# RESTORATION OF AN AFRICAN IRON-AGE FURNACE

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In August 1998 a furnace dating from the South African Late Iron Age. + 1 100 AD, was vandalized at the Melville Koppies Nature Reserve in Johannesburg. The Melville Koppies Management Committee and the SA National Monuments Council asked one of us (Grant) to attempt a restoration. XRF and XRD analyses proved that the local soil was used in construction and that the furnace bottom had been vitrified at approximately 1 100 °C, but that the furnace wall had been heated only to 400 °C or less. There is thus a possibility that this furnace was used as a forge, and not for smelting. Some months were spent, both in SA and the UK, in identifying possible solvents and consolidants, and in testing for the best method of application, concentration of consolidant and drying time of the pieces to be refitted. As consolidant we selected B67 (poly-isobutyl methacrylate), and solvents industrial methylated spirits (IMS) and cyclohexanone, which produced good strengths and minimal colour change on drying. The furnace has been successfully restored. The consolidant/solvent combinations worked well. causing only minimal darkening in places. These results should be of use to anyone who has to repair or restore a fired-earth structure.

Keywords: AFRICA, IRON AGE, SMELTING, FORGING, RESTORATION

# 1. Introduction

Melville Koppies (a 'koppie' is a hill in Afrikaans), situated near the city centre of Johannesburg, South Africa, is an important pre-colonial national historical site. In the 1960's the remains of a low-shaft, non slag-tapping, bellows-blown furnace were discovered here (Friede, 1979). The furnace is situated near the crest of the koppie, and 'seems to have been placed in regard to consistent updrafts rising from the valley upwards towards the crest of the slope' (Mason, 1986). Initially only about a centimeter of the

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furnace shell projected above present surface level, but after excavation it was described thusly (Figure 1):

"The furnace resembles a wide mouthed globular pot in shape nearly 3 ft. in maximum external diameter and slightly over 2 ft. in depth. The furnace has two tuyere apertures about 6 inches wide and 1 ft. deep. The material is hard baked, reddish, clayey soil with slag crusted on parts of the interior" (Kusel, 1974).

Radiocarbon dating (Y-1338) of material collected from the furnace floor yielded an age of AD1060  $\pm$  50 years. The furnace was 'associated' with pottery resembling middle Iron-Age assemblages from several nearby sites (Mason, 1986). This was one of the best-preserved Iron-Age furnaces in South Africa. We say 'was' because in August 1998, the site was entered illegally, and the glass casing protecting the furnace destroyed by persons unknown. A heavy glass panel fell onto the furnace itself, causing extensive damage near one tuyere port (Figure 2). Several broken pieces of furnace wall and many smaller shards were retrieved by the University of the Witwatersrand Archaeology Department (Figure 3).

The Melville Koppies Management Committee and the SA National Monuments Council asked one of us (Grant) to attempt a restoration. It was decided to adopt a multidisciplinary approach, involving experts from the fields of ceramics restoration (Botha) and cement and concrete technology (Makovy). It soon became obvious that we did not understand how the furnace was constructed. Parts of the furnace wall and many of the smaller shards were extremely crumbly, similar in texture to the surrounding red soil. In contrast, the large pieces seemed well consolidated. Some sort of binding agent appeared to have been applied, although there apparently is no published reference to a previous restoration. The furnace bottom appeared ashy and vitrified. We decided to use XRD and XRF to try and identify the material used in the construction, and to quantify the degree of vitrification.

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# 2. XRD and XRF

A colour change between the surface and the middle of the large shards, resembling some form of chemical coating, is clearly visible in Figure 3. XRF analysis (Table 1) showed a difference in  $SO_3$ , CI and  $K_2O$  concentrations between the outer wall, middle and inner wall of the shards. We concluded that there was evidence of chemical stabilization. Our analytical chemist (Butcher) made a guess that the adsorbed agent was a polyorganic material containing some chlorine. It may never be known with certainty who applied it and when, but whatever its' chemical nature, it did not interfere with the restoration.

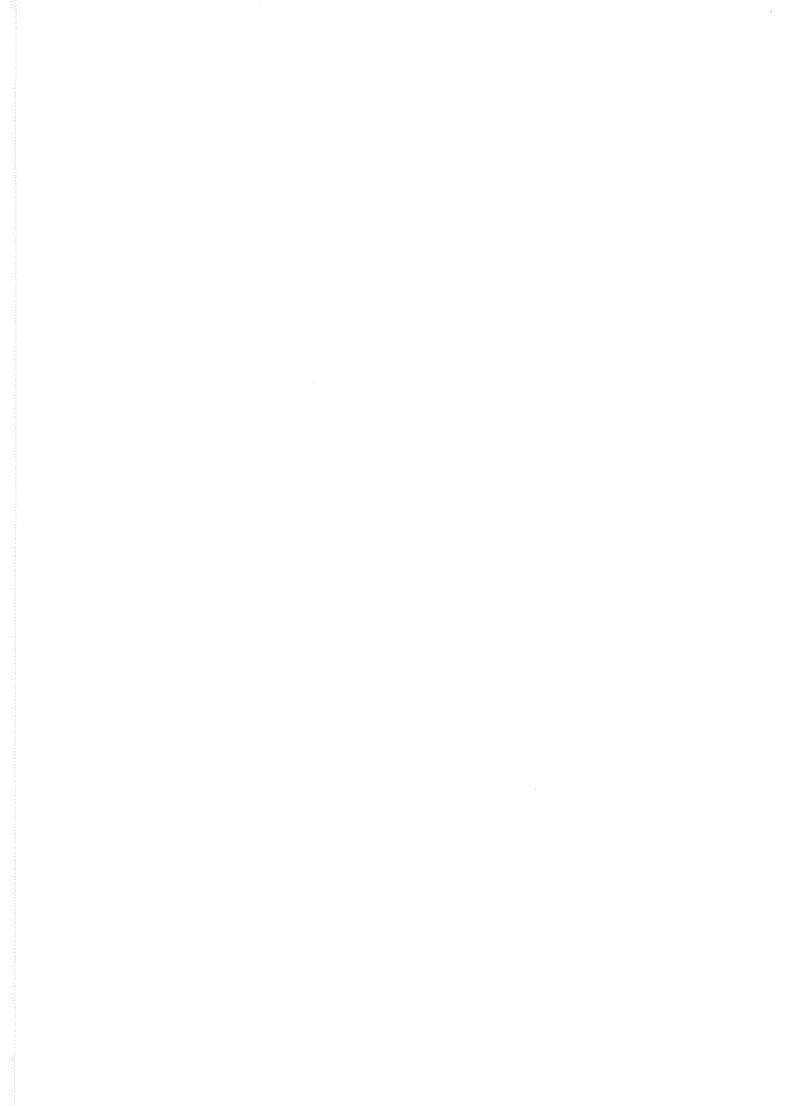
Samples of the bottom and the largest shard were analysed by XRD together with the surrounding soil. Results are shown in Figures 4-6. In Figure 4, it is clear that the large shard is made from raw soil. If this shard is representative, it appears that the furnace wall may have been heated to a moderate temperature only (possibly dried at  $\pm$  400 °C), but not so high a temperature as to change the XRD signature of the clay minerals. Samples of raw soil were then heated to 1000 °C and 1200 °C in a laboratory muffle. The XRD signature for raw soil heated to 1000 °C is similar to that of the furnace bottom (Figure 5). However, at 1200 °C mullite ( $3Al_2O_3$   $2SiO_2$ ) appears. This is not present in the furnace bottom. In addition, tridymite (a high temperature variety of quartz) appears in the XRD spectrum of the soil heated to 1200 °C, and quartz is beginning to disappear (Figure 6). The furnace bottom showed neither of these effects. We estimate that the maximum temperature the furnace bottom could have been subjected to was approximately 1100 °C.

# 3. Strength Development

Local soil was slurried with 2 consolidant solutions — B67 + IMS and B67 + cyclohexanone. Samples of both slurries were placed into polythene bags for 0-8 weeks. At the end of the prescribed period (0,6 and 24 hours, 3 days, 1,2, 4 and 8 weeks), individual packets were opened and the sample washed with acetone to terminate the hydration reaction. The residues were dried at 30 °C and presented to the XRD spectrometer. Significant strength development only appears to be present at 4 and 8 weeks, when both sets of specimens dried and hardened appreciably. The developed peaks in the XRD spectrum appeared to be clay (muscovite, kaolinite and halloysite) hydrates only. However, it is not recommended in future to use an acetone wash as this could have removed reactants.

# 4. Technical Conservation Process

Both the shards and the main structure needed to be consolidated before reconstruction. The shards were placed into solvent-resistant containers. Small holes, just big enough to introduce the pipette droppers, were drilled into these shards and infused with cyclohexanone. Pre-wetting assisted with the absorption of the consolidant solution. The consolidant [B67 in solution with cyclohexanone] was introduced into the shards along the lines where reconstruction was to occur (Figure 7). Infused pieces were then covered in plastic wrap to retard evaporation and allowed to dry for several weeks. The main body of the furnace was drilled, infused and wrapped in the same way.



