mr-XNIL26SF Series: Solvent Free UV-curable Formulation for Nanoimprint Lithography with Excellent Release Properties

Characteristics

mr-XNIL26 is a liquid UV-curable resist system with a high curing rate designed for UV-based nanoimprint lithography (UV-NIL). It consists of highly fluorinated components leading to excellent release properties. The mr-XNIL26 series is provided as ready-to-use solutions for various film thickness ranges and also as a solvent-free type. The cured polymer has a very low surface energy, high thermal and mechanical stability. This particularly enables imprinting and etching of dense nanostructure designs in the sub-100 nm range.

Processing Scheme

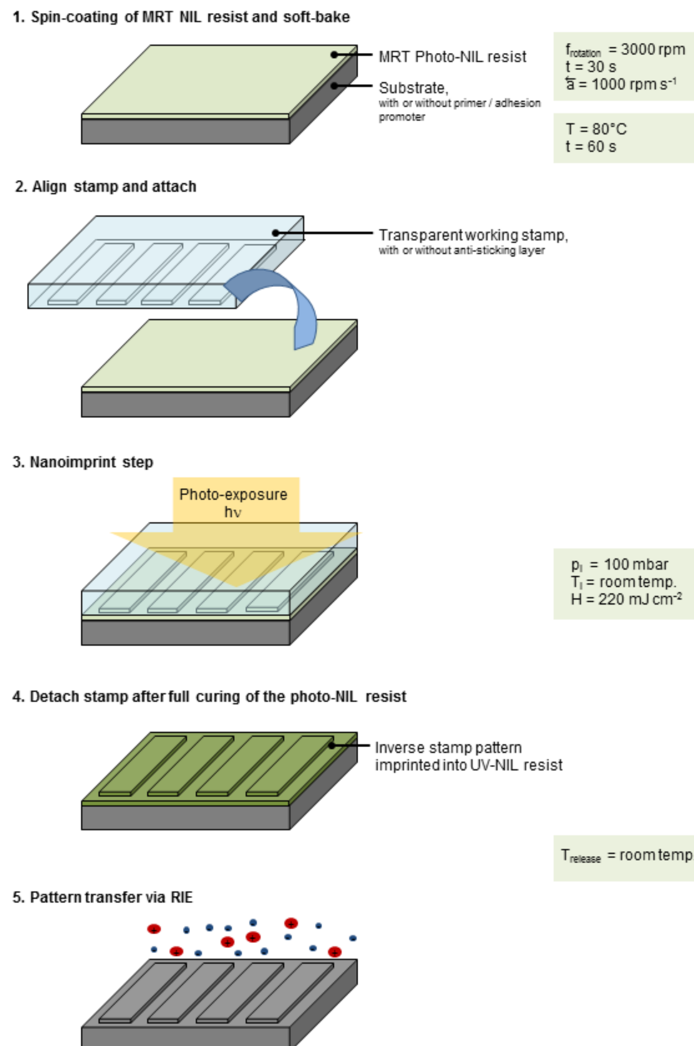

Fig.1: Process scheme of a Photo-NIL process with mr-XNIL26SF.

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.

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Processing Details

Resist formulations	mr-XNIL26SF
Film thickness in µm	**4.8 ± 0.2**
Substrate preparation	Spin clean with 2-propanol and dehydrate on a hotplate at 200 °C for 5 min or apply O_2 plasma treatment Depending on pattern size and substrate material mr-XNIL26SF may require a primer. mr-APS1 is recommended.
Coating spin speed [rpm] time [s] acceleration [rpm/s]	3000 30 1000
Prebake (hotplate) [°C] [min]	80 1
Imprinting parameters	
Imprint Temperature [°C] Imprint Pressure [bar] Exposure dose [mJ/cm^2] Release Temperature [C°]	Room temperature 100 mbar 220 Room temperature

Standard Processing Conditions

Best results are accomplished at temperatures of 20–25 °C and a relative humidity of 40–46 %. The specific process parameters depend on substrate material, equipment, and application.
mr-XNIL26 is light-sensitive and should not be exposed to direct daylight. mr-XNIL26 bottles have to be handled under yellow light.

