Patinal®

Evaporation Materials

PRODUCT INFORMATION

Substance H4 Patinal®

EMD Chemicals Inc. Phone 856-423-6300
Internet: www.emdchemicals.com Mail: optics@emdchemicals.com

Substance H4 Patinal®

NOTES AND EXPLANATIONS

We advise our customers regarding technical applications to the best of our knowledge within the scope of the possibilities open to us, but without obligation. Current laws and regulations must be observed at all times. This also applies in respect to any protected rights of third parties. Our suggestions do not relieve our customers of the necessity to test our products, on their own responsibility, for suitability for the purpose envisaged. Quotations from our literature are only permitted with our written authority, and the source must be stated.

The products information sheet is based on our own investigations and on literature data. The properties of the materials in thin coatings are affected by the choice of deposition conditions. For this reason, coating properties quoted are to be understood as being typical values and cannot be guaranteed. As far as possible, the conditions under which the coating properties were achieved are indicated.

QUALITY CONTROL

During quality control of the products analyses were performed with the aim to measure those properties of the products which are of major importance for the applicability of the products and the properties of the coatings. Chemical analysis is performed to determine the specified impurities. Furthermore application testing is performed to analyse the behaviour of the product during melting and evaporation. Also thin coatings are made to analyse transmittance and refractive index. The values listed in the specification are guaranteed impurity limits determined on representative samples of each production batch.

SAFETY NOTE

Working safety requires that products which are formed during evaporation to be kept in the closed system. If fine dust develops during cleaning of evaporation equipment suitable respiratory protection must be provided (approved respirators).

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GENERAL INFORMATION

Substance H4 has been developed with the aim to provide a material for vacuum deposition of highly transparent homogeneous high index layers for antireflection coatings and multilayers on mineral glasses, crystalline and plastic substrates in VIS and NIR.

The transmittance range of layers of Substance H4 is from about 360 nm to about 7 µm. The material can be used on heated and on unheated substrates. Substance H4 is provided in form of granules in several sizes.

Substance H4 is protected by patents in various countries including Germany, USA, Japan and China.

ITEMS AND PACKING SIZES

1.08332.0150	Substance H4 granules about 1-4 mm Patinal® 100 g pack
1.08332.1050	Substance H4 granules about 1-4 mm Patinal® 1 kg pack
1.15591.0150	Substance H4 granules about 0.1-2 mm Patinal® 100 g pack
1.15591.1050	Substance H4 granules about 0.1-2 mm Patinal® 1 kg pack

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SPECIFICATION

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

1.08332	Particle size (1-4 mm)	≥ 80%
1.15591	Particle size (0.1-2 mm)	≥ 80%

Application test conforms

RoHS information:

Cd (Cadmium)	≤ 0,01 %
Cr (Chromium)	≤ 0,1 %
Hg (Mercury)	≤ 0,1 %
Pb (Lead)	≤ 0,1 %
PBB (polybrominated biphenyls)	≤ 0,1 %
PBDE (polybrominated diphenyl ethers)	≤ 0,1 %

The Chromium(VI) concentration (RoHS requirements: ≤ 0.1 %) is always smaller than or equal to the total chromium concentration.

PROPERTIES OF THE BULK SUBSTANCE

Chemical composition:	Titanium oxide / lanthanum oxide mixture
Appearance:	dark grey granules
Melting temperature:	about 1800 °C
Evaporation temperature:	2200 – 2300 °C

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EVAPORATION PROCEDURE

Evaporation temperature of Substance H4 is about 2200 °C. H4 should preferably be evaporated with electron beam from a Molybdenum liner, not directly from a water-cooled copper crucible. The use of a hot liner will improve the flatness of the molten surface and improve the yield. Intensive cooling should be avoided.

It is possible to deposit thin layers of H4 from a Mo-boat, for example when plastic substrates should be protected against direct exposure to E-Beam radiation.

H4 can be used without pre-treatment. For optimum flatness of the melt it is recommended to use a low frequency (~3-6 Hz) circularly sweeping, softly defocussed electron beam around the outer border of the liner. Without an overlap in the center of the liner.

