



CONSTGLASS



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1-Pilot object

Pilot object: Cologne Cathedral,
N VI, choir clerestory, "Cycle of Kings" (ca. 1300)

Picture



internal face



external face

Identification of the panel:

NVI, choir clerestory

Panel: 4bl
external face

Treatment and other materials:

Product: ORMOCER® (inorganic-organic hybrid polymer)

Application:

- in 1988 (stained glass studio DBV);
- ORMOCER® protective system coats the external face of the panel.

- the coating system was applied in several different layers:

1. base lacquer (50% ORMOCER® + 50% Paraloid® B 72, solved in ethyl acetate 1:3), twice applied;
2. protective lacquer (50% ORMOCER® + 50% Paraloid®, solved in ethyl acetate 1:10), 3 x applied; in each layer inorganic pigments (glass flakes) were inserted;
3. covering layer: Paraloid® B 72, solved in Toluene 1:9.

Cleaning:

Before the ORMOCER®-treatment the glass surface was cleaned with brushes and fibreglass-brushes to remove all corrosion products.



sampling area (external face)

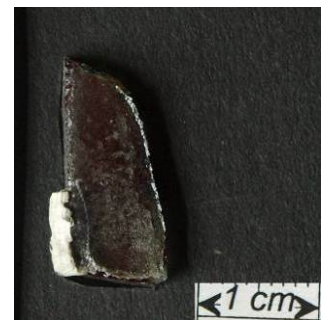


sample Col_ORMOCER




(The above pictures show the complete sample, that was afterwards divided in separated pieces)



Col_ORMOCER_separated_1



Col_ORMOCER_separated_2

	 <p data-bbox="411 181 711 226">CONSTGLASS</p>	
	<p data-bbox="411 293 707 338">Table of results</p>	
<p data-bbox="100 427 209 456">Remarks</p>	<p data-bbox="767 427 1497 611">The external surface of the panel is completely coated with the ORMOCER[®] protective system. The coating is in a good condition. Here and there bubbles are visible and several parts of the coating (thick application) have a milky aspect. The putty around the glass pieces was coated with ORMOCER[®], too. From the conservator's point of view there is no need to remove the coating.</p>	



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2-Results

sample reference: Col_ORMOCER_separated_1,
red flashed glass treated with the ORMOCER[®] protective system

Questions	Techniques	Answers
<p>Morphology</p> <p><i>What is the morphology of the coating? How is the bonding between coating and corroded glass?</i></p>  <p>Col_ORMOCER_separated_1</p>	<p>Optical Microscope (DBV)</p>	<p>Visibly the surface of the ORMOCER[®]-coating is intact, but some areas have a milky aspect (insufficient adhesion between the coating and the glass underneath? Micro-cracks in the ORMOCER[®]-layer? Progress of corrosion?).</p>
<p>Typical phenomenon of the ORMOCER[®] coating (bubbles), exemplified by another segment of the treated panel</p> 	<p>(DBV)</p>	 <p>external face (reflected light)</p>  <p>external face (transmitted and reflected light)</p> <p>Partially the ORMOCER[®] coating shows bubbles.</p>

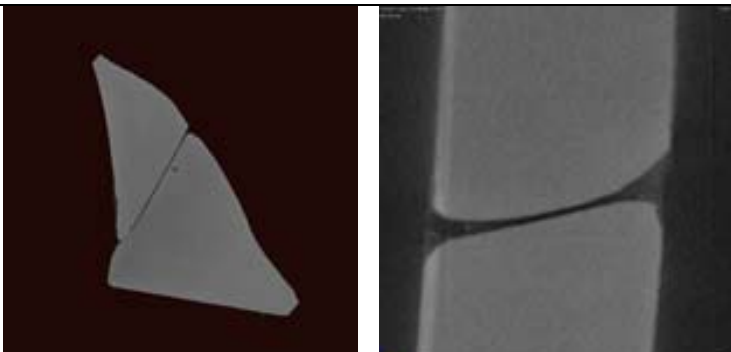
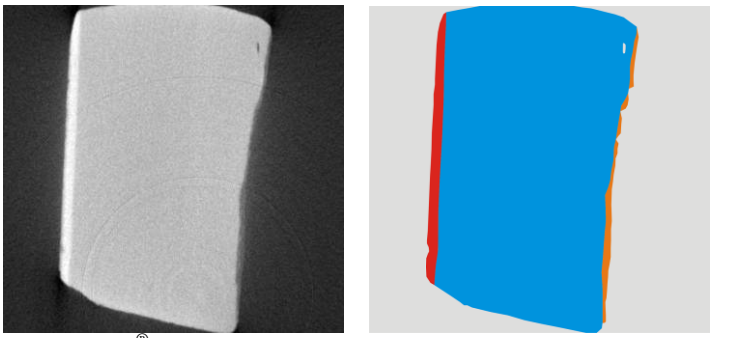


