

Bharat Heavy Electricals Ltd. Tiruchirappalli 620014

Advanced Technology Products Operation Planning & Control OP&C: PROC: RPVB3: 002

PROCEDURE FOR ZINC CHROMATE COATING

Revision no and date	No of	Description	Prepared by (BHEL)	Reviewed by (BHEL)	Approved by (BARC)
	pages			San Contract Contract	
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16-12-2016			(OP&C)	Rajasekaran (Plant Lab)	
01	12	Re-submitted for approval	Ajass	the hint	
07-02-2020		based on RPVB2 procedure as commented by BARC. Procedure no. given	Apage 07.02.20 20 (ATP/OPRe)	9/01/11/11	
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1. Scope

This procedure details the process of Zinc Chromate coating for components made of Carbon steel & Low Alloy steel.

Applicable specifications – IS 1573 and IS 9839.

Refer Annexures-1 for list of components to be Zinc chromate coated.

2. Quality Control

- 2.1. Details of the chemicals, test methods and the equipment proposed to be used shall be submitted by the firm while giving the offer. The exact designation of all proprietary materials proposed for use shall be stated in offer.
- 2.2. All machined components shall have been subjected to LPT prior to coating.
- 2.3. The offer shall include a detailed method of control, with limits for time, temperature and pH values, and all other pertinent details that will ensure compliance with the requirements of this Procedure.
- 2.4. No deviation from the approved Procedure shall be permitted without prior written approval of BHEL/QC and Customer/QS.
- 2.5. Process parameters shall be established by the S/C firm initially on mock-up coupons provided by BHEL/QC. Mock-up coupons are representative of actual job materials.
- 2.6. All the coating stages, Testing and Inspection shall be carried out the presence of BHEL/QC and Customer/QS.
- 2.7. Only after process parameters have been established on mock-up coupons and accepted by BHEL/QC and Customer/QS, job production can be taken up.
- 2.8. Production Test coupons supplied by the manufacturer shall be processed concurrently with each batch of job and shall be sufficient to conduct inspection/testing as per paragraph no. 4.0 of this procedure. Only when results of testing conducted on Production Test Coupons in the presence of BHEL/QC and Customer/QS are satisfactory, it can be concluded that phosphate coating process is satisfactory. Actual job shall also be subsequently inspected as per

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- paragraph no. 4.0 of this procedure. Spare quantity of job identified by Purchaser can be taken as Production Test Coupons.
- 2.9. Approval of process, materials and equipment implies no guarantee of acceptance of the results obtained after testing. Acceptance of the test results for Production Test coupons does not guarantee acceptance of the phosphate coating on components and component will be accepted only after complete inspection of the same. Test specimens shall be used only once and then preserved only for records.
- 2.10. Care shall be taken at all stages to prevent contamination of the surfaces being treated by touching with bare hands, splashing with undesirable liquids or the condensation of moisture on parts after drying.
- 2.11. Test specimens shall be used only once and then preserved only for records.
- 2.12. Care must be taken to ensure that parts are kept in process baskets/pallets such that air entrapment in part internals is minimized during bath immersion.
- 2.13. Carryover of solution from one chemical process tank to next shall be totally avoided.
- 2.14. Chromate conversion (passivated) Zinc coatings contain hexavalent Chromium which may irritate the skin & cause ulcers on skin. Cotton, nylon or rubber hand gloves may be used to prevent skin ulceration and/or finger print corrosion of coatings while handling coated parts.

3. <u>Coating stages</u>

- 3.1. Consecutive stages shall follow one another without delay.
- 3.2. Major stages along with the associated equipment are listed below: -

Stage	Equipment required						
I – Pre-treatment	Electric Furnace (applicable for material grade						
	38XH3MFA only)						
II – Surface preparation	Degreasing plant						
	Rust or scale removing solution tank						
III – Rinsing after cleaning	Cold/Hot water tank (with constant water overflow)						
IV – Electroplating	Electroplating column/tank (SS), heated by gas, steam or electricity						

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V – Rinsing	Cold water rinse tank (with constant water overflow)					
VI - Drying	Force drying / Drying oven (applicable for material grade 38XH3MFA only)					
VII – Hydrogen	Electric Furnace (applicable for material grade					
Embrittlement Relief	38XH3MFA only)					
VIII – Chromate	Hot-chromate dip tank					
conversion						
IX – Rinsing	Cold/Hot water rinse tank (with constant water					
	overflow)					
X - Drying	Force drying / Drying oven					

