

PIEZO-ELECTRIC QUARTZ CRYSTAL -BASED DOSIMETRY AND DAMAGE ASSESSMENT OF PARCHMENT AND ARTISTS' VARNISH

M.Odlyha¹, S.Jakiela², J.M.Slater¹, L.Bozec³, C. J.Bergsten⁴, T.Gronthoft⁵, E.Dahlin⁵,
M. P. Colombini⁶, I.Bonaduce⁶, D.Thickett⁷, R.Larsen⁸, and M. Scharff⁸

¹ Institute of Structural and Molecular Biology, Dept. of Biological Sciences
Birkbeck, University of London, UK m.odlyha@bbk.ac.uk

ABSTRACT

This paper describes work performed within three EU funded projects and traces the development and applications of a piezo-electric quartz crystal (PQC)-based dosimeter. Indoor monitoring using the prepared dosimeters took place initially within museums and historic castles in the framework of the MIMIC project (Microclimate Indoor Monitoring for Cultural Heritage Preservation, EVKV-CT-2000-00040). Dosimeters were placed in the proximity of painted works of art. In the two following projects the dosimeters were adapted for use in space restricted enclosures which included organ pipes and microclimate frames containing paintings. This was performed in the framework of the SENSORGAN (Sensor system for Detection of Harmful Environments for Pipe Organs, contract no. 022695) and the PROPAINT (Improved protection of paintings during exhibition, storage and transit, FP6 SSPI no.044254) projects respectively. The development of the dosimeter involved miniaturization of electronic components, and development of software to allow continuous monitoring and data acquisition, also using battery operated units. Two types of coatings for the PQC crystals were used: natural resin mastic varnish (MIMIC, PROPAINT) and lead metal (SENSORGAN, PROPAINT). In addition to the dosimetry work, methods for damage assessment of materials, in particular for collagen-based parchment and varnish as performed in the IDAP (Improved Damage Assessment of Parchment EVK4-CT-2001-00061) project and PROPAINT projects respectively will be reported.

Keywords

Dosimetry, piezoelectric quartz crystals, damage assessment, parchment, atomic force microscopy, artists' varnish.

1. Introduction

1.1 Dosimetry

The rationale for using dosimetry technology is to provide a tool for indoor monitoring in museums and showcases where cultural heritage objects are displayed. Methods for monitoring air quality in museums have not yet been standardized and are not extensively used [1]. Several factors such as lack of sensitivity, high cost of some techniques, and no clear correlation between level of pollutants and their impact on the collection have discouraged monitoring on a large scale. For monitoring of microclimates there is the additional complication of limited volume for air sampling. Dosimeters can be prepared which show enhanced sensitivity to the main cause of damage and the synergistic effect of contributing factors. The PQC-dosimeter, as described in the MIMIC [2][3], SENSORGAN [4][5] and PROPAINT[6] projects is responsive to volatile organic acids and photooxidizing effects, depending on the

nature of coating used on the crystals. The change monitored is of the chemical reaction between pollutant and coating and is irreversible. The larger the measured change the greater the degradation of the coated dosimeter, and with prolonged exposure, the degradation of the objects themselves (lead-based) and natural varnishes on paintings.

1.2 Damage Assessment of Parchment

The IDAP project introduced the idea of a multianalytical approach to assess damage in collagen-based parchment. The rationale for using a range of analytical techniques was based on the fact that collagen has a hierarchical structure and the objective was to determine whether damage could be observed at different structural levels. Damage assessment procedures were developed on accelerated aged samples and applied to archival parchment. Atomic force microscopy (AFM), microthermal analysis (μ -TA), and controlled environment Dynamic Mechanical Analysis (DMA) were among the techniques used [7][8]. Parchment samples were subjected to accelerated ageing, using a range of values of RH, T and inorganic pollutants (NO_x , SO_2) [9]. In the current MEMORI project the work has been extended to include samples of parchment and leather subjected to accelerated aging using various levels of volatile organic acids, in particular, ethanoic acid. In the IDAP project accelerated aged and archival samples were studied by AFM to determine the effect of ageing on the periodic D-banding structure of collagen. In addition to measurements of D-banding, a procedure was developed for quantification of the degree of intactness of the collagen D-banding as observed in the AFM images. This gave a damage parameter (peak area) which correlated with measured shrinkage temperatures of accelerated aged and historical samples [10].

