LASER MICROSCOPY - FAMM

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Introduction

FAMM^{TM} (fluorescence alteration of multiple macerals) is a technique developed in CSIRO during 1989-1991 by R. Wilkins and J. Wilmshurst for the assessment of thermal maturity of coals and dispersed organic matter. It has been especially designed to deal with the problems posed by

- Suppression (and enhancement) of vitrinite reflectance.

It is also a very useful tool in cases were vitrinite is rare or absent The technique can be applied to material in the rank range of 0.4% - 1.2% vitrinite reflectance (R_r).

The CSIRO purpose built microprobe uses a 488 nm argon ion laser for excitation and projects a probe spot of about 2-micrometer diameter on to the sample to be analysed. The instrument consists of three modules, the microprobe, the laser/electronics module, and a computer system. The instrument uses fibre optics for laser delivery to the analysis point. The built-in focus aid allows the operator to easily focus on a sample (conventional microscopical polished block). An active safety system protects the operator from accidental exposure to the laser beam.

The microprobe consists of an Olympus BX60 microscope and a fibre optic based laser delivery system. See Figure 1 for details.

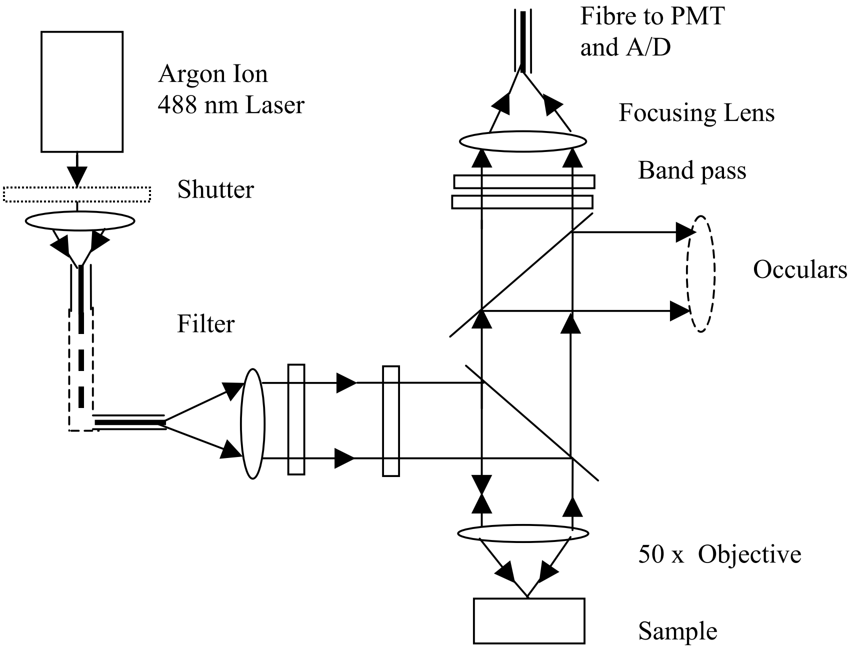

Figure 1. Simplified diagram of the microprobe

Fluorescence alteration

The first detailed studies on fluorescence alteration were published by Teichmüller & Ottenjann (1977). Since then several workers have presented results of alteration analyses on vitrinites, liptinites and inertinites.

Fig. 2 shows the typical change in shape of vitrinite alteration curves with increasing depth for the Australian North West Shelf well Dampier-1. Although the shape of the alteration curve gives a rough indication of rank, the curve shape and position is also affected by the H/C ratio of the vitrinite, which can be unusually high for the given rank (perhydrous) or low (subhydrous).