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Chemical Composition	Desktop tomography (UGhent)	 <p>ORMOCER[®] coated sample (treatment 1988): 2D slice through the glass sample. The crack is clearly visible on the CT data, due to the high attenuation of the glass and low attenuation of the crack and the glue.</p>
		 <p>ORMOCER[®] coated sample (treatment 1988): Code for the left picture: on the left: red flashed glass layer, in the middle: bulk glass, on the right: ORMOCER[®] protective layer with glass flakes. Colour code for right picture: blue area is bulk glass, the red area is the red flashed glass and the orange area is the ORMOCER[®] coating.</p>
	Phase-contrast tomography on Synchrotron (Institute)	not foreseen
	SEM/EDX (Institute)	n/a (before re-treatment with Paraloid [®])
Organic component composition	FTIR (Institute)	n/a (before re-treatment with Paraloid [®])
Microbiology	RAMAN (FCS)	n/a (before re-treatment with Paraloid [®])



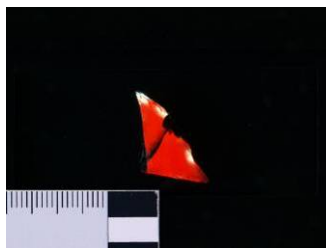
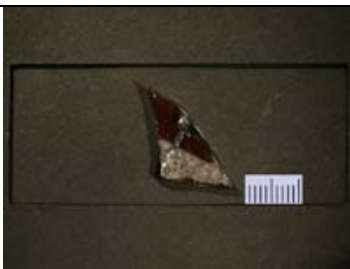


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	Molecular biology ATP measurements (LBW)	n/a
<p>Reversibility <i>How can I remove the coating without damage?</i> <i>Which kind of solvent can I use?</i> <i>Can I remove all flakes?</i></p>  <p>Col_ORMOCER_separated_1 external face before elimination of the ORMOCER®</p>	<p>Test studies elimination (DBV)</p>	<p><u>Note:</u> For the reversibility/re-treatability tests the Constglass-consortium decided, that exclusively the solvent MEK should be used.</p> <p>Treatment: MEK-gel (5% Klucel® G) / compress. Duration: 4 x 10 and 15 min. (altogether 45 minutes) and a subsequent MEK-cleaning with cotton swabs and a dry brush. Result: ORMOCER® was removed by the MEK-compress. Additionally, remains of the embedded glass flakes had to be removed with MEK-soaked cotton swabs; to remove every flake was impossible; there are still remains on the surface.</p> <p>Pictures after the elimination of ORMOCER®</p> <div style="display: flex; justify-content: space-around;"> <div style="text-align: center;">  <p>Col_ORMOCER_separated_1 external face after elimination (reflected light)</p> </div> <div style="text-align: center;">  <p>Col_ORMOCER_separated_1 external face after elimination (transmitted light)</p> </div> </div>
<p>Re-treatability</p>	<p>Test studies re-treatability (DBV)</p>	 <p>sample retreated with Paraloid® B72 (doped with Sartomer® for μCT)</p>



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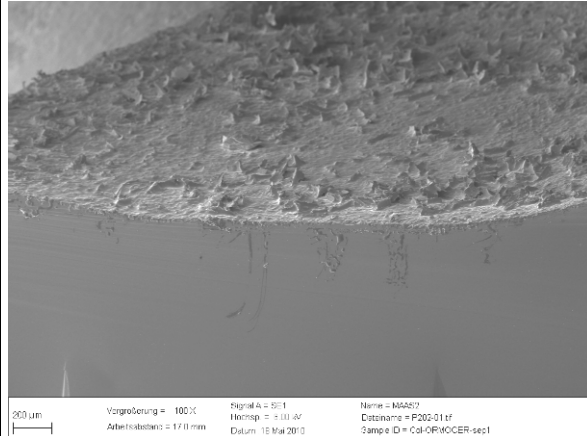
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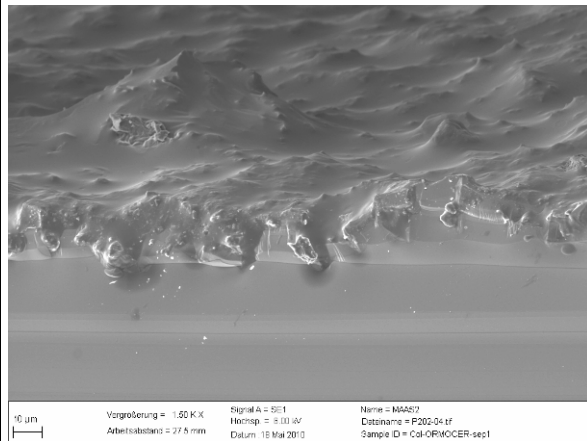
Organic component composition

