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Procedia Chemistry 8 (2013) 307 – 316

Procedia Chemistry

Youth in Conservation of Cultural Heritage, YOCOCU 2012

Research on corrosion of lead printing letters from the Museum Plantin-Moretus, Antwerp

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Abstract

A number of lead alloyed printing letters in the collection of the Museum Plantin-Moretus show corrosion problems. A research project was set up to tackle the reasons for the corrosion issues. This first phase of the project was designed to evaluate a number of facts which can be related to the metal problems and to evaluate how and which analytical techniques are the most appropriate to perform sequel research. Microscopy, XRF, SEM-EDX, XRD and electrolytic techniques were used in combination with an adapted Oddy test. Corrosion with an exceptional high volume increase is reported on lead-antimony alloys.

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Keywords: Lead; Antimony; Corrosion; Conservation; Printing; Types; Collection; Acetic acid; Formic acid

1. Introduction

The Museum Plantin-Moretus in Antwerp[1] is known for its broad history of book printing. The museum holds the original workshop, presses, library and the family house of Plantin and Moretus, which covers a printing history from the 16th century until the 19th century. From 1877 onwards, the complex was turned into a museum. In 2005, the museum was added to the UNESCO's World Heritage List.

The museum includes also a huge amount of lead alloyed printing letters, which are stored in wooden boxes or were once assembled to printing blocks, then wrapped in paper for storage. Some of these printing blocks are exhibited in showcases in the museum. These standing types show in some cases an exceptional corrosion form, including a fierce expansion and decomposing of the metal alloy. The 336 wooden letter boxes on the other hand were sealed with hardboard lids in the year 2000, to protect the content against vandalism. However, during

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doi:10.1016/j.proche.2013.03.038

visual inspection on last year, a severe corrosion phenomenon (Fig. 3) was observed on some of the letters. This paper presents the first stage in search for the reasons of these corrosion phenomena and why it occurs only on some letters whilst others seem unaffected.

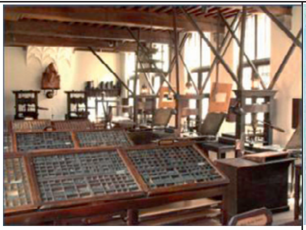

Fig. 1 Interior, print room

Fig. 2 Example of one of the letterboxes; red painted bottom plate

Fig. 3 Two corroded letters, length 28 mm, width 3 mm, depth 1,2 mm

On request of the museum, a study was performed on this complex corrosion problem. A research set up was designed to detect the precise cause of the corrosion processes and to suggest a possible treatment to prevent further damage on the letters. For this research, which was limited in time and is considered to be the first overviewing step towards an in-depth research project, several types of collecting data were chosen. This way, facts which can be connected to the problem enable further research choices to be made more accurately.

2. Methods

2.1. Selection of letterboxes

A partial selection of wooden boxes, holding the lead letters on which corrosion formation had been detected, was made. From the 336 wooden boxes, containing thousands of lead alloyed printing letters, 19 boxes were chosen for examination. The selection was made on the historical period of the printing type, the storage position of the boxes in the museum and on the wood types, of which the boxes consist. Where available, letterboxes with capital letters and small letters (Fig. 2) of the same type were selected together since it was suspected from the museum staff, that smaller letters were more prone to corrosion than larger ones. The letterboxes were selected from two rooms, the print room (Fig. 1) and the adjacent storage room. The printing types vary between 16th and 19th century letters, although at least a number of them were at some point in the history recasted in the original molds, which too are a part of the museum collection. The boxes were mainly made from oak, but are often combined with other wood types such as beech, pine or multiplex for the bottom plates, or hardboard lids. For some boxes, paper was glued onto the bottom plate, or it was painted red (Fig.2).

2.2. Study of the letterboxes materials

All available historical, object material and environmental data was collected and put in Excel tables to detect possible correlations between different parameters. Besides the facts from the box materials, the overall appearance and corrosion which was on the individual letters visually observed, was noted. Dimensions of the letters were measured and put in the table as five size categories. The letters holding important remnants of black or red printing inks, were marked as well. The facts from the boxes, the letters and the macroscopic and microscopic investigation were put together in an elaborate and complex table. Together with these observations also 95 letters were selected on corrosion appearance (see further chapters 2.3, 2.4, 3.2 and 3.3).