Dilution of mr-XNIL26SF

mr-XNIL26SF resist formulations can be diluted to achieve lower film thickness upon spin coating as originally specified. Specific film thickness ranges can be obtained by variation of the diluent/solution mass ratio. The processing details for the diluted solutions are the same as for the original formulations (substrate preparation, coating, imprinting).
It is highly recommended to filter the self-diluted resist formulations in order to effectively avoid particle contamination in the spin-coated film. Syringe filters with pore size in the range 0.1–1 µm can be applied. The recommended diluent is ma-T 1050.

Mass ratio data for dilution [w/w]	
Film thickness @ 3000 rpm	**mr-XNIL26SF / ma-T 1050**
100 nm	0.10 / 0.90
200 nm	0.20 / 0.80
300 nm	0.25 / 0.75

Substrate Preparation

The substrates have to be free of impurities and moisture. Silicon substrates should be spin-cleaned with acetone / 2-propanol, baked at 200 °C for 5 min. and cooled down to room temperature immediately before coating. Alternatively, short oxygen or ozone plasma cleaning is recommended.
Depending on the structure size and the applied substrate material, mr-XNIL26SF may need a primer or adhesion promoter. mr-APS1 form micro resist technology GmbH is recommended. For processing of mr-APS1 adhesion promoter please refer to the separate processing guidelines.

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Coating

Uniform coatings are obtained by spin coating of the resist formulations. A spin time of at least 30 s is recommended. Extended spin duration time is considered to decrease the film thickness and may improve also the film thickness homogeneity.

Prebake

Spin-coated mr-XNIL26SF resist films are baked on a hotplate at 80 °C for 1 min. The prebake step removes residual solvent in the spin-coated film.

Stamp Preparation

Hard stamps like Si, Quartz, Ni, etc.: For defect-free imprints and low release forces using silicon, nickel or quartz stamps, it is highly recommended to treat and equip the stamp surface with a release agent. A well-suited release agent for silicon or silicon dioxide is F_{13}-TCS (trichloro-(1*H*,1*H*,2*H*,2*H*-perfluorooctyl)-silane, CAS number [78560-45-9]), that is commercially available from many suppliers of specialty chemicals.
Polymer stamps: mr-XNIL26SF can also be used in combination with hard polymeric working stamps like the commercially available product OrmoStamp®. Please follow the separate processing guidelines for the stamp preparation with OrmoStamp®. Other options as working stamps are PFPE, PUA, or other polymer systems, whereas, the relatively high pressure and temperature during mr-XNIL26SF processing needs to be considered. mr-XNIL26SF is not compatible to oxygen permeable stamp materials like PDMS.

Imprint Conditions

Main factors determining the imprint conditions are the rheological behaviour of the polymer, the mould layout (feature size, density of the patterns etc.), the residual layer thickness to be attained and the imprinting tool. Typically nanoimprint polymers are heated to 50–80K above T_g. The mr-XNIL26SF series can be imprinted in any tool suitable for doing thermal nanoimprint lithography. Commercial nanoimprint tools as provided e.g. by EV Group (AUT), SUSS MicroTec (GER), NIL Technology (DEN), Obducat (SWE), or others may be used.
It is recommended to imprint on the lower end of the process window and to choose rather low pressure and temperature values for the first trials. mr-XNIL26SF performs well because of its optimised flow properties. Very high imprinting pressures are proven to be a source of defects and should be avoided.

Residual polymer layer removal

The residual layer remaining in between the imprinted patterns is removed by oxygen reactive ion etching (RIE) in order to open the window to the substrate.

Removal / Stripping

Plasma-based removal: A full removal of processed resist residues is preferably achieved by applying oxygen plasma. Since mr-XNIL26SF only contains organic components, there are no residuals left over on the substrate after plasma treatment with pure oxygen.
Wet-chemical stripping: As the mr-XNIL26SF will be fully cured after imprinting it is insoluble in standard organic solvents. Hence, comparatively harsh wet chemical stripping conditions need to be applied, e.g. piranha solution.

Storage

Storage at temperatures of 15 – 25 °C is recommended. Storage in a refrigerator is not required. Keep mr-XNIL26SF bottles closed when not in use. Under these conditions, a shelf life of 6 months from the date of manufacture is ensured.

