



Multi-layer coating system for circular steel hollow sections, comprising a three-layer polyethylene coating and a polyamide-based top coat— Requirements and tests

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Author Q, Dr. Hocks Date:	Checked by: Q, Dr. Löbbe Date:	Approved by: Q, Weil Date:



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**Multi-layer coating system for circular steel hollow sections, comprising a three-layer polyethylene coating and a polyamide-based top coat—
Requirements and tests**

Preface

This Mill Standard has been issued for a multi-layer coating system comprising a polyethylene (PE) coating and a top coat of polyamide (PA), which is used on circular steel hollow sections. The design does not provide for adhesion between the polyethylene coating and the polyamide top coat. The coating system is also suitable for use on offshore installations.



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Previous editions

— None



Multi-layer coating system for circular steel hollow sections, comprising a three-layer polyethylene coating and a polyamide-based top coat— Requirements and tests

1 Scope

This Mill Standard defines the requirements on a multi-layer coating system which consists of a polyethylene coating and a polyamide top coat. The coating system is suitable longitudinally welded and spiral-weld steel tube & pipe as well as steel fittings. It can be used at service temperatures of between -40°C and +80 °C.

2 Normative references

The following are normatively referenced documents are indispensable in the application of this Mill Standard. In the case of dated references, only the edition cited applies. With undated references, the latest edition of the referenced document (including all amendments, if any) applies.

DIN 30670, Polyethylene coatings of steel pipes and fittings — Requirements and tests

DIN EN 10204, Metallic products — Types of inspection certificates

DIN EN ISO 306, Plastics — Thermoplastic materials — Determination of the Vicat softening temperature (VST)

DIN EN ISO 307, Plastics — Polyamides — Determination of the viscosity number

DIN EN ISO 527-1, Plastics — Determination of tensile properties — Part 1: General principles

DIN EN ISO 527-2, Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion masses

DIN EN ISO 868, Plastics and ebonite — Determination of indentation hardness by means of a durometer (Shore hardness)

DIN EN ISO 1133-1, Plastics — Determination of the mass-flow rate (MFR) and the melt volume flow rate (MVR) of thermoplastics — Part 1: Standard method

DIN ISO 1183-1 Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pycnometer method and titration method

DIN EN ISO 2808, Paints and varnishes — Determination of film thickness

DIN EN ISO 4892-2, Plastics — Methods of exposure to laboratory light sources — Part 2: Xenon-arc lamps

DIN EN ISO 9001, Quality management systems — Requirements

DIN EN ISO 11357-1, Plastics — Differential scanning calorimetry (DSC) — Part 1: General principles

DIN EN ISO 11357-3, Plastics — Differential scanning calorimetry (DSC) — Part 3: Determination of temperature and enthalpy of melting and crystallization

E DIN EN ISO 15512, Plastics — Determination of water content

DIN EN ISO 21809-1, Petroleum and natural gas industries - External coatings for buried or submerged pipelines used in pipeline transportation systems - Part 1: Polyolefin coatings

ISO 20340, Paints and varnishes -- Performance requirements for protective paint systems for offshore and related structures



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3 Terms and definitions

The terms and definitions used in this Mill Standard are in accordance with DIN 30670 and DIN EN ISO 21809-1.

4 Symbols and abbreviation

A ₀	Elongation at break in %
W	Impact energy in J
L ₀ , L ₁	Length values for the determination of elongation (same unit, i.e. m or mm)
DSC	Differential Scanning Calorimetry
S	Surface area in m ²

5 Multi-layer coating

The corrosion protection coating consists of three layers: an epoxy resin primer, an adhesive agent, and an extruded polyethylene coating to DIN 30670 or DIN EN ISO 21809-1. The epoxy resin primer is applied as a powder. The adhesive agent can either be applied as a powder or extruded over the primer. The polyethylene layer is applied by either blown film extrusion or the extrusion wrapping process.

As an additional protection against adverse weather effects and mechanical damage, a top coat of polyamide is extruded over the three-layer corrosion protection coating.

6 Ordering information

6.1 Mandatory information

The following information is mandatory:

- Number and dimensions of the pipes or fittings to be coated;
- Reference to this Mill Standard;
- Technical delivery conditions for the polyethylene coating.

6.2 Ordering options

The following information may be requested as optional information:

- Deviating layer thicknesses;
- Test certificate, e.g. to test certificate 2.2 to DIN EN 10204 or other types of test certificates to DIN EN 10204;
- Deviating test frequencies.



