sample mounting stubs of the SEM. The samples were given a thin coating of gold by means of sputter coater of the SEM. The final samples were examined at various magnifications upto a max. of 5000 X.

3.3.7. ESCA analysis for electroless coated powders :

Dispersion of silver on cadmium oxide and zinc oxide surfaces of electroless coated Ag-CdO and Ag-ZnO powders was studied by surface sensitive technique ESCA. A Vacuum Generator's ESCALAB MK II Spectrometer with $MgK\alpha$ radiation source (1253.6 eV) was used for recording of x-ray photoelectron spectra (XPS). Powder samples dusted on double-sided adhesive tape and mounted on sample holder were analysed using x-ray source of 100W (10 KV x 10 mA) power at a vacuum better than 5×10^{-8} mbar during analysis. The spectrometer was calibrated using photoelectron line at 932.7 eV which gave the binding energy of C (1S) photoelectron line from pump oil contamination at 285.0 eV. This C (1S) at 285.0 eV was used as reference for charge correction. Photoelectron spectra were collected using an Apple II e microcomputer interfaced to the spectrometer, which permitted signal averaging on repeated scans to improve signal to noise ratio. The data stored as a series of data files on floppy disks, were analysed by a second Apple II e microcomputer. Reported values of binding energies for Ag(3d), Cd(3d) and Zn(2p) for photoelectron lines are accurate to ± 0.01 eV and are average of atleast three different runs.

3.3.8. DTA/TGA studies :

Spray-coprecipitation technique and freeze-drying routes gave rise to formation of precipitates of mixed carbonates or bicarbonates of Ag and Cd or Ag and Zn at an intermediate stage of powder processing. They are further decomposed by firing to yield silver-cadmium oxide and silver-zinc oxide composite powders. Representative samples of mixed carbonates of Ag and Cd as well as Ag and Zn were therefore subjected to DTA/TGA on Seiko SSC 5200 and TG/DTA 220 system of Seiko Instruments Inc., Japan upto a max.temperature of 800° C to study their decomposition behaviour. The

analysis was done at a heating rate of 10° C per min. High purity alumina was used as reference and normal air was used as the atmosphere during analysis.

3.4. Compaction and sintering of powders :

Ag-MeO composite powders were pressed on a 100 Ton capacity hydraulic press of M/s.Lawrence & Mayo (India) Pvt. Ltd.,Bombay, model LM-17-510.

Green compacts of 15mm dia. x 1.5mm ht. and 8.3mm dia x 1.5mm ht. were pressed at 400MPa pressure using two different die-sets of WPS grade of steel having following dimensions :

<u>Parameter</u>	<u>15mm dia.die</u>	<u>8.3mm dia. die</u>
Bore dia. (mm)	15.00	8.30
O. D. of die (mm)	80.00	75.00
Height (mm)	60.00	55.00
Punch dia. (mm)	14.95	8.28
Punch ht. (mm)	60.00	55.00
Bottom pin dia. (mm)	14.95	8.28
Bottom pin ht. (mm)	14.00	14.00

15mm dia. samples were used for evaluation of bulk physical properties whereas 8.3 mm dia. samples were used for electrical contact property evaluation. Two-layer compacts of Ag-MeO powders backed with silver powder were pressed using 8.3mm

dia. die to facilitate brazing of the final contact tips to copper backings. The green compacts were subjected to green density measurements.

Both 15mm dia. and 8.3mm dia. green compacts were sintered in a tube furnace fitted with nichrome heating element at 930°C in air for 60 min. M/s. Wild Barfield Ltd., UK make tube furnace (model TM3) equipped with a microprocessor based temperature programmer controller (model WEST 2050) of M/s. Toshniwal Brothers (Bombay) Pvt. Ltd. at a heating rate of 6°C per min. was used. Fig. 3.7 shows the photograph of the sintering furnace alongwith programmer controller. The sintered compacts after evaluation of their physical properties, were subjected to repressing at 830 MPa pressure using the same die sets to improve their density. Repressing was followed by annealing of compacts at 600°C for 30 min. in air in a muffle furnace.

3. 5. Hot pressing of sintered compacts :

The 15mm dia. sintered compacts were hot pressed at 585 MPa pressure & 400°C temperature by using hot pressing die assembly fabricated for this purpose. Fig. 3.8 shows the photograph of the hot pressing set-up. A 15mm bore dia. die of H 11 grade of die steel with a heating-pad wrapped around it was used. The overall dimensions of the press tools were 50.0 mm dia. x 54.0 mm ht. of die, 14.40 mm dia. x 55.00 mm ht. punch with a bottom pin of 14.40 mm dia. x 15.0 mm ht.. The temperature of hot pressing was controlled by means of a SUNVIC type relay control switch. The settings of the SUNVIC control and the corresponding temperatures inside the die were pre-calibrated before the use of hot pressing system.



Fig. 3.7 : Photograph of sintering furnace along with temperature programmer-controller.

