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## THE MICROTOPOGRAPHY OF PENCIL LEAD IN DRAWINGS

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The following is, for the most part, a summary of a paper which has been published elsewhere, and a more complete discussion of this study will be found there.<sup>1</sup>

Pencils have been used for drawing from the latter part of the 16th century to the present. The earliest pencils consisted simply of sawn pieces of natural graphite placed in various types of holders. The most highly valued graphite came from mines near Borrowdale in Cumberland, England, and it was the discovery of these deposits in the mid-16th century which led directly to the introduction of the pencil as a common writing and drawing tool. The Cumberland mines served as the only major source of pencil graphite in Europe for the next two or more centuries. As the supply of high quality material from these mines dwindled, methods were sought by which poorer grades of graphite could be made into usable pencils. Before the end of the 18th century, several methods had probably been tried, all of which involved the mixing of ground graphite with a binder, such as gum, resin, lime, or molten sulfur. However, all of these yielded very hard and generally unsuitable pencils.

In 1794, the French Bureau of Mines commissioned N.J. Conté to find a substitute for natural graphite pencils, and later that year he patented a process which involved mixing together finely divided graphite and clay, drying the mass, and firing it in a furnace. Conté's process was soon adopted throughout Europe, and it is essentially the method still used to make pencils today. Although natural graphite pencils were still available after the time of Conté's invention, the less expensive synthetic pencils probably replaced them to a great extent shortly afterward.

The purpose of this research project is to establish whether synthetic and natural pencil leads can be distinguished from one another on the basis of their microtopography. These distinctions could be used to answer certain questions of authenticity or attribution in pencil drawings. The study is being carried out primarily by scanning electron microscopy.

The structure of graphite consists of parallel sheets of fused benzene rings; bonding between the sheets is due only to Van der Waals forces. As a result of its structure, graphite readily cleaves along layers, but is much harder across these layers. As a stick of natural graphite is drawn

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across a piece of paper, the mark is usually somewhat uneven. There is a tendency for graphite particles to break off in unevenly sized pieces. The larger fragments are plate-like and often rest on their faces on the page. The larger of these smooth faces have a distinctive metallic reflection when viewed at magnifications of a few hundred times.

The suitability of a particular sample of natural graphite for use as a pencil lead depends both on the purity of the material and its physical state. Harder mineral impurities in the graphite, such as quartz, feldspars, and so forth, produce striations or scratches in the pencil lines. Graphite in which the particle sizes are large or inhomogeneous will not leave uniform marks, and due to the anisotropy of the mineral, a stick of graphite may behave quite differently depending on its orientation as it is drawn across the paper. Of all the deposits every known, Borrowdale graphite seems to have best combined the most desirable features for a pencil lead.

The graphite in synthetic pencils has been and continues to be derived from natural sources. It is normally first purified by flotation or other techniques and then thoroughly ground and mixed with a high-purity, finely divided clay. The particle sizes of both graphite and clay are probably on the order of a few microns or less, at least in present-day pencils.

The fired mass tends to fracture rather uniformly into particles which are roundish, and on a drawing it is possible to produce sharper lines of greater density more readily with the softer grade synthetic pencil leads than with most natural graphites. At magnifications of a few hundred times, the highly reflecting plates sometimes visible in natural graphite lines are absent. There are, however, considerable variations in the lines produced by synthetic leads, dependent on the nature of the paper or support surface and the pressure exerted by the draughtsman, as well as the grade of the pencil.

Although distinctive differences between the marks of natural and synthetic pencil leads may occasionally be observed in situ on drawings at magnifications of a few hundred times, this is not often the case. The morphological distinctions become clear at magnifications obtainable only with a scanning electron microscope, which requires samples to be taken from the drawings. Ideally, the sampling procedure should be one which does not damage the paper (or other support) and which produces no visible change in the appearance of the pencil line. After several trials, the following sampling procedure was adopted. A very thin film of Acryloid B-72 resin (Rohm & Haas) in xylene and toluene was cast on a piece of mylar and allowed to completely dry. Small squares approximately 1-2 mm by 1-2 mm were cut and placed resin-side down on the graphite line to be sampled. By gently pressing the square onto the paper with a microneedle, graphite particles are pulled off of the page. Since the square is transparent, the amount of graphite actually transferred is readily evident at all times.