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7 Coating application

7.1 General

The coating materials shall be selected by the coating manufacturer, because they can judge best which materials are optimally suited in conjunction with their equipment and processes to meet the requirements specified in the applicable standards. While the three-layer corrosion protection coating calls for due consideration of the epoxy resin primer, the adhesive agent and the extruded polyethylene layer, the multi-layer coating system dealt with in the present Mill Standard features an additional polyamide top coat for protection against adverse weather conditions and mechanical impact. The color of the polyamide coat should be clearly distinguishable from that of the polyethylene coating.

7.2 Coating structure

7.2.1 Polyethylene coating

The three-layer polyethylene coating is subject to the requirements of the agreed-upon technical delivery conditions.

7.2.2 Polyamide top coat

Table 1 below provides an overview of the technical data specified in the delivery documents and the technical datasheets for the polyamide top coat. The minimum layer thickness of the mechanical protection is 2 mm. For particularly high mechanical loads, a greater thickness can be agreed upon. The layer thickness check is carried out as specified in 0.

Table 1 — Mandatory information in the product manufacturer's datasheets and delivery documents

Material	Property	Test basis	Mill certificate: Certificate of conformance (COC) or batch certificate(BC)	Technical datasheets
General information	Material designation	-	COC, BC	ja
	Product manufacturer	-	COC, BC	ja
	Storage conditions	-	-	ja
	Batch number	-	BC	-
	Melting point DSC (ΔT_{q2})	DIN EN ISO 11357- 1/3	BC	ja
	Yield stress	DIN EN ISO 527	-	ja
	Elongation at break	DIN EN ISO 527	-	ja

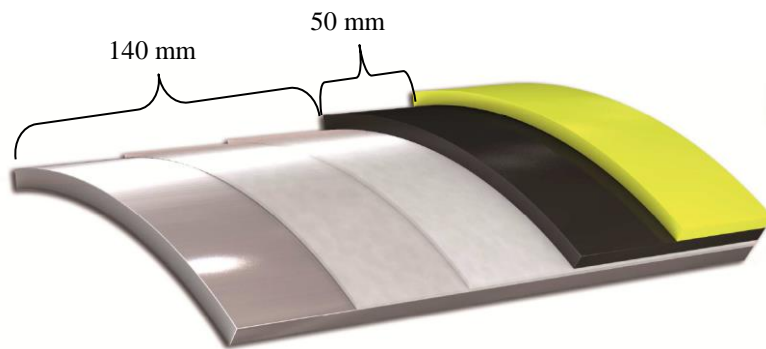


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Material	Property	Test basis	Mill certificate: Certificate of conformance (COC) or batch certificate(BC)	Technical datasheets
	Ultimate stress	DIN EN ISO 527	-	ja
	Shore hardness D	DIN EN ISO 868	-	ja
	Density	DIN EN ISO 1183-1	C	ja
	Water content	E DIN EN ISO 15512	C	ja

7.2.3 Pipe end design

The requirements for the polyethylene coating at the pipe ends are defined as part of the agreed-upon technical delivery conditions. The polyamide top coat on the polyethylene coating is cut back over a length of at least 50 mm. Beveling of the polyamide top coat is not required.



8 Requirements

8.1 General

The requirements on coatings in accordance with this Mill Standard apply at the time of delivery and serve for quality assurance of the coating materials used and for monitoring the of the coating process.

8.2 Material properties

The requirements on the three-layer coating for corrosion protection based on polyethylene are defined in the agreed-upon delivery conditions.



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The properties of the polyamide top coat and the related tests are compiled in Table 2. DSC measurements are performed on polyamide granules as part of the incoming inspection. The melting point shall be compared with the data given in the delivery documents.

Table 2 — Properties of polyamide granules and polyamide top coats

Property	Specified requirement	Test according to:	Notes
Melting point DSC (ΔT_{g2})	-	Appendix A	Incoming inspection of raw material (polyamide granules). The melting point must match the specifications given in the delivery documents.
Layer thickness	min. 2 mm	Appendix B	
Shore hardness D	min. 65	DIN EN ISO 868	Shore hardness is determined on the pipe coating.
Elongation at break (23 °C ± 2 °C)	min. 200 %	Appendix C	
Low-temperature impact resistance (-40 °C ± 2 °C)	min. 10 J/mm	Appendix D	25 kV test: There must be no crack formation.
Indentation resistance (23 °C ± 2 °C)	max. 0.075 mm	Appendix E	
UV resistance	min. 150 %	Appendix F	Testing of elongation at break
Thermal ageing resistance	min. 150 %	Appendix G	Testing of elongation at break

8.3 Production monitoring

8.3.1 Polyethylene coating

Before applying the polyamide top coat over the polyethylene coating, it must be ensured that the latter complies with the customer's order specifications.