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RECOMMENDED COATING CONDITIONS

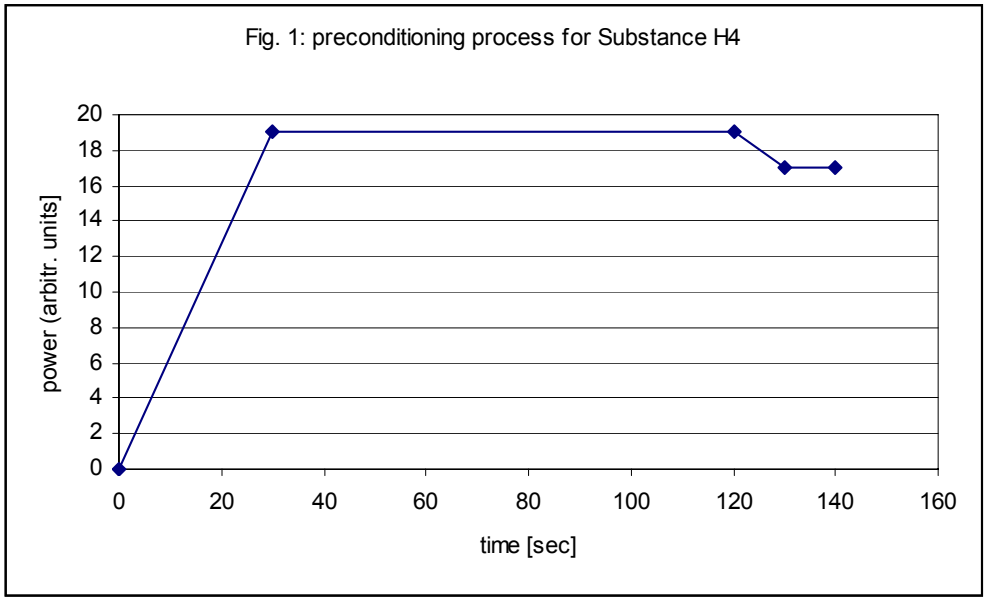
Evaporation source	Electron beam copper crucible, water-cooled with warm/hot molybdenum liner
Oxygen partial pressure	$0.8 – 2 * 10^{-4}$ mbar
Deposition rate	0.2 – 0.8 nm/sec
Substrate temperature	30 – 300 °C
Density setting	5.9 g/cm^3
Z-ratio	1.0

Substance H4 melts at a temperature below the evaporation temperature and therefore forms a flat melted surface. So a homogeneous thickness distribution can easily be maintained. The following procedure is recommended:

The material is filled into the crucible and pre-melted in vacuum below a shutter. If necessary fresh material can be refilled and pre-melting is repeated. One crucible filling can be used for several consecutive layers without influence on the stability of the refractive index. Evaporated material can be replenished as well.

The following figure shows the preconditioning process used for Substance H4.

