

Substances H4 and H4 HD Patinal®

GENERAL INFORMATION

Substances H4 and H4 HD Patinal® are high refractive index evaporation materials for the vacuum deposition of highly transparent homogeneous thin film coatings. They are composed of a mixture of titanium oxide and lanthanum oxide to form a stable LaTiO_3 phase. Substances H4 and H4 HD Patinal® allow for very stable process conditions as well as stress compensation in multilayer stacks and are thus very well suited for high precision optical coatings.

AREAS OF APPLICATION

- High quality AR and other optical multilayer coatings for NUV, VIS and NIR on glass, crystalline substrates and polymers
- High precision filters requiring low stress levels
- Low absorption coatings on temperature sensitive substrates such as polymers
- Protective and reflection enhancing coatings on metals in combination with e.g. SiO_2

MAIN FEATURES OF SUBSTANCE H4 HD PATINAL® vs. SUBSTANCE H4 PATINAL®

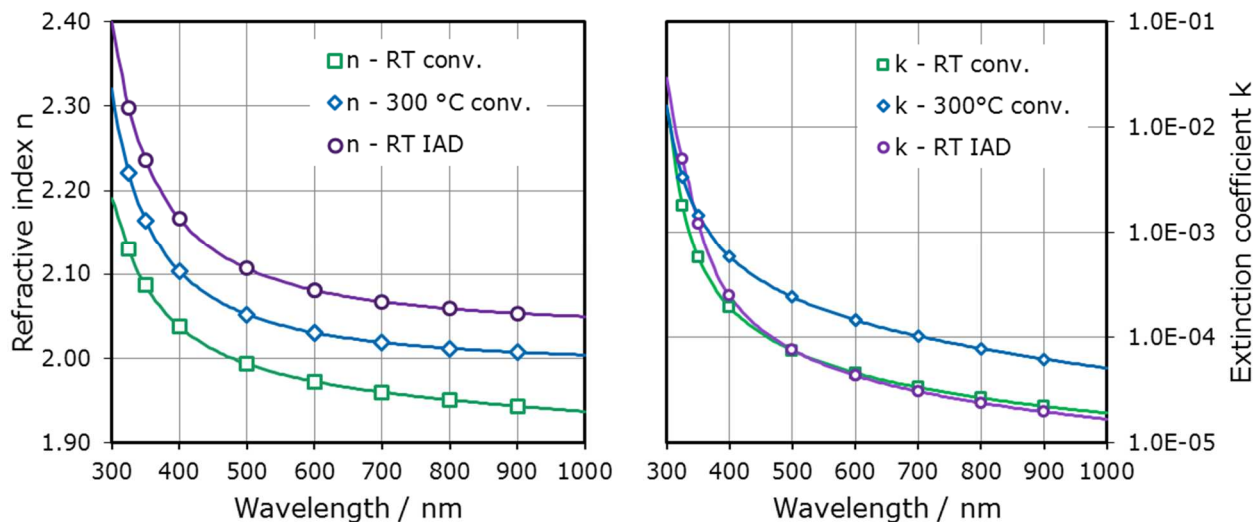
- High apparent density (almost doubled compared to Substance H4 Patinal®) can free up crucible positions for other materials or allows to produce thicker layer systems in one coating process, autofeeder systems only need half the volume compared to Substance H4 Patinal®
- Easier processing due to eliminated sintering contraction during premelt
- Reduced process preparation time as premelting requires fewer refills
- Improved material and layer homogeneity



THIN FILM PROPERTIES

Range of transparency	350 nm – 7 μm
Refractive index at 500 nm	
• conventional $T_s = 300\text{ °C}$ / no IAD	~ 2.05
• conventional $T_s = 50\text{ °C}$ / no IAD	~ 2.00
• IAD – $T_s = \text{RT}$	~ 2.10
Thin film stress	Neutral – low tensile