1.3 Damage Assessment of Artists' Varnish

In the MIMIC project resin mastic varnish coated steel strips were also exposed alongside the coated PQC crystals in the proximity of artworks. Levels of pollutants (inorganic) were measured at the sites as well as RH, T and light levels. The parameter used to evaluate damage was the change in glass transition temperature (T_g) of the varnish films, as measured by dynamic mechanical analysis, and the broadening of the carbonyl peak, as measured by reflectance FTIR. In PROPAINT the selection of varnishes was extended to include dammar with and without Tinuvin 292, the polycyclohexanone resin MS2A (with and without Tinuvin 292) and B72 (copolymer of ethyl methacrylate methyl acrylate). Dynamic mechanical analysis was performed together with gas chromatography/mass spectrometry (GC/MS). The accelerated aging programme included aging on exposure to various levels of NO_2 O_3 and ethanoic acid at different RH values. Site exposure of the strips was performed at locations where

environmental conditions were monitored. Varnish strips were placed within and outside the paint frames [12].

2. Main Objectives of the projects

The aim of the MIMIC project was to provide dosimeters where the measured signal was related to changes in the chemical composition of artists' varnish, in particular resin mastic. Calibration was performed with respect to known levels of light and also NO₂ at selected values of RH. Exposure of the PQC dosimeters at a number of sites where environmental conditions were recorded for periods up to two years was also made

The objectives of the SENSORGAN project were to make available new instrumentation for monitoring harmful environments for pipe organs through development of sensors for real-time measurement. The system contained three different sensors to detect (a) levels of organic acids harmful to lead-based organ pipes (b) environments damaging to wooden parts of organs (c) possible dew formation inside organ pipes. The use of lead-coated PQC crystal dosimeters for detecting levels of organic acids was suggested since lead coupons were already used in museums as dosimeters for volatile organic acids [13]. The design of the PQC dosimeters had to take into account that they needed to be placed in the organ without disturbing playing or affecting the sound [5].

In the PROPAIN project the objectives were to monitor conditions within microclimate frames for paintings and to compare obtained values for levels of inorganic and organic pollutants with those outside the frames. Three types of dosimeters were used EWO, glass and piezoelectric quartz crystals [6]. In the case of the latter both lead and resin mastic coated crystals were used to monitor effects due to acids and due to photo-oxidizing agents. Monitoring was performed in locations where environmental parameters had been measured [11]. Moreover the design was to be made compatible with the narrow space available between frames and paintings.

3. Methodology

3.1 The PQC dosimeter technology

At the start of the MIMIC project varnish-coated crystals were placed in specially designed holders made of Delrin (Figure 1), and these are also described elsewhere [2].

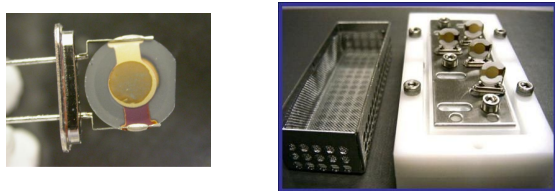


Figure 1 The PQC lead-coated crystal (left) and mounted in specially designed holders (7cm x 5cm) made of Delrin (right)

Measurements of crystal frequency were made before

and after exposure. The difference was expressed in terms of the frequency shift Δf (Hz) relative to the frequency shift caused by the coating (F (kHz)).

