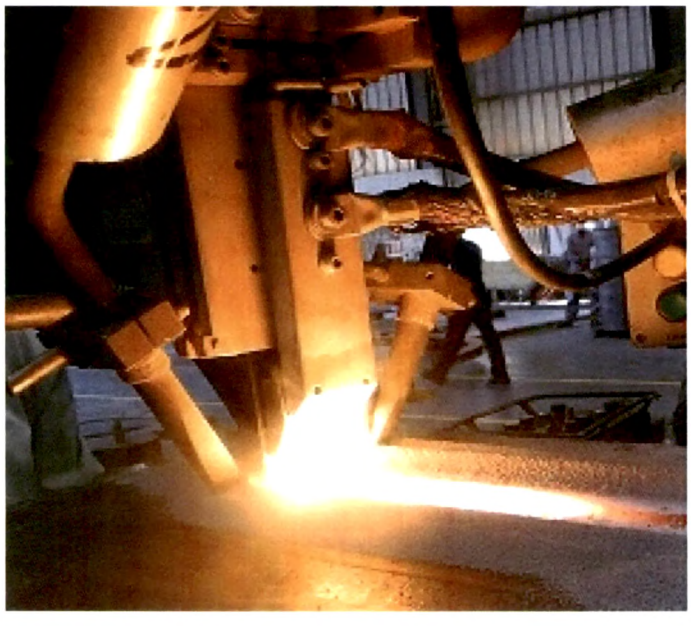
CHAPTER -3

EXPERIMENTAL WORK



Experimental Procedure:

3.1 Development of weld overlays:

The base material of 55 mm thick plate of Cr-Mo steel (SA387 Gr11 Cl2) received in the normalized, accelerated cooled and tempered condition. Chemistries of the parent metal and the electrode strips are listed in table-3.1. As shown in fig. -1 first Strip was cut at an angle of 45^0, so it strikes the arc easily by developing higher current density at the tip of the electrode. Then it scratches on the base metal so initiate arcing between the electrode & the base metal. The small quantities of flux and strip start melting before traveling the welding head. As soon as sufficiently thick layer of molten slag is formed, arc was extinguished but strip electrode was continuously fed into a shallow layer of molten electro conductive slag. The heat which is needed to melt the strip, slag forming flux & the surface layer of base metal was generated by the joule effect as a result of the welding current flowing through the liquid electro conductive slag. The interior temperature of the bath is in vicinity of 1925^0 C while the surface temperature is approximately 1650^0 C .This feature ia shown in fig 3.1 & 3.2.

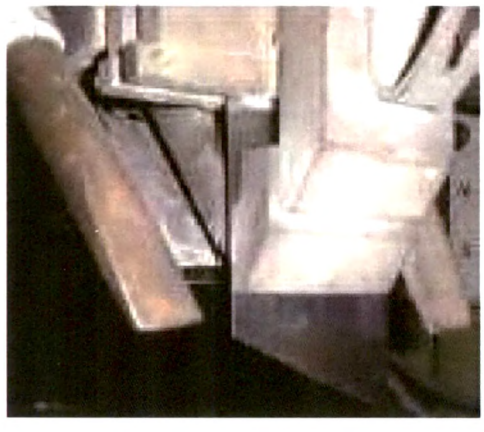


Fig 3.1 . Strip cut at 45^0 angle

Fig 3.2 Flux on both the side of the strip while staring the weld

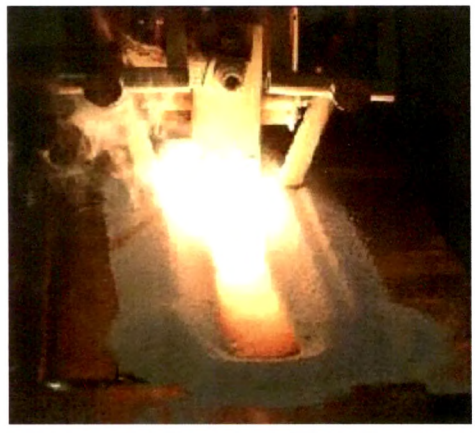


Fig 3.3 Continuous cladding by ESSC Process

Fig 3.4 weld overlay developed by ESSC Process

Table-3.1: chemical Composition of base metal and strip electrodes (weight%)

Type	C	Cr	Ni	Mo	Si	Mn	Nb	N2	P	S
Base Metal	0.15	1.2	0.16	0.5	0.6	1.2	0.001	-	0.003	<0.001
309L	0.005	21.0	11.0	<0.03	0.5	1.8	-	0.036	0.012	0.0005
309L Nb	0.008	21.0	11.0	<0.03	0.5	1.8	0.52	0.036	0.011	0.0006

As shown in fig. 3.4 single layer technique was adapted to developed weld overlays of 309 L & 309L Nb austenite stainless steel on Cr-Mo steel using electroslag strip cladding process with welding conditions which is tabulated in table- 3.2 & table- 3.3.

Table-3.2: Design of Experiments:

Exp. No	Strip Electrode	Welding Current (Amp)	Welding Voltage (volt)	Travel Speed (mm/min.)
1	Single layer 309L	1100-1300	24-26	160
2	Single layer 309L	1100-1300	24-26	180
3	Single layer 309L	1100-1300	24-26	200
4	Single layer 309L Nb	1100-1300	24-26	160
5	Single layer 309L Nb	1100-1300	24-26	180
6	Single layer 309L Nb	1100-1300	24-26	200

Table-3.3: Strip welding conditions:

Polarity	DECP
Electrode extension (Stick out)	35 mm max.
Height of flux	20-25 mm .
Preheat temperature	125^0 C min.
Interpass temperature	200^0 C max.