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Disposal

Dispose of as halogen-containing liquid. The fluorinated components in the mr-XNIL26 formulations are non-hazardous materials. They comply with the international 2010/2015 PFOA Stewardship Program and do not contain any PFOA derivatives or precursors.

Environmental and Health Protection

mr-XNIL26SF should be handled with the same care as usual for all chemicals. Ensure that there is adequate ventilation while processing the materials. Avoid contact with skin and eyes and breathing solvent vapours. Wear suitable protective clothing, safety goggles and gloves. The product related material safety data sheet is delivered with the product and should be read carefully.

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Attachment

Specifications of the liquid mr-XNIL26SF Formulation

Parameter		mr-XNIL26SF
Appearance/colour		slightly yellow
Film thickness [1)]	[µm]	4.8 ± 0.2
Density @ 25 °C	[g cm^{-3}]	1.414 ± 0.003
Dynamic viscosity @ 25 °C	[mPas]	147 ± 1.0
Refractive index n_D^{25}	[2)]	1.391 ± 0.002

[1] Spin-coated at 3000 rpm for 30 s [2] refractive index of the resist formulation is measured at 589 nm and 25 °C

Physical Parameters of the mr-XNIL26SF Polymer

mr-XNIL26SF before imprinting		
Glass transition temperature T_g	[°C]	-
Thermal behaviour		Liquid formulation
Processed mr-I 8000R polymer (imprinted)		
Glass transition temperature T_g	[°C]	no
Thermal behaviour		Duromeric

Film Thickness and Spin Curves

The thickness data of spin-coated films refer to an open spin-coating system. For film thickness measurements using ellipsometric methods the following Cauchy coefficients can be used:

n_0 = 1399, n_1 = 43, n_2 = -8. Cauchy formula: $n_{(\lambda)} = 10^{-3}\, n_0 + 10^{2}\, n_1 / \lambda^2 + 10^{7}\, n_2 / \lambda^4$.

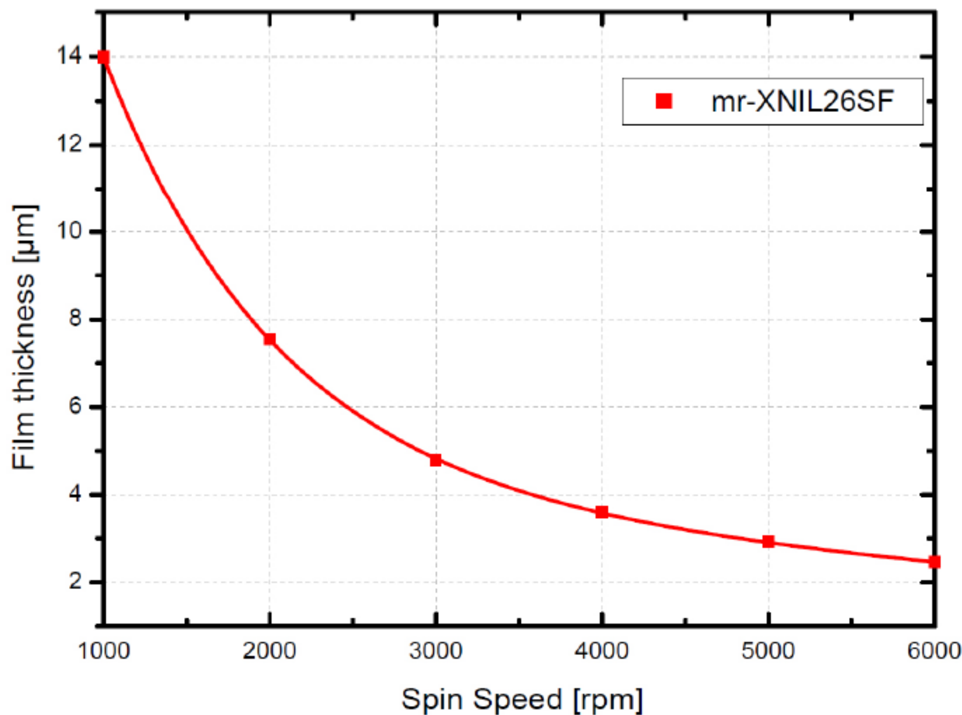

Fig.2: Spin curves of the mr-XNIL26SF formulation, 30 s spin time.

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