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#### **1-Pilot object**







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VITRÒCENTRE ROMONTE LUNTER: SCENE DE SUEREMANNE SUR LE VITIMEL ET LES-ARTES AUG VANNE

#### 2-Results

sample reference:	Doubling red flashed glass with Araldite <sup>®</sup> .	
Questions	Techniques	Ancware
Questions	Termques	Allowers
Morphology	Optical Microscope	
Why is the Araldite <sup>®</sup> deterioration so different (vellowing in different		2-
stages / crizzling and stable	The results of the microscopic	
surfaces) on the same piece of plass?	analyses can be	E MESSING MARKEN
8.000	found at the end of the document	
When peeling off, does the Araldite <sup>®</sup> hurt the glass surface?	ANNEXE 1.	
induite martine gauss surjuce.		
How far did the Araldite <sup>®</sup>		5 1
surface?		
<i>Can you detect and alfferentiale several preparations of Araldite</i> <sup>®</sup>		
on the samples from these		I
objects?		SI
On these samples you can see several steps of this process, as		
well as our corresponding,		
provisional classification and		
We propose that the various		
stages of changing of the material		
on this large sample are		
investigated and described		
the analyzing methods available		
in the project: visual		
physical properties, interfaces		
properties.		
		SI
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VITROCENTRE ROMONT L'ANTER SERVICE EL RECHERINGER SUR LE VETENRE LE LES ARTES NO VARIAL

#### Stage 3:



Clear and transparent resin filling, beginning of or advanced yellowing. The plated glass compound has still a dark shining aspect. The whitish "micro-bubbling" is getting denser. Due to the yellowing of the material, the bubbles can have a brown-ochre colour, under reflected light the areas can also look "milky". The adhesion starts to weaken in these parts, but in general it is still very strong.

Stage 4:



Changing aspect from bright, white, shiny aggregations of points to iridescent surface areas. The Araldite<sup>®</sup> surface does not look "deep dark" any more, but rather white on dark (mostly to be seen on edgings or cracks, where mechanical impacts and movements may have occurred).





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VITROCENTRE ROMONT I ERITER SCENE DE REGERENSEE SUR LE VETRE ET LES AREES DU VERBE







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VITROCENTRE ROMONTE KURTER, SCONE, DE REGERERGERE SUR LE VETRIER, LE LES, ARTES SUD VARIANT.

 	1
SEM	n/a
Desktop tomography	n/a
Phase-contrast tomography on Synchrotron	Sample CSRIV_01 The sample has been analysed with phase-contrast micro tomography, with photon energy of 27 keV and a sample-to-detector distance of 66 cm, in order to enhance the contribution of the low-absorption consolidant.





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VITROCENTRE RENTER SEISNE DE REALEMENCEE SUR LE WYDNEL DE LES ARDES DED VARME







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VITROCENTRE ROMONT KERTER SEEDSE DIE SUS-ERES STUD VARIANE SUIR LE WÜRMIE LET LES ARTES STUD VARIANE

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		With the exception of the edges of the fragments, the Araldite <sup>®</sup> sticks rather well to the glass, even if there are some cracks from the surface to the glass. The green parts could be, in principle, either Araldite <sup>®</sup> or glass, but the second hypothesis is more correct. The small metal parts can come both: from the grisaille or, in general, from some pigments, but sometimes come from the lead frames.
		The situation is pretty much the same both for the opaque and the transparent Araldite <sup>®</sup> forms, and the loss of adhesion is usually confined to the edges of the fragment, even on the other tip of the glass segment.
		Particular observations: The plating glass has been detached; its surface seems to have been smoother than the surface of the original.
		The crack in the epoxy layer occurs at the border between parts of different thickness. This confirms an observation on larger samples: The effect could be due to shrinking, but also to the different mechanical stresses due to thermal expansion (glasses, but especially the resin itself).
		In the crack area (top of the image, old crack reopened for sampling), the well adhering epoxy infill has stripped off a part of the adjacent glass – evidence for the risks of de-restoration.
	Optical computer tomography OCT (Piotr Torgorski, Turn Poland)	For the results of OCT, see the report of Pavel Karaskiewicz below ANNEXE 2
Chemical Composition	SEM/EDX	n/a
Organic component composition	FTIR	n/a Surface of the sample is too rough for FTIR-spectroscopy.
	RAMAN	n/a