- 3.3. Stage I Stress relief HT (Pre-treatment for high strength steels)
- 3.3.1. Steels of specification $38XH3M\Phi A$ shall be given stress-relieving treatment before phosphating.
- 3.3.2. The parts should be heated for at least 30 ± 10 minutes at a temperature of $190\pm10^{\circ}\text{C}$.
- 3.3.3. Minimum duration of heating -1 hour for every 25.4 mm (1 inch) of thickness but not less than 0.5 hour for thicknesses less than 12.7 mm (0.5 inch).

3.4. <u>Stage II – Surface preparation</u>

- 3.4.1. Before processing, all scale, rust, grease oil & foreign matter shall be removed from surface to be treated by a method, or combination of methods, suitable for meeting the requirement.
- 3.4.2. Choice of the appropriate process depends upon the type and degree of contamination and the size and shape of the parts. Method of cleaning or a combination thereof shall be finalized after discussions with Purchaser.
- 3.4.3. For material grade 38XH3MΦA, non-electrolytic alkaline or anodic alkaline cleaners or mechanical cleaning procedures are preferred to avoid the risk of producing hydrogen embrittlement during cleaning
- 3.4.4. At the end of immersion, the items shall be lifted above the solution level to allow draining of excess chemicals.

3.5. <u>Stage II – Surface preparation</u>

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- 3.5.4. At the end of immersion, the items shall be lifted above the solution level to allow draining of excess chemicals.

3.6. Stage III - RINSING AFTER DEGREASING AND RERUSTING

- 3.6.1. Items shall be rinsed thoroughly in cold water rinsing tank both after de-scaling and after de-rusting. Items shall be drained of excess water.
- 3.6.2. Care should be taken to ensure that the water supply itself is sufficiently free from harmful salts as given below:
 100 ppm total chlorides (calculated as Cl⁻)
 200 ppm total alkalinity (calculated as CaCO₂)
- 3.6.3. Adequacy and effectiveness of cleaning shall be demonstrated on the mock-up coupons. Cleaned components are to be offered to BHEL and Customer/QS before any further processing.
- 3.7. <u>Stage IV Zinc Electroplating</u>
- 3.7.1. Application method Acid Zinc plating.
- 3.7.2. Material for tank SS (preferred) or low-carbon steel.
- 3.7.3. The material used in the construction of the electroplating plant shall be such that they have no adverse effect on the quality of the coating produced. Care should be taken to avoid use of unsuitable material in plant construction, for

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- example, copper or brass heating coils, which adversely affect the quality of coating formed.
- 3.7.4. In addition, parameters viz. Acidity of the Electroplating bath, Concentration of inhibitors, depolarizers and oxidizers, if any; free acid and total acid points shall be controlled.
- 3.7.5. Electroplated parts shall not be allowed to dry between the electroplating solution treatment and the subsequent cold running water rinse. Fog sprayers or other means for prevention of drying shall be provided.

3.8. Stage V – Water rinsing

- 3.8.1. After electroplating, thorough dip rinsing with water with continuous overflow maintained by the addition of fresh water shall be done in order to remove any residues of the solution. Items shall be immersed only as long as necessary to meet the requirements of this specification.
- 3.8.2. The flow of rinse water shall be adjusted, regulated or controlled in order to prevent contamination at any time beyond a time limit that is such as to assure no detrimental effect on the coatings produced.
- 3.8.3. Care should be taken to ensure that the water supply itself is sufficiently free from harmful salts, limits for which are listed below: -
 - 70 ppm total chlorides (calculated as Cl⁻),
 - 200 ppm total alkalinity (calculated as CaCO₃)

3.9. <u>Stage VI – Drying</u>

- 3.9.1. Parts of material grade 38XH3MFA after rinsing, shall be dried thoroughly (preferably force-dried).
- 3.9.2. Precautions shall also be taken to avoid local accumulation of solid residues, such as may be formed on the surface, when hard water is used for rinsing. Special attention being paid to the parts that contain pockets crevices.
- 3.9.3. In case Compressed air drying is used, air should be dry and free from oil and dirt. The pressure of air shall not exceed 6.3 kgf/cm².