The material chosen for adhesion and gap filling was B67 + IMS, mixed with fine soil dust (Figures 8). The gap filling mortar contained approximately ¼ volume of glass beads to prevent the mixture from making a smooth paste (Figure 9). This imitates the original textural qualities of the furnace. Joins between furnace body and pieces (Figure 10) should hold the structure firmly together, but are not quite as strong as the original fabric of the structure. Should it be required at a future time, joins should yield again in the same place if any deliberate forces are applied to them. Restoration was completed in August 1999 (Figure 11). Details of the selection of solvents and consolidant and a step-by-step procedure for restoration will be presented in a forthcoming paper (in preparation).

### 5. Conclusions

We needed to know the chemical composition and state of vitrification of the furnace walls and bottom in order to understand how the furnace was constructed. XRD and XRF gave us this information, but we were unable to determine whether or not the furnace was used for smelting or forging, although the latter seems more probable. Strength development after consolidation appears to be due solely to the formation of clay hydrates, but the acetone used to terminate the hydration reaction may have washed out reactants. The restoration complies fully with the National Monuments Council requirement that the

reconstruction be reversible, and the procedures developed should be useful for the repair or restoration of large fired-earth structures.

Acknowledgements

We would like to thank Mr. John Gomersall, Managing Director of PPC Ltd., and Mr. Barry Butcher, Chief Chemist - PPC, for generous assistance with XRD and XRF, and Mrs. Trish Urquhart and Ms. Rochelle Hatley of the Guild for the tireless hours they put-in on the restoration.

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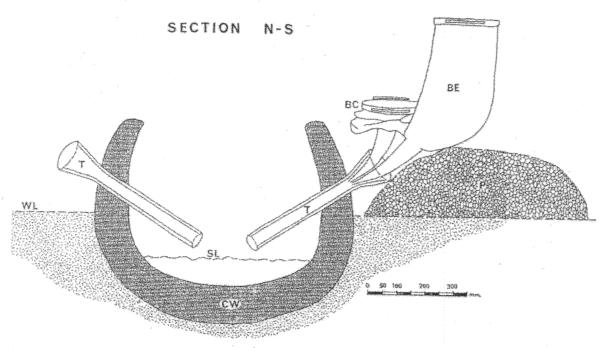


Fig. 1 - Reconstruction of iron-smelting furnace 7/63 from Melville Koppies Reserve, Johannesburg WL Excavated working level BC Bellows compressed BE Bellows extended CW Clay walls

P Platform

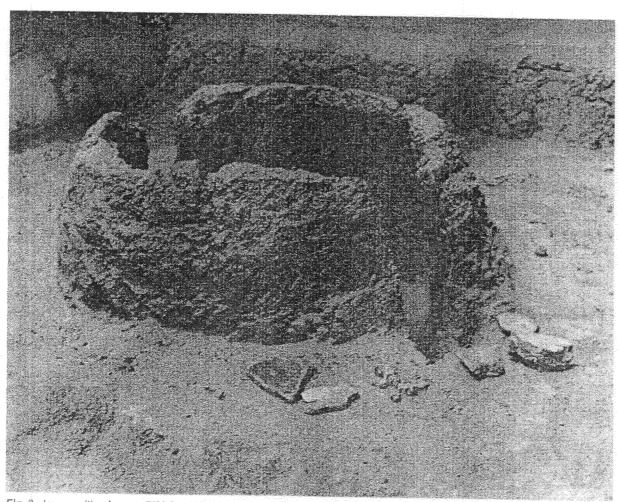
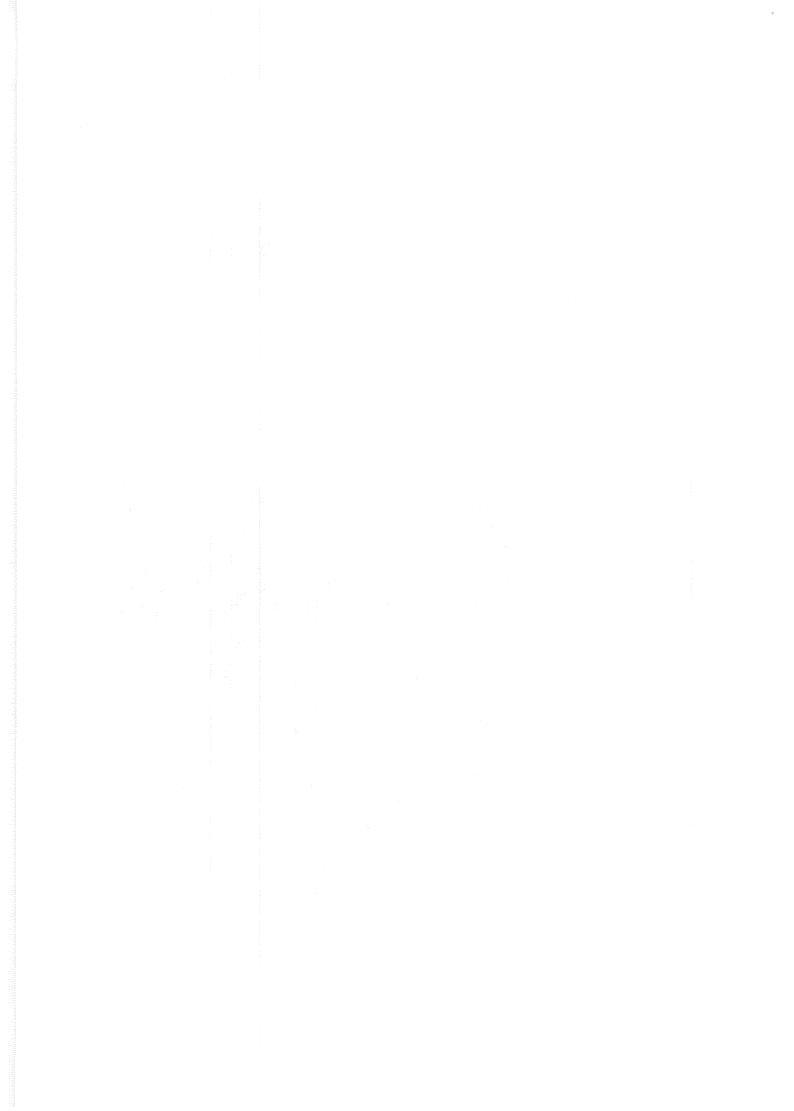


Fig. 2 - Iron-smelting furnace 7/63 from Melville Koppies Reserve, Johannesburg

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Figure 1: Appearance of the furnace of the when first excavated (Friede, 1979)



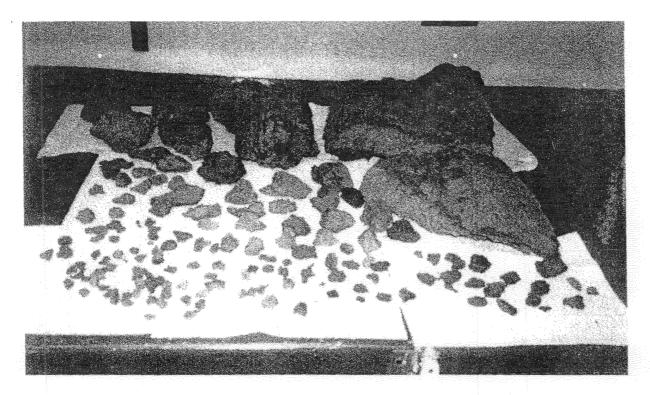
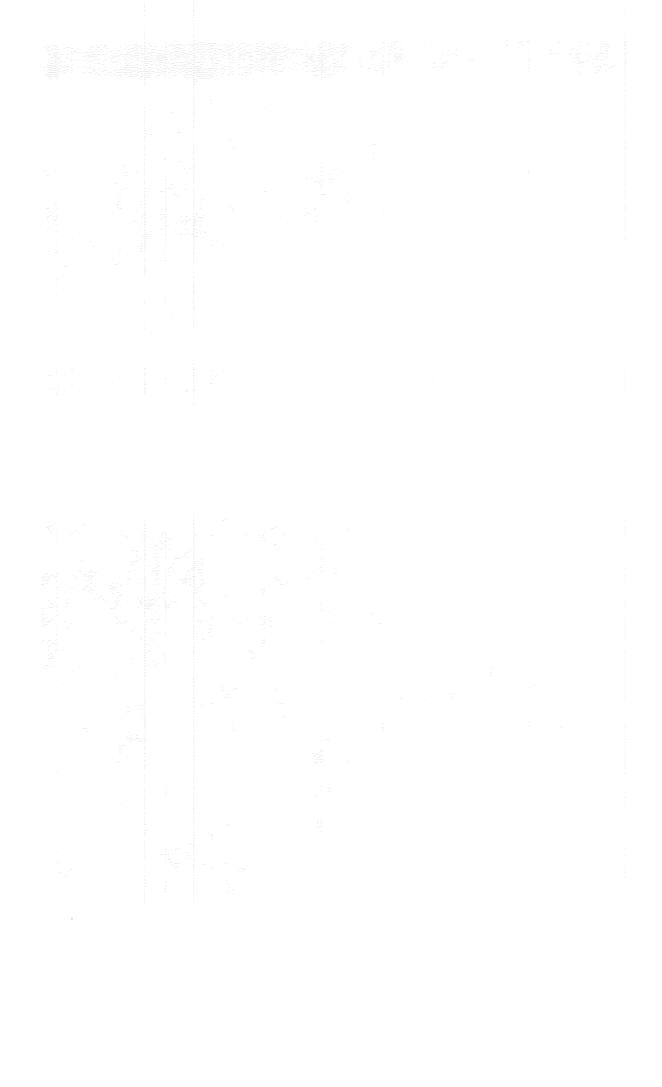


Figure 3: Large and small shards in laboratory prior to consolidation.



Figure 2: Broken Furnace, after large shards and glass were removed



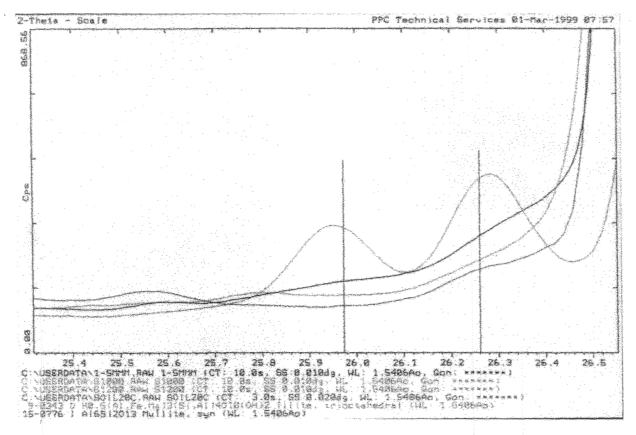


Figure 5: XRD spectra of raw soil (red) compared with furnace bottom material (black) heated to 1000 °C (green) and 1200 °C (blue).

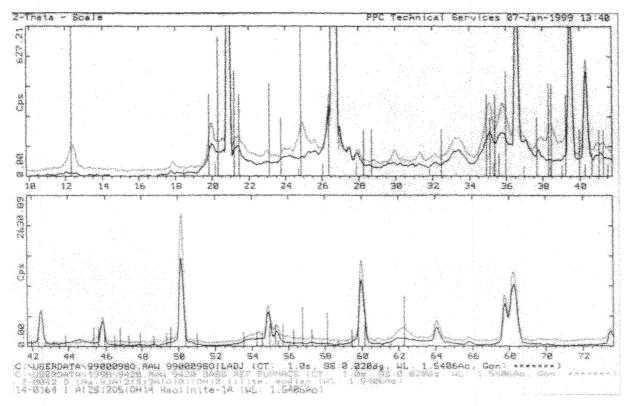
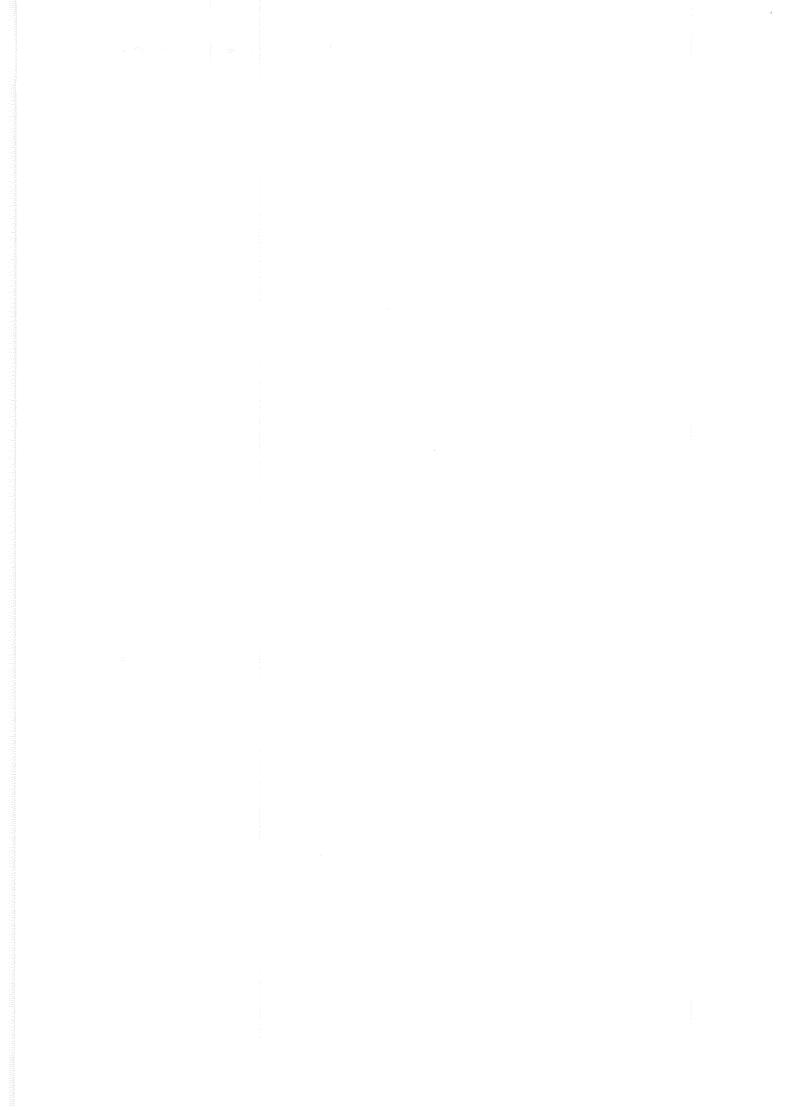


Figure 4: XRD spectra of raw soil (black) and material from large shard (green).



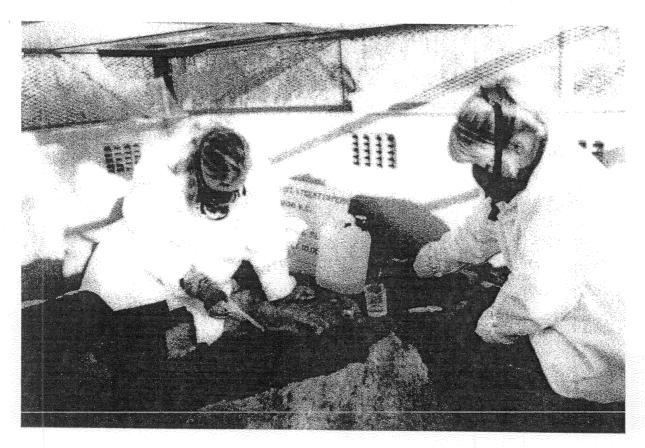


Figure 7: Trish Urquhart and Rochelle Hatley applying consolidant solutions.

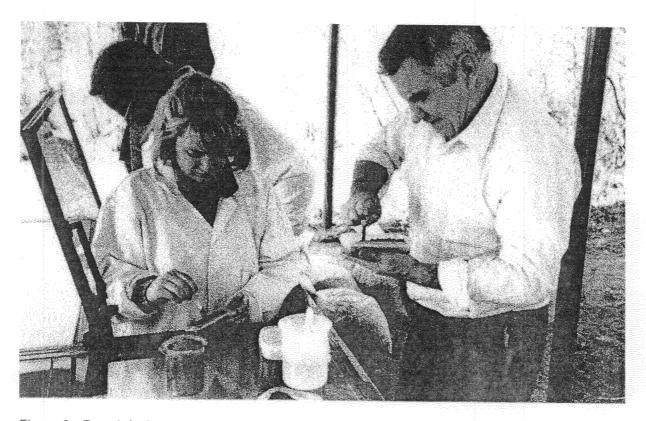
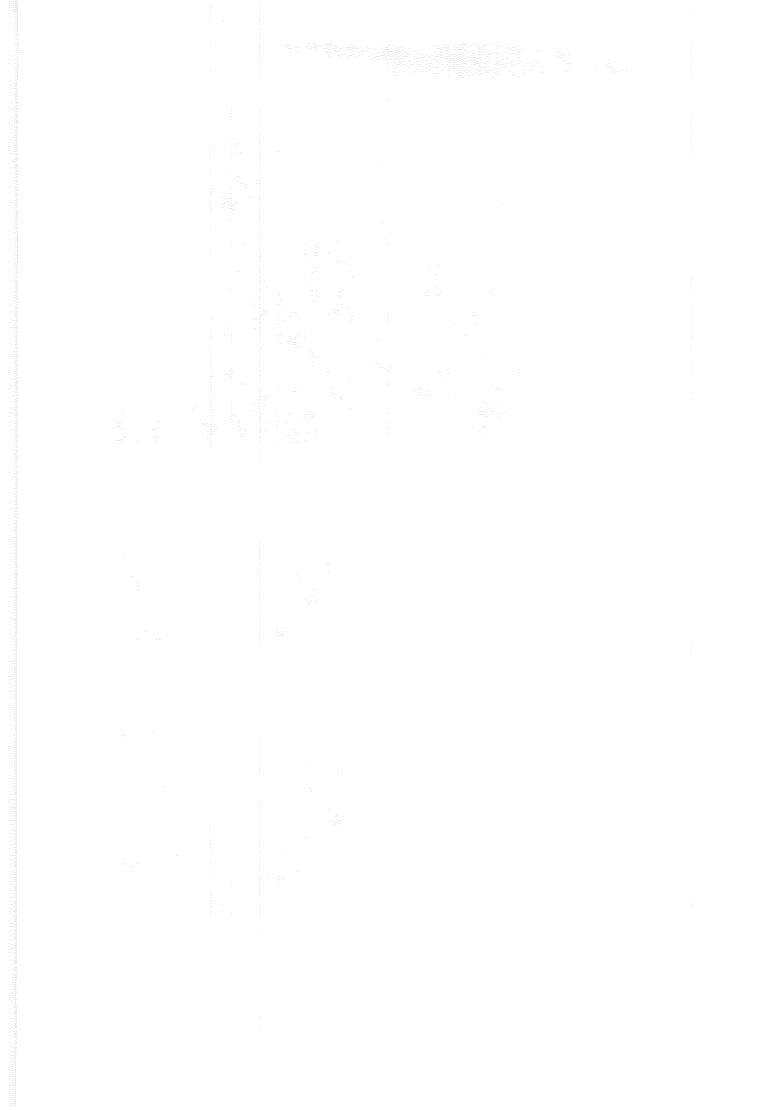


Figure 8: From left, Grant, Rochelle Hatley and Makovy preparing the gap-filling mortar.