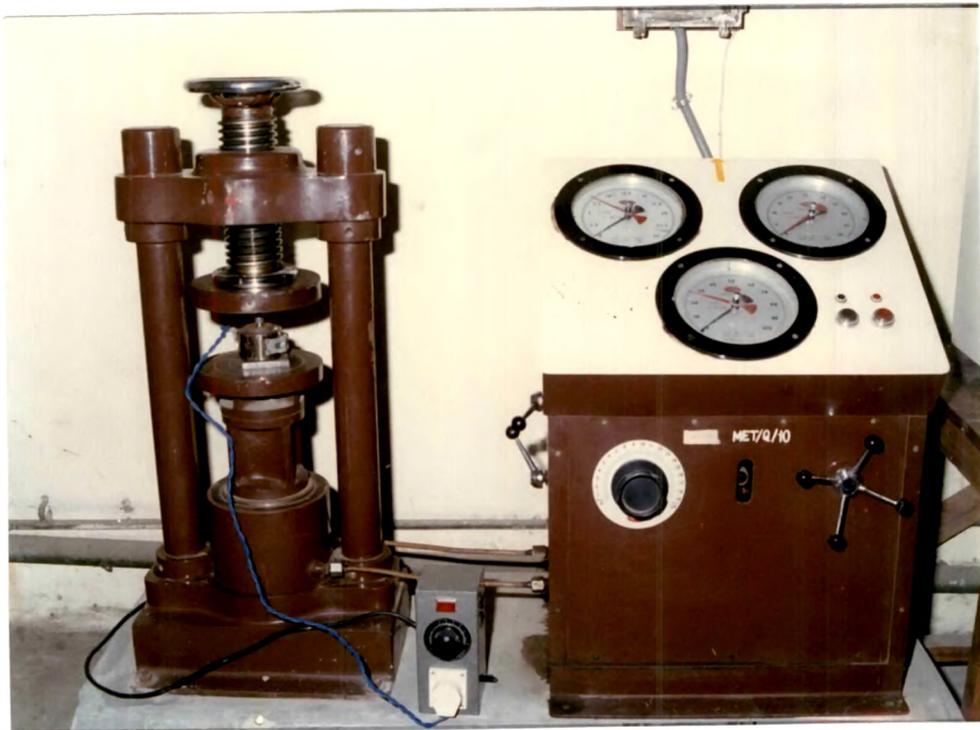


Fig. 3.8 : Photograph of the hot-pressing set up.

The 8.3mm dia. sintered and repressed compacts were hot pressed by first heating them in a muffle furnace to 600°C temperature for 5 min. after which they were quickly transferred to die and hot pressed.

Finally, hot-pressed compacts were subjected to property evaluation.

3. 6. Structure and property evaluation :

3. 6.1. Density measurement :

The density of Ag-MeO compacts was measured after compaction, sintering and hot pressing. The density was found by dividing measured weight of compact by its volume. Since the samples in this investigation had geometrically simple shape (that of a pellet or cylinder), precise estimation of density was possible in this way. Few representative sintered and hot pressed samples were subjected to density measurements as per the procedure based on Archimede's principle for a crosscheck. Such measurements gave marginally higher values of densities than the former. The results of density measurement comprise of actual density and percentage theoretical density for respective composition.

3. 6 .2. Microhardness estimation :

Microhardness data for sintered and hot-pressed Ag-MeO samples was obtained on ground and polished samples using NEOPHOT microhardness attachment at a load of 65 grams using diamond indenter. Samples used for optical microscopy have been used for this purpose. Vickers hardness number (VPN) is calculated by the following equation

$$VPN = (1854 \times P) / (d \times 0.28)^2$$

where P = load applied in grams

d = indent diagonal in mm

An average of 3 - 4 measurements was taken for each sample.

3. 6.3. Electrical conductivity measurement :

The electrical conductivity was measured on 15 mm dia. as-sintered and final hot pressed compacts using Type 757 conductivity meter of M/s. Technofour Ltd., Pune based on the principle of induced eddy currents. Same optical microscopy samples were used for these measurements to ensure proper contact between the measuring probe and the surface of the test sample for reliable and consistent results. The meter reads the conductivity directly in % IACS units. The consistency and reliability of results was confirmed by periodic verification on standard samples of known conductivity.

3. 6.4. Optical microscopy :

Final hot-pressed compacts of 15 mm dia. were subjected to optical microscopy with a view to study morphology and dispersion of oxide phase in silver matrix. The 15 mm dia. Ag-CdO and Ag-ZnO samples were polished on emery papers of successively fine grit size. Followed by polishing on emery papers, samples were polished on 1 micron size diamond paste on napless polishing cloth using kerosene. The polished samples were washed and thoroughly cleaned with acetone and then examined under UNION microscope at magnifications ranging from 100x to 500x. The samples were examined and photographed in un-etched condition only. Utmost care was taken during photography to minimise the effect of shadowing developed as a result of high reflectivity of polished surfaces of silver.

3.6.5. Image analysis of final compacts :

In order to quantify the morphology and degree of dispersion of oxide phase in silver matrix, polished samples of silver-metal oxide were subjected to image analysis using Leco 300 Metallograph coupled with Leco 2001 Image Analyser of Leco Instruments Inc., USA. Imaging was done at 1000x magnification for Ag-15 wt % CdO and Ag-10.8 wt % ZnO final hot-pressed compacts processed by different processing routes. The samples processed by conventional PM route (involving blending) were, however, examined at 500x. The image analysis constituted estimation of oxide phase count leading to area and volume fraction; measurement of degree of roundness of oxide phase indicative of shape morphology and the feret measurements of oxide phase particles revealing the isotropy or anisotropy of oxide phase.