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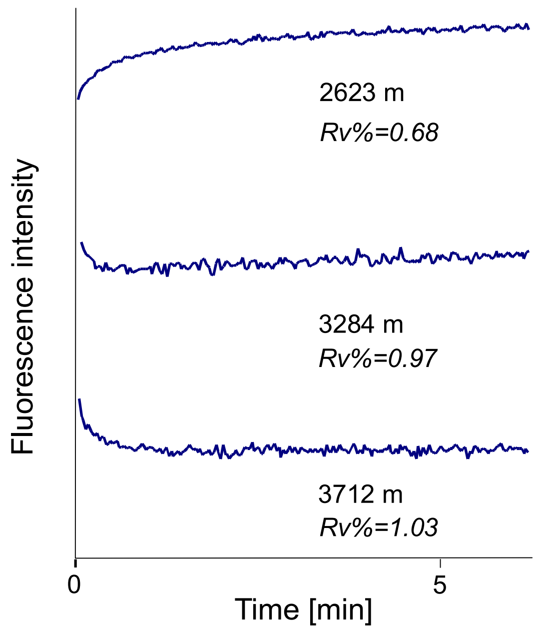

Figure 2. Fluorescence alteration curves for vitrinites from the well Dampier-1, North West Shelf, Australia illustrating changes in shape from positive (2623m) to negative (3712m) alteration with increasing depth. [after Wilkins et al., 1992]

Oxidation effects

Oxidation of samples is an important potential source of error in FAMM^{TM} analyses. Oxidation of surfaces by air exposure results in a decrease in fluorescence intensity of the macerals (Hagemann et al., 1989). In porous rocks such as many coals and sandstones the interior of the samples may be affected as well as the surfaces.

Figure 3 shows the change of fluorescence of coal vitrinite in epoxy resin mounts measured soon after preparation and again after 18 months exposure to air. These data show

- The rate of oxidation of coal is related to rank as well as to composition for coals of similar rank.
- Oxidation most affects high and medium volatile bituminous coals, especially those with perhydrous compositions, and least affects sub-bituminous and probably low volatile bituminous coals.

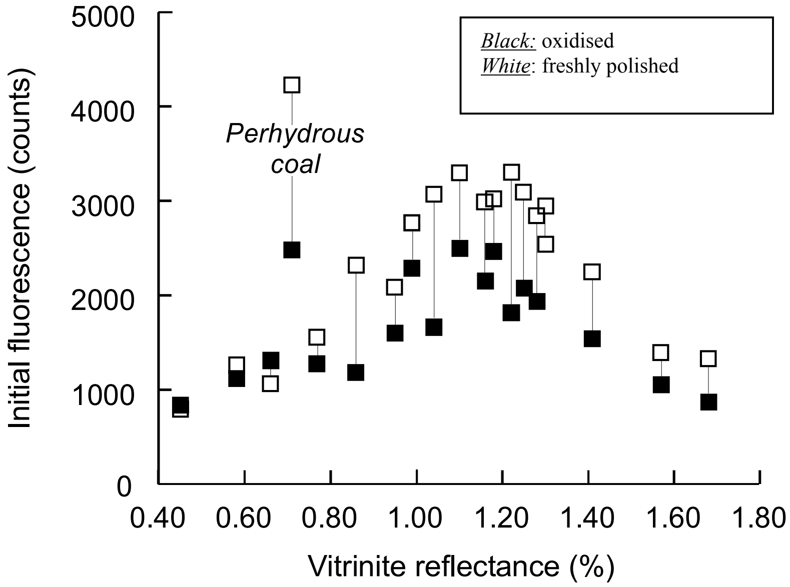

Figure 3. Influence of oxidation on the initial fluorescence intensity of polished surfaces of coal measured after freshly polishing, and again after 18 months exposure to air without repolishing [after Wilkins et al., 1998].

- A significant reduction in fluorescence intensity occurs after just one or two days air exposure and the intensity continues to fall with further exposure.

The problem of oxidation can be overcome by analysing freshly ground and polished samples.

Fluorescence Alteration – Rank, Suppression and Enhancement.