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- 3.10. Stage VII Hydrogen Embrittlement relief
- 3.10.1. Subsequent to drying after electroplating, parts of material grade 38XH3MFA shall be given embrittlement relief treatment immediately without any of the parts developing cracks.
- 3.10.2. The heat treatment shall commence as soon as possible, preferably within 1 hour but not later than 3 hour after electroplating has been completed.
- 3.10.3. Heat treatment has to be done at 190 220 °C with 8 hours soaking time. For parts > 25 mm thick with threads, do heat treatment at 190 220 °C with 24 hours soaking time.
- 3.10.4. This heat treatment has to be done after electroplating and before application of chromate conversion coating or stressing operation.
- 3.10.5. Hardness shall be checked on mock-up and production test coupons subsequent to heat treatment to determine any unacceptable reduction in hardness. In such circumstances, treatment may be carried out at a lower temperature, but not less than 130 °C, for a minimum period of 8 hours. It may be noted that a low temperature treatment can adversely affect the fatigue strength of the article.
- 3.10.6. Electroplated parts passivated due to hydrogen embrittlement relief treatment shall be reactivated before receiving the supplementary treatment by immersing in a dilute acid solution. Parts shall be activated as soon as possible following this treatment and handled carefully to avoid contamination.
- 3.11. Stage VIII Chromate Conversion Coating (Supplementary treatment)
- 3.11.1. Immersion in acidic chromating solution containing hexavalent or trivalent chromium salts, together with other salts shall be used to achieve different appearances on articles as listed in Annexure 1.
- 3.11.2. The grades of chromic acid used in the preparation of chromate rinse as also the quality of water shall be of such purity that the provision relating to freedom from corrosive residues shall be met.
- 3.11.3. The item shall be subjected to supplementary treatment for at least 60 seconds.

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3.11.4. Chromate conversion coatings harden with age by gradual dehydration. The articles shall be handled carefully for the first 24 hours after treatment, and any tests (including corrosion tests) shall be deferred until the expiry of that period.

3.12. Stage IX - Rinsing

- 3.12.1. If hot water is used as the final rinse after the chromating process, the time of rinsing should be kept as short as possible, for hexavalent type of coating if used, in order to prevent the dissolution of the hexavalent chromium, if present.
- 3.12.2. Precautions shall also be taken to avoid local accumulation of solid residues, such as may be formed on the surface, when hard water is used for rinsing.

3.13. Stage X - Drying

- 3.13.1. After the final rinsing, the components shall be dried thoroughly at a temperature compatible with the type of chromating used in order to prevent cracking due to dehydration of the chromate coating.
- 3.13.2. Force drying is preferred. Special attention being paid to the parts that contain pockets crevices. Compressed air should be dry and free from oil and dirt. The pressure of air should not be above 6.3 kgf/cm².
- 3.13.3. Maximum drying temperature shall be restricted to 60 °C.

4. Inspection, Testing & Acceptance

All tests, including accelerated corrosion testing, shall be carried out at least 24 h after chromate conversion treatment.

4.1. Process Examination

- 4.1.1. The entire Zn9Cr coating process along with the associated inventory shall be subject to examination at the discretion of BHEL/QC and Customer/QS to verify conformance with this specification.
- 4.1.2. Sampling for process control testing shall be subject to the approval of persons from BHEL/QC and Customer/QS. The sampling plan shall be sufficient to

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- demonstrate adequate control of the process and conformance of products processed.
- 4.1.3. Tests shall be performed at the frequency established from mock-ups and approved by persons from BHEL/QC and Customer/QS.
- 4.2. Finish & Appearance of article after zinc plating
- 4.2.1. Items shall comply with the finish or surface roughness requirements on the drawing prior to application of the phosphate coating. Acceptable finish after zinc plating is bright finish.
- 4.2.2. Over the significant surface, the plated article shall be free from clearly visible plating defects such as blisters, pits, roughness, nodules, cracks, burning or unplated areas and shall not be stained or discoloured.
- 4.2.3. On articles, usually where a contact mark is inevitable, its position shall be decided by mutual agreement and shall be excluded for inspection of appearance.
- 4.2.4. Superficial stains that result from rinsing or slight discoloration resulting from drying or heating operation to relieve hydrogen embrittlement shall not be the cause for rejection.
- 4.3. Finish & Appearance of article after chromate conversion
- 4.3.1. Appearance of the supplementary treated article shall be as per Annexure I.
- 4.3.2. In the case of iridescent yellow color, the combination of colors may vary according to the process conditions like pH, conditions of the base material and zinc deposit, temperature, time of reaction, agitation and composition of the chromate conversion bath.
- 4.3.3. The chromate film shall be free of bare patches.
- 4.4. <u>Dimensions of coated items</u>
- 4.4.1. Items shall comply with dimensional requirements of the drawings after application of coating.
- 4.4.2. Threaded items shall answer GO and NO GO gauges as per ISO 1502 after coating.
- 4.5. <u>Test for coating thickness</u>