The basic operation of the piezoelectric quartz crystal (PQC) can be summarized as follows: when an alternating electric field is applied to a PQC it vibrates at its resonant frequency (in this case 10MHz). If a coating is applied the crystal will become heavier and will vibrate at a lower frequency. The circuit was designed to measure a difference between the exposed coated crystal and an uncoated reference crystal. A reactive lead coating changes mass rapidly following deposition until it forms a passivating layer of lead oxide. On exposure to acids this layer then breaks down and further reactions with mass increase and corresponding lowering of frequency occur. The relation between mass of coating and frequency of oscillation is given by the Sauerbrey equation.

$$\Delta m = \frac{\Delta f \cdot A}{2.3 \cdot 10^6 \cdot F_0^2}$$

where Δf is the change in frequency in Hertz, F_0 is the fundamental frequency of the device in MegaHertz, Δm is the change in mass in micrograms and A is the active area in cm².

The crystals used in the MIMIC project have a resonant frequency (F_0) of 10MHz. In the previous ERA (Environmental Research for Art Conservation, EV5V-CT94_0548) project, poly(ethyleneimine) (PEI) coated crystals were used for measurements of gradients in RH across a selected painting [14]. In the case of PEI the coating behaved in a reversible manner on moisture sorption, provided high values of RH (e.g. >90%) were avoided. Shifts in oscillation frequency occurred with changes in the mass of the coating and they were calibrated to read values of RH.

In the MIMIC project dosimeters were used mainly as passive samplers where the varnish coated PQC crystals were held in metal clamps and then exposed to the microclimate for selected periods. Measurements were made before and after exposure. Towards the final year of the project continuous monitoring was made possible with powered units (14cmx10cm) (Figure 2) on which the array of eight crystals was mounted.

The system utilised proven electronics and data handling from a commercial instrument (QTS-1, Quartz Technology Ltd, Hemel Hempstead (UK), (<http://www.quartztec.com>) that had been modified by the company for this application by the development of new software and dosimeter chambers. The core of the instrument was a 20MHz PIC Microcontroller that collected digital and analogue signals from various sensors and operated firmware that translated the signals into a suitable data stream for transfer via an RS232 link to the host computer. The units (QTS-2 and QTS-3) incorporated a reference crystal (uncoated) and the output signal was then presented as a frequency

difference between the uncoated and coated crystals. This provided basic temperature compensation for the underlying quartz crystal device. The QTS-2 was a larger instrument and was mainly used for laboratory calibration, and QTS-3 was used for site exposures in the MIMIC project. One of the test sites was Charlottenburg Castle, Copenhagen, Denmark. Continuous monitoring for selected time intervals was made for periods of up to 3 months, and data were read using the dedicated SENSOR SCAN software.



Figure 2 Powered unit (QTS-3) used in the MIMIC project for continuous monitoring (left) and adapted crystal holder for monitoring environment within organ pipes (right) as mounted in St Botolph without Aldgate (London).

Resulting damage to the coating was obtained from the Δf values and then expressed as a ratio ($\Delta f/F$), i.e. change in Hz per kHz of coating.

3.2 Adaptation of PQC dosimeters

Modifications were made to the sample holder so that it would support coated crystals in contact with the air passing through the organ pipes (Figure 2, right). The sample holder was designed to house three crystals. This was again made of Delrin and contained the electronics which were miniaturized for this application.

A schematic of the experimental arrangement is shown in Figure 3.

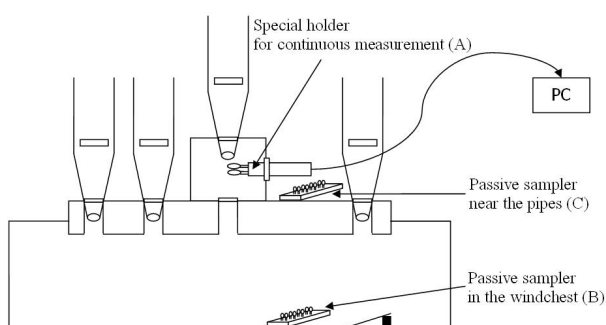


Figure 3 Outline of experimental arrangement for monitoring within organ pipes. This shows how the adapted holder was placed in an optimal way for monitoring within pipes, compared to the passive holders used at the start of the project.