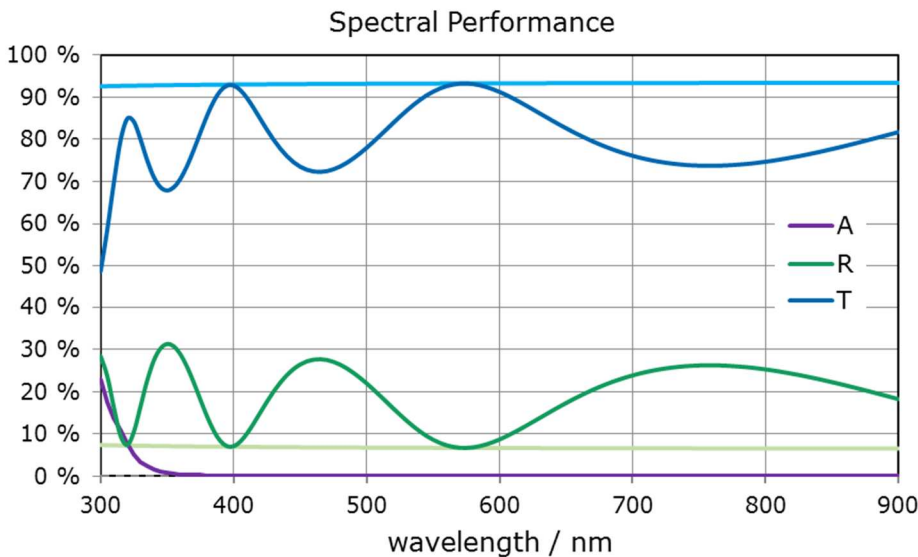
Optical Dispersion



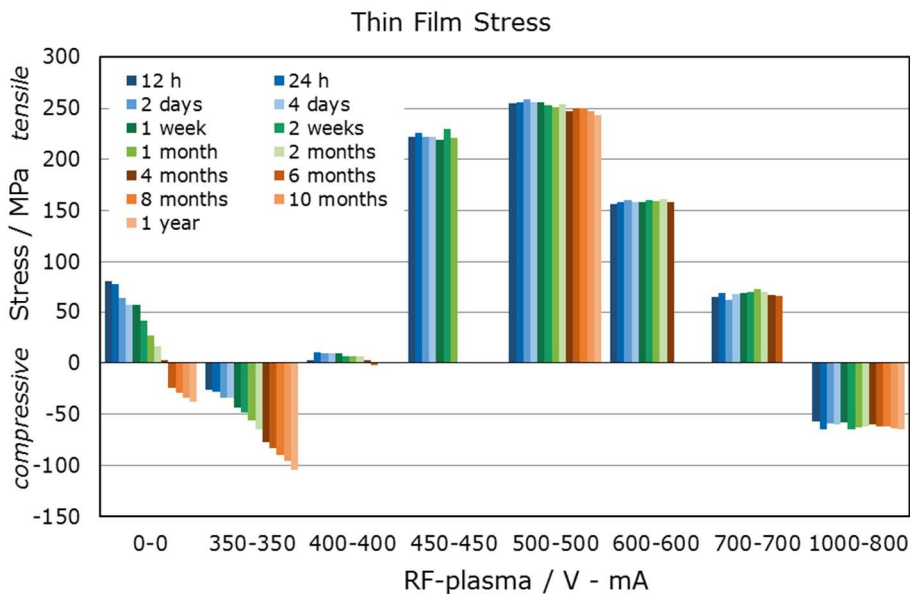
wavl / nm	300	350	400	500	650	800	1000
n - RT conv.	2.19	2.09	2.04	1.99	1.97	1.95	1.94
n – 300 °C conv.	2.32	2.16	2.10	2.05	2.02	2.01	2.00
n - RT IAD	2.40	2.24	2.17	2.11	2.07	2.06	2.05
k - RT conv.	1.5E-02	5.7E-04	1.9E-04	7.5E-05	3.9E-05	2.7E-05	1.9E-05
k – 300 °C conv.	1.6E-02	1.5E-03	5.9E-04	2.4E-04	1.2E-04	7.8E-05	5.1E-05
k - RT IAD	3.0E-02	1.2E-03	2.5E-04	7.6E-05	3.6E-05	2.4E-05	1.7E-05



The following figure shows adsorption, transmittance and reflectance spectra typical for films of Substance H4 Patinal® (thickness ~275 nm, substrate temperature 280°C, substrate fused silica).