3.2 Sample Preparation:

- Cladded weld overlay has been cut in the form of slice of 10mm wide and it were taper grinded on milling machine at angle of 10-15 degree for various testing.

Fig. 3.5 Taper grinding of cladded samples

3.3 Post Weld Heat Treatment (PWHT) cycle for weld overlays

- It was performed to relieve the stresses of the base metal after cladding process. It was carried out in coil heating furnaces with temperature controlling devices which was also connected to graph plotting device as show in fig.3.6 & 3..7

Fig: 3.6 Coil Heating Furnace **Fig: 3.7 Graph Plotting Devices**

3.3.1 Post Weld Heat Treatment (PWHT) Cycle:

1. Rate of Heating from Room Temperature to 400°C: 100°C/Hr
2. Rate of Heating from 400°C to 690°C: 40°C/Hr
3. Soaking Temperature: 690°+14°C
4. Soaking Time: 30Hrs
5. Rate Of Cooling from 690°C to 400°C: 40°C/Hr
6. Switch off the furnace after reaching 400°C and allow it to cooling furnace itself.

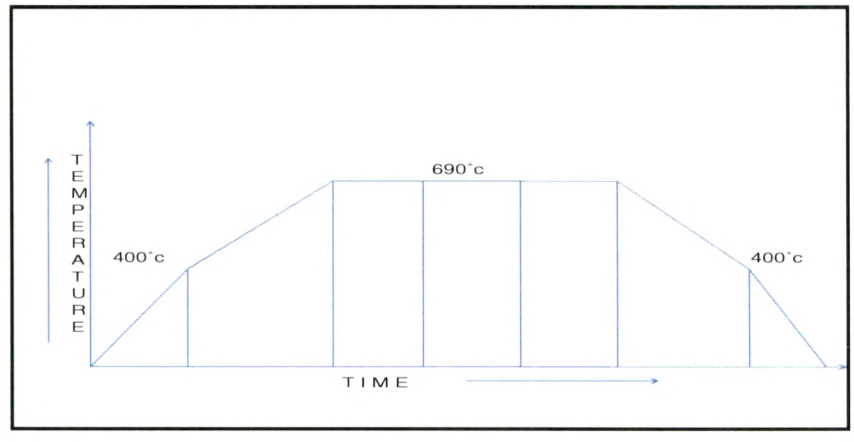


Fig 3.8 PWHT cycle for both weld overlays

3.4 Testing & Evaluation of weld overlays

3.4.1 Microstructure Evaluation of weld overlays

3.4.1.1 Optical Microscopic Analysis

- Neophot – 2 Microscope is used to observer micro structural changes at grain boundary as well as at interface using
- Etchant for Base Metal: Nital (98% Methanol & 2% HNO_3) solution.
- Etchant for cladded region: Aquzregia (75% HCl & 25% HNO_3) solution.
- All samples were viewed at 1000X Magnification.

3.4.1.2 Electron Microscopic Analysis

- To reveal various inter-metallic compounds SEM studied were performed on both the weld overlay while percentage element present were determine by EDAX analysis.

3.4.2 Determination of Ferrite Content of the weld overlays

- Ferrite content was measured for both weld overlays by using Fischer Ferrite-scope MP 30 for all types weld overlays samples.

3.4 .3 Determination of Hardness of weld overlays

- Hardness values at different location of weld overlay were performed with the help of Vickers Pyramid Hardness Testing method using Wilson Wolpert Micro Vickers – 401MVD Instrument with load of **100 Kg.** Hardness value determine by the used of standard chart with the help of load range and diagonal length of indentation.(see Fig.3.10)

Fig 3.9 Vicker Hardness Test

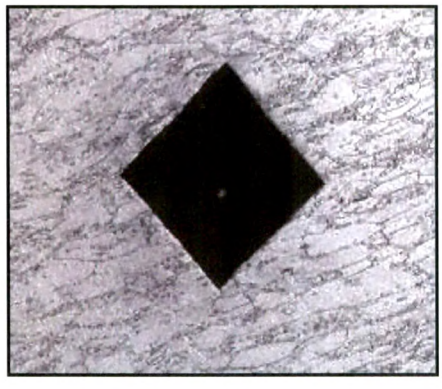


Fig. 3.10 Square base Indentation

3.4.4 Corrosion Testing of weld overlays :

3.4.4.1 Description of Instrument (Potentiostat/ Galvaneostat/ZRA)

- Model: EG&G PAR 273A & Gamry Potentiostate (reference600)
- Software: M273 & M398
- Attachment: Computer system for data storage
- Corrosion Cell consist of
  - Reference electrode: standard calomel electrode ($Ag/AgCl_2$)
  - Auxiliary electrode: graphite rod
  - Working electrode: test sample

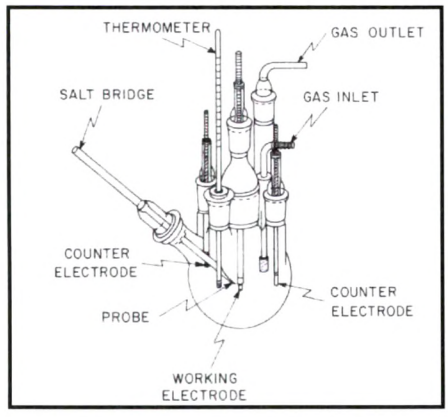


Fig. 3.11 Corrosion cell

Fig. 3.12 Experimental Setup of corrosion testing

3.4.4.2 Sample Preparation for corrosion testing

All samples has been masked using water proof cello tap to get exact exposure area (5mm x 5mm) for corrosion testing at base metal, interface and cladded Material separately. During testing of at one location other two locations were kept covered with masking tap.

