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High Temperature Adhesive: Eccobond-104

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Various process shops have dealt with the properties, preparation, application, curing and inspection of Eccobond 104A/104B. The present paper takes in to count various requirements of preparation,application, curing and testing of the adhesive. In the present study, which is based upon Various practices followed by different organizations in the world and author's own experience of working with this high temperature adhesive. After curing the adhesive retains good strength up to 220deg C. The shear strength is found to be :

- 17.4 MP $_a$ at 24 deg C.
- 12.5 MP $_a$ at 150 deg C.
- 7 MP $_a$ at 230 deg C.
- (1 MP $_a$ =145psi).

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I. Introduction

Eccobond 104 is supplied in two parts, part A is liquid and part B is fine powder . This is supplied in sealed container having not more than 1 litre and 1 kg of part 'A' and part 'B' respectively. Part "A" is liquid and is available in three viscosities:

Standard Grade : 30,000 to 40,000 cps.

Filled Grade : 50,000 to 60,000 cps.

Putty Grade : 750,000 to 85,000 cps.

Unless otherwise stated, standard grade part 'A' is to be used for all applications. In sealed containers, under dry conditions, the shelf life is 2 years. However the shelf life for the materials unopened, is indefinite provided the materials pass the laid down tests. This is a high temperature adhesive that retains good strength up to temperature of 220 deg C. The paper deals with application of this adhesive with particular reference to wide range of material that are frequently required to be bonded and thereafter are required to work under high temperature going up to 220 deg C. The paper explains in detail the methodology to be used for Preparation, Application, Curing, Handling and Inspection of test piece. This is lucid and handy document useful for all practicing Engineers technologists and industries where bonding of high temperature adhesive is a frequent requirement.

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II. Preparation

a) Batching

The following procedure and precautions are required to be taken while doing the batching.

i. The liquid must be taken from a container which has been stirred to counteract "layering".
ii. The powder must be taken from its container under dry conditions because it is highly hygroscopic and will deteriorate.
iii. The mix ratio by weight is to be:
   - 64 parts of part "B"
   - 100 parts of liquid "A"
iv. The quantities sufficient for normal work load batches, should be repacked in new containers, preferably not less than 39 gms of part "A" and 25 gms of part "B"in order to ensure proper proportioning.
v. The screw top glass bottle for the powder should be of size that will hold 25 to 40 gms when completely filled. The container for the liquid must be of such a size that will be 1/3rd full when it contains the exact amount of part "A", necessary for correct mixing ratio.
vi. Each item is to be sealed, dated and identified including co-relation of paired container.
vii. From each new incoming batch of materials, a sample is to be mixed and used for testing as per the prescribed procedure.

b) Mixing

Mix thoroughly to a smooth flowing consistency. Care is to be taken to ensure that whole bottle of powder is added to the associated liquid and none is lost. It may be noted that finely divided powder is easily blown about. Warm the mixture to 60 deg C (approx) to improve the flow characteristics.

Stages	Key Points
Thoroughly part "A" resin before using.	It is important to invert the tins Containing part "A" occasionally to ensure that resin does not settle too much.
Weight out 100±1/2 parts "A", 64 ±1/2 parts of part "B"	
Warm the mixture to 60deg C to help mixing.	
Mix thoroughly the two parts Ensuring that all particles of Powder are mixed mixed with Resin. (Eccobond 104 "B" should be sieved to separate the lumps of powder)	It is preferable to use disposable un waxed cups and wooden Spoon. Mix together to smooth consistency.
When mixed and while still warm, De-aerate by placing in a vacuum oven of 2 mm Hg or less.	This is best carried out in vacuum oven held at 60 deg C.
The mix will initially froth, and space must be allowed in the mixing cups, but after a few minutes the froth will collapse.	The mix should be held in vacuum for 30 min after collapse to ensure complete de-aerate. This mixture should be de-aerated after every 6 hrs.

