Firing temperature estimation of early medieval glass crucibles

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Brief

To provide an equivalent firing temperature estimate for two early medieval glass crucibles: V4845 and V4846.

Methodology

The two crucible samples were prepared as polished specimens to allow THE examination of the microstructure of the clay filaments within the ceramic fabric. By observing the degree of vitrification exhibited by the filaments at 2000x magnification using the secondary electron imaging of a scanning electron microscope or an electron microprobe, it is possible to estimate an equivalent firing temperature (Maniatis and Tite 1981: 72; Tite 1995: 37-40). Individual clay filaments were analysed by a spot analysis using EDS (beam diameter approximately 1-2 microns) enabling the characterisation of the clay into calcareous or non-calcareous, which in turn assists with the equivalent firing temperature estimate.

If the degree and extent of the vitrification of the clay is unclear, then small fragments of the ceramic can be re-fired to known temperatures using a firing cycle with a 200^{o}C per hour rate of heating and a soaking time of one hour at the maximum temperature (Maniatis and Tite 1981: 72). The re-fired samples are then examined in the same manner as the original samples.

Results

The EDS spot analyses of individual filaments in V4845 show that it was formed from a low- to non-calcareous clay (Figures 3 and 4 exhibit a small peak for Ca), and therefore would behave as a non-calcareous clay during firing. A clay will behave as non-calcareous during firing if it has a composition with less than 5% Ca (Maniatis *et al.* 1981: 263; 1984: 211; Kilikoglou 1994: 70-5). The vitrification microstructure observed in V4845 consists of very fine filaments forming a continuous network across most of the sample, which is described as an extensive vitrification microstructure (Figures 1 and 2). However, there ware also some areas that appear to consist of isolated smooth-surfaced filaments (Figure 1: left of centre), which is characteristic of the initial vitrification microstructure that occurs at a lower temperature than the extensive vitrification microstructure. This suggests that the vitrification microstructure is intermediate between the two stages and that the extensive vitrification microstructure has not fully formed.

The EDS spot analyses of individual filaments in V4846 show that it was formed from a low- to non-calcareous clay (Figures 7 and 8 exhibit a small peak for Ca), and therefore would also behave as a non-calcareous clay during firing. The vitrification microstructure observed in V4846 also consists of very fine filaments forming a continuous network of the extensive vitrification microstructure across the whole sample (Figures 5 and 6).

There is a slight difference in the EDS spectra between the core and the edge of both samples. The EDS spectra for the edge of the samples includes a small peak for lead (which is absent for the spectra from the core), which may indicate the presence of material that has leached in or been deposited in the pores from the contents of the glass crucible.

The usual equivalent firing temperature estimate for non-calcareous clay with an initial vitrification microstructure is 750-800^{o}C and with an extensive vitrification microstructure is 800-900^{o}C (Maniatis and Tite: 1981: 68; Aloupi 1993: 16; Kilikoglou 1994: 70-5). However, these samples exhibit very fine filaments, which is unusual for a non-calcareous clay, and thus it is uncertain whether the usual firing temperature estimates for these vitrification stages would apply in these cases. In order to check at which temperature the visible degree of vitrification formed during firing, two refiring experiments were undertaken. A small fragment of V4846 was refired at 800^{o}C and a small fragment of V4845 was refired at 900^{o}C.

The fragment of V4846 refired at 800^{o}C showed no change in the vitrification microstructure from the original sample (Figure 9). This suggests that 800^{o}C is either lower or the same as the original firing temperature. The sample of V4845 refired to 900oC exhibited an extensive vitrification microstructure together with a few thicker filaments (Figure 10), which suggests that this temperature is above the original firing temperature. These results suggest that, although the microstructure may appear unusual for non-calcareous clays, that the standard equivalent firing temperature estimates are valid. Therefore the extensive vitrification microstructure exhibited by V4846 would suggest an equivalent firing temperature estimate of 800-900^{o}C. The intermediate initial vitrification/extensive vitrification microstructure suggests an equivalent firing temperature estimate of about 800^{o}C.

Summary of Results

The equivalent firing temperature estimate for V4845 is about 800^{o}C.
The equivalent firing temperature estimate for V4846 is 800-900^{o}C.