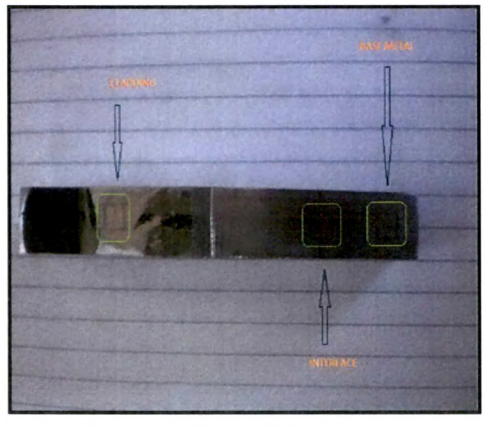


Fig.3.13 location of corrosion test

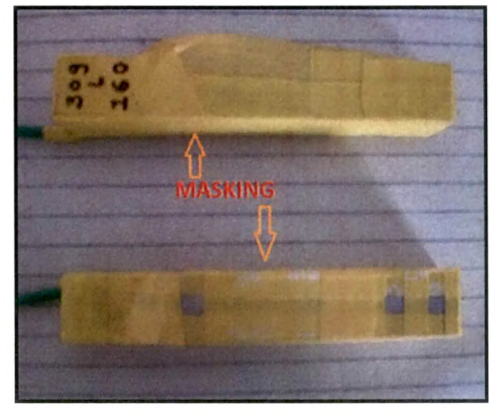


Fig. 3.14 Masking of Sample

3.4.4.3 Potentiodynamic Testing

Potentiodynamic studies were carried out on both weld overlays in 0.1 N HCl solution, 0.1 N HNO_3 solution, 0.1 N H_2SO_4 solution & 3.5 % NaCl solution as per ASTM G-5 standard, which is "Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements" using potentiostat of Gamry

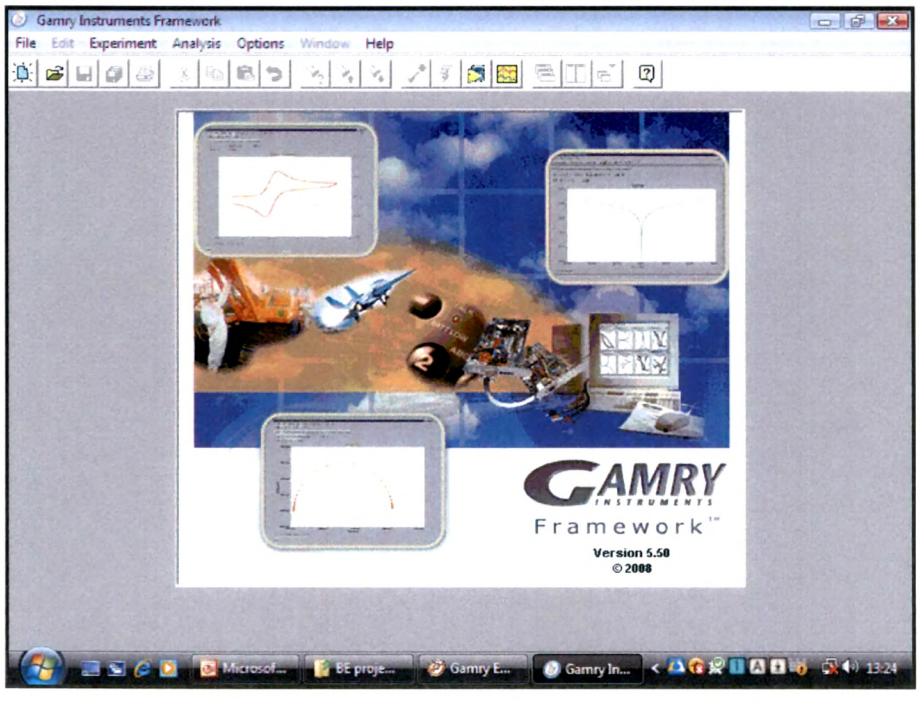
reference 600. Samples were subjected to anodic & cathodic polarization using following operating parameters which are tabulated in table no-3. 4.

Table - 3.4 : Operating Parameters for Potentio-dynamic test

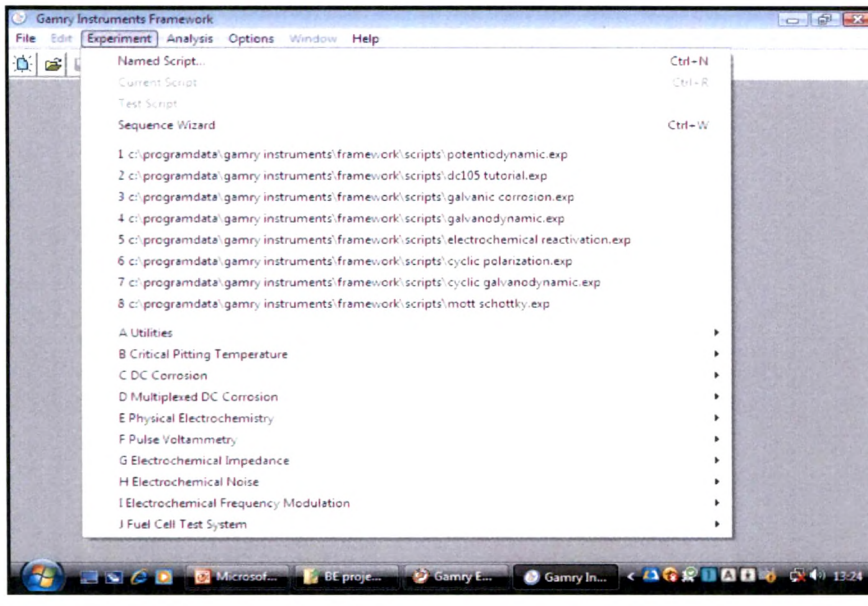
Sr.No	Potentio-dynamic test
1	Initial voltage: -0.5 V with respect to open circuit potential
2	Final voltage: 1.7 V with respect to reference electrode
3	Scan rate: 5 mV / sec
4	Conditioning time: 60 sec at -1.0 V
5	Initial delay : 60 sec
6	Sample exposure area: 0.25 cm^2
7	Density : 7.87 gm / cm^2
8	Equivalent weight: 22.92 gm

3.4.4.3.1 Sequence of Potentio-dynamic test:

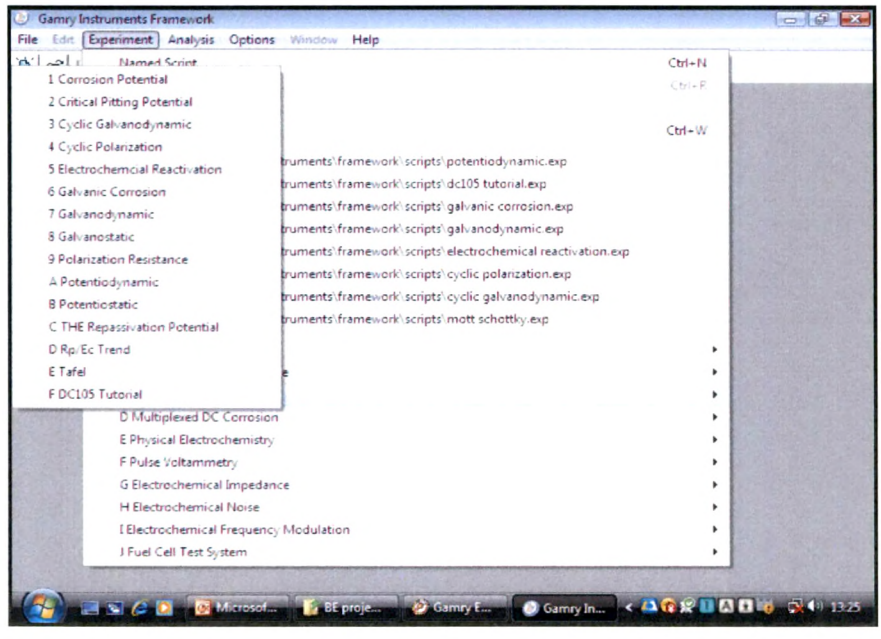
1.0 Gamry Instruments Frame Work



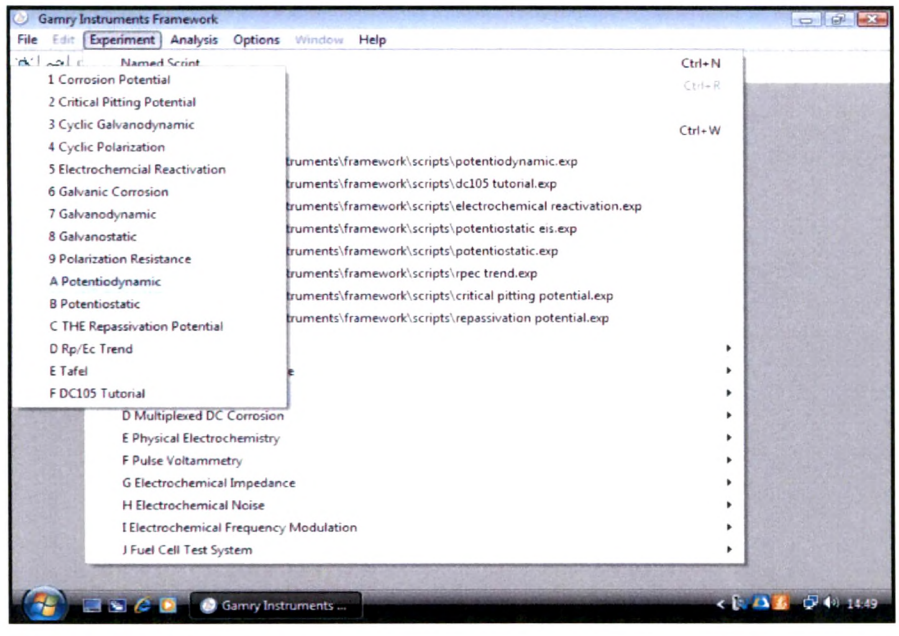
2.0 Experiment



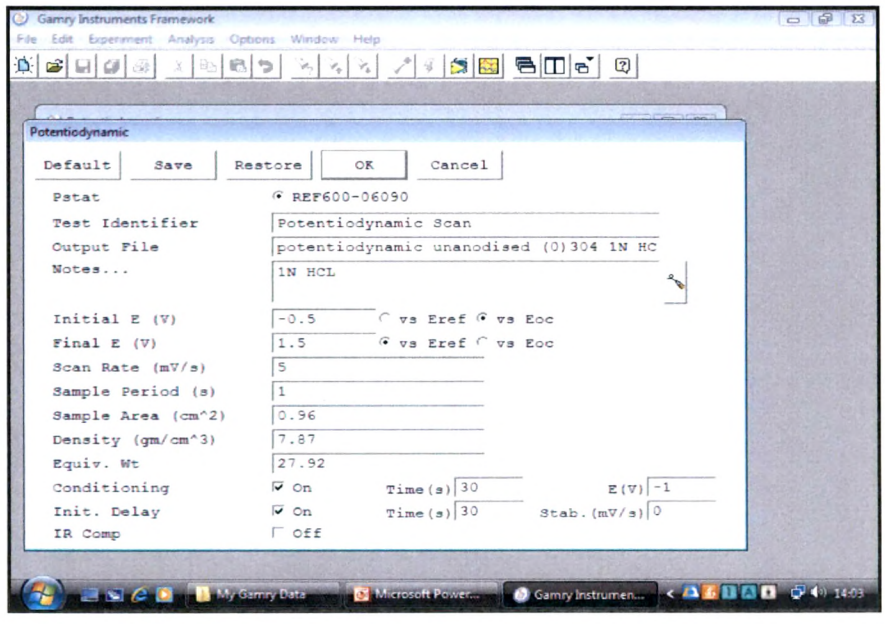
3. 0 DC Corrosion



4.0 Potentiodynamic



5.0 Get Setup & Run



3.4.4.4 Cyclic polarization Testing

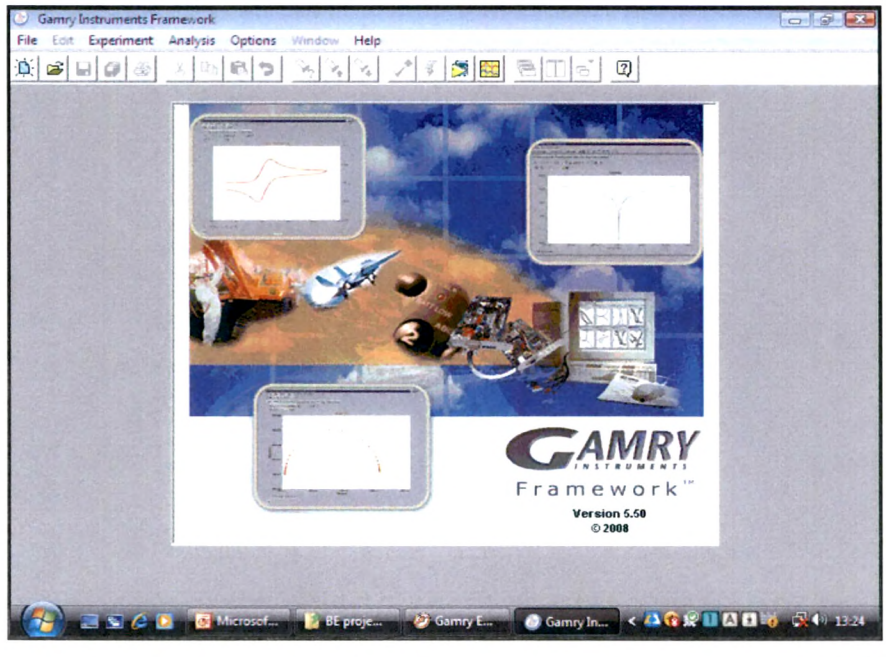
The pitting behaviour of both weld overlays were carried out by cyclic polarization study in 6 % $FeCl_3$ Solution by using Potentiostat Gammry Reference 600 as per as per ASTM Standard G-61 which is a "Standard Test Method for Conducting Cyclic