Figure 4 The sample holder as set up in the organ in the cathedral in Olkusz (Poland) (left) and the full setup including the PC (right)

The resulting signals from the crystals were then passed through a Handyscope and the output was read in purpose-written software (SENSOR SCAN). The system still required mains power. This was possible in the organ pipes that were monitored in the churches in St Botolph without Aldgate (London), Olkusz (Poland) (Figure 4) and Oergryte New Church (Sweden).

Further modifications both to the sample holder and to how the system was powered had to be made in the PROPAIN project to ensure that the unit could be inserted within frames containing paintings.

The sample holder, which again accommodated three crystals, was changed to a flat rectangular shape to fit inside the frames. (Figure 5). The holder was then connected by leads to the control box which housed the data collection hardware and replaced the bulky Handyscope. Also it became possible to power the system using batteries.



Figure 5 Sample holder adapted for monitoring within paint frames is shown on the left (control box is placed outside the frame and is on top of the frame). Detail of the holder and control box, which also contains the batteries, is shown on the right. Paint frame was located in the Statens Museum (Copenhagen) and exposure made during the PROPAIN project

4. Results

4.1 Varnish coated PQC dosimeters

In the MIMIC project a total of seven non-conditioned sites were investigated which included northern and southern European locations in urban and semi-rural environments. One air-conditioned site (British Library) in central London was included for comparison. The results from exposure of about three months at the sites were summarized by plotting the dosimeter output against days of exposure [3]. The lowest values were

obtained from the conditioned site and also at an urban site where the monitoring was performed during the winter months (Charlottenburg Castle, Copenhagen) and where light levels were relatively low. Amongst the highest values of change in the varnish-coated dosimeters was that of the dosimeter exposed in the Petrie Museum of Egyptian Archaeology (London) during the monitoring period autumn to winter. A distinct difference was observed, however, for a dosimeter exposed in a showcase in the Petrie museum. The level of change was similar to that observed for the conditioned site. Analysis of accelerated aged samples in MIMIC and previous ERA projects [16] demonstrated that the chemical effect of inorganic oxidizing pollutants (e.g. NO_x) enhanced the damage produced from light aging alone. So this should be taken into account when reading the values.

Measured changes could be correlated with magnitude of doses received of oxidizing agents and light, the higher the dose levels the higher the response. Temperature also affected the response as seasonal differences were observed. In the PROPAIN project behaviour of the varnish coated crystals was correlated with that of the EWO dosimeter, and a dose response function was calculated [6]. In both the MIMIC and PROPAIN projects, exposure was made at sites where levels of inorganic pollutants were measured, in addition to RH, T and light. Where the MIMIC project showed variation for example due to location, such as uncontrolled environments versus climate-controlled locations, the PROPAIN project showed variation not only due to location but also to the type of frame used and the climate within the frame which was affected by properties such as its air exchange value and the materials used to make the frame. Where the frames had a lower protective action, and higher values of inorganic pollutants were registered, then changes in the values of the varnish-coated PQC crystals were also higher for similar light levels.

4.2 Lead coated PQC dosimeters

In the SENSORGAN project, lead-coated crystals showed differences in response between the three sites tested St Botolph, Olkusz and Oergryte. The latter site showed the highest level of change in the lead-coated crystals. The organ in Oergryte New Church was completed in 2000 in the style of 17th century north German Baroque. It was constructed in accordance with historical traditions and materials, and consequently the wooden parts were made of oak. Measured levels of emitted organic acids were higher than at the other two sites. In the case of St Botolph the air within pipes in some locations in the proximity of recent restoration work which included introduction of fresh pine wood was found to give a higher response than in the locations where pipes were standing over older wooden parts. The lowest measured response was in the historic organ at Olkusz, Poland (Figure 6) where there was no restoration work involving fresh wood. Seasonal variations in the response of the lead-coated crystals

were also observed. In all cases measured change was higher in the summer months when the RH and T values were higher. Higher values have been shown to affect the extent of emissions of volatile organic acids [15].



