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# Firing Experiments to Create Black Ceramics

## An Attempt to Understand the Making of Pottery at the Neolithic Site of Sipplingen B, Osthafen (Lake Constance)

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Project: Mobilities, Entanglements and Transformations in
Neolithic Societies of the Swiss Plateau (3900–3500 BC)

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Gletterens FR, CH. Gisela Thierrin-Michael

# 1. Introduction

In the course of the SNSF-research project „Mobilities, Entanglements and Transformations in Neolithic Societies of the Swiss Plateau (3900 – 3500 BC)“, we repeatedly noticed pottery with black surfaces in Late Neolithic lake settlements of the northern Alpine Foreland  - such as in Sipplingen, Osthafen (Lake Constance, Germany) - which seem to have been fired in reduction. Since no pottery kilns or other firing devices are known from this time until today, we have carried out a small experimental archaeological pilot-study to gain a deeper understanding of the possible firing process. Its preliminary results are presented here.

In January 2018, we performed experimental firings in order to recreate black ceramics, similar to the Neolithic potteries from Sipplingen, Osthafen with materials collected around Sipplingen. Three firing tests were first carried out in electrical kilns in order to test different thermocouples. Several briquettes were then fired at different temperatures in these kilns under laboratory controlled conditions. Finally, we performed an experimental firing in a bonfire in the “Village lacustre” of Gletterens with briquettes of the same composition as above, while recording the temperature evolution inside and outside a pot with thermocouples and an infra-red camera.

# 2. Material

## 2.1. Clay preparation (03-05.01.18)

Material

NLT 266B (D17013): siderolithic clay found in Malm sinkholes (only the yellow part, probably containing less iron, was used), Büttenhardt.

NLT 267 (D17029): clay from a stream in Sipplingen, Süssenmühle; very fine-grained, homogeneous, plastic texture. GPS coordinates:

- Germany: 2 733881E / 5299034N / 423 m
- Switzerland: 726078E / 294301N / 423 m

NLT 268 (D17030): clay from a stream in Sipplingen (Gewerbezone Ost); coarse-grained, spongy-sandy texture, containing organic remains (plant stems, etc.) and gravel. Coordinates:

- Germany: 2 733362E / 5299435N / 425 m
- Switzerland: 725567E / 294712N / 425 m

*Fig. 2.1. a. Starting the preparation of clay NLT 266B; b.  ball of plastic clay 266B; c. clay on a gypsum plate.*

#### 2.1.1 Preparation of clay NLT 266B (03-04.01.18)

The yellow part of dry clay 266B was weighed (922 g) and put into a bucket. 400 ml water were added on top of it to make it malleable (Fig. 2.1a). After one day, the mixture clay-water was too liquid so we put it on a gypsum plate for a few hours to absorb some of the water (Fig. 2.1b). At the end of the day, the clay was plastic enough to form a ball (Fig. 2.1c), stored in a plastic bag (~850 g).

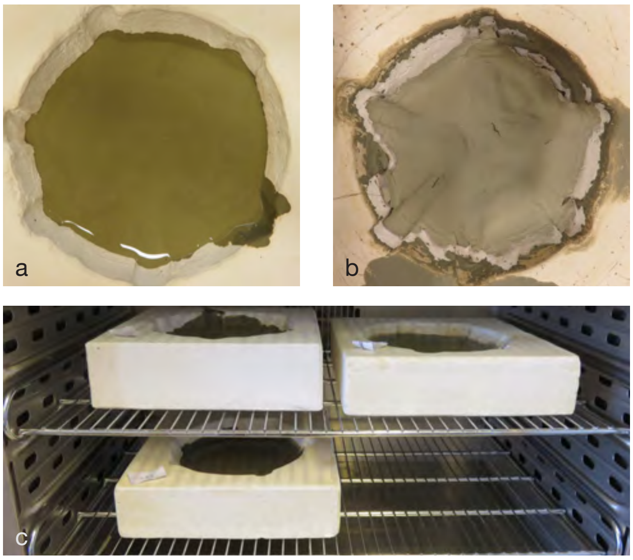



*Fig. 2.2. a. Liquid clay; b. dry clay; c. moulds in the oven.*

#### 2.1.2. Clay-mineral enrichment of clays NLT 267 and 268 (03-05.01.18)

~500 g of clay NLT 267 and ~500 g of clay NLT 268 were weighed. Small fractions of clay were put in a large bowl and covered with water. After a few minutes, when the larger/heavier particles had settled down, the liquid containing lighter particles (clay) was poured into a gypsum mould (Fig. 2.2a) and put into an oven at 60°C for a few hours (Fig. 2.2b) in order to absorb/evaporate the water. What remained was fine-grained clay, which could be detached quite easily from the mould (Fig. 2.2c). However, some white particles, assumed to be gypsum from the mould stuck on the clay. The parts with too much white particles were not kept, as gypsum can make the ceramic shatter during firing (remark: Neolithic sherds from lake dwellings may also contain gypsum, due to a secondary formation during the burial in anoxic environment). The clay was then modelled into a ball and stored in a plastic bag to prevent drying.

This clay-mineral enriched part of clay NLT 267 (“NLT 267 T”) will be used as barbotine for pot “267 G FB”, whereas NLT 268 T will be used to form briquettes.

### 2.2. Determining the water content of clays NLT 267 and NLT 268 (12-19.01.18)

Small balls of untreated clays NLT 267 and NLT 268 were prepared by spraying some water or letting them dry in order to obtain a good plasticity. They were weighed, then put in an oven at 100°C for three days (12-15.01.18) and weighed again. The water content required for a good plasticity was calculated from it. However, the plastic stage of clay contains a whole range of water contents, thus the values indicated below (Table 2.1) only represent a rough estimate of the plasticity index. After four more days in the oven at 100°C, the balls were weighed again and showed the same weights.

| Clay | Wet | Dry | wt% water |
|---|---|---|---|
| NLT 267 | 107.8 g | 76.3 g | 29.2 % |
| NLT 268 | 89.4 g | 64.4 g | 28.0 % |

*Table 2.1. Estimation of plasticity indexes (wt% water) for clays NLT 267 and NLT 268.*

### 2.3. Grog preparation (temper) (04-11.01.18)

#### 2.3.1. Preparation of briquettes for grog

Five briquettes (4.1 x 8.1 x 1.2 cm) were made from clay NLT 266B (too liquid, not plastic) and five others from clay NLT 268 (Fig. 2.3a). After one day in the open air, they were dry enough to be removed from their moulds (3 briquettes made with NLT 266B cracked while drying, possibly because the clay was too liquid when moulded and too many large pores formed during drying) and were placed on their smallest side for a few days to dry further (Fig. 2.3b).

#### 2.3.2. Firing and crushing of the briquettes

After one week drying at room temperature, the briquettes were fired in a Nabertherm N11/HR electrical kiln (hereafter: N11) at 550°C (10-11.01.18; Fig. 2.4), then ground (11.01.18) with a mortar and sieved to keep the fraction smaller than 2 mm (Fig. 2.5).

Program for the electrical kiln (N11):

- T1: 550°C  t1: 165’
- T2: 550°C  t2: 60’  (soaking time)

### 2.4. Rock preparation (temper) (10-12.01.18)

A gneiss boulder collected in Sipplingen was fired in a Nabertherm Ceramotherm N100 electrical kiln at 850°C (hereafter: N100; 10-11.01.18) together with the thermocoupled briquette NLT 267 (see section 2.5.2). During the cooling phase, the boulder was taken out of the kiln at ~400°C and put into a metal bucket filled with cold water (Fig. 2.6a) in order to cause a thermal shock. This process made the crushing of the boulder easier. When the boulder temperature was low enough to be touched, a hammer was used to break the boulder (Fig. 2.6b), and the fragments obtained were then crushed in a metal mortar (Fig. 2.6c). It was not sieved, so some large particles may remain.

Program for the electrical kiln (N100):

- T1: 850°C  t1: 255’
- T2: 850°C  t2: 60’  (soaking time)

*Fig. 2.3. a. Briquettes of clay NLT 268 and NLT 266B; b. briquettes drying on their smallest side.*

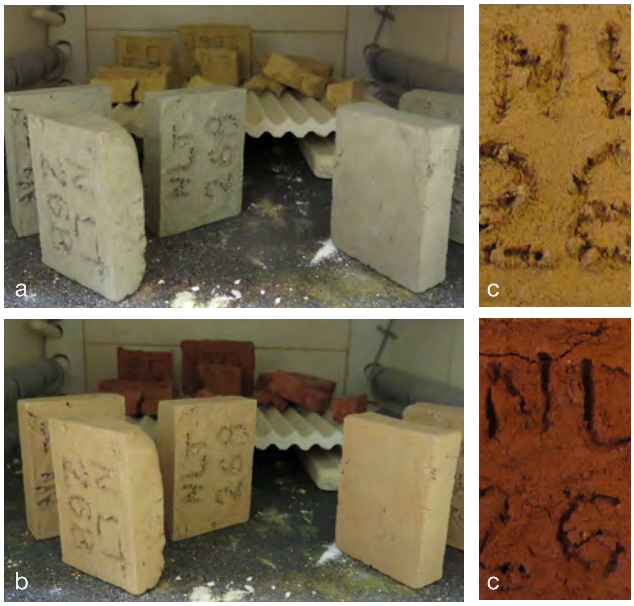


*Fig. 2.4. Briquettes in the oven before (a) and after (b) firing; c. fired briquettes NLT 268 and 266B.*

*Fig. 2.5. a. Crushing of briquettes NLT 266B; b. fraction < 2 mm of briquettes NLT 266B.*


*Fig. 2.6. a. Boulder in water; b. breaking of the boulder; c. crushing of the boulder.*

# 3. Thermocouples testing (04-19.01.18)

## 3.1. Material

Three firing tests were carried out in two electrical kilns fired at different temperatures in order to test and compare the different types of thermocouples before recording temperatures in the field. For this purpose, three different types of thermocouples (TC) were used:

Type K thermocouples:

Four TC (type K: Ni/NiCr) connected to a multi-way switch were used (Fig. 3.1), to record the evolution of firing temperatures. This type of TC can record temperatures as high as 1300°C. Each type K TC consists of a positive nickel-chromium wire and a negative nickel wire, which are placed in the multi-way switch. The multi-way switch was connected to a computer by USB port. Christoph Neururer prepared the same computer program as used for the 2004-2008 bonfire experiments (Maggetti et al. 2011) and extended two of the wires, in order to be able to fire two objects in two different kilns/bonfires at the same time (all the recordings were finally done in one kiln/bonfire at a time). For the firing tests in the lab, 4 different TC were prepared: one with soldered wires (Fig. 3.1b) and three with a golden bead (Fig. 3.1c). For the experimental firing in Gletterens, a Cu-shell was set at both ends of each wire, as described below:

Repairing a TC after burning:

1. Cut the burnt part of the TC and remove the protection of each wire by scratching with a knife on a length of ~2 cm.
2. Twist the two wires together on ~5 mm from the base (out of the protection). The wires should not touch each other outside the twist. Cut the part that is not twisted.
3. Crimp a Cu-lug in order to cover the whole twist.
4. Also crimp a Cu-lug at the other end of the thermocouples (the one that enters the multi-way switch).

Type S thermocouples in electrical kilns:

Two electrical kilns equipped with type S TC were used to fire several briquettes. Type S TC consists of a positive wire of 90% platinum and 10% rhodium used with a negative wire of pure platinum. They are highly resistant to oxidation and corrosion, but can be contaminated by H, C and many metal vapours. They can record temperatures as high as 1600°C. Kilns used were N11 and N100.

Fluke® Thermocouple Thermometer 54 II B:

A thermometer equipped with a type K TC was also used. It did not save temperatures, thus we noted readings regularly by hand on paper.

## 3.2. Firing tests

### 3.2.1. First test: firing of briquette NLT 267 in kiln N100 at 850°C (05-11.01.18)

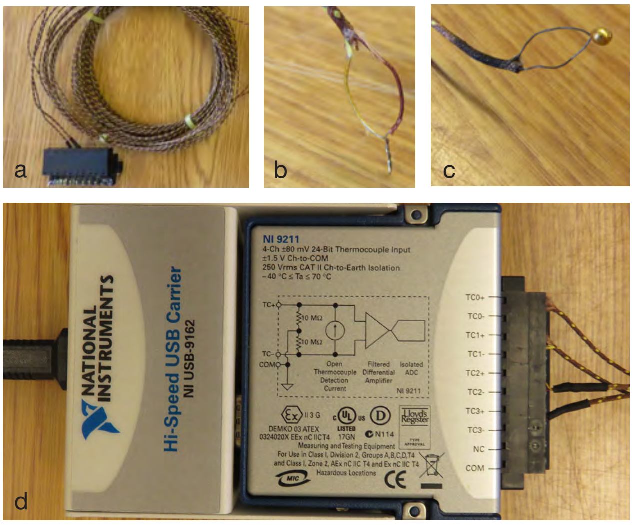



*Fig. 3.1. a. Type K TC; b. TC with soldered wires; c. TC with golden bead; d. multi-way switch.*

A briquette of clay NLT 267 was formed (untreated clay; 6.5 x 16.1 x 2 cm; 05.01.18; Fig. 3.2). The type K TC with only soldered wire was inserted inside the briquette, whereas a type K TC with a golden bead was inserted through it, so that the metal lay on top of the clay.

The briquette dried for ~1 week and was then fired at 850°C in the larger electrical kiln (N100; together with the gneiss boulder; 10-11.01.18), while recording the temperatures every 15 s. The computer program was stopped on 12.01.18.

Program for the electrical kiln (N100):

- T1: 850°C  t1: 255’
- T2: 850°C  t2: 60’  (soaking time)

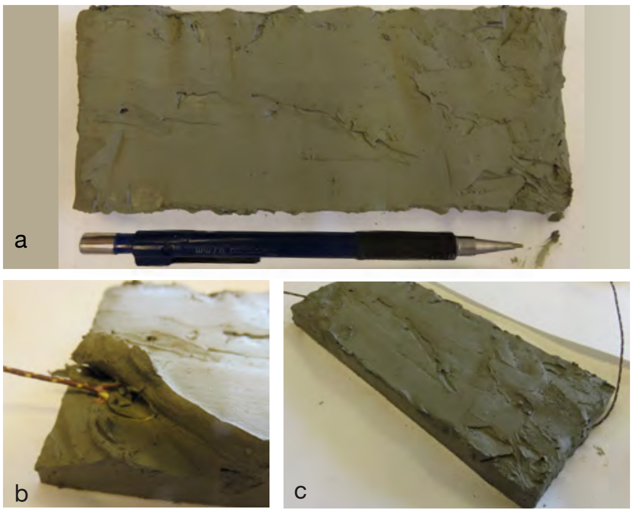



*Fig. 3.2. a. Briquette of clay NLT 267; b. inserting the TC inside the briquette; c. briquette with TC.*

#### 3.2.2. Second test: firing of briquette NLT 266B in kiln N100 at 550°C (05-15.01.18)

A briquette was made from clay NLT 266B (4.1 x 8.1 x 1.2 cm; 05.01.18; Fig. 3.3). A type K TC with a golden bead was placed inside it, and another on top of it. It dried for ~2 week and was fired at 550°C in the electrical kiln N100 (15-16.01.18) with other briquettes ("NLT 267 C" and "NLT 268"). This thermocoupled briquette 266B was then crushed to produce more grog.

Program for the electrical kiln (N100):

- T1: 550°C t1: 165'
- T2: 550°C t2: 60' (soaking time)

#### 3.2.3. Third test: comparison of different temperature recordings in kiln N11 (19.01.18)

Three TC (type K; two with golden beads and one with soldered wires) linked to a computer were put in electrical kiln N11, in the zone were the briquettes had been fired. As they had not been repaired after previous firing in briquettes (firing tests 1 and 2), they could be damaged and thus not work accurately. A Fluke® Thermocouple Thermometer 54 II B (type K TC) was put in the kiln as well, close to the three TC (Fig. 3.4). The kiln was fired at 750°C with a 10 min-soaking time.

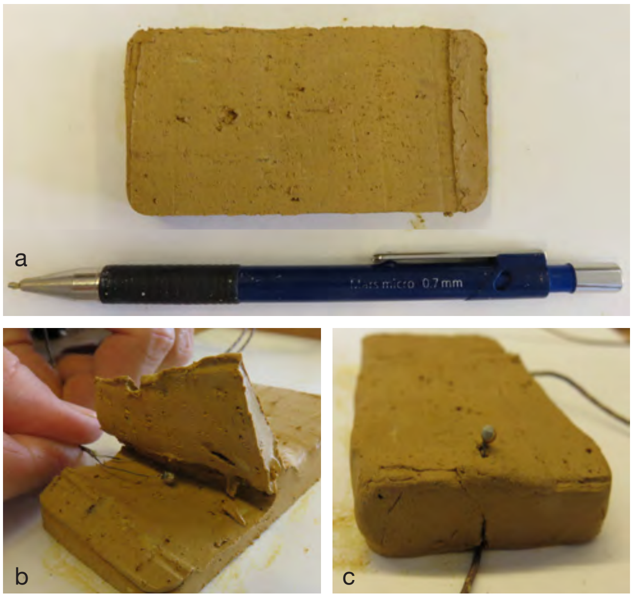



*Fig. 3.3. a. Briquette of clay NLT 266B; b. inserting the TC inside the briquette; c. briquette with TC.*

Program for the electrical kiln (N11):

- T1: 750°C t1: 225'
- T2: 750°C t2: 10' (soaking time)

### 3.3. Results of firing tests

#### 3.3.1. Firing tests 1 and 2: firing of briquettes with thermocouples in kiln N100

For both tests 1 and 2, the temperatures recorded by type K thermocouples show a shift of +40°C compared to the temperature indicated on the electrical kiln (Fig. 3.5 and Fig. 3.7). Indeed, instead of measuring 850°C resp. 550°C during the soaking time, as programmed, the TC recorded temperatures up to 890°C resp. 590°C. We suggest that the temperature in the kilns is not uniformly distributed, so

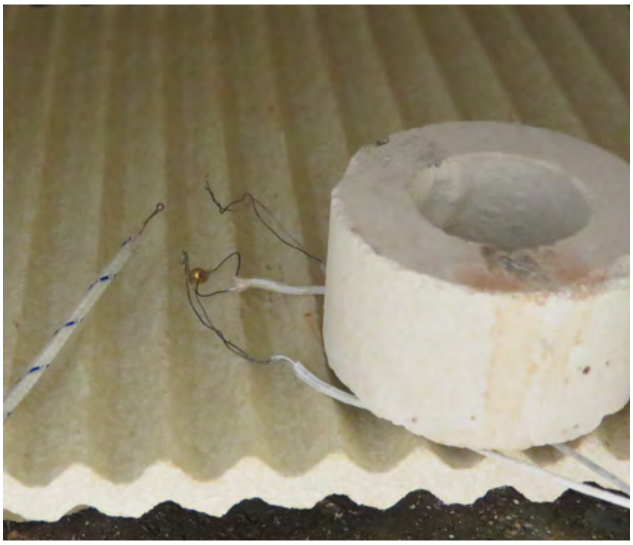


*Fig. 3.4. Location of the thermocouples in electrical kiln N11.*

that the TC of the kiln registered other temperatures than the mobile TC, which were placed at the extreme opposite from the TC of the kiln.

There is also a shift between the temperatures recorded by the TC on the briquette surface (in fact the air at the surface) and the TC in the briquette body, as can be seen in Fig. 3.6 and Fig. 3.8. The air at the briquette surface reacts much more quickly to temperature changes, particularly visible during the soaking time, where the oscillations correspond to the pulses produced by the heater of the kiln. On the other hand, the briquette body has more heat capacity than air, thus its temperature increases after that of the briquette surface, and remains high longer than on the briquette surface. Maximal temperature of briquette body was approximately 5°C less than that of briquette surface, for firing test 1. No substantial difference between the temperature of briquette body and surface was observed for firing test 2, possibly due to the location of the thermocouples.

The temperature drop at the brick surface around 09:20 was caused by the opening of the kiln to remove the gneiss boulder. This was however too short to significantly affect the TC in the brick body.

### 3.3.2. Firing test 3: comparison of different temperature recordings in kiln N11

Firing test 3 (Fig. 3.9) shows that the TC1, TC2 and the thermometer, all placed at the same location in the kiln, have temperature variations of only ± 5°C, suggesting a good reliability of these three thermocouples. TC4 did not function very well, especially before 02:18:00 and after 04:50:00, probably due to damage related to its former use in briquettes. Temperature indicated on the kiln is ~40°C lower than TC1, TC2 and thermometer, which is consistent with the results from firing tests 1 and 2.

Remarks: due to manipulation errors, temperature recording of TC1, TC2 and TC4 was started after 1 hour firing, whereas temperatures from the thermometer were correctly recorded only after 02:54:00. The thermocouples were abruptly removed from the oven after ~5 hours firing, as seen by the strong drop of temperature in Fig. 3.9 (except for non-working TC4).

### 3.3.3. Remarks about the three firing tests

The three firing tests performed in the laboratory suggest that the type K thermocouples (including the thermometer) are reliable, or at least that all these type K thermocouples are consistent with each other. As they record different temperatures than the electrical kilns (type S thermocouples), further analyses would be needed to ascertain the accuracy of these type K and S thermocouples. Such analyses could consist of firing a material that transforms into another phase at a given temperature, followed with XRD analyses to verify if this material has transformed or not (e.g. Maggetti 1982; Maggetti et al. 2011).

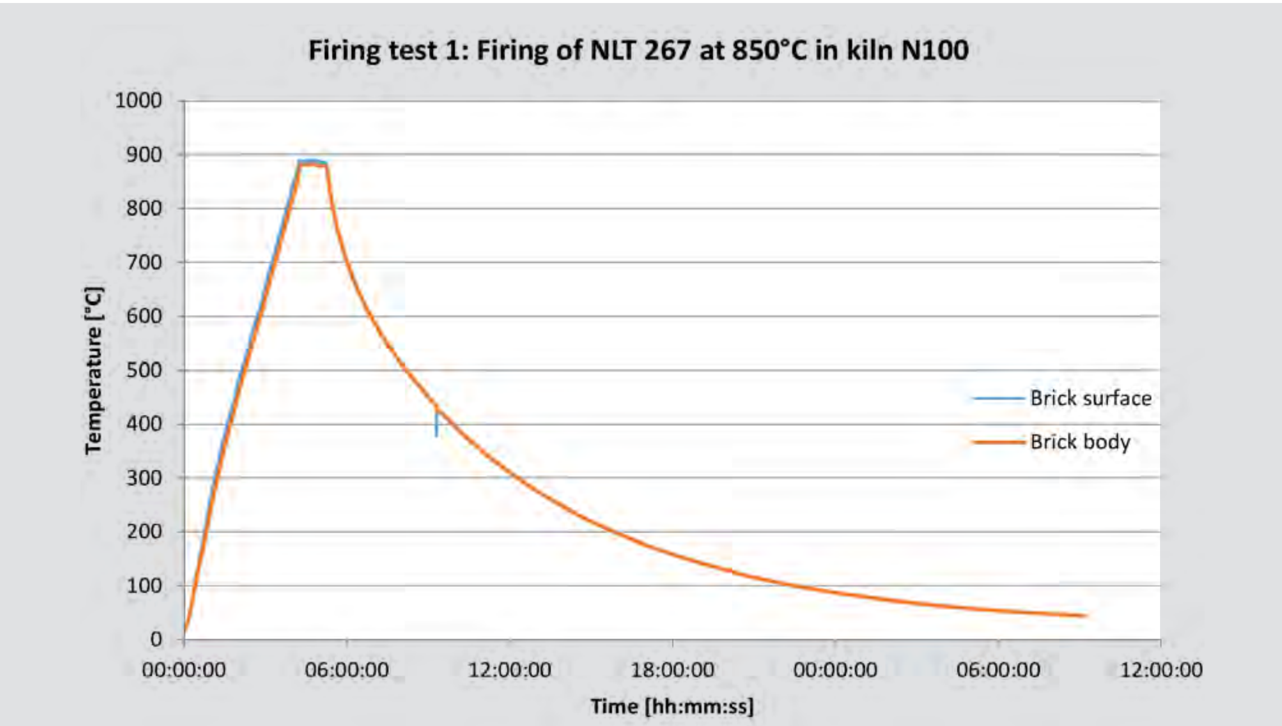



*Fig. 3.5. Firing test 1: temperature evolution recorded by two type K thermocouples.*

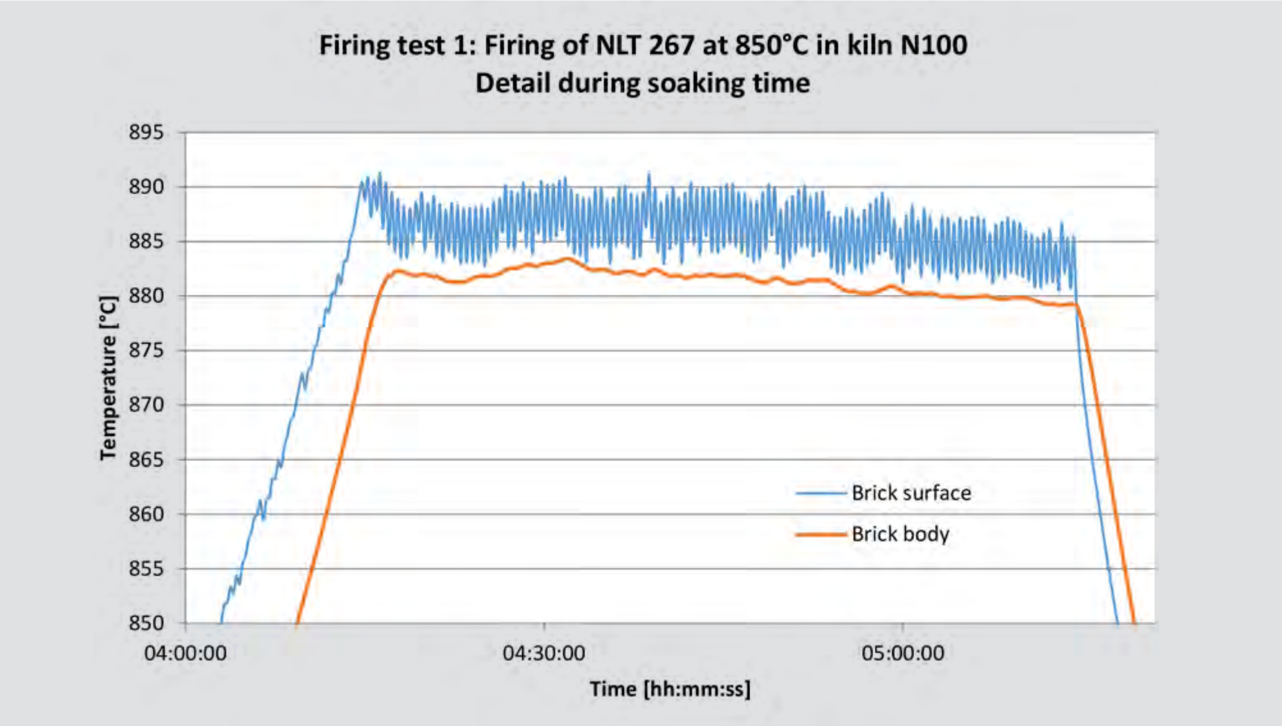



*Fig. 3.6. Firing test 1: detail of the temperature evolution during soaking time.*

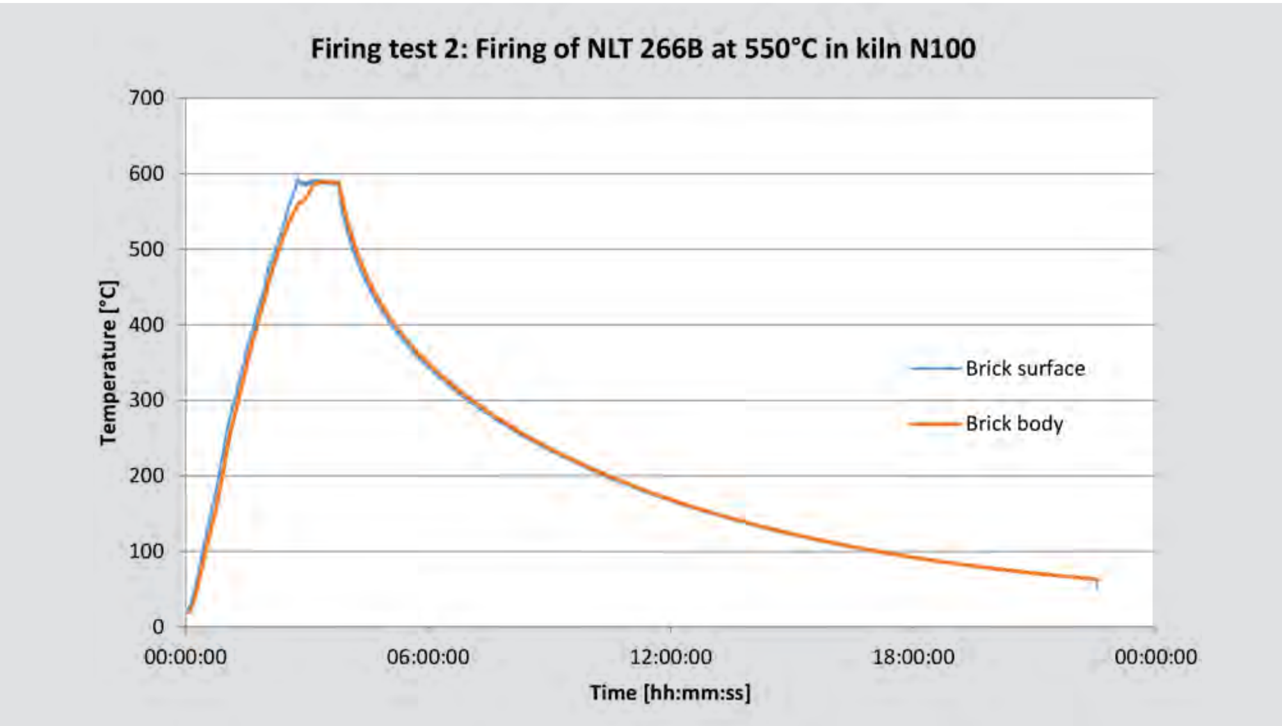



*Fig. 3.7. Firing test 2: temperature evolution recorded by two type K thermocouples.*

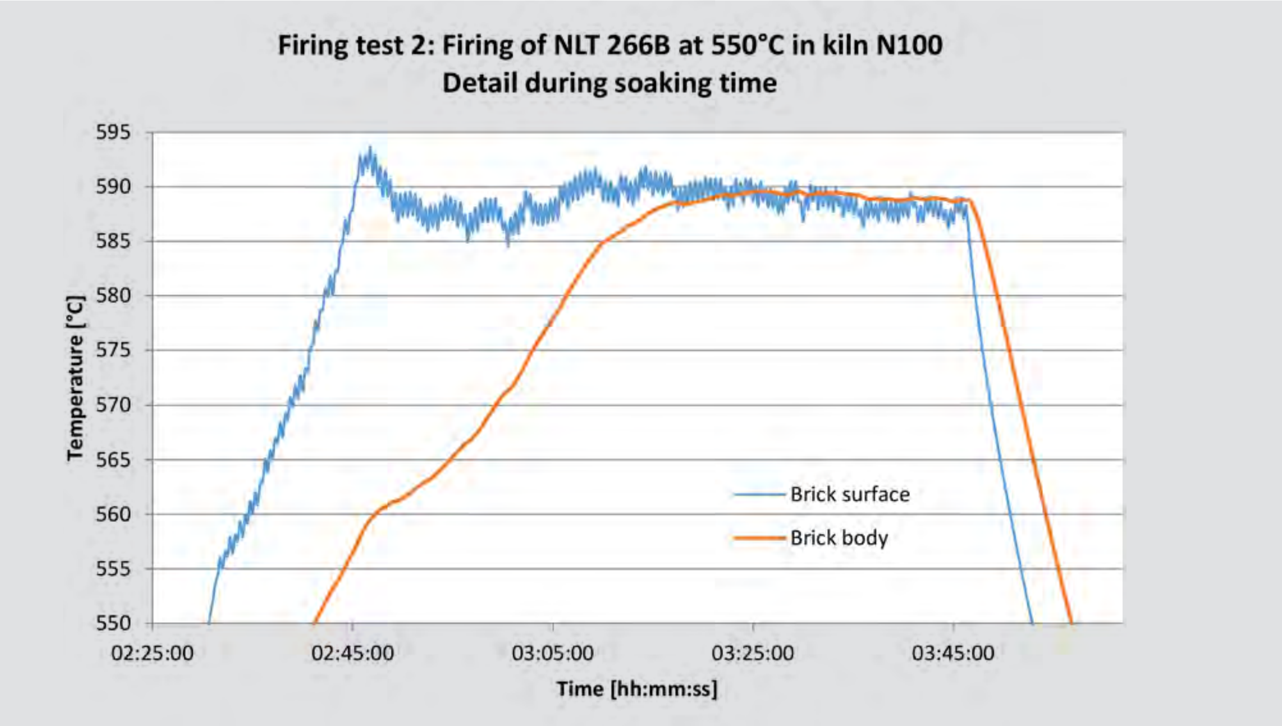



*Fig. 3.8. Firing test 2: detail of the temperature evolution during soaking time.*

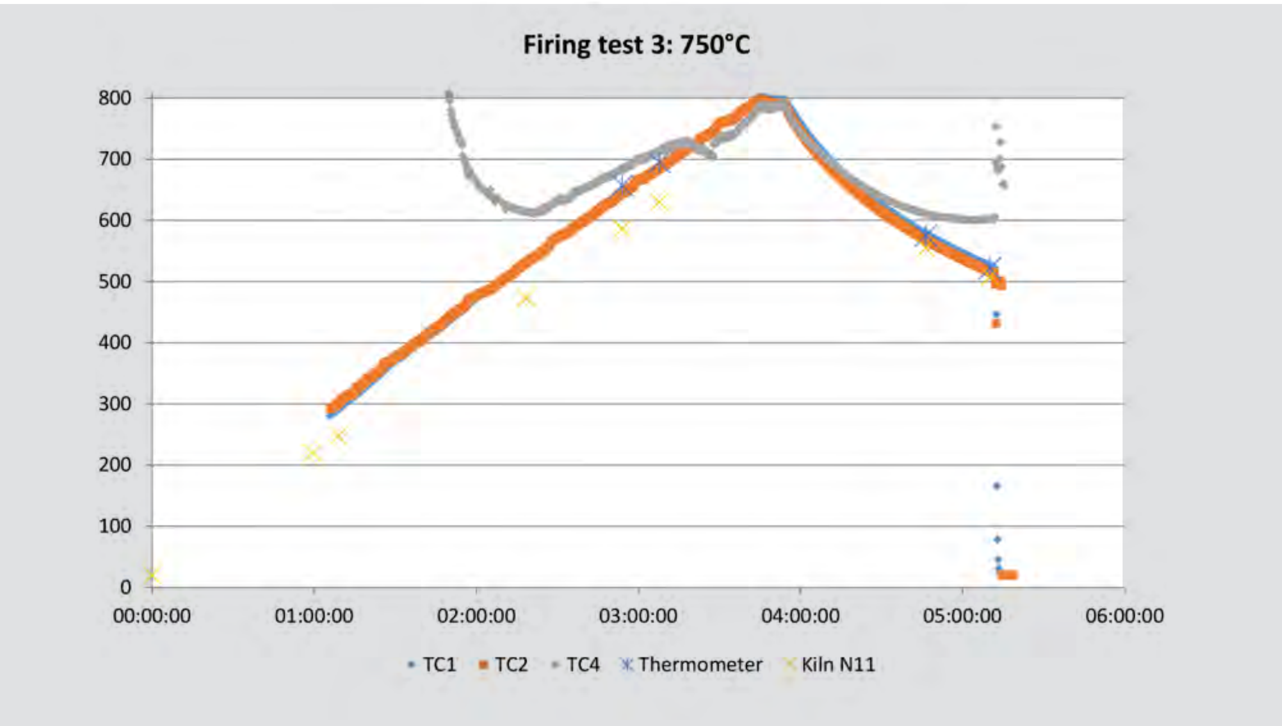



*Fig. 3.9. Temperature evolution recorded by three type K thermocouples, a thermometer with a type K thermocouple and electrical kiln N11 with a type S thermocouple.*

## 4. Firing experiment of briquettes and pots (10-26.01.18)

### 4.1. Preparation of briquettes and pots

Briquettes (4.1 x 8.1 x 1.2 cm) were formed from clays NLT 267, NLT 268, NLT 268 T (treated) and clay from Guin, adding different tempers (10 wt % of grog, 10 wt % of calcite, or 15 wt % of crushed rock) as indicated in Table 4.1 (“crushed rock” was more easily incorporated than calcite and grog, thus we added 15 wt% of it). Several briquettes of each clay-temper mixture were prepared in order to fire them at different temperatures (Table 4.1). As we only had a small quantity of clay NLT 268 T, only four half briquettes were made from this clay. Four pots were also made from clay NLT 267 and grog or crushed rock (two pots with each temper).