Polarization Measurements for Localized Corrosion Susceptibility of Iron-, Nickel- or Cobalt Based Alloys" using potentiostat of Gamry reference 600. Samples were subjected to cyclic polarization scan using following operating parameters which are tabulated in table No -3. 5.

Table-3.5: Operating Parameters for Cyclic polarization test

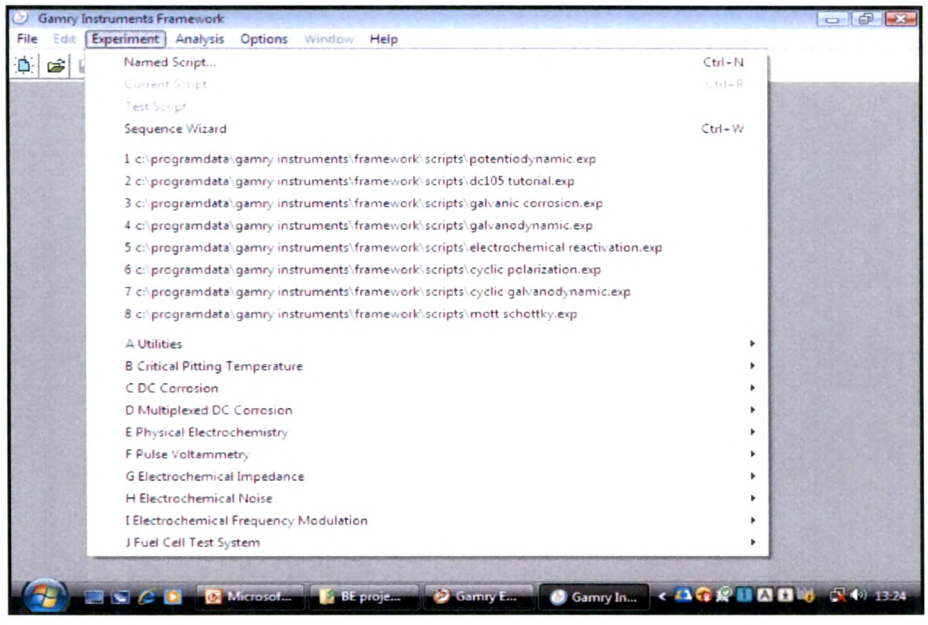
Sr.No	Cyclic polarization test
1	Initial Voltage=0.5 V
2	Forward Scan=2.5 mV
3	Reverse Scan=2.5 mV
4	Final Voltage=1.0 V
5	Apex Current= 1000 mA / cm^2
6	Sample area = 0.25 cm^2
7	Density = 7.87 gm / cm^2
8	Equivalent weight = 22.92 gm