Fig. 1: preconditioning process for Substance H4



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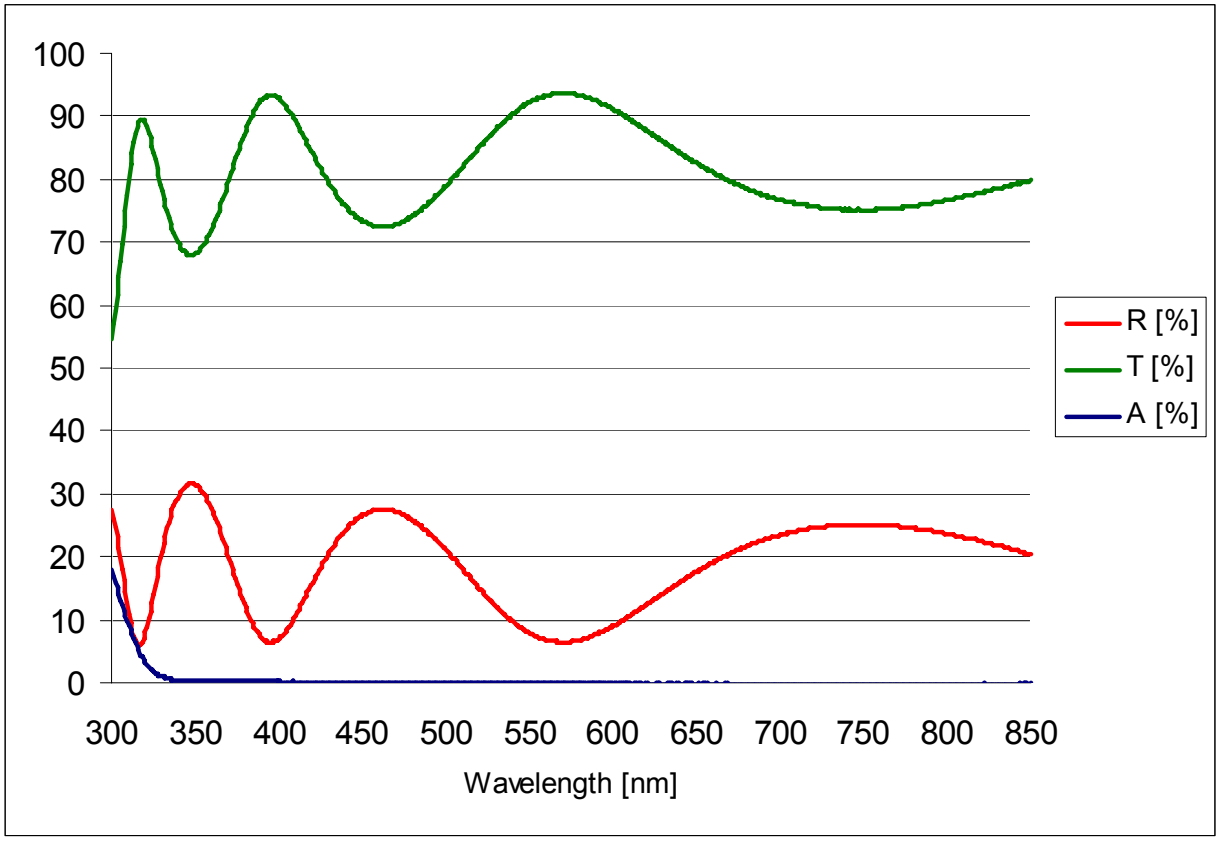
PROPERTIES OF THIN FILMS

The following figure shows transmittance and reflectance spectra typical for films of Substance H4.



The physical thickness of the film is about 275 nm. The film was deposited on a fused silica substrate at about 280 °C substrate temperature. The short wavelength cut off is at about 330 nm, the transmittance in IR extends to about 7 µm.

The influence of oxygen pressure during deposition on the optical properties is small as compared to oxides like TiO_2 or titanium suboxides, Ta_2O_5 or Substance H2. Absorption is not detectable by spectroscopic methods above 400 nm for coatings deposited with oxygen pressures in the range of $8 * 10^{-5} – 2 * 10^{-4}$ mbar (detection limit about .1 %, absorption index about $2 * 10^{-4}$). Only at pressures of $5 * 10^{-5}$ mbar and less the films become absorbing.

