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Clay mineral evidence for low-grade Caledonian and Variscan metamorphism in south-western Dyfed, south Wales

D. ROBINSON, R. A. NICHOLLS

Department of Geology, The University, Bristol, UK

AND

L. J. THOMAS

Department of Geology, The University, St. Andrews, Fife, Scotland

SUMMARY. Illite crystallinity determinations on Palaeozoic pelitic rocks, whose stratigraphic range runs from Lower Cambrian to Westphalian, indicate that anchimetamorphism has affected both the Lower and Upper Palaeozoic sequences. Two metamorphic episodes are in evidence, with the earlier, Caledonian, being of slightly higher grade. The higher anchizone crystallinity values are recorded from the Fishguard area in which mineral assemblages of the prehnite-pumpellyite facies have recently been recognized in basic igneous rocks. The later metamorphic episode has affected rocks to the south of the Variscan front. Here crystallinity values are low anchizone but some straddle the boundary with the diagenetic state. The Pembroke coalfield lies in this southern area and has coals largely of anthracite rank with volatile matter contents of between 10.1 and 5%. Grade of metamorphism as indicated by crystallinity and by coal rank data from the Pembroke coalfield shows anomalous results to that described from the main South Wales coalfield. A neo-formed $2M$ illite from the Variscan spaced cleavage is described with analytical and X-ray diffraction data.

THE south-western region of Dyfed, consisting largely of Palaeozoic rocks, can be divided into two broad zones disposed north and south of the Variscan front which is marked by the junction of the Caledonides and Variscides (Dunning, 1964). To the north (fig. 1) there is a largely Lower Palaeozoic clastic succession dominated by rocks of Ordovician age and with considerable volumes of Caledonian igneous rocks. These Lower Palaeozoic rocks underwent deformation during the Caledonian orogeny and it is generally accepted that low-grade metamorphism accompanied the deformation (Rast, 1969), but there is little evidence on the matter. Recently, however, pumpellyite-bearing metamorphic assemblages have been described from the Fishguard Volcanic Group (Bevins, 1978) and the grade of metamorphism is given as within the prehnite-pumpellyite facies.

The rocks to the south of the Variscan front are largely Upper Palaeozoic in age, and in the Marloes Sands section (localities 21-40, fig. 1) the quasi-conformable transition from the marine Silurian into ORS facies rocks is seen (Allen and Williams, 1978). The rocks in this area too have undergone considerable deformation, with the development of major east-west folds (Tringham, 1979). A spaced cleavage is present in mudstones and sandstones of all ages, but becomes of regional character and of a pervasive nature only in the southern part of the Pembroke coalfield. This area is shown as unmetamorphosed on the metamorphic map of Europe (Zwart and Sobolev, 1973).

Experimental. Illite crystallinities were measured on a total of 67 pelitic samples (mudstone/shale/slate). The dominant whole-rock mineral assemblage is quartz-illite-chlorite, with minor feldspar and pyrophyllite in some of the Lower Palaeozoic samples and kaolinite in some of the Upper Palaeozoic samples. Mixed-layer clays were recognized in a number of Carboniferous samples but these samples were not included in the determinations. The $< 2\ \mu m$ fraction from mechanically powdered (porcelain ball-mill) samples was separated by centrifugal and subsequent filtration techniques. The crystallinity determinations were made on air-dried smear mounts of the separated material, using a Phillips PW1010 diffractometer with Ