

A METHOD OF MINIMIZING FORMATION OF SPHERULITES IN CARBOWAX-IMPREGNATED CLAYS

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Clay soils are commonly impregnated with 'Carbowax 6000' prior to fabrication of thin sections for fabric study on the petrographic microscope (Mitchell, 1956). The 'Carbowax' replaces pore water and reduces drying shrinkage which may cause unacceptable fabric change. Carbowax 6000 is derived from ethylene glycol and like many polymer compounds of this sort it tends to crystallize in the form of spherulites (Fig. 1). This structure can sometimes be seen in thin section superimposed upon the clay fabric (Fig. 1b). This complication undermines confidence in the reliability of results, casting considerable doubt upon the authenticity of the fabric. This note describes a method by which the formation of spherulites can be reduced drastically.

Very rapid cooling of a liquid frequently inhibits crystallization and the material solidifies in the glassy state. This occurs when molten Carbowax 6000 is plunged into Freon 22 supercooled in liquid nitrogen to a temperature in the range -100 to -130°C . Within this range temperature variation had little influence on the result. The supercooled Carbowax devitrifies rapidly when the temperature rises to normal values, but instead of spherulites in the 0.1 – 1 mm size range only small (~ 25 – 50 μm) isolated spherulites form in a cryptocrystalline groundmass (Fig. 1c). This microstructure remains stable for several weeks and perhaps indefinitely. Supercooling by direct immersion in liquid nitrogen is less effective because a layer of nitrogen gas forms surrounding the sample; this layer has an insulating effect and the rate of cooling is reduced. In this case the spherulites are larger and the microcrystals of the groundmass are coarser (Fig. 1d).

Little or no spherulite formation has been observed in thin sections made of Carbowax-impregnated clays which have been quickfrozen in supercooled Freon (Fig. 1e,f). It is probable that vitreous Carbowax is only formed in a relatively thin layer close to the surface of the sample. Hence, it is well to remove as little of the sample as possible when pre-grinding the surface to be mounted on the glass slide.

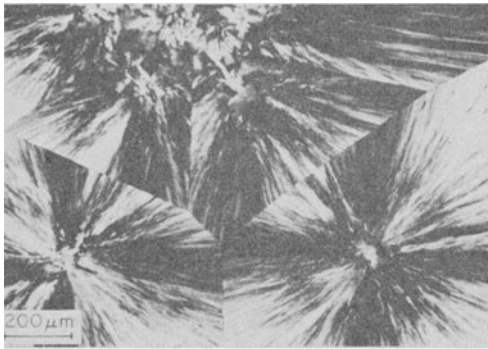
Spherulites are not always observed in Carbowax-impregnated clays which have slowly cooled to room temperature and it seems not unlikely that in some cases the soil acts rather like a filler in inhibiting crystallization of the polymer. Possibly the strength of the bonds between the clay crystallites of which the skeletal fabric is composed is the deciding factor. When bonds are weak, the Carbowax exerts a sufficient force of crystallization to displace the host crystals; whereas when clay-to-clay particle bonds are strong, crystallization of the Carbowax has to accommodate itself to the clay fabric.

*Department of Civil Engineering,
The University of Calgary,
Calgary, Alberta, Canada*

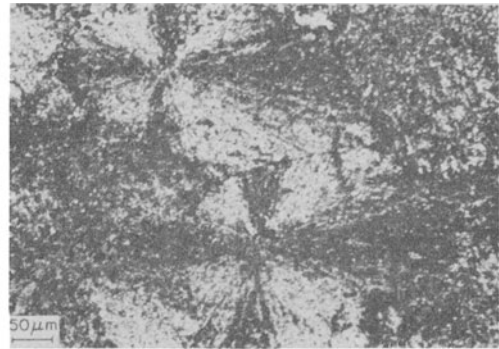
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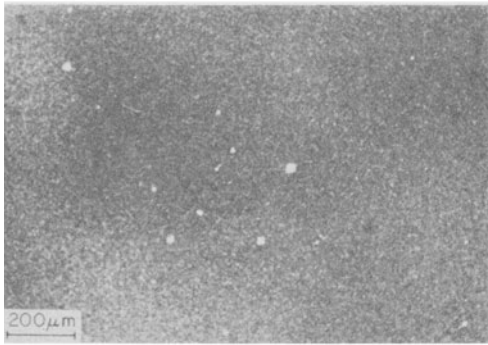
- Mitchell, J. K. (1956) The fabric of natural clays and its relation to engineering properties: *Proc. Highway Res. Board*, **35**, 693–713.



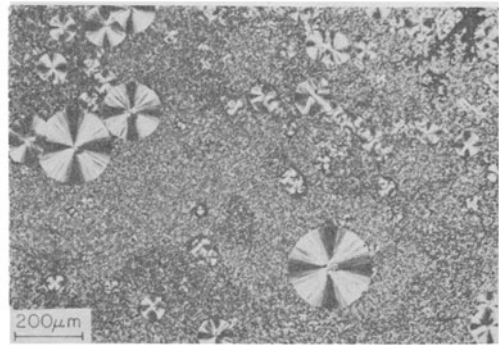
(a) "CARBOWAX 6000"-SLOW COOLING IN AIR



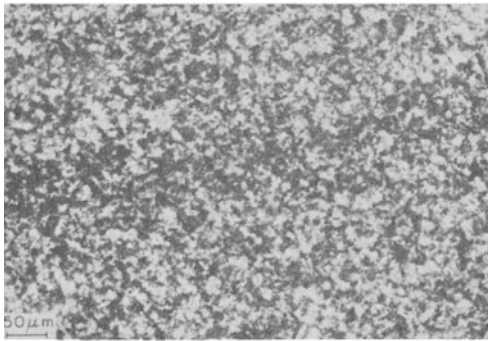
(b) LEDA CLAY-BRECKENRIDGE, QUE. IMPREGNATED "CARBOWAX 6000" SLOW COOLING IN AIR



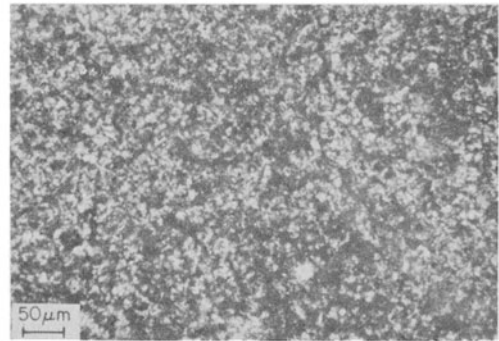
(c) "CARBOWAX 6000"-ULTRA-RAPID COOLING IN SUPERCOOLED FREON 22



(d) "CARBOWAX 6000"-COOLED IN LIQUID NITROGEN



(e) LEDA CLAY-BRECKENRIDGE, QUE. IMPREGNATED "CARBOWAX 6000" SUPERCOOLED IN FREON 22



(f) LEDA CLAY-GLOUCESTER, ONT IMPREGNATED "CARBOWAX 6000" SUPERCOOLED IN FREON 22

Fig. 1. Optical micrographs of carbowax and carbowax-impregnated clay.