c) Pot Life

24 hours minimum at 25 deg C. This time will be reduced at higher temperature.

d) Surface Preparation

This is carried out to produce a satisfactory adhesive bond.

i. The bonding should be carried out within 24 hours of surface preparation. Where this is impracticable, an additional degreasing operation immediately prior to bonding shall be carried out. The degreasing is to be done as per the process detailed for the appropriate material. If nothing is quoted, degrease process shall be ;

Degrease surfaces to be reactivated as per Para e) Method-1 clause a)

e) Operating Procedure- Metals and Alloys

i. *Aluminium and Al. Alloys*

- *Method - 1*

a) *Degrease with trichloroethylene vapor as per the following sequence*

i) Immerse components slowly in vapour compartment(not in boiling Liquor)

ii) Leave for 1-5 minutes according to weight of the components. A heavy component will require a longer time to attain the same temperature as the vapor, this is essential for complete degreasing.

iii) Withdraw slowly.

b) *Alternatively*

Where vapor degreasing is impracticable, degreasing using clean liquid trichloroethylene is permissible.

1. *Abrade the surface with wet stone.*
2. Degrease with trichloroethylene vapor as per para a) above.

- *Method - 2*

a) Dry Hone.

- *Method - 3*

a) Chromate film to be given as per the approved method.

- *Method - 4*

This method is used for all aircraft structures and components where corrosion protection is required. Anodise to appropriate chromic acid method of anodizing viz DEF- 151 Type-2.

Note : The bonding should be carried out within 16 hours of anodizing if practicable. If for any reason it is practically not feasible to complete the bonding process within 16 hours, then the surfaces should be reactivated as per the following scheme.

a) Scheme for Reactivation of Sealed/ Contaminated Anodic Surfaces

This scheme covers the application of acid based solution for the reactivation of anodized surfaces which have become sealed contaminated.

i. *Approved Solutions*

- Ardrox 1074 supplied by M/s Ardrox Ltd., Brentford Middlesex, U.K.
- Metal 'Prep' which shall have the following formulations and shall be prepared by the material lab.

Phosphoric Acid (85%)	35 ml
Teepol	05 ml
Butul Cellosolve	62.5 ml.
Water	147.5 ml

For bonding purposes, the preferred solution for reactivation is Metal 'Prep' as above.

- *Process*

- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)
- Scrub the surface to be reactivated, with a clean cheese cloth charged with the reactvation solution. The surface must remain wet for 1 to 2 minutes before wiping dry with a clean cheese cloth. If the solution has dried, make a fresh application and wipe off immediately.
- If the cheese cloth shows signs of contamination, re- prepare the surface as above using reactivation solution diluted with the equal volume of water. Repeat until a clean wipe is obtained.
- Wash the surface using a clean cheese cloth and warm distilled de-ionized water until testing with blue litmus paper indicates no acid is present. If the litmus paper turns red, the acid is present, and further swilling is required.

- At this stage, the surface should be covered with a uniform film of water, i the water breaks occur, this indicates that the reactivation is not complete and the process must be repeated as above.
- Before bonding,, the component must be either
  1. Over dried 10 to 20 minutes at 55 deg C to 65 deg C and cooled to below 30 deg C.
  2. Wipe with a mixture of equal volume of Industrial Methylated Spirit (IMS) or deionised water and allowed to dry at room temperature for 30 minutes.
  3. Bonding must commence within 8 hours.
- Where the anodic surface to be reactivated has been locally repaired with alochrom solution, application of reactivation solution must be continued until the alochrom treatment has been removed i.e. Il traces of alochrom treatment has been removed. Such areas may then be retreated with alochrom but without rescuffing the surface. In such case, the bonding and painting shall commenc within i hour.
- Cheese cloth must not be redipped in reactivation solution of swill water, a new cloth must be used every time.
- Care must be taken to avoid flooding the surface with reactivation solution or swill water to ensure that none is trapped in crevices or other parts of the component.
- Operator shall wear rubber gloves to prevent reactivation solution contact with skin.
  a. *If the assembly contains components which are to be machined after bonding, the same may be allowed, Masking of the bonded areas, before chromic acid anodizing is not required.*