A surface treatment was applied to the objects that were to be fired in bonfires. The briquettes and pots for the bonfire with capsule (FBK, *Feldbrand mit Kapsel*) were first smoothed with a wet finger, then dried for a few hours until they got a leathery texture, and finally polished (back side of briquettes and outside and rim of half pots; Fig. 4.1a-b) with a fine grained-pebble. The pots for the bonfire without capsule (FB, *Feldbrand)* were not polished, but covered with barbotine of clay NLT 267 (pot NLT 268 R; Fig. 4.1.c) resp. NLT 267 T (pot NLT 268 G).

*Fig. 4.1. a. Pot 267 G FBK with polished part (left side) and smoothed part (right side); b. Example of the polished surface of briquette NLT 267 R FBK (note: the upper left edge could not be polished, because too dry); c. Pot 267 R FB with barbotine NLT 267 (untreated).*

| | Temper | Firing type | Sample name | Polished | Sawn | XRD | Thin sections | SEM |
|---|---|---|---|---|---|---|---|---|
| Briquettes Untreated clay NLT 267 | - | FB | 267 FB | x | x | x | x | |
| | G 266B | 650 | 267 G 650°C | | x | x | x | |
| | G 266B | 850 | 267 G 850°C | | x | x | x | |
| | G 266B | FB | 267 G FB | x | x | x | | |
| | G 266B | FBK | 267 G FBK | x | | x | | |
| | C | 550 | 267 C 550°C | | x | | | |
| | C | 650 | 267 C 650°C | | x | x | x | |
| | C | 750 | 267 C 750°C | | | | | |
| | C | 850 | 267 C 850°C | | | | | |
| | C | FB | 267 C FB | x | x | x | | |
| | C | FBK | 267 C FBK | x | | x | | |
| | R | 650 | 267 R 650°C | | x | x | x | |
| | R | 850 | 267 R 850°C | | x | x | x | |
| | R | FB | 267 R FB | x | x | x | | |
| | R | FBK | 267 R FBK | x | x | x | x | |
| Pots Untreated clay 267 | G 266B | FB | pot 267 G FB | x | | x | | |
| | G 266B | FBK | pot 267 G FBK | x | x | x | x | x |
| | C | FB | pot 267 C FB | | x | x | x | |
| | C | FBK | pot 267 C FBK | | x | x | x | |
| | R | FB | pot 267 R FB | x | | x | | |
| | R | FBK | pot 267 R FBK | x | x | x | x | x |
| Briquettes Untreated clay NLT 268 | - | 550 | 268 550°C | | x | | | |
| | - | 650 | 268 650°C | | x | | | |
| | - | 750 | 268 750°C | | | | | |
| | - | FB | 268 FB | x | | | | |
| | - | FBK | 268 FBK | x | | | | |
| | G 266B | 650 | 268 G 650°C | | x | | | |
| | G 266B | 750 | 268 G 750°C | | | | | |
| | G 266B | FB | 268 G FB | x | | | | |
| | G 266B | FBK | 268 G FBK | x | | | | |
| | C | FB | 268 C FB | x | | | | |
| | C | FBK | 268 C FBK | x | | | | |
| | R | FB | 268 R FB | x | | | | |
| | R | FBK | 268 R FBK | x | | | | |
| Briquettes Clay -rich part of NLT 268 | G 266B | FB | 268 T G FB | x | x | | | |
| | G 266B | FBK | 268 T G FBK | x | | | | |
| | R | FB | 268 T R FB | x | x | | | |
| | R | FBK | 268 T R FBK | x | | | | |

*Table 4.1. Summary table indicating sample preparation types and analyses.*

All objects were first dried for ~1 day on a gypsum mould and thereafter put in an oven at 100°C for three days. After drying for 3 days in an oven at 100°C, the briquettes were fired either in electrical kilns at given temperatures or in two bonfires (with and without capsule) in Gletterens.

Two more pots were made from clay NLT 267 and calcite, one for each type of bonfire. Thermocouples were inserted in the body and on the surface of pot "267 C FBK". No surface treatment was applied for these pots, in order to leave the thermocouples undisturbed. Pot "267 C FB" dried for 3 days at 60°C, while pot "267 C FBK" dried for ~24h at 50°C.

### 4.2. Programs for the electrical kilns

**Firing at 950°C (N11; from 23.01.18, 15h25 to 24.01.18, 8h30 at ~200°C)**

- T1: 600°C t1: 240'
- T2: 600°C t2: 30' (pause for Quartz transformation)
- T3: 950°C t3: 105'
- T4: 950°C t4: 60' (soaking time)

**Firing at 850°C (N100; from 23.01.18, 15h25 to 24.01.18, 8h45 at ~170°C)**

- T1: 600°C t1: 240'
- T2: 600°C t2: 30' (pause for Quartz transformation)
- T3: 850°C t3: 75'
- T4: 850°C t4: 60' (soaking time)

**Firing at 750°C (N11; from 24.01.18, 11h to 25.01.18)**

- T1: 600°C t1: 240'
- T2: 600°C t2: 30' (pause for Quartz transformation)
- T3: 750°C t3: 45'
- T4: 750°C t4: 60' (soaking time)

**Firing at 650°C (N100; from 24.01.18, 11h to 25.01.18)**

- T1: 600°C t1: 240'
- T2: 600°C t2: 30' (pause for Quartz transformation)
- T3: 650°C t3: 15'
- T4: 650°C t4: 60' (soaking time)

### 4.3. Firing experiments in Gletterens (FR) (26.01.18)

At around 8h30, two fires were started in Gletterens: one outside (FB) and the other under a roof (FBK). Fireplaces were quite wet. Pinewood was used to start the fires, then straw, dead leaves and some logs from a mixture of wood (beech, maple, oak and ash) were added. The fires were then let to die, to produce embers.

#### 4.3.1. Bonfire without capsule (FB, *Feldbrand*)

In fireplace FB (Fig. 4.2a), briquettes and pots (most of them face up) were directly placed on the embers (Fig. 4.2b) and covered with branches of pine. Temperatures were manually recorded, using a thermocouple linked to a thermometer (Fig. 4.2c). Fire was restarted at 11h10 (Fig. 4.2d), under a light rain. At 11:50, fire was extinguished by putting sand on it (Fig. 4.2e). (In order to have good reducing conditions, we should have put straw before sand, so as to provide fuel.)

#### 4.3.2. Bonfire with capsule (FBK, *Feldbrand mit Kapsel*)

In fireplace FBK, embers were covered with wood (mixture of beech, maple, oak and ash) and straw, on which briquettes and pots (including the pot connected to two thermocouples) were placed and covered with sawdust (Fig. 4.3a). Pots were placed upside-down. A third thermocouple was placed on top of the straw. Newspapers were placed all around it (Fig. 4.3b). The whole was covered with a capsule (40 cm-diameter earthenware flowerpot) at 10h37 (Fig. 4.3c). A fourth thermocouple was placed on top of the capsule and stabilised with a pebble. Another pebble was used to close the hole on the capsule (Fig. 4.3c). Beech wood and newspapers were placed around the capsule (Fig. 4.3d). Fire was restarted at 10h45 (Fig 4.3e-f), although recording of temperatures started at 10h35 with the thermocouples and a FLIR T450sc infrared camera (Fig. 4.3g). When the fuel was entirely consumed, we let the capsule remain above the ceramic objects for some time during the cooling phase (Fig. 4.3h), and then removed it (Fig. 4.3i). Temperatures recording stopped at 13h11 for the TC and around 12h35 for the infrared camera (total: 2h36 of recording with TC and 2h with infrared camera).

Fig. 4.2. a. Fireplace FB; b. placing of objects; c. temperature recording with thermometer; d. firing; e. fire extinction with sand.

Fig. 4.3. a. Placing of objects; b. putting newspaper; c. covering with an earthenware flowerpot and wood; d. restarting of the fire; e. firing; f. fireplace FBK; g. temperature recording with infrared camera; h. cooling phase; i. fired objects.

## 4.4. Temperatures recorded in the field

### 4.4.1. Bonfire without capsule (FB)

For the bonfire without capsule, temperatures were manually recorded with a Fluke® Thermocouple Thermometer 54 II B (Table 4.2 and Fig. 4.4). For the first 40 minutes, no fuel was added to the fire. Then, a fire was restarted on the embers, as observed with the peak at 650°C. After 1h, temperatures reached 950°C. In this type of fire, temperatures cannot be controlled and thus strongly fluctuate.

| time | T [°C] | |
|---|---|---|
| 00:00:00 | 47 | |
| 00:20:00 | 130 | |
| 00:30:00 | 200 | |
| 00:40:00 | 310 | |
| 00:40:30 | 650* | * Restarting of fire |
| 00:45:00 | 575 | |
| 00:53:00 | 850 | |
| 01:00:00 | 930 | |
| 01:07:00 | 950 | |
| 01:13:00 | 800** | ** Removing of logs (put into FBK) |
| 01:22:00 | 223 | |

*Table 4.2. Temperature evolution in bonfire without capsule.*

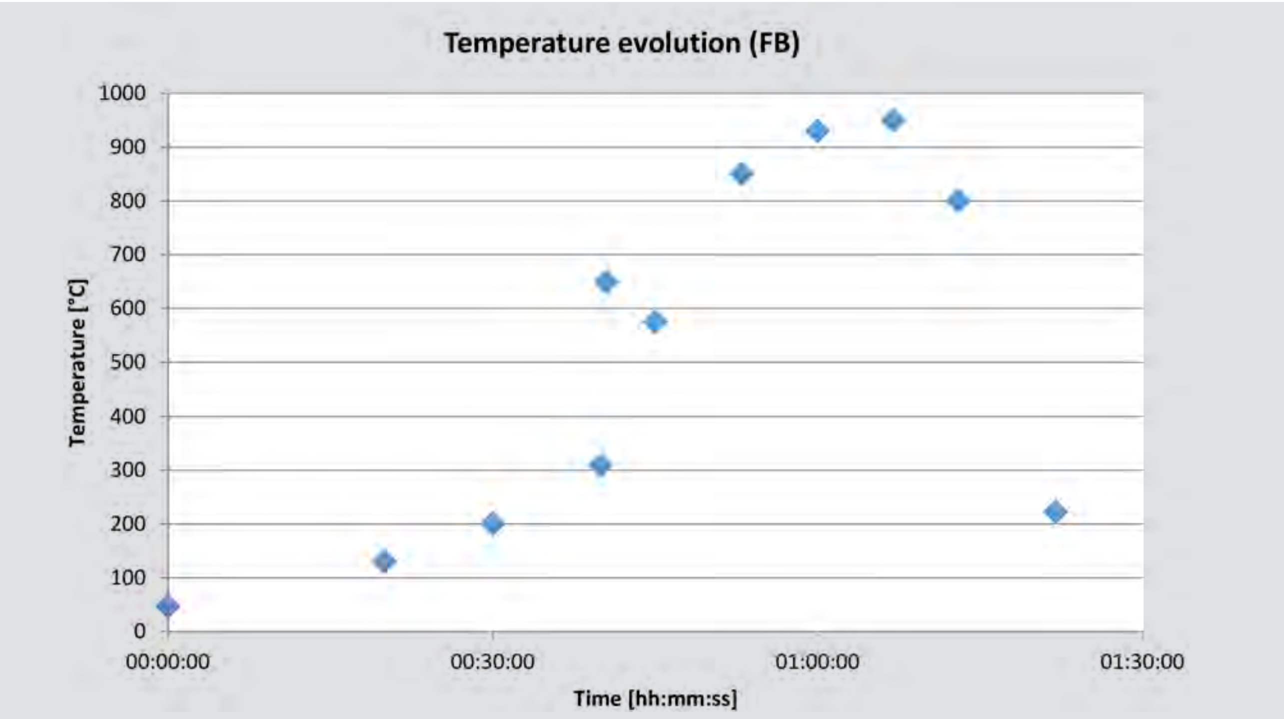



*Fig. 4.4. Temperature evolution in bonfire without capsule, as recorded with a thermometer (type K thermocouple).*

### 4.4.2. Bonfire with capsule (FBK)

The following graphs (Fig. 4.5) show the temperature evolution recorded during the firing experiment with the four type K thermocouples, from 10h35 to 13h10. The fire was started at 10h45 (00:10:00), which can be observed by the slow temperature increase in- and outside the capsule, and especially by the temperature jump from ~16°C to 225°C resp. 47°C in the pot body resp. the pot surface. Around 00:40:00, logs were added to the fire, causing a strong and rapid temperature rise.

During the temperature rise, pot body was hotter than pot surface. Only after 01:30:00, shortly before the cooling phase, did the situation reverse, with cooler pot body than pot surface. This situation is opposite to the results from the firing tests in the electrical kiln, possibly because the thermocouple on the pot surface was the furthest away from the fire, as the pot was placed upside-down, while the pot body, only 5 mm-thick, was in almost direct contact with the temperature inside the capsule.

It seems that the pot (body and surface) has a stronger heat capacity than the inside of the capsule, as it reacts much more slowly to temperature rise and decrease.

Maximal pot temperatures reached 600°C for 15-20 minutes. The inside of the capsule never reached temperatures higher than 660°C, whereas the outside (in direct contact with the fire) went as high as 830°C with strong fluctuations, especially when logs were added or moved. The capsule enabled temperatures to remain quite regular with almost no oscillations.

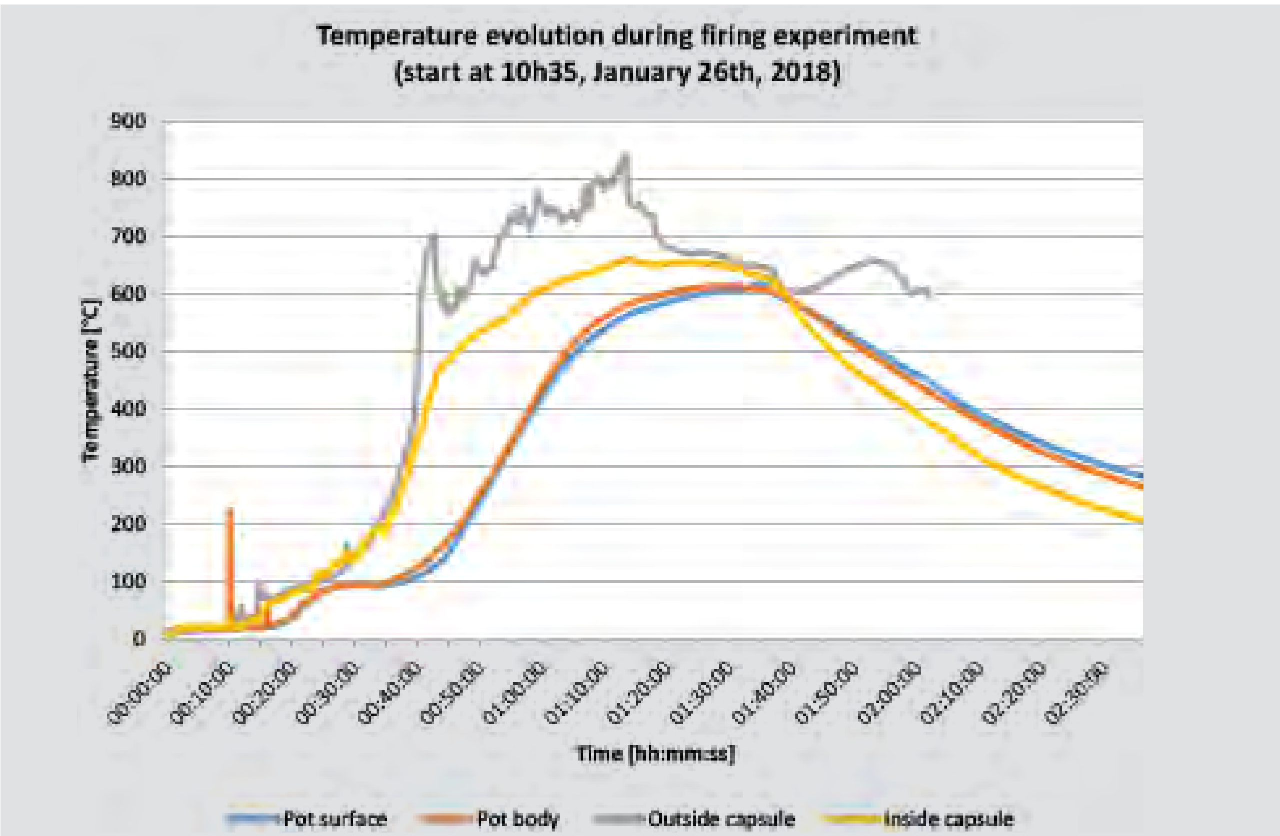



*Fig. 4.5. Temperature evolution in bonfire with capsule (FBK), as recorded with four type K thermocouples.*

#### 4.4.3. FLIR T450sc infrared camera

Temperature recording with the FLIR T450sc infrared camera started at 10h35, for ~2h15. Figure 4.6 shows infrared images taken at different times. Maximal fire temperature was about 900°C, so slightly above the temperatures recorded with thermocouples outside the capsule. After ~1h40 of temperature recording, a pan was put in the fire to boil water, moving some of the logs.

We believe that the combination of temperature recordings with type K thermocouples and with a FLIR T450sc infrared camera is a good way to obtain a more precise idea of the temperature evolution within a bonfire. Indeed, these two methods are complementary, as the thermocouples recorded the temperature of the pot, inside the capsule and outside of the capsule, whereas the infrared camera filmed the temperatures of the fire. We observe relatively similar values for the thermocouples outside the capsule and for the infrared camera.

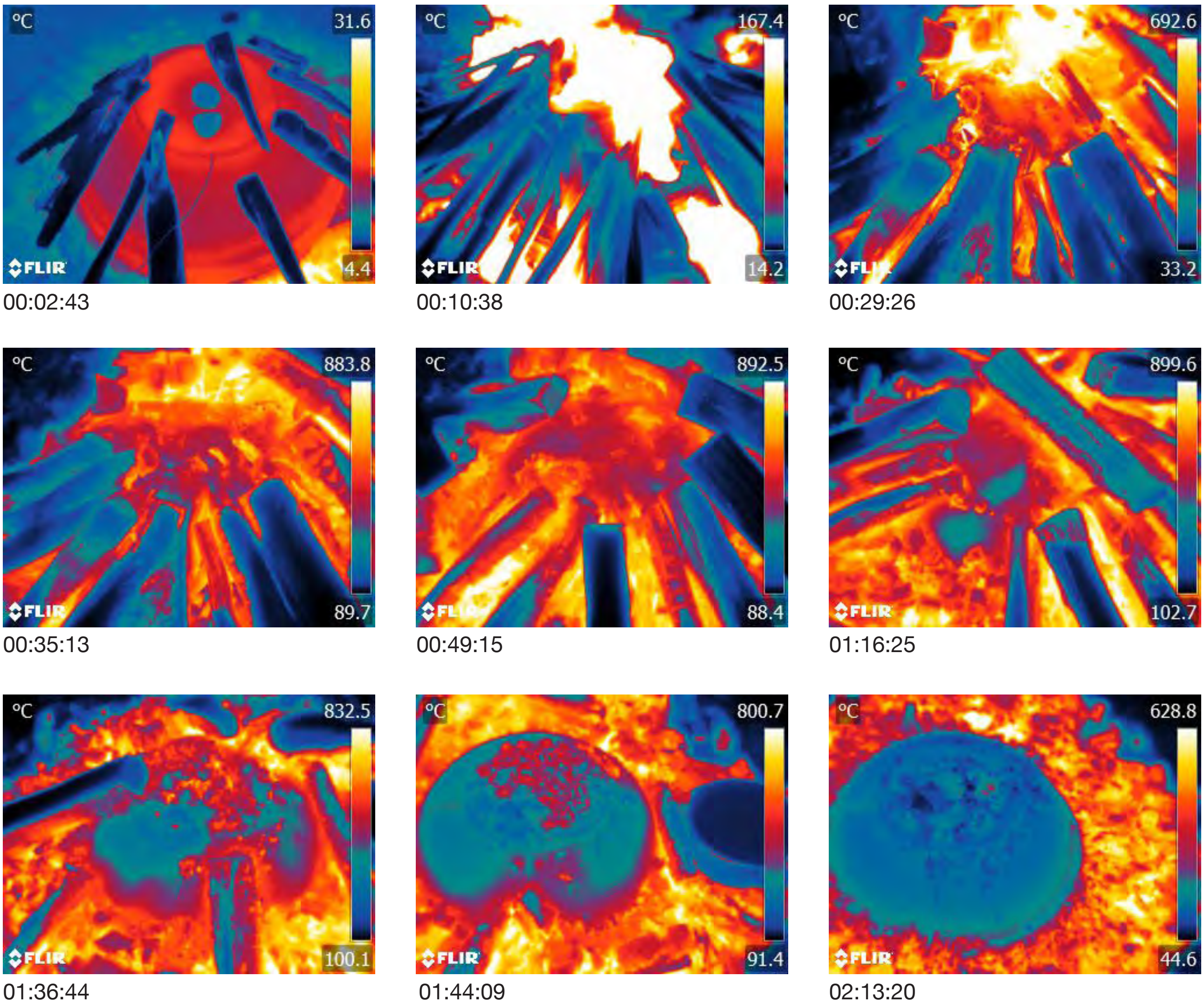
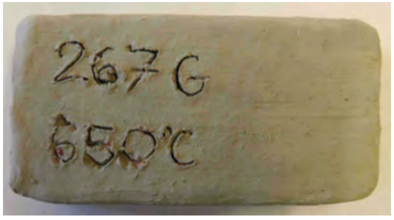
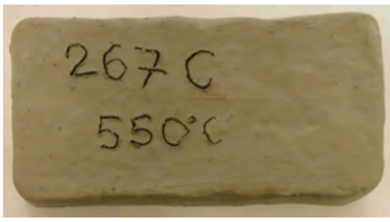
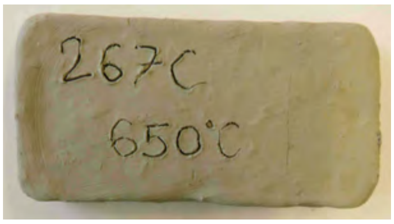
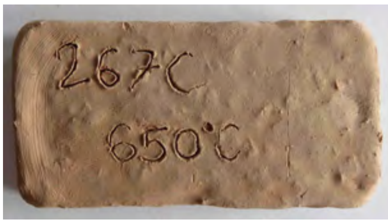
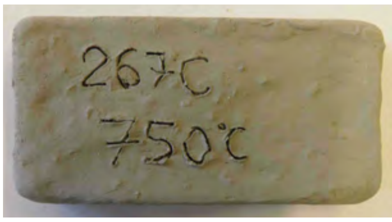
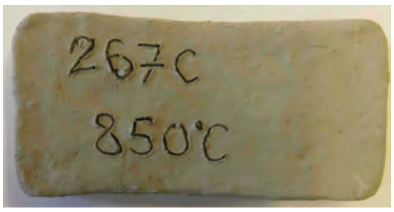


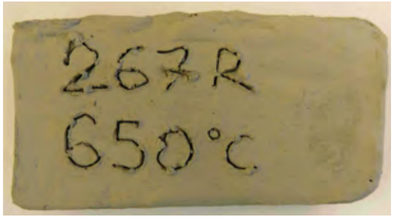
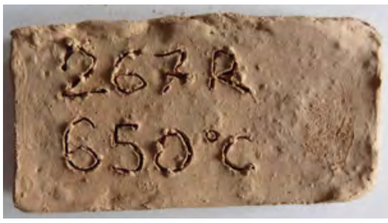
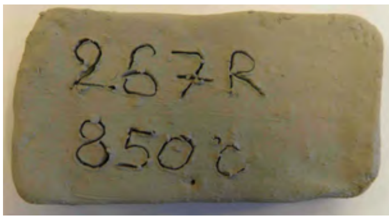
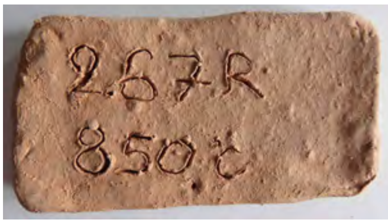
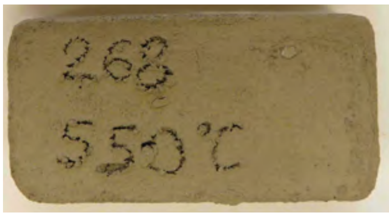
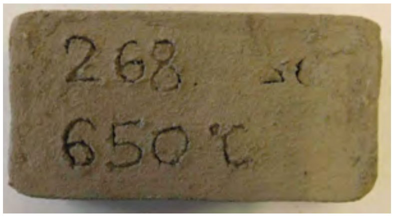
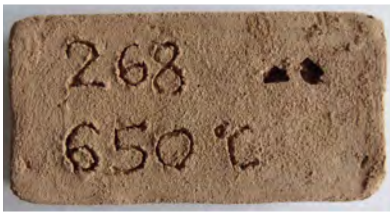
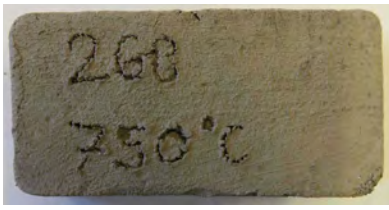

*Fig. 4.6. Infrared images made during the firing. Note the different temperature scale for each image*

### 4.5. Pictures of briquettes and pots

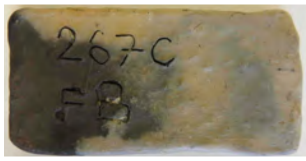
This subchapter displays pictures of briquettes and pots taken before and after firing, as well as their cross-sections. Two briquettes fired in electrical kilns were not sawn neither broken (NLT 267 C 750°C and NLT 267 C 850°C), thus instead of a cross-section, a picture of the whole briquette taken a few days after the firing is shown. Note: briquette size is ~4.1 x 8.1 x 1.2 cm.

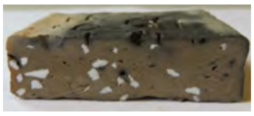
#### 4.5.1. Briquettes fired in electrical kilns

| | Raw | Fired | Fired (cross-section/ state after a few days) |
|---|---|---|---|
| NLT 267 G 650 |  |  |  |
| NLT 267 G 850 |  |  |  |
| NLT 267 C 550 |  |  |  |
| NLT 267 C 650 |  |  |  |
| NLT 267 C 750 |  |  |  |
| NLT 267 C 850 |  |  |  |

| | Raw | Fired | Fired (cross-section/ state after a few days) |
|---|---|---|---|
| NLT 267 R 650 |  |  |  |
| NLT 267 R 850 |  |  |  |
| NLT 268 550 |  |  |  |
| NLT 268 650 |  |  |  |
| NLT 268 750 |  |  |  |
| NLT 268 G 650 |  |  |  |
| NLT 268 G 750 |  |  |  |

#### 4.5.2. Briquettes fired in bonfire without capsule (FB)

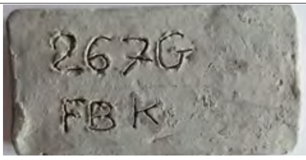
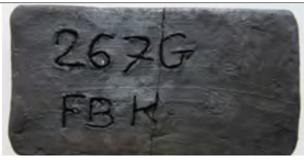
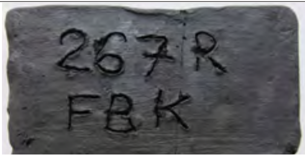
| | Raw (front) | Raw (back) | Fired (front) |
|---|---|---|---|
| NLT 267 FB |  |  |  |
| NLT 267 G FB |  |  |  |
| NLT 267 C FB |  |  |  |
| NLT 267 R FB |  |  |  |
| NLT 268 FB |  |  |  |
| NLT 268 G FB |  |  |  |
| NLT 268 C FB |  |  |  |
| NLT 268 R FB |  |  |  |
| NLT 268 T G FB |  |  |  |
| NLT 268 T R FB |  |  |  |

| | **Fired (back)** | **Fired (cross-section)** |
|---|---|---|
| NLT 267 FB |  |  |
| NLT 267 G FB |  |  |
| NLT 267 C FB |  |  |
| NLT 267 R FB |  |  |
| NLT 268 FB |  |  |
| NLT 268 G FB |  |  |
| NLT 268 C FB |  |  |
| NLT 268 R FB |  |  |
| NLT 268 T G FB |  |  |
| NLT 268 T R FB |  |  |

#### 4.5.3. Pots fired in bonfire without capsule (FB)

| | Raw | Fired | Fired (cross-section) |
|---|---|---|---|
| NLT 268 G FB |  |  |  |
| NLT 268 C FB |  |  |  |
| NLT 268 R FB |  |  |  |

#### 4.5.4. Briquettes fired in bonfire with capsule (FBK)

| | Raw (front) | Raw (back) | Fired (front) |
|---|---|---|---|
| NLT 267 G FBK |   |  |   |
| NLT 267 C FBK |   |  |   |
| NLT 267 R FBK |   |  |   |
| NLT 268 FBK |   |  |   |
| NLT 268 G FBK |   |  |   |
| NLT 268 C FBK |   |  |   |
| NLT 268 R FBK |   |  |   |
| NLT 268 T G FBK |   |  |   |
| NLT 268 T R FBK |   |  |   |

| | Fired (back) | Fired (cross-section) |
|---|---|---|
| NLT 267 G FBK |  |  |
| NLT 267 C FBK |  |  |
| NLT 267 R FBK |  |  |
| NLT 268 FBK |  |  |
| NLT 268 G FBK |  |  |
| NLT 268 C FBK |  |  |
| NLT 268 R FBK |  |  |
| NLT 268 T G FBK |  |  |
| NLT 268 T R FBK |  |  |

#### 4.5.5. Pots fired in bonfire with capsule (FBK)

| | Raw | Fired | Fired (cross-section 1) |
|---|---|---|---|
| NLT 267 G FBK |  |  |  |
| NLT 267 C FBK |  |  |  |
| NLT 267 R FBK |  |  |  |

## 4.6. Description of the fired objects

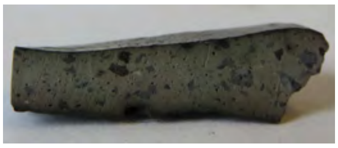
### 4.6.1. Objects fired in electrical kilns

All the briquettes fired in the electrical kilns (oxidizing atmosphere) got a brick-red colour (note: the photos do not reproduce the accurate colour of the briquettes). Briquettes made with clay NLT 267 have a very fine, compact texture, whereas those made with clay NLT 268 are sandy. After a few days, briquettes NLT 267 C 850°C, NLT 268 750°C and NLT 268 G 750°C started to disaggregate. After four weeks, briquette NLT 267 C 750°C is still very solid (it cannot be broken by hand), but calcite appears through the surface, suggesting a beginning of disaggregation. This experiment shows that briquettes made with clay NLT 267 are, for the most part, very solid, except in the cases with addition of calcite that were fired higher than 750°C. On the other hand, clay NLT 268 does not make solid briquettes, especially when fired from and higher than 750°C, even when no calcite was added (calcite naturally occurs in this clay).