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1 www.museumplantinmoretus.be

Since the wood types of which the boxes consist are considered significant for corrosion initiation[1], the wood types were investigated on their organic vapor release towards the letters. For this purpose, an adapted Oddy test[2] was performed in a dual way. First a set of lead (Pb) and two selected alloys were placed together in each of the recipients to evaluate the influences of the different wood types. For the alloys, an antimony (Sb)-enriched and a tin (Sn)-enriched alloy was chosen, considering recipes for the casting of letters[2] and previous XRF-measurements which were performed on a number of letters (methodology comparable to the descriptions in 3.3.1). Alloys were subsequently composed of Pb 70, Sb 25, Sn 5w% and Pb 70, Sb 5, Sn 25w%. The metals were melted in an electric furnace fitted with a graphite crucible and casted in steel ingots with a width of 2,5 mm, producing casted sheets of approximately 20 x 50 mm. The casted pieces were cut to smaller size by sawing, thus leaving the cast structure of the metal which enhances the comparison to casted printing letters.

Oak, pine, beech, multiplex and hardboard from the used lids were the identified wood types on the boxes. These were cut in 1cm^3 blocks with a total weight of 15g for each wood type. Subsequently, each wood type was placed on the bottom of a 500ml airtight container, together with 5ml distilled water in a separate inner recipient and together with the Pb and the two alloys. The metal samples were each dimensioned 10 x 10 x 2,5 mm, giving 300mm^2 surface (Fig.4). Each sample was positioned on the wood, giving maximum influence of wood vapours to the bottom side of each sample, whilst the upper surface was free for the container atmosphere reactions.

Fig. 4: Container with wood

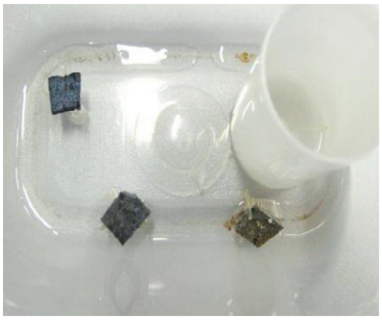

Fig. 5: Container with acid solution

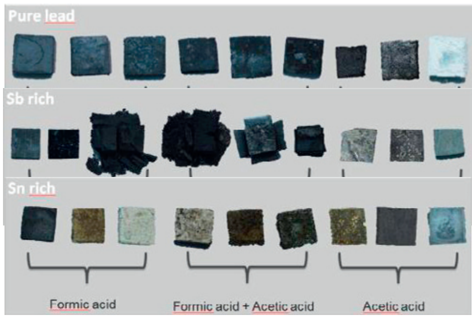

Fig. 6: Results of acids on Pb and two alloys

Secondly, in a number of recipients the wood was replaced by certain volume percentages of acetic and/or formic acid solutions. Acetic and formic acid were both calculated to their maximum evaporation properties in respect to the 1:100 ratio of the inner recipient (5ml) to the 500ml container (Fig.5). Highest concentrated solutions were for formic acid to water 4:1 ml and for acetic acid to water 1,5:3,5 ml. Both acids were diluted respectively 10 and 100 times to study the differences in reaction towards the metal and alloys. Another set of containers held mixtures of the formic and acetic acids in different ratios: 75:25, 50:50, 25:75 v% of the most concentrated solutions. The series in Fig.6 show the results of corroded samples from the Oddy test, on top pure lead samples, middle antimony-rich alloy, bottom tin-rich alloy. Each reads for the acidic environment from left to right: Formic acid solution 1%, 10%, 100%; Mixtures formic acid/acetic acid 75-25%, 50-50%, 25-75%; Acetic acid solution 100%, 10%, 1%.

All containers were kept in a temperature controlled furnace (Heraeus Thermo T12) at 60°C for 28 days. Product specifications: Containers Curver 500ml Polypropylene, distilled water VWR Prolabo, Acetic acid RPL Technical and Formic Acid 98+% Acros. Control samples were placed in a separate container, only holding water to maintain a maximum relative humidity (RH).

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[2] A standard Oddy test consist of air tight containers, which hold a metal specimen and a substance to be checked for its corrosive vapours. In the recipient, a maximum RH is maintained whilst the set is placed in an oven at 60°C for 28 days.

2.3. Macroscopic and microscopic observations on the letters

Extensive macroscopic and microscopic observations were performed on selected letters from the random obtained collection. From the 19 selected boxes, 95 letters with macroscopically different corrosion aspects on their surface were chosen. A Leica M50 stereoscope was used up to 60x enlargement and an Olympus Bx41 was used in bright- and darkfield modus for 100-500x magnifications. Samples and cross-sections of letters were embedded with Technovit 2000 LC, grinding and polishing was executed with a Buehler Alpha-grinder with a Vector specimen holder polishing head. Because the very low hardness of lead often causes smearing of the surface, etching of the samples was performed simultaneously on the last polishing step by using a mixture of glycerol, acetic acid and nitric acid (84/8/8 ml ratio) on a Chemometh I cloth.[3]

2.4. Analytical instruments

XRF, SEM-EDX and XRD were used for elemental and corrosion analysis on the letters. The instrument used for XRF measurements was an Innov-X Alpha-4000, fitted with a tantalum tube, Analytical mode, 0-30 keV. Spectra were fitted with Axil. SEM-EDX spectra were obtained from a Jeol JSM 6300, which was used to study a limited selection of alloys, following the XRF results and to study corrosion phenomena, derived from the microscopic images. The samples, embedded for the optical microscopy were used here again. XRD was performed with an instrument type Juber G670 Guinier camera in the range 4-100°, 2θ. Spectra were matched with data from the International Centre for Diffraction Data (ICDD).