The square is then transferred to a carbon planchet covered with double-sided adhesive tape. All operations are carried out under a binocular microscope, and even at these magnifications (ca. 30 X) the sites sampled are not visibly altered.

In most cases, the samples were first coated with a conductive coat of carbon and X-ray data collected from several particles in each sample. Before photographing, the samples were further coated with approximately 100 Å of gold-palladium alloy, which substantially improves the resolution. Usually one or two scanning electron micrographs were taken of each sample, but many particles were examined in each sample to insure that the photographed particles were representative. In addition, two and sometimes more samples were taken from most of the drawings examined.

At this stage, a number of drawings from the 16th century to the present have been sampled, but many more samples will be necessary to compile a useful reference collection of photomicrographs of various types of pencils. As a part of this stage of the study, several natural graphites of known geological provenance as well as several modern synthetic pencils have also been examined. Plate-like particles, whether in natural graphite or synthetic pencil leads, are usually preferentially oriented on their faces by the sampling procedure used. Synthetic pencil leads in particular may appear rather differently if scrapings from the lead are directly examined, since many of the flakes in these leads are more randomly oriented in scrapings. Consequently, lines were drawn on a piece of paper with the geological samples as well as the modern synthetic pencils and the lines sampled in the same manner as the drawings.

For the purposes of this summary, only two photomicrographs are reproduced. Figure 1 is a scanning electron micrograph of natural graphite from Borrowdale in Cumberland, England; it is typical of natural graphites in general. The graphite appears as a series of sheets with extended smooth surfaces; often these sheets are folded under one another and are usually broken at sharp angles. The secondary electrons which produce the image are generated from a depth of as much as 100-200 Å in spite of the gold-palladium coating, and this causes the apparent transparency of some of the sheets. Figure 2 is a Staedtler Mars-Lumograph 200 2H pencil lead, a relatively soft pencil lead in which the smooth, folded surfaces of graphite are evident, particularly near the lower right corner. These are interspersed with rather amorphous, rounded particles which may be partially sintered clay, as well as some jagged petal-like flakes which are similar in appearance to unsintered clay particles.

It should be emphasized that these two photomicrographs put the most favorable light on the differences in the microtopography of natural and synthetic pencil leads, and samples from drawings are often more difficult to interpret. From the examination of samples from several Ingres drawings, it appears that the early synthetic pencils contained graphite which was

not as well-ground as that in modern pencils, and subsequently there are rather large flakes of graphite visible in the photomicrographs.

Natural graphites always contain at least small amounts of impurities (clays and other minerals), and poorer grades may contain enough of these materials that they do present the "clean" graphite image of Figure 1.

It is possible that with careful X-ray fluorescence analysis of particles in the samples, the "clay (or other impurity) fraction" of a natural or synthetic pencil lead can be more readily separated from the "graphite fraction" than is possible on the basis of microtopography alone. A few experiments we have carried out recently suggest that transmission electron microscopy may also prove valuable, although the sampling problems associated with this technique are more complex than those associated with the scanning electron microscope.

Although more work certainly needs to be done, the scanning electron microscopic technique makes it possible to distinguish between natural graphite and synthetic pencil leads in many cases. Additional studies may lead to further possibilities in distinguishing between the pencil leads in drawings.

#### Reference

1. Richard Newman, "The Microtopography of Pencil Lead in Drawings: A Preliminary Report," Papers Presented at the Art Conservation Training Programs Conference, April 28 & 29, 1980, University of Delaware, Newark, Delaware, pp. 31-46.

