

tion, and liptinite macerals can be recognized, especially when associated with vitrinite.

Coal facies can be defined using the following criteria: (1) lustre, (2) texture, (3) sedimentary structure (degree of lamination), (4) mineral matter (type and approximate amount), (5) Maceral composition.

The rapid appraisal of criteria 1 through 4 can be carried out at a mesoscopic scale of examination (Fig. 1). The thickness of each coal facies can be measured and the nature of bounding contacts recorded. Having established that coal facies defined at a mesoscopic scale represent the microscopic composition, the maceral and microlithotype composition of several examples from each facies can then be examined, eliminating the need to examine the whole seam-section microscopically.

SUMMARY

The use of a binocular microscope and incident light source to examine whole, oriented coal samples at a mesoscopic ($\times 10$) scale allows the rapid appraisal of coal type. Microscopic studies may subsequently be used to support and amplify the mesoscopic facies. This method has the following advantages over other techniques of coal facies analysis:

- 1) It is rapid and not hampered by subjective macroscopic brightness criteria that may bear little relationship to the maceral content.

- 2) The orientation, sedimentary structure and texture of the coal is preserved, and the relationships of coal facies with associated mineral matter are retained.
- 3) Stratigraphic information such as facies association, facies sequences, and the nature of facies boundaries can be used to interpret the origin of coal types.
- 4) Microscopic studies can be carried out on a few examples of each facies identified at a mesoscopic scale, thus decreasing the amount of time necessary for analysis.

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A CATHODOLUMINESCENCE MICROSCOPE FOR LOW INTENSITY LUMINESCENCE¹

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INTRODUCTION

A cathodoluminescence instrument equipped with a cold cathode and an oblique (Technosyn Ltd., Cambridge, U.K.) or deflected (Nuclide Corporation, Acton, MA 01720, U.S.A.) electron beam may be suitable for investigating carbonates and certain feldspars. However, these conventional instruments (Gallup 1936; Smith and Stenstrom 1965; Long and Agrell 1965; Sippel 1965; Zinkernagel 1978; Steyn et al. 1976) do not allow detection of the faint and rapidly-changing short-lived luminescence typical for authigenic quartz, chalcedony and anhydrite. They work with an excessively high beam current density which leads, within a short time, to a change in the luminescence intensity and color (Fig. 1A, B).

In this paper we describe the design and working conditions of an improved cathodoluminescence microscope using a layout similar to that described by Zinkernagel (1978). The high sensitivity of the hot-cathode instrument provides the unique capability to detect weak and

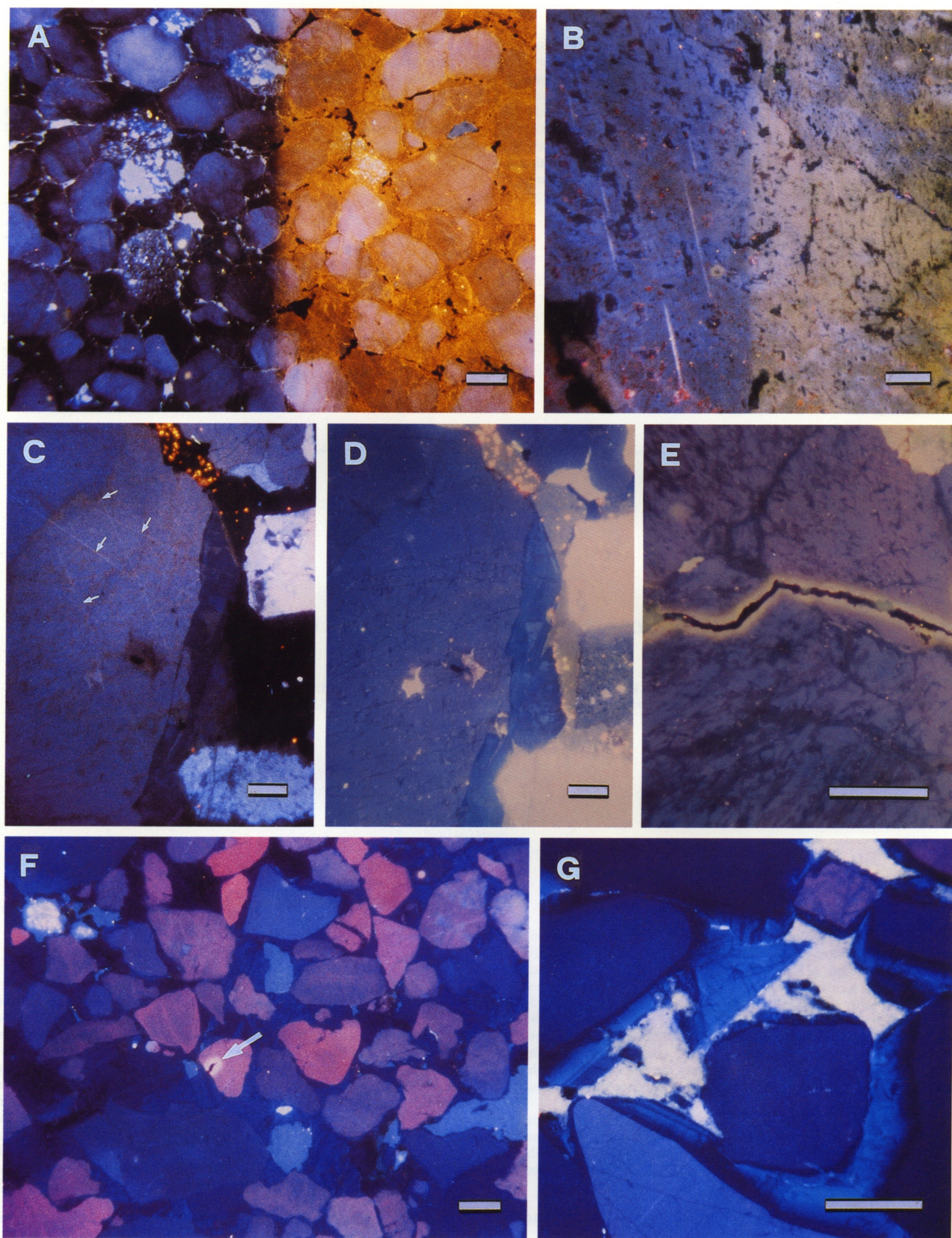
short-lived luminescence in minerals with virtually no deterioration of the luminescence during observation (Fig. 1D).