2.5. Environmental study

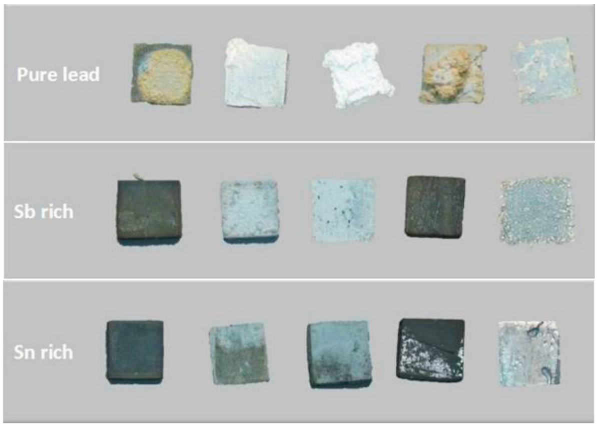
A part of the project was to survey the ambient environment in the museum itself. For this, sets of Pb and the two selected alloys (see 2.2) were placed in the museum as reacting coupons on which subsequently electrolytic measurements were performed. Measurements were carried out using a Palmsens potentiostat; voltammetry mode; electrolyte 0,1 M Na_2SO_4; values V/MSE reference electrode; scan rate 0,01 V/s; measuring area 0,4 cm^2. Also, a cooperation with the University of Antwerp who performed measurements towards particulate matter was beneficial to compare results.

3. Results

3.1 Letterboxes

The different kinds of wood of the letterboxes play undeniably an important role in the corrosion of the contained letters[4], as can be seen also in the results of the Oddy test with the wood samples. Furthermore, the observed corrosion development is importantly different between the lead coupons and the Sb-enriched and Sn-enriched alloys (Fig.7). Since there are many letters in each letterbox and only on some severe corrosion was discovered, it is to be suspected that alloying elements play an important role. This is also observed in the Oddy test where the Sb- and Sn-enriched alloys show in comparison to the pure lead samples a totally different corrosion development under identical atmospheric influences in the containers with the acid solutions (Fig. 6) but also from the wood types (Fig.7-9).

The results of extreme corrosion expansion (Fig. 11) on the Sb-rich Pb-alloy, which developed a 1000% volume increase, were repeated in an equal adapted Oddy test setup, only at room temperature. In this test, the same extreme corrosion was developed, although it took about three times longer to reach a similar grade of corrosion (example in Fig.10) compared to the samples at 60°C from the standard Oddy test. Lower concentrations of formic acid in water or mixtures with less formic acid than acetic acid, all gave results which were in direct relation to the formic acid presence (Fig.6).