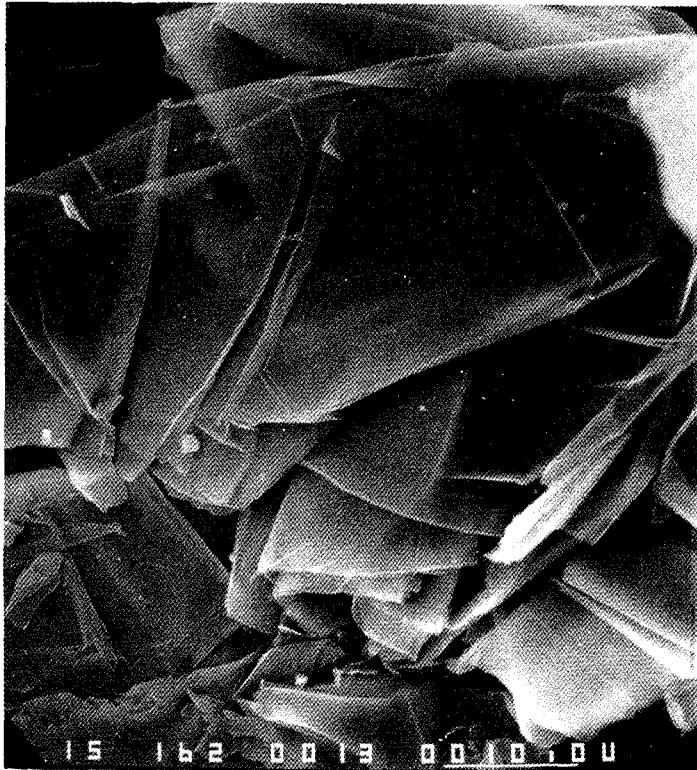


Figure 1

Scanning electron micrograph of natural graphite from Borrowdale, Cumberland, England (Geology Museum, Harvard University, sample no. 108076).

The number in the lower right corner in this and the following figure gives the scale of the micrograph: in this case, the white bar equals  $10\ \mu$ .

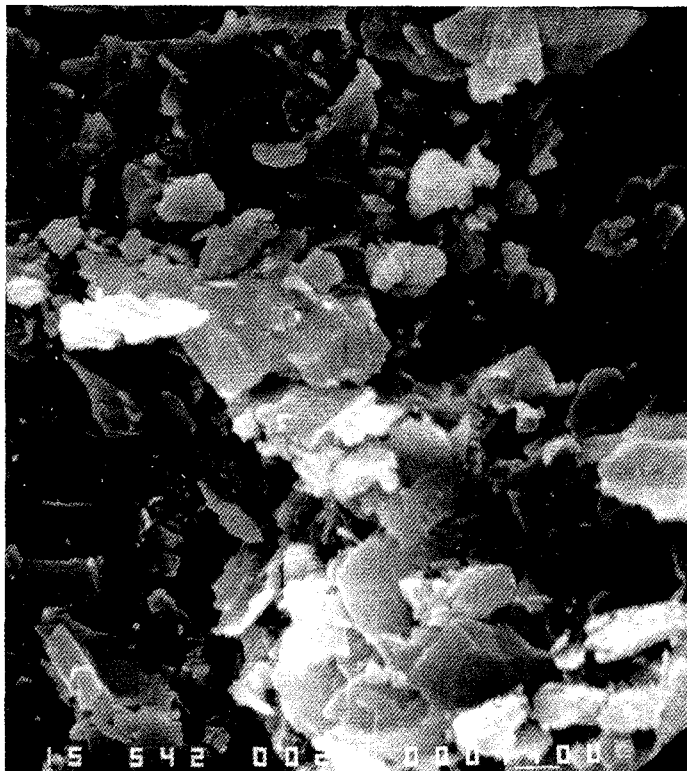


Figure 2

Scanning electron micrograph of Staedtler Mars-Lumograph 200 2H pencil lead. White bar equals  $1\ \mu$ .