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Microbiology		Molecul ATP measure

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## **SLASS**



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### results

Microbiology	Molecular biology ATP measurements	No results
Reversibility We found out by taking out the test samples, that even when the Araldite <sup>®</sup> seems to be in a bad condition, it's still sticking to the carrier glass. On this cartography you can see several steps of detachment. - In the green zone, the adhesion is lost. - Zone yellow shows parts where the adhesion starts to weaken, but in general it is still very strong. - The red zone shows Araldite <sup>®</sup> in very good condition. It would be hard or even dangerous to taking out this glass pieces. That's why we decide to keep them like it is and not to reverse the back plating.	Test studies Elimination	
Re-treatability	Test studies Re- treatability	In this case, we don't re-treat the panel.



#### **ANNEXE 1: Optical Microscope**

Preface to the damage characterization of Araldite<sup>®</sup> plating

The historic glass segment of a church window exists of three fragments of red flashed glass. The plating was made at the non-flashed side. The thickness of flat glass was about 1.5 mm; the Araldite<sup>®</sup> plating was around 200 µm thick. The following damage characterization was made with light microscopy with a 50 fold magnification. Both reflected light (RL) and transmitted light (TL) was used; also combined with dark field (DF).

The corrosion description of the plating contains the phenomena between the cover glass and the Araldite<sup>®</sup> as well as the one between the Araldite<sup>®</sup> and the original glass. Also the original glass shows damages, caused by corrosion. These must not be confused with the one from the plating, but could be a cause of defective plating. To clarify this comparison with the original glass before the plating would be necessary.

Corrosion phenomena, which can be found under the yellow aged Araldite<sup>®</sup> plating, looking yellowed discoloured, depending on the light. Without plating these phenomena looks grey-whitish. The original glass shows often this damage.

Microscopic pictures of original glass with plating with two different lights.



Microscopic pictures of original glass **without** plating with two different lights.



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#### Damage characterization of Araldite<sup>®</sup> plating

Samples: Switzerland, Parish Church west – Panel of fragments 3a, 3b, 3c Stage 3

- <u>Cover glass-Araldite<sup>®</sup></u>: Good connection between cover glass and Araldite<sup>®</sup>. Araldite<sup>®</sup> shows yellowing and conditional of manufacturing bubbling (RL/DF and TL/DF), locally small iridescent areas (RL).
- Araldite<sup>®</sup>-original glass: no abnormalities visible

Area: Cover glass/Araldite <sup>®</sup>	Area: Araldite <sup>®</sup> /original glass
DI	No abnormalities
<u>200 µm</u> Same area, RL/DF	



#### Stage 4

- <u>Cover glass-Araldite<sup>®</sup>:</u> Connection between cover glass and Araldite<sup>®</sup> is partially broken. The new very thin gap between both media looks iridescent (RL) or milky (TL/DF), because of light optic phenomena.
- <u>Araldite<sup>®</sup>-original glass:</u> Connection between Araldite<sup>®</sup> und original glasses is in smaller areas broken. The Araldite<sup>®</sup> looks whitish crystalline; the phenomena are only visible in dark field.



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#### Stage 5:

- <u>Cover glass-Araldite<sup>®</sup></u>: Proceeding of gap formation between cover glass and Araldite<sup>®</sup>. Iridescent decrease because of the increasing of gap width. Grey-blue emerging damage phenomena (RL). In dark field only the border line are visible, on other areas it is transparent (DF).
- <u>Araldite<sup>®</sup>/Original glass:</u> Araldite<sup>®</sup> is dissolving in bigger areas, but not laminar, from the original glass. Increasing of the whitish crystalline areas mainly visible in dark field. In reflected light as dark and partly iridescent areas weakly visible.







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VITROCENTRE ROMONT L'AUTRE SCENE DE SUPERENCER SUR LE VETRORE LE LES AREES SUR VAN

#### Stage 6:

- <u>Cover glass/Araldite<sup>®</sup></u>: The adhesion properties of the Araldite<sup>®</sup> have changed significantly: uneven appearance, formation of rough segments which adhere either at the cover or on the original glass (RL+DF). Maybe the appearance has changed because of the contraction respectively the expansion of the plating material. On the inner side of the cover glass partly crystalline structures are visible (DF).
- <u>Araldite<sup>®</sup>/Original glass:</u> Araldite<sup>®</sup> adheres mainly on the original glass. Only isolated smaller golden yellow areas visible. These could be a hint for the beginning of delamination (image presentation is difficult).