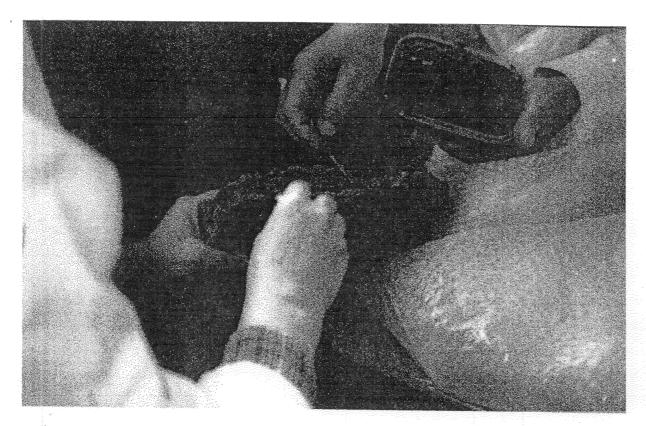


Figure 9: Mortar applied to the join of a large shard.

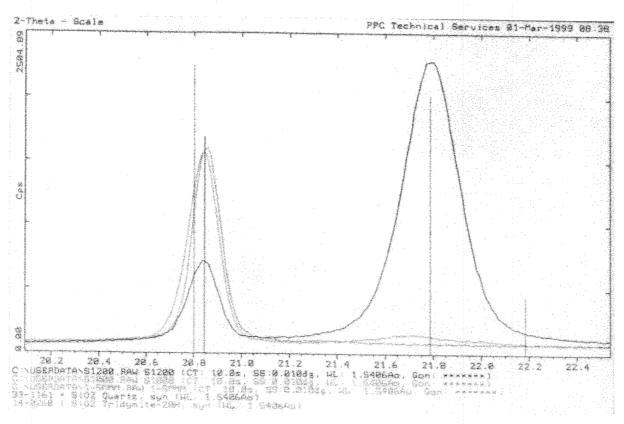


Figure 6: XRD spectra of raw soil heated to 1000  $^{\circ}$ C (green) and 1200  $^{\circ}$ C (black), compared to spectrum of furnace bottom material (blue).

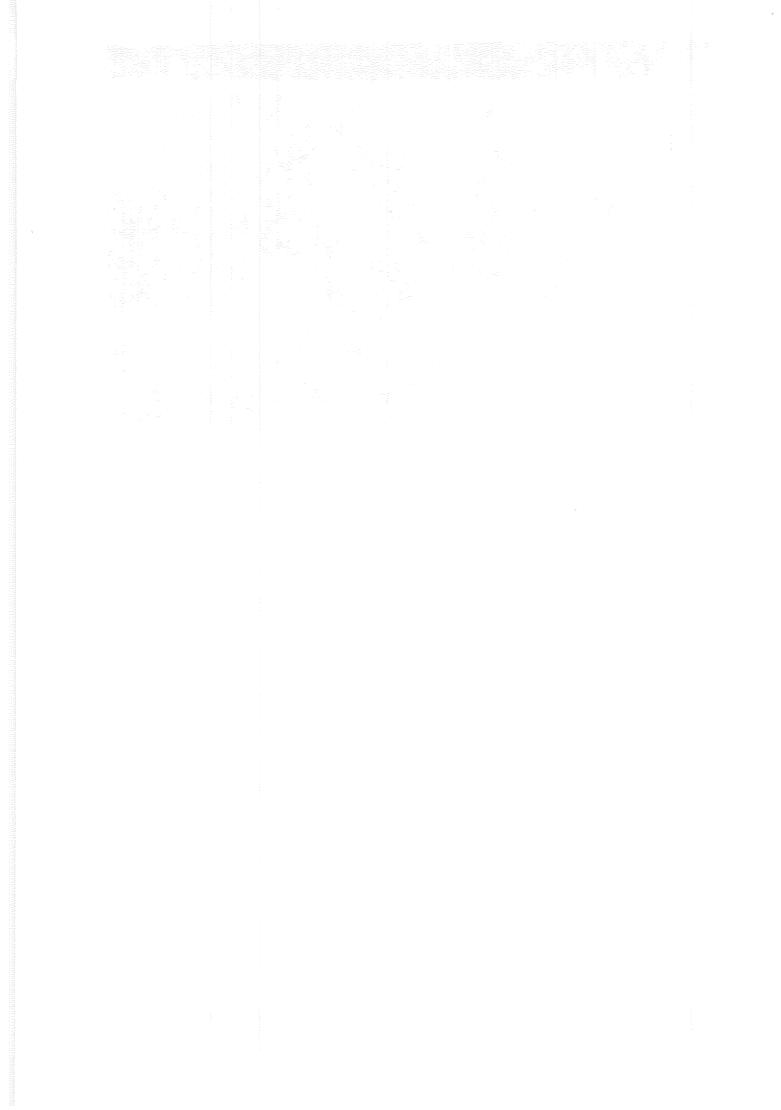




Figure 10: Ballasting the newly cemented shards.

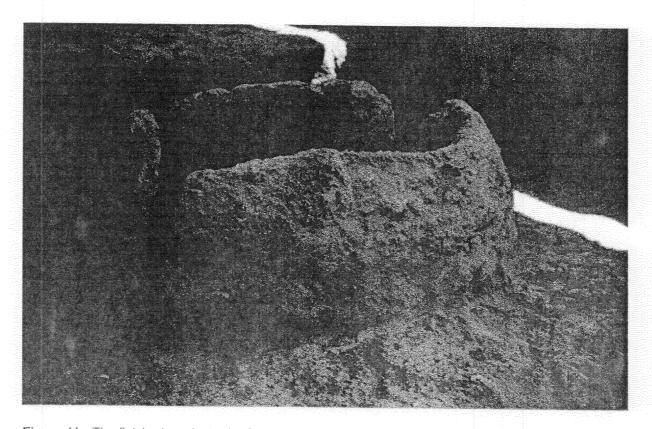
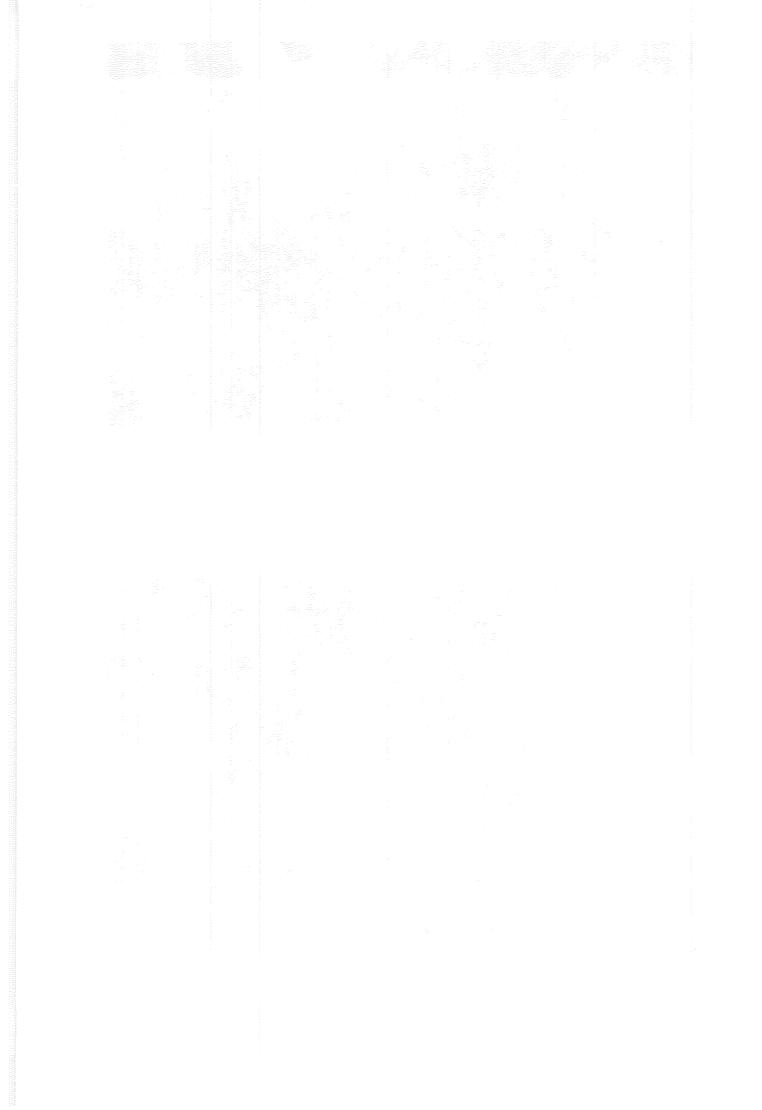


Figure 11: The finished product - the furnace today, after 8 months of experiment and restoration.