References

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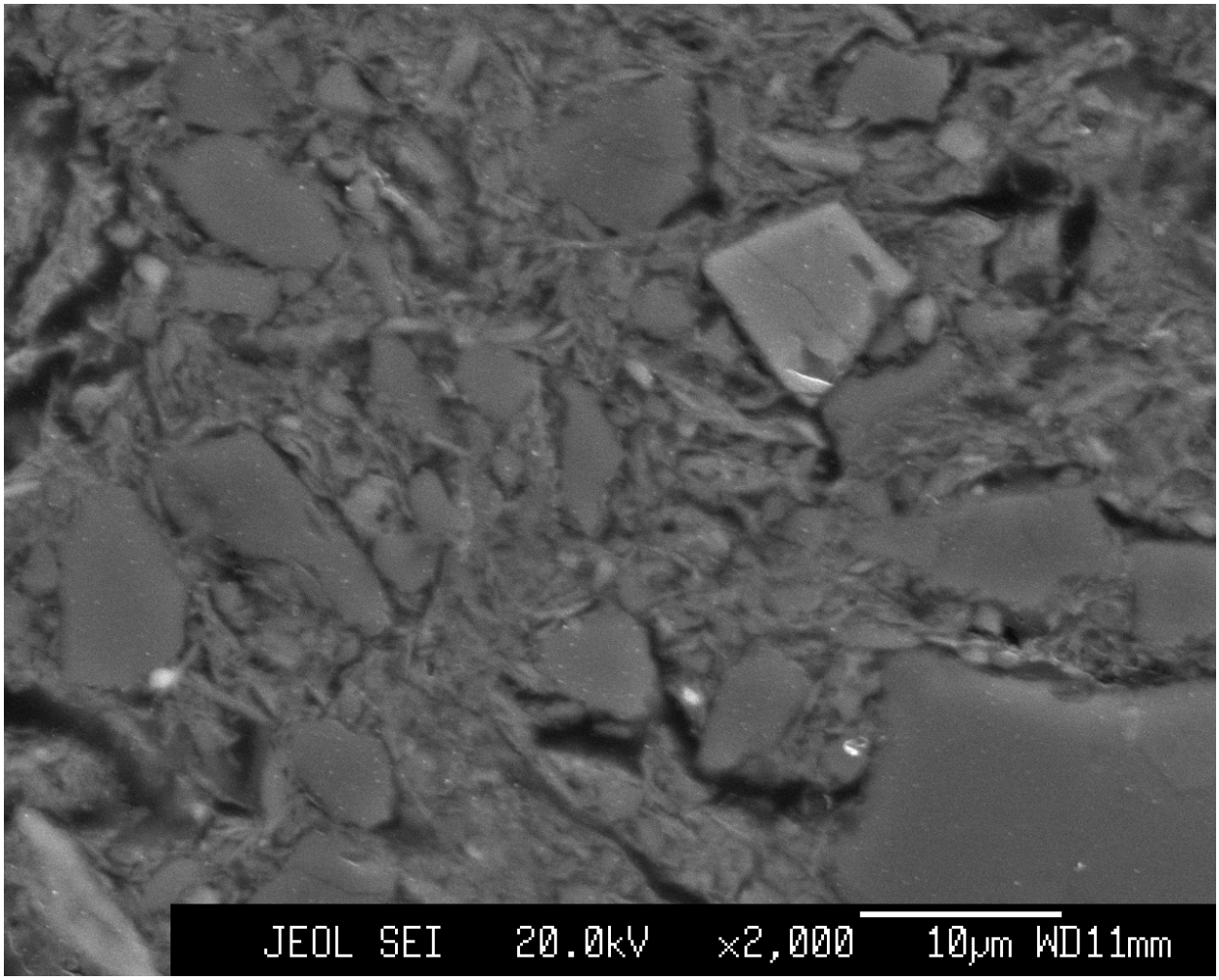

Fig 1. V4845 body.