Pine Oak Beech Hardboard Multiplex

Fig. 7: Wood types from left to right in each series above

Fig. 8: Pb sample on beech wood

Fig. 9: Pb sample on hardboard

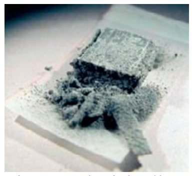


Fig. 10: Sb-rich alloy After 21 days room t°

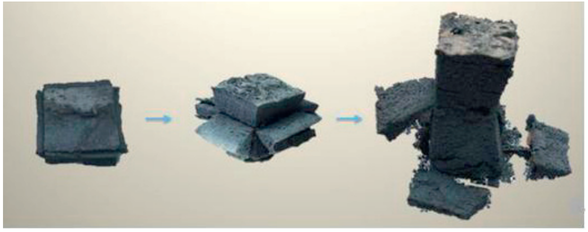
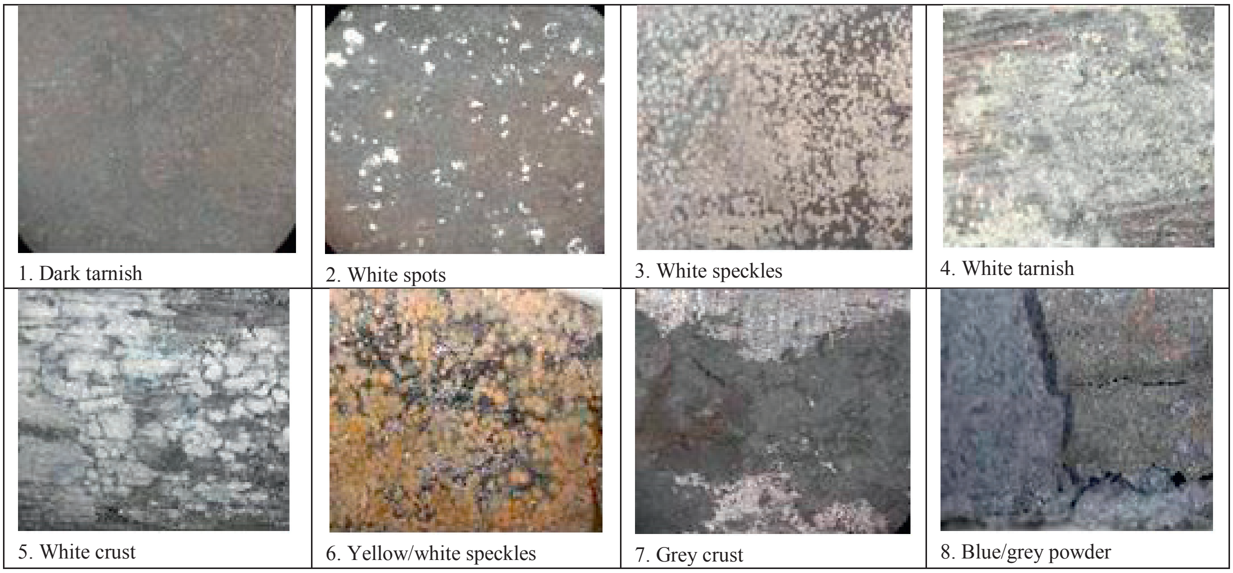


Fig. 11: Extreme corrosion development of Sb-rich Pb alloy

3.2 Macroscopic and microscopic investigations

The selected 95 letters showed under binoculars (60x magnification), a number of groups with visually different corrosion appearances. A wide but consistent variation of eight corrosion types, from thin dark layers over yellow and powdery white towards grey and totally decomposed alloys, was noted. Hence a classification of eight corrosion types, based on the visual morphology, was made (Fig.12).

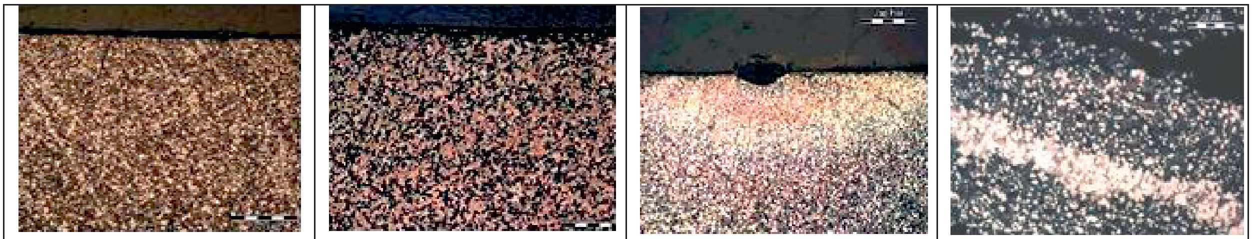


1. Dark tarnish
2. White spots
3. White speckles
4. White tarnish
5. White crust
6. Yellow/white speckles
7. Grey crust
8. Blue/grey powder

Fig. 12 Classification of eight corrosion morphology types observed on the selected letters, binocular images on the surface, 60x

The relevancy of this method for selecting corrosion types was investigated with following analytical techniques. If these corrosion appearances are to be related to different corrosion forms, the advantage of selecting on visual appearance is significant for a collection of many thousands of letters. It could enable the museum staff to evaluate the condition of the letters on a regular basis, assisted by these comparative images.

Microscopic study of the selected letters shows that all corrosion forms are limited to a relative thin surface layer (examples Fig.13a, 13b). Exceptions are to be found with the so called ‘white spots’ (Fig.12, Type 2) and ‘blue/grey powder’ (Fig.12, Type 8) corrosion. ‘White spots’ is a severe corrosion, concentrated at certain points on the surface (Fig.13c), also evaluated with SEM-EDX (3.3.2, Fig.17b). The ‘blue/grey powder’ (Fig.13d) is for certain the most destructive corrosion type for the complete alloy, which decomposes in its structure to a grey powder (3.3.2, Fig. 17a).