Consolidation with Paraloid B67 in IMS & Cyclohexanone

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#### ABSTRACT:

This paper follows investigations into the strength development of slurries used in the conservation of the Melville Koppies iron-age smelting furnace (Grant, Makovy 2000). In this project the polymer, Paraloid B 67 (isobutyl methacrylate) was used in conjunction with two solvents respectively: industrial methylated spirits (IMS) and cyclohexanone. It was found that the polymer slurries inadvertently produced sample pellets of distinctly different strengths when the solvents were interchanged. The slurry prepared with IMS as solvent at a 10% w/v polymer-solvent solution, cured considerably harder than the one prepared with cyclohexanone as a solvent at the same solution. The developed peaks in the XRD spectrum appeared to be clay (muscovite, kaolinite, halloysite) hydrates only' (Grant, Makovy 2000). In order to establish more conclusively the reasons for the different strengths, it was decided to study the resulting samples using a scanning electron microscope (SEM). These results showed the IMS prepared solution to have formed "bridges" between the particles comprising the slurry, whereas the cyclohexanone prepared samples showed the polymer to have consolidated the individual particles, forming far fewer connecting "bridges".

#### SAMPLE PREPARATION:

While the conservation of the furnace was in progress, it was found that the rate of drying of the prepared filler medium (employing IMS as solvent), could not be controlled / retarded sufficiently to avoid some darkening of the fills. This also resulted in an undesirably hard fill, incompatible with the rest of the structure, which is very friable. A decision was made to reduce the polymer concentration to 2%, and then to add this solution until there was just enough polymer to hold the slurry particles together on drying. Though the discolouration was successfully reduced, the resulting gap-fill was still very hard.

The samples were prepared to simulate the on-site process. Some dry material was taken from a redundant furnace sherd and two separate, but identical volumes were placed on a glazed ceramic tile. A 2% w/v solutions of the polymer in IMS and cyclohexanone respectively were pipetted onto the two samples and worked with a wooden spatula, as had been done on site. The slurries were left to dry at room temperature but remained crumbly on drying. The solution was introduced again and this was repeated until the cured slurries were fully consolidated and presented structurally solid bodies able to withstand vigorous hand-applied pressure. It was found that the IMS slurry reached this stage after the fourth application, whereas the cyclohexanone slurry remained brittle up to the sixth application. Both samples received ten applications of polymer/solvent solution in total, to simulate the saturation levels that had been achieved on the actual furnace. The samples were allowed to cure for five days at room temperature before they were presented for SEM analysis.

#### SEM RESULTS AND DISCUSSION:

The samples were viewed uncoated using a Philips XL30 environmental scanning electron microscope (ESEM) using a large field gaseous secondary detector (LDF) at 0.9 Torr.

Figure 1 below: A view of the untreated sherd at x120 magnification.

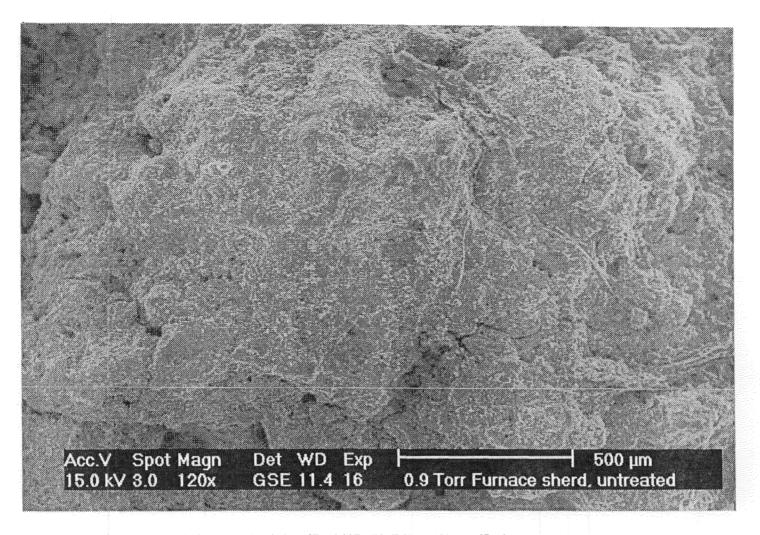
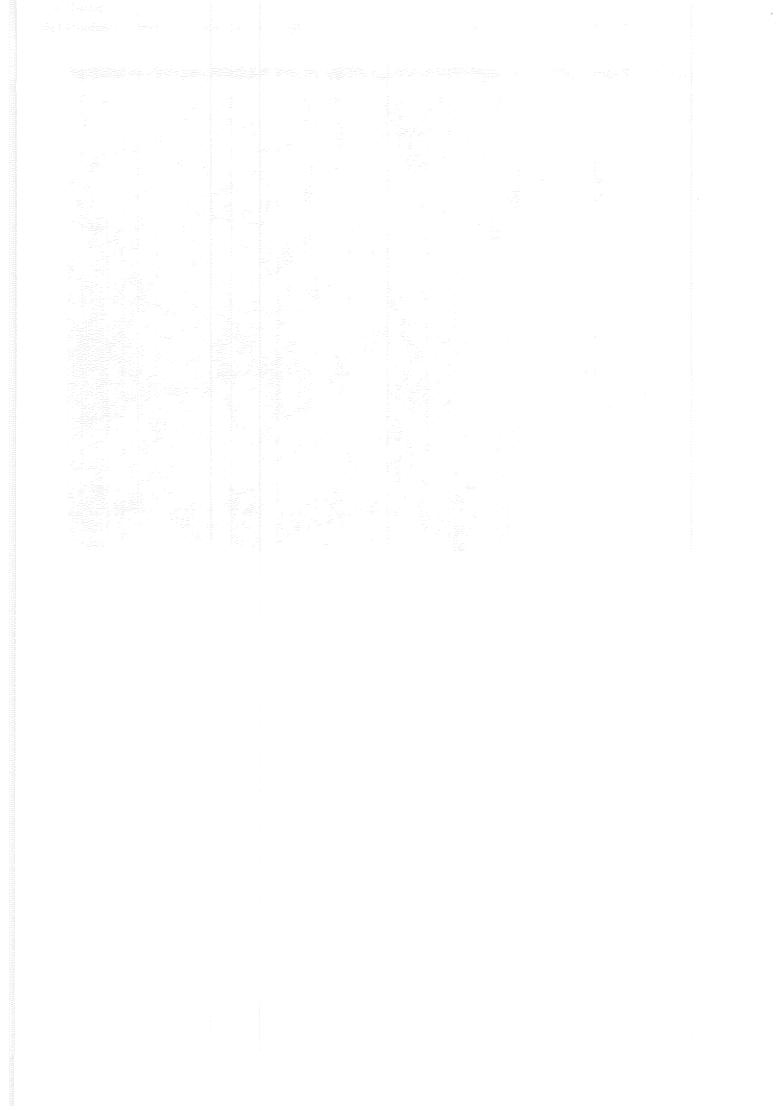


Figure 2 below: The sample treated with a 2% w/v solution of Paraloid B 67 in IMS at x120 magnification

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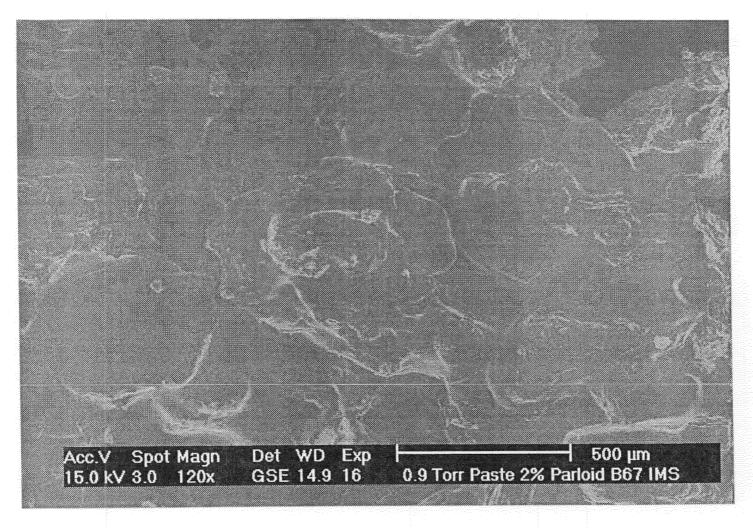
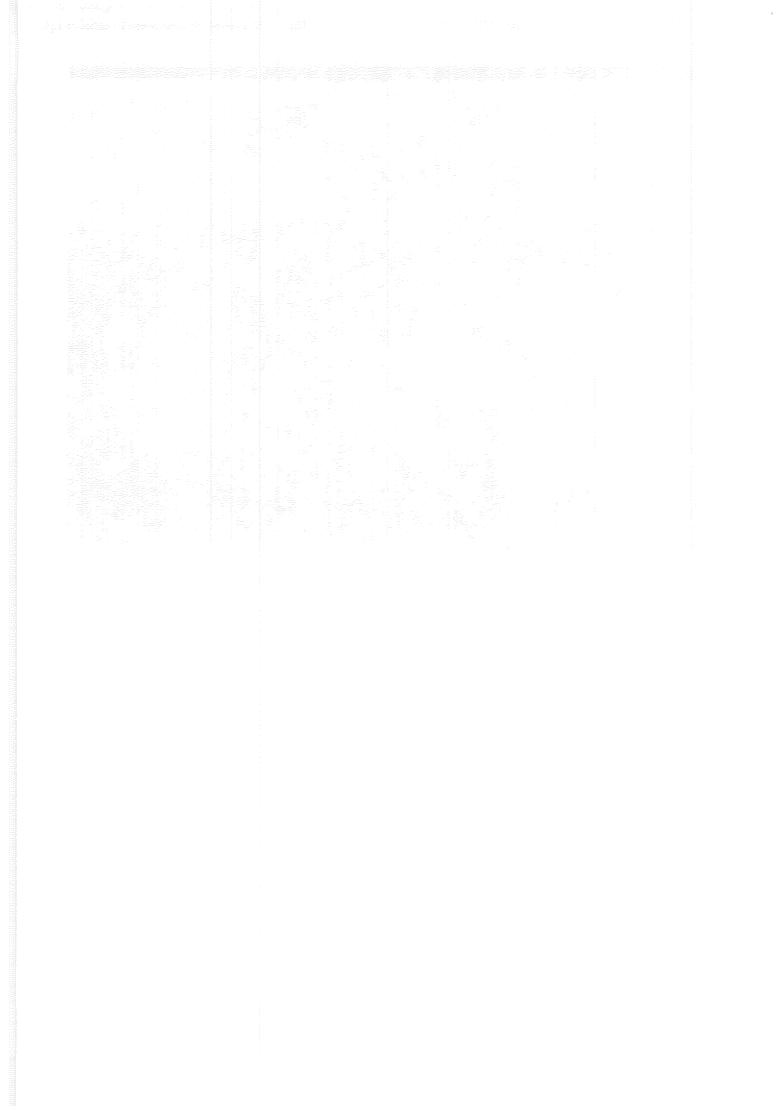