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Stage 7:

- <u>Cover glass/Araldite<sup>®</sup></u>: The damage phenomena of stage 6 are increasing and the segmentation gets finer. The connection between Araldite<sup>®</sup> and cover glass is only locally existent. This is good visible on the light-grey and large-scaled areas (RL).
- <u>Araldite<sup>®</sup>/Original glass:</u> The Araldite<sup>®</sup> appears golden yellow, large-scaled and crumbling. This damage is a hint for the increasing brittleness of the Araldite<sup>®</sup>. The connection of the Araldite<sup>®</sup> to the original glass was also decreased.







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Stage 8:

- <u>Cover glass/Araldite<sup>®</sup></u>: Connection between cover glass and Araldite<sup>®</sup> is totally lost. Brittleness of Araldite<sup>®</sup> leads to a fine segmentation and to a bigger crack structures and holes. Small whitish structures can partly be found on the Araldite<sup>®</sup> (DF).
- <u>Araldite<sup>®</sup>/Original glass:</u> Connection between Araldite<sup>®</sup> and original glass is totally lost. The demonstration of this corrosion phenomenon is only hardly possible with microscopic techniques.





#### ANNEXE 2: OCT: Optical Coherence Tomography

Optical Coherence Tomography is based on the recording of changes of optical properties of materials transparent for chosen wavelength (in the case of this research IR radiation 810nm was used). The scans are recorded as series of .jpg pictures (about 150 for one scan) which are shown as an .avi movie from which an interesting .jpg picture can be extracted for interpretation as well as a set of protocols for separated pictures.

The sample analyzed was a piece of flashed red glass from Romont with a painted layer on it stuck with an adhesive (probably epoxide) to the glass support approx. 2 mm thick.

- Both sides of sample were scanned:
- 1. The original glass surface: named  $\mathbf{face}$
- 2. The supporting glass named: **back**

The 10 scans of the Romont sample were executed:

number	scan number	side	additional
			interpretation
1	145339	back	
2	145815	back	
3	150757	face	
4	105650	face	
5	110632	face	
6	111233	back	
7	112113	back	.avi
8	112940	back	.avi
10	113722	back	.avi

Three scans (7, 8, 10) for clarity the scans have been reinterpreted and those scans are most suitable for interpretation.



From the .jpg pictures two were chosen for interpretation. They have to be treated as an example and help for possible further evaluation of the OCT results.

The typical scanning result is depicted on fig.1 where:

1. The final scan

2. The scanned area (yellow line depicts the scanning line)

3. The picture of the sample showing the spot of spot of scanned area

4. The written information is the description of scanning conditions. The most important one is:

**Wymiar** (**X**,**Y**,**Z**) [**mm**] with information on of dimensions of scanned area. In example below: X=11.6 mm; Y=12 mm and Z=1.85 mm. Z is the deep of light penetration.

Bear in mind that the X value is on vertical axis and Y on horizontal one.

fig. 1. Typical OCT scan report of the sample of Romont glass



The raw results depicted above do not show the real scan view and need to be adjusted. The final picture is shown below (fig.2).



fig. 2 Scan of the Romont sample from the back

Fig 2 shows the OCT scan from the back of the sample, i.e. from the backing glass. The depth of penetration of IR radiation is about 2 mm (cf. the scale) and reaches through the glass and adhesive to the back surface of the old glass. The interface backing glass - adhesive gives only information that there is no delamination between them, but on the old glass surface several phenomena are seen: 1. corrosion 2. delamination of the glass surface which might have been caused by adhesive's contraction.

The OCT scanning from the face of the sample is easier to analyze as IR do not penetrated deep into the glass, probably because of red copper containing layer (copper absorbs IR radiation). Typical example of such a scan shows fig.3.



fig. 3. OCT scan from the face of the sample



The scan depicted on fig.3 shows a (1) glass breaks with slightly uneven joint of glass edges and (2) the copper flashed surface.

Above depicted examples show only the possibilities of the method. The OCT for multilayered samples is still on its development stage and interpretation is difficult and time consuming. These two examples however show the way of interpretation and the potential of the method.

Pawel Karaszkiewicz

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