### 4.6.2. Objects fired in bonfire without shell (FB)

Once the fires were extinguished, the briquettes and pots were put one after the other inside a bucket with water a few seconds. If the ceramics were not fired high/long enough, they would break down and return to the malleable clayey state. Some parts of the students' ceramics were indeed not sufficiently fired (f.ex. bottom of large pot, placed upright in the fire).

These objects display various colours, from black to orange and grey. The polished surfaces appear smooth, although no particular feature can be distinguished. All these briquettes can easily be broken by hand, except briquette NLT 267 FB. However, a fragment of this briquette burst out, probably because this clay without temper was too fat. Its cross-section let appear pores with a kind of black halo. Other briquettes made from clay NLT 267 display similar pores, although in smaller quantity. Grog added as temper in briquette NLT 267 G FB remained red. Despite the

| | Fired (cross-section 2) |
|---|---|
| NLT 267 G FBK |  |
| NLT 267 C FBK |  |
| NLT 267 R FBK |  |

diversity of colours on the surface, the inside of these briquettes is mostly greyish.

Briquettes made from clay NLT 268 are all particularly sandy and not very solid. The colour of these briquettes is the same at the surface and in the inside. In briquette NLT 268 G FB, the grog became blackish in the black area, and remained red in the greyish zone. The treated, clay-enriched part of clay NLT 268 made briquettes of a slightly finer texture. In this case, the grog became blackish in the black as well as in the greyish zones.

Barbotine that covers pots NLT 268 G FB and NLT 268 R FB cracked during drying. Indeed, as the barbotine contains less temper, it shrunk more than the pot itself while drying. This is especially visible for pot NLT 268 G FB, which is covered with the treated, clay-enriched part of clay NLT 267 T, and displays stronger cracks than the untreated barbotine on pot NLT 268 R FB. The cross-section of the former also shows a clear limit between the barbotine (outside layer) and the pot.

#### 4.6.3. Objects fired in bonfire with capsule (FBK)

Like the objects mentioned above, those fired in FBK were put into water to determine if they transformed into ceramics.

These objects all have a black surface. Briquettes and pots made from clay NLT 267 that had been polished prior to firing display a shiny, metallic surface. The inside of briquettes made from clay NLT 267 is greyish, whereas the inside of briquettes made from clay NLT 268 is uniformly black. Grog remained red in briquette made from clay NLT 267, but became black in briquettes made from clay NLT 268 and NLT 268 T. Interestingly, it became black as well in pot NLT 267 G FBK, possibly because the walls of this pot are thinner than those of the briquettes made from clay NLT 267, or because of a larger quantity of sawdust surrounding the pots.

A fragment of briquette NLT 267 C FBK burst out during firing, maybe due to the presence of calcite as temper, although we observed that adding 10 wt% of limestone as temper did not have a negative impact on the ceramics (briquettes and pots) fired in FB and FBK (firing temperatures remained mostly largely under 750°C). All the briquettes made from clay NLT 268 have a sandy texture and are very friable. Those made from the treated clay NLT 268 have a finer texture and are slightly more solid. However, all the objects can be broken by hand, indicating that they were not fired long enough.

## 5. Analyses

### 5.1. Water absorption measurements (26-30.01.18 and 22.03.18-27.03.18)

A fragment of each of the briquettes and pots fired in FBK was weighed and put into water (26.01.18, 15h30). Four days later (30.01.18, 10h45), the samples were removed from the water, wiped with a wet sponge and weighed again. Water absorption (wt%) was calculated from the dry and wet weights, according to the following equations:

$$d = weight_{wet} - weight_{DRY}$$
$$water\ absorption = (100*d)/weight_{wet}$$

The results of the water absorption show values between 1.76 and 17.86 (Table 5.1). This wide range of values may be partly explained by the diversity of sample sizes: small samples are less reliable than larger sample. Due to high fluctuations between the results from the water absorption measurement made in January, we performed this measurement again in March with larger fragments of five samples (Table 5.1), put into water on 22.03.18, 9h30, and removed and weighed on 27.03.18, 13h. The second water absorption measurement (in italics in Table 5.1) has much higher values than those from the first measurement (in red in Table 5.1), except for Pot 267 C FBK (13.76 instead of 14.16). In the description below, we do not consider the five red values from the first measurement.

We can distinguish three groups, which correspond to the different types of clays. Samples made from the treated part of clay NLT 268 (NLT 268 T) show the highest values, around 17 wt%. Water absorption seems to be higher for briquettes made from clay NLT 268 (~12-16 wt%) than those made from clay NLT 267 (11-13 wt%), which are more porous. However, the pots made from clay NLT 267 have similar values as briquettes NLT 268, possibly because pot walls are relatively thin and thus can absorb more water than briquettes. The different types of temper do not seem to have an effect on the water absorption.

| Sample | $Weight_{Dry}$ [g] | $Weight_{Wet}$ [g] | Water absorption [wt %] |
|---|---|---|---|
| NLT 267 G FBK | 29.00 | 32.80 | 11.59 |
| NLT 267 C FBK | 9.90 | 10.80 | 8.33 |
| | *25.78* | *29.79* | *13.46* |
| NLT 267 R FBK | 23.90 | 27.10 | 11.81 |
| NLT 268 FBK | 28.20 | 32.30 | 12.69 |
| NLT 268 G FBK | 14.54 | 17.30 | 15.95 |
| NLT 268 C FBK | 8.50 | 9.90 | 14.14 |
| NLT 268 R FBK | 19.80 | 20.60 | 3.88 |
| | *17.30* | *20.16* | *14.19* |
| NLT 268 T G FBK | 4.60 | 5.60 | 17.86 |
| NLT 268 T R FBK | 7.30 | 8.82 | 17.23 |
| Pot NLT 267 G FBK | 4.00 | 4.20 | 4.76 |
| | *15.50* | *18.44* | *15.94* |
| Pot NLT 267 C FBK | 5.70 | 6.64 | 14.16 |
| | *16.73* | *19.40* | *13.76* |
| Pot NLT 267 R FBK | 5.60 | 5.70 | 1.76 |
| | *27.56* | *31.76* | *13.22* |

*Table 5.1. Water absorption of samples fired in FBK. Five samples were measured a second time: in red are the values from the first measurement (January), and in italics are those from the second measurement (March).*

## 5.2. X-Ray Diffractometry (XRD) (31.01.18-08.02.18)

Twelve briquettes and the six pots were analysed by XRD, in order to determine the mineralogical composition of the fired ceramic. As some of the briquettes were not homogeneous, we chose arbitrarily to sample a ~1.5 cm-large slice from the left part of each briquette. We also took a ~2 $cm^3$-sherd of each pot. Picture of the samples were taken (Fig. 5.1). Each sample was first crushed with a hammer, then ground with a disc mill for 1 minute with a speed of 960 $min^{-1}$. The powder was put into a sample holder.

The analyses were performed with a Rigaku Ultima IV X-ray diffractometer, which was operated at 40 kV and 40 mA with a Cu-anode, from 5 to 70°, 0.5°/min and a 0.02°-step width.

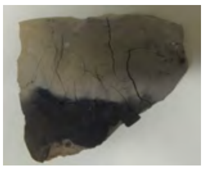
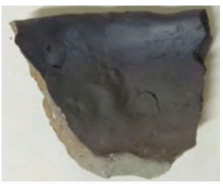
| | FB | FBK | 650°C | 850°C |
|---|---|---|---|---|
| NLT 267 |  | | | |
| NLT 267 G |  |  |  |  |
| NLT 267 C |  |  |  | |
| NLT 267 R |  |  |  |  |
| Pots NLT 267 G |  |  | | |
| Pots NLT 267 C |  |  | | |
| Pots NLT 267 R |  |  | | |

*Fig. 5.1. Samples used for XRD analysis.*

The diffractograms obtained (Figs. 5.3-5.9) were then processed with the Rigaku PDXL-2 software in order to identify the mineral phases (Table 5.2 and Appendix 1). As a comparison, XRD analyses of raw clays NLT 267, NLT 268 and NLT 266B from a previous experiment are also displayed (Fig. 5.2). Some phases were difficult to identify, because their peaks overlapped with those of other phases. For example, illite peaks were mostly hidden by muscovite peaks, so this phase was not identified in many briquettes, although it was certainly present. Kaolinite (001) overlaps with clinochlore (002), so only clinochlore was identified, although a large clinochlore (002) peak can be explained by the presence of kaolinite. Furthermore, the relative intensity of clinochlore peaks changes with temperature.

Similar mineral phases were identified for the briquettes fired at 650°C or in bonfires: quartz, calcite (sometimes also dolomite), muscovite, clinochlore and feldspars (mostly albite). In briquettes fired at 850°C, changes of mineral assemblages can be observed: clinopyroxenes, hematite and gehlenite appear, replacing carbonates and clinochlore; pyrite was not found in our samples, although it is often present in Neolithic ceramics from lake settlements, due to secondary formation during burial.

Some differences between the briquettes fired in kiln and in bonfire can be observed, in particular regarding the intensity of the peaks of the clay minerals:

NLT 267 G:

- The peak of chlorite (001) is very intense for 650°C, small for FB and FBK and absent for 850°C.
- The peak of muscovite/illite at ~8.7 is much more intense for 650°C, FB and FBK than for 850°C.
- The peak of chlorite (002)/kaolinite (001) is present for FB and FBK, but absent for 650°C and 850°C.

NLT 267 C:

- The peak of chlorite (001) is more intense for 650°C and FB than for FBK.
- The peak of chlorite (002)/kaolinite (001) is present for FB (very high) and FBK (smaller), but absent for 650°C.

NLT 267 R:

- The peak of chlorite (001) is very intense for 650°C, very small for FB and FBK and absent for 850°C.
- The peak of muscovite/illite at ~8.7 is more intense for 650°C, FB and FBK than for 850°C.
- The peak of chlorite (002)/kaolinite (001) is present for FBK only.

Pots NLT 267 G, C and R:

- The peak of chlorite (001) is slightly higher for FBK than for FB.
- The peak of muscovite/illite at ~8.7 is intense for FB and FBK.
- The peak of chlorite (002)/kaolinite (001) is more intense for FBK than for FB.

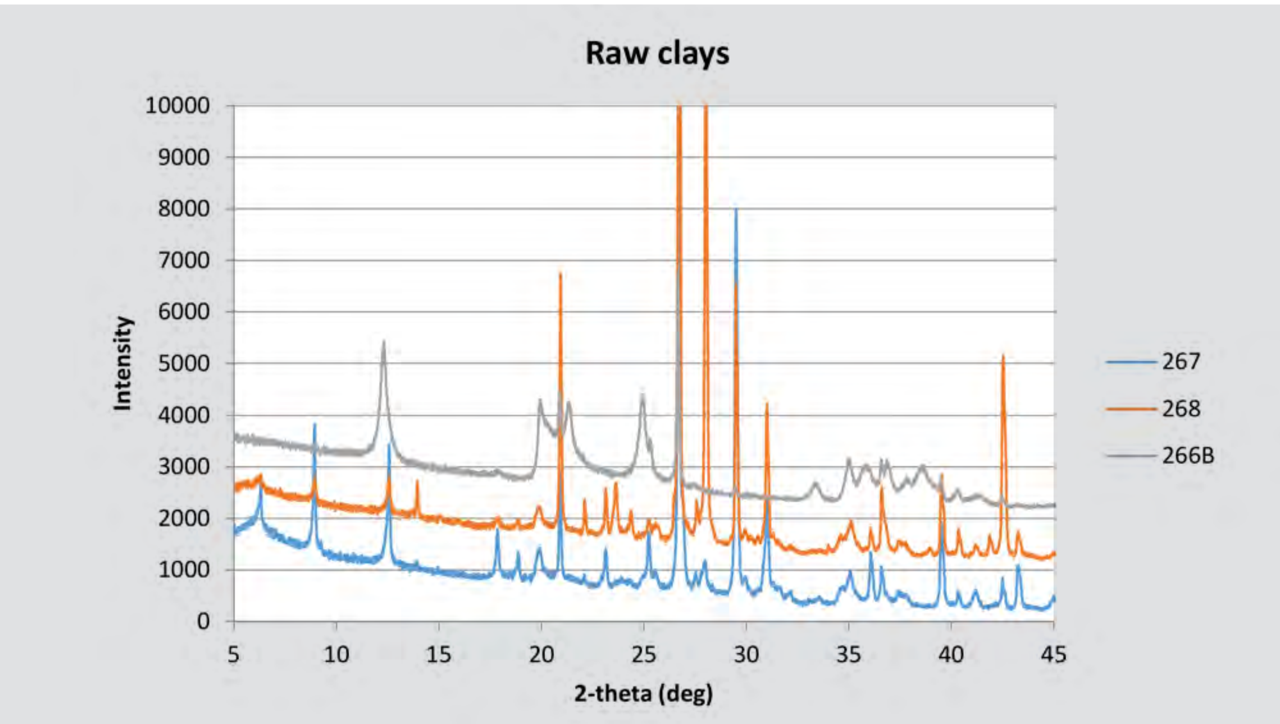



*Fig. 5.2. Diffractogram of raw clays NLT 267, NLT 268 and NLT 266B.*

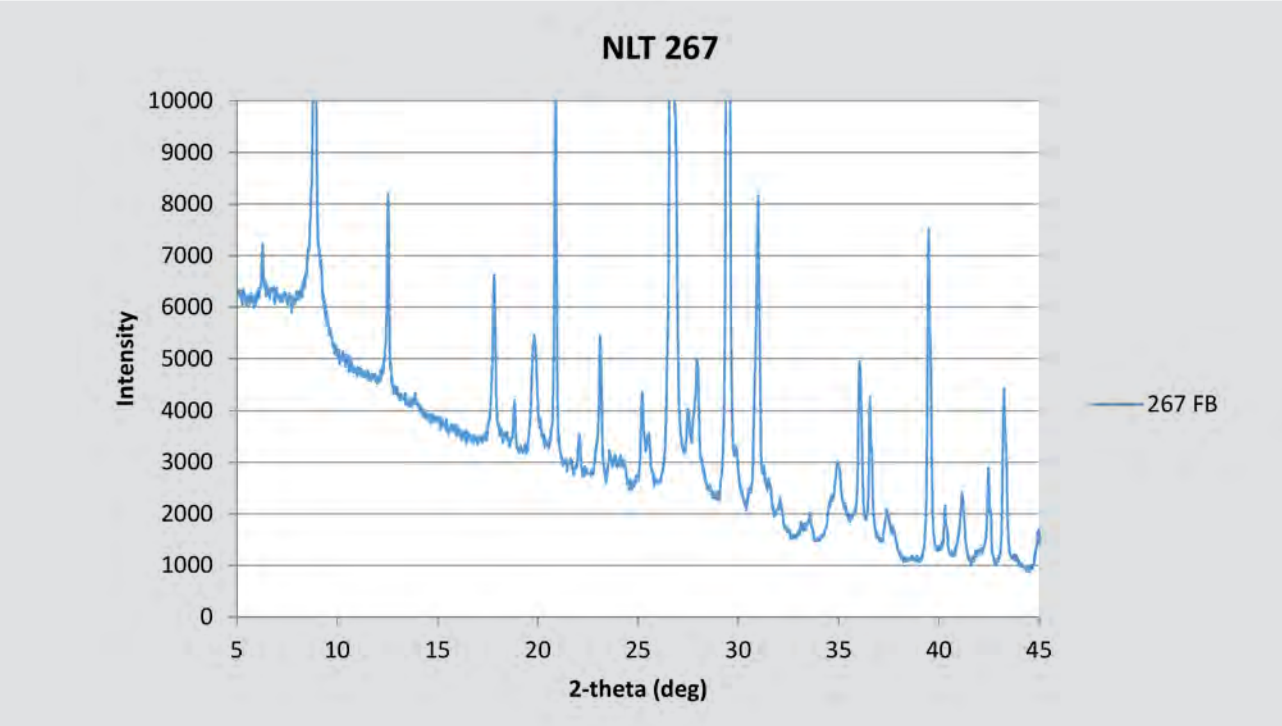



*Fig. 5.3. Diffractogram of briquette made with clay NLT 267.*

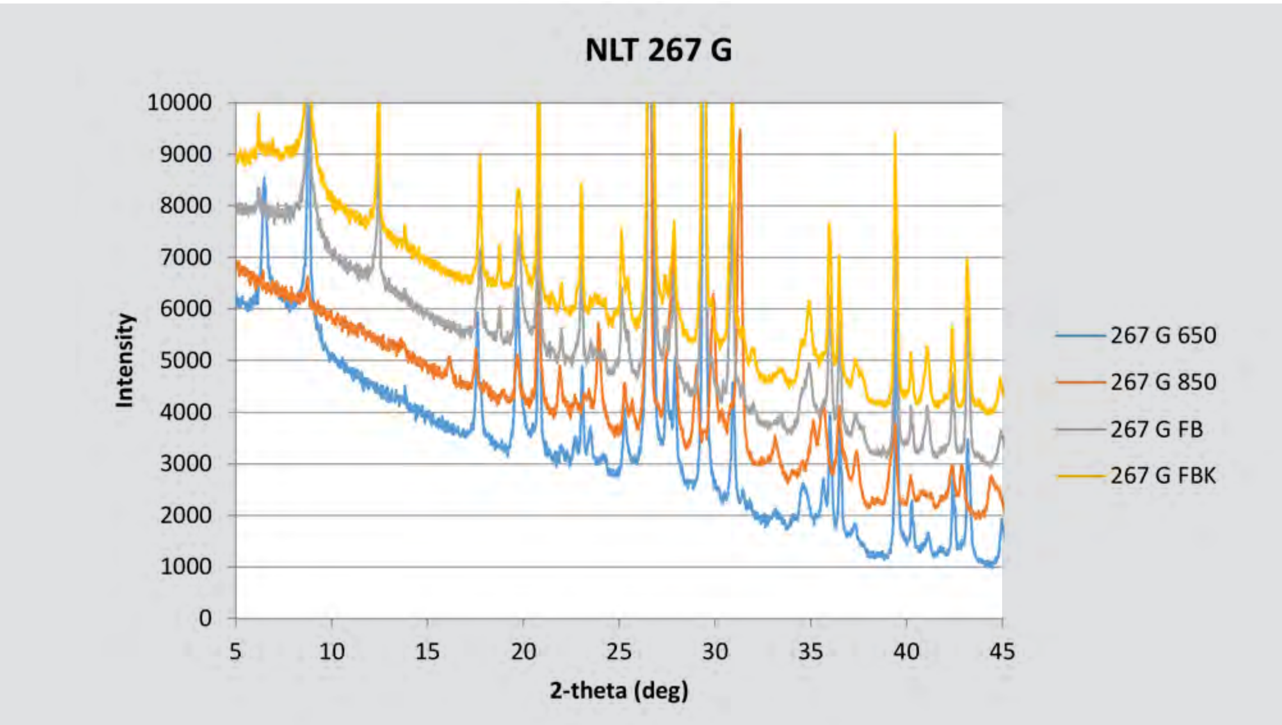



 *Fig. 5.4. Diffractograms of briquettes made with clay NLT 267 G fired at different temperatures.*

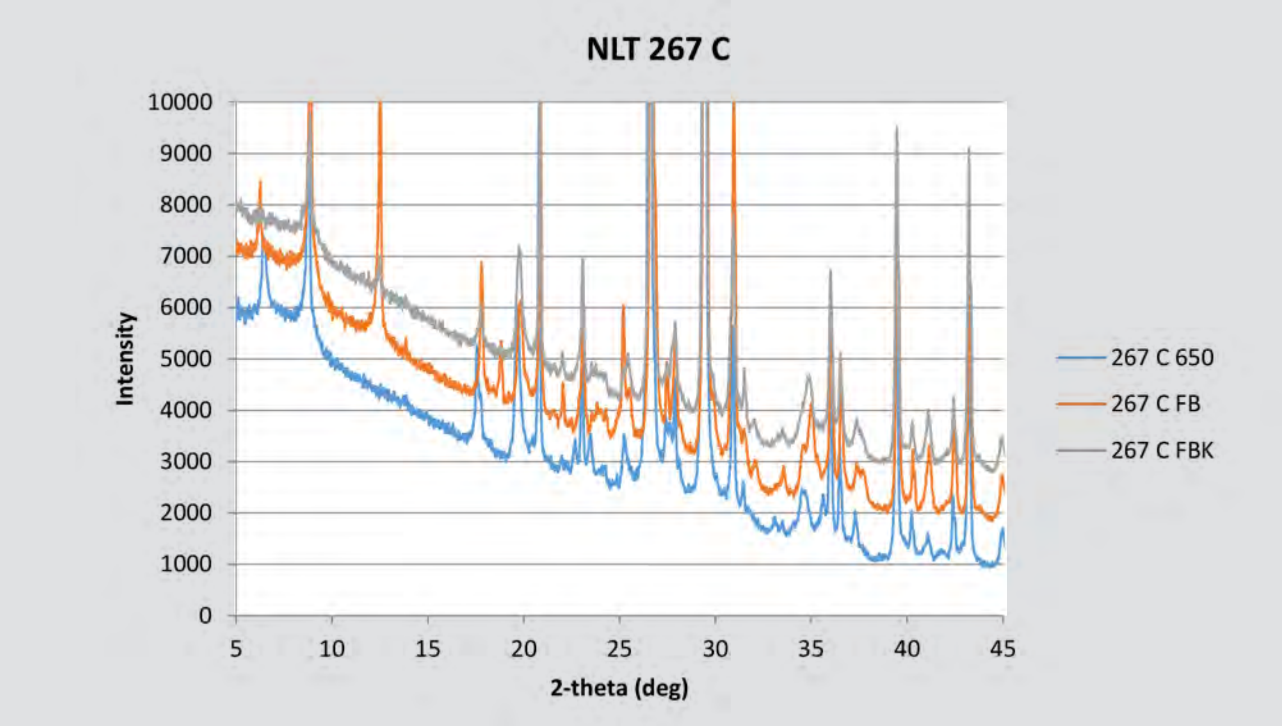



*Fig. 5.5. Diffractograms of briquettes made with clay NLT 267 C fired at different temperatures.*

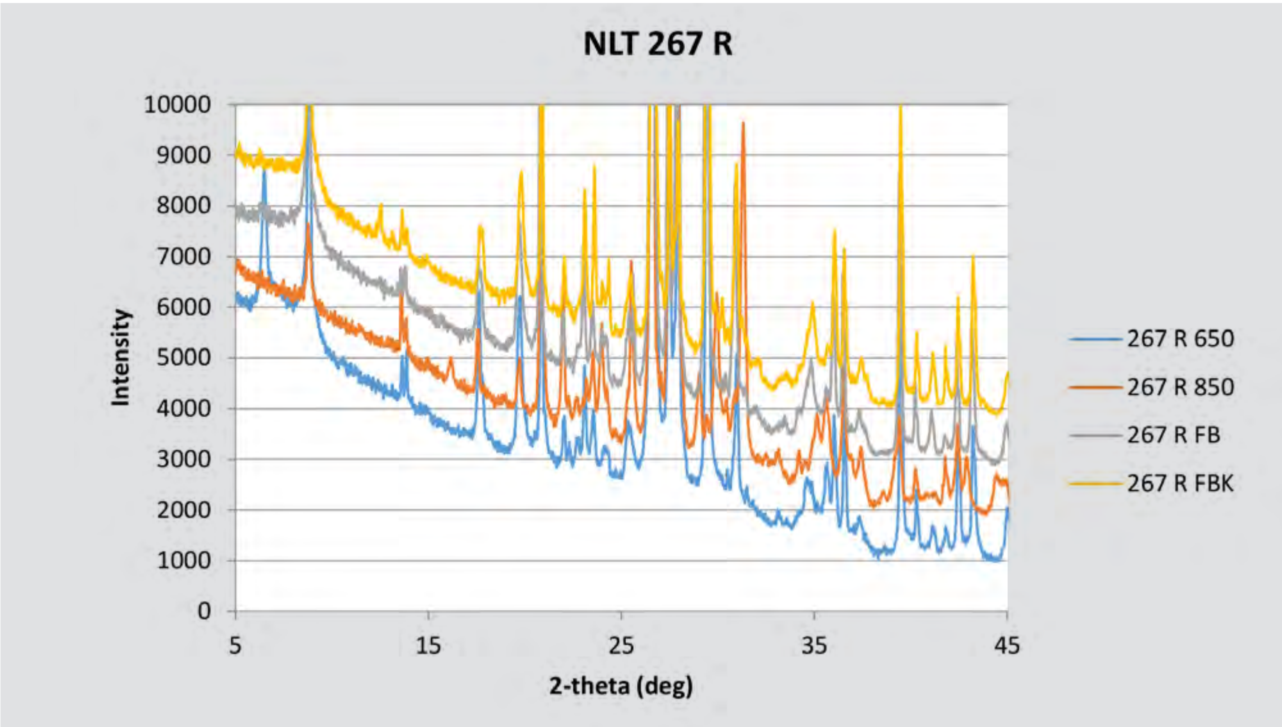



*Fig. 5.6. Diffractograms of briquettes made with clay NLT 267 R fired at different temperatures.*

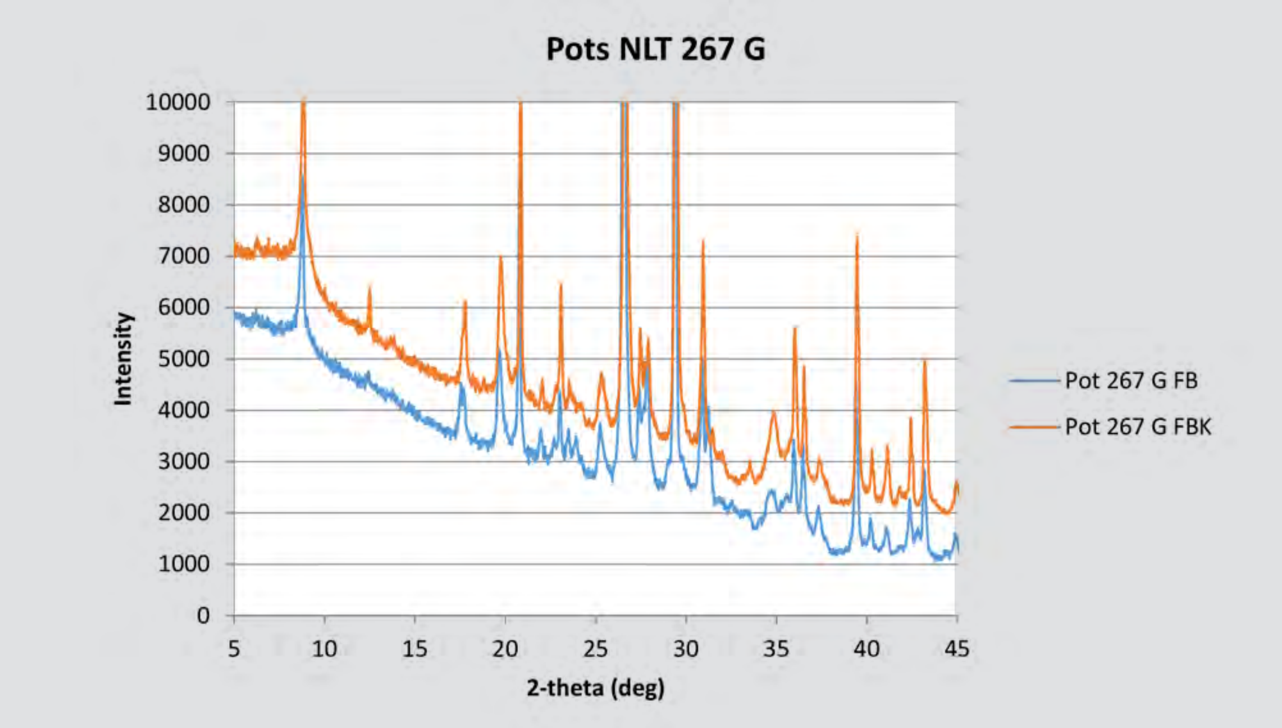



*Fig. 5.7. Diffractograms of pots made with clay NLT 267 G fired at different temperatures.*

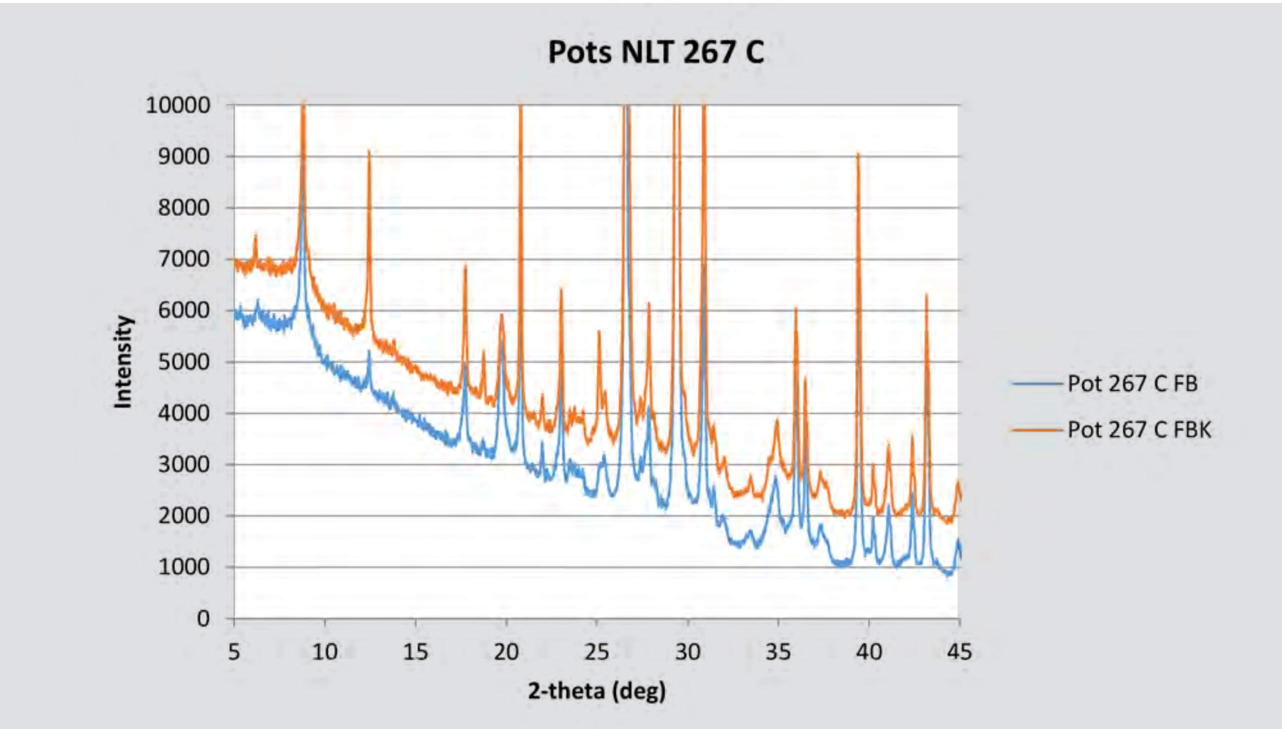





*Fig. 5.8. Diffractograms of pots made with clay NLT 267 C fired at different temperatures.*

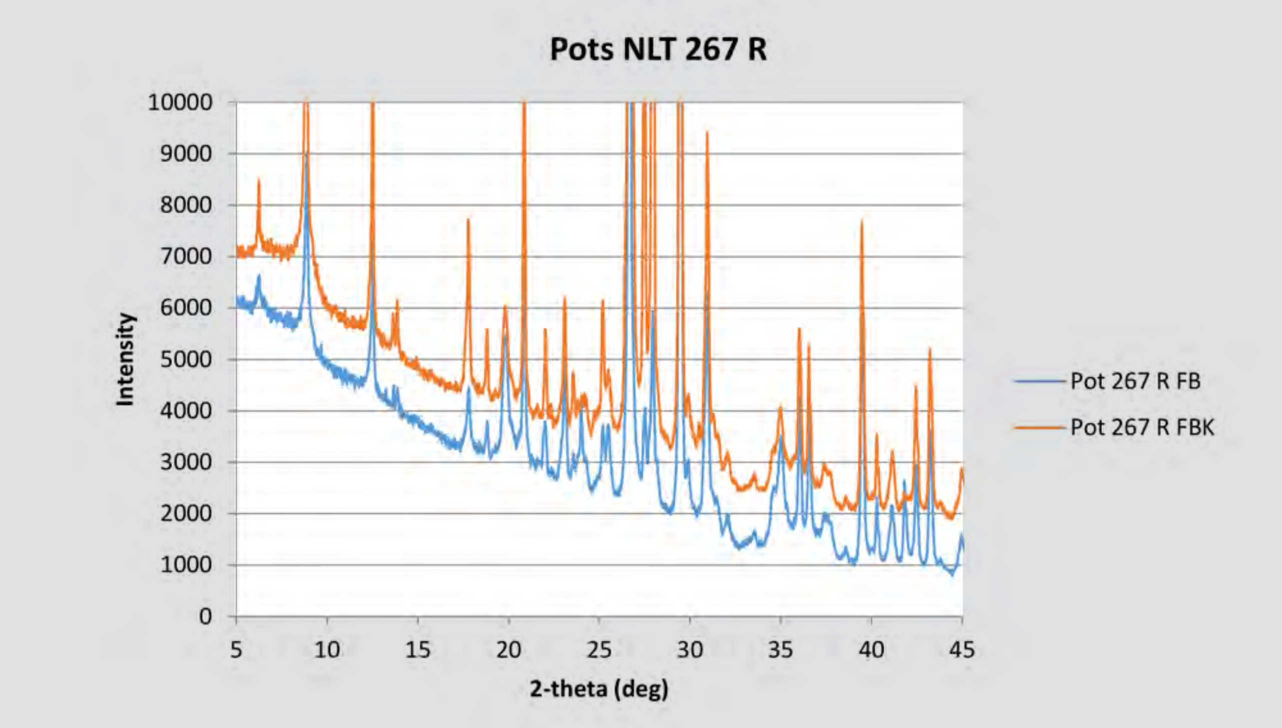



*Fig. 5.9. Diffractograms of pots made with clay NLT 267 R fired at different temperatures.*

| Samples | Quartz | Calcite | Dolomite | Illite | Muscovite | Clinochlore (001) | Clinochlore (002) | Albite | Orthoclase | Microcline | Hematite | Gehlenite | Diopside |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 267 FB | x | x | x | x | x | x | xx | x | | | | | |
| 267 G 650 | x | x | | x | x | x | | x | | | | | |
| 267 G 850 | x | | | | x | | | x | x | | x | x | x |
| 267 G FB | x | x | x | x | x | x | xx | x | | | | | |
| 267 G FBK | x | x | x | | x | x | xx | x | | | | | |
| 267 C 650 | x | x | x | x | x | x | | x | | | | | |
| 267 C FB | x | x | x | | x | x | xx | x | | | | | |
| 267 C FBK | x | x | x | | x | x | xx | x | | | | | |
| 267 R 650 | x | x | x | | x | x | | x | x | | | | |
| 267 R 850 | x | | | | x | | | x | | x | x | x | x |
| 267 R FB | x | x | | | x | x | | x | x | | | | |
| 267 R FBK | x | x | x | x | x | x | xx | x | x | | | | |
| Pot 267 G FB | x | x | x | x | x | x | x | x | | | | | |
| Pot 267 G FBK | x | x | x | | x | x | xx | x | | | | | |
| Pot 267 C FB | x | x | x | x | x | x | x | x | | | | | |
| Pot 267 C FBK | x | x | x | | x | x | xx | x | | x | | | |
| Pot 267 R FB | x | x | x | | x | x | xx | x | x | | | | |
| Pot 267 R FBK | x | x | x | | x | x | xx | x | | | | | |

*Table 5.2. Identification of the mineral phases in the briquettes and pots. In the case of clinochlore, two peaks are represented (001 and 002); the more intense of the two is indicated with "xx".*

### 5.3. Characterisation of pot surfaces with binocular images (14-15.02.18)

A Nikon SMZ18 binocular was used to acquire images of several fragments of the pots fired in the bonfire with capsule (FBK; Figs. 5.10-5.12), as well as of pot NLT 267 C FB (Fig. 5.13).