8.3.2 Polyamide top coat

The relevant parameters of the extrusion line and process and of the appearance of the top coat must be regularly checked. The top coat must be uniform in color and surface appearance. It must also be free from pores and other defects that affect the quality of the coating system.



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9 Testing

9.1 General

The tests for the determination of the coating and coating material properties are described in the Appendices to this Mill Standard. A distinction must be made between production tests and system tests.

In the system test, the material properties and functional requirements of the coating components are verified and documented in line with this Mill Standard. Coating producers who comply with the requirements of DIN EN ISO 9001 or an equivalent management system can carry out the system test themselves.

Production monitoring is ensured by the coating producer or an authorized material testing institute. If the requirements specified in this Mill Standard are not met during these in-process inspections, the test shall be repeated on twice the number of samples from the same batch. If these also fail to meet the requirements, the whole batch shall be rejected.

9.2 Test certificates

A test certificate 2.2 to DIN EN 10204:2005-01 shall be issued as verification that all the specified requirements have been met. Other certificate types are subject to special agreement.

9.3 Testing type and frequency

The testing type and frequency for the three-layer polyethylene coating is defined in the agreed-upon technical delivery conditions. The testing type and frequency for the polyamide top coat are listed in Table 3. Other test procedures are subject to special agreement.

Table 3 — Testing type and frequency for the polyamide top coat

Testing type	Frequency	In-process test	System test
Melting point DSC (ΔT_{g2})	1/batch	X	X (1 sample)
Layer thickness	3/shift	X	—
Shore hardness	1/shift	X	X (5 samples)
Elongation at break	1/shift	X	X (5 samples)
Low-temperature impact resistance	1/shift	X	X (1 sample)
Indentation resistance	1/shift	X	X (3 samples)



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Testing type	Frequency	In-process test	System test
UV resistance	—	—	X (1 set of samples)
Thermal ageing resistance	—	—	X (1 set of samples)

10 Marking

For identification purposes, a label is affixed to each pipe. It shall contain the following information:

- Name of pipe manufacturer;
- Reference to the applicable pipe standard;
- Reference to the standard applicable to the corrosion protection coating;
- Reference to this Mill Standard.



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Appendix A
(normative)

DSC measurements

A.1 General

The incoming polyamide granulate is inspected by differential scanning calorimetry (DSC) thermal analysis as described in DIN EN ISO 11357-1 and -3. The measurements shall be performed in accordance with these standards, unless otherwise specified in the present Appendix.

A.2 Test equipment

The test equipment consists of the following components:

- DSC tester with cooling device;
- Aluminum sample carrier with lid;
- If necessary, nitrogen gas supply, dry, analytical grade.

A.3 Process description

A.3.1 Measuring procedure

The measurement, performed on a granule taken from the delivered batch, comprises the following steps:

- Heating of the sample from 25 °C to 230 °C at a rate of 20 K/min, followed by rapid cooling to 25 °C and an isothermal phase of 1.5 minutes;
- Heating of the sample from 25 °C to 230 °C at a rate of 20 K/min.

A.3.2 Evaluation

The first heating step serves primarily to remove residual moisture from the sample. In the second step, the melting point of the polyamide is determined as shown in Figure A.1.