3.7. Effect of lithium addition to Ag-ZnO system :

Role of lithium as an additive to silver-cadmium oxide contact material has been well documented in the literature as reported in section 2.6.1. Similar studies were planned to investigate the effect of Li on Ag-ZnO system. Lithium was added as lithium nitrate dissolved in methanol to spray-coprecipitated Ag-10.8 wt % ZnO powder. 0.5 wt% LiNO₃, 1.0 wt % LiNO₃ and 2.0 wt % LiNO₃ were added to stoichiometric amounts of spray-coprecipitated Ag-10.8 wt % ZnO powders in three different beakers and stirred ultrasonically for 5 min. to ensure uniform dispersion. The methanol was evaporated and LiNO₃ coated Ag-ZnO powders were then sieved through 100 mesh sieve. These powders were then subjected to AAS, ESCA, particle size analysis and XRD measurements. These powders were subsequently pressed to 50% theoretical density at 10 MPa pressure to get 15 mm dia. green compacts. The green compacts were first subjected to decomposition of LiNO₃ to Li₂O by their slow heating to a temperature of 650°C followed by holding at this temperature for 120 min. Finally the compacts were

heated to 930° C for 60 min. in air in the same sintering furnace. The sintered compacts were hot-pressed at 585 MPa pressure in hot pressing die at ~ 400° in air. The compacts were subjected to measurements of density, microhardness, electrical conductivity, optical microscopy and image analysis at various stages as per procedures discussed earlier.

3. 8. Life testing of contacts :

3. 8.1. Design and fabrication of life testing set up for AC contacts :

Two types of test facilities were developed for life testing of Ag-MeO contacts in AC mode. One consisting of performance evaluation on a pair of contacts and another on an electrical device such as a contactor.

Test set up for a pair of contacts :

In this set up the contact make and break operation was ensured by means of a vibration generator (of M/s. Industrial Engg. Instruments, Bangalore) housing an electromagnet which was energized by a power oscillator (of M/s. Industrial Engg. Instruments, Bangalore). The to-and-fro vertical movement of the electromagnet's spindle ensured making and breaking of the moving contact with respect to the fixed one. The contact tips of 8.3 mm dia. x 1.5 mm ht. of Ag-MeO/Ag double-layered type were brazed to EC grade high conductivity (99 % IACS value) copper lugs using M/s. Advani Oerlikon's A - 316 low - melting silver alloy filler rod alongwith F - 316 flux. The lugs alongwith tips were gripped inside a EC grade copper sample-holder having split-type design. The moving contact tip alongwith the sample holder was electrically isolated from vibration generator assembly by means of a teflon insulator. The fixed contact tip was also fitted in the sample holder in a similar manner. A cross-hole of 1.5 mm dia. at a distance of 5

mm below the working face of fixed contact was drilled to insert a PT 100 RTD thermocouple for temperature rise measurements. The gap between the fixed and the moving contact could be adjusted by means of a threaded stud of 16.5 mm dia. x 115.0 mm length which acted as gap trimming mechanism. A teflon insulator piece was inserted between the sample-holder and the stud to ensure the electrical isolation from the rest of the system. The entire mechanical assembly was done using slotted angles. A top plate holding the gap trimming stud and having the adjustable holes was used for final alignment of fixed and moving contacts before the start of make-break operations.

The power oscillator coupled to the vibration generator is designed to vary the frequency of vibration from 1-10KHz. The amplitude of vibrations (and thereby the stroke length of moving contact and hence the making force) can be varied within a certain limited span using the amplitude control knob provided on the front panel of the power oscillator. The contact making force can be measured by using a load cell of 0-10 N capacity (of M/s. Industrial Engg. Instruments, Bangalore) which is coupled to a

Table 3.4 : Measured load versus amplitude setting

Amplitude	Load in N
0.0	0.2
0.5	0.4
1.0	0.6
1.5	0.9
2.0	1.1
2.5	1.3
3.0	1.6

digital load indicator (M/s. Industrial Engg. Instruments, Bangalore). Prior to the use of the system, the making load value for different amplitude settings can be predetermined by fixing the load cell between the fixed contact and the gap trimming stud. Fig.3.9 shows the photograph for the load measurement and Table 3.4 gives the values of measured contact making load for different amplitude settings.

Terminals are provided on both sample holders, housing fixed and moving contacts for connection to 230 V AC supply, through a suitable load such as resistive load. Fig.3.10 shows pictorial view of this system and Fig. 3.11 shows the photograph of the brazed contact tips alongwith copper lugs.

Test set-up for testing a contactor :

Facilities were also developed to test the pressed- sintered- hot pressed contacts of different process routes by installing them on a commercial contactor. For this purpose a 32 A / 230 V / single phase AC commercial contactor of L & T make model ML-2 with a coil voltage of 230 V was used. The original contact tips already existing on the mounting base of the contactor were removed by grinding them on a wheel grinder and in their place our tips were brazed as per details given earlier. The brazed tips were given a strike silver plating in an electroless mode by giving them a dip in the silver plating bath of following composition for 5 min.