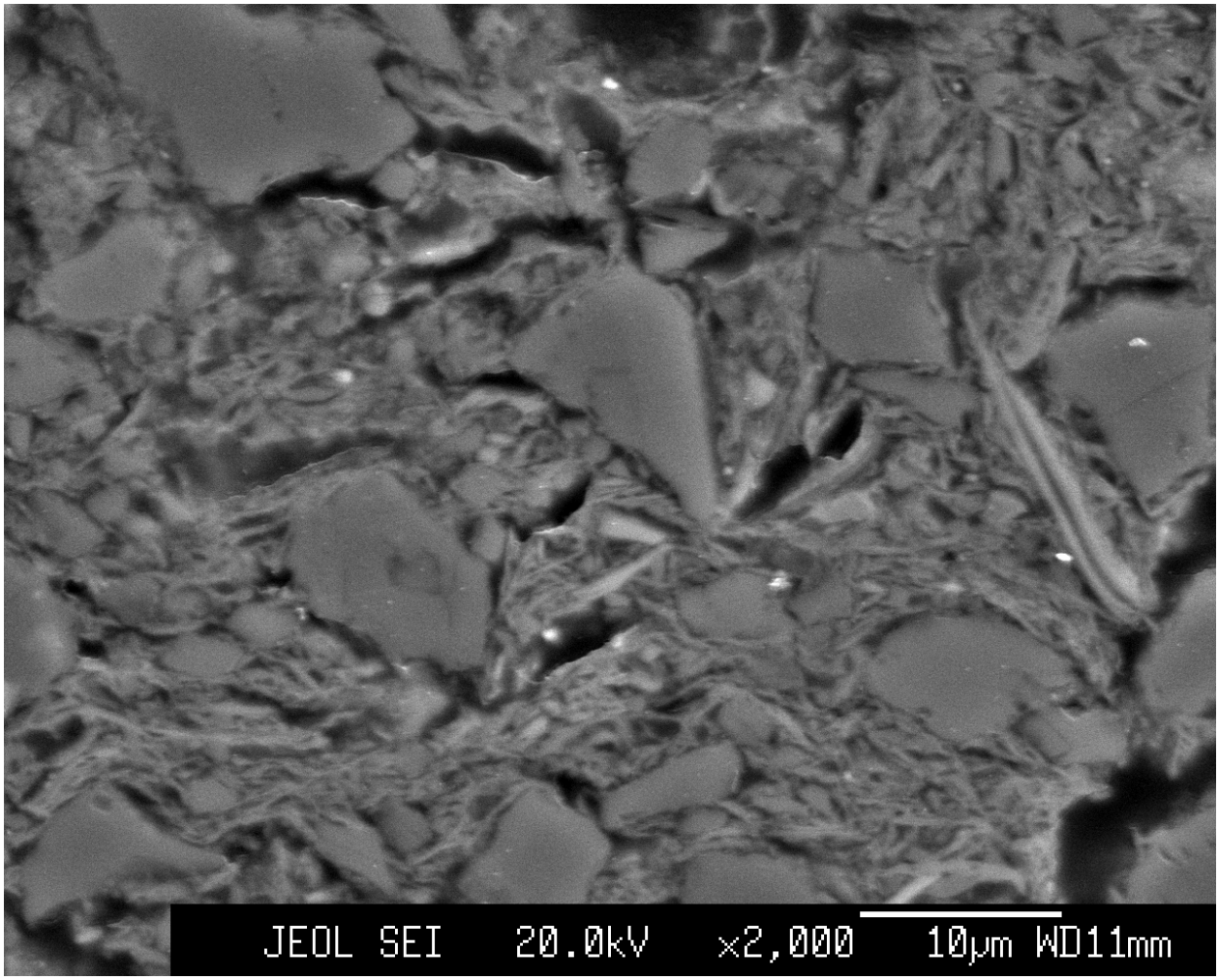

Fig 2. V4845 body.

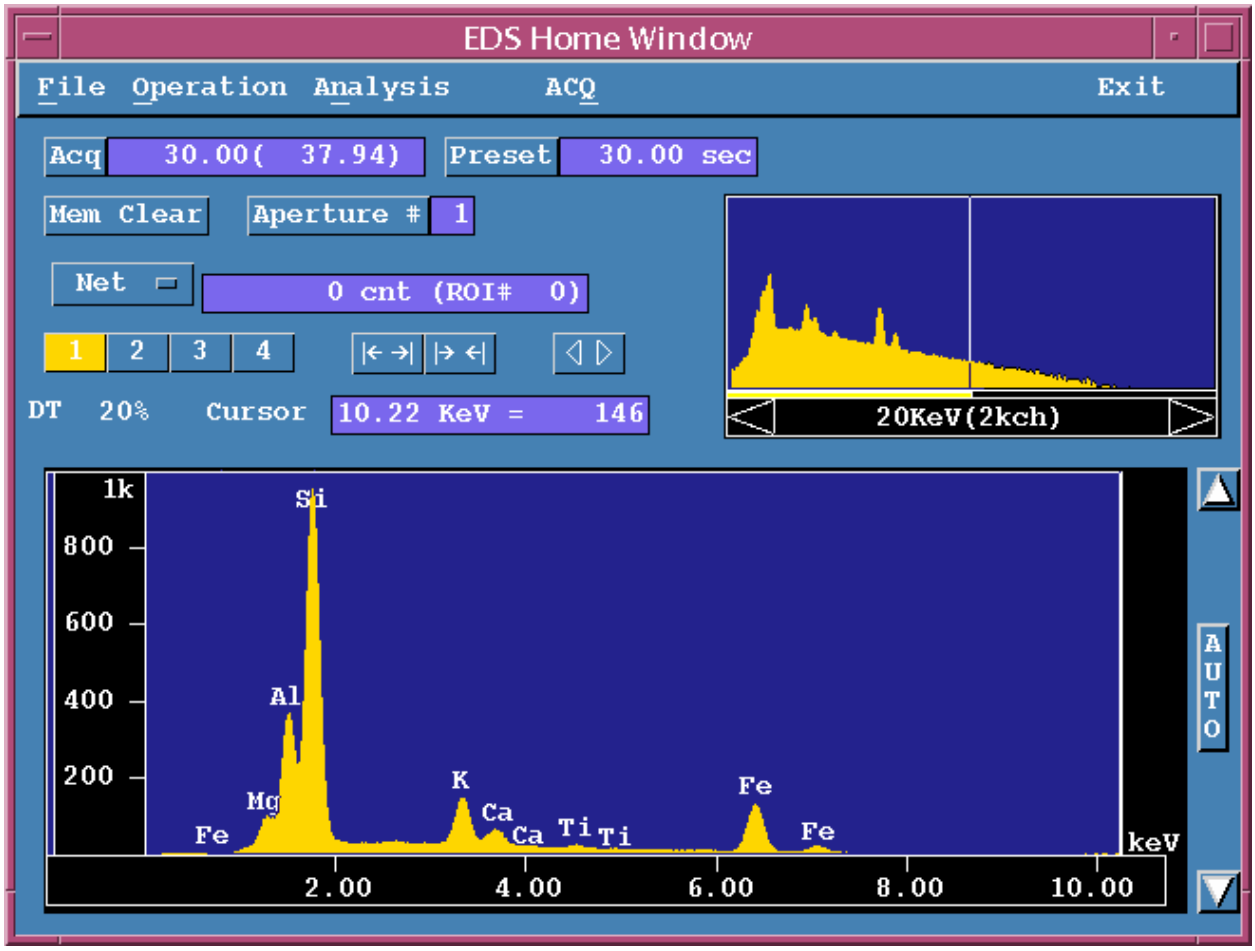

Fig 3. V4845 core.

