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# Qualification of a Corrosion-Protection Coating System for Transport and Storage Casks: Requirements and Results

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## INTRODUCTION

In the FRG, the storage of spent fuel in transport/storage casks is a relatively expensive solution. Cost-reducing manufacturing methods are highly desirable, in order to retain competitiveness. The spent fuel is stored in these casks in dry condition under an inert gas atmosphere for several years. For this reason, the requirements made on a corrosion-protection coating for the inner cavity must be appropriate to the average level of operating loads. Expensive overlay-welding of stainless steel lining are to be replaced by cheaper technology.

## REQUIREMENTS

The requirements made on a corrosion-protection system for the inner cavity, which must be adhered to, in order to obtain validation and approval by the competent authority, can be derived from the necessities of

- Manufacture (selection of method)
- In-service conditions and
- Approval and validation procedure.

The most important requirements, which must be fulfilled in accordance with specified criteria and tolerances, are:

- corrosion resistance
- thermal resistance
- He-tightness: Typ (B) requirements
- possibility for vacuum drying
- decontaminability
- testability of quality
- adhesion

- repairability
- machinability
- surface without defects

In particular, the requirements made on the sealing faces - HELICOFLEX metal gaskets are used - and thus, also, on the corresponding test methods, have led to the refinement of test systems such as eddy-current testing, dye penetrant and ultra-sonic tests.

#### SELECTION OF METHOD

From the multitude of possible methods and materials for manufacturing of a corrosion-protection coating, the following two methods were shortlisted after consideration of the above-mentioned requirements and performance of tests:

- chemical nickel plating
- thermal spraying.

Since not only the dominant cost criterion, but also the mobility of the method and environmental aspects had to be taken into account during the selection phase, the conclusion has been drawn that the thermal spraying system possesses the necessary potential.

#### DESCRIPTION OF THE PROCESS

A corrosion-resistant alloy is deposited on the surface of the inner cavity requiring coating by means of thermal spraying. The application of this multi-layer coating is effected by means of an automatic, programm-controlled system. The required coating thickness, including a certain oversize for machining, is adjusted to accord with the requirements for the individual cask areas (sealing faces, circumference and bottom area).

Compressed air is used as the working gas for the adhesion layer, and argon for build-up of subsequent layers.

The deposited coating is refined by means of hammering, which converts existing tensile stresses into compressive stresses; porosity is largely compacted, and the surface is smooth, and its area thus reduced, leads, in total, to a decisive improvement in corrosion resistance.

If required, the coating can also be machined by means of turning or grinding, with similar positive results as obtained under hammering.

#### TESTING OF THE PROCESS

After performance of numerous deposition tests on experimental plates and intensive examination of the products in the laboratory, the first large-scale coating tests were performed:

An NiCr coating was applied automatically by means of electric-arc deposition to the internal surface of a cast-iron cask of the following internal dimensions:

Length = 2,980 mm  
Diameter = 720/970/1,240 mm

with an initial wall thickness of approx. 360 mm.

According to the requirements of the individual cask sectors, and including an average allowance of approx. 0.2 mm, coating thickness was between 0.7 and 1.5 mm.

The allowance specified was reduced by means of subsequent hammer-compaction, i.e., the thickness of the original coating was reduced by some 20 to 30 %. In the sealing face areas, the coating was turned down to nominal thickness.

After machining of the coating, surface roughness was approx. 5  $\mu$ Ra, and the coating was homogeneous, manifesting scarcely any pores or inclusions, and with no bonding defects in the transition area between the parent material and the deposited material.

Vacuum drying and measurement of residual moisture in the interior of the cask were used to furnish the proof that the condition of the surface fulfills the following requirement:

Perfect drying within a reasonable period of time must be possible after filling of the cask with water and drainage of the water.

#### QUALIFICATION PROCEDURE

The qualification procedure has been agreed with the Bundesanstalt für Materialforschung und -prüfung (BAM), the independent expert of the competent authority. The procedure comprises development and qualification of control procedure, demonstration of fulfilment of Type B(U) requirements (leak tightness, resistance to - 40 °C drop test, and accidental thermal loads), and verification of a correlation between the container-coating and simultaneously processed samples.

A test program is to be performed for validation of the procedure. The test bodies needed for this purpose are to be produced both from a test cask to be coated, and from parallel-coated specimens.

The program consists in detail of the following tests and experiments:

#### Suitability of the Test Procedures Selected

- Coated Test Body with Artificially Induced Defects.

Test: Dye penetration test, eddy-current test, ferroxyl test.

- Heating-up Test

Repeated heating from room temperature to 160 °C

Test: Visual assessment, dye penetration test, eddy-current test, metallography.