Silver cyanide : 7.5 g per litre.

Potassium cyanide : 15.0 g per litre.

The brazed contact assemblies were then thoroughly washed, cleaned with acetone, dried and weighed precisely on a microbalance (M/s. Anamed Instruments Pvt. Ltd., Bombay ; model EAB - 2200) and finally installed on the test contactor. Fig. 3.12 shows the photograph of the contactor, housing our contact tips and a single contact tip, separately.



Fig. 3.9 : Photograph of the contact force measurement set-up

1. Load Cell
2. Load indicator
3. Electromagnetic vibrator
4. Power oscillator



Fig. 3.10 : Photograph of the contact testing set-up for a pair of contacts.

1. Fixed contact
2. Moving contact
3. Electromagnetic vibrator
4. Power oscillator
5. Resistive load

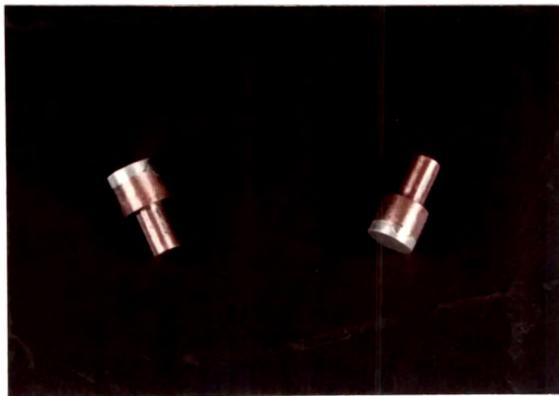


Fig. 3.11 : Photograph of the brazed contact tips alongwith copper lugs.

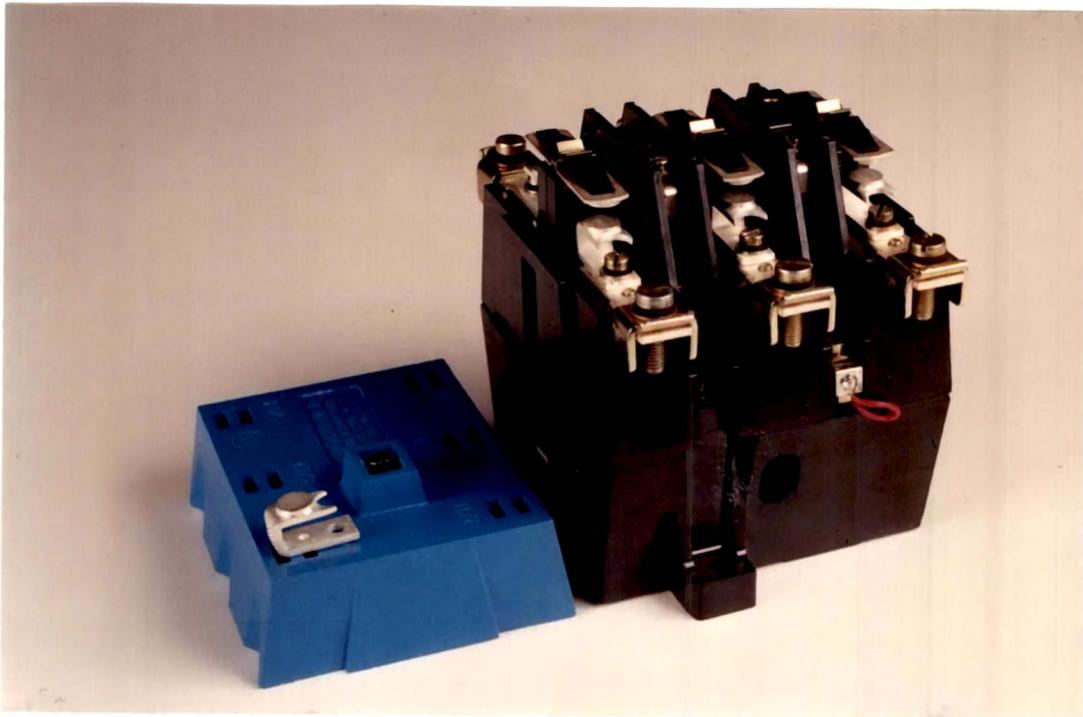


Fig. 3.12 : Photograph of the test-contactor assembly and a single contact tip shown separately.

The input power to the contactor was 230V AC single phase drawn through a resistive load of 23 ohms per phase. The resistive load consisted of Kanthal wire of 1.2 mm dia. in the form of a coil of 42 mm dia.x 370 mm length. Out of the three pairs of contacts (six fixed and six moving), only two pairs (i.e.four fixed and four moving contacts) were used as test-contacts, whereas third pair was used for connections to 8085 microprocessor based control circuit. Fig.3.13 shows the circuit diagram of the AC testing set up for testing of contactor and Fig. 3.14 shows the pictorial view of the same. The detailed program flowchart is given in Fig.3.15 and the actual assembly language program used has been given in Appendix-A3. The program has a provision to change ON time, OFF time; the continuous ON period at the end of stipulated number of make-and-break operations, etc. The ON and OFF times can be varied from few milliseconds to several seconds. The continuous ON period given at the end for temperature rise measurements may also be varied from few minutes to an hour. The program is equipped to set the desired number of make-and-break operations & the operating frequency. The system is also equipped to display the number of failures due to contact welding during the course of make-break operations. The loading of input parameters and program control is done through 8085 microprocessor kit of M/s .Dynalog Systems Pvt. Ltd., Ahmedabad. The set up is designed to carry out the contact testing at 10A/230V AC single phase at a power factor of 1.0.