Figures 5.10-5.12 show a distinct black surface layer of various thicknesses, between a few µm (Figs. 5.10b, 5.11c, 5.12b) to almost 1 mm (Fig. 5.12d). For polished pots NLT 267 G FBK and NLT 267 R FBK, this surface has a metallic shine, sometimes along with tiny dots, such as in Figures 5.10c-d and 5.12a-b.

Pot NLT 267 C FB (Fig. 5.13) displays a light-coloured border, distinct from the rest of the fragment. It is a few µm-thick and is directly followed by a darker area.

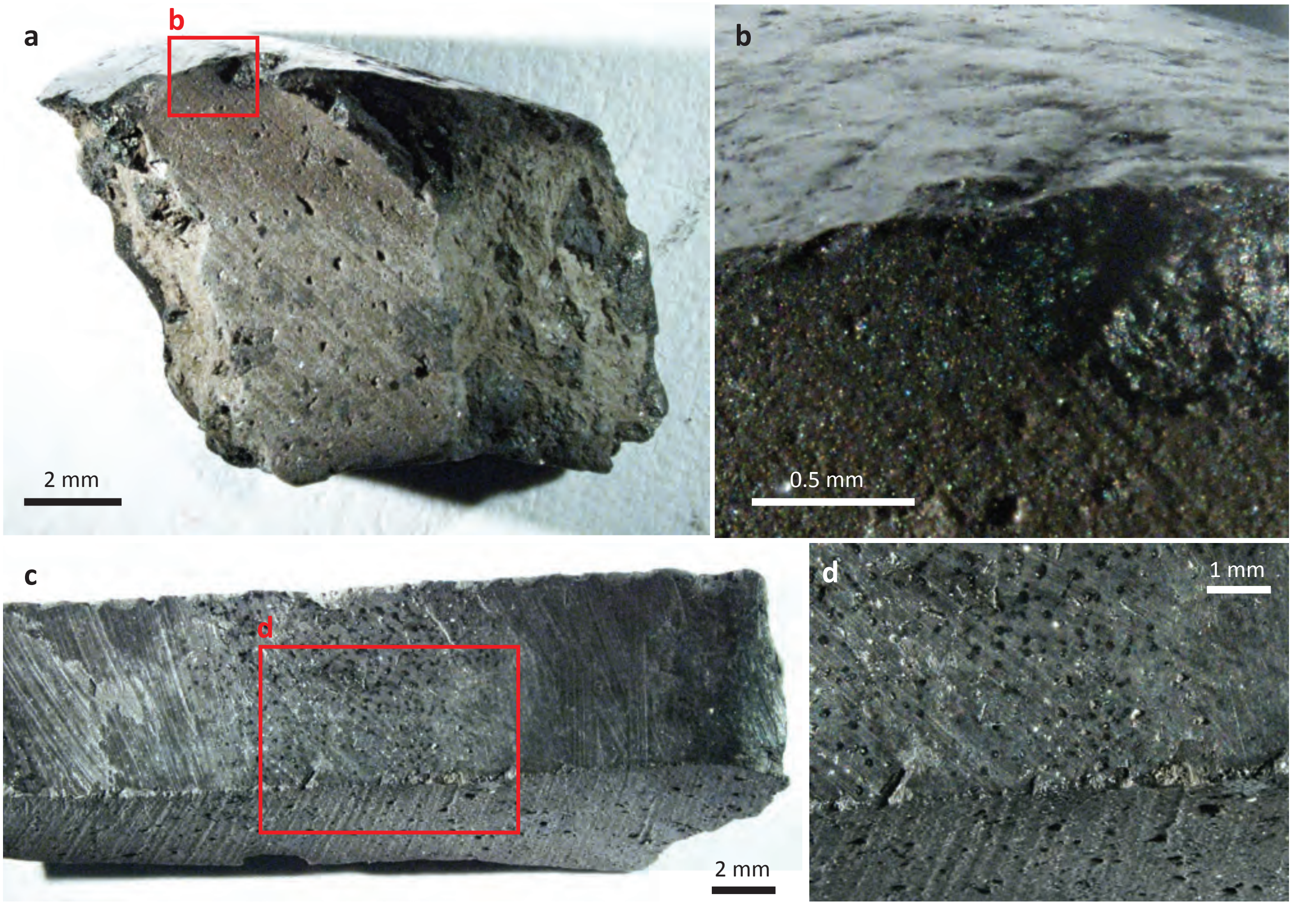



*Fig. 5.10. Binocular images of two fragments of pot NLT 267 G FBK.*

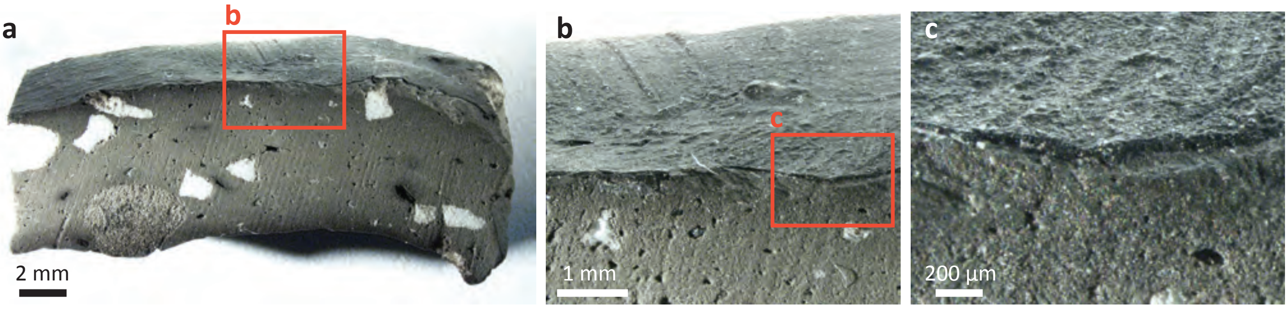



 *Fig. 5.11. Binocular images of a fragment of pot NLT 267 C FBK*

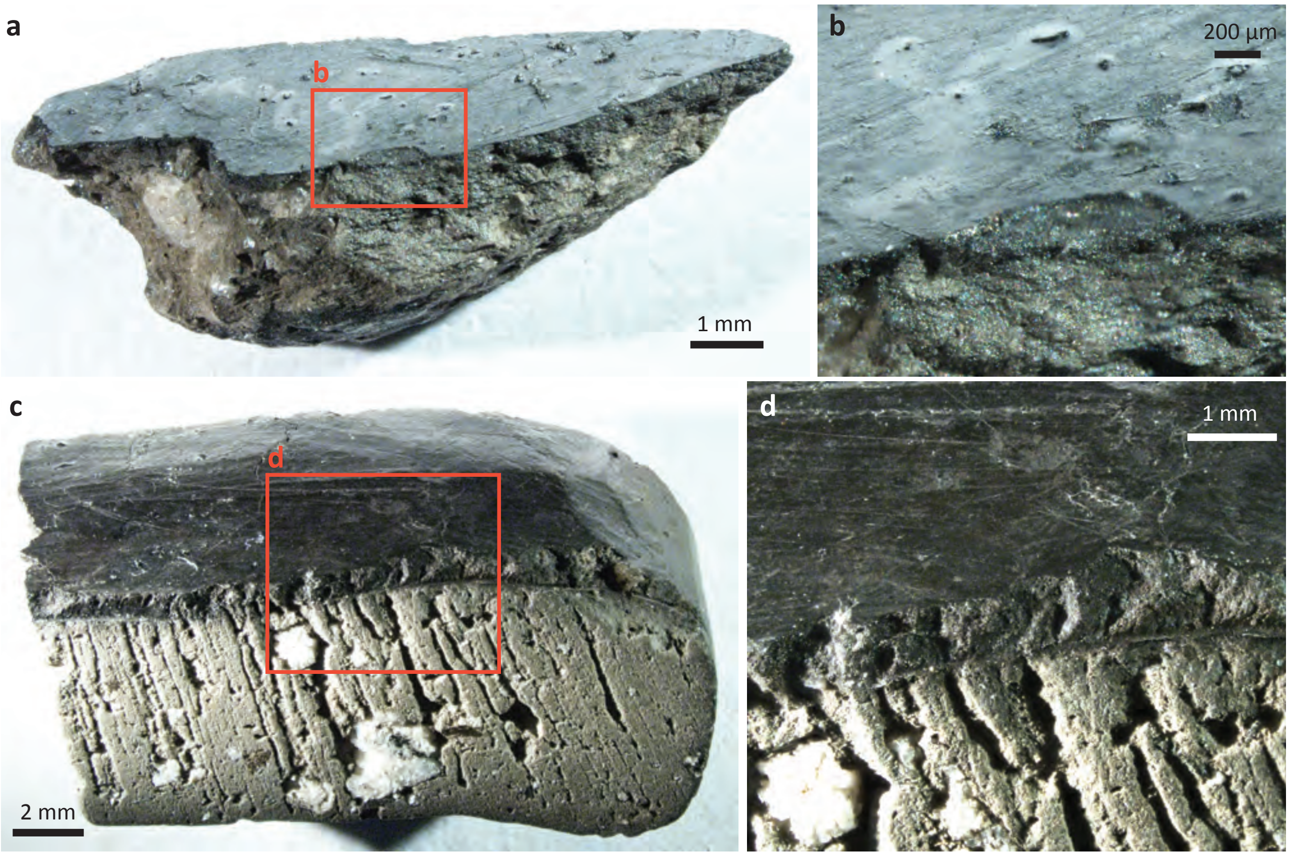



*Fig. 5.12. Binocular images of two fragments of pot NLT 267 R FBK.*

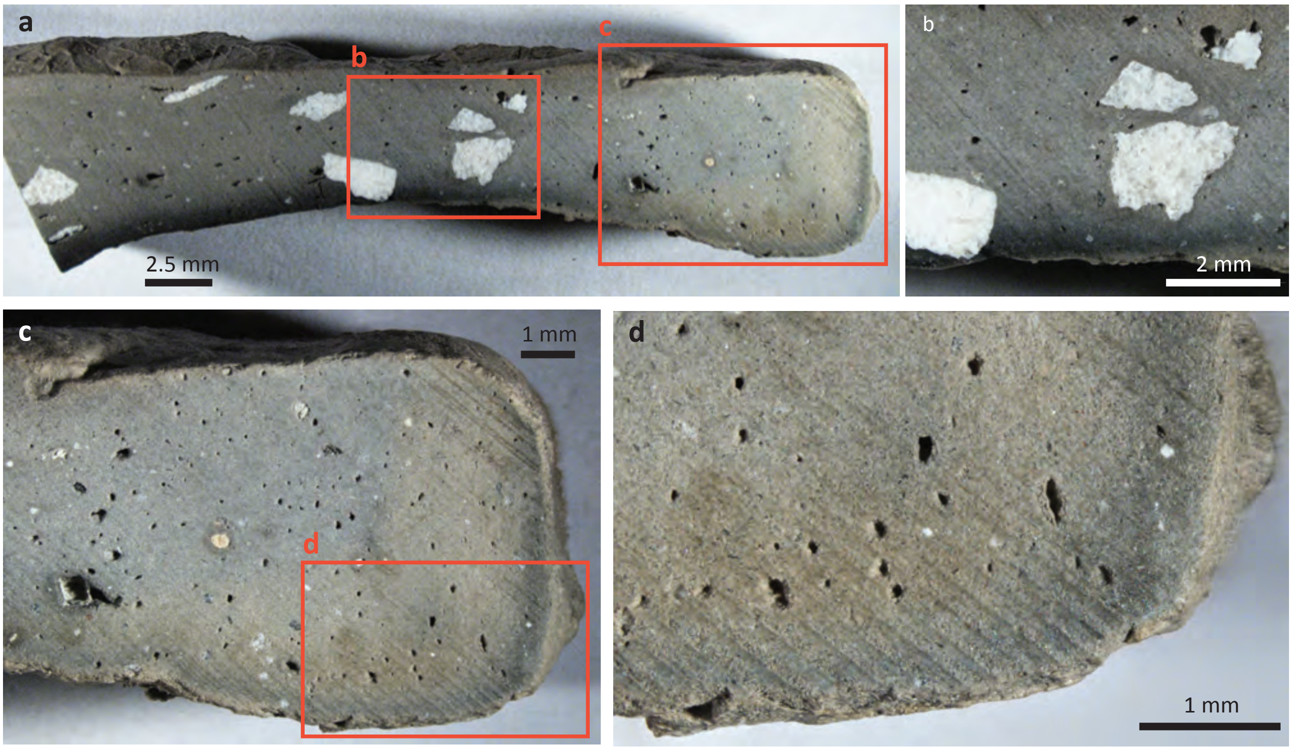



*Fig. 5.13. Binocular images of a fragment of pot NLT 267 C FB.*

## 5.4. Scanning electron microscope (SEM) coupled with energy-dispersive spectrometry (EDS) (13-18.02.18)

SEM analysis was performed on fragments of the polished parts of pot NLT 267 G FBK ("NLT 267_1") and pot NLT 267 R FBK ("NLT 267_2") in order to get images of the micro-structure at the surface and in the cross-section, as well as to do an EDS analysis and mapping of the elements. Samples were previously covered with a 30 nm-thick carbon coating. Results were processed by Christoph Neururer (Appendix 2).

### 5.4.1. Fragment of pot NLT 267 G FBK ("NLT267_1")

Nine sites were investigated with SEM and EDS for this pot fragment. Measuring parameters for Sites 1_1 to 1_6 were 20 kV and Spot 5.0, and 6 kV for Sites 7-11. Site 1_7 was measured with a Spot 3.0, and Sites 8-10 with a Spot 4.0. Site 1_2 does not exist.

### 5.4.2. Fragment of pot NLT 267 R FBK ("NLT267_2")

First, 16 SEM images from different areas were taken of this pot fragment (Figs. 5.14-5.17), followed with an EDS mapping of two sites.

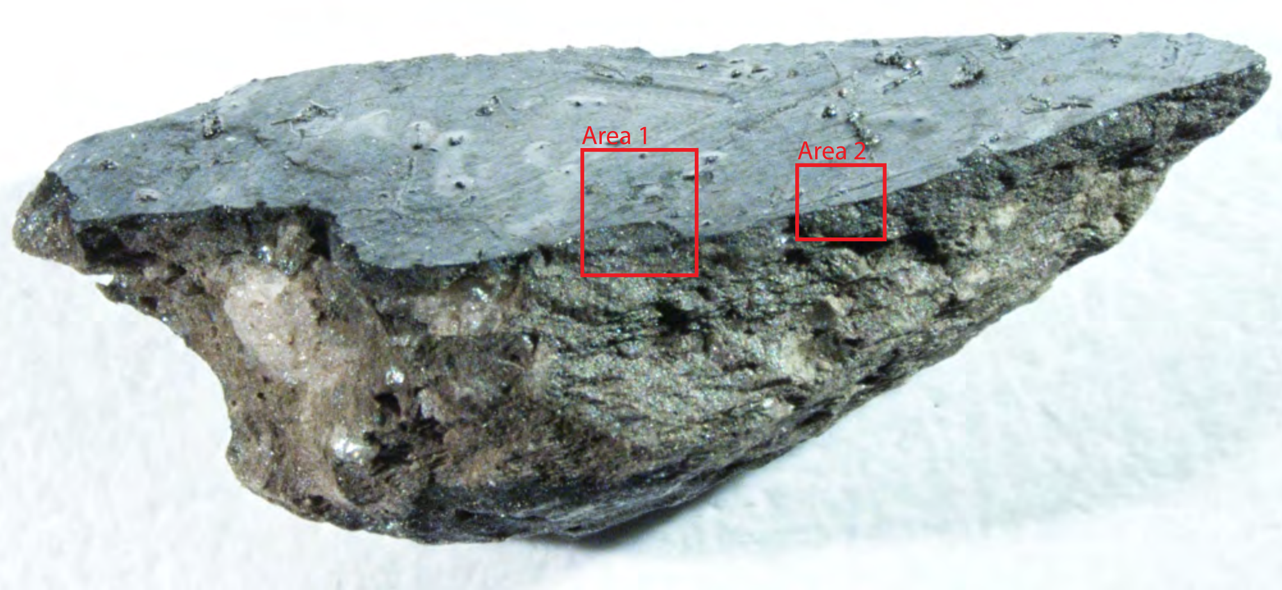



*Fig. 5.14. Binocular image of pot fragment NLT 267 R FBK, showing areas 1 and 2.*

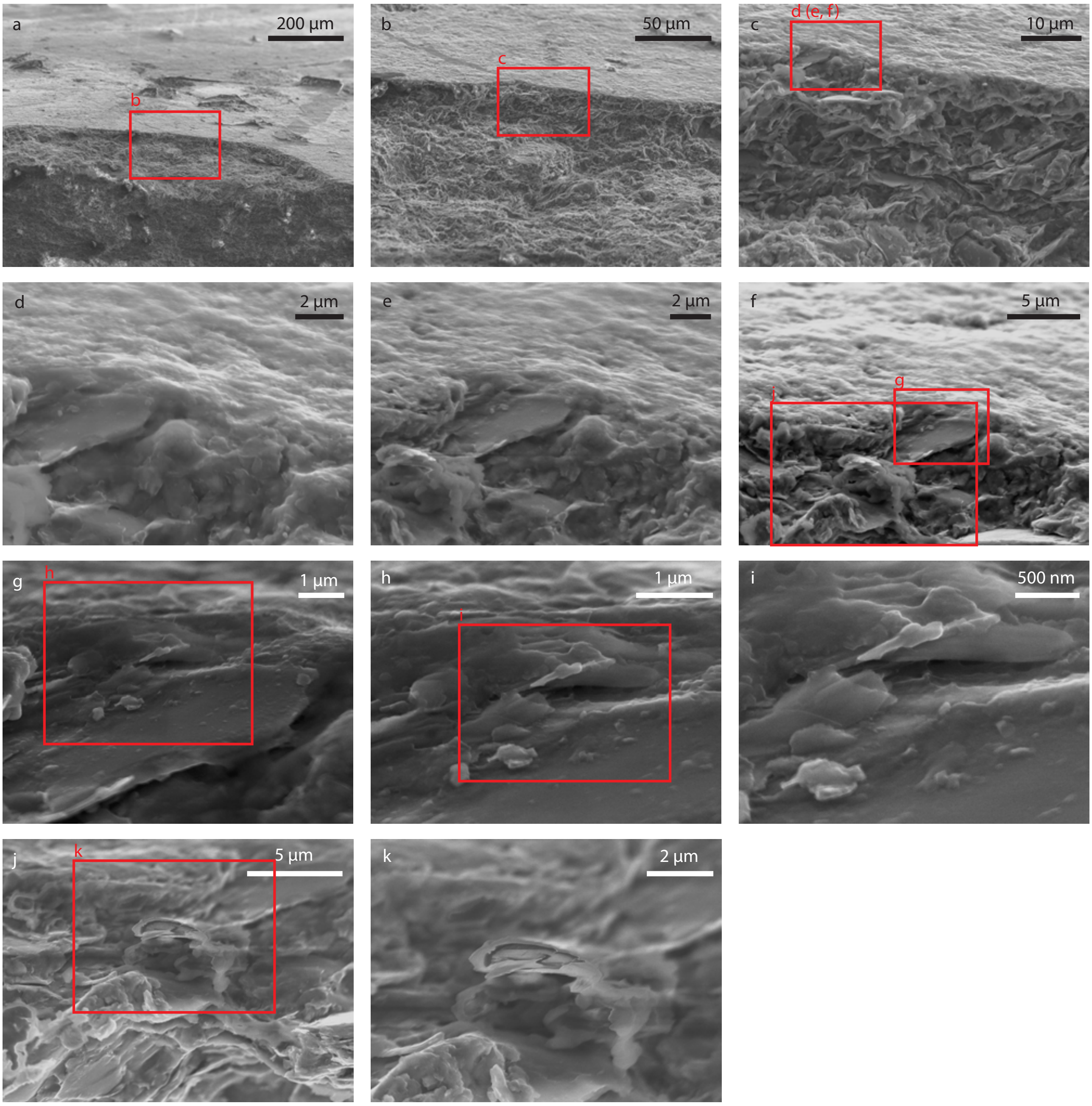



*Fig. 5.15. SEM images of area 1 of pot fragment NLT 267 R FBK; a-d: tension of 10 kV, spot 4.0; e: tension of 6 kV, spot 2.0; f: tension of 6 kV, spot 3.0; g-k: 6 kV, spot 3.0, ultra-high resolution; artefacts are present in j-k.*

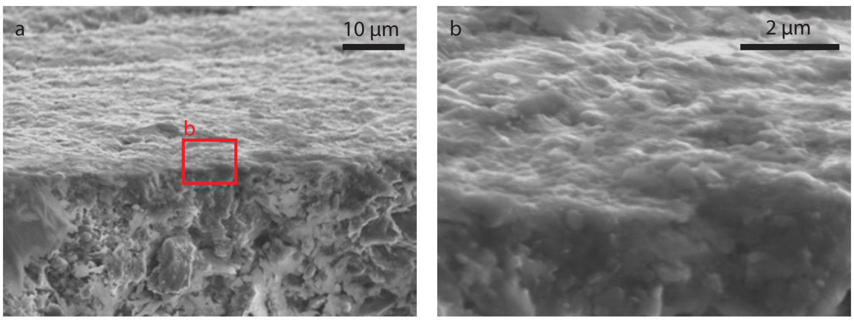



*Fig. 5.16. SEM images of area 2 of pot fragment NLT 267 R FBK; a: tension of 10 kV, spot 4.0; b: tension of 6 kV, spot 2.0.*

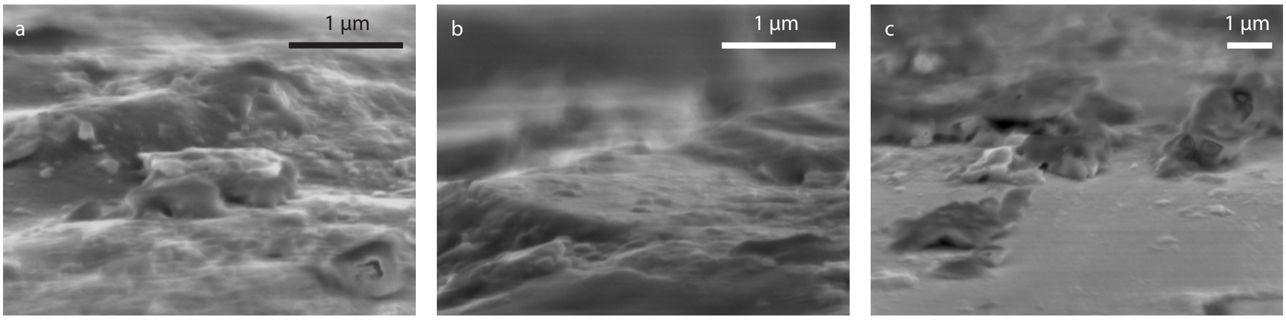
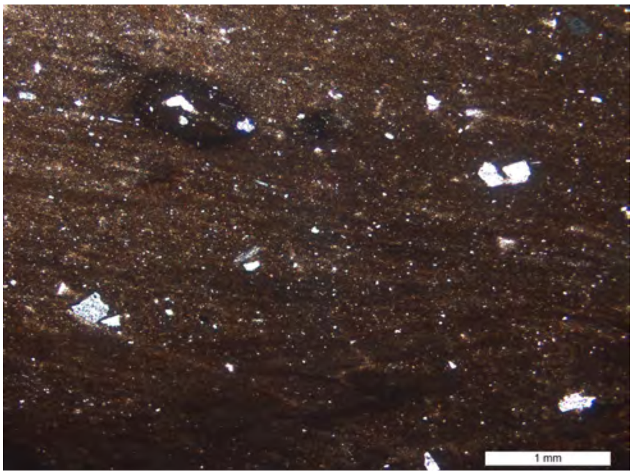


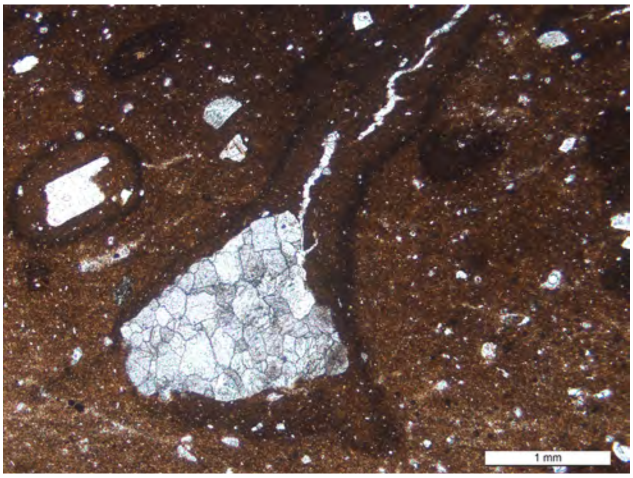

*Fig. 5.17. SEM images of unknown area(s) of pot fragment NLT 267 R FBK; a-c: tension of 3 kV, spot 3.0, ultra-high resolution; artefacts are present in these images.*

### 5.5. Thin sections (microscopy)

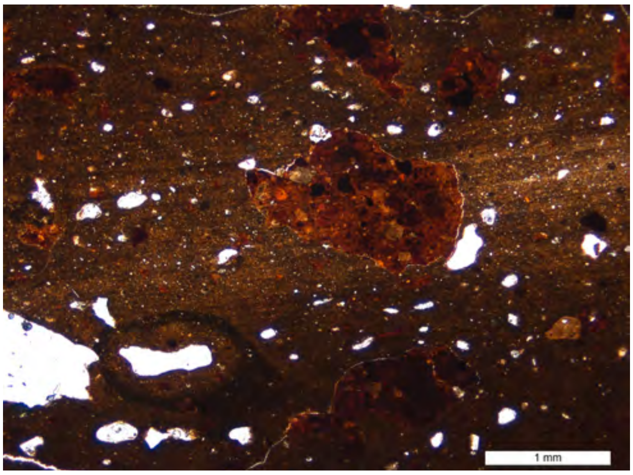
Eleven cross-sections of briquettes and pots were embedded in resin and polished to make 30 µm-thin sections. They were thereafter observed with a Leica DM4500 P petrographic microscope, coupled to a Leica DFC500 digital camera, to get information about the texture of the ceramics. Images were acquired with the Leica Application Suite V4.6 software (see below).