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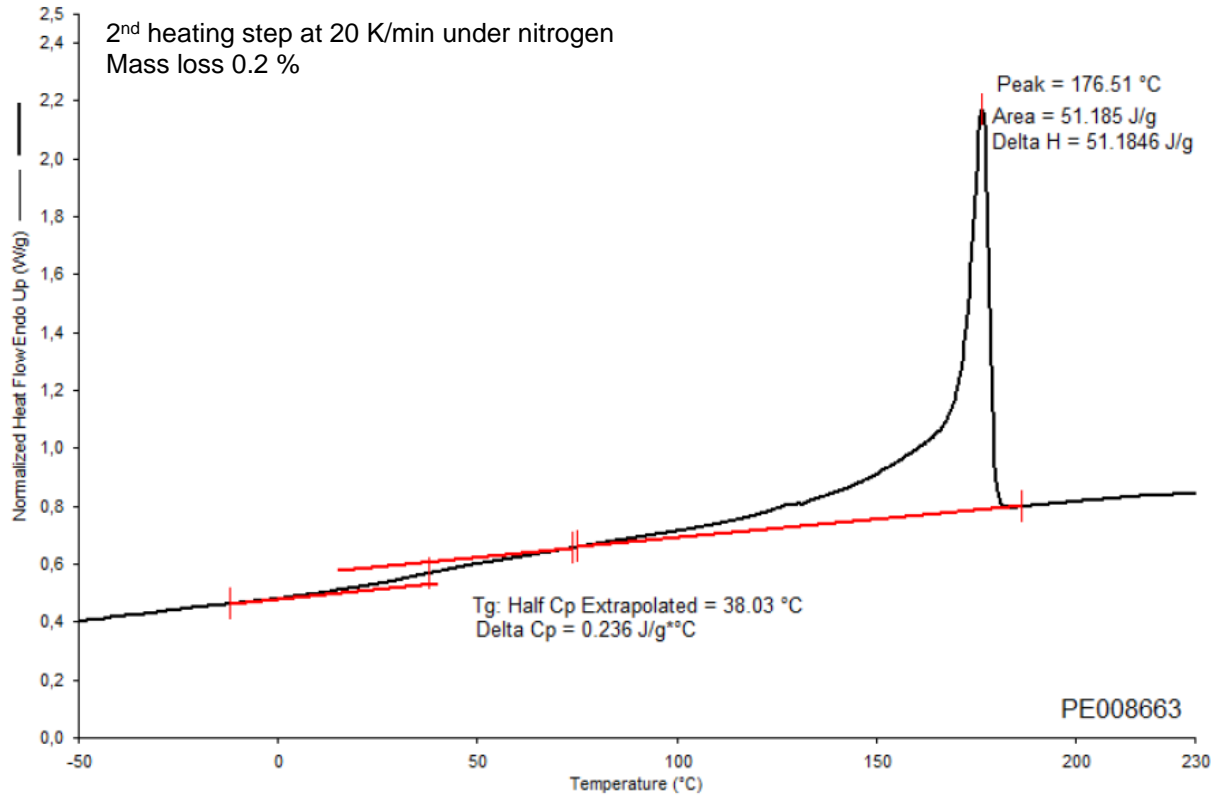


Figure A.1: Determination of the melting point during the second heating step



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Appendix B
(normative)

Thickness check

The thickness of the polyamide top coat is equal to the difference between the total coating thickness and the thickness of the corrosion protection coating. Alternatively, it can be measured directly when cutting it back at the pipe ends. The thickness of the corrosion protection coating is measured at the pipe ends.

Procedures for coating thickness checks are described in DIN EN ISO 2808. The thickness of the complete coating system and of the corrosion protection coating is checked using a nondestructive method in accordance with DIN EN ISO 2808.

This nondestructive check is performed with the aid of an electro-magnetic, eddy current or ultrasonic measuring device with an accuracy of $\pm 10\%$. The device must be designed and calibrated for the thickness range concerned. To determine the total coating thickness, a minimum of twelve measurements must be performed on each pipe to be examined.



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Appendix C
(normative)

Elongation at break

C.1 General

Elongation at break is tested in accordance with DIN EN ISO 527-1. The test samples are produced and selected in accordance with DIN EN ISO 527-2.

C.2 Test equipment

The test shall be performed using a tension device that allows the recording of a force-path diagram.

C.3 Process description

C.3.1 Sample preparation

The test shall be performed on samples taken from the polyamide top coat. After a circumferential incision, the polyamide top coat can be easily separated from the polyethylene coating.

C.3.2 Measuring procedure

Elongation at break is tested on sample types 1B, 5A or 5B (cf. Figs. 1 and A.2, DIN EN ISO 527-2) at a temperature of $23\text{ °C} \pm 2\text{ °C}$.

The following test speeds according to Table C.1 below shall be observed.

Table C.1 — Test speeds

Sample type to DIN EN ISO 527-2	Test speed to DIN EN ISO 527-1 mm/min
1B	50
5A	25
5B	10



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C.3.3 Evaluation

Elongation at break (A_0 in %) is determined according to the following equation:

$$A_0 = \frac{L_1 - L_0}{L_0} \cdot 100$$

where

A_0 = Elongation at break in %;

L_0 = Initial length (distance) between measuring points;

L_1 = Distance between measuring points at sample break.

The test result is calculated as the arithmetic mean of five samples.