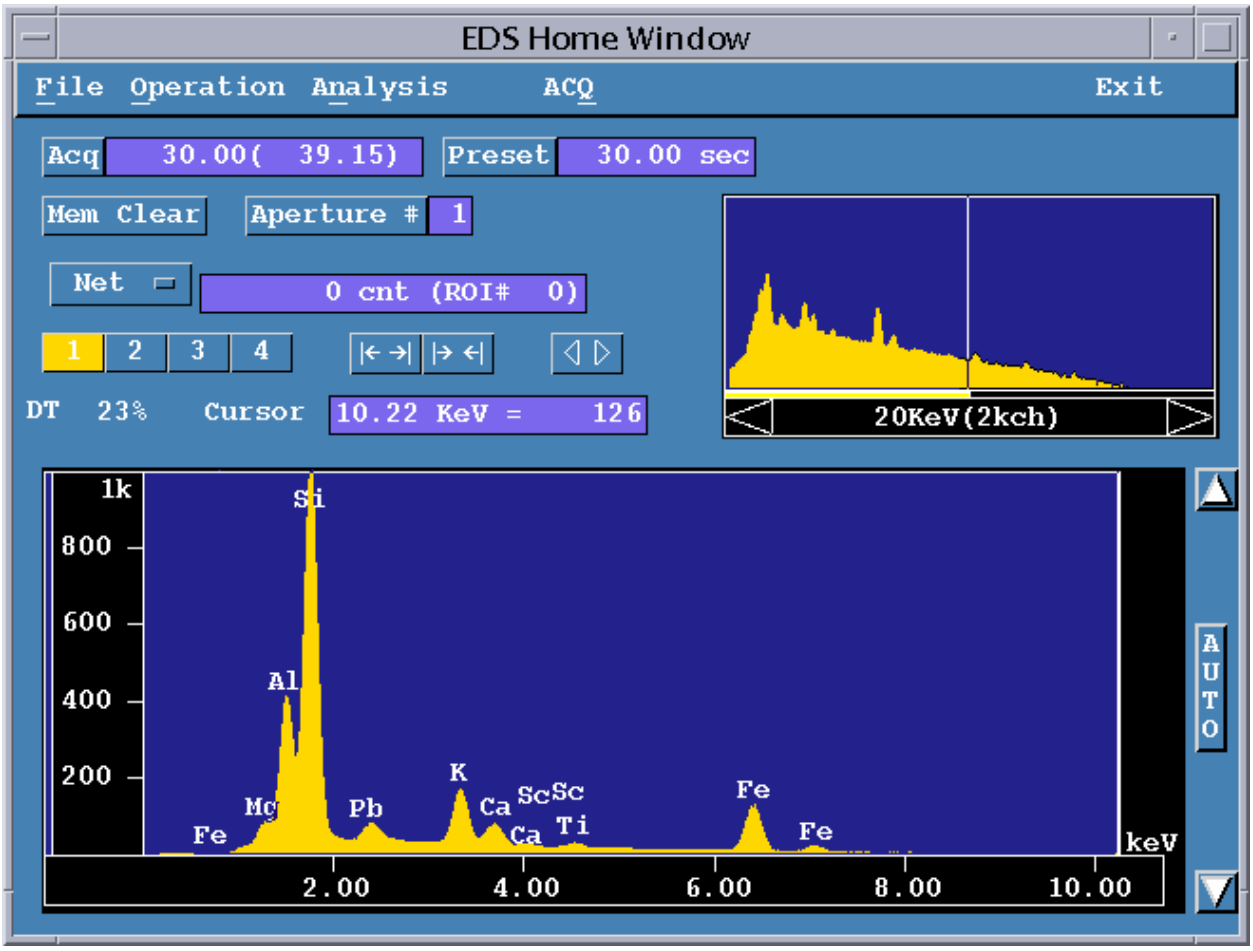

Fig 4. V4845 edge

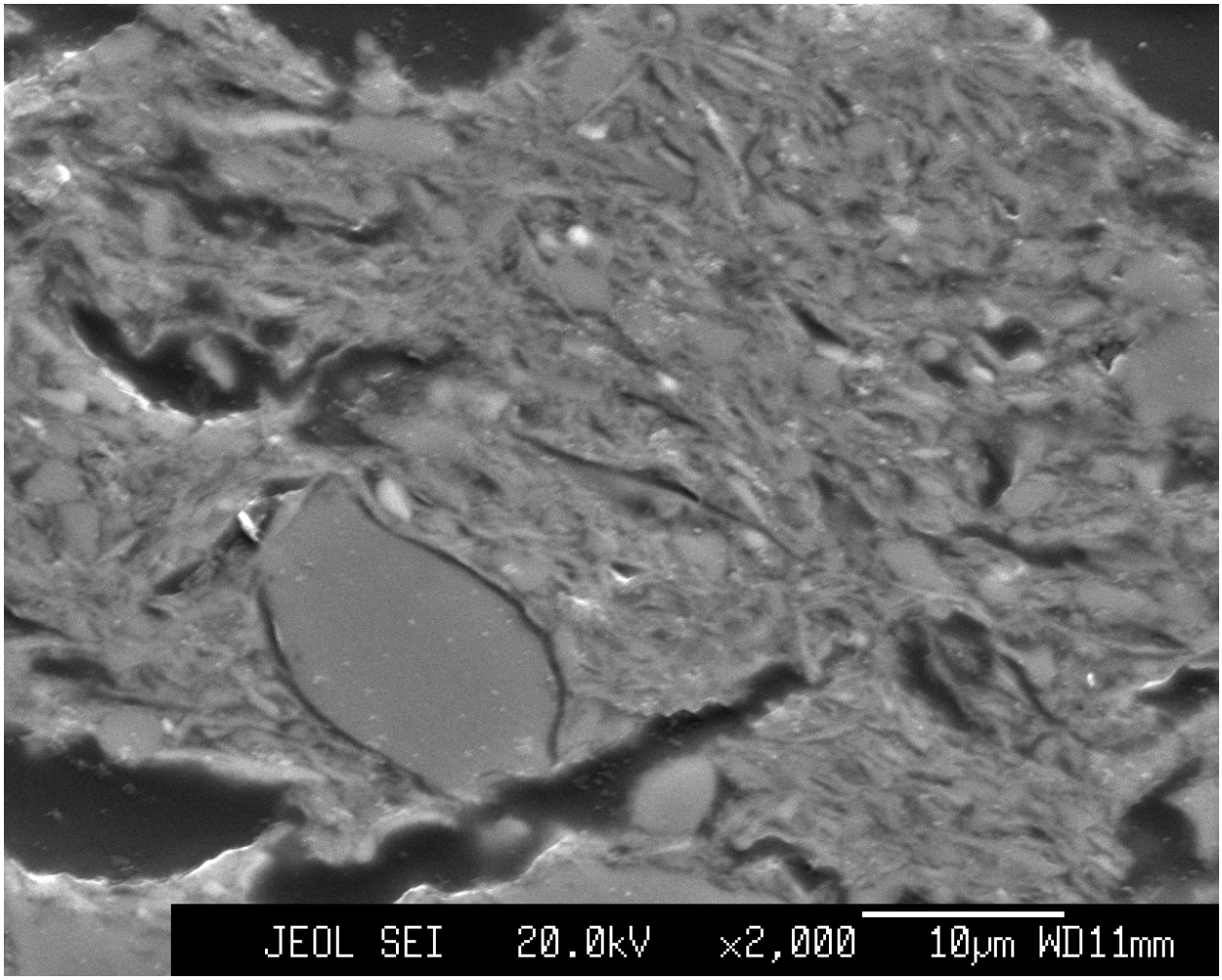