3.4.4.4 .1 Sequence of Cyclic polarization Testing

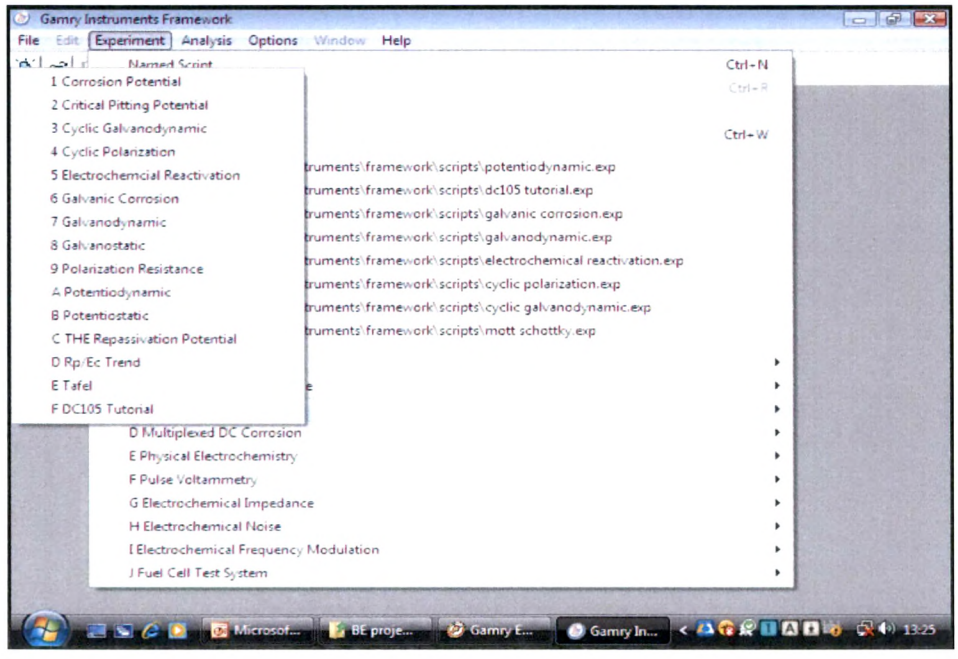
1. Gamry Instruments Frame Work



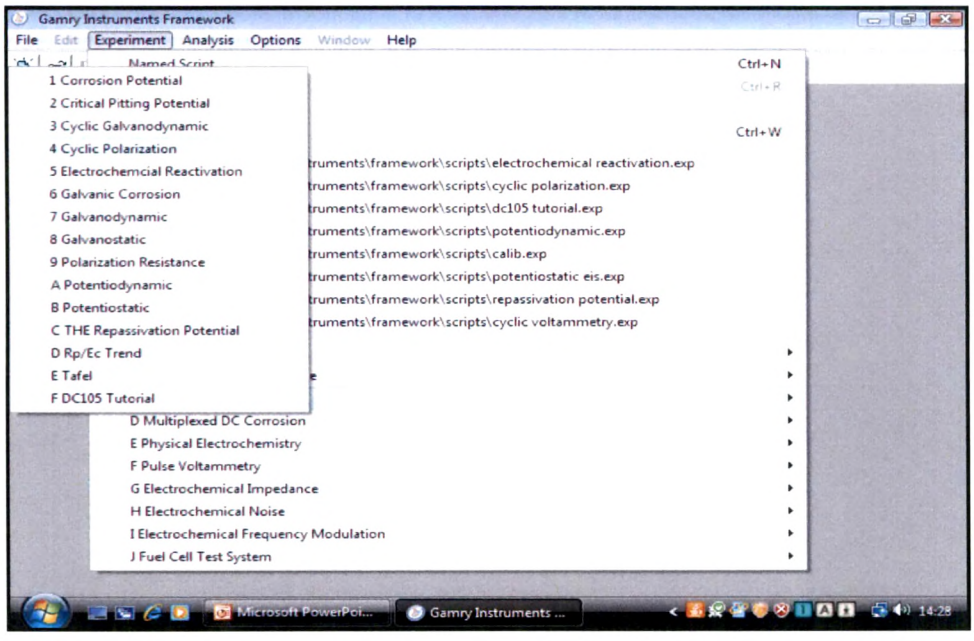
2.0 Experiment



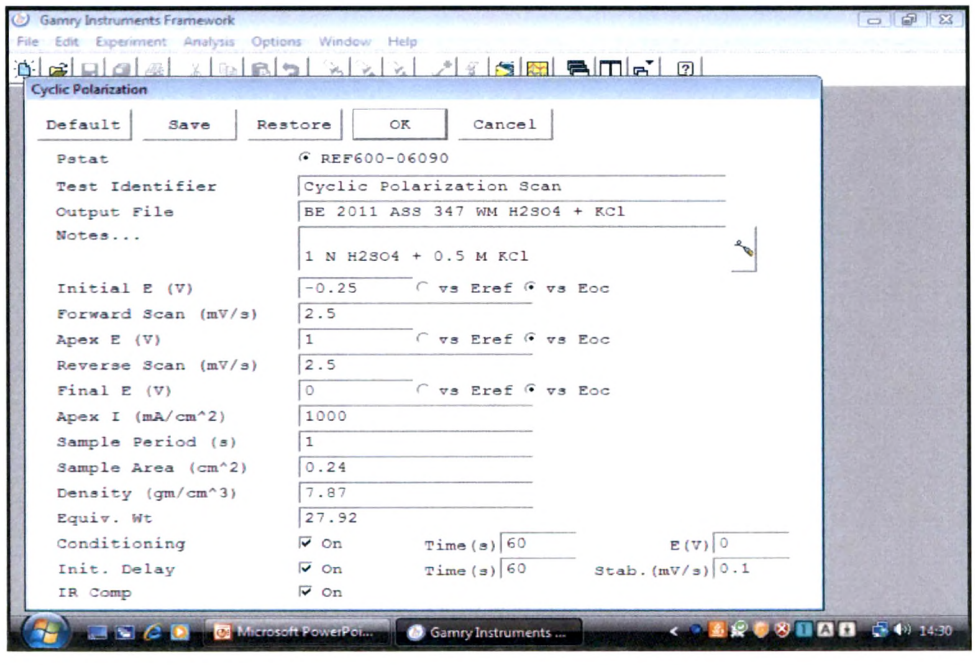
3. 0 DC Corrosion



4.0 Cyclic polarization



5.0 Get Setup & Run



3.4.4.5. Inter-granular Corrosion Testing:

3.4.4.5.1 Electrochemical Potentiokinetic Reactivation (EPR) Test

EPR test were performed on both weld overlays before and after the post weld heat treatment (PWHT) at different locations mainly cladded region, weld interface and base metal in 0.5 N H_2SO_4 & 0.01 M NaCl to quantify the degree of sensitization of in term of peak current density.

Fig 3.15 Experimental Setup for EPR test.

Fig 3.16 Argon purging

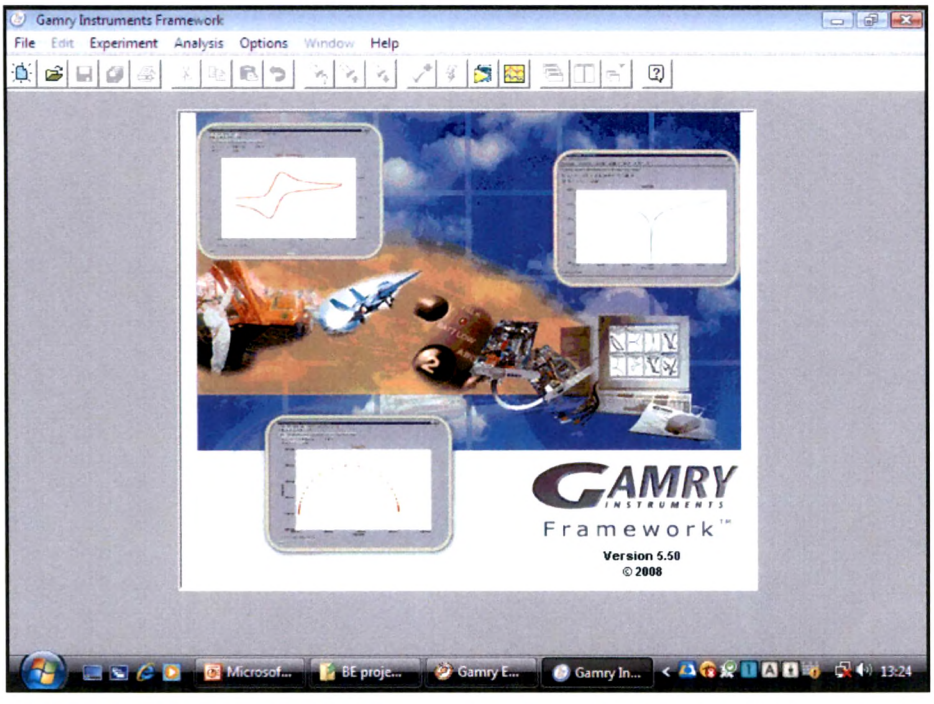