Figure 6 The historic organ dating from 1611 in the Minor Basilica of St. Andrew the Apostle, Olkusz, Poland.

An example of the experimental output is shown in Figure 7 below. The response for the lead coated PQC crystal is shown to increase over a period of over two months. Comparison of the values of the overall change over selected time periods and of the rate of change was made between sites. The rate of change increased significantly at sites where RH values exceeded 60% RH.

In Figure 7 the values for RH lie within 35-40% RH. The period of monitoring was from the start of Feb. 2007 to end of April, when there was still heating within the church.

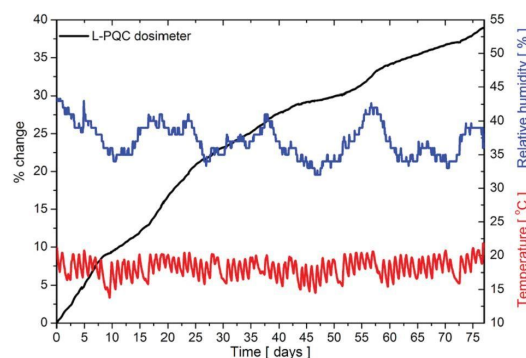


Figure 7 Lead PQC crystal response (black) expressed as change (%) vs. time (days) for exposure in St. Botolph within a Hautboy pipe standing on the toeboard (pine) in the upper swell box made of pine (recently replaced during restoration).

In the PROPAIN project values for the lead-PQC dosimeters were found to be higher, for all locations tested, inside microclimate (mc) frames than in the rooms. This was consistent with the higher measured levels of organic acids in frames than in the rooms, in particular ethanoic acid [6]. Values were found to exceed 1000 µg/m³ in some frames, and this was significantly higher than in the rooms.

The PROPAIN project also allowed the possibility of

testing the lead-coated PQC dosimeter together with the glass dosimeter. Correlation was found between the two dosimeters, and a dose-response function for the dosimeters was calculated [6]. The lead PQC dosimeter was also used to test the effect of introducing sorption agents into micro-climate frames. Reduction in emissions was observed with concomitant decrease in the change recorded by the dosimeter. Testing within frames also included exposure in front of and behind the painting. With the adapted dosimeter it was possible to place a dosimeter in front of and behind the painting. It was found that the response behind the selected panel painting in a microclimate frame was higher than in front [12].

4.3 Damage assessment of parchment

In the IDAP project the effect of accelerated aging using RH,T showed that there was a reduction in the extent of D-banding of the parchment. This was more severe in samples exposed to pollutant aging, such as exposure to SO₂ for periods of 2 to 16 weeks. The reduction in extent of intact D-banding of the collagen as measured at the nanoscale level by AFM also produced a change in the mechanical response of parchment when exposed to increase in RH [7]. It was also found that damage in archival samples could be ranked, and that damage was more severe in bookbindings than in the manuscripts [9].

Currently it is not known how exposure of organic-based materials, such as parchment, to levels of VOCs above 1000µg./m³ for prolonged periods affects such objects. The preservation target set for ethanoic acid for museum, gallery, library and archival collections is 1000µg/m³ and RH between 50-60% for one year [1]. This is the length of time during which objects can be exposed to this level of pollutant with minimal risk of deterioration. However, this value is taken from what is known about the effect of VOCs on metals, and still needs to be determined for organic-based objects. Part of the work in the current FP7 MEMORI “Measurement, Effect Assessment and Mitigation of Pollutant Impact on Movable Cultural Assets – Innovative Research for Market Transfer” project is to determine whether such levels do produce physicochemical changes in parchment, leather, and varnishes.