Fig 5. V4846 body.

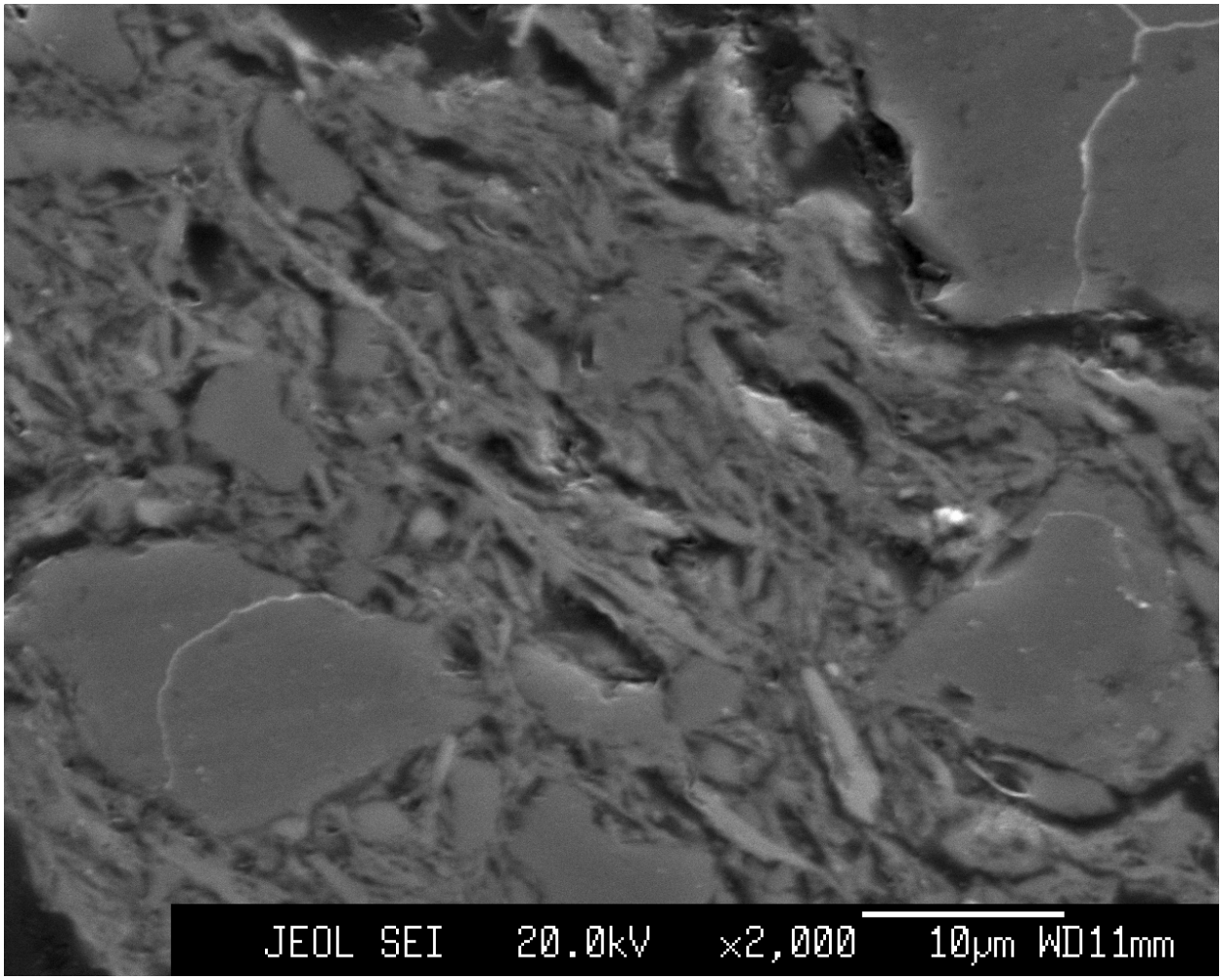

Fig 6. V4846 body.