A circuit was also developed for the measurement of contact resistance in terms of mV drop using a PT 100 RTD thermocouple and a digital multimeter. Fig. 3.16 shows the circuit diagram of the set up used for mV measurement at fixed contact.

3.8.2. Design and Fabrication of life testing setup for DC contacts :

Set up for DC contact testing on a contactor was developed by making some modifications in the AC contact test set up described in section 3.8.1. The input power

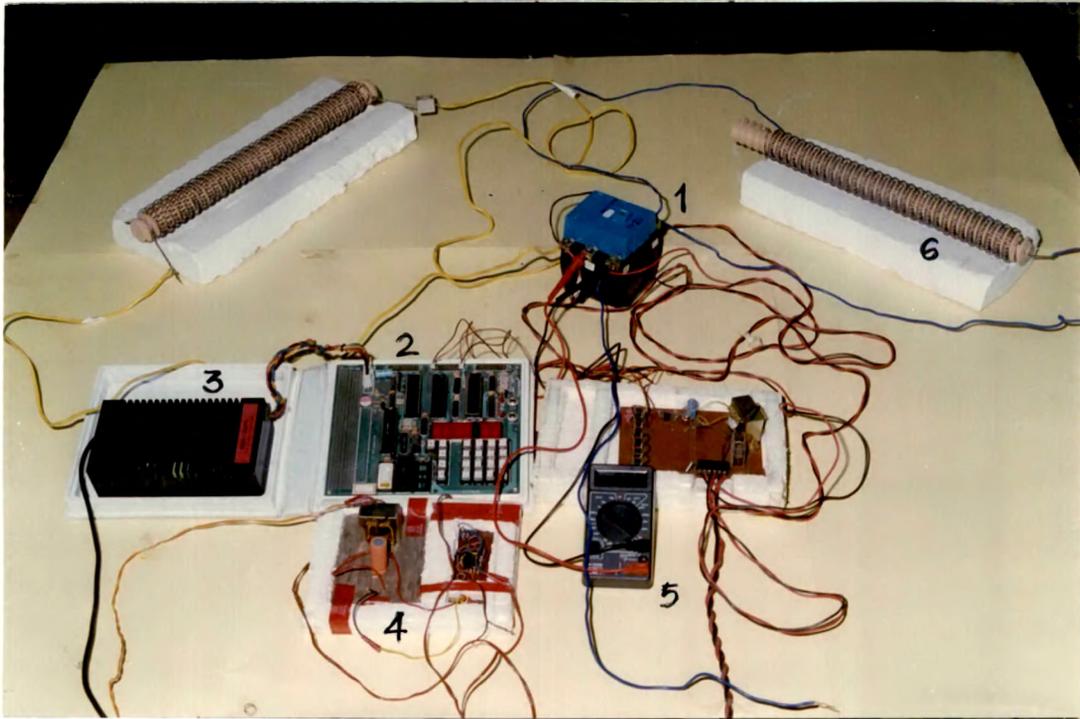


Fig. 3.14 : Pictorial view of the AC contact testing set-up.