Figure 3 below: The sample treated with a 2% w/v solution of Paraloid B 67 in cyclohexanone at x120 magnification.



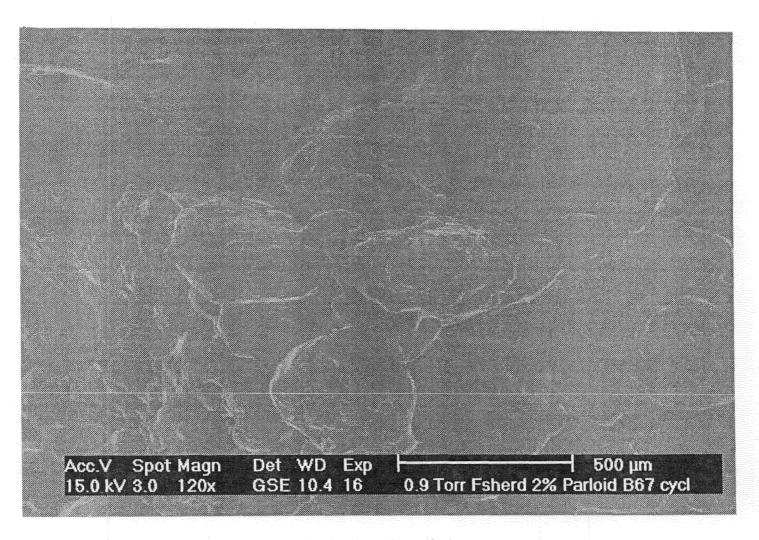
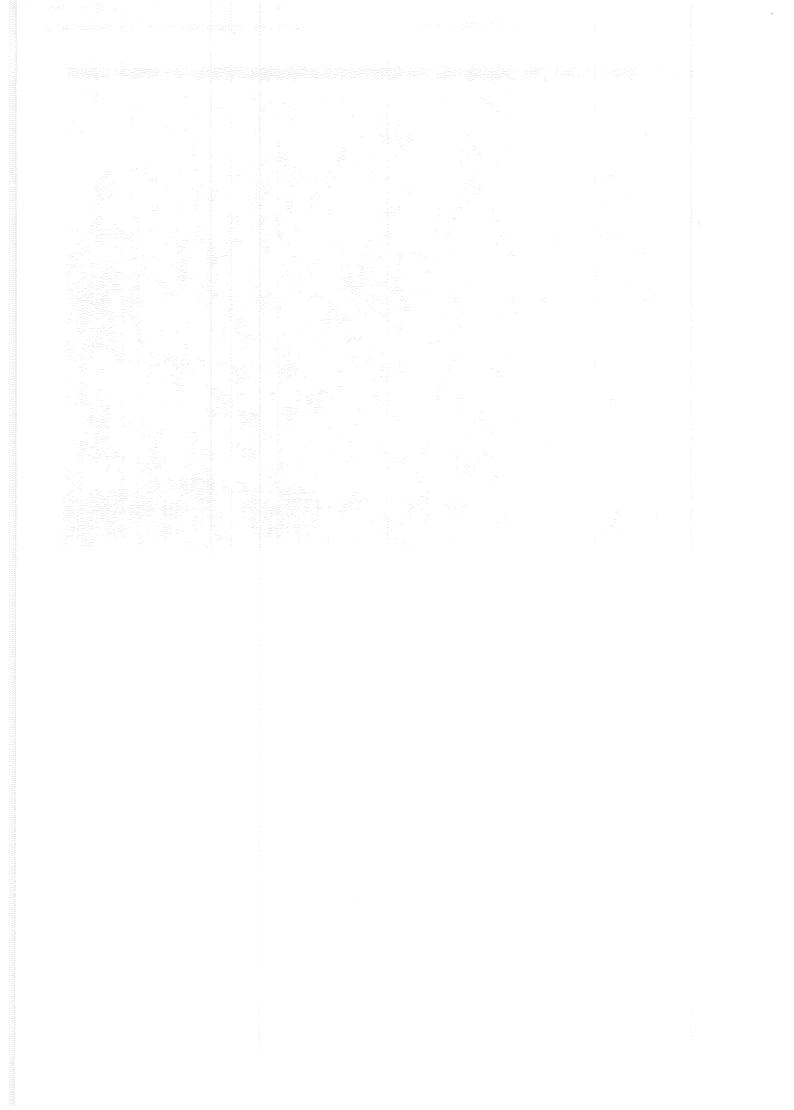


Figure 4 below: The surface of the Paraloid B 67 in IMS coating viewed at x120 magnification.



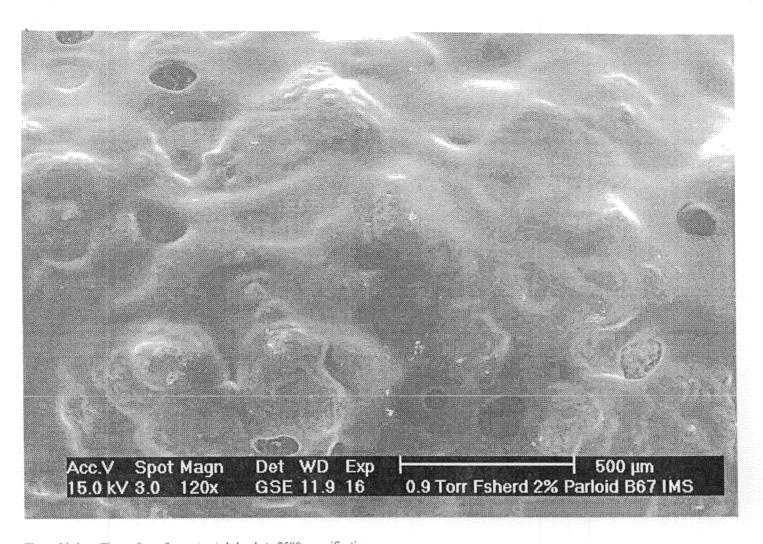
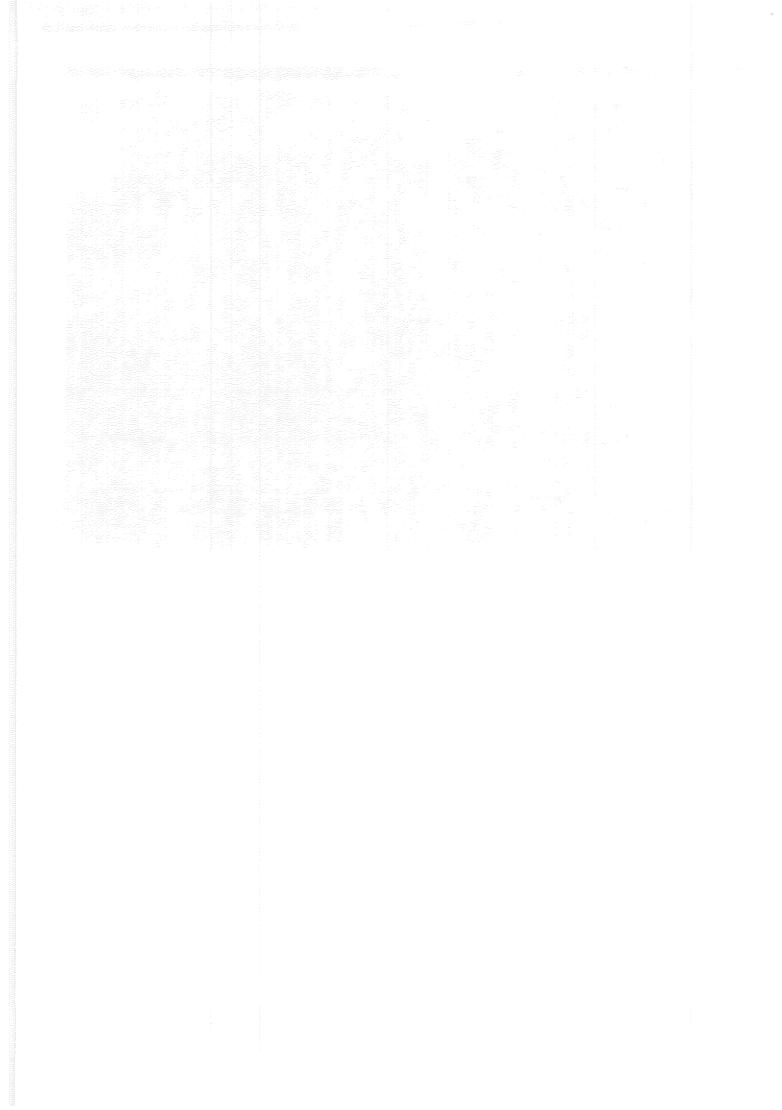


Figure 5 below: The surface of an untreated sherd at x2500 magnification.