13a: Dark tarnish, 100x, Bright Field | 13b: Grey crust, 100x, Bright Field | 13c: White spots, 200x, Bright Field | 13d: Blue/grey powder, 200x, BF

Fig. 13 Images of corrosion layers in cross section

3.3 Analytical research

3.3.1 The XRF data was at first instance mainly used to determine similarities or differences in alloy composition between the letters of a defined printing type and corresponding historical period. Also, the uniformity or differences within each letterbox, was checked. Very often, museum conservators have the assumption that letters from a certain type, period or cast all have a similar composition. The different corrosion problems however, presented together in a same letterbox, suggest differently. Since the alloy quality was only to be checked poorly in former times and the old letters were used over and over again to cast new and sharp ones, it is important to know that every letter can be in fact another alloy.

The obtained results on the 95 measured letters vary in an important way, which leads to the conclusion that the letters do not show consistent relations in alloy composition, historic period, letter type or letterbox they were kept in.

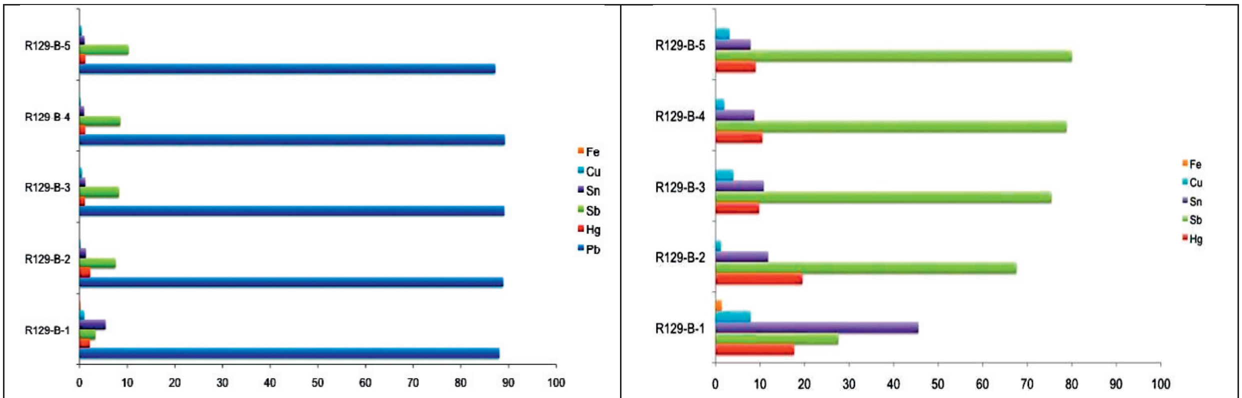


Fig.14a: Five letters from box R129-b, all elements | Fig.14b: Box R129-b, alloying and trace elements only

Although lead is the foremost alloying component, with an average of 88% (% to the fitted and normalized spectra data) from all measured letters, Fig. 14a and 14b show the variations of the other alloying elements. As an example here, the type ‘Nonpareille’ by Haultin with sample coding R-129-b-5 to R-129-b-2 show a high content of Sb, whilst Sn is low. R-129-b-1 however shows a high Sn value and lower Sb. More similar results were drawn from other sets. A very important note has to be made on the occurrence of mercury in these letters. It is uncertain why Hg is detected as an element in these printing letters and should be investigated further. Besides Pb, Sb, Sn and Hg, also Cu and Fe are found in very low concentrations, supposing they are not added intentionally as alloying elements. Fe may be present due to the use of moulds, castings spoons or mixing rods.

Other correlations could however be detected from the XRF-results. A group of 40 samples, selected and classified by corrosion appearance (see chapter 3.2), show very similar alloy compositions. The letters, which developed 'white spots' (Fig.12, Type 2) show higher values of Sn besides the Sb presence (Fig. 15a and 15b). On the other hand, the 'blue/grey powder' corrosion type (Fig.12, Type 8), contains high values of Sb and very little Sn (Fig. 16a and 16b). Since these two corrosion types seem to be the most severe, it is important to concentrate on the presence and on the ratios of Sb and Sn in the Pb-alloys.

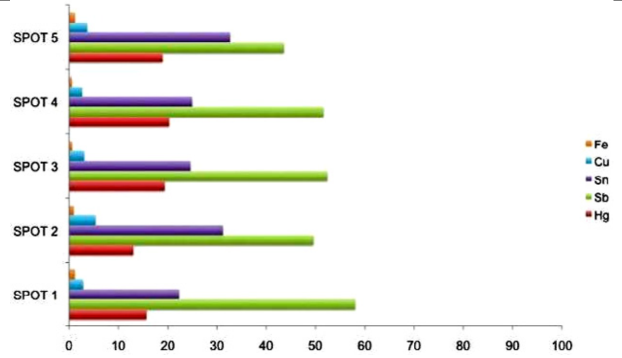


Fig.15a: Alloy elements of letters with 'White spots' corrosion

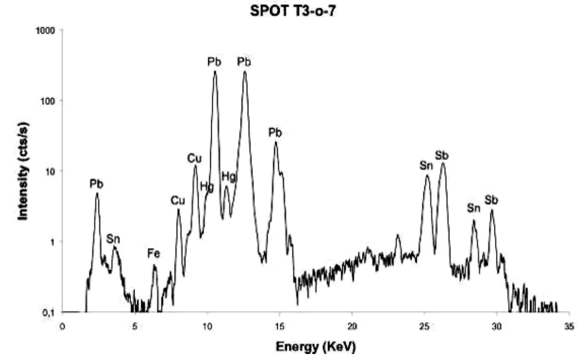


Fig.15b: XRF-Spectrum, sample T3-o-7 (with SPOT corrosion)