SEM of re-treated samples (ISC)

For SEM the samples were only broken not mounted and cut, what the normal procedure would be. After breaking the samples were coated with a thin layer of gold to make the surface conductive.



SEM picture of re-treated sample, broken via backside. Perspective view to surface and bulk material (below). On the top of the sample the glass flakes, which were original included in the coating material are visible. Obviously, not all flakes were removed with the cotton swabs.



SEM-picture: The Glass flakes were enclosed by the Paraloid[®], which was applied during the re-treatment.



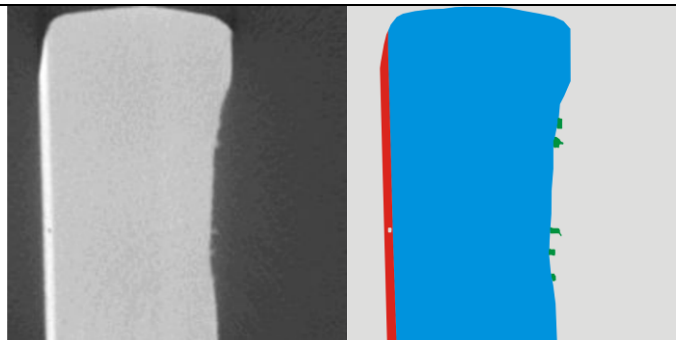
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Test studies re-treatability (UGhent)



After the removal of ORMOCER[®] and afterwards re-treatment with Paraloid[®] B72. For better contrast the Paraloid[®] was doped with Sartomer[®] and Iodine.

Colour code for right picture:

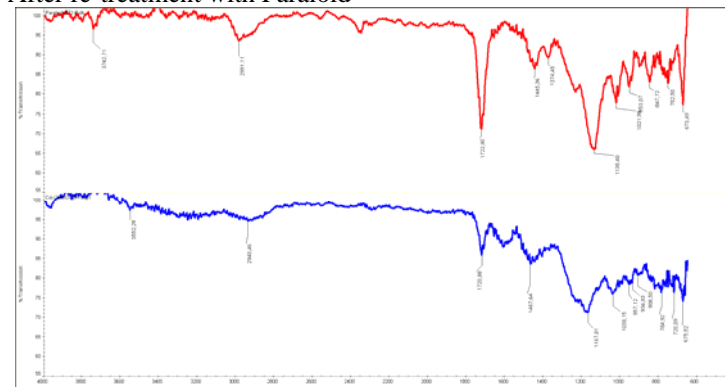
Blue: glass

Red: layer of red flashed glass

Green: traces of Paraloid[®] or rest of glass flakes, formerly embedded in the ORMOCER[®] coating?

FTIR measured with ATR-FTIR (ISC)

After re-treatment with Paraloid[®]



The Col_ORMOCERe_separated_1 spectra (blue) - measured at the top of the re-treated area - is comparable with a Paraloid[®] B72 reference spectrum, upper one in red colour.

RAMAN (ISC)

Analyzes with μ -Raman spectroscopy was not possible on re-treated Col_ORMOCERe_separated_1. The doped Paraloid[®] produced fluorescence, so that analyzes with μ -Raman were not possible.