FAMM results are normally presented as the final (400s) fluorescence intensity versus the ratio of the final to the initial fluorescence intensity,

- The final (400 s) fluorescence intensity is predominantly related to the H/C ratio of the maceral, and
- The ratio of the final to the initial fluorescence intensity is a rank indicator.

Vitrinites of orthohydrous ('normal' with regard to their rank) composition plot on or close to the sub-vertical line (the J-curve or 'normal' vitrinite line), which represents the maturation pathway for orthohydrous telovitrinite. The normal vitrinite line is derived from a suite of well-defined reference coals it is used to calibrate the diagram in terms of equivalent mean random vitrinite reflectance (EqVR). In the original form of this diagram, the calibration was derived from a suite of mainly Australian Permian coals (Fig. 4). For coals and dispersed organic matter containing 'normal vitrinite', it is found that points on the diagram representing a range of vitrinite, inertinite and liptinite macerals fall close to a parabolic curve that intersects the 'normal' vitrinite line at EqVR. An example of the fluorescence alteration diagram for such a coal is shown in Fig. 5. Note that whereas the orthohydrous (normal) vitrinites plot near the centre of the diagram, the inertinites plot in the low fluorescence intensity field and the liptinites plot in the high fluorescence intensity field.

In summary: orthohydrous vitrinites plot on the J-curve, subhydrous vitrinites and inertinites plot on the left side of the J-curve, perhydrous vitrinites and liptinites on the right.

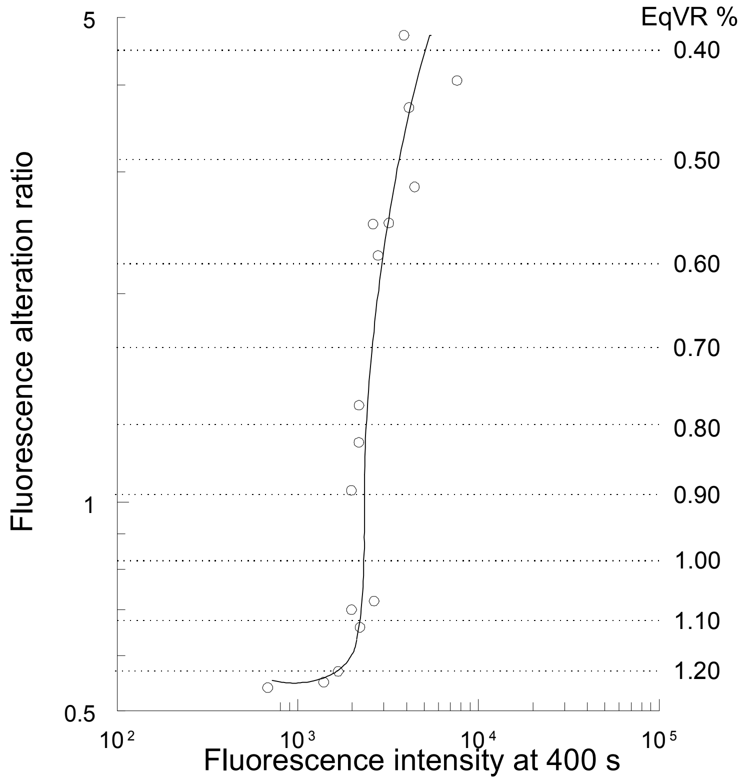

Figure 4. Determination of the 'normal' vitrinite line for a suite of mainly Permian coals from Eastern Australia. Each point defining the curve is the average value of 10 analyses on telovitrinite per coal sample

A number of calibrated fluorescence alteration diagrams have been prepared for coals of different age and geographical distribution.

These diagrams include samples from the

- Australian Permian-Triassic
- Australian Jurassic
- Indonesian Tertiary
- Netherlands Carboniferous.

Others are in process of preparation. The basic framework of all diagrams is the same with differences being accommodated by small differences in the lateral (left-right) position of the 'normal' vitrinite line and the accompanying suppression iso-correction curves. (See Fig. 6) The reasons for the differences between these diagrams are poorly understood but may at least in part due to differences in flora precursors.