1. Test contactor
2. Microprocessor Kit
3. Power supply
4. mV measurement circuit
5. Millivoltmeter
6. Resistive Load

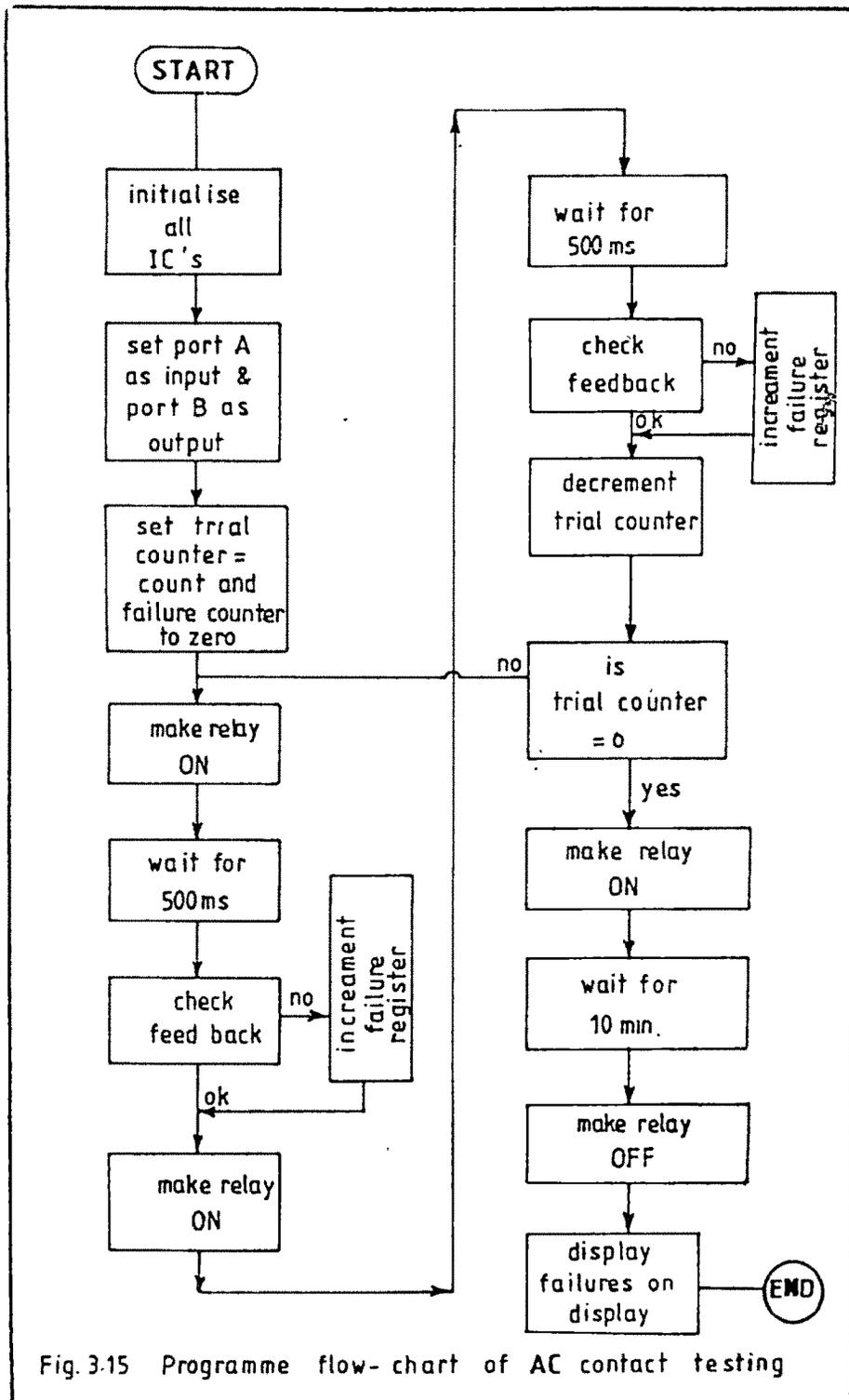


Fig. 3.15 Programme flow-chart of AC contact testing

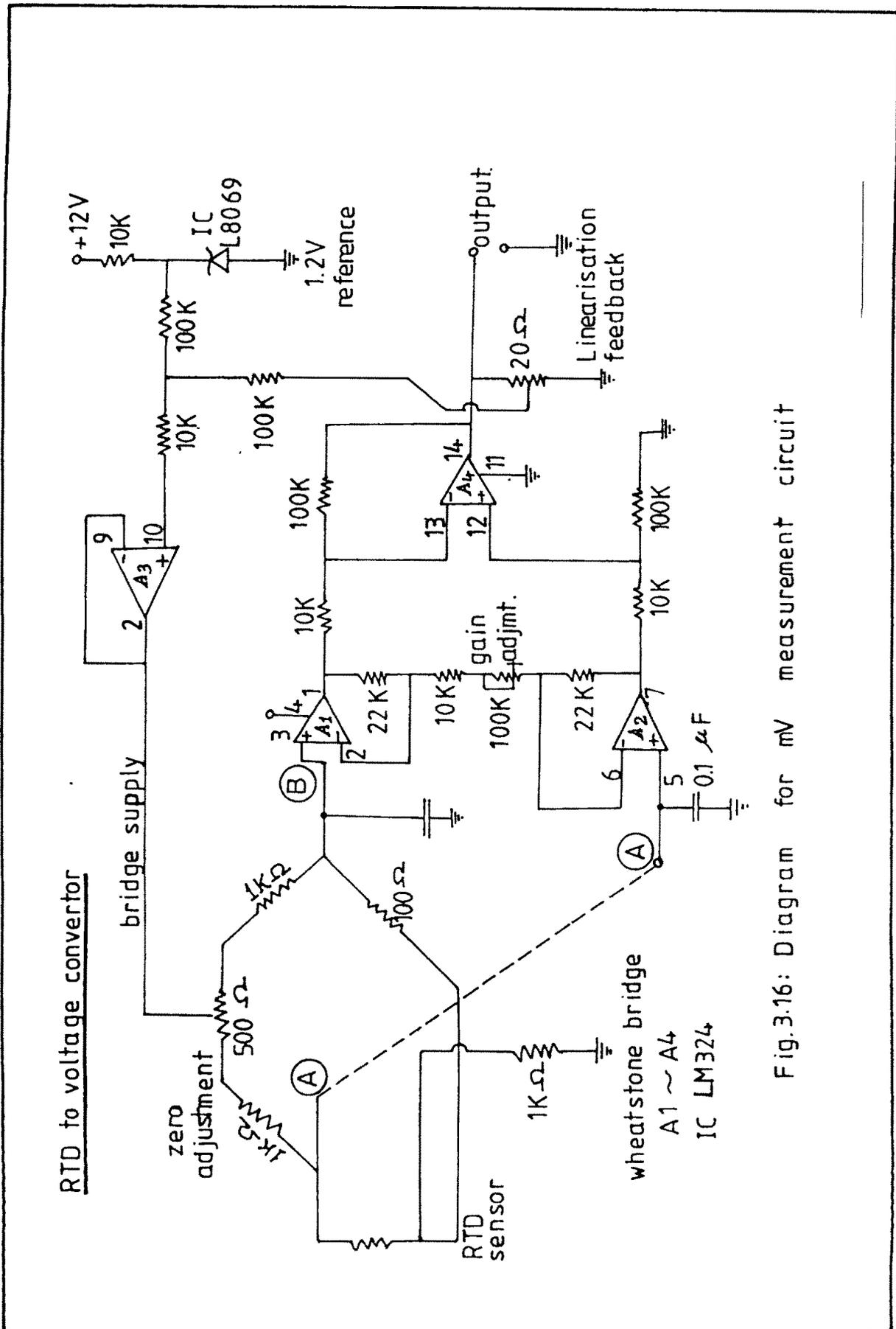


Fig.3.16: Diagram for mV measurement circuit

to the DC test contactor was a low voltage-high current supply subjected to rectification. This was achieved by installing an AC transformer designed and developed for this purpose. The transformer has input rating of 230V AC single phase with tapings for drawing 23V-20A, 23V-40A, 23V-60A and 23V-80A AC output from its secondary. This low voltage high current AC supply from transformer secondary was subjected to DC rectification by means of a rectifier circuit consisting of a bridge rectifier alongwith heat sink. The DC rectification was however done only for 23V-20A output rating in view of high cost of rectification. However, it is possible to expand it upto 23 V-80 A as and when needed.

The load for DC testing consisted of a load bank of four numbers of Kanthal wire coils of 1Ω resistance each having the wire diameter of 2.7 mm and the overall coil size of 28 mm dia.x 300 mm length each. The provision is made in the design to expand the capacity of resistive load by an additional 25%. Rest of the control circuit is same as that used for AC testing. Photograph of DC test set up is given in Fig. 3.17.

3.8.3. Life testing of AC contacts :

Contacts were evaluated for their electrical performance (i) on the set up developed in the laboratory (ii) at M/s. Jyoti Ltd., Baroda (iii) at National Metallurgical Laboratory, Jamshedpur and (iv) at Electrical Research and Development Association (ERDA), Baroda.

(i) Life testing (endurance test) was done on the set up developed as per section

3.8.1. for contacts developed by following processing routes.

- (a) Ag-7.1 wt % ZnO by spray-coprecipitation
- (b) Ag-7.1 wt % ZnO by electroless coating
- (c) Ag-10 wt % CdO by conventional PM route.



Fig. 3.17 : Photograph of the DC contact testing set-up

1. Test contactor
2. Microprocessor kit
3. Power supply to kit
4. mV measurement circuit
5. Millivoltmeter
6. Resistive load
7. Step-down transformer