The thin film stress of coatings made with Substances H4 or H4 HD Patinal® can be adjusted over a wide range by applying a plasma or ion source for further densification of the layers. Unlike many other high refractive index materials, fully densified layers made of H4 show a tensile stress mode. Substance H4 and H4 HD Patinal® can be used to compensate compressive stress of SiO₂ layers in a coating design.



RF-plasma / V – mA	1 day	1 month	1 year
0 – 0 (conv.)	78 MPa	27 MPa	-38 MPa
350 – 350	-28 MPa	27 MPa	-104 MPa
400 – 400	11 MPa	7 MPa	-
500 – 500	256 MPa	251 MPa	244 MPa
700 – 700	69 MPa	73 MPa	-

NOTES FOR EVAPORATION

Evaporator source	Electron beam evaporator
Liner	Copper crucible or Mo-Liner
Melting temperature	1800 °C
Evaporation temperature	2200 – 2300 °C
Deposition rate	0.2 – 0.8 nm/s
Oxygen partial pressure	0.8 - 2.0·10 ⁻⁴ mbar
IAD settings (RF Source)	IAD with O ₂ , 350 mA / 350 V to 700 mA / 700 V, max. refractive index > 450 mA / 450 V
Substrate temperature	30 – 300 °C
QCR-settings	Density 5.9 g/cm ³ , z-ratio 1.0

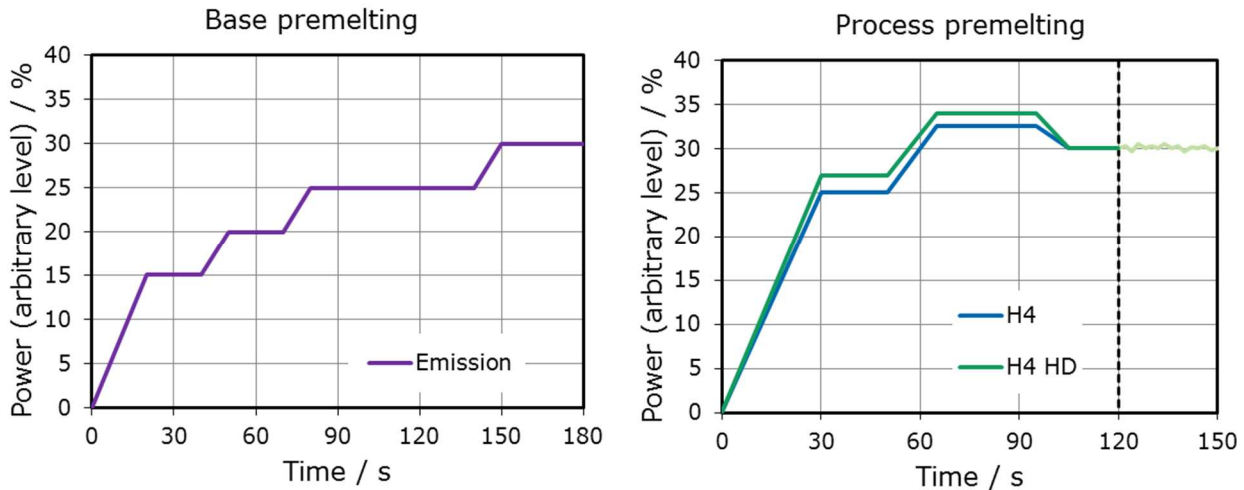
Substances H4 and H4 HD Patinal® should preferably be evaporated by electron beam deposition from a molybdenum liner, not directly from a water-cooled copper crucible. For optimum flatness of the melt a circular sweep pattern at low frequency (< 20 Hz) is recommended with a softly defocussed electron beam. A beam overlap in the center of the liner should be avoided (ring shaped pattern). Other sweep techniques depend on the available equipment.