If a J-curve for the analysed sample is not available, the degree of suppression/enhancement is easily determined by subsequent VR measurements on the same grains, which were used for the FAMM analyses.

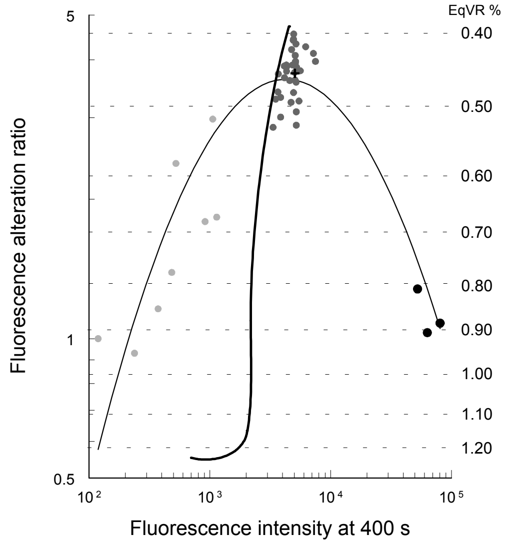

Figure 5. Fluorescence alteration (FAMM™) diagram for an Australian orthohydrous coal with a measured vitrinite reflectance of 0.42 %.

Light grey: inertinite, *medium grey:* vitrinite, *black:* liptinite

Where possible a second-order polynomial curve (multi-maceral curve) is fitted to the data. The curve allows to read the EqVR from its maximum (when the curve is curving upwards; up to EqVR values of ~0.9%) and its minimum (when the curve is curving downwards) and indicates according to the position of the maximum relative to the J-curve if the reflectance is suppressed/enhanced or not. An additional advantage of this method is that it does not require the unequivocal identification of a maceral and it is theoretically possible to derive an EqVR value for a sample that does not contain vitrinite or at least derive an estimate. This graphical approach normally works well for coals but may cause some difficulties with dispersed organic matter, where not always sufficient amounts of in situ macerals from all three maceral groups are present. In these cases the EqVR is calculated from the mean of the vitrinite data points on the fluorescence alteration diagram.

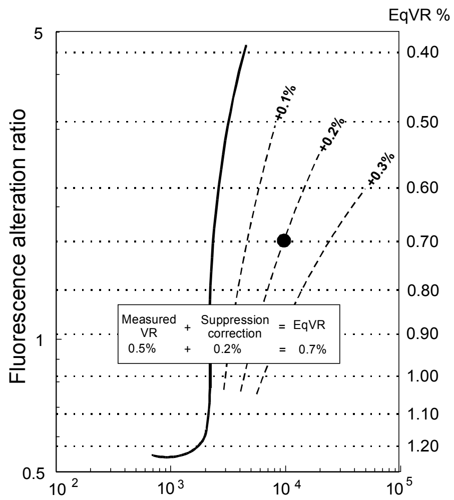

Figure 6. Fluorescence alteration (FAMM™) diagram showing generalised suppression iso-correction curves and an example for a vitrinite reflectance suppression of 0.2 %. [after Wilkins et al., 1995]

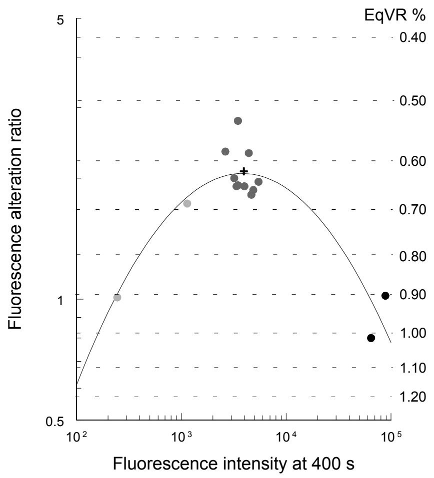

Figure 7. FAMM diagram for an Oligocene coal from Mequinenza, Spain, measured VR: 0.30% R_r EqVR: 0.61% .
Light grey: inertinite, *medium grey:* vitrinite, *black:* liptinite