*NLT 267 FB x25*




*NLT 267 C 650°C x25*

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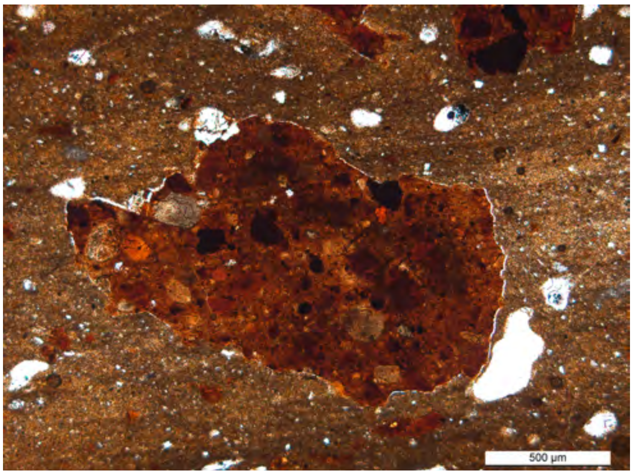


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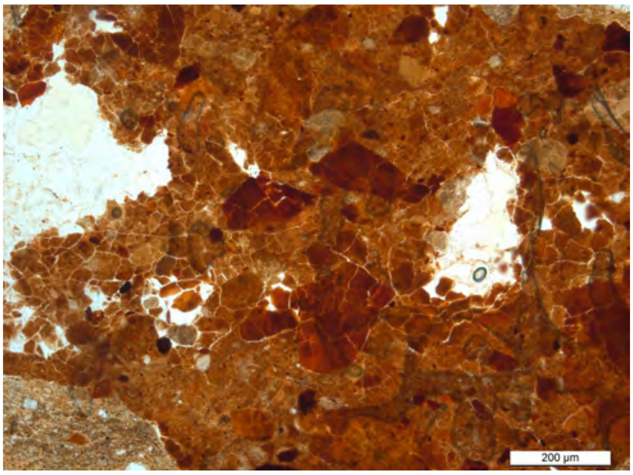


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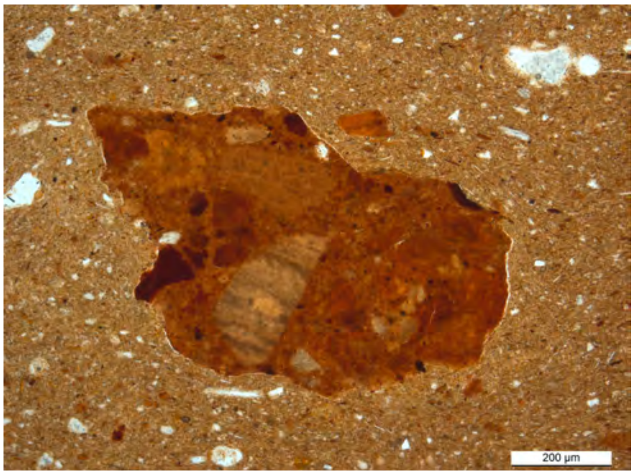


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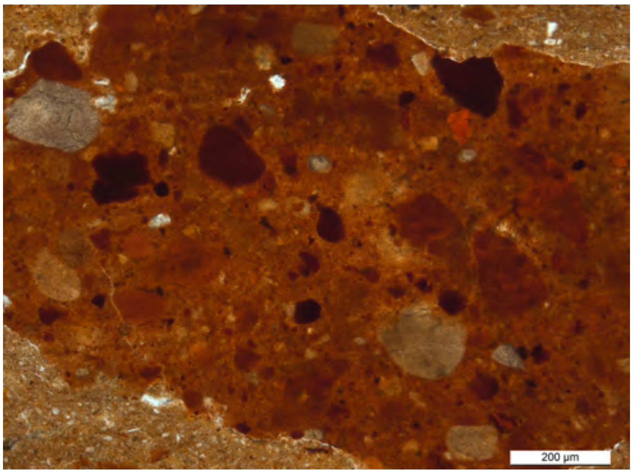
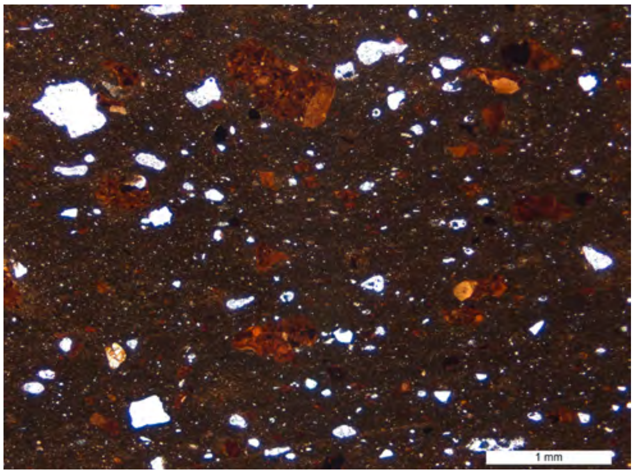
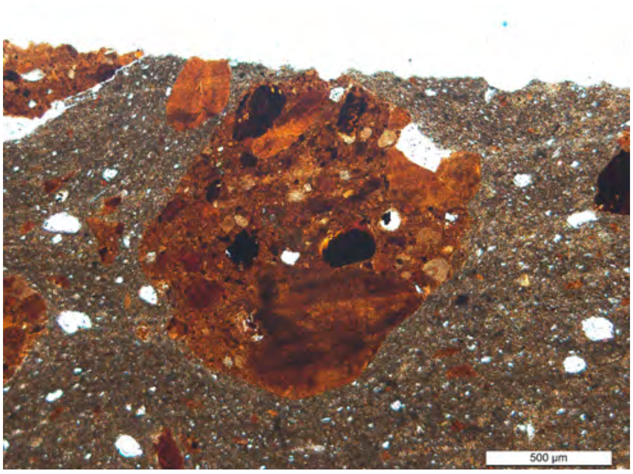



*NLT 267 G 650°C x100*




*NLT 267 G 850°C x2*

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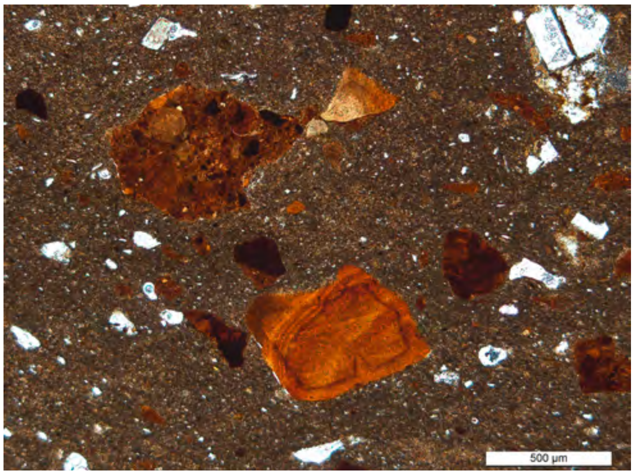


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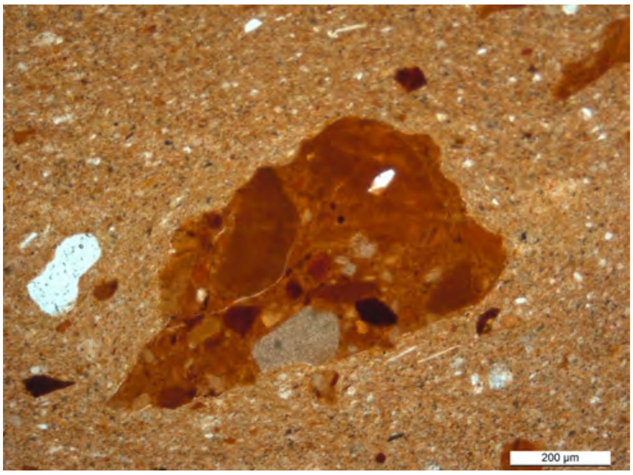


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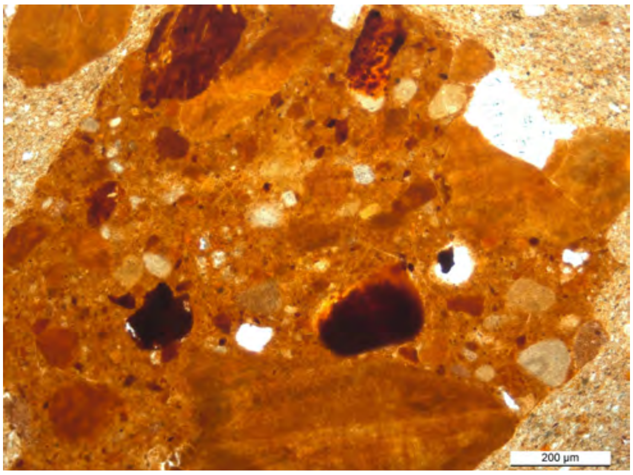


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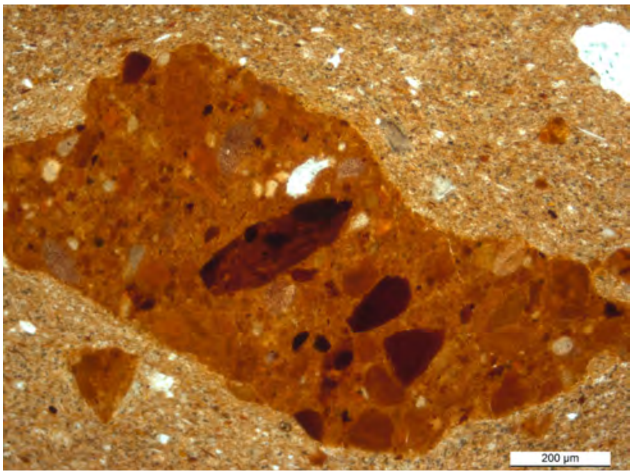


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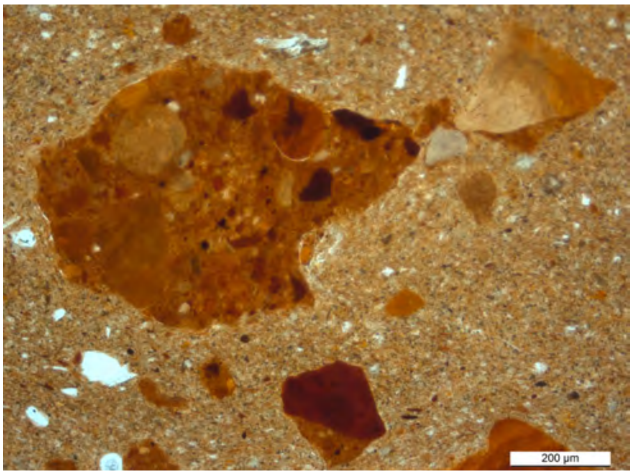


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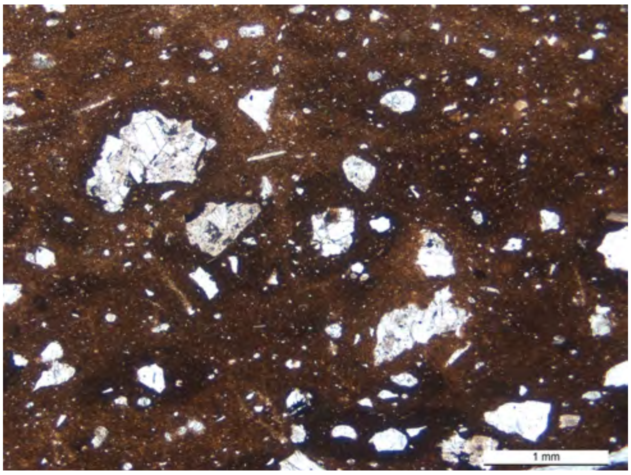
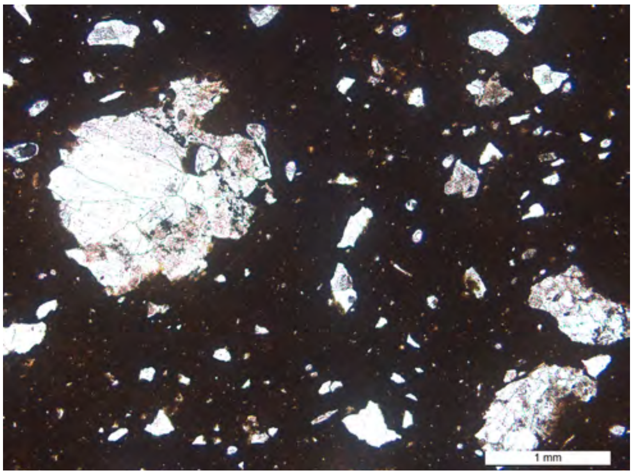



NLT 267 R 650°C x25

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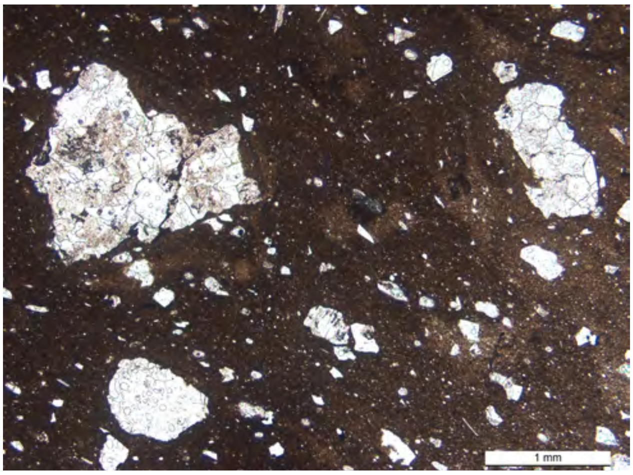


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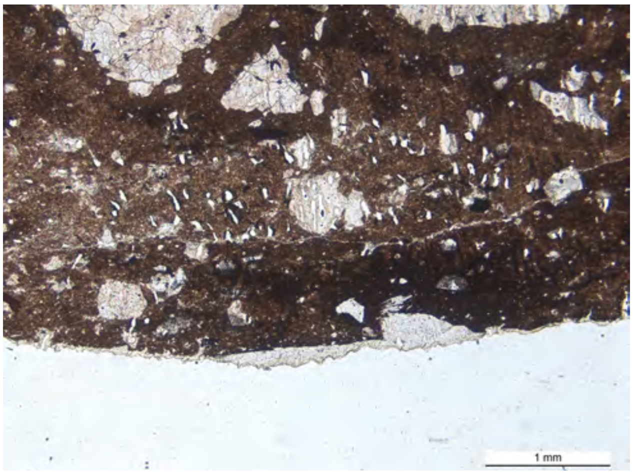


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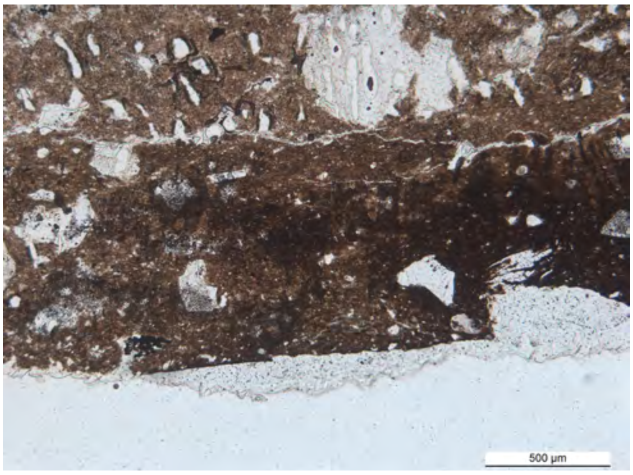


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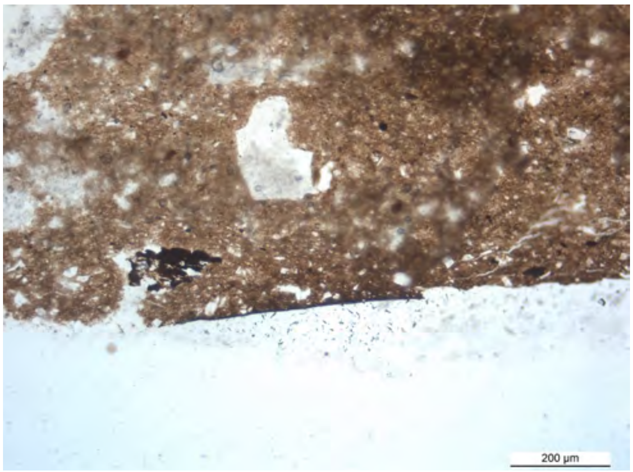


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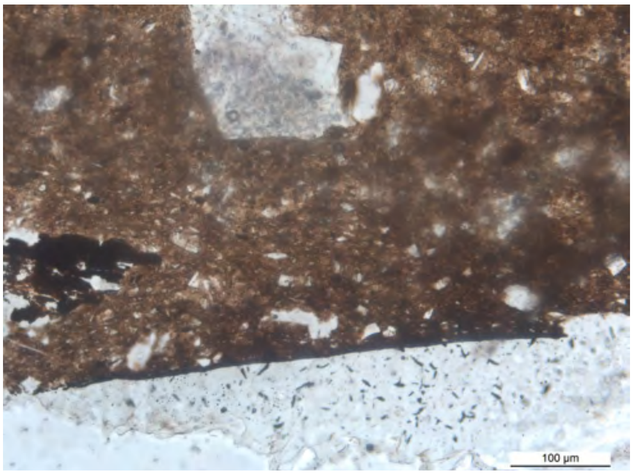


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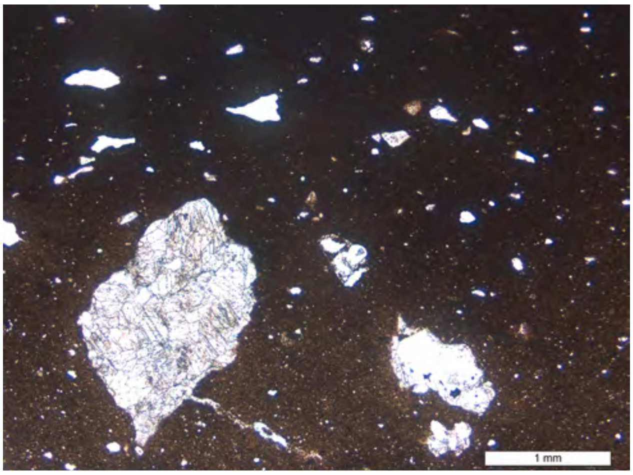
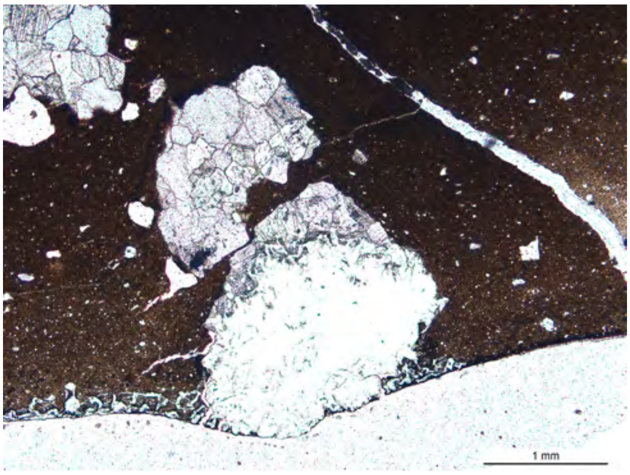
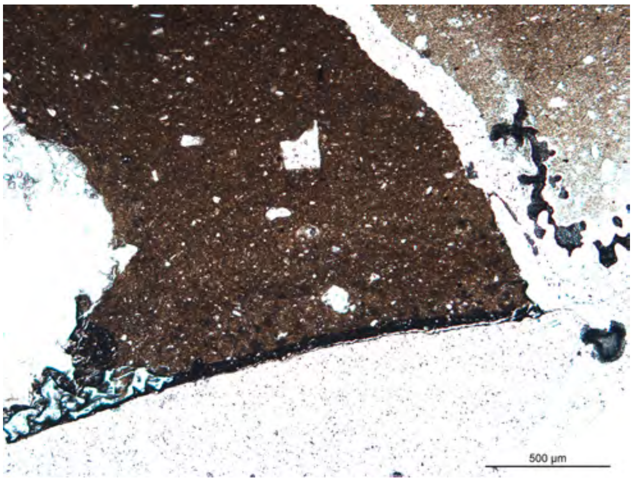


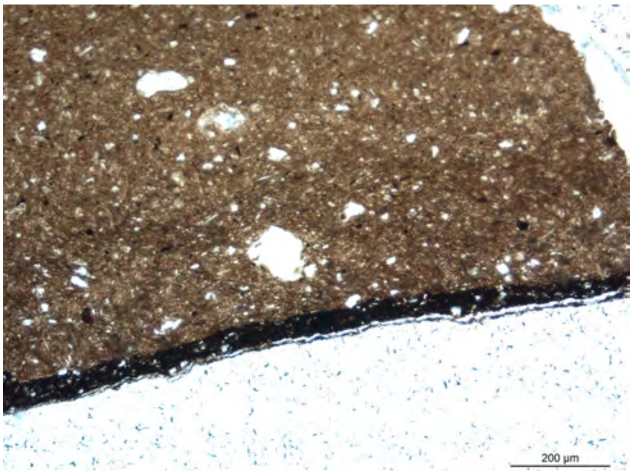

*Pot NLT 267 C FB x25*



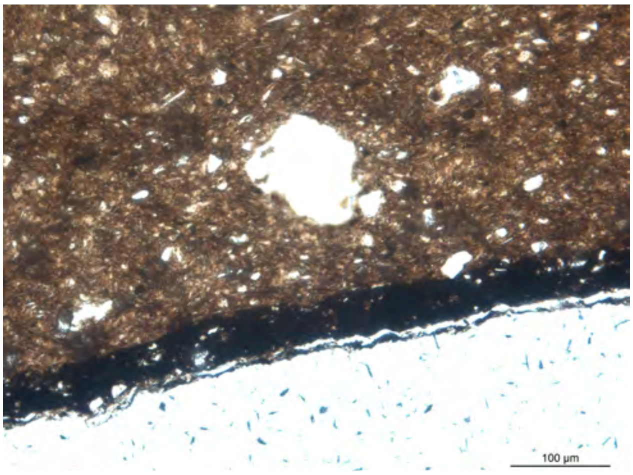

Pot NLT 267 C FBK x25



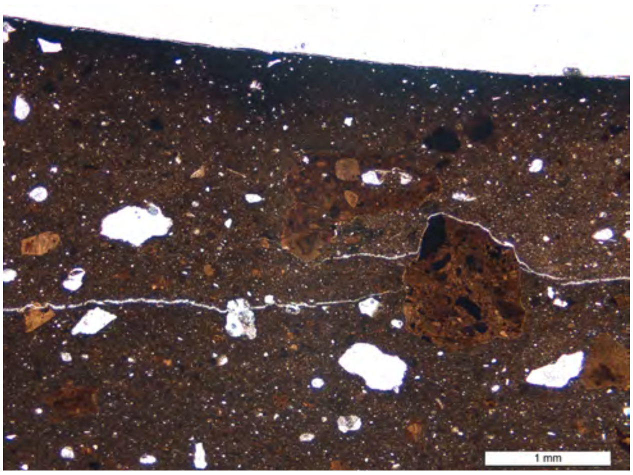

Pot NLT 267 C FBK x50




Pot NLT 267 C FBK x100




Pot NLT 267 C FBK x200

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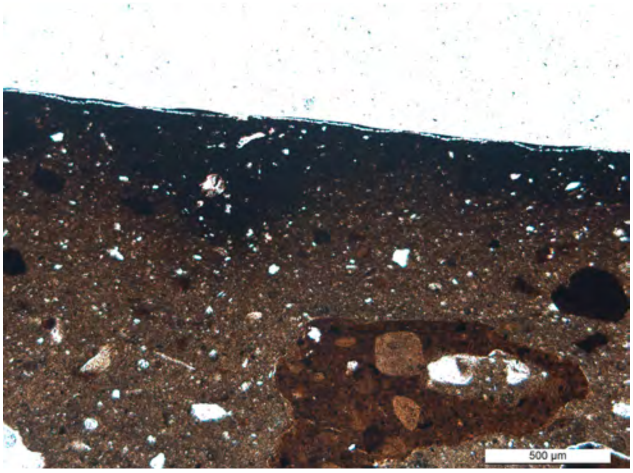
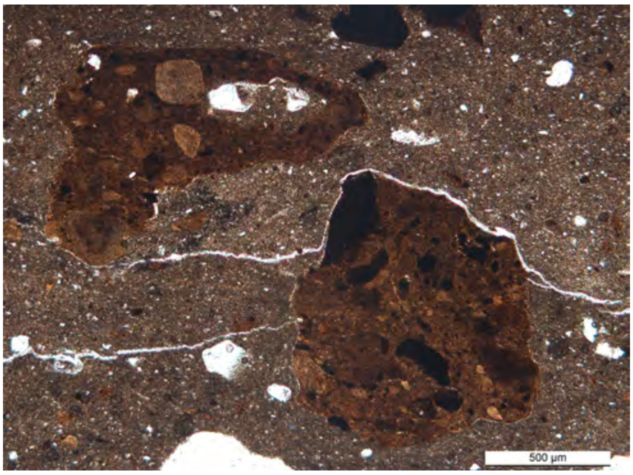



Pot NLT 267 G FBK x50

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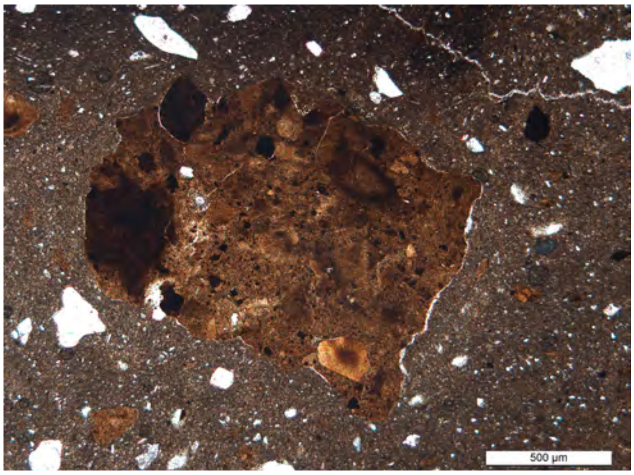
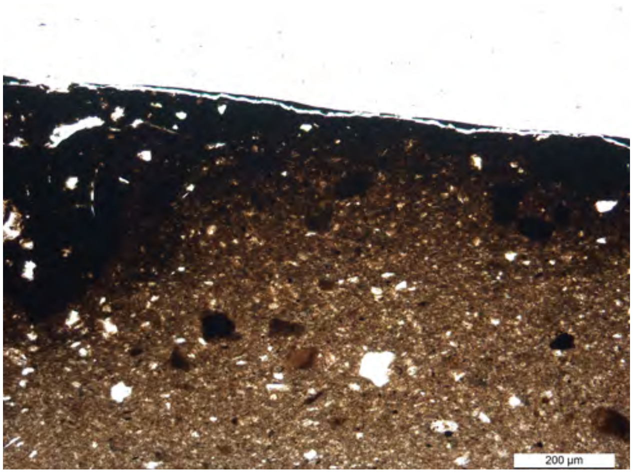



*Pot NLT 267 G FBK x50*

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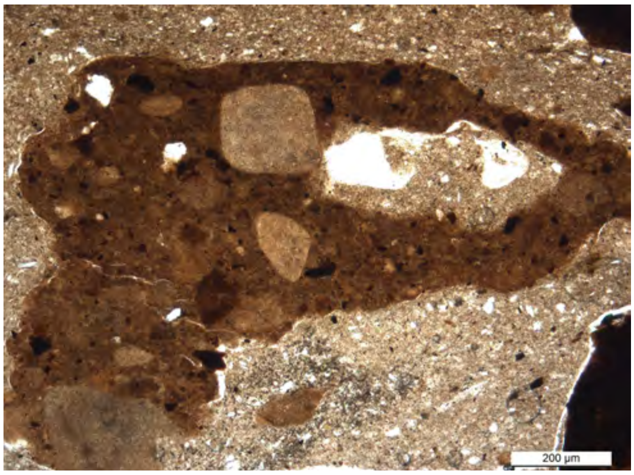


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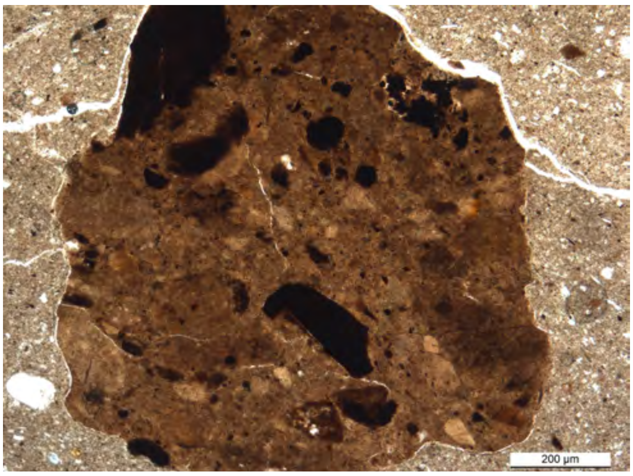


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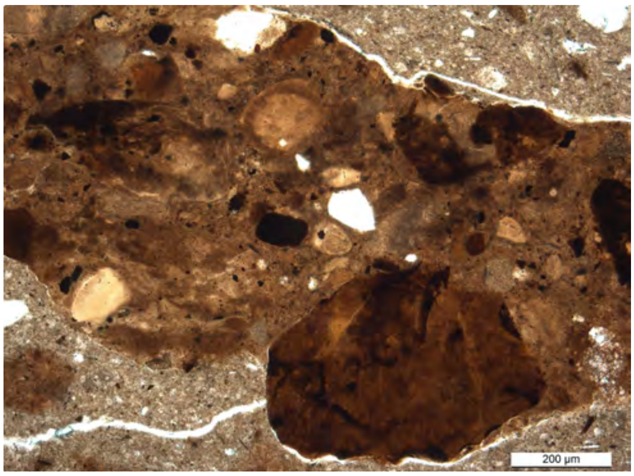
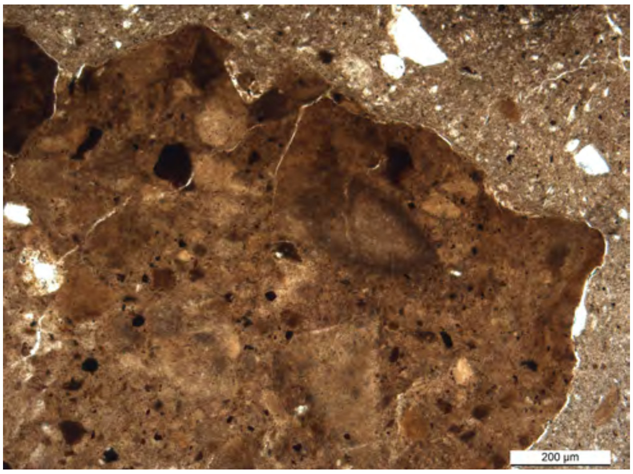



*Pot NLT 267 G FBK x100*

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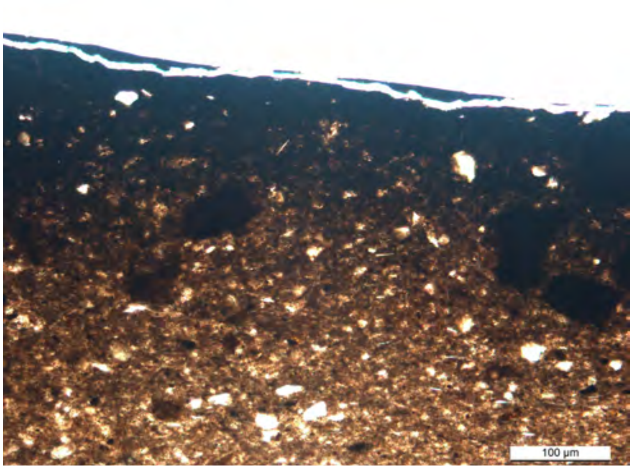
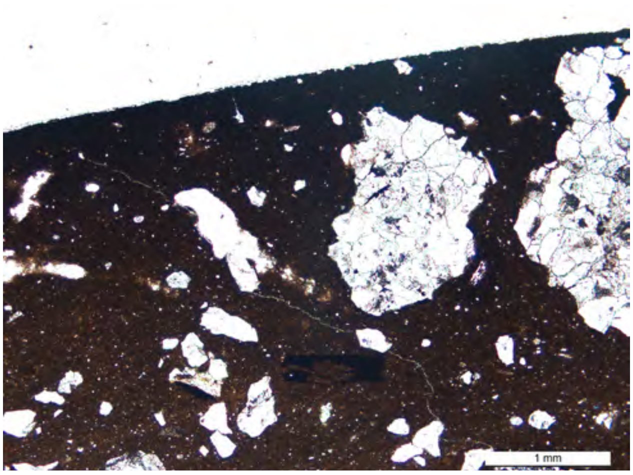
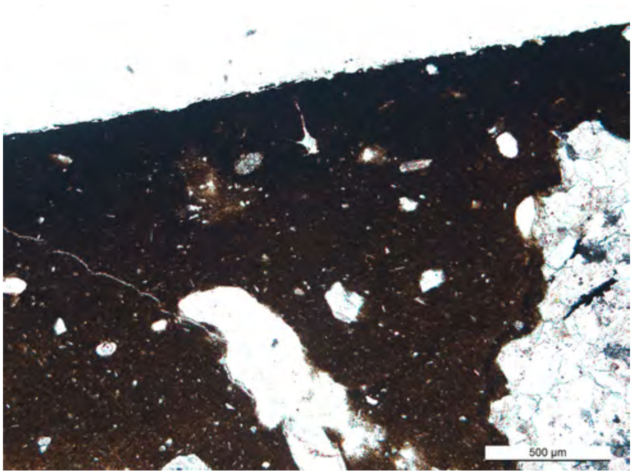


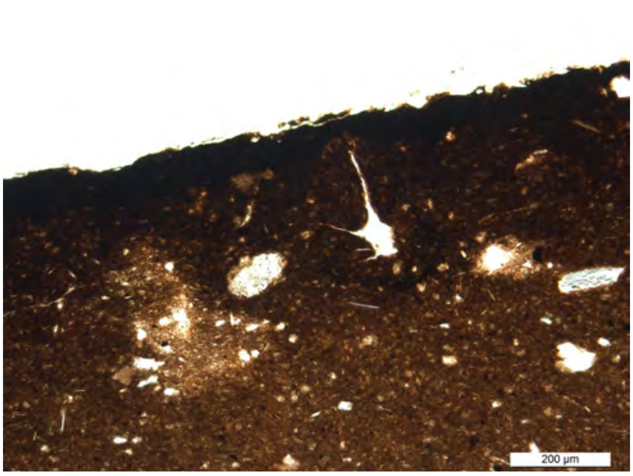

*Pot NLT 267 G FBK x200*



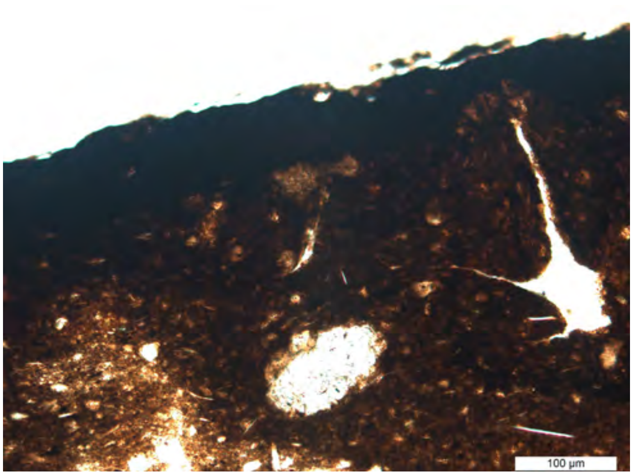

*Pot NLT 267 R FBK x25*




*Pot NLT 267 R FBK x50*




*Pot NLT 267 R FBK x100*




*Pot NLT 267 R FBK x200*

## 6. Conclusion

The firing experiments conducted in Gletterens led to interesting results. One of the most apparent is the metal-like surface of the polished pots and briquettes made from clay NLT 267, fired under a capsule. Indeed, the polishing of the raw clay compacted and set the minerals in the same direction, creating a smooth surface on which carbon platelets could fix in a parallel orientation (“glossy carbon”) during firing (Tobias 2013; Heimann & Maggetti 2014; Noll & Heimann 2016).