INSTRUMENT

Figure 2 shows a schematic cross section of the instrument. Bombardment of the thin section with electrons takes place from the sample side and observation of the luminescence from the reverse side. The observed luminescence intensity is proportional to the inverse square of the working distance; thus our array is superior to commercially-available set-ups with observation and electron-bombardment from the same side, as it provides a much smaller working distance between the luminescence source and objective lens. Use of a hot cathode with a directly-heated tungsten filament as the electron source produces a stable, easily adjusted beam but has the concomitant disadvantage of requiring a vacuum better than 10^{-3} Pa, only obtainable with a high vacuum pumping system. The choice of a turbomolecular pump has the advantage of a fast, clean high vacuum system that is automatically vented in case of power failure.

Free electrons produced by thermal emission from the heated tungsten

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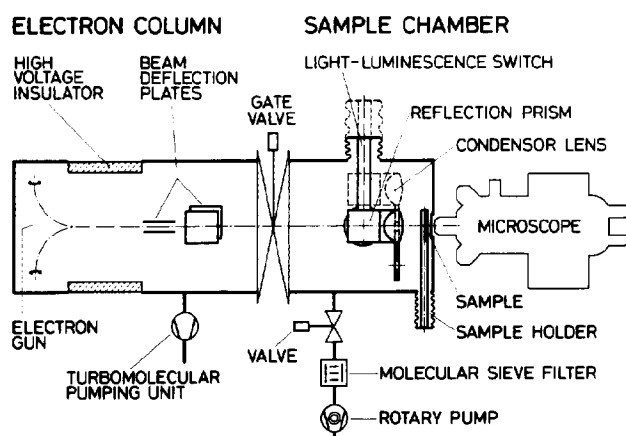


FIG. 2.—General design of the high sensitivity cathodoluminescence microscope.

filament (typical lifetime 3,000 h) are deflected by 90 degrees to separate them from the filament glow. The main electron acceleration field functions as a convex lens, so that a focused electron beam is emitted at any acceleration potential from 2.5 to 50 kV. Beam current densities between 5 nA/mm² and 10 μA/mm² can be obtained at the sample location.

The electron column containing the electron gun and four beam deflection plates are separated by a gate valve from the sample chamber with the sample holder. During sample exchange the electron column is kept under high vacuum. The whole procedure is automated and takes less than 15 minutes. An optical system (reflection prism and condenser lens) is moved into the path of the electron beam for observation in transmitted light. Vacuum interlocks switch off high voltages and filaments at pressures above 10⁻³ Pa. A lead casing around the sample chamber protects the operator from X-rays produced by the electron bombardment of the sample.