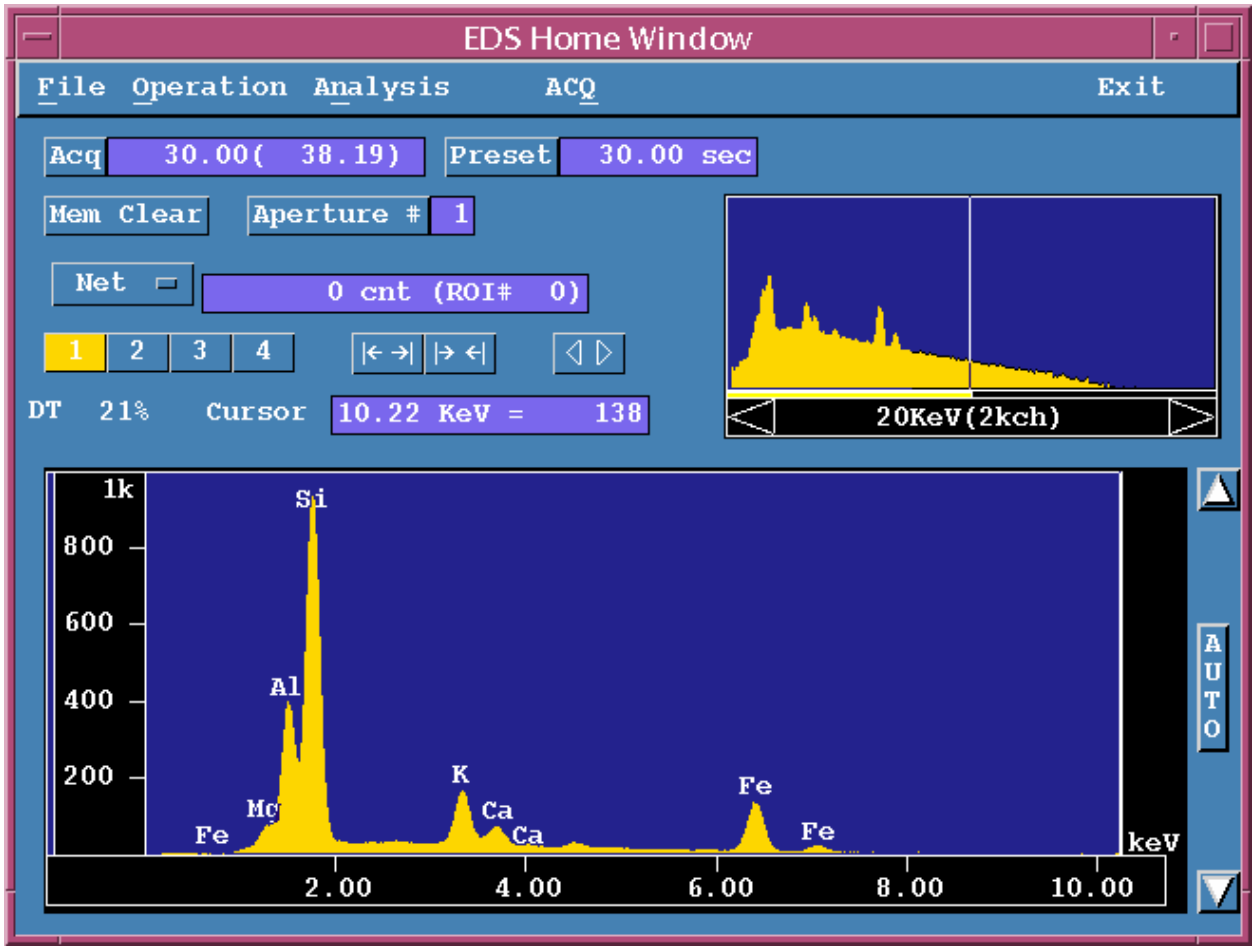

Fig 7. V4846 core.tif

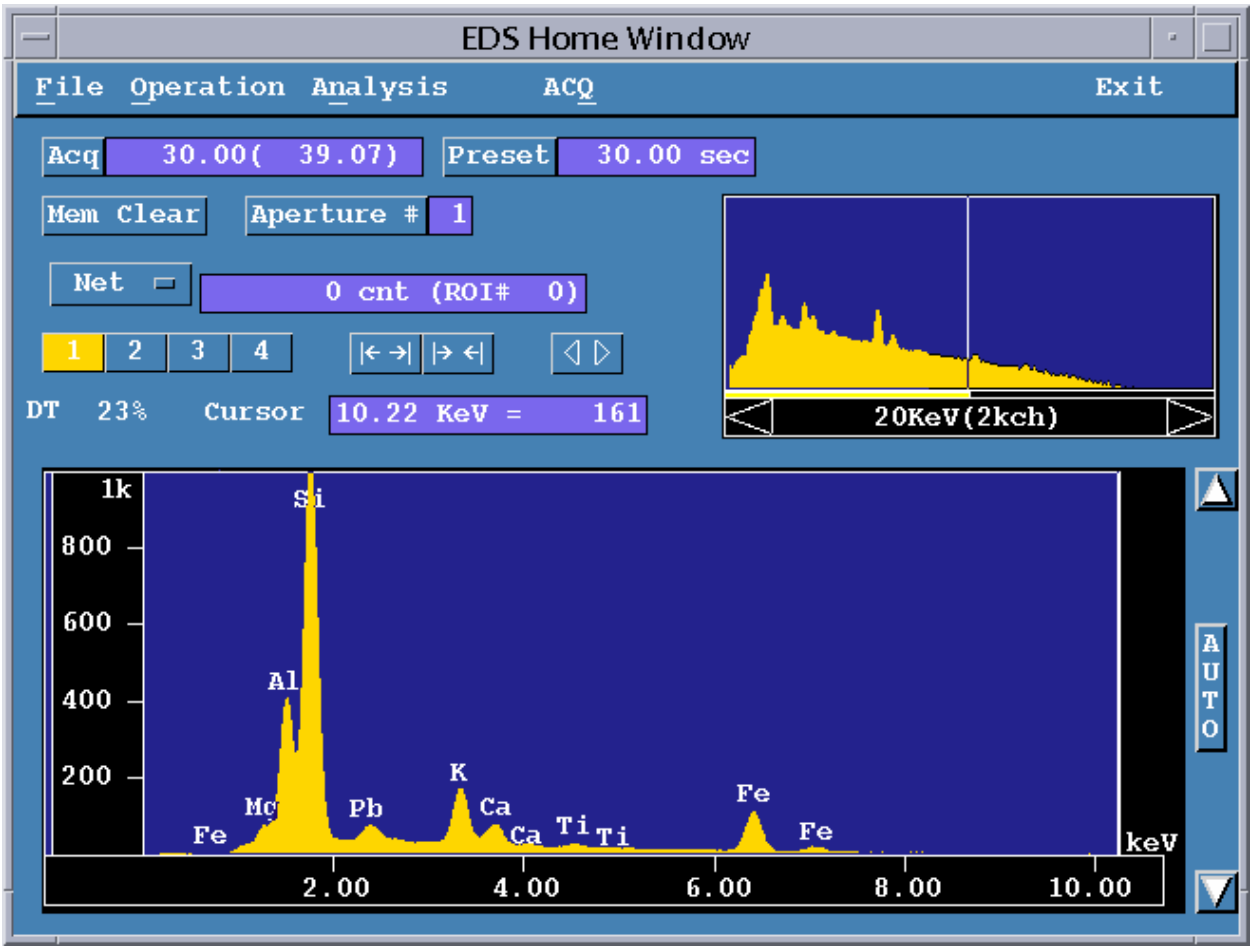

Fig 8. V4846 edge.