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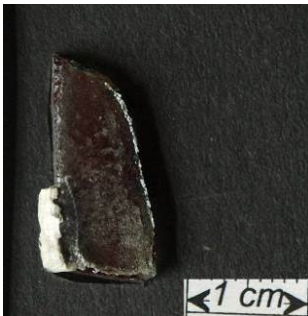
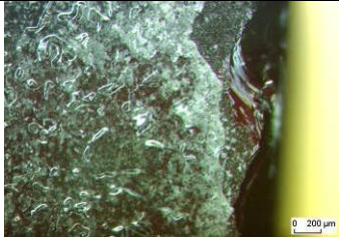
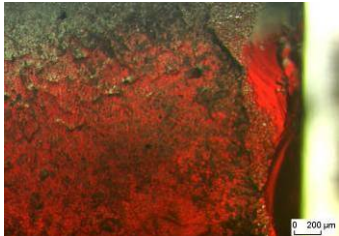
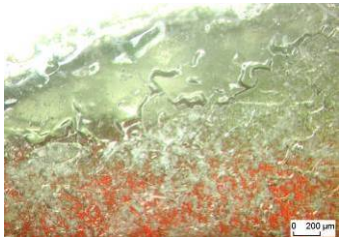


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2-Results

sample reference: Col_ORMOCER_separated_2,
Red flashed glass treated with the ORMOCER[®] protective system

Questions	Techniques	Answers
<p>Morphology <i>What is the morphology of the not weathered coating?</i> <i>How is the bonding between coating and corroded glass?</i></p>  <p>Col_ORMOCER_separated_2</p>	<p>Optical Microscope (ISC)</p>	 <p>Reflecting light: coating looses adhesion and flakes off only at the borders of the sample, whitish glass corrosion crystals underneath the coating do not affect the coating system.</p>  <p>Transmitting light: same detail as above, remnants of glass corrosion products (dark structures) underneath the coating, no micro fissures in the coating.</p>  <p>Reflecting light: Areas with a thick Paraloid[®] top coating (upper border of the sample) appear more transparent than parts of thin Paraloid[®] application (milky appearance).</p> <p>Visibly the surface of the ORMOCER[®]-coating is intact. The adhesion between the coating system and the glass underneath is sufficient. Flaking of at the borders is probably due to mechanical stress during taking the samples. There are no micro-cracks in the ORMOCER[®]-layer.</p> <p>The milky aspect appears in areas of very thin Paraloid[®] top coating and due to the corrosion of glass flakes (which are not well embedded) and / or a rough surface of the ORMOCER[®] layer. Moreover, despite the cleaning process before treatment remnants of glass corrosion products can be detected underneath. They also have an influence on the appearance of the coating system. There are no signs for the development of new corrosion crystals (detachment of the coating, micro fissures etc.).</p>



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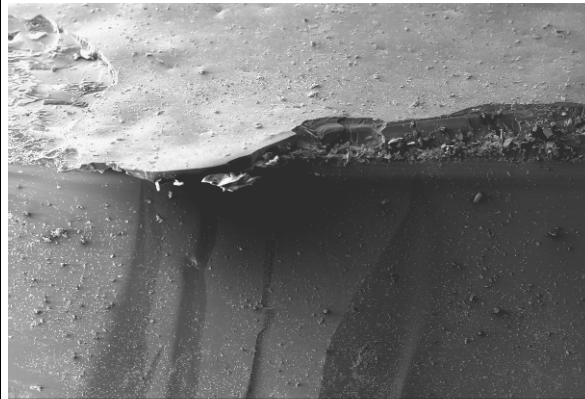


Table of results



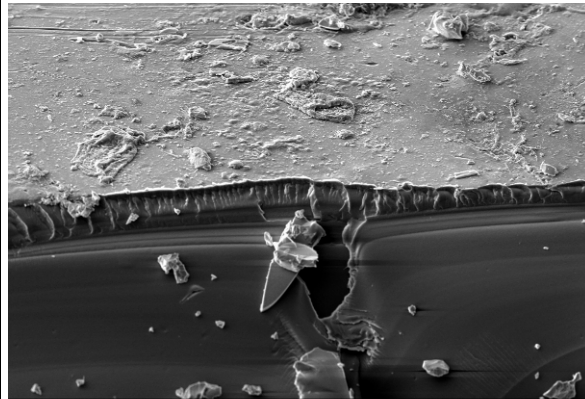
SEM
(ISC)

For SEM the samples were only broken not mounted and cut, what the normal procedure would be. After braking the samples were coated with a thin layer of gold to make the surface conductive.



100 µm Vergrößerung = 100X Signal A = SE1 Name = MAA57
Arbeitsabstand = 7.0 mm Hochsp. = 8.05 kV Dateiname = P201-01.tif
Datum = 17. Mai 2010 Sample ID = Col-ORMOCER-se2

SEM picture: The ORMOCER[®] shows, even after the exposure a good adhesion to glass. No fissures between glass and ORMOCER[®] are detectable. The embedded glass flakes are almost covered by the ORMOCER[®] and Paraloid[®].