Fig . 3.15 & 3.16 shows the experimental setup which the same set up that were used for potentiodynamic testing and cyclic polarization testing, only additional argon purging is required for de-aeration of the test solution to remove dissolved oxygen.

3.4.4.5.1 .1 Experimental Procedure of EPR test:

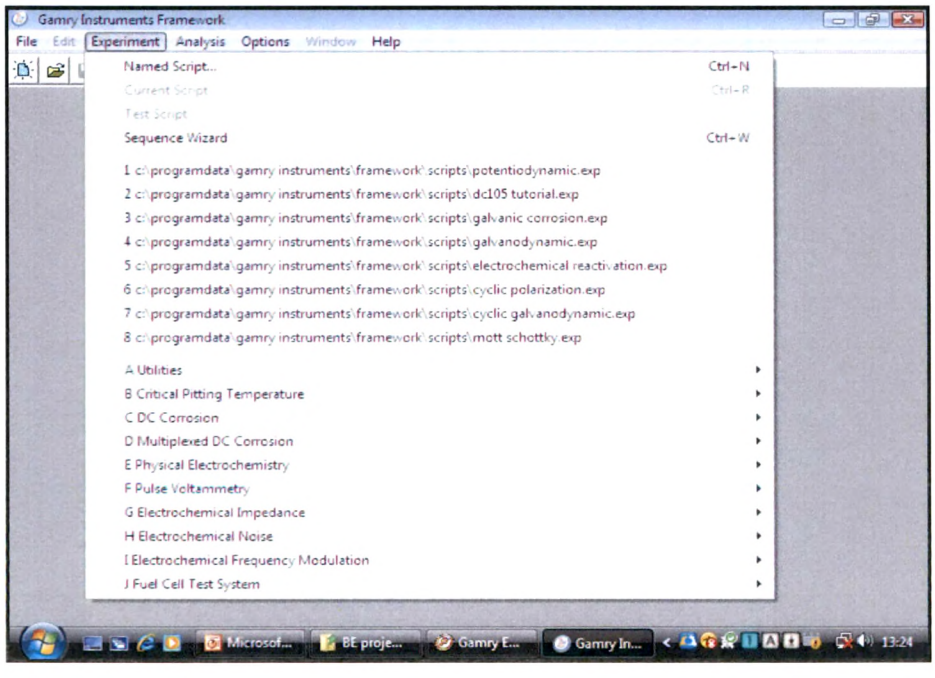
- Prepared sample with exposure area of 5 mm X 5 mm.
- The experiment is carried out in mixture of 0.5 N H_2SO_4 & 0.01 M NaCl.
- Solution is de-aerated with argon gas before and during the test.
- Temperature of the solution must be maintain constant.
- Anodically polarized the test specimen from OCP to + 300 mV(SCE) with scan rate of 5 mv / sec and immediate after reaching + 300 mV(SCE) catholically polarization was done up to OCP.
- The Maximum current of loop is measured