Located next to the sample chamber is a specially-adapted Zeiss petrographic microscope (Fig. 3) for visual or photographic observations under luminescence or transmitted light (plane- or cross-polarized). Both analyzer and polarizer are connected to a low torsion flexible shaft and turn simultaneously in the crossed position (Zinkernagel 1978). The need to turn both polarizer and analyzer simultaneously dictates a dou-

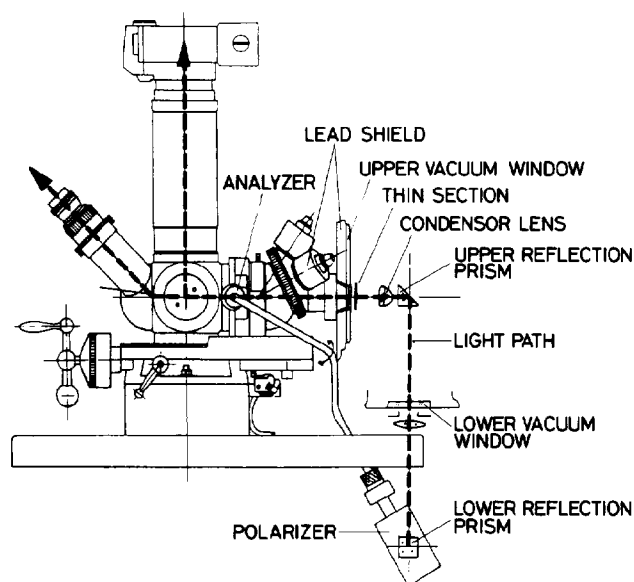


FIG. 3.—View of the optical system.

ble reflection of the polarized light en route between polarizer and analyzer. The effects of these two reflections are compensated for by employing a 3-dimensional reflection path. The microscope is mounted on a precision sliding table with a crank gear permitting change of objectives and focusing. A 10× photographic ocular gives magnifications of 7.7×–83× on a 24 × 36 mm film.

PARAMETERS INFLUENCING THE LUMINESCENCE INTENSITY OF QUARTZ

The relationship between electron energy, beam current and luminescence intensity for quartz (data from Coy-Y11 1969) is such that, for a given acceleration potential, there is a saturation point beyond which an increase in beam current does not produce a further increase in luminescence intensity. To clarify this relationship, the luminescence intensity in quartz was determined for 56 different combinations of beam current densities (0.1–0.6 μA/mm²) and electron energies (2.5–30 keV). A 3-dimensional plot (Fig. 4) of electron energy, beam current density and luminescence intensity shows that the most favorable conditions for the highest luminescence are at 0.4–0.5 μA/mm² and 25–30 keV. The luminescence characteristics of K-feldspar, calcite and detrital or authigenic quartz are differentiable even with an electron energy of 3 keV and a beam current density of 0.3 μA/mm² (Fig. 1C).

An additional increase in the luminescence intensity is obtained by evaporation of a conductive aluminium layer onto the thin section surface. This metal layer not only prevents surface charging but also acts as a mirror and thus enhances the luminescence intensity by reflecting the backward-directed (i.e., towards the electron gun) component of the luminescence.

The resolution of the luminescence pattern depends not only on the intensity of the luminescence but also on the electron penetration depth. At low electron energies (< 5 keV), the maximum electron penetration in quartz is only a few tenths of μm, whereas at higher electron energies (> 30 keV) this depth increases to more than 8 μm (Weber 1964) leading to a decrease in resolution.

In addition, luminescence generated by high energy electrons gives inferior resolution at oblique grain contacts or with very fine grains. However, if the penetration depth is small (e.g., Fig. 1C), all the surface irregularities are imaged. Good results at low voltages are thus only attainable from highly polished thin sections.

EFFECT OF ELECTRON BOMBARDMENT ON LUMINESCENCE COLORS

The visible effect of prolonged or heavy electron bombardment on quartz luminescence is alteration of the initial violet, blue, bottle-green,

FIG. 1.—Cathodoluminescence photomicrographs from quartz, K-feldspar and anhydrite. Scale bar = 250 μm.

A) Effects of electron bombardment on luminescence color and intensity in quartz. The left side of the sample was exposed to the electron beam (30 keV, 0.2 μA/mm²) for 1½ minutes, whereas the right side was bombarded with electrons for 40 minutes.

B) Effects of prolonged electron bombardment on K-feldspar crystallized at low temperature. The left side of the sample is a normal exposure (8 s at 30 keV and 0.3 μA/mm²), whereas the right side had previously been bombarded with electrons for one minute.

C) Luminescence of detrital and authigenic quartz, K-feldspar and low temperature albite obtained with an electron energy of 3 keV. The small penetration depth of electrons causes the very shallow (< 0.1 μm) surface scratches (arrows) to be visible.

D) Same field of view as in C, but obtained with 30 keV electron energy.

E) Natural alteration of the luminescence color in quartz. The alteration along the fracture is caused by α-radiation.

