1. Application

1.1 Study of soil fragments or of polished sections with the scanning electron microscope (SEM) is the final step in the sequence from macromorphological study of soil in the field, through morphological observations of soil samples at low magnification under the stereomicroscope, to study of thin sections, and finally to SEM. SEM makes possible the study of microfabrics involving particles and voids finer than approximately 5 μm (Smart and Tovey, 1982). Combined with energy dispersive X-ray analysis (EDXRA) it is a powerful tool for determining the elemental composition of soil features such as fine particles and thin coatings in situ (Bisdom, ed. 1980).

2. Apparatus and Materials

2.1 Apparatus and materials for preparing thin sections are listed in 84-047.
2.2 Nylon or Texmet polishing cloth, 6 μm and 1 μm diamond paste and 0.3 μm aluminum oxide
2.3 Scanning electron microscope (Cambridge Stereoscan)
2.4 Apparatus for carbon coating or gold coating of samples
2.5 Energy dispersive X-ray analyser (Kevex 5100)
2.6 Polaroid camera for photomicrography
2.7 Polarizing microscope
2.8 SEM stubs, silver cement
2.9 Other materials such as marking pens, micro spatulas, etc.

3. Reagents - none

4. Procedure

4.1 Studying fabric of 'undisturbed' fragments
4.1.1 Select suitable fragments under a stereomicroscope to show the feature of interest. For example, if a ped coating is to be studied, select fragments with the coating intact. Brush some of the fragments to expose a cross section of the coating on the coated matrix.
4.1.2 Dry the fragments and mount them with the surface of interest positioned upwards on 1 cm SEM stub. Use silver paste to cement the fragment to the stub. If the dry fragment is not coherent, partially impregnate it by allowing a suitable solution (agar, collodion) to soak in by capillarity, and cure.
4.1.3 Coat the fragment with carbon, gold, or both so as to prevent charging.
4.1.4 Study the fabric under the SEM at low (50X) to high (20,000X) magnification depending on the nature of the feature. To avoid bias, observe and photograph the feature at regular intervals along one or more transect across the feature. It is essential to examine the feature of interest in several fragments.
4.2 SEM-EDXRA of Polished Sections

4.2.1 Using normal procedures for thin section preparation (84-047), bring the slide to about 35 µm thickness. Clean in ultrasonic cleaner containing petroleum ether.

4.2.2 Cover back of slide with a thin layer of grease or vaseline and insert in holder. Using 9 µm aluminum oxide and distilled water on plate glass, bring the surface condition closer to the polishing stage. This should be done by hand and in a straight one direction movement.

4.2.3 Thoroughly clean the sample and holder. Charge the nylon or Texmet polishing cloth with a 3-4 cm bead of 6 µm diamond paste. Place sample on polisher and run until surface appears to be flat and polished. Check about every 15 minutes. Always grind samples with similar hardness in groups. Use all three weights supplied with polisher during this step.

4.2.4 After thorough cleaning, run sample for about 15 minutes using 1 µm diamond paste and two weights.

4.2.5 After thorough cleaning polish sample for about 30 seconds using 0.3 µm aluminum oxide and distilled water. Use one weight during this step. The polishing cloth should have a thick nap similar to billiard cloth.

4.2.6 Thoroughly clean sections and take to microscope for evaluation. The degree of polish depends almost entirely on the type of sample, polishing times, abrasive size, amount of abrasive, and polishing cloth type, and not the type of polisher.

4.2.7 Study the polished section under the polarizing microscope and circle features to be analyzed using ink containing an element that will show clearly on the SEM (circle approximately 2 mm diameter, Koh-i-noor 3084F ink). Photograph the features of interest within the circle. Cut out with a diamond saw approximately 1 cm² of the section including the circled area, mount the sample on an SEM stub with silver cement and coat the chip with carbon, gold or both to prevent charging. Locate the circled area under the SEM and then locate features of interest with the aid of the photographs. Analyze several microareas within each feature by EDXRA and relate the spectra to those of suitable standards (McKeague and Wang, 1980).

5. References