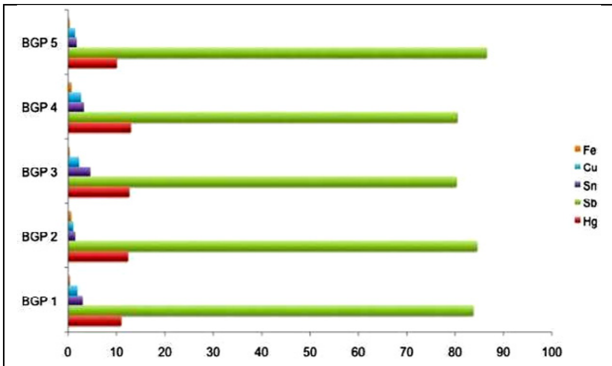


Fig.16a: Alloy elements of letters with 'Blue/grey powder' corr.

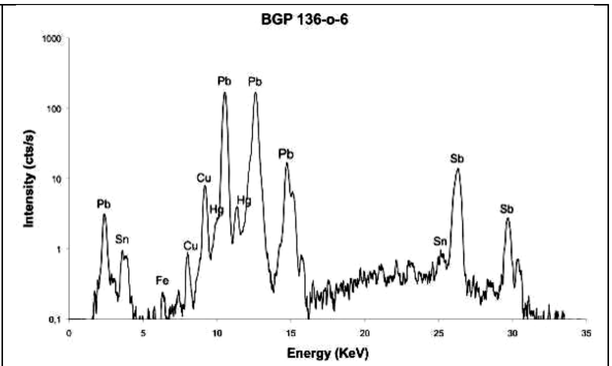


Fig.16b: XRF-Spectrum, sample 136-o-6 (with BGP corrosion form)

It might be concluded from these findings, that each kind of corrosion is directly related to a specific alloy. Since the letters with different kinds of corrosion occur in a same letterbox, and are therefore influenced by identical circumstances (kinds of wood composition of the box, acid emissions, temperature and RH-values and fluctuations), this assumption seems important.

3.3.2 SEM-EDX was used to study a limited selection of self-casted alloys and letters, following the XRF results and to study corrosion phenomena, derived from the microscopic images. The samples, embedded for the optical microscopy (3.2) were reused.

Consistent mappings and thorough work on the obtained results was unfortunately not possible in the short notice of time, given to this first step overview research project. Follow-up research must deliver more data to be compared to the other findings. The image in Fig. 17a shows the cross section of the corrosion Type 8 (Blue grey powder), where the metallic antimony is visible in the corrosion layer. Fig. 17b shows the cross section of corrosion Type 2 (White spots), on which the local pitting is to be observed. Fig. 17c is an image on the surface of corrosion Type 7 (Grey crust), whereof XRD results (see below) show Cerussite, Hydrocerussite and metallic Pb and Sb elements.

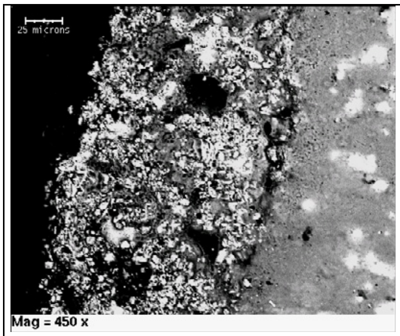


Fig. 17a: Backscattered image of BGP (Type 8)

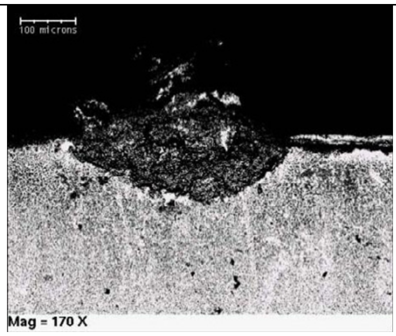


Fig. 17b: Backscattered image of SPOT (Typ 2)