F) Anhydrite-cemented quartz sandstone. The short-lived dark blue luminescence color in anhydrite cement exhibits growth zonation. The arrow points to a detrital quartz grain containing a zircon inclusion. Alpha-particles resulting from natural radioactive decay have produced the light luminescence halo in the quartz around the zircon.

G) Zoned quartz overgrowth as revealed by short-lived cathodoluminescence. The well-rounded detrital grains are easily distinguished from their angular quartz overgrowths.

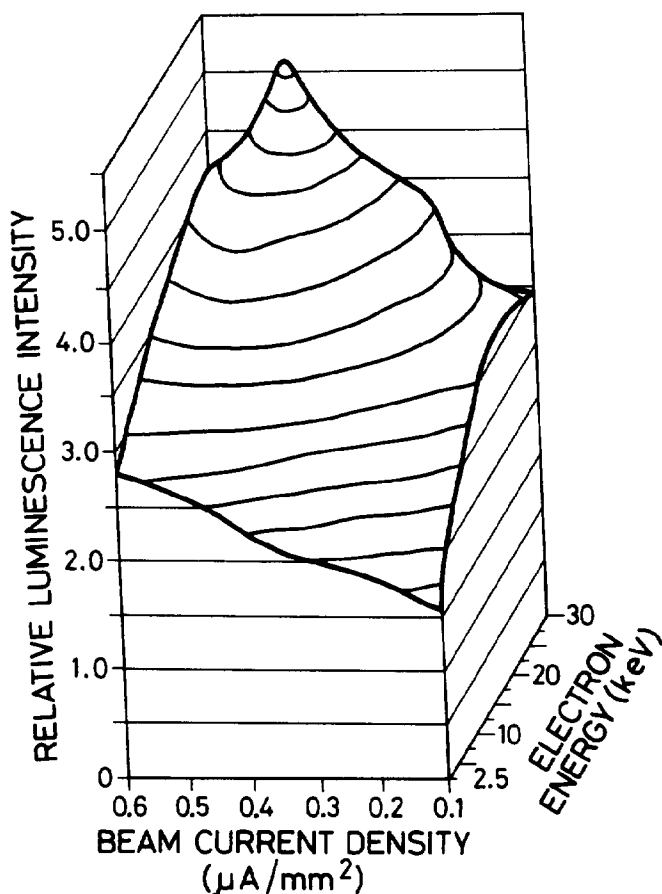


FIG. 4. — Relationship between electron energy, beam current density and relative luminescence intensity.

or yellow luminescence colors as well as non-luminescent quartz to various shades of reddish-brown (Fig. 1A, B). This alteration is non-reversible at room temperatures and starts immediately at the onset of electron bombardment (Ramseyer et al. 1988). This phenomenon (first documented by Zinkernagel 1978) is marked by an increase in intensity of the yellow-red range combined with a decrease in the blue range of the spectrum. The resulting brown luminescence color represents a broad peak in the visible spectrum centered around 650 nm wavelength with a half width of approx. 150 nm (Coy-Yll 1969; Koyama et al. 1977).

Continued electron bombardment of authigenic quartz or anhydrite causes a rapid decay of the short-lived luminescence in the depth range of electron penetration (Ramseyer 1983). Only this short-lived luminescence displays growth characteristics such as zonation in quartz or anhydrite (Fig. 1F, G).

Our experiments using temperature-indicating labels on the reverse side of the thin section glass indicate that the temperature during con-

tinuous electron bombardment at 30 keV and $0.3 \mu\text{A}/\text{mm}^2$ was between 88 and 93°C. We estimate the temperature of the thin section to be < 20°C higher than this value. Thermal effects are thus unlikely to cause the observed luminescence alterations; indeed, heating quartz to 450°C for one hour restores the luminescence.

Crystal lattice effects are more likely to control luminescence colors in quartz, as shown by the brown-luminescing alteration haloes around uranium- or thorium-bearing zircon inclusions (Fig. 1E, F). Calculations of the penetration depth of α - or fission particles (Marmier 1980) in quartz confirm that α -particles from the uranium or thorium decay series are able to penetrate to depths similar to the value of $28 \pm 8 \mu\text{m}$ measured for the brown-luminescing alteration haloes. Furthermore, the brown-luminescing layer produced by prolonged electron bombardment at 30 keV can be removed by planing off the uppermost 5–10 μm of the sample. We conclude that particle bombardment and not thermal effects causes the natural and induced alterations in luminescence.

The alteration of luminescence color and the loss of short-lived luminescence are thus both caused by the destructive effect of electron bombardment on the crystal lattice. It is therefore important to specify the exact working conditions when comparing luminescence results.

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