8. Bridge rectifier

The test parameters were as under :

Test current	:	10 A
Test voltage	:	230V AC single phase
Power factor	:	1.0 (resistive load)
No. of make-break operations	:	1×10^5
Frequency of operation	:	2700 operations per hour
ON time	:	500 ms
OFF time	:	830 ms

For estimation of weight loss due to erosion during make and break, the contacts alongwith the mounting base were first precisely weighed on microbalance prior to commencement of the test and then periodically after every 12,500 operations. At the end of 12,500 operations the rated current of 10A AC was passed for 10 min. continuously and the mV were measured by placing the tip of the PT 100 RTD thermocouple just adjacent to the fixed contact. This measurement was done using the millivolt measurement circuit and the digital multimeter. The current and the voltage at various points in the circuit during the test were varified using a clip-on meter.

(ii) Ag-8.6 wt % ZnO contacts processed by spray-coprecipitation and electroless coating routes were subjected to similar life testing (using a similar contactor) at R & D Centre's Switchgear Laboratory of M/s. Jyoti Ltd., Baroda on their life testing rig.

Test current	:	30A AC
Test Voltage	:	415V AC 3 phase
Power factor	:	0.95
No. of make break operations	:	1×10^5

Frequency of operation : 1200 operations per hour.
 ON time : 100 ms
 OFF time : 2.9 sec.

The erosion studies were done in the same manner as stated above by periodic weighing of contacts at every 10,000 operations interval. The contact resistance was measured after every 20,000 operations using a digital micro-ohm meter of Motwane, model LR - 205, at 1A DC. Measurements were done on all the six fixed contacts. Besides this a temperature rise test of contactor at 30A AC at a rated voltage of 4V single phase with all the main contacts connected in series was carried out. This test was carried out after zero, 50,000 and 1,00,000 operations during electrical endurance test. Rated current of 30A was passed continuously for 6 hrs. each time and the temperatures were measured at various strategic points on the contactor by means of a digital temperature indicator with a specially developed contact type sensor for this purpose.