- Drop Test to Verify Tightness as per Type B(U) Requirements

9 m drop (test body: - 40 °C)

Test: System tightness, dye penetration, eddy-current test, metallography.

- Compression of a Metal Gasket

Test body from drop test

Test: Measurement of surface roughness, with rubbing, metallography.

- Repair of Defects

Coated test body with artificially induced defects, repair of defects using various methods.

Test: Dye penetration test, eddy-current test, ferroxyl test, metallography.

- Surface Quality of Machined Layer

Test body from tests mentioned above

Test: Surface roughness measurement.

coating in the cask and that on the parallel-coated specimens. This is the precondition for restriction of quality-assurance testing mainly to simultaneously coated specimens in coating of series-production casks.

The qualification procedure has at present reached the stage of preliminary tests for clarification of detailed questions with regard to the coating of the qualification cask and the development and manufacture of the test bodies for drop tests.

Since the qualification cask itself has not yet been coated, no results can as yet be submitted on this.

The results to be anticipated can, however, be derived from the coating of an experimental cask, as described in "TESTING OF THE PROCESS".

Tests performed on co-coated specimens produced the following results:

- Metallographic Examinations

Metallographic examinations were used to investigate layer structure and for documentation of possible defects, such as pores and cracks in the spray-applied coating.

Typical coating structures can be seen in figure 1. The lower coating, applied using compressed-air as working gas manifests, as anticipated, relatively severe oxidation, while, in the upper coating zone, which was produced under an argon atmosphere, the oxidic content is substantially smaller.

In each case, four specimens were taken parallel and perpendicular to the direction of spray-application from the individual test specimen, for the purpose of microsection examination. No cracks were found on the micrograph.

On a specimen featuring a repaired layer (see fig. 2), it was impossible to microscopically distinguish the transition from the original spray-applied layer to the repaired zone. In the lateral microsection, too, only a slightly greater oxidation was detectable.

The average hardness of the layer sprayed under argon was:

- in the direction of spraying 305.6 HV<sub>0.1</sub> (standard deviation: 30.6)
- perpendicular to the direction of spraying 264.5 HV<sub>0.1</sub> (standard deviation: 25.3)

- He-tightness, Test Layer

Standard He-leak rate less than  $10^{-7}$  mbar l/s

- Corrosion Test

Leaching test in boric acid and deionate, test period: approx. three months.

- Leaching of a Porous Specimen

Long-term behaviour of a defective coating.

- Testing of Machinability

Machining of a coating (turning, grinding).

- Testing of Adhesion

Test as per DIN 50 160.

- Coating Thickness Measurement

Eddy-current testing.

Some tests were also performed on specimens with local repairs, in order to simultaneously validate suitable repair methods.

## RESULTS

The tests supplies information on:

- corrosion behaviour
- testing for internal defects
- He-tightness of the coating, including performance after exposure to accident stresses
- repair methods
- mechanical properties.

The objective of the test program is, in addition to the basic validation of the process, to show confirmation of the correlation between

- Layer Thickness Measurement

The layer thickness was measured under a photomicroscope on a metallographic microsection from the three test objects provided for metallographic and corrosion testing. Average thickness was, for test object 1: 0.993 mm, for test object 2: 0.671 mm and for test object 3: 0.450 mm. The manufacturer had claimed thickness of approx.

0.5 and 0.3 mm respectively for the NiCr 80 20 coatings on the respective test objects.

- Corrosion Testing

The corrosion behaviour of the coatings was investigated by means of exposure of specimens in dilute aqueous boric acid solution (3 grms/1  $H_3BO_3$  = 0.15 N  $H_3BO_3$ ). The test period was eleven days.

After removal from the test medium, no signs of corrosive attack could be microscopically determined on the surfaces of the specimens. Transverse microsections of the exposed specimens were then made and examined photo-microscopically.

- Adhesion

Adhesion was determined in accordance with DIN 50 160. It was an average of  $29.1 \text{ N/mm}^2$ , (standard deviation  $2.6 \text{ N/mm}^2$ ). Adhesion of the repaired layer was  $27.2 \text{ N/mm}^2$ .

The adhesion figures were thus above the required minimum value of  $25 \text{ N/mm}^2$ .

- Summarizing Assessment of the Results

The coating characteristics specified by NUKEM and tested by an independent expert were achieved. This statement is qualified only with regard to the freedom of the coatings from pores, since isolated pores could be observed both in the NiCr 80 20 coating applied with air as working gas and in that applied using argon. However, the diameter of such pores remained at an order of magnitude of a few  $10 \text{ }\mu\text{m}$ . Porosity, however, does not penetrate through, as the corrosion tests which were performed prove.