f) Copper and Copper Alloys

Prepare the surface with following method

- *Method*
- *Degrease with trichloroethylene vapor as per the following sequence*
  (a) Immerse components slowly in vapour compartment(not in boiling Liquor)
  (b) Leave for 1-5 minutes according to weight of the components. A heavy component will require a longer time to attain the same temperature as the vapor, this is essential for complete degreasing.
  (c) Withdraw slowly.
- *Alternatively*

(i) Where vapor degreasing is impracticable, degreasing using clean liquid trichloroethylene is permissible.

(ii) Abrade the surface with wet stone.

(iii) degrease with trichloroethylene vapor as per clause (i) above.

- Etch at room temperature for 5 minutes with neutraclean 68 diluted 2:1 by volume with distilled water. The solution must be stirred throughout the etching process.
- Rinse thoroughly with cold tap water.
- Dry using clean kim-wipe hot air circulating oven or hand air drier.
- Apply a thin film of DZ-80 to the surfaces, which are to be bonded and dry for 30 minutes at 80 deg C+5 deg C.
- Allow to cool and abrade with 320 grade silicon carbide paper wet or dry Tri-Mite untill the surface is matt.
- Wipe with kimwipe soaked in IMS to remove traces of abrasive.
- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)

g) Iron and Ferrous Alloys (Except Corrosion resis Steels) and laminations

- *Method - 1*
- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)
- Etch at room temperature with ferric chloride solution for 10 seconds.
- Rinse in cold tap water.
- Wipe, while wet, with damp cotton wool to remove any residue.
- Dry using clean kimwipe, hot air circulating oven or hand hair drier.
- Caution : Where rusting may occur e.g. mild steel parts, hot air drying should not be used.

- *Method - 2*

a) Flash nickel plate as per the appropriate standard and treat as follows

- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)
- Surfaces may be bonded up to 2 weeks after plating.

h) Steel, Corrosion Resistant

- *Method*
- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)
- Etch at room temperature with ferric chloride solution for 2 minutes.
- Rinse in cold water.
- Wipe, while wet , with damp cotton wool to remove any residue.
- Dry using clean kim wipe, hot air circulating oven or hand air drier.

i) Cadmium, Passivated and Unpassivated

- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)

Surfaces may be bonded within 2 months after plating.

j) Nickel

- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)

- Surfaces may be bonded within 2 weeks after plating.

i. *Silver*

Method - 1

- Apply Argentoplatine with distilled or demineralized water.
- Rinse in cold distilled or demineralised water. And stove dry at 100 deg C.

Method - 2

Where ultimate strength is required:

- Passivate to appropriate specification.
- This method has minimum effect on contact resistance.
- Surfaces may be bonded within 24hours of surface preparation.

k) Surfaces which are Tin plated

- Metal (Tin) not suitable for adhesion.
- Locally remove the plating if plated.
- After removal Tin plating from the base metal, the surfaces may be prepared as per the method laid down for the base metal.

l) Magnetic Materials

- Dry hone or vapor blast.
- Vapor blast to appropriate spec.
- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)

m) Glass/ Plastics

a. *Glass*

- *Grind or short blast surface.*
- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)

b. *Delran (Polyacetal)*

1. Degrease surfaces to be reactivated as per Para e) Method-1 clause a)
2. Etch at 100 deg C for i minute in the following solution. Ensure that the components are at 100 deg C before immersion.
3. 0.3 gm Toluene P-Sulphuric acid.
4. 150 cm^3 Per chloroethlene.
5. 3 cm^3 1.4 Dixon.
6. Remove the components from hot solution and immediately heat the wet components for 3 minutes at 100 deg C.
7. Rinse thoroughly in warm water.
8. Dry using kimwipe, hot air circulating oven or hand hair drier.