10 µm Vergrößerung = 1.50 KX Signal A = SE1 Name = MAA57
Arbeitsabstand = 7.0 mm Hochsp. = 8.05 kV Dateiname = P201-08.tif
Datum = 17. Mai 2010 Sample ID = Col-ORMOCER-se2

SEM picture with higher magnification.

Desktop tomography
(Institute)

performed on separated_1 (result UGhent; see above)

Phase-contrast tomography on Synchrotron
(Institute)

not foreseen

Chemical Composition

SEM/EDX
(Institute)

Braking glass samples gives no even surfaces, which are needed for SEM/EDX



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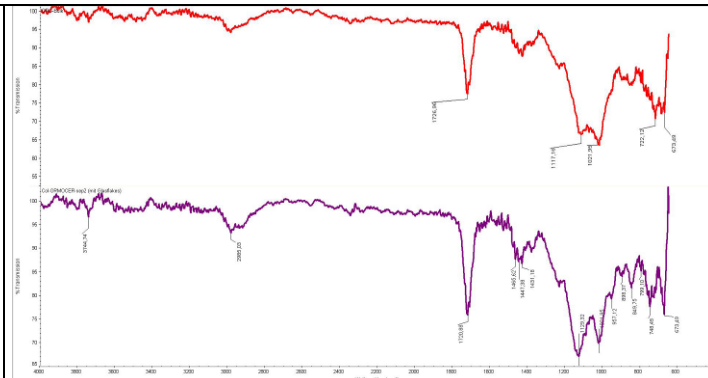


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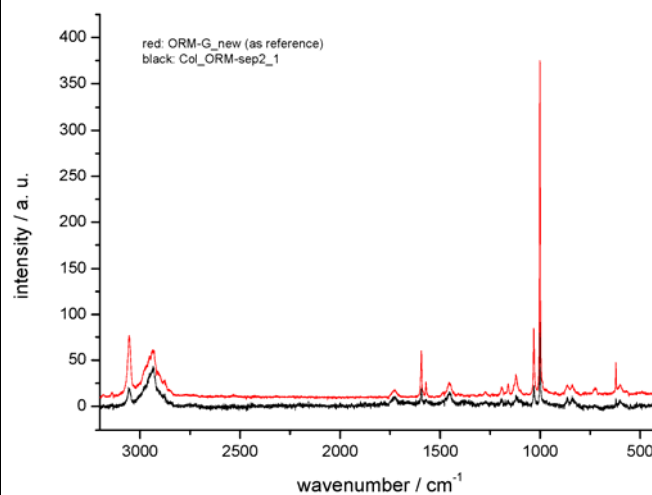
Organic component composition

FTIR (ISC)



ORMOCER[®] bulk material as reference (red line) in comparison to Col_ORMOCERe_sep_2, several layers of ORMOCER[®] (applied 1991)

RAMAN (ISC)



The spectra of the weathered Col_ORMOCERe_separated_2 (black spectra below) layer is comparable with a fresh applied ORMOCER[®] (upper red spectra). No chemical changes of the ORMOCER[®] during over 20 years of exposure are observable.

Microbiology

Molecular biology ATP measurements (Institute))

Not foreseen for this sample; investigation on fresh ORMOCER[®] coating materials: no “unusual” biological contamination detectable (additional test with fresh materials)



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Reversibility

How can I remove the coating without damage?

Which kind of solvent can I use?

Can I remove all flakes?

Test studies elimination (DBV)

A second elimination test was carried out on a segment in the panel, as a comparison to the treatment of the separated segment Col_ORMOCER_separated_1.

Treatment:

MEK gel (5% Klucel® G) / compress.

Duration: 3 x 10 min (altogether 30 minutes) and a subsequent MEK-cleaning with cotton swabs and a dry brush.

Result: ORMOCER® was removed by the MEK-compress.

Additionally, remains of the embedded glass flakes had to be removed with MEK-soaked cotton swabs; to remove every flake was impossible; there are still remains on the surface. Close to the framing lead profiles there are remains of ORMOCER®, too.

Pictures after the elimination of ORMOCER®



NVI 4br, reflected light



NVI 4br, transmitted light

Re-treatability

Test studies re-treatability

Not intended