We also observed that the ceramics fired in bonfires, especially those close to the ground, were not fired long enough and are relatively fragile. As we had a limited amount of wood (~25 kg of firewood and one dried Christmas tree (pine)), the fires stopped when we ran out of wood. Future experiments should consider to have more wood available in order to be more flexible with the duration of the experiment.

The combination of different analyses such as XRD, SEM, binocular images and microscopy enables to better understand the processes that occurred during the firing of the ceramics. The black layer formed during firing in a reducing atmosphere (FBK) could thus be investigated in several ways, giving information about its thickness, micro-structure and composition.

Regarding the SEM analysis, no precise quantitative analysis of the elements could be performed, due to the C-coating and the coarse topography of the samples, although the SEM-EDS distribution maps indicate that the surfaces of the pots are richer in C than their cross-sections. (In order to better determine the presence of carbon, in particular at the surface of the pots, we suggest using a Au-coating for future analyses. However, there are other disadvantages to this covering.) A SEM analysis comparing polished with non-polished samples from the same pot could also bring important information about the surface layer.

## 7. References


Heimann, R.B., & Maggetti, M. 2014. Ancient and Historical ceramics, Stuttgart.

Maggetti, M. 1982. Phase analysis and its significance for technology and origin. In: Olin, J.S. and Franklin, A.D. (eds.) Archaeological ceramics. Washington: 121-133.

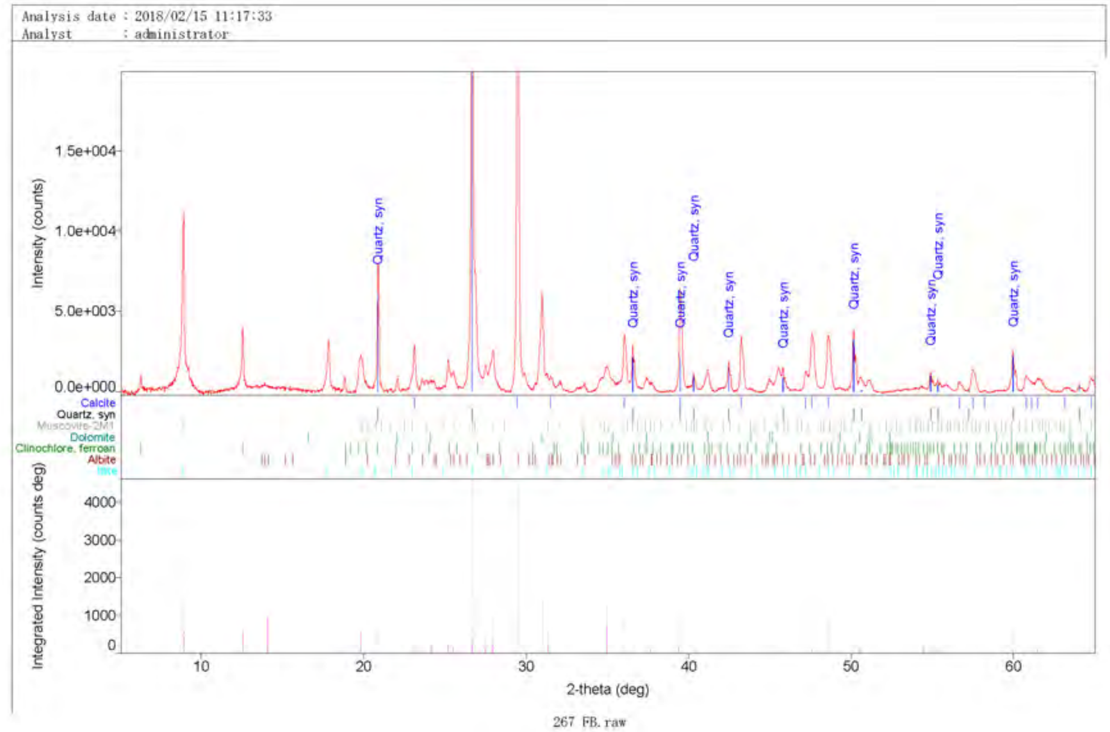
Maggetti, M., Neururer, Ch., Ramseyer, D. 2011. Temperature evolution inside a pot during experimental surface (bonfire) firing. Applied Clay Science 53: 500-508.

Noll, W., & Heimann, R.B. 2016. Ancient Old World Pottery: Materials, technology, and decoration, Stuttgart.

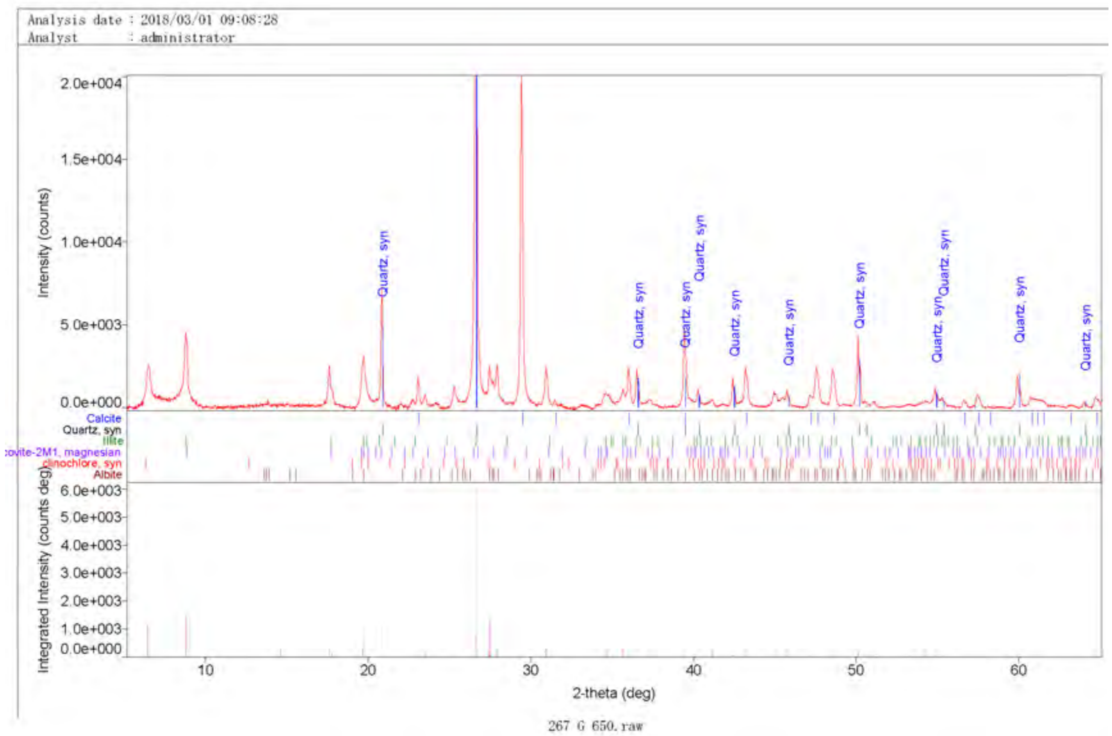
Tobias, W. 2013. Schwarze Keramik in Portugal. Books on Demand.

# 8. Appendix 1: Diffractograms with phase identification

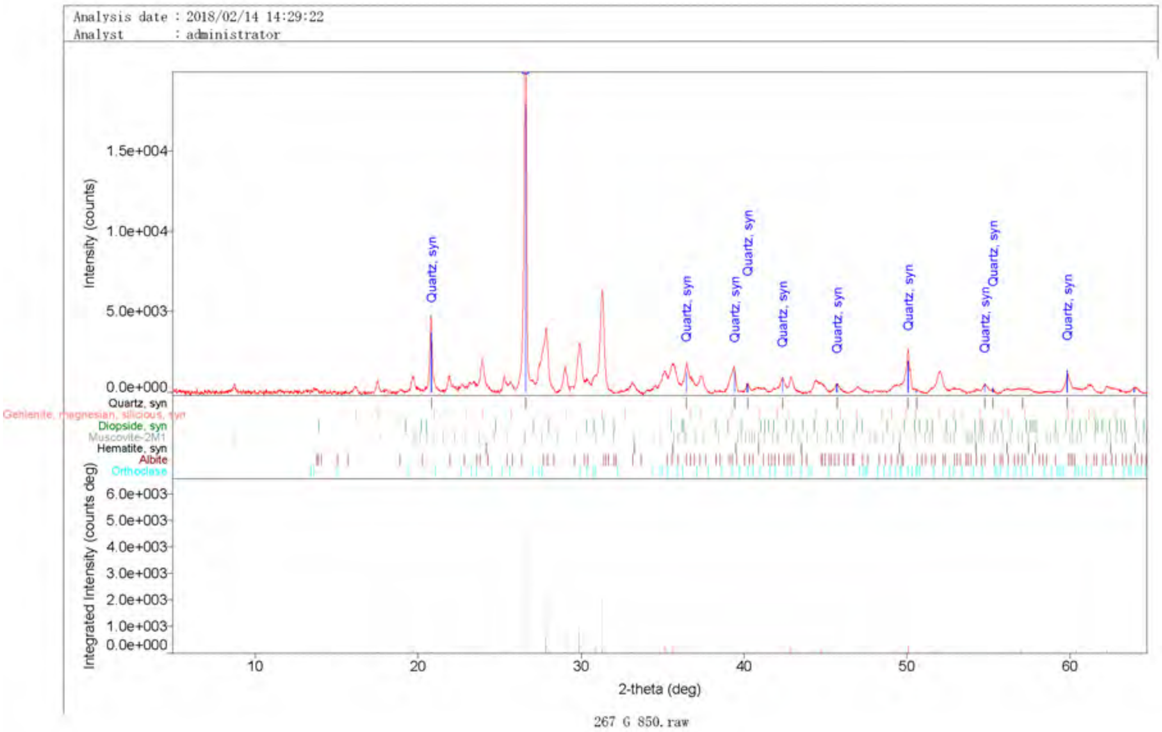
NLT 267 FB



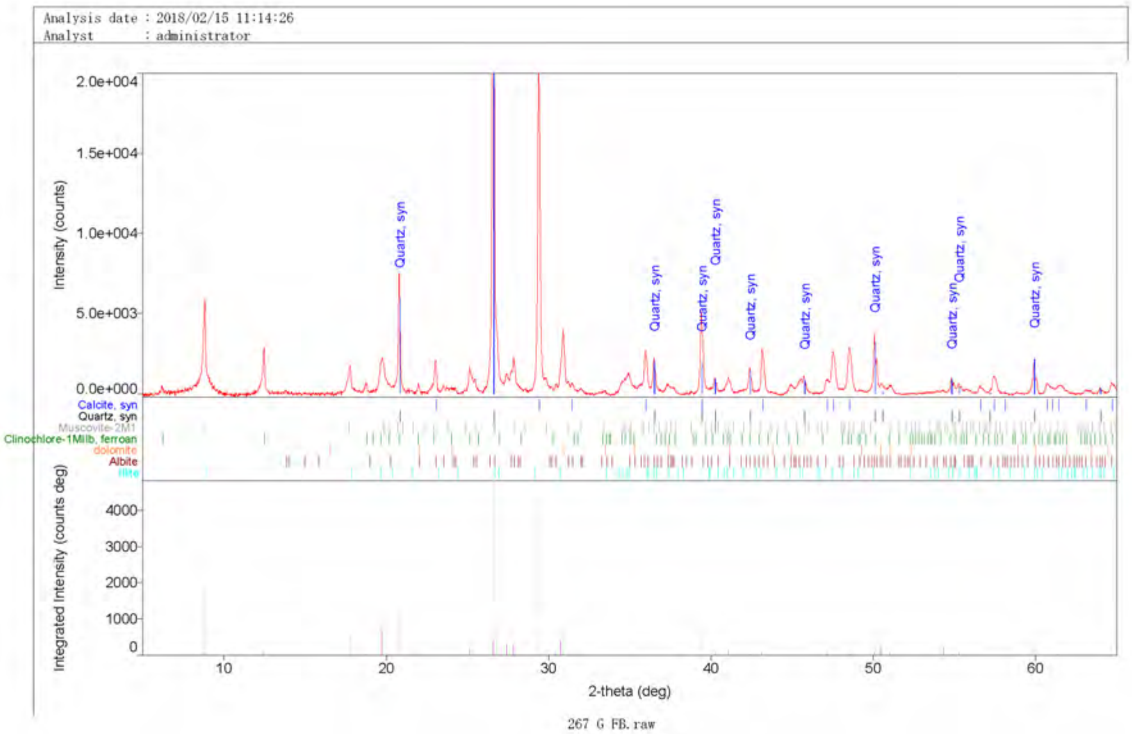

NLT 267 G 650°C



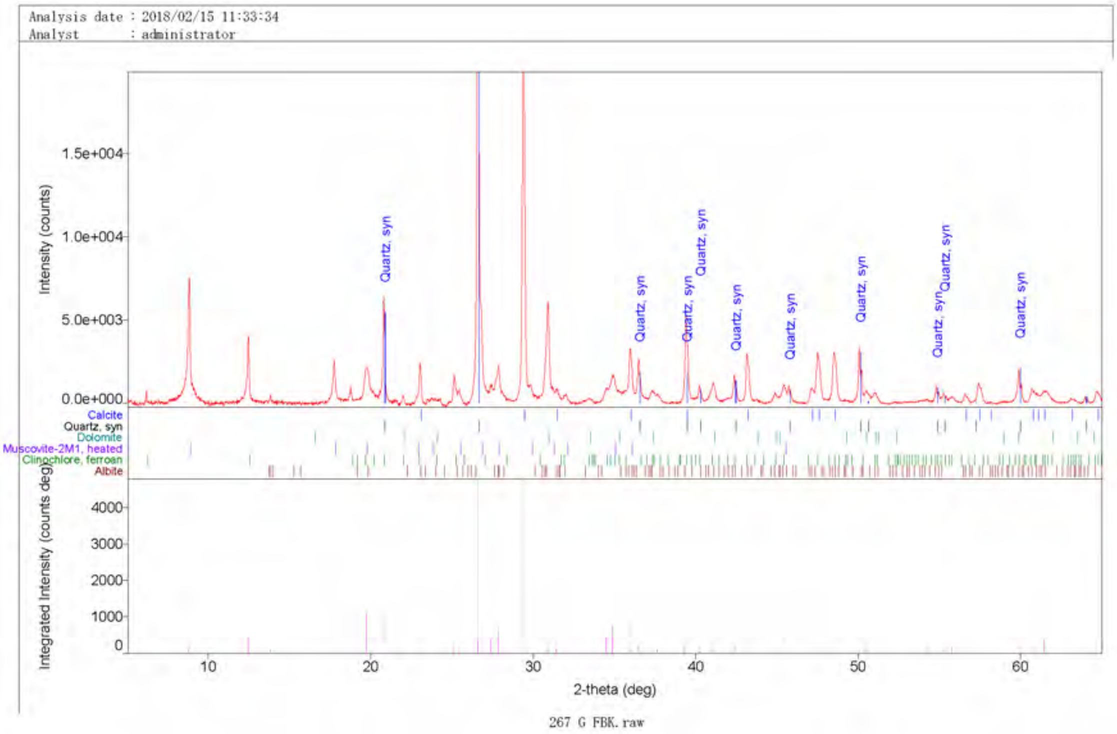
NLT 267 G 850°C



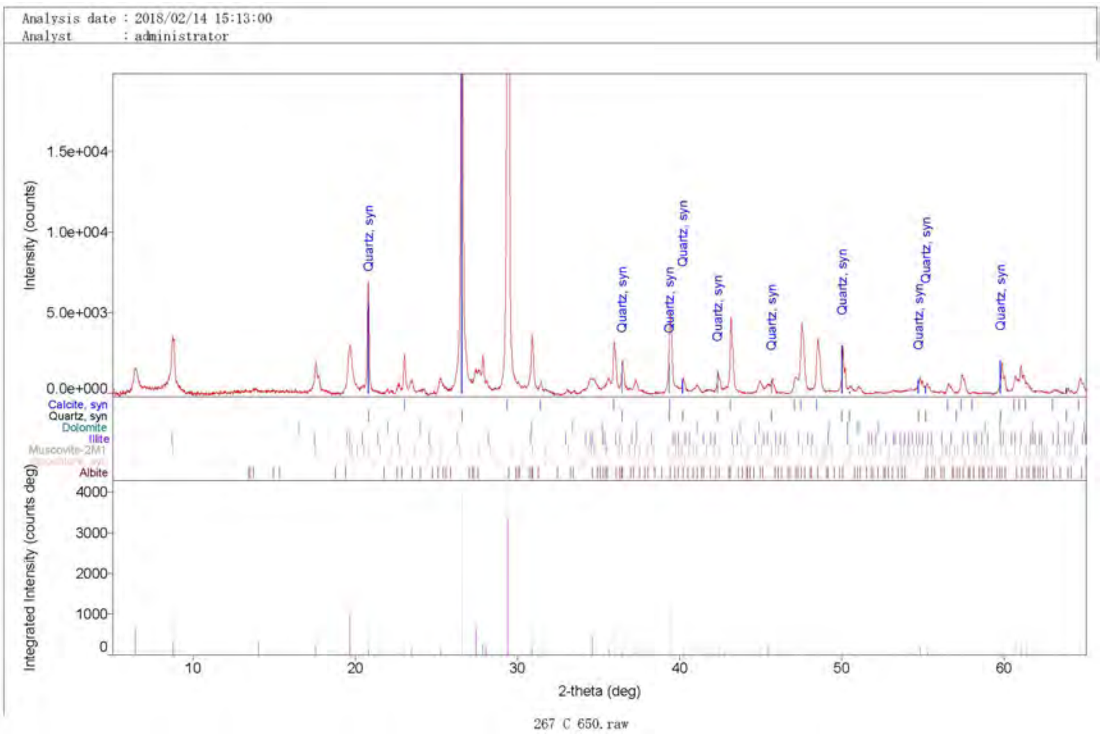

NLT 267 G FB



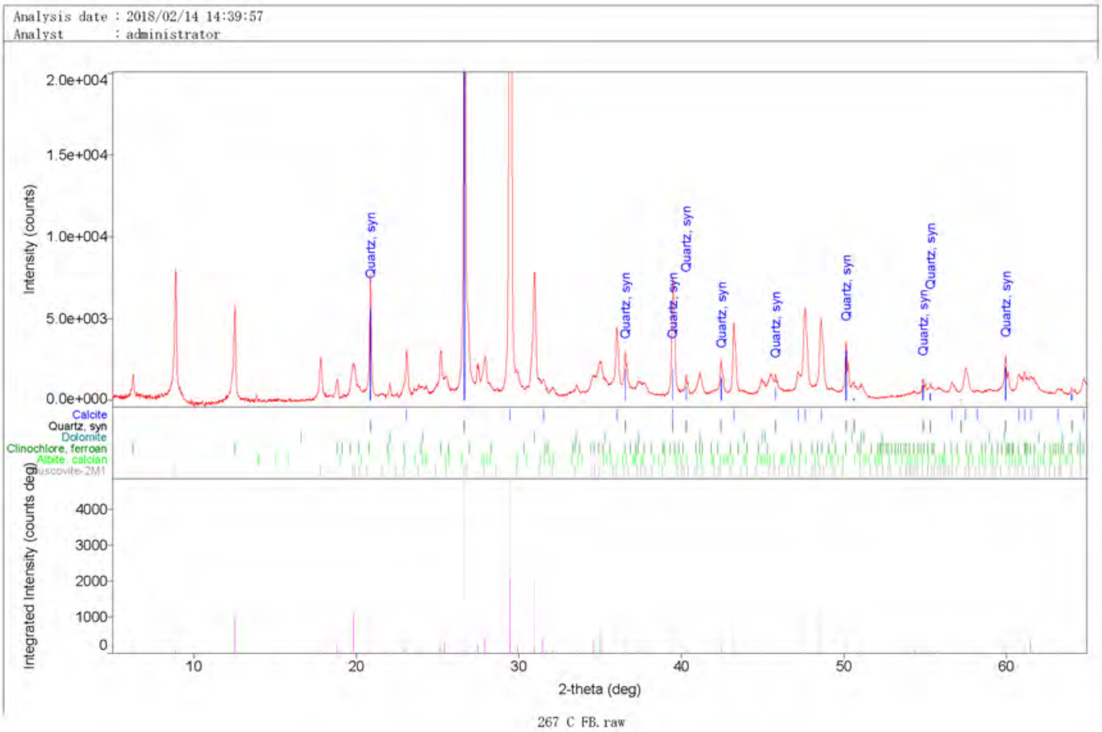
NLT 267 G FBK



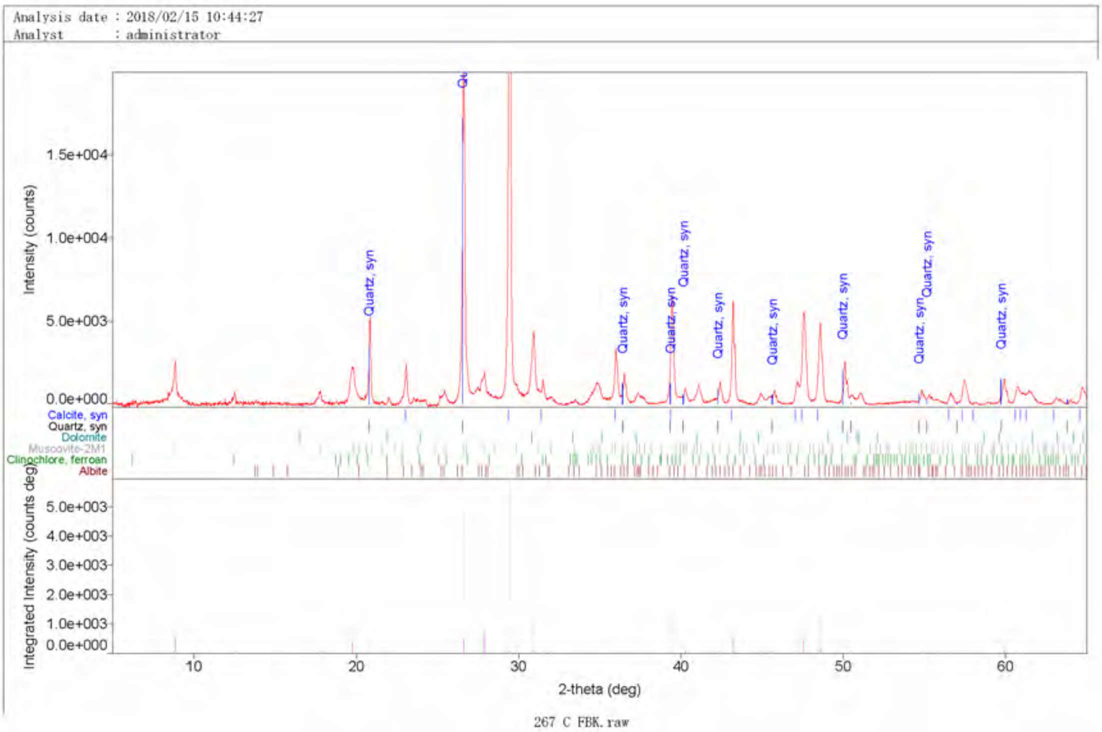

NLT 267 C 650°C



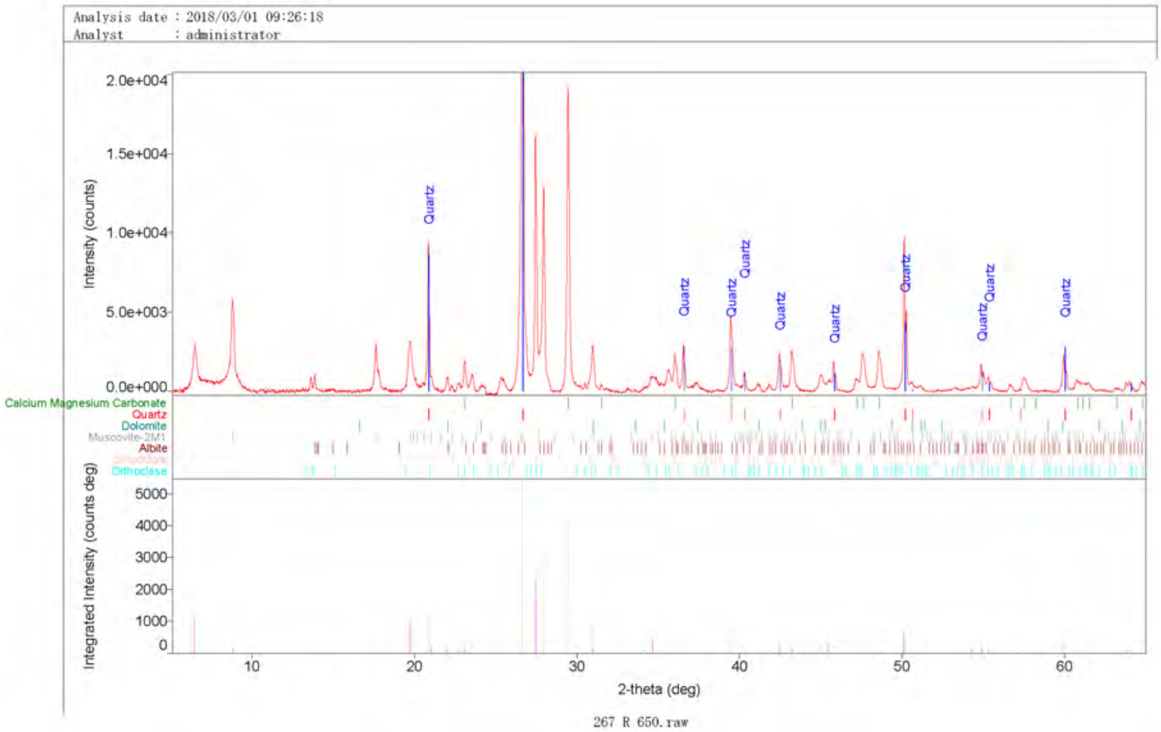
NLT 267 C FB



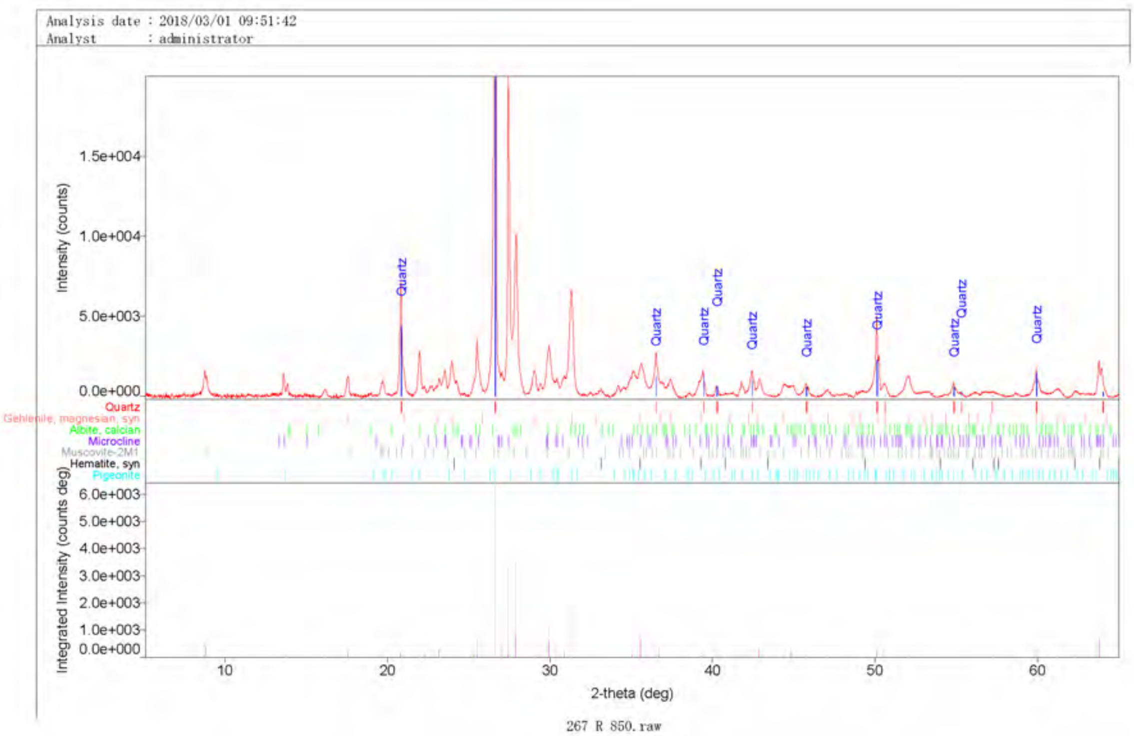

NLT 267 C FBK



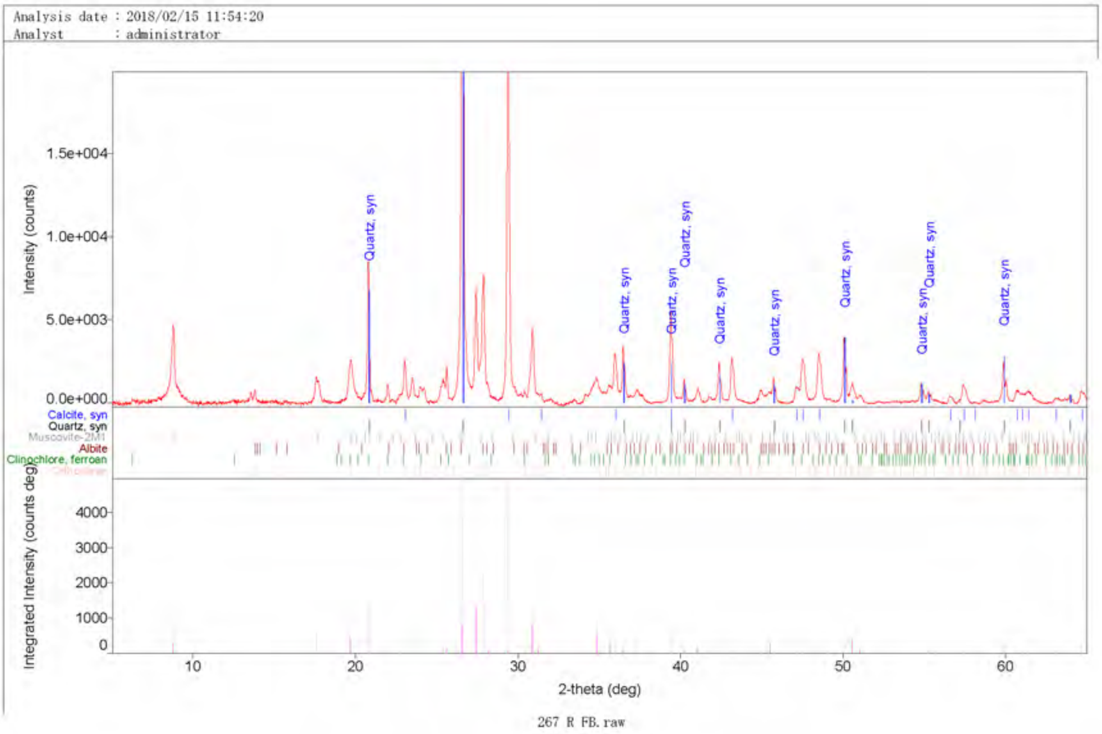
NLT 267 R 650°C




NLT 267 R 850°C



NLT 267 R FB

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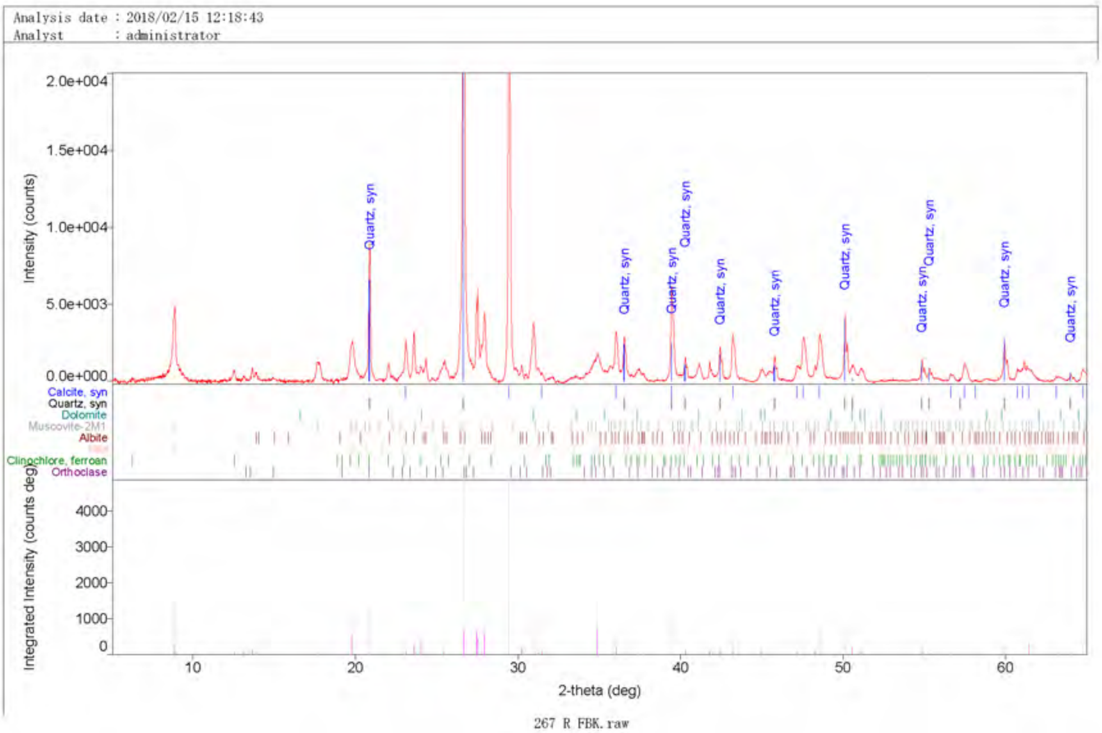
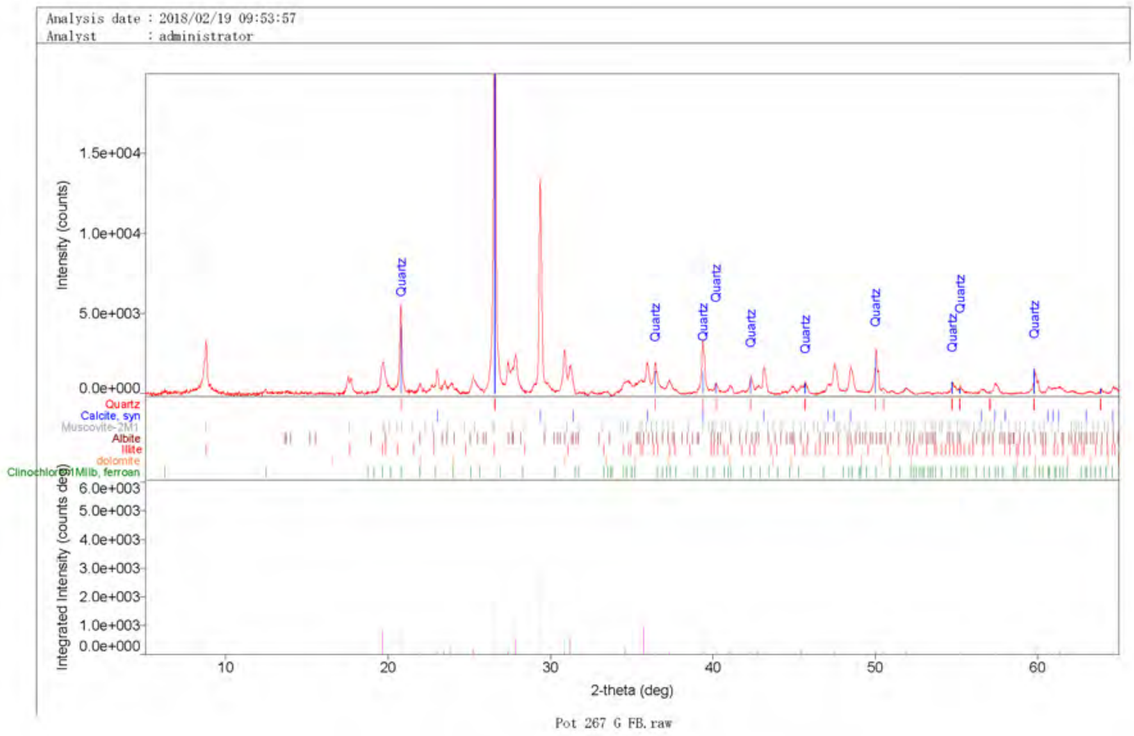