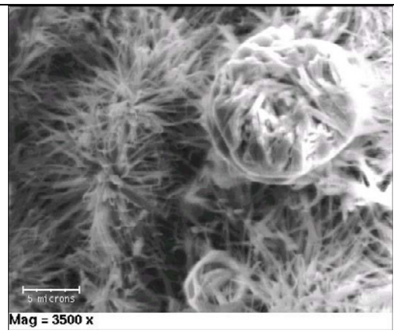


Fig. 17c: SEI of corrosion type 'grey crust'

3.3.3 XRD results showed mainly a number of lead corrosion products, although some peaks in the spectra could not be identified. The obtained results on powdered corrosion samples from selected original letters show a range of lead corrosion products: Cerussite ($PbCO_3$), Hydrocerussite ($2PbCO_3.Pb(OH)_2$), Lead Acetate Oxide Hydrate ($Pb_3(CH_3CO_2)2Pb.H_20$) and Lead Formate ($PbC2H_2O_4$). In three samples a crystalline phase with the structure of Sb is present. Powder XRD does not allow to determine the exact chemical composition of this alloy and other methods like SEM or TEM EDX should be used to characterize. These findings are however matching with the microscopic images which show clear metallic elements present in the corrosion bulk.

It could be concluded from the measurements that the samples (i.e. the original letters) were exposed to formic and acetic acid. If the letters were exposed to a combination of the two acids (as can be expected from the long term environmental conditions), unknown corrosion products are formed which can explain unidentified intense peaks in the spectra. These peaks could not be indexed with data from the ICDD. Full details of this research part are to be published.

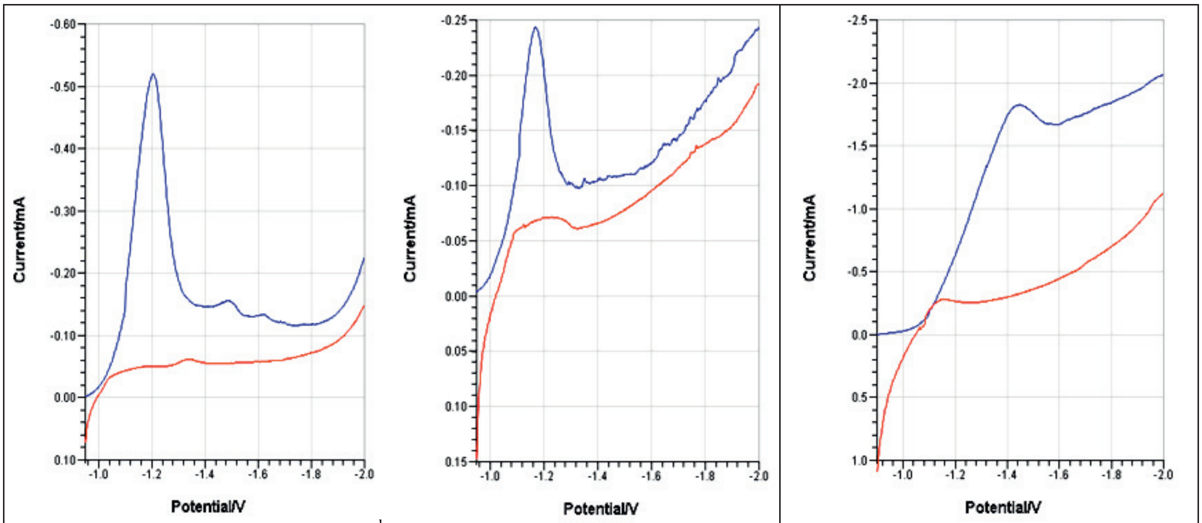
3.4 Environmental study

Electrolytic measurements on metal coupons[5], after being placed in the museum for four months (see 2.5), were used to evaluate the ambient and general level of corrosive elements in the interior atmosphere. Since the letters consist mainly of lead, with additions of antimony and/or tin, sets of three coupons were composed to be placed at different spots in the museum. Each set holds pure Pb and two Pb-alloys: Pb 70; Sb 25; Sn 5 and Pb 70; Sb 5; Sn 25 w% (see 2.2).

Different spots in the museum, matching storage places for the historical letter boxes, were selected in the print room and the adjacent storage room, including a showcase where standing types and their prints are shown to the public. Also on these standing types, corrosion was in some cases detected by the museum staff.

The results from the electrolytic measurements show clearly that the pure Pb coupon from within the showcase (location IV) showed a more severe corrosion, compared to those placed at other spots in the museum (locations I-III, V-VII). It is clear that on the coupon from the showcase, the Pb-oxide peak is much more substantial in current (-1,8 mA) and in surface area, extending towards the H_2-development at the end of the curve (Fig. 18c).