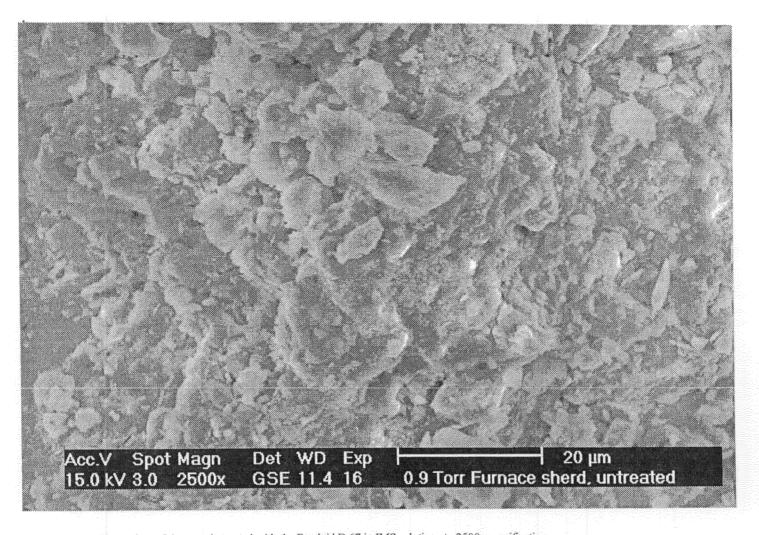
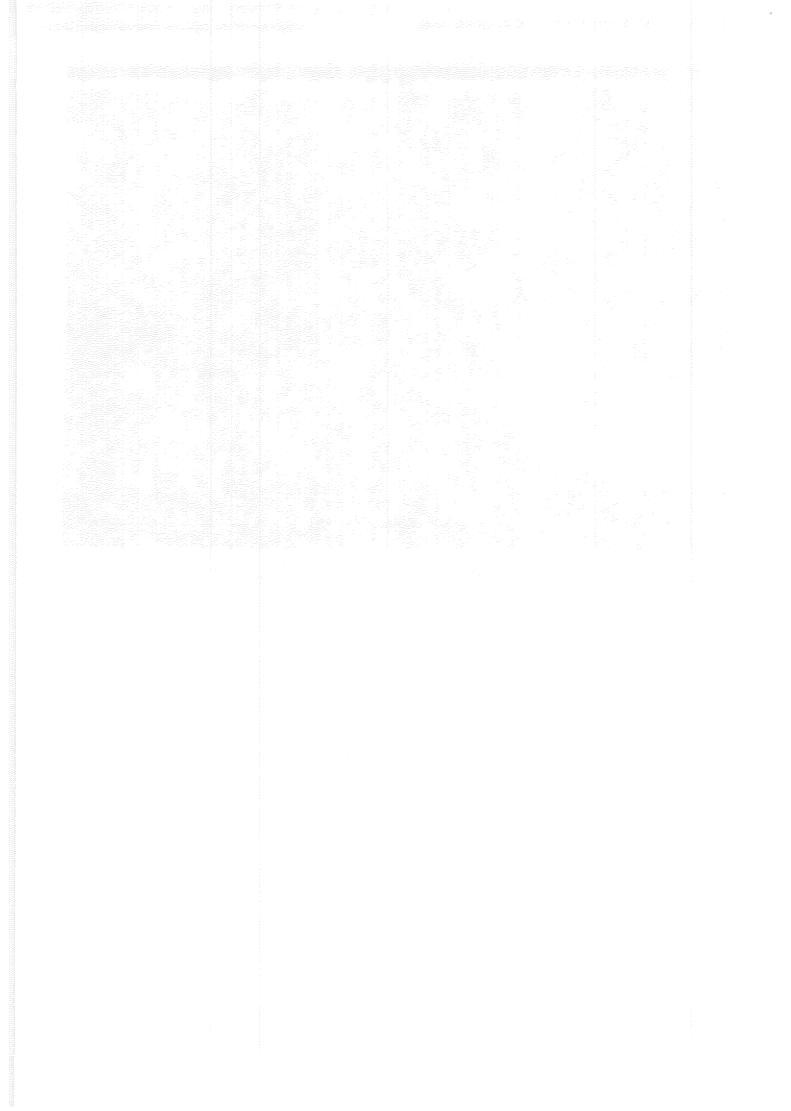


Figure 6 below: The surface of the sample treated with the Paraloid B 67 in IMS solution at x2500 magnification.



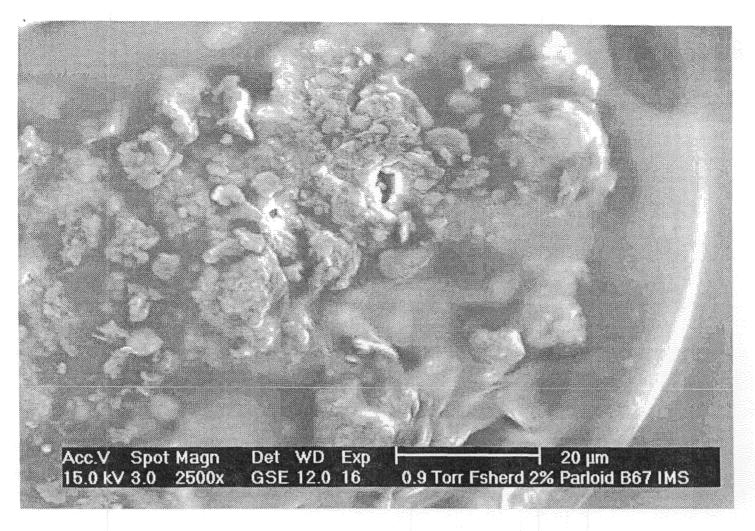
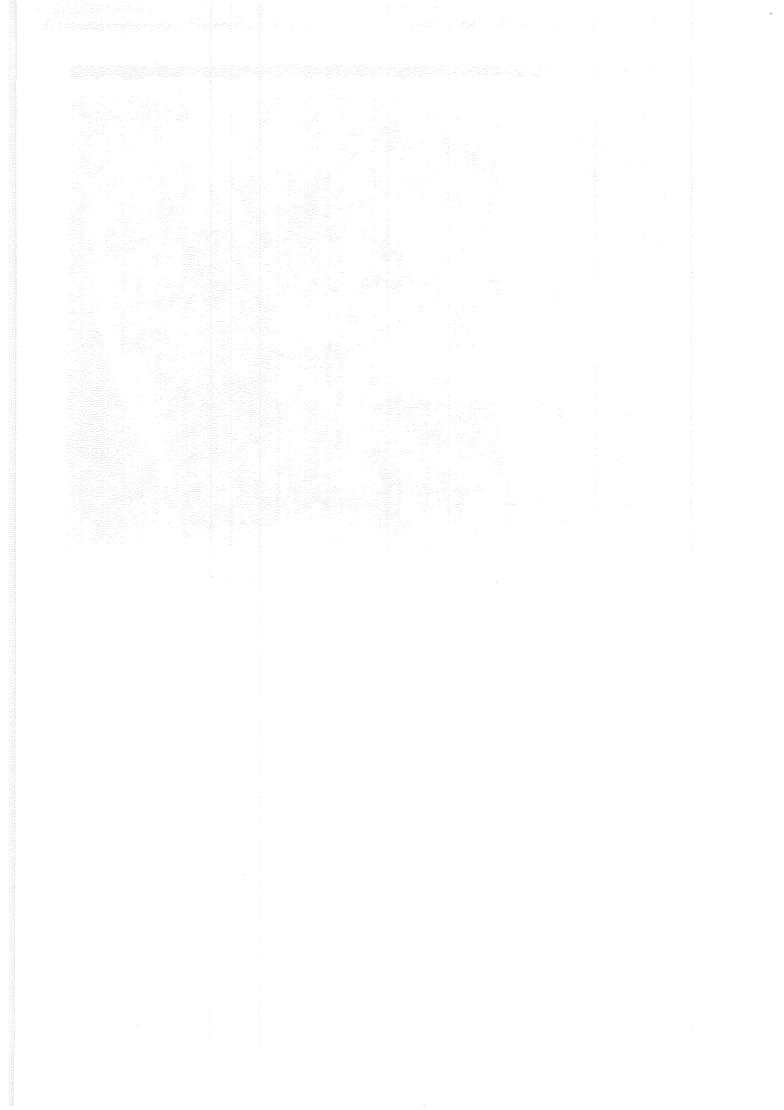
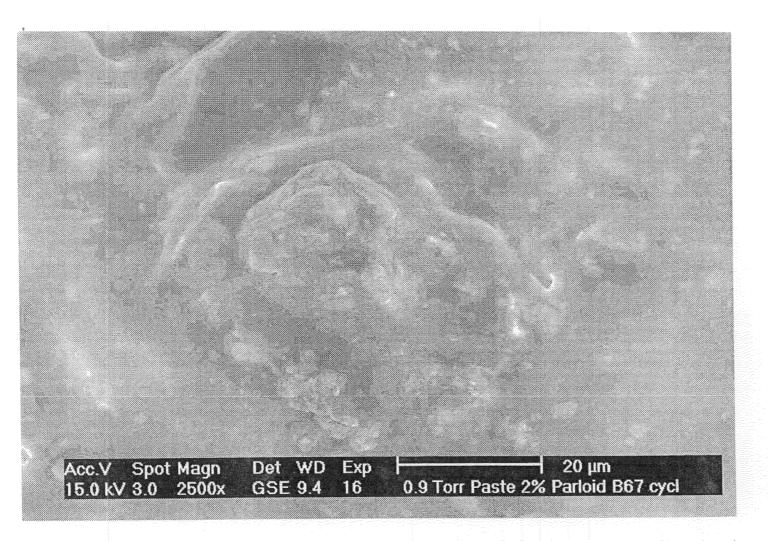