Notes:

- This process should be carried out in a well ventilated area, where means of extraction are available for any health hazard.
- During operations (2), the apparatus should be designed with vapor cooling ring., as due to evaporation a concentrated solution will result. Stainless steel solution is suitable material for the cooling ring.

c. *Etched Maline*

Method

Degrease surfaces to be reactivated as per Para e) Method-1 clause a)

d. *Nylon*

Method

- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)
- Abrade the surface with 240-320 grade silicon carbide paper wet or dryTri-M-ite or vapour blast as per the appropriate spec.
- Wipe off the residue from abrasion with damp cotton wool.
- Dry, using clean kimwipe, air circulated oven set at 70 deg C or hand hair drier.
- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)

e. *Fluoro Plastics (P.T.F.E., F.E.P. etc), if not pre-etched*

Method

General method where ultimate adhesion is not required, i.e. where mechanical fasteings provide the main strength.

- Allow the tetra etch solution to reach room temp.
- Apply tetra-etch solution by dipping or liberally brushing the surfaces to be bonded.
- When the surfaces become milk chocolate brown in colour (approx 5 seconds), remove the tetra etch by wiping with clean cloth soaked in I.M.S.
- Ensure that the bottle of tetra-etch solution has been kept tightly closed and stored at -10deC to 0 deg C. It must not be left at room temp aftwer use.
- Ensure that the bottle has been in use for less than six months. Record the date on the bottle when it is initially opened.
- The bottle must be atleast one third full. Bottles which are partially used can be combined provided neither has been used for more than 4 months. Pour the newer solution in the older dated bottle.

Caution

The above precautions to remove moist air are necessary because this solution reacts with water very readily ceases to be capable of etching.

f. Resin Bonded Board

Method

- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)
- Abrade the surface with one of the following methods
- 240-320 grade silicone carbide paper (Wet or dry Tri-M-Ite).
- Vapour blast to appropriate Spec.
- Wash off the residue from abrasion in cold water.
- Dry, using kimwipe, air circulating oven at 70 deg C or hand hair drier.

- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)

g. *Silicone Resin Bonded Glass Board*

This surface is unsuitable for adhesion.

h. *Thermoset Mouldings*

- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)
- Abrade the surface with one of the following methods:
- 240-320 grade silicone carbide paper (Wet or dry Tri-M-Ite).
- Vapour blast to appropriate Spec.
- Wash off the residue from abrasion in cold water.
- Dry, using kimwipe, air circulating oven at 70 deg C or hand hair drier.
- Degrease surfaces to be reactivated as per Para e) Method-1 clause a)

i. *Rubbers Natural, Nitrile, Neoprene (Excluding Silicone Rubber)*

Method

- Carefully place in concentrated sulphuric acid at 20 deg C to 25 deg C for the following period of time.

Natural rubber	: 1 minute
Nit rile Rubber	: 2 minutes
Neoprene Rubber	: 5 minutes

Ensure that the surface to be treated is in contact with acid.

Caution

Silicone rubber is not suitable for bonding with epoxy adhesive.

n) Painted Surfaces

Wipe the surfaces which are to be bonded with clean cloth soaked in SBP. Care must be taken to ensure that the surfaces which are not to be bonded should not be wiped.

i. *Carbon*

Abrade with 120 grit emery paper and ultrasonically clean to appropriate spec.

a. Ultrasonic Cleaning

This method specifies requirements for cleaning components with ultrasonic equipment which is designed for process where;

- The components are immersed in a tank containing a transmission fluid of either water or kerosene.
- The container is partly immersed in tank containing a transmission fluid of either water or kerosene.
- This method is not suitable for Rubber/-Seals/Bellows/Bellow Assys/Impregnated electrical coil.

b. Abrading of bonded threads is not required. Surfaces 32 micro inches or inner surfaces,sharo edges must be protected against abrasion. Ensure complete freedom from abrasive particles before bonding.