Alternatively, H4 Patinal® can also be deposited by thermal evaporation from a Mo-boat as well, which is recommended for example when sensitive polymer substrates have to be protected against direct exposure to e-gun radiation (e.g. UV).

The thin film stress of layers made from Substance H4 and H4 HD Patinal® can be adjusted to a permanent and longterm stable tensile mode. Shift free and dense layers require an RF plasma power of greater than 400 mA / 400 V. For the highest refractive index of about 2.1 a RF plasma power of at least 450 mA / 450 V is required. Setting the plasma power to high levels can result in coating damage and an increased absorption.



The following figure shows the preconditioning process used for Substance H4 and H4 HD Patinal®. For Substance H4 HD Patinal® the initial power settings are slightly higher due to the higher heat capacity of the dense granules.



Preparation of the base melt requires thorough melting to avoid any remaining loose granules, especially for the first fillings. Residual granules can initiate the formation of holes and cavities in the melt. One crucible filling can be used for many consecutive layers without affecting the refractive index. The highest repeatability of the layer thickness distribution can be achieved by regular refilling of the crucible to an identical starting level before the next coating run.



PRODUCTS

Substance H4 and H4 HD Patinal® are available as granules in different size distributions.

Product Code	Description	Purity*	Dimensions
1.08332	Substance H4 Granules Patinal®	≥ 99.95 % (3N5)	Granules, about 1 – 4 mm
1.15591	Substance H4 Granules Patinal®	≥ 99.95 % (3N5)	Granules, about 0.1 – 2 mm
1.00230	Substance H4 HD Granules Patinal®	≥ 99.95 % (3N5)	Granules, about 0.1 – 2 mm
1.02437	Substance H4 HD Granules Patinal®	≥ 99.95 % (3N5)	Granules, about 0.8 – 4 mm

*The purity values are based on the specified trace metals.

Appearance

1.08332	Dark grey granules
1.15591	Dark grey granules
1.00230	Dark grey granules
1.02437	Dark grey granules



SPECIFICATION

Cobalt (Co)	≤ 0.001 %
Copper (Cu)	≤ 0.001 %
Chromium (Cr)	≤ 0.005 %
Iron (Fe)	≤ 0.005 %
Vanadium (V)	≤ 0.01 %

RoHS information

The RoHS compliance information is part of the Certificate of Analysis (CoA) for each batch of Patinal® material.

Sizes

1.08332	Granules 1 - 4 mm ≥ 80 %
1.15591	Granules 0.1 - 2 mm ≥ 80 %
1.00230	Granules 0.1 - 2 mm ≥ 80 %
1.02437	Granules 0.8 - 4 mm ≥ 80 %

Application test

Each batch has to pass a specific application test assessing its evaporation behaviour.



Quality assurance

Research, production and sales of our Patinal® evaporation materials take place under a certified DIN EN ISO 9001 quality management system and DIN EN ISO 14001 environmental management system. The quality of the materials is assured by our manufacturing processes, in-process controls and quality tests. Each batch is released only after passing our chemical analysis and application tests designed to confirm the suitability of the material for the evaporation process.

Handling precautions

Product safety information required for safe use is not included in this document. Before handling, read product and safety sheets and container labels for safe use, physical and health hazard information. The material safety data sheet is available online at www.patinal.com, from your EMD representative or distributor, or by calling your global Merck KGaA, Darmstadt, Germany, contact.

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