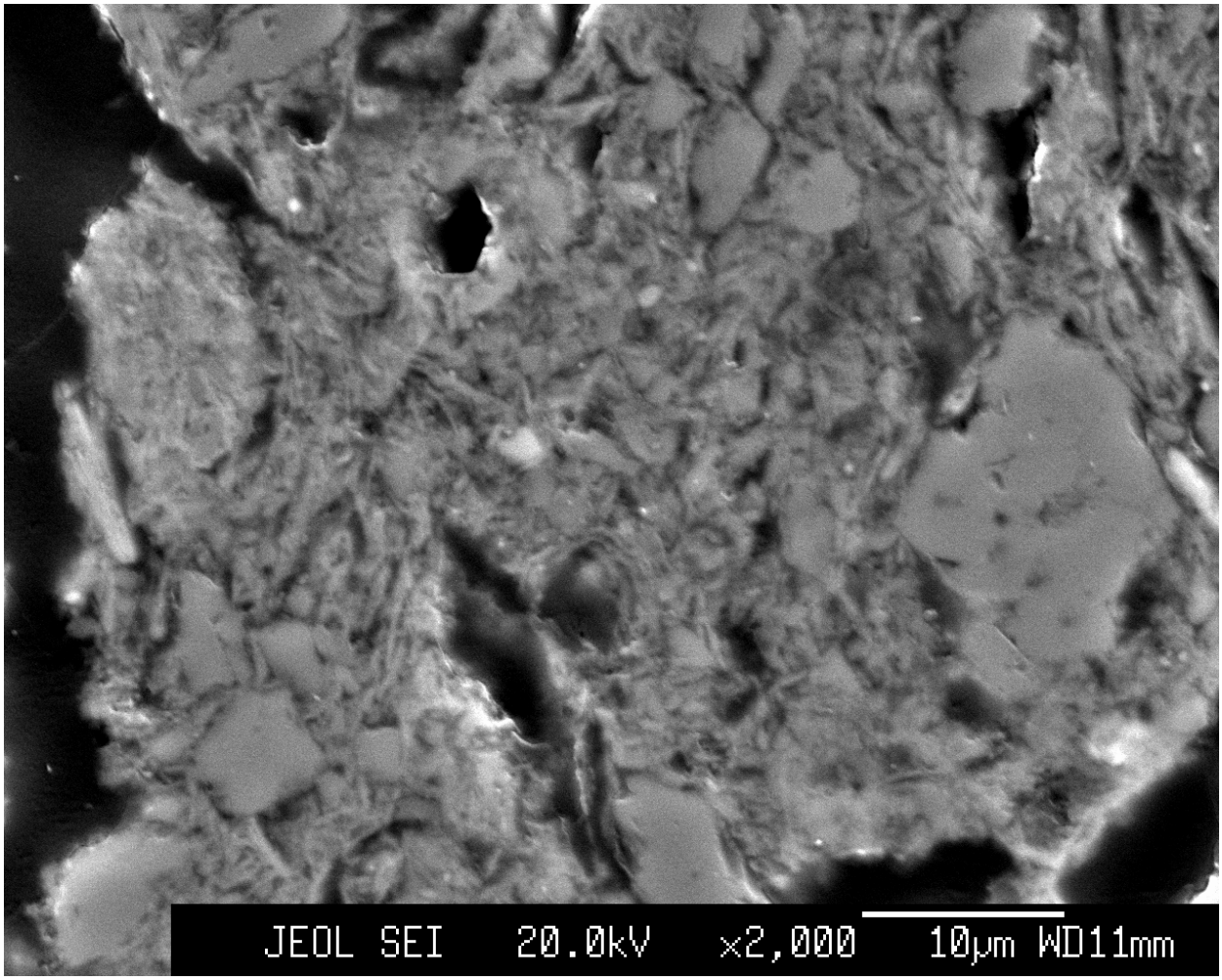

Fig 9. V4846 refired 800 x2000.