Pot NLT 267 G FB

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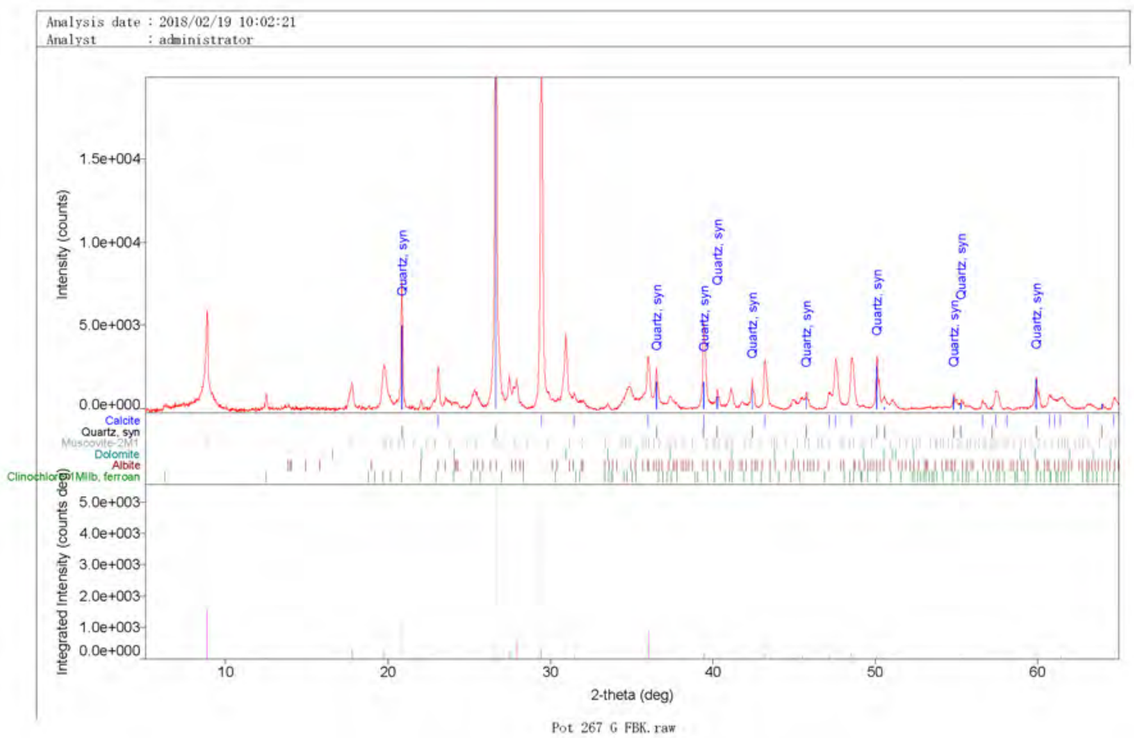

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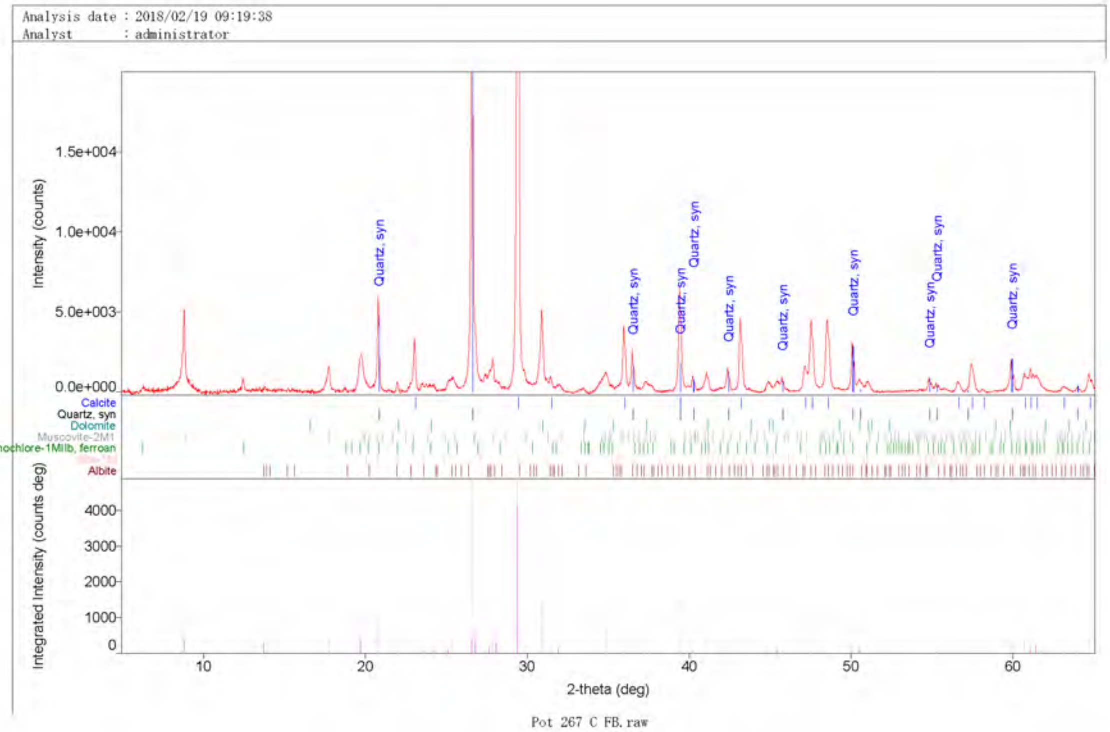


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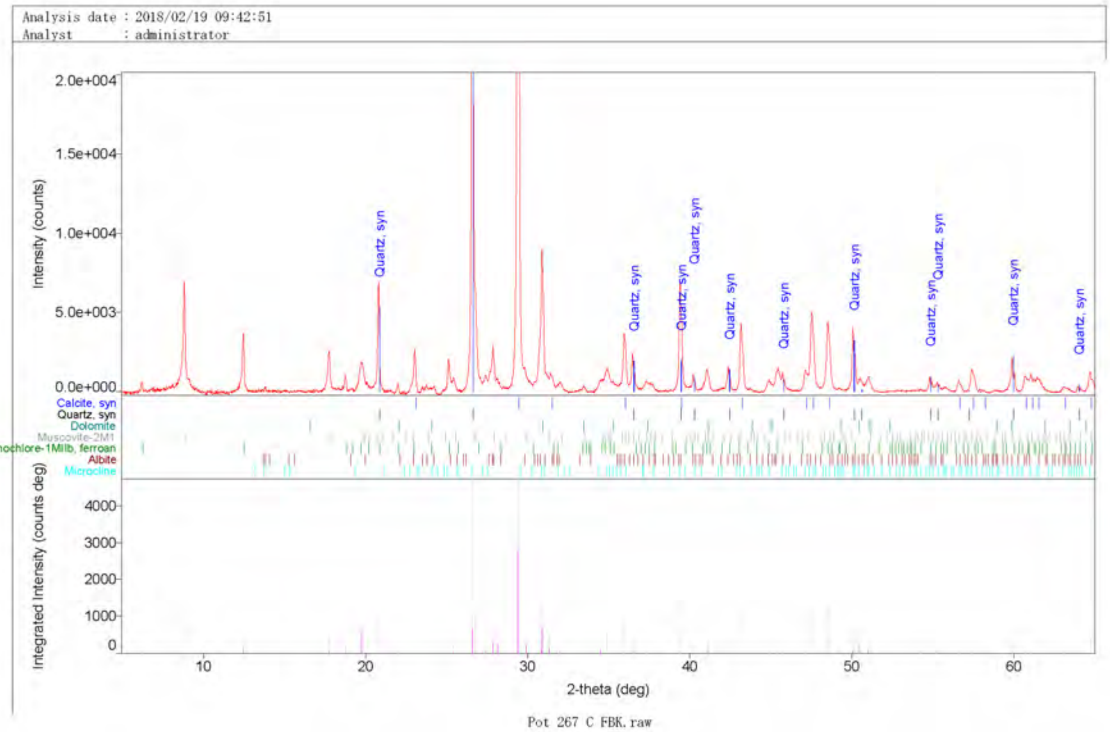

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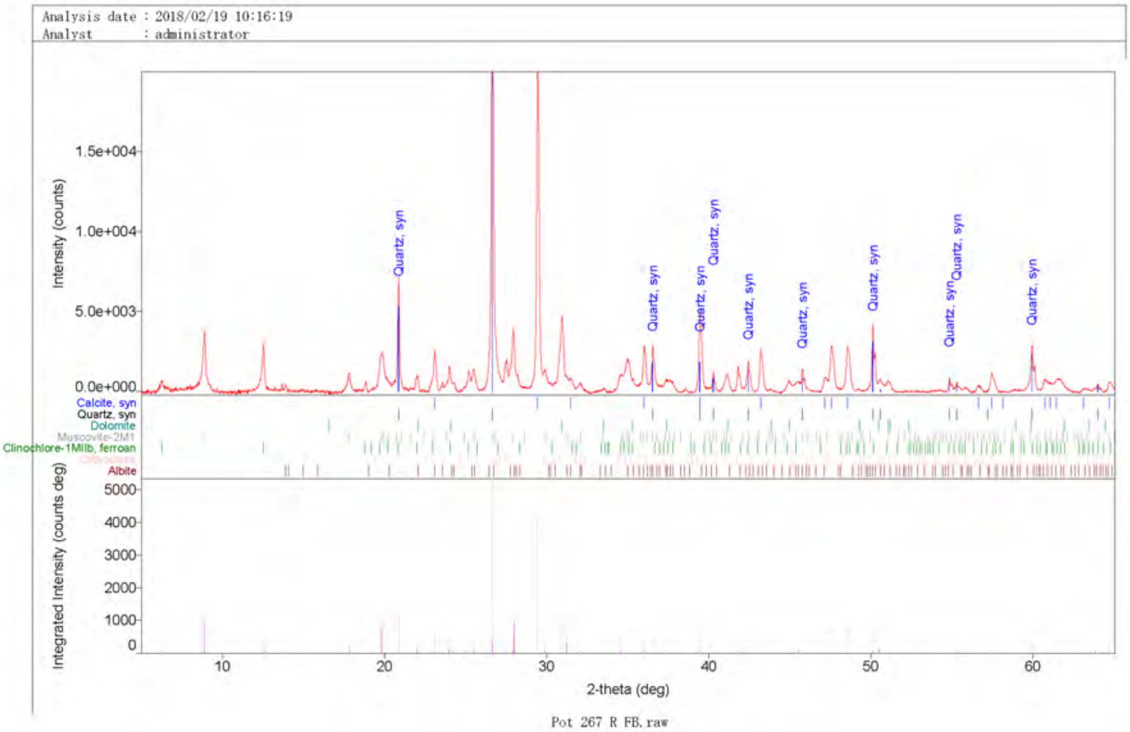
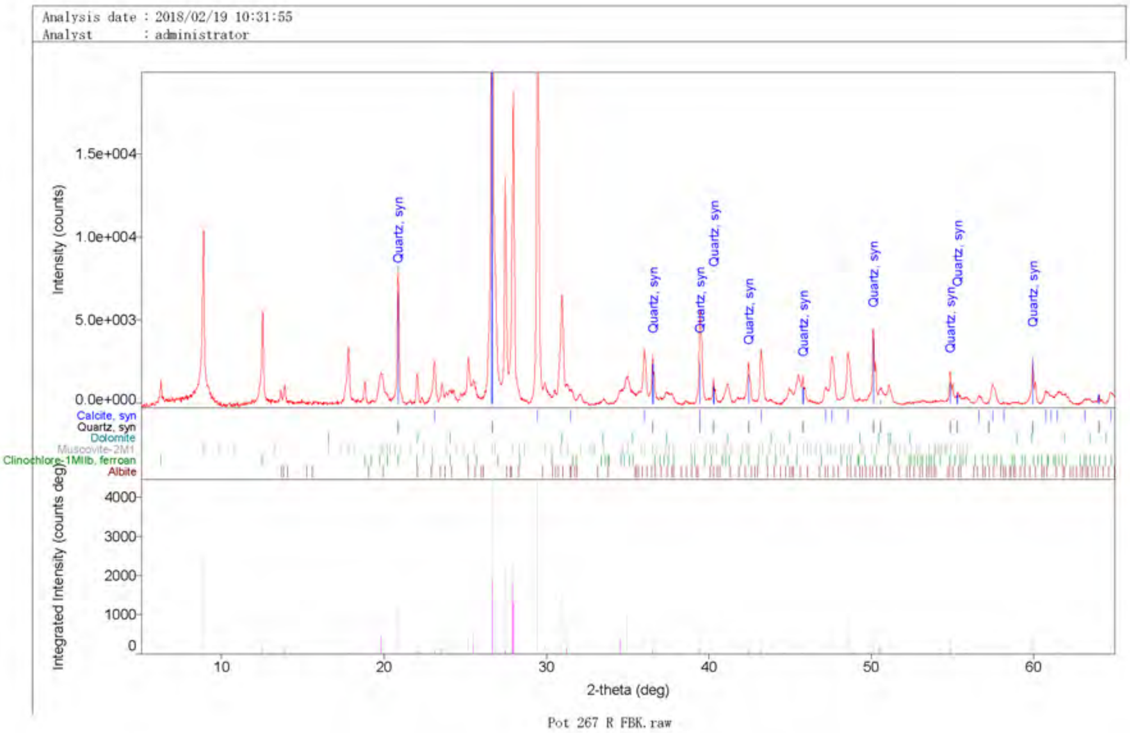



Pot NLT 267 R FBK

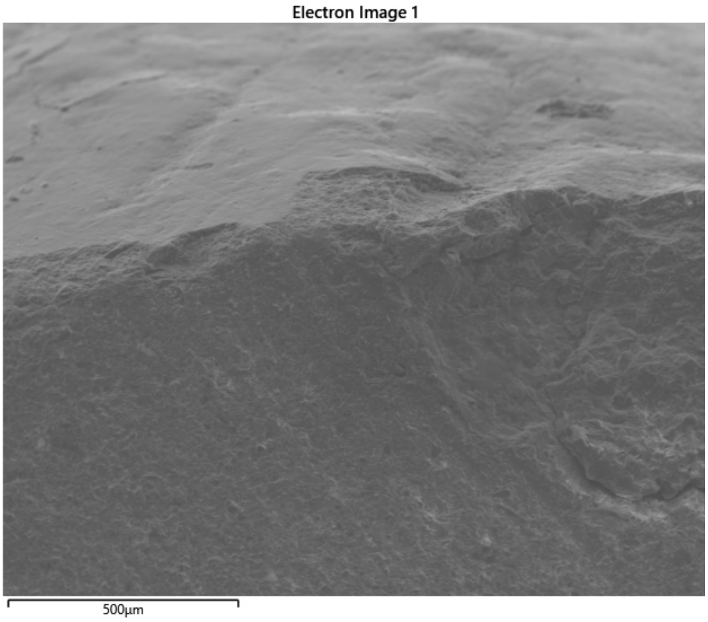


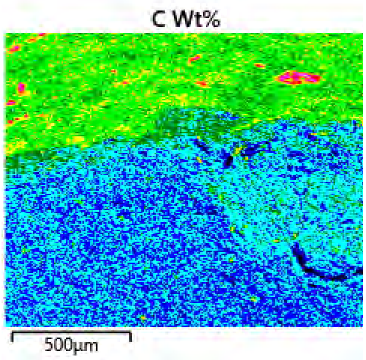
# 9. Appendix 2: Results of the SEM-EDS analysis (by Ch. Neururer)

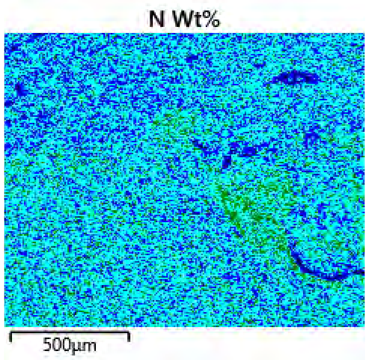
The following figures show the results of the SEM-EDS analyses, performed on two samples (NLT 267 G FBK and NLT 267 R FBK) at different sites with different measuring parameters. The distribution maps indicate clearly that the surfaces of the samples are richer in C than their cross-sections.

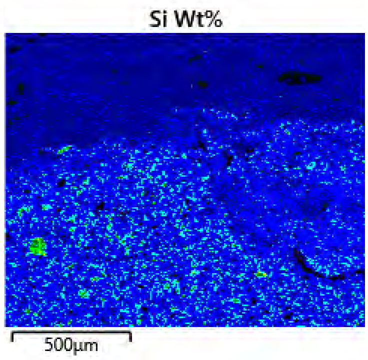
**NLT 267 G FBK ("NLT267_1")**

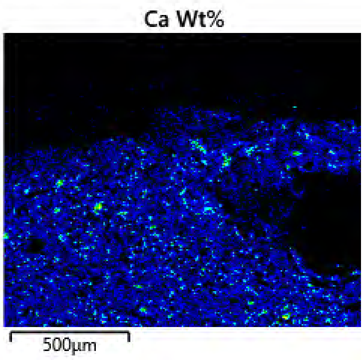
Site 1

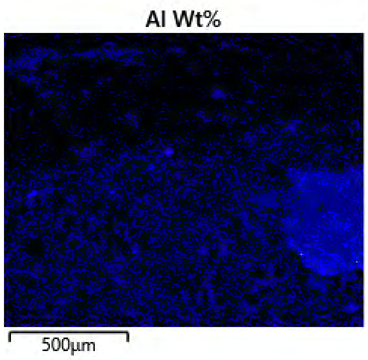


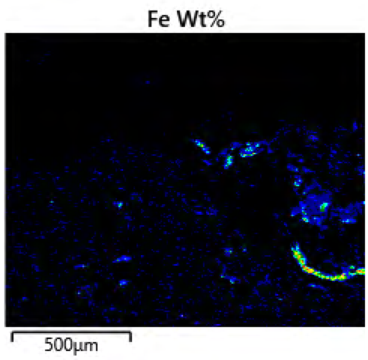



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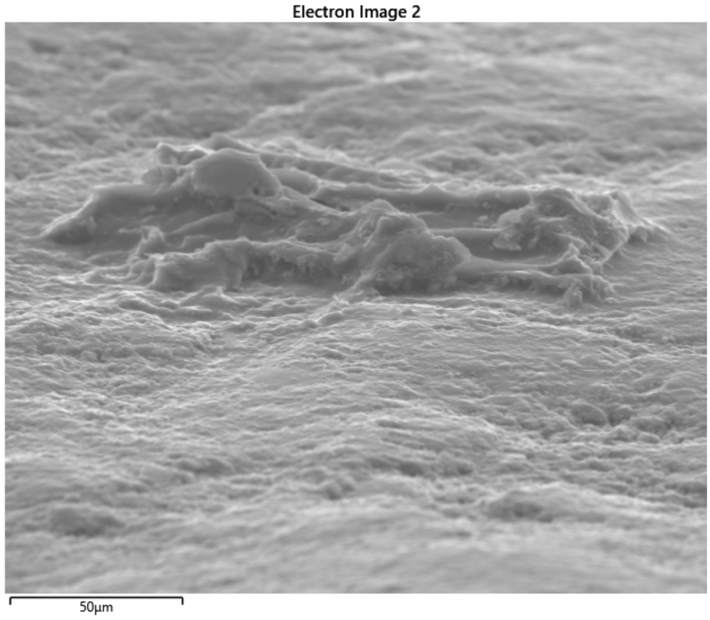
Electron Image 2
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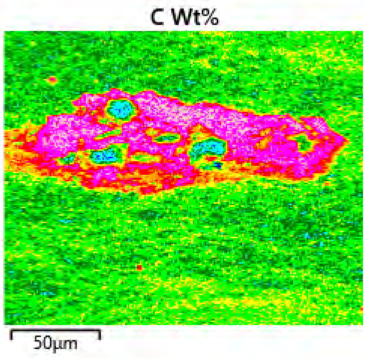
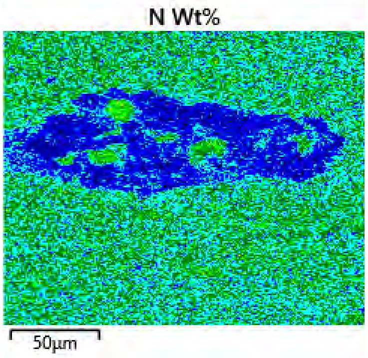
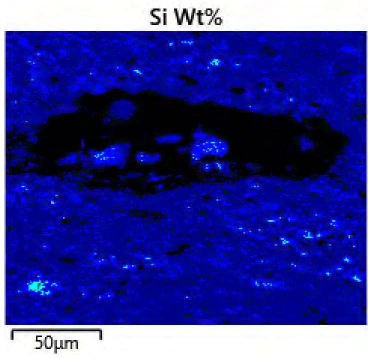

C Wt%
50µm


N Wt%
50µm


Si Wt%
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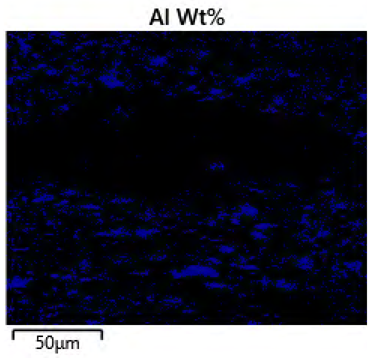
Al Wt%
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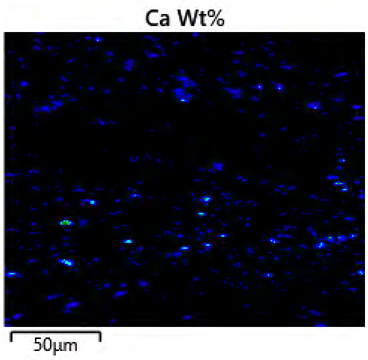
Ca Wt%
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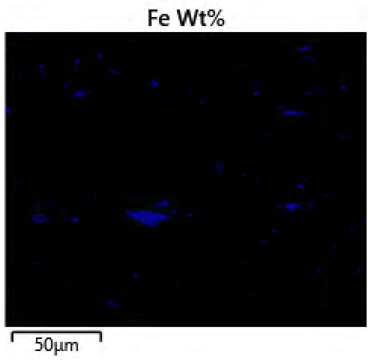
Fe Wt%
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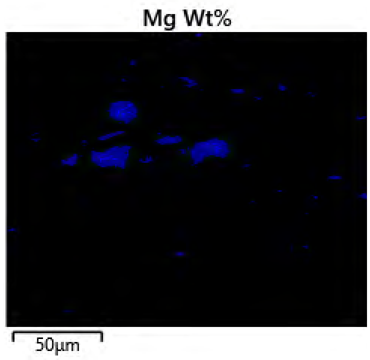
Mg Wt%
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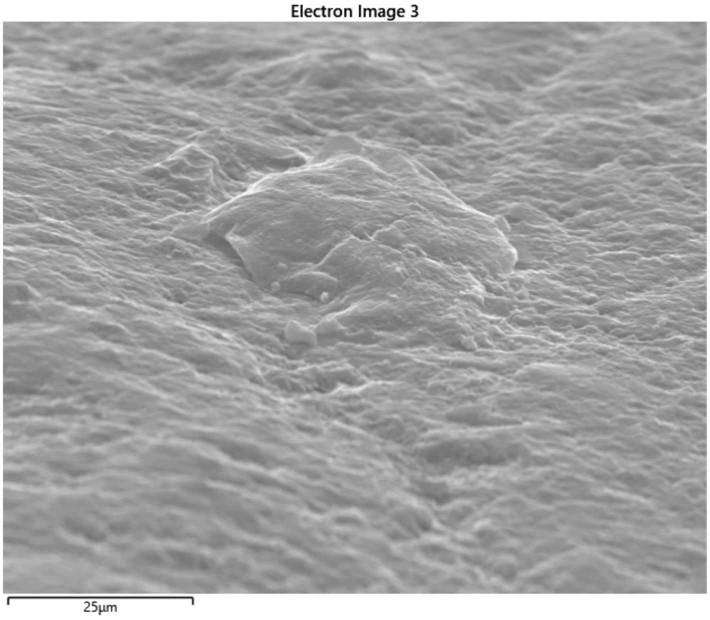
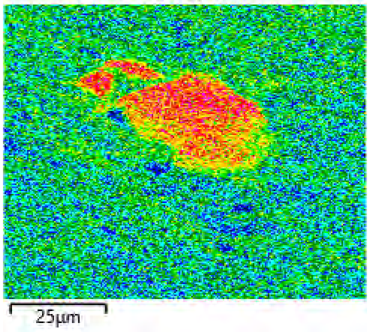
Site 4

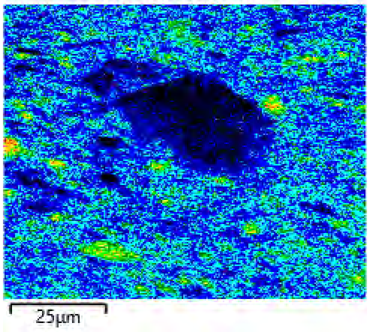
Electron Image 3



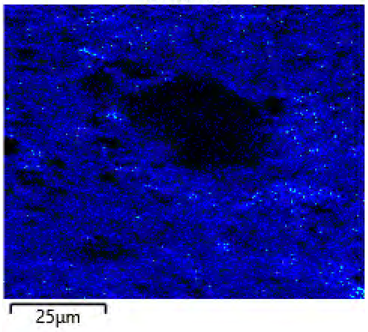

C Wt%




Si Wt%




Al Wt%

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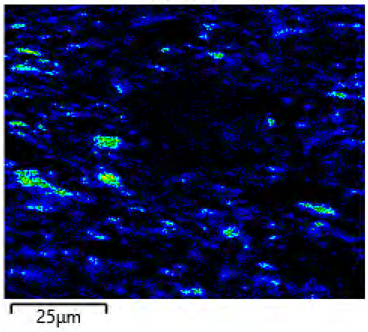
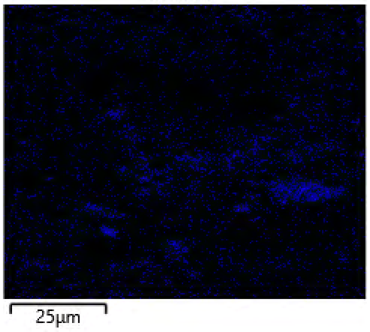
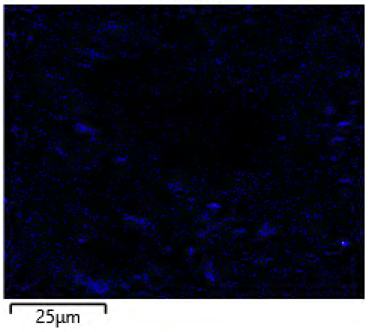


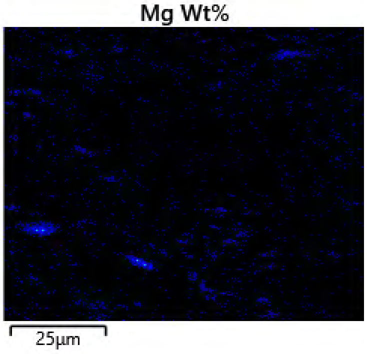

K Wt%



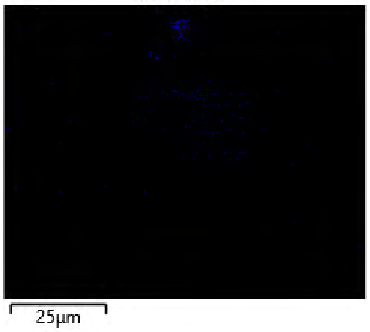

Fe Wt%



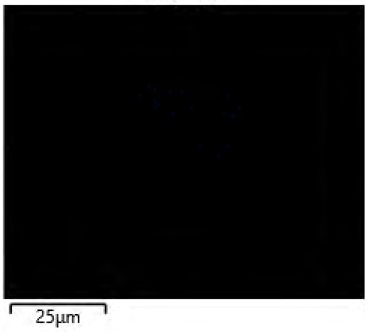

Mg Wt%



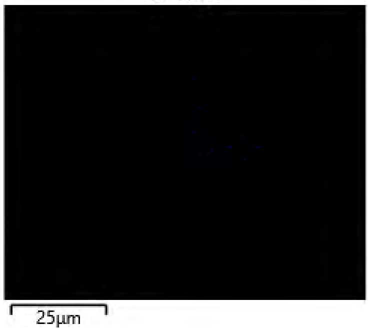

Na Wt%




S Wt%

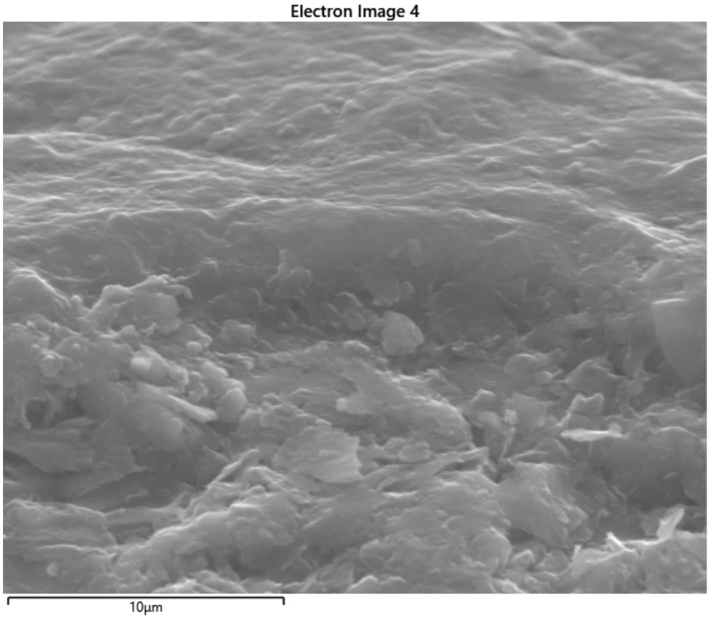



Cl Wt%



Site 5

Electron Image 4

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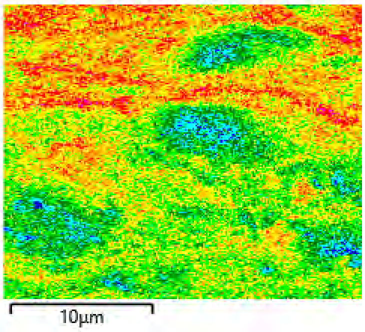