Recent preliminary observations have been made on modern parchment (goat). Samples were exposed to ethanoic acid vapour (c.1500µg/m³ and 74%RH) for periods of 15 and 33 days. Non-invasive testing using atomic force microscopy (AFM) was performed. Figure 8 shows the images (5µm x 5µm) of these samples. Exposure of up to 33 days caused loss of the periodic D-banding of the collagen structure and wrinkling.

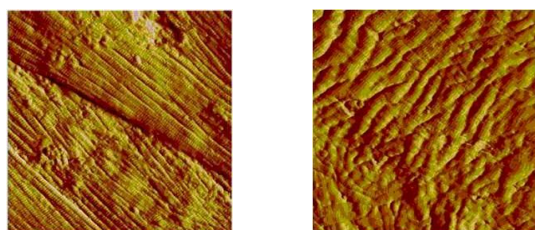


Figure 8 AFM images (5µmx5µm) of modern parchment (goat) exposed 15days (left) and 33 days (right) to 0.6ppm (c1500µg/m³) HAc at 74%RH

4.4 Damage assessment of varnish coated strips

In the MIMIC project the *T_g* of varnish-coated strips showed larger shifts for samples exposed in locations where photooxidizing conditions exceeded recommended levels [3]. In PROPAIN gas chromatography/ mass spectrometry (GC/MS) measurements were also performed on the natural and synthetic varnish strips placed within and outside the frames [12]. For example the resin mastic placed in the frame in the Statens Museum (DK) showed that the *T_g* of the varnished strip in the frame was greater than the *T_g* of the varnished strip outside the frame, in the room. Levels of ethanoic acid in the frame were higher than in the room. In this case the frame offered no protection. Increase in *T_g* and indications of increased crosslinking were confirmed by GC/MS data [12]. In the case of Tate Britain the *T_g* of the varnish strip in the room was higher than in the frame, due to higher NO₂ levels in the room. So in this case the frame had a protective function and the painting was not subjected to the higher dose of NO₂. Both DMA and GC/MS showed that natural varnishes after their application and initial curing undergo rapid oxidation and cross linking. The rate of the oxidation and cross linking, which produced a more brittle and less soluble varnish, was shown to depend on the pollutant doses (NO₂, O₃, acetic acid) both in laboratory exposures and in site exposures [12]. In the current MEMORI project further studies are in progress, focussing on the effect of volatile organic acids, in particular ethanoic acid on artists' varnishes.

5. Conclusion

Piezoelectric quartz crystal dosimeters have been developed and applied in several projects They have demonstrated their potential for discriminating between environments of differing levels of corrosivity caused by the presence of either photooxidizing agents or volatile organic acids.

Damage assessment in collagen-based materials has been shown to be possible using atomic force microscopy. Damage assessment of varnishes has been demonstrated using the combination of DMA and GC/MS techniques.

6. Affiliation of authors

S.Jakiela, Institute of Physical Chemistry PAS 44/52 Kasprzaka, 01-224 Warsaw, Poland

J.M.Slater, Institute of Structural and Molecular Biology, Dept. of Biological Sciences Birkbeck, University of London

L.Bozec, Biomaterials and Tissue Engineering UCL Eastman Dental Institute, University College 256 Gray's Inn Road, London, WC1X 8LD UK

C. J. Bergsten Göteborg Organ Art Centre University of Gothenburg, Sweden

T. Grønhoft and E. Dahlin Norwegian Institute for Air Research (NILU) P.O. Box 100, NO-2027 Kjeller, Norway

M.P. Colombini and I. Bonaduce, Department of Chemistry and Industrial Chemistry, University of Pisa via Risorgimento 35, I-56126 Pisa, Italy

D. Thickett. English Heritage Rangers House Chesterfield Walk, London, SE10 8QX

R. Larsen and M. Scharff School of Conservation Esplanaden 34DK-1263 Copenhagen K

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