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- 4.5.1. The minimum thickness of zinc coating shall be as per Annexure 1.
- 4.5.2. Coating thickness shall be determined by microscopic examination at 100X minimum.
- 4.5.3. Thickness measurement of zinc coatings on mock-up coupons shall be made before and after application of supplementary treatment. The chromate film may be removed by using a very mild abrasive (as paste of levigated alumina rubbed on with the finger).
- 4.5.4. The minimum local thickness of the zinc coating shall be measured for all the significant surfaces. Average thickness shall be determined for small parts and fasteners where minimum local thickness cannot be determined.
- 4.5.5. Definition of "Significant" and "Non-significant" surfaces on parts shall be as per IS specifications.
- 4.6. <u>Burnishing Test for adhesion</u>Method of testing and acceptance shall be as per Appendix F of IS 1573.
- 4.7. <u>Humidity Test for chromate coating performance</u>
 Method of testing and acceptance shall be as per Appendix C of IS 1573.
- 4.8. <u>Test for resistance to corrosion</u>
- 4.8.1. Mock-up coupons shall be subjected to neutral salt spray (fog) test for determination of accelerated corrosion resistance as per ASTM Test Standard B117.
- 4.8.2. Refer Annexure 1 for
 - Duration of neutral salt spray test before red rust appears.
 - Duration of neutral salt spray test before white rust appears.
- 4.9. <u>Chemical test for chromate film</u>Method of testing and acceptance shall be as per Appendix D of IS 1573.
- 4.10. Coating surface density for chromate conversion coating

 The approximate coating surface density and typical chromium content shall be as per Annexure 1.

5. Packing

- 5.1. Coated products shall be individually packed in polyethylene cover followed by soft packing so as to avoid any damage during handling or shipping. Parts/articles cleared for shipment but found damaged after receipt shall be accounted as "Rejected". Marking of items shall be on tag.
- 5.2. The following completed documents for mock-up, Production Test Coupons & actual job shall be supplied along with the job: -
 - OPS, Inspection and Testing records
 - Process parameters & log records
 - Stress relief and Hydrogen embitterment relief treatment records

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Duration of Neutral salt spray test (in hrs)	Before zinc coating corrosion (white rust) begins	72		72		48		27		24		
Duration of spray test (in hrs)	Before base material corrosion (red rust) begins	144		144		72		144		96		
Minimum coating thickness (microns)	Avg	9 to 12		9 to 12	9 to 12		4 to 6		11 to 15		11 to 15	
Minimum coating th (microns)	Local (max)	10		10		ν.		12 .		12		
Approx. film density (gram/m²)	Typical Chromium content (mg/m²)	0.5 to 1.5	50 to 100	0.5 to 1.5	50 to 100	0.5 to 1.5	50 to 100	0.5 to 1.5	50 to 100	Up to 0.5	5	
Арреагансе		Yellow Iridescent		Yellow Iridescent		Yellow Iridescent		Yellow Iridescent		Black		
Threads available on the component		M20 x 1.5 - 8g		M20 x 1.5 – 80	M20 x 1.5 – 8g		M8 – 7H		M105 x 3 – 7H M27 x 1.5 – 7H M16 x 2 – 7H		M27 x 1.5 – 6g	
BHEL Drawing No.			00545-R01	2-PV-629- 00545-R01		2-PV-629- 00545-R01		2-PV-629- 00545-R01		2-PV-629- 00545-R01		
(zmerg) idgisW leubivibal			1156		25		1800		8800		700	
Qty. (nos.)			28		28		35		13		55	
oberg leiroteM			SA 105		SA 105		SA 105		38ХН3МФ А		38ХНЗМФ А	
Part			Kod	Plug - 7		Bush - 6		Flange - 3		Stud M27x1.5		
Sr. No.			_	(1	,	2		4		0	