The other six measurements show mainly a single Pb-oxide peak with a moderate current (resp. -0,53 and -0,24 mA) and a well-defined position, outlined by a sharp peak. (Fig. 18a and 18b)



18a: Location I, Pb oxide peak, -0,53 mA | 18b: Location VII, Pb oxide peak, -0,24 mA | 18c: Location IV, very large peak area, -1,8 mA

Fig. 18: Compared results from voltammetry measurements on the metal coupons placed in the museum.

Besides these findings, measurements on the alloyed coupons also show that it is very difficult to interpret the voltammetry curves from the Sb- and Sn-rich Pb-alloys as there is too little data available on this and the metals in the alloy and their possible corrosion products interfere in the reading of the curves. Also, these alloys did not react in a more important way then the pure Pb did, therefore it seems not beneficial to use these alloys as coupons for environmental controls, in contrast to the very different results which were gathered from the adapted Oddy test.

The same alloys and the pure lead were also used in the simultaneously performed Oddy-test of which the conclusions are complementary to the observations on the museum objects. The first important result from this setup was the fierce corrosion reaction towards lead alloys from the beech wood, along with the expected corrosion reactions from the oak, multiplex and hardboard. Pine was least affecting the pure Pb nor the selected alloys. The second significant result was the extremely strong corrosion reaction with a total decomposition of the Sb-rich alloy under formic acid environments, with or without the presence of acetic acids. Under the same conditions, pure Pb was only corroded in a much lesser way. This indicates that the presence of high Sb-amounts in Pb-alloys may act as a predominant factor to initiate extreme corrosion under strong formic acid conditions.

4. Conclusions

However the amount of letters which are considered to be endangered is relatively small, the survey shows the need for examining the whole collection of lead printing letters on corrosion problems. The project ensures that the different corrosion forms are to be distinguished by macroscopic and simple microscopic means, which is beneficial for the museum staff to go through the complete and vast collection of letters.

Different alloys of the letters appear to be present in the different boxes, but also in every box itself and even between the same kind of letters. Therefore it seems not possible to link any of the original types to certain historical periods and there is no correlation to be found between the actual letters and recipes from the time.

Also, the recasting of letters, possibly also partial, disturb any correlation between original alloys which the printers may have used, and what is to be found as letters today.

It is shown that the lead alloy letters, containing high antimony and low tin levels are the most prone to severe corrosion. High levels and prolonged exposure of acids are to be found and lie at the basis of the metal deterioration. Besides this, it was remarkable that the letter boxes which were made with a beech wood bottom, showed the most and strongest corrosion formation. The adjacent Oddy-test showed an exceptional fierce expanding corrosion form, especially on the high Sb, low Sn Pb-alloy, in a formic acid atmosphere.

The environmental conditions in the museum are part of the corrosion problem, as there are constantly rather high acid levels present. It is however very difficult if not impossible to be avoided, as the main material for the museum interior and many of the machines and furniture consist out of oak, which is historically related to the interior. It is however possible to limit the amounts of added acids by modern materials by sensible selection for temporary exhibitions and by investigating how controlled ventilation can diminish the problem.

The findings of this first stage project give a clear view on the importance of the damage to the collection and leads to more defined ways of collecting useful data to tackle the causes of the problems and to enable conservators to design a dedicated preventive conservation plan. Also, possible conservation and/or restoration actions on the corroded objects can be derived from the results in this and in sequel researches.

Follow-up research on corrosion is to be performed by I. Martini, University of Genova (Prof. dr. P. Piccardo) in cooperation with Prof. dr. C. Martini (Instituto di Metallurgia, Università di Bologna). At Antwerp University it is expected to have a continuing research towards the active and preventive conservation treatments to ensure the important historical collection in its future.

Acknowledgements

The project was initiated and supervised by the author. The practical research work and the oral presentation at Yococu conference was performed by master students Emilie Lieten and Marjan Jacobs, Metal conservation department of the Artesis University College. Fitting the XRF results with Axil was performed by Dr. O. Schalm. The Oddy test was performed by master student I. Martini on Erasmus exchange from the University of Genova, under Prof. dr. P. Piccardo, Chemistry Department. There was a beneficial cooperation with the University of Antwerp, research group of Environmental studies, Dept. of Chemistry with Prof. dr. K. De Wael and dra. B. Krupinska, with their research on particular matter and air quality within the Museum Plantin-Moretus. SEM-EDX was performed at MiTAC, Antwerp University, Chemistry Departement, Prof. Dr. K. Janssens and dra. W. Anaf. XRD analysis was performed by EMAT, Antwerp University, Physics Department with Prof. dr. D. Schryvers, dr. A. Abakumov and dra. E. Grieten. The research was made possible through the kind admission of the Museum Plantin-Moretus, Conservator Mrs. I. Kockelbergh and the City of Antwerp, vzw Erfgoed en Musea, Head Mr. D. Aerts.

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