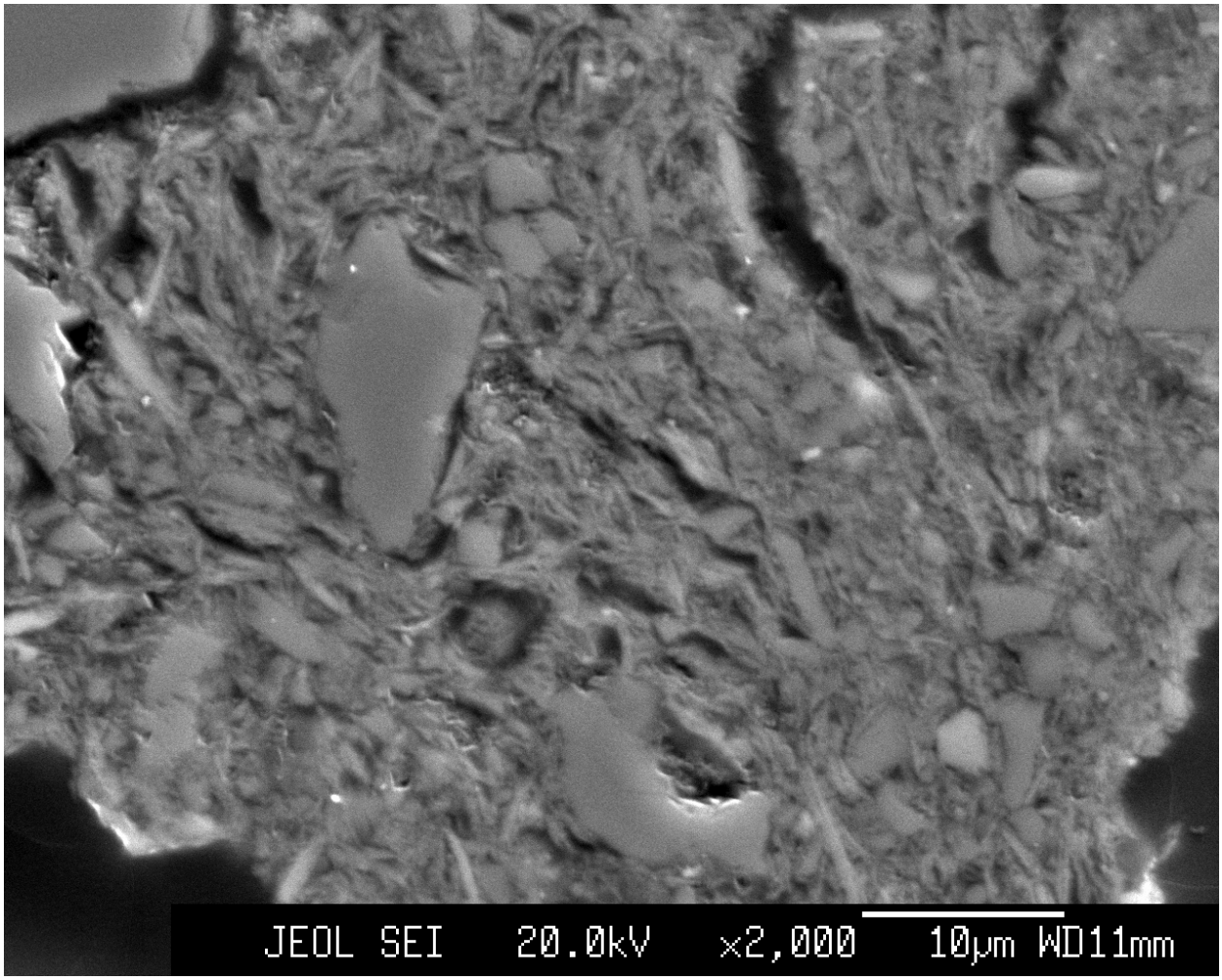

Fig 10, V4845 refired 900 x2000