

Processing Guidelines – OrmoClear®FX

OrmoClear®FX

Characteristics

OrmoClear®FX is an inorganic-organic hybrid polymer for micro optical applications like micro lenses or diffractive optical elements.

The solvent-free and UV-curable ready-to-use formulations can be processed by UV molding or UV lithography. PDMS-based molds can be used for UV molding applications. Detailed physical parameters of the processed hybrid polymers can be found in the attachment.

After processing the OrmoClear®FX hybrid polymer shows

- Excellent transparency in the near UV and visible wavelength range
- High chemical and physical stability,
- High thermal stability up to 270 °C
- High resolution to sub-100 nm pattern dimensions

Processing Options

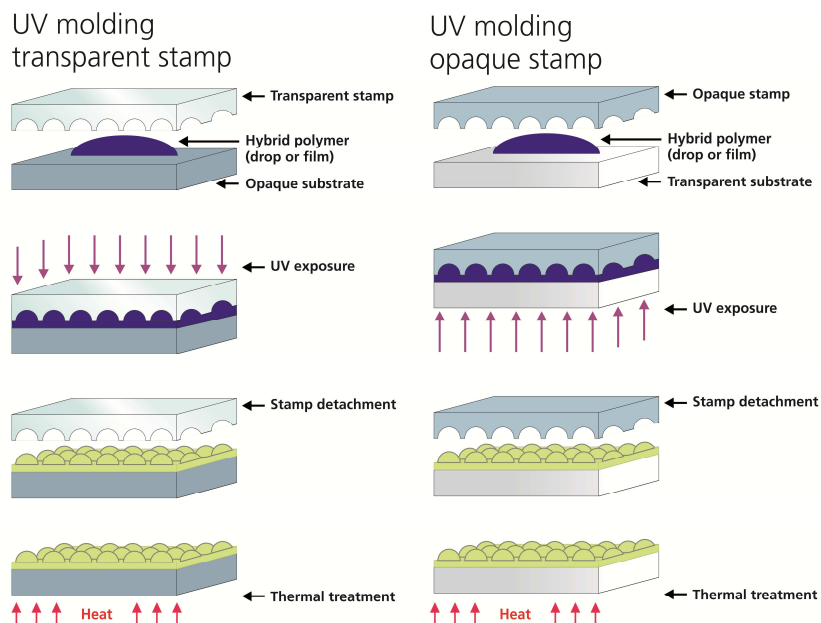


Fig. 1: Process flow for UV molding with a transparent stamp or a transparent substrate.

Standard Processing Conditions

Best results are accomplished at temperatures of 20–25 °C and a relative humidity of 40–46 %. **OrmoClear®FX has to be processed under yellow light.** The guidelines relate to standard processing on silicon, glass, or silicon dioxide. The specific process parameters depend on substrate, application, and equipment.

This information is based on our experience and is, to the best of our knowledge, true and accurate. It should inform you about our products and their application processes. We don't guarantee special features of our products or use for a concrete process.

Processing Guidelines – OrmoClear®FX

Processing Details

Solvent-free types		OrmoClear®FX	
Film thickness (after UV curing)	[µm]	20 ± 5	
Substrate preparation		Spin clean with 2-propanol and dehydrate on a hotplate at 200 °C for 5 min or apply O ₂ plasma treatment	
Spin coating	spin speed	[rpm]	3000
	time	[s]	30
	acceleration	[rpm/s]	1000
Prebake (hotplate, optional)		[°C]	80
		[min]	2
Exposure dose ¹	[mJ/cm ²]	1000 – 3000	
Post exposure bake		[°C]	130
		[min]	10
Development with OrmoDev ²	[s]	30 – 180	
Hardbake		[°C]	150
		[h]	3

¹ UV broadband exposure, intensity measured at $\lambda = 365$ nm

² Optional step, immersion development, duration may vary depending on feature size, necessary only when UV lithography is applied

Substrate Preparation

For glass or metal surfaces it is advisable to use an adhesion promoter such as OrmoPrime®08. For processing information please see the OrmoPrime®08 processing guidelines. The adhesion of the material to Si and SiO₂ may be sufficient in certain cases. In this case the substrates have to be free of impurities and moisture. They should be spin-cleaned with acetone/ 2-propanol, baked at 200 °C for 5 min and cooled to room temperature immediately before coating. Alternatively, short oxygen or ozone plasma cleaning is recommended. Pre-cleaning with a gentle etching agent (e.g. acetic acid) will also improve the adhesion to glass.

Coating

Uniform coatings are obtained by spin coating of OrmoClear®FX. Increasing spin duration decreases the film thickness and increases homogeneity. For UV molding processes, also dispensing can be applied.

Prebake

Spin-coated OrmoClear®FX films are baked on a hotplate at 80 °C for 2 min. Such a prebake step is optional for these solvent-free formulations. It improves film homogeneity and adhesion to the substrate.