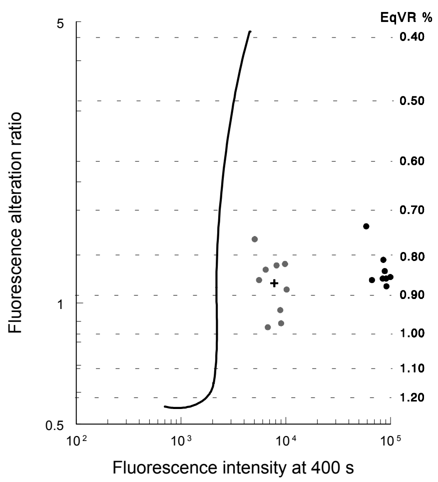

Figure 8. FAMM diagram for a Permian coal from the Leping Mine, Jianxi Provence, China
Measured vitrinite reflectance: 0.57%R_r;
EqVR: 0.85%.
Medium grey: vitrinite, *black:* liptinite

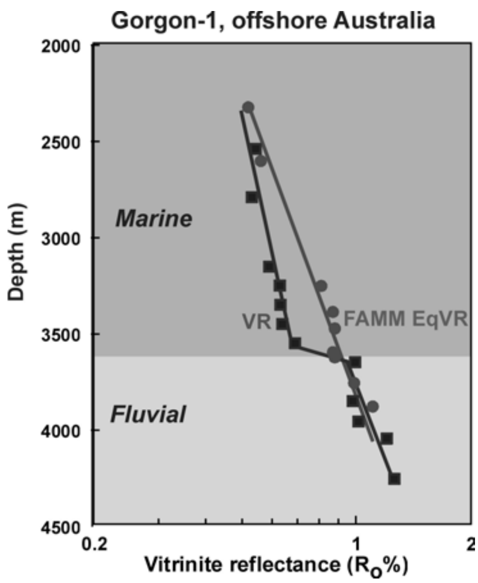

Figure 9. FAMM and VR data from a marine and a fluviatile sequence offshore Australia, the VR in the marine sequence being significantly suppressed. Wilkins et al. (1994).

Figures 7 – 9 show examples of FAMM analysis results and their application. In Fig. 7 the FAMM diagram of an Oligocene coal sample from Mequinenza (Spain) shows the effect of high suppression due to a highly reducing and bacterial rich depositional environment (for details see: Querol et al., 1995), which may account for hydrogen-rich peatification/coalification products, (Teichmüller and Teichmüller, 1982). Carbon content, moisture and calorific value and spectral fluorescence data indicate a rank of a sub-bituminous to high volatile bituminous coal whereas the reflectance is significantly lower (0.30%R_r). The available J-curves do not apply to the reflectance suppression in this sample but though no normal vitrinite calibration is available, the EqVR seems to indicate the rank accurately.

Fig. 8 shows an example of a cutinite rich coal (65% cutinite; for details, see: Pickel et al., 1993) - from the LePing district in China, which also has a significantly suppressed vitrinite reflectance, probably due to the impregnation of vitrinite with newly generated hydrocarbons from the cutinite. This vitrinite reflectance suppression is clearly detected by FAMM.
The graph in Fig. 9 demonstrates the importance of the control on suppressed vitrinite reflectance for the assessment of accurate thermal maturity data.

Summary

FAMM^{TM} offers an easily applicable and reliable method for rank determination esp. in cases where it is of importance to exclude the influence of vitrinite reflectance suppression or enhancement. The degree of suppression/enhancement can be quantified.
It has the additional advantage that the measurement area is very small (1.8 micron) due to the use of a laser as an excitation source. Thus in contrast to 'classical' petrological methods, even very small macerals can be analysed.

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