(iii) Ag-10.8 wt % ZnO contacts processed by spray-coprecipitation and electroless coating routes were evaluated at NML, Jamshedpur as per following conditions :

Test current : 15 A
 Test voltage : 220V AC single phase
 Power factor : 1.0
 No. of make-break : 1×10^5
 operations
 Frequency of operation : 1 per sec
 ON time : 500 ms
 OFF time : 500 ms

The endurance test was carried out by installing the contacts on a standard contactor of Cutler Hammer India Ltd. of 16 A / 220 V AC rating. Fig. 3.18 shows the pictorial view

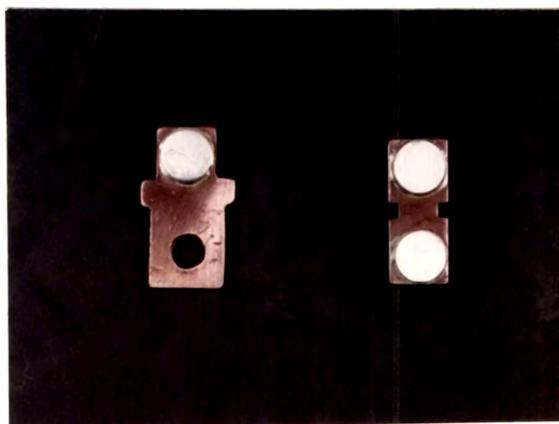


Fig. 3.18 : Photograph of the brazed contact assembly tested at NML

of contacts brazed on copper base plates used in this testing. The erosion measurements were done by weighing the contacts after every 25,000 operations and finding the weight loss in milligrams. Contact resistance measurements in milli ohms were done using a 10A DC source and the mV drop across the contacts was measured.

(iv) Ag-10.8 wt % ZnO contacts processed by Mechanical Alloying (MA) route were also subjected to life test as per IEC 947 - 4 - 1 (1990) for AC-3 utilization category at ERDA, Baroda. Testing was done by mounting our contact tips on a commercial contactor as stated in section 3.8.1.

The following tests were conducted :

1. Verification of temperature rise
2. Verification of ability to withstand overload currents
3. Verification of rated making and breaking capacities
4. Verification of conventional operational performance for 7000 operations at a rate of 1200 op.per hour.

The details of the contactors (2 nos.) used for these test are :

Make	: L & T] (with our contact tips of Ag-10.8 ZnO (MA) composition installed)
Type	: ML 2	
No. of poles	: 3	
Rated operating voltage (Ue)	: 415 V AC	
Rated operational current (Ie)	: 30 A	
Utilization category	: AC 3	
Conventional free-air thermal current	: 40 A	
Rated frequency	: 50 Hz	
Rated control supply voltage (Us)	: 240 V, 50 Hz	

Temperature rise test was conducted by passing conventional free air thermal current of 40 A through main circuit and measuring temperature at top and bottom terminals.

Overload current of $8 \times I_e$ i.e. equal to 240 A AC was passed for 10 sec. through the contactor for verification of ability to withstand overload currents.

Two separate tests were conducted for verification of making and breaking capacities

- a) making capacity test and
- b) making and breaking capacity test

(a) Making capacity test was conducted as per following specifications :

Applied voltage $U = U / U_e = (1.05 \pm 20\%) = 436 \text{ V}$

Applied current $I = I / I_e = (10 + 5\%) = 302 \text{ A}$

Power factor = $(0.45 \pm 0.05) = 0.424$

No. of operating cycles

25 with 85 % U_s

and 25 with 110 % U_s

On-time of test : 50ms

Off-time of test : 10 seconds

(b) Conditions for making and breaking capacity test were :

Applied voltage $U = 415 \text{ V}$

Applied current $I = I_c / I_e = (8 + 5\%) = 242 \text{ A}$

Power factor = $(0.45 \pm 0.05) = 0.441$

Recovery voltage $U_r = U_r / U_e = (1.05 + 5\%) = 436 \text{ V}$

On-time of test : 50ms

Off-time of test : 30 seconds

No. of operating cycles = 50

Similarly verification of operational performance capability was done under following set of conditions:

Applied voltage $U = (415 \text{ V} + 5 \%) = 415 \text{ V}$

Applied current $I = I_c / I_e = (2.0 + 5 \%) = 62 \text{ A}$

Power factor $= (0.45 \pm 0.05) = 0.471$

Recovery voltage $U_r = U_r / U_e = (1.05 + 5 \%) = 436 \text{ V}$

No. of operations = 7000 at 1200 op. per hour with 50 ms on-time

3.8.4. Life testing of DC contacts :

Ag-10.8 wt % ZnO and Ag-15 wt % CdO contacts processed by Mechanical Alloying route were subjected to life testing on a set up developed in the laboratory as described in section 3.8.2. The same type of contactor as used in earlier discussion was used for DC testing. DC testing was conducted as per IEC 947 - 4 - 1 (1990) for DC -1 utilization category.

The operating parameters of the test were :

Test current : 10A DC per pair of contacts.

Test voltage : 23V DC

Power factor : 1.0

No. of make-break : 7000 (minimum 6000 operations as per
operations IEC specifications)

Frequency of operation : 360 operations per hour

ON time : 500 ms

OFF time : 9.5 sec.

Erosion rate was measured by weighing the contacts after every 1500 operations. Same way mV measurements were also done after every 1500 operations. The polarity of contacts was reversed after 3000 operations as per IEC specifications.