Exposure

OrmoClear®FX is sensitive for UV broadband, i-line (365 nm), and h-line exposure (405 nm). Keep the substrates always horizontal until the resin is cured. Applying an UV overdose during curing does not affect the OrmoClear®FX hybrid polymer properties. UV curing leads to volume shrinkage in the range of 3–4 % for OrmoClear®FX (see attachment).

UV exposure in general:

- If high stability is required, we recommend a flood exposure after development when lower doses were used for the preliminary curing step. Higher doses will also improve the adhesion to the substrate.
- UV curing using monochromatic light instead of broadband UV light needs longer exposure time or higher UV doses. Using h-line (405 nm) requires higher UV doses than using i-line (365 nm).

This information is based on our experience and is, to the best of our knowledge, true and accurate. It should inform you about our products and their application processes. We don't guarantee special features of our products or use for a concrete process.

Processing Guidelines – OrmoClear®FX

UV molding applications:

- Please note that the UV exposure dose influences the resolution. If resolution is not critical, we recommend applying UV doses > 1000 mJ/cm².
- Generally, hard molds, e.g. based on quartz or Si, should be coated with a release layer (anti-sticking layer) to guarantee defect-free release as well as low release forces. The most common release agent for silicon or silicon dioxide is “F₁₃-TCS” (1H,1H,2H,2H-perfluorooctyl-trichlorosilane, CAS number [78560-45-9], available from common suppliers of specialty chemicals).
- OrmoClear®FX is compatible with PDMS molds.

Post Exposure Bake

OrmoClear®FX is baked on a hotplate at 130 °C for 10 min after UV exposure. Higher temperature and longer bake time both increase the adhesion to the substrate. If an oven is used, increase baking time by 15 min.

Development

A wet-chemical development step is optional. It is only mandatory, if uncured material has to be removed. Ready-to-use developer OrmoDev is recommended. The temperature of the developer should be 20-25 °C. The developed films are thoroughly rinsed with 2-propanol and then gently blow-dried using nitrogen.

Hardbake

A hardbake is required to increase the thermal and environmental stability: 150 °C for 3 h on a hotplate or in an oven. To improve the transparency of the OrmoClear®FX hybrid polymer it is advisable to do the hardbake in an oven under inert gas atmosphere or in vacuum (< 10 mbar) at 150 °C for 3 h.

Removal

As the OrmoClear®FX hybrid polymer forms a three-dimensional polymer network during curing, drastic conditions for removal are necessary. The solvent PGMEA or NMP-based solvents in an ultrasonic bath at higher temperature (40–60 °C) for several hours will usually result in a peel off. Hot piranha etch is also suitable. Alternatively O₂/CHF₃ plasma can be used to remove the cured hybrid polymer.

Do not use pure oxygen plasma! Porous SiO₂ will be formed.

Storage

We recommend 5–15 °C as standard storage temperature (refrigerator), whereby 18–25 °C is the regular working temperature. Under these conditions a shelf life of 6 months from the date of manufacture is ensured. When OrmoClear®FX has not been annealed to 18–25 °C prior to processing, its viscosity is higher. Therefore, processing results may deviate from the data indicated in the tables above. Storage conditions deviating from the recommendations can lead to pre-aging (e.g. higher viscosity or insufficient curing upon UV exposure after room temperature storage for several weeks). OrmoClear®FX and any unprocessed material have to be stored under yellow light. Keep the bottles closed when not in use.

Disposal

Unexposed material: dispose of as halogen free solvent.

Exposed material: dispose of as solid chemical waste.

This information is based on our experience and is, to the best of our knowledge, true and accurate. It should inform you about our products and their application processes. We don't guarantee special features of our products or use for a concrete process.

Processing Guidelines – OrmoClear®FX

Environmental and Health Protection

OrmoClear®FX is a non-hazardous material. Nevertheless, it should be handled with same care as all chemicals. Ensure that there is adequate ventilation while processing the materials. Avoid contact with skin and eyes and breathing solvent vapours (in case of diluting with thinner). Wear suitable protective clothing, safety goggles and gloves.

Attachment

Specifications

Liquid material (uncured)		
Type		OrmoClear®FX
Viscosity 25 °C (solvent-free)	[Pa·s]	1.5 ± 0.3
Refractive index n_p^{25} (589 nm)		1.535 ± 0.002
Spectral sensitivity (UV curing)	[nm]	300 – 410
Filtration level	[µm]	0.8

Physical Parameters of Processed Hybrid Polymers

Cured hybrid polymer (UV-exposed and baked) ¹		
Type		OrmoClear®FX
Thermal behaviour		Duromeric
Refractive index n_p^{25} (589 nm)		1.555 ± 0.003
Optical dispersion		See Fig.3 below
Abbe number		34
Volume shrinkage ²	during UV curing [%]	3 – 5
	during hardbake [%]	< 1
Modulus of elasticity	[GPa]	n.d.
dn/dT (589 nm)	[10 ⁻⁴ /K]	-2.7.
Hardness (by indentation)	[MPa]	n.d.