The tests also furnished proof that repair spraying can be performed without impairing the specified requirements of the coatings.

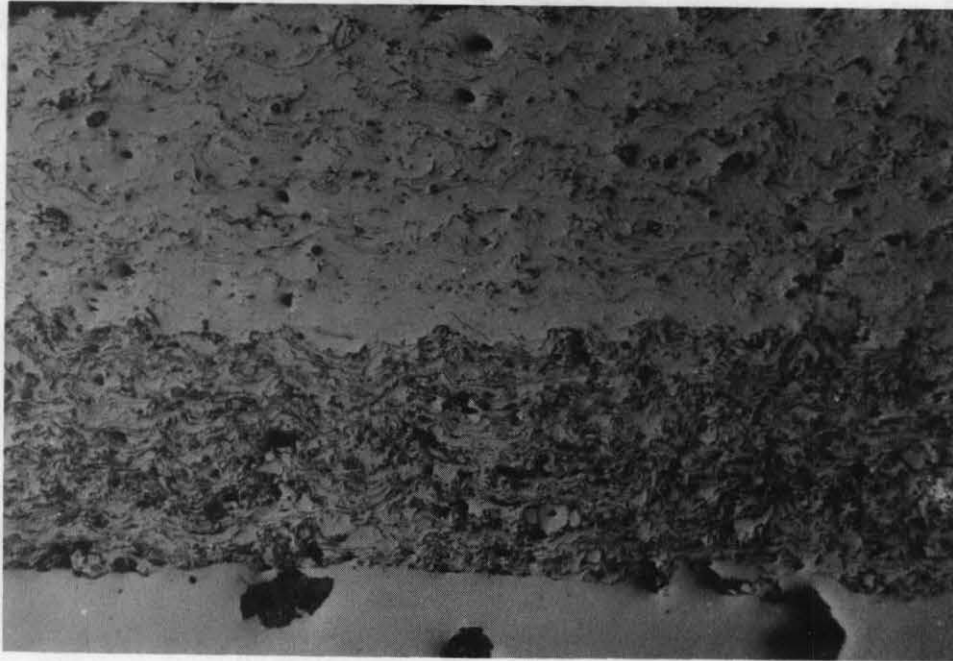


Fig. 1. Typical structure of a hammered surface  
(parent material, adhesion layer, covering layers)  
enlargement 60:1

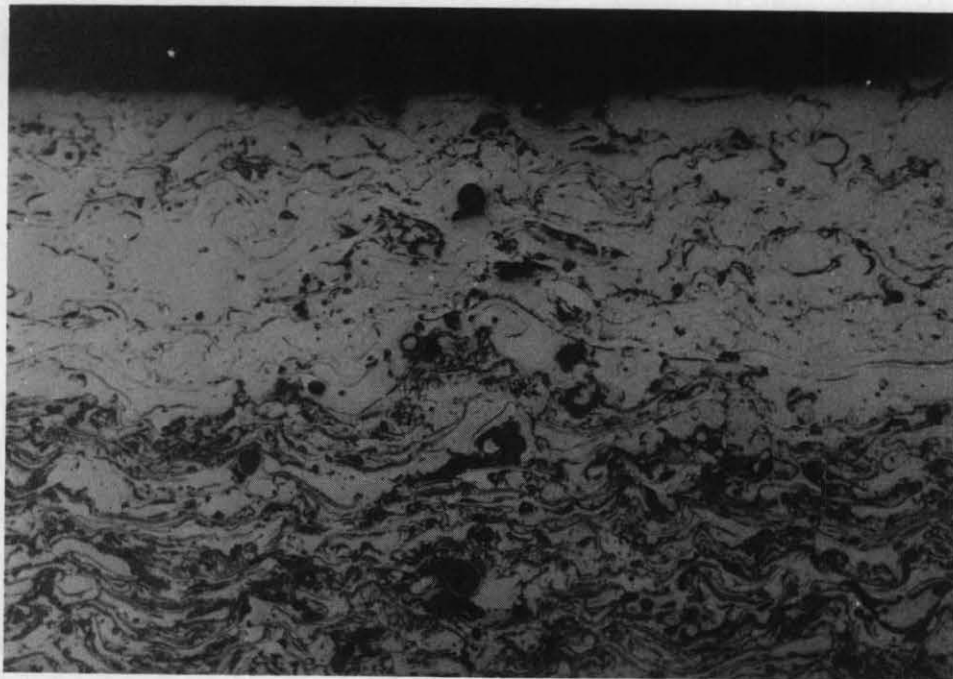


Fig. 2. Transition area from a repaired defect  
to the original coating  
enlargement 60:1