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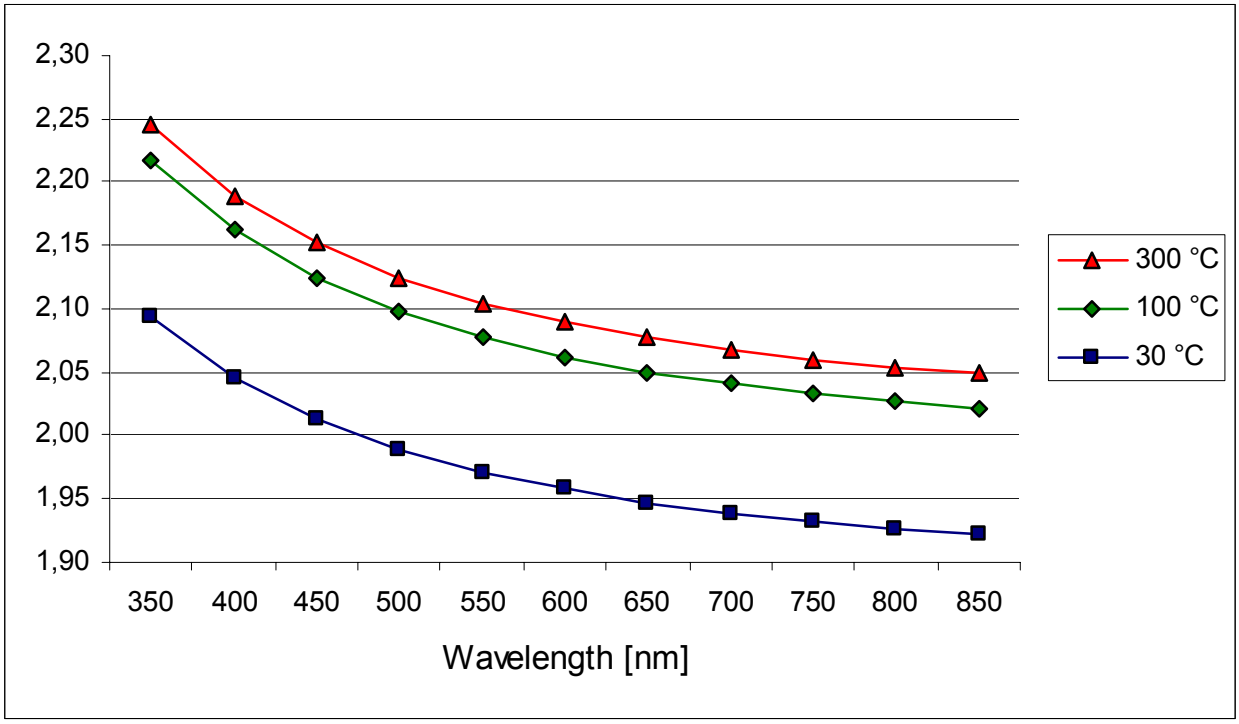
The stability of the optical properties for subsequent deposition was tested by running 4 layers of 270 nm physical thickness each from one melt. Refractive index for these 4 layers was 2,10 ± 0,01 at 500 nm, absorption at wavelengths above 400 nm was not detectable.

The optical properties of Substance H4 films are influenced by the substrate temperature. Deposition on unheated substrates results in layers with refractive index 1.96 at 500 nm, at 300 °C temperature the index is 2.1. In both cases there is no absorption detectable for wavelength above 380 nm.

The dispersion of refractive index for different substrate temperatures was derived from transmission curves of these layers and is shown in the following figure:



The dispersion can be described by following Cauchy formulae:

$n(\lambda) = 1.887 + 25410/\lambda^2$ for unheated substrate (RT)
$n(\lambda) = 1.982 + 28920/\lambda^2$ Substrate temperature 100 °C
$n(\lambda) = 2.009 + 28880/\lambda^2$ Substrate temperature 300 °C

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Layers of 630 nm thickness on silicon, germanium and zinc selenide show some absorption at 2.9 µm due to water incorporation. The long wavelength cut-off was measured at around 10 µm.

Layers of Substance H4 made with ion-assisted deposition without heating of substrates showed no water absorption bands at 2.9 µm.

STABILITY OF COATINGS

Films on fused silica and glass (BK7) deposited at 300 °C substrate temperature proved to be stable under different storage conditions (100 °C in dry air, 25 °C in 80 % humidity). Coatings on polycarbonate passed the Scotch-tape test.

AREAS OF APPLICATION

Substance H4 is especially suitable for production of AR coatings and optical multilayer coatings for NUV, VIS and NIR.

REFERENCES

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US patent application US 5 340 607

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