3.4.4.5.1. 2 Sequence of Electrochemical Potentiokinetic Reactivation (EPR)

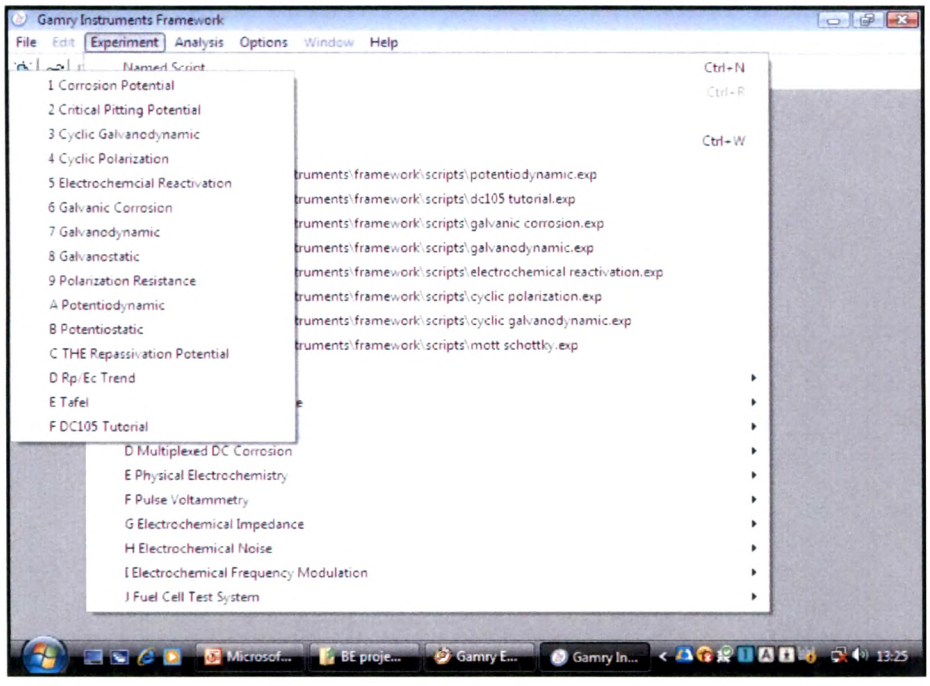
1. Gamry Instruments Frame Work



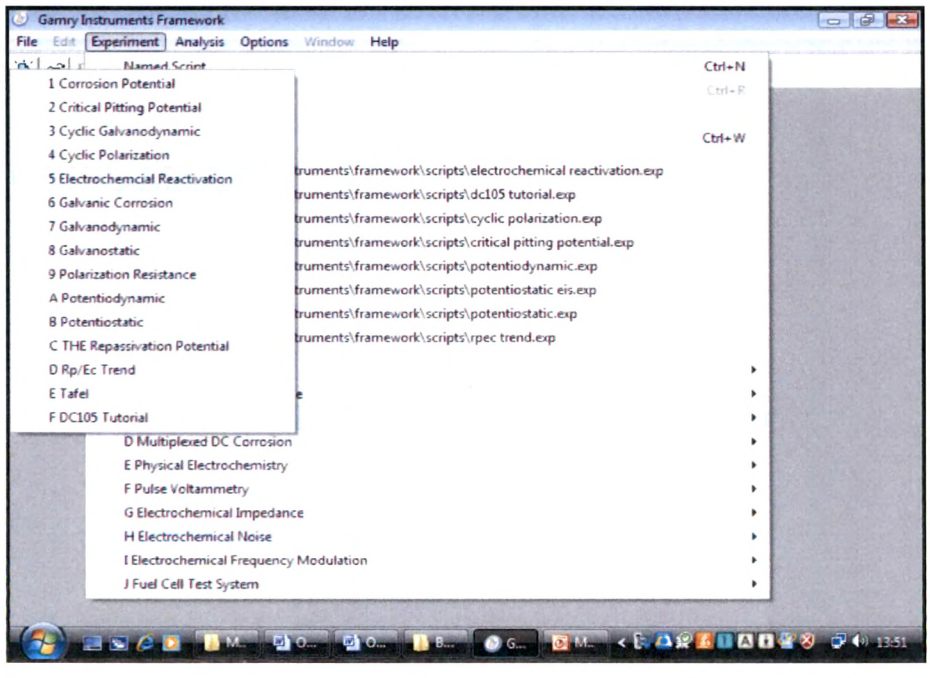
2.0 Experiment



3. 0 DC Corrosion



4.0 Electrochemical Potentiokinetic Reactivation



5.0 Get Setup & Run