c. Absolute freedom from grease and finger marks is essential. Check wetability with distilled water, dry off and clean with solvent. Do not touch. Clean with cold solvents as per appropriate spec.

d. For adhesive mixture used in every batch of parts being bonded, test piece is to be prepared, processed and tested as follows:

- The test piece to consist of two strips of steel each 11/12 cm long,24.6/26.2 cm wide X 0.85/1.3 cm thick. The edges must be free from burrs.
- The surfaces to be bonded are to be prepared by abrasive blasting, using aluminum oxide grit, 120/220 mesh to produce a finish not finer than 120 micri inch.
- Clean to ensure that the surfaces are free from grease and finger mark. This may be carried out by cleaning with cold solvents as per the appropriate standard.

1. Rubber seals should be cleaned with Kerosene and not subjected to any other solvent.
2. Bellows and bellow assy's be cleaned with Kerosene nd not subjected to any other solvent.
3. Cleaning must be carried out immediately prior to bonding.

- With the aid of suitable fixture, bond with an overlap of 12.7/14.3 cms and the long edges parallel following the procedure as laid down in para 4 of this paper.
- When pulled on testing machine at a rate of loadin of 1.3/2.7 KN per minut, the joint is to withstand a load of 4.5 KN (1000 lbf.) without failure.
- Pulling of test piece shall be carried out by Physical testing lab under the supervision of quality control deptt.

III. Application

- The adhesive will normally be applied to both contacting surfaces. However there will be cases such as plugging of blind holes where to avoid entrapment of air, an adequate pool of adhesive is to be applied to the hole only and excess is exuded by carefully pressing the plug to bottom. Preferred thickness of the layer of the adhesive is between 0.5 to 1.5 mm depending upon the requirement.
- Assemble cylindrical parts with a twisting motion, if possible, to ensure joint coverage. Suitable fixtures must be used to ensure that the position of the parts is not disturbed. Viz mandrels, rods with spring loaded washers.
- With flat surfaces, bring the surfaces together and loghtly squeeze out excess material.
- After mating the parts, carefully wipe away excess adhesive preferably with cotton buds.
- Place in cool oven (50 deg C max.). Cylindrical joints should be arranged to be vertical so that

surface tension will help the assembly to maintain concentricity and equalise the joint thickness. Flat joints to be arranged such as to maintain the parts in their relative positions but no pressure is to be applied except only light clamping.

IV. Curing

- Curing may be accomplished in accordance we on of the following schedules.

Time (Minimum)	Temperature
6 hours	120 ± 5 deg C
3 hours	150 ± 5 deg C
2 hours	180 ± 5 deg C
1 hours	200 ± 5 deg C

- General guide lines for curing different materials are as follows:

1. Steel, Nickel, Copper :1 hour 190/200 deg C.
2. Alloys or carbon. :2 hours 175/185 deg C.
3. Al Alloys :4 hours 150/160 deg C.
4. Assy's Containing Rubber and plastics :Refer Drawing of the component/ Assy.

- If the assembly is to be used at temp. Above 250 deg C, it shall be given a further cure of 12 hrs. (min) at 260 deg C.
- *Eccobond may break at 400 deg C. At this temp. It can cause distortion and change of properties. So, care must be taken for all these factors.*

V. Handling Precautions

- Avoid skin contact and inhalation of vapours. The use of barrier cream is recommended.
- Working area must be well ventilated and ovens extracted to outside atmosphere.

VI. Inspection

a) Material Control

i. When received, the parts 'A' and 'B' shall be passed through "Quarantine Inspection Deptt" to the laboratory. The laboratory shall repack the material as per para II. Preparatrion a) Batching

ii. Identification of contents and correlation of paired containers to para II. Preparation a) Batching Para iv, v and vi must be clearly visible on each jar. Where the identification label is missing from a jar or not legible or the seal is broken, the contents must not be used.