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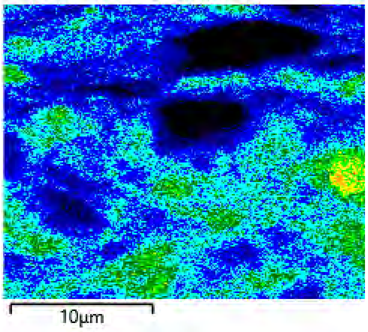


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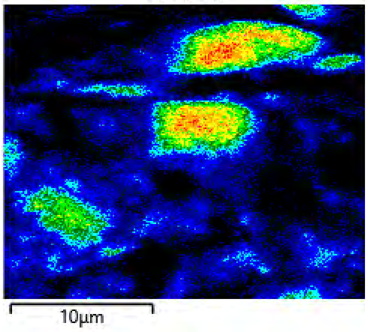


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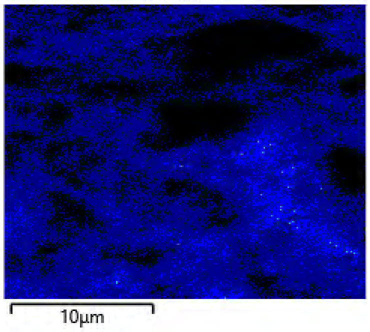
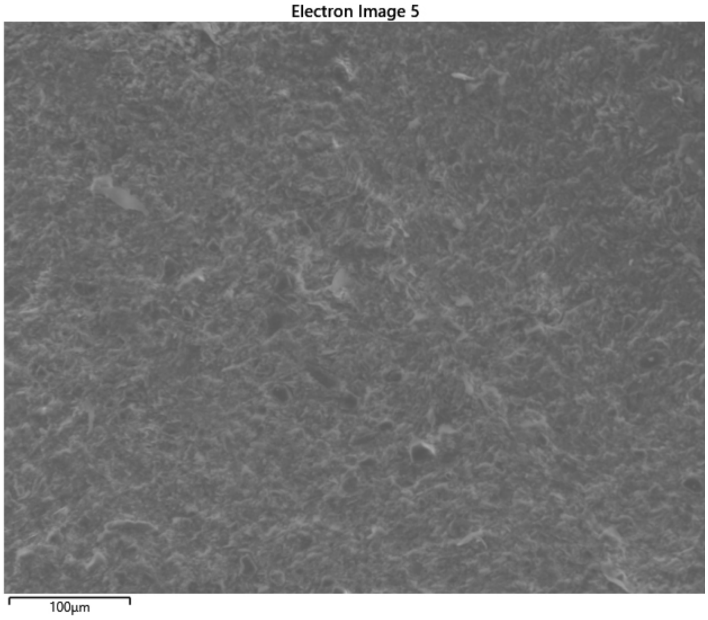


Site 6

Electron Image 5

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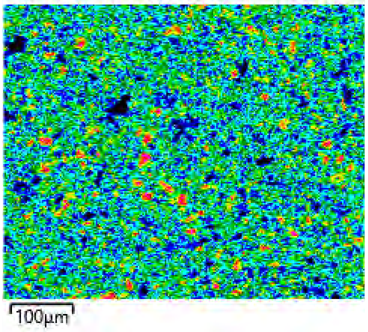
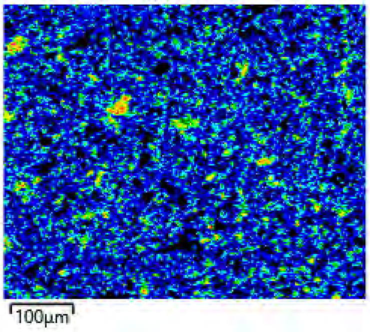
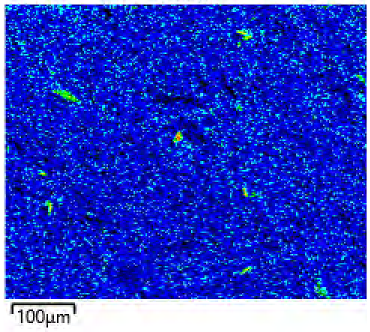


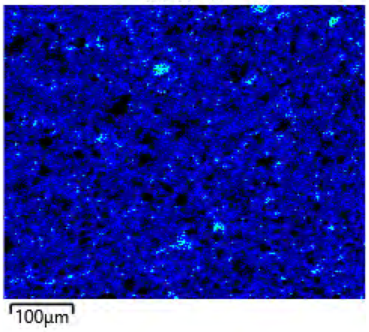

Ca Wt%



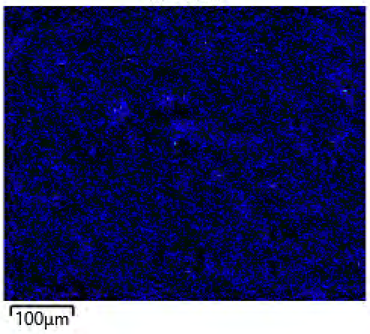

C Wt%



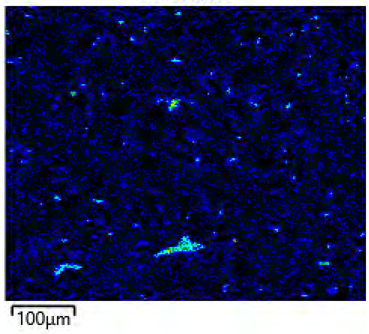

Al Wt%




N Wt%

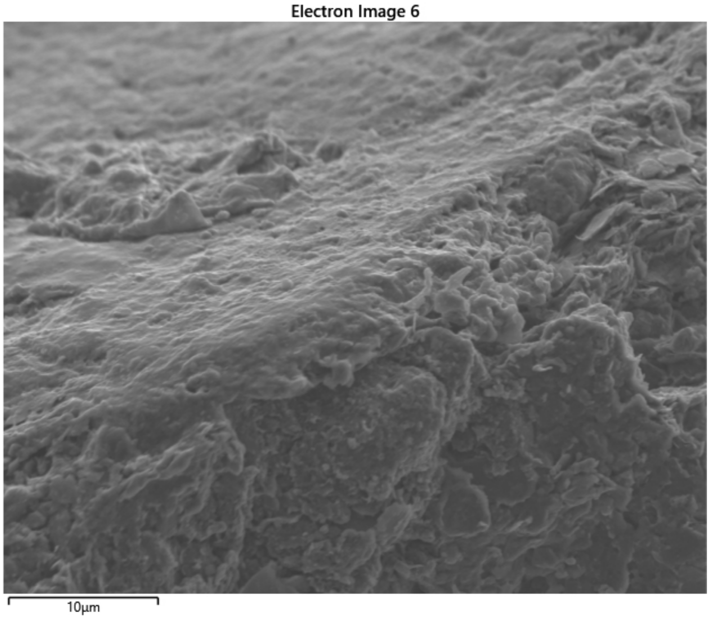



Fe Wt%

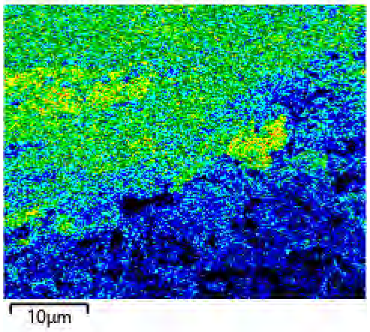


Site 7

Electron Image 6




C Wt%

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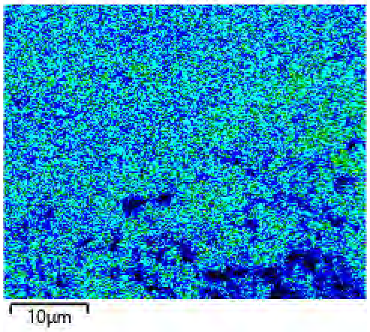


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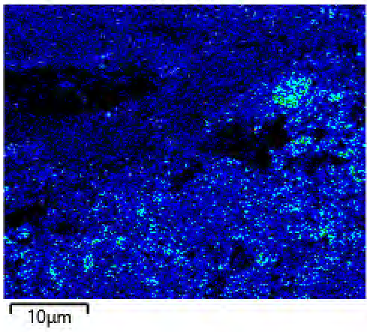
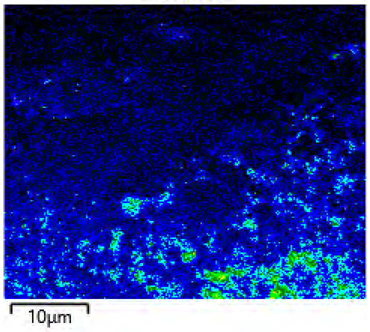



Ca Wt%

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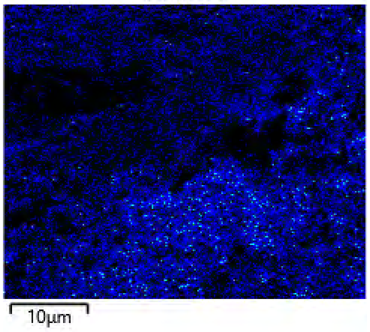


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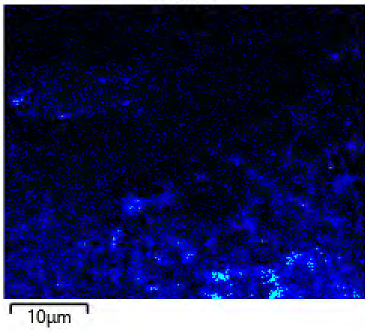


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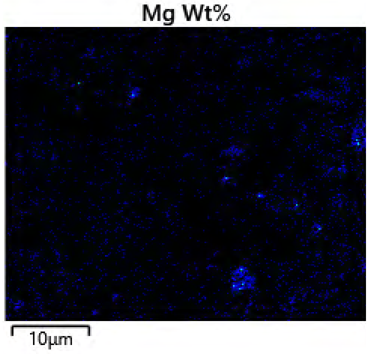
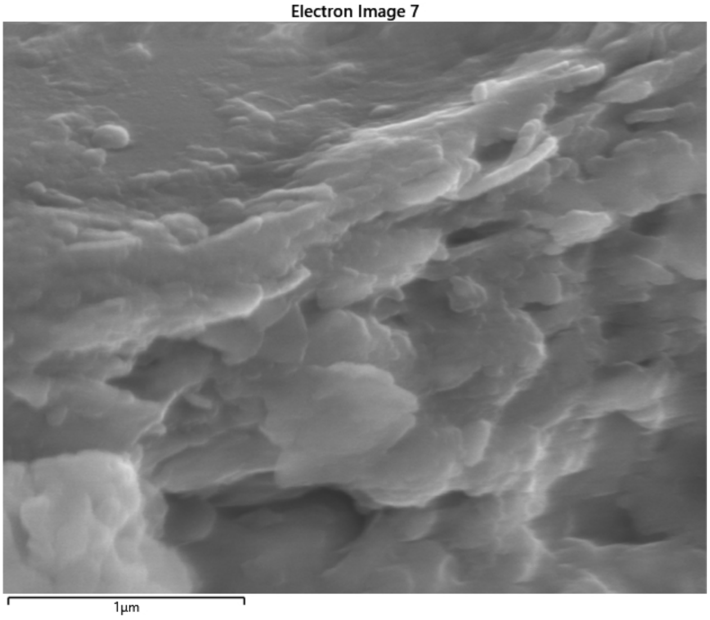


Site 8

Electron Image 7

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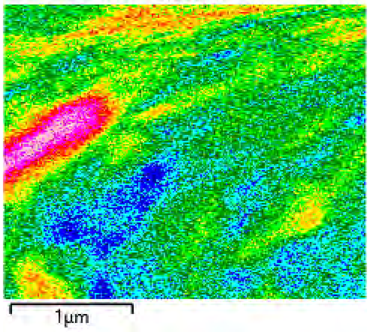


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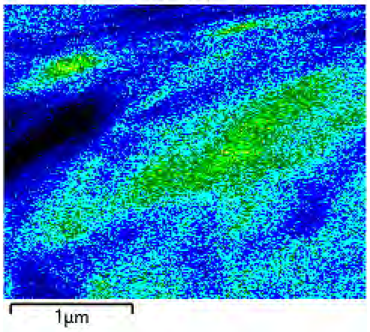


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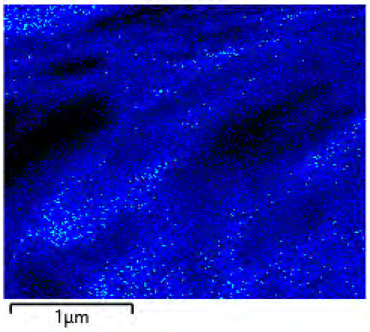


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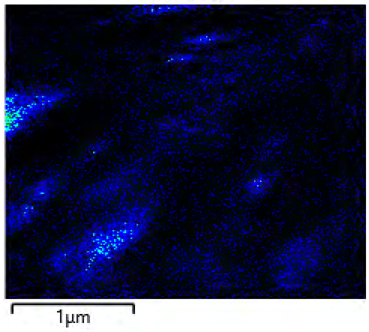


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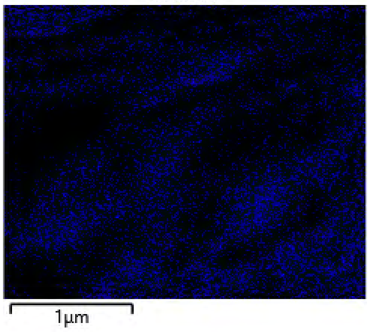


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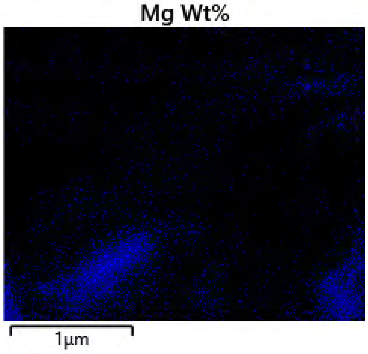


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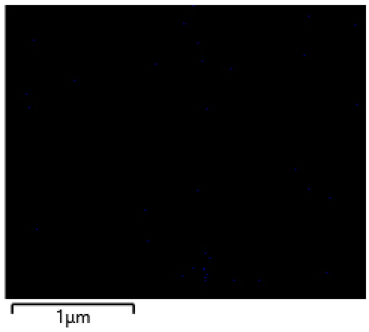
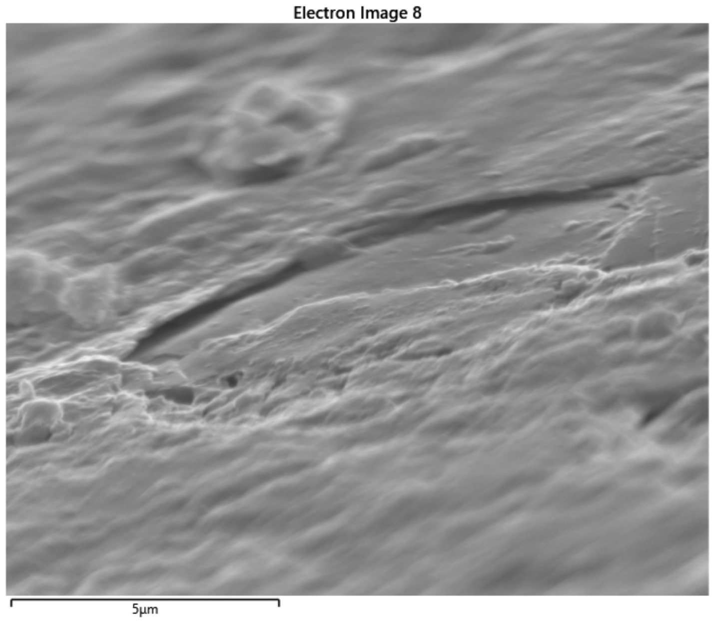
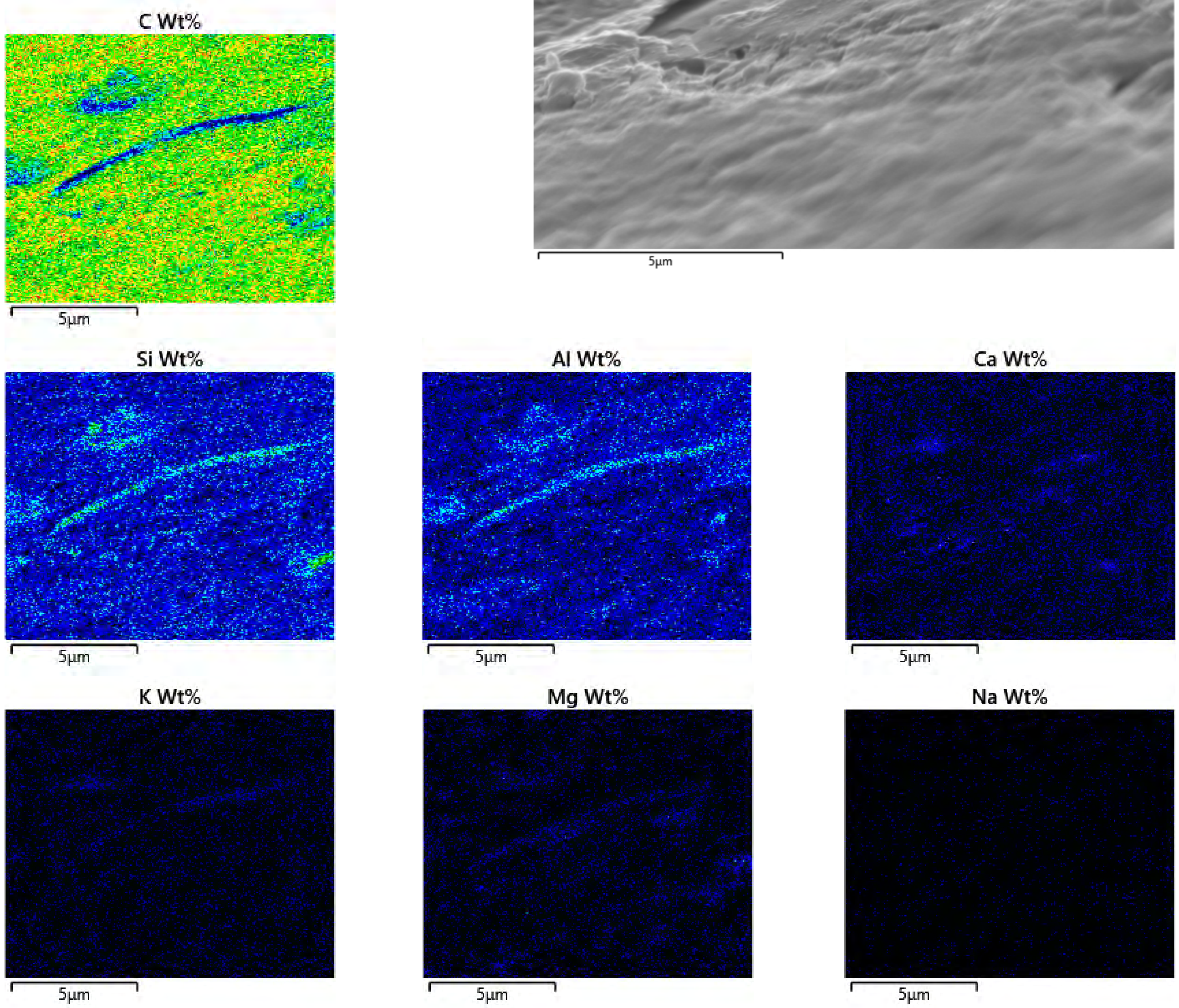


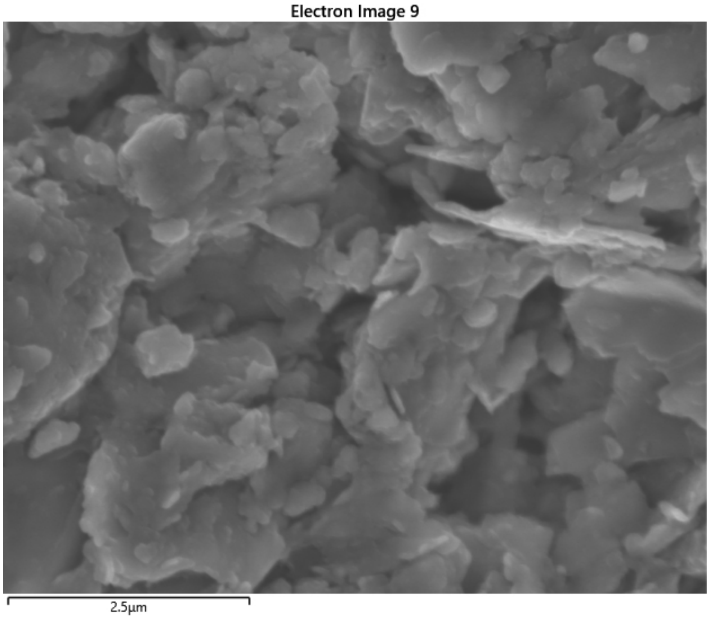
Site 9


Electron Image 8
5µm


C Wt%
5µm
Si Wt%
5µm
Al Wt%
5µm
Ca Wt%
5µm
K Wt%
5µm
Mg Wt%
5µm
Na Wt%
5µm

Site 10

Electron Image 9

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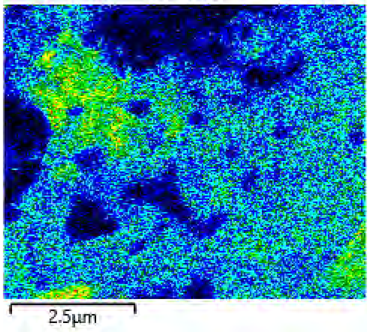


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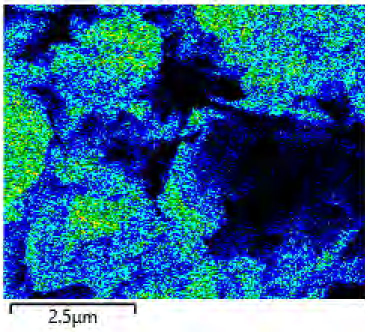


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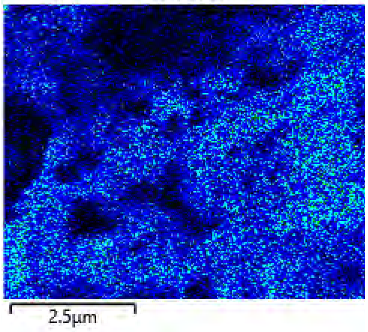


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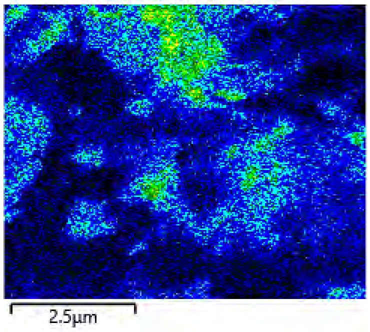


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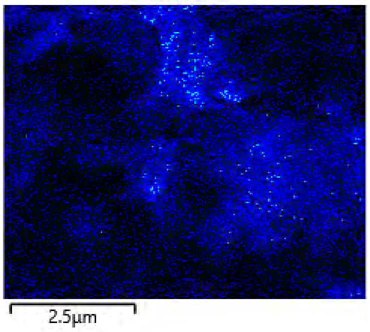


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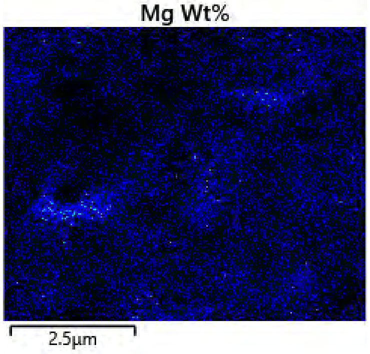


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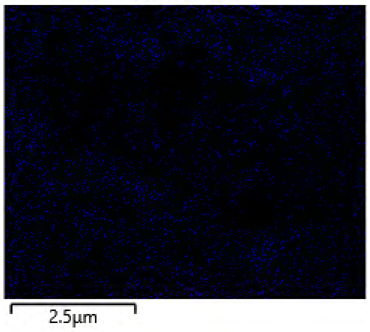
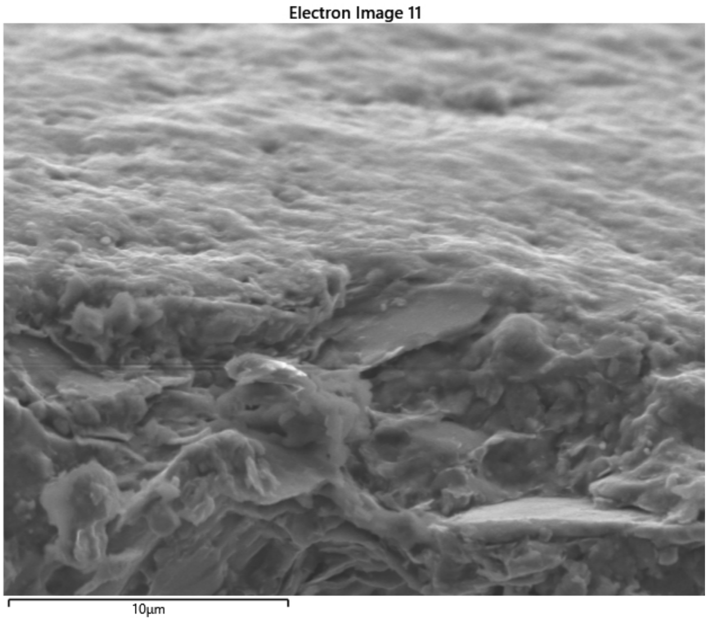
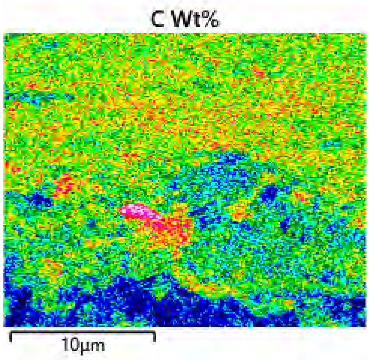
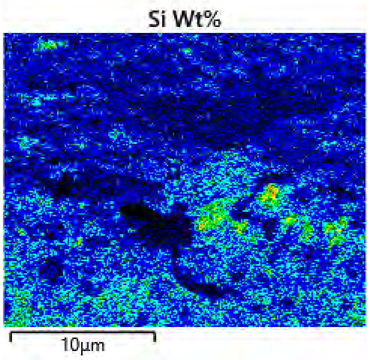


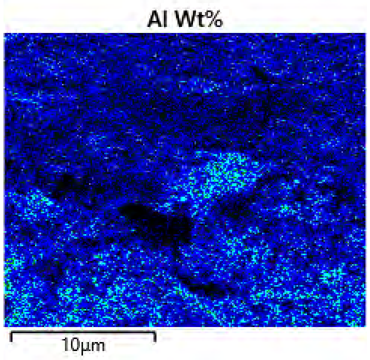
**NLT 267 R FBK (“NLT267_2”)**

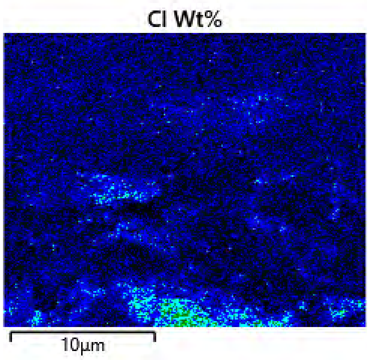
Site 1

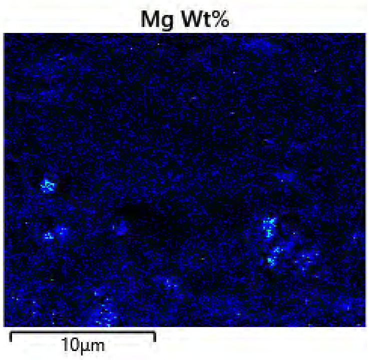


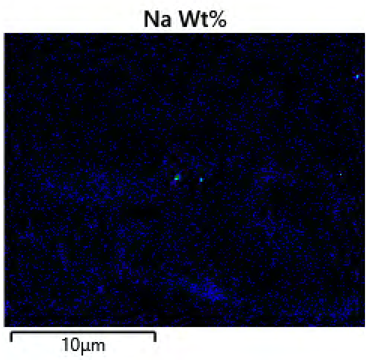



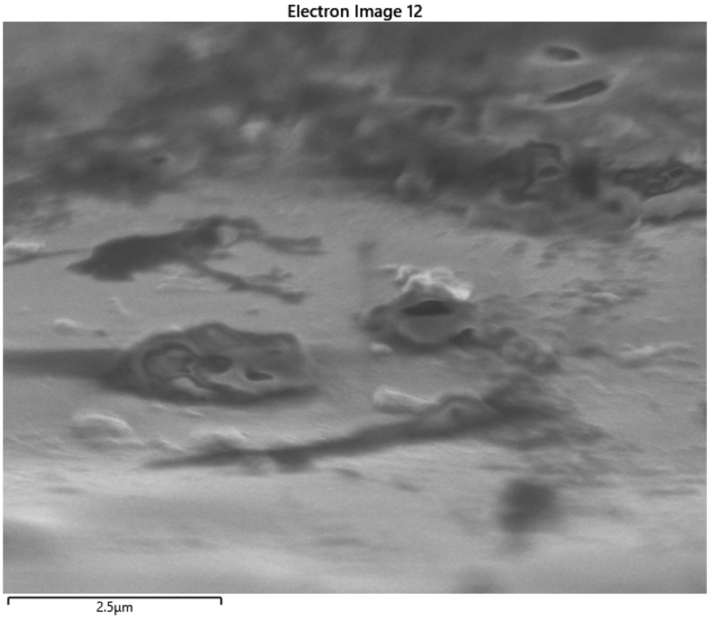



Site 2

Electron Image 12

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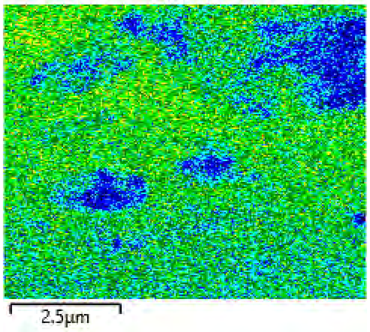


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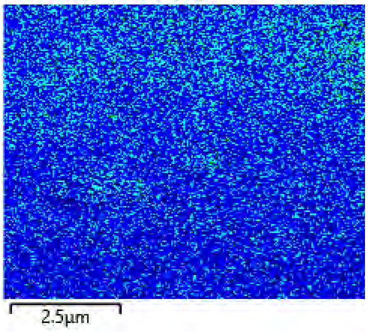


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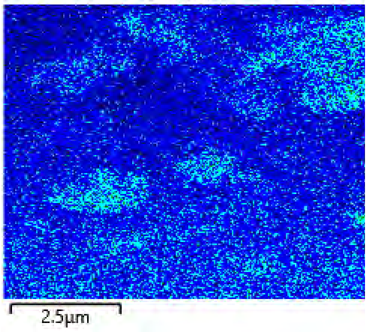


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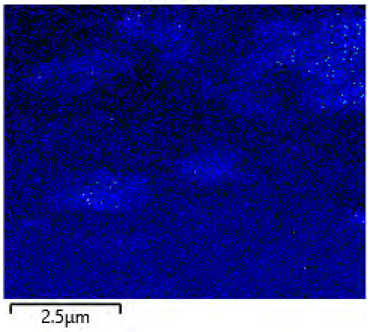


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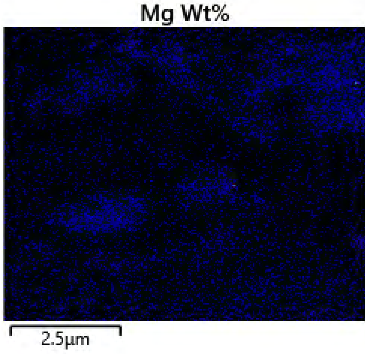


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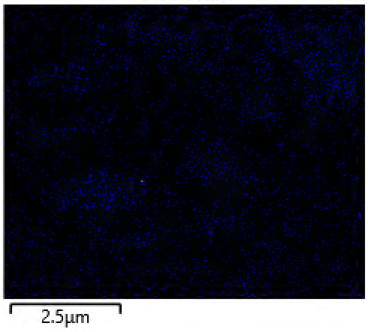

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