¹ Sample processing: (1) UV dose 1000 mJ/cm², (2) post exposure bake: hotplate 10 min @ 130 °C, (3) hardbake: hotplate 3 h @ 150 °C

² Exact degree of shrinkage depends on particular processing conditions and on pattern size

This information is based on our experience and is, to the best of our knowledge, true and accurate. It should inform you about our products and their application processes. We don't guarantee special features of our products or use for a concrete process.

Processing Guidelines – OrmoClear®FX

Film Thickness and Spin Curves

The thickness values of spin-coated films were determined after UV exposure (1 J/cm²) and post exposure bake (10 min @ 130 °C). The data refer to an open spin-coating system.

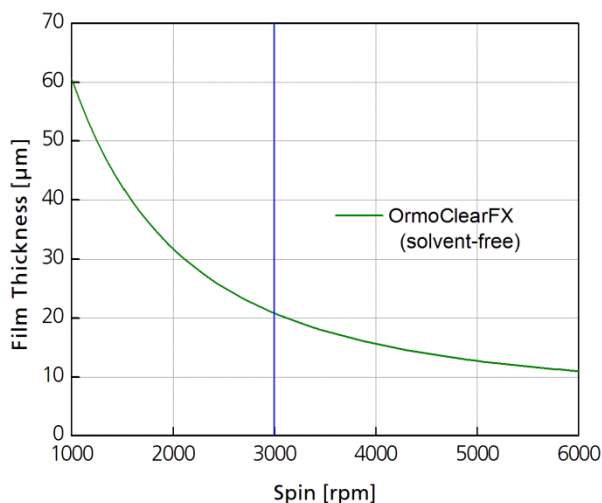


Fig.2: Spin curve of solvent-free OrmoClear®FX, 30 s spin time (film thickness determined after UV exposure and post exposure bake).

Transparency

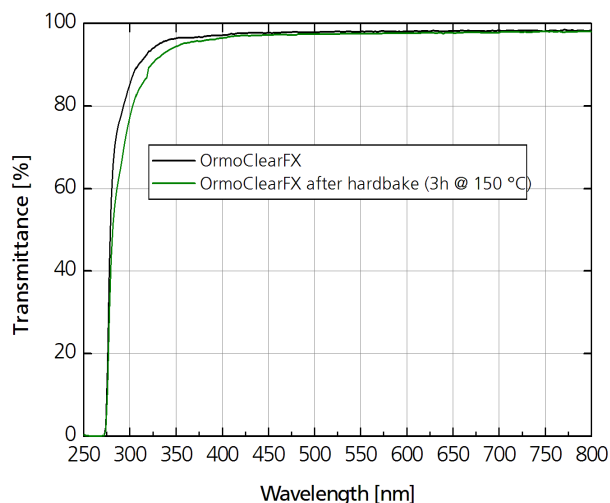


Fig.3: Transmittance of OrmoClear®FX after standard processing with and without subsequent hardbake (3 h @ 150 °C).

This information is based on our experience and is, to the best of our knowledge, true and accurate. It should inform you about our products and their application processes. We don't guarantee special features of our products or use for a concrete process.

Processing Guidelines – OrmoClear®FX

Optical Properties

The refractive index data for OrmoClear®FX were determined after UV exposure (1000 mJ/cm²) and post exposure bake (10 min @ 130 °C).

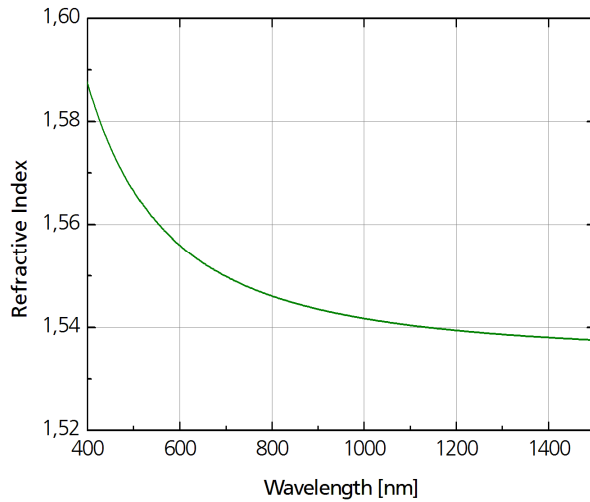


Fig.4: OrmoClear®FX dispersion curve (refractive index vs. wavelength) at 25 °C.

OrmoClear®: DE 30 210 075 434; IR 1 091 359; TW 100030628 (application)
OrmoPrime®: DE 30 210 075 436

This information is based on our experience and is, to the best of our knowledge, true and accurate. It should inform you about our products and their application processes. We don't guarantee special features of our products or use for a concrete process.