Figure 7 below: The surface of the sample treated with the Paraloid B 67 in cyclohexanone solution at x2500 magnification.





The image in Figure 2 show a much more significant deposit of Paraloid B 67 between the particles when IMS was used as the solvent. Figure 3 shows that the Paraloid B 67 has penetrated the particles thus consolidating them, before starting to form deposits between the particles. The Paraloid B 67 acts as "bridges" between the particles, thereby reinforcing and binding them into a stable structure. In the sample prepared with IMS as solvent, polymer Paraloid B 67 is deposited in larger quantities around the particles, possibly explaining why the IMS treated sample achieved structurally solidity sooner. In the case of the cyclohexanone sample, this could only start once sufficient Paraloid B 67 had been introduced to fully consolidate the particles, thereafter allowing the surplus to form the required "bridges" to hold the particles together. Figure 4 shows a fairly uniform layer of polymer, featuring some pores. This slightly porous "skin" was deposited when IMS was used as solvent, while no such deposits were found in the cyclohexanone sample. The formation of skins such as these is thought feasiblely consistent with the degree of hardness that had been observed.

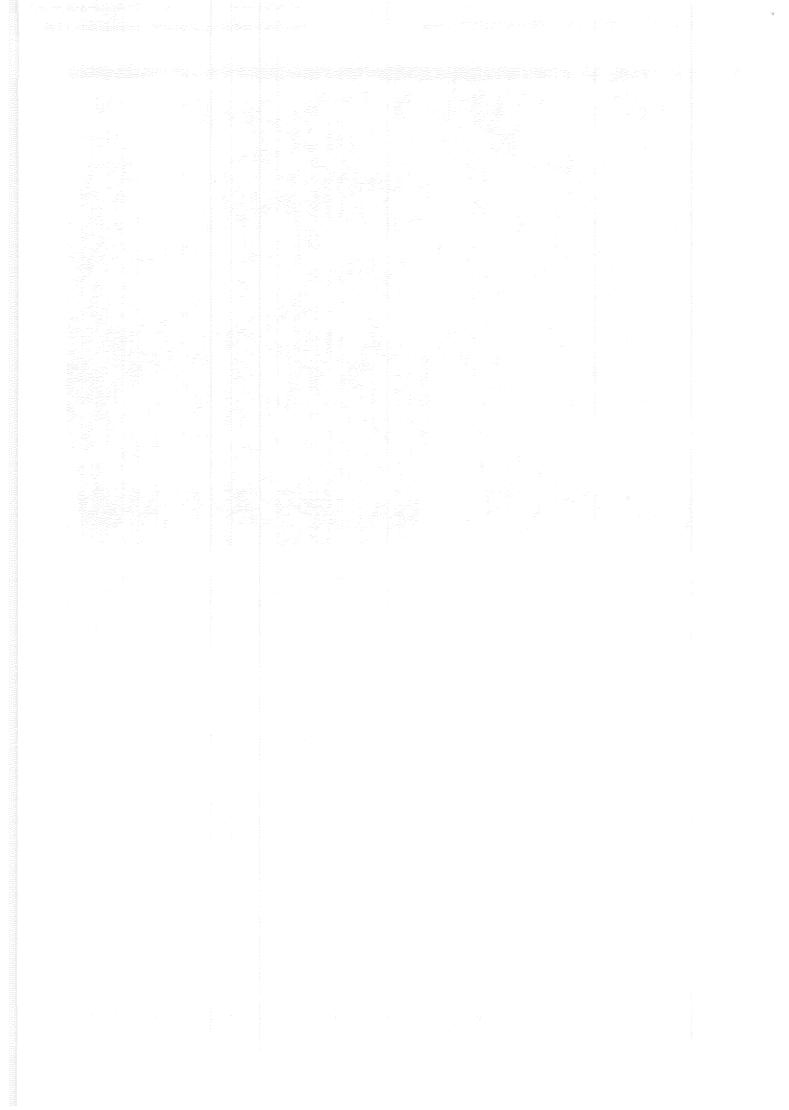
At a larger magnification, Figure 6 shows the Paraloid B 67 in a heavier deposit surrounding the particle whereas Figure 7 shows the Paraloid B 67 forming a skin over the particle rather than adjacent to it, lending further support to the conclusions above.

A tentative conclusion for the dissimilar behaviour of the polymer, Paraloid B 67, in the different solvents, is as follows; [Doménech-Carbó and Auro-Castro, 1999]

"Controlled evaporation: The polymer in the initial solution is dissolved in a mixture of solvent and nonsolvent. As the solvent is more volatile than the nonsolvent, the composition of the polymer solution varies during the evaporation process so that it becomes increasingly richer in polymer and nonsolvent. If this method is performed by exposure of the cast film to the surrounding atmosphere, water sorption from the atmosphere will occur simultaneously with evaporation of the solvent. In this case a film with a thin layer over a thick, more or less porous sub-layer will be obtained."

The IMS used to prepare the Paraloid B 67 contains water and therefore introduces a nonsolvent into the "casting film" - leading to the formation of a porous sub-layer as mentioned above. This would explain the formation of "bridges" between the particles as they are in fact held within the pores of the sub-layer. Figure 4 of the same article [Domenech-Carbo and Auro-Castro, 1999], shows a SEM image of the polymer Ultrason E2010 PES exposed to an atmosphere of 39% RH: Although the casting film in this case does not contain nonsolvent, the topography of this polymer strongly resembles that of Figure 4 featured in this article, indicating that a thin film formed in this treatment was also porous. The assumption is therefore that the presence of water (introduced by the IMS and atmospheric conditions) has been sufficient to cause porosity in the polymer - thereby increasing the overall strength of the slurry as explained.

Of further interest was our field and laboratory observations that a reduced concentration of polymer did not significantly alter the hardness of the end result, corresponding with observations by Doménech-Carbó and Auro-Castro.



"The skin is very thin (<3.6Fm) and has a dense structure. In spite of the different concentrations of both casting solutions the films obtained show a very similar morphology, which would seem to indicate that this factor does not have much influence on the final structure of the films, at least in the interval of concentrations considered here. (Between 15% and 25% concentrations)"

With the sample prepared with cyclohexanone as solvent there was no nonsolvent present, which would have suggested that the porous structure of the polymer will be reduced. There is however a further factor that significantly influences the resulting morphology of the polymer, namely the rate of solvent evaporation. The cyclohexanone has a low boiling point and therefore evaporates very slowly especially in comparison to IMS. [Thomas 1988]

"...The first falling rate period begins when the liquid cannot be supplied to the surface as quickly as the solvent evaporates and it is, therefore, pulled from the centre of the porous object. During this period pressure drops over the pore length and is the driving force that pulls the liquid to the surface..."

also [Doménech-Carbó and Auro-Castro, 1999]

"This behaviour is probably due to the loss of the more volatile ethyl acetate, which leaves behind the lower boiling point solvent DMF, which plasticizes the cellulose nitrate gel as it evaporates, giving rise to a highly dense homogeneous structure."

Both these statements support the behaviour of the Paraloid B 67 in the cyclohexanone where the polymer stays in the individual particles and only when these are saturated does the polymer collect between the particles thereby adhering them together (Figure 3 and 7).

#### CONCLUSION:

Although, overall, the conservation of the furnace was successful, it would have been more suited to use a slurry that yielded a softer, more friable end result that more accurately matched the overall texture and properties of the original furnace material. In this single respect, the polymer used and tested in this project did not satisfy. The other specifications were however all met. On account of the extremely porous nature (and size) of the structure, the risk of unsightly darkening had proven one of the most difficult to manage and surmount, but was nevertheless achieved.

#### ACKNOWLEDGEMENTS:

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#### PRODUCTS AND EQUIPMENT:

Philips XL30 environmental scanning electron microscope (ESEM): University of Natal, Pietermaritzburg Campus, Centre for Electron Microscopy.

Paraloid B 67, solvents, other restoration materials and all tools: The South African Academy of Ceramics Conservation, Joubertina, South Africa

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