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Appendix D
(normative)

Low-temperature impact resistance

D.1 General

Impact resistance is determined with the aid of a weight dropped with a defined impact force (cf. Table 2).
The polyamide top coat is tested at -40 °C.

D.2 Test equipment

The impact resistance test is carried out using a device with a drop weight that impinges vertically onto the polyamide top coat removed from the pipe. The drop weight device shall be guided to ensure minimum friction. The accuracy of the test should not be impaired by any air build-up. The front face of the drop weight is part of the surface of a sphere with a diameter of 25 mm. The drop height of the weight is about 1 m.

D.3 Process description

D.3.1 Sample preparation

The test shall be performed on samples from the polyamide top coat. After a circumferential incision, the polyamide top coat can be easily separated from the polyethylene coating.

D.3.2 Measuring procedure and evaluation

The sample is subjected to five impacts. The distance between two impact points should be at least 30 mm. Using a high-voltage test device, the sample is then checked for defects. Punctures are not permissible.



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Appendix E
(normative)

Indentation resistance

E.1 General

This test consists of measuring the indentation of a punch in the coating under defined conditions in terms of temperature and the load applied.

E.2 Test equipment

The test equipment comprises a 250-g metal rod with an additional weight. A metal pin with a flat face shall be fitted at the center of the lower end of the rod. The diameter of the flat face shall be 1.80 mm \pm 0.05 mm, corresponding to an indentation area of 2.5 mm². The total mass shall be 2.5 kg, resulting in a surface pressure of 10 N/mm². Furthermore, a penetrometer with a dial gauge having an accuracy of 0.1 mm is required. The test shall be conducted at room temperature.

E.3 Process description

E.3.1 Sample preparation

The test shall be performed on samples from the polyamide top coat. After a circumferential incision, the polyamide top coat can be easily separated from the polyethylene coating.

E.3.2 Measuring procedure and evaluation

After conditioning the test sample at test temperature (RT) for about 1 h, the punch (without the additional weight) shall be lowered slowly and carefully to the sample surface, and the zero value set at the penetrometer within 5 s. Then the additional weight shall be attached to the punch and, after a loading time of 24 h, the depth of penetration read from the penetrometer.



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Appendix F
(normative)

UV resistance

F.1 General

To test their resistance to UV ageing, polyamide samples are exposed to continuous irradiation under xenon light at defined temperature and humidity conditions. Ageing-related changes are assessed on the basis of the change in elongation at break according to Appendix C.

F.2 Test equipment

The test equipment shall enable the samples to be aged under the specified test conditions and shall consist of an irradiation chamber fitted with a xenon arc lamp (cf. DIN EN ISO 4892-2, Method A).

F.3 Process description

F.3.1 Sample preparation

The test shall be performed on samples taken from the polyamide top coat. After a circumferential incision, the polyamide top coat can be easily separated from the polyethylene coating.

F.3.2 Measuring procedure

Measurements shall be carried out in accordance with DIN EN ISO 4892-2, Method A, Exposure Cycle no. 1. The requirements specified in DIN EN ISO 21809-1 shall be observed.

Samples for determining elongation at break according to Appendix C shall be taken both before exposure to irradiation with 7 GJ/m² (initial value) and afterwards.

F.3.3 Evaluation

The variations of the arithmetic mean values shall be documented and evaluated after exposure to 7 GJ/m².

Note: The specification of 7 GJ/m² applies to pipe for buried pipelines with a view to their storage before pipe-laying. ISO 20340 for offshore structures specifies a lower, i.e. less exacting, value.



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Appendix G
(normative)

Thermal ageing resistance

G.1 General

To test resistance to thermal ageing, polyamide samples are exposed to dry heat in a circulating-air oven. Ageing-related changes are assessed on the basis of the change in elongation at break according to Appendix C.

G.2 Test equipment

The test shall be performed on samples that have been aged in a thermostatically controlled circulating-air oven capable of maintaining a test temperature constant within ± 2 °C.

G.3 Process description

G.3.1 Sample preparation

The test shall be performed on samples from the polyamide top coat. After a circumferential incision, the polyamide top coat can be easily separated from the polyethylene coating.

G.3.2 Measuring procedure

The samples are exposed to a temperature of 100 °C for 200 days (4,800 h). Samples for measuring elongation at break according to Appendix C are taken before the start of exposition (initial value) and after every 800 h of exposure to heat.

G.3.3 Evaluation

The variations of the arithmetic mean values shall be documented. The final evaluation shall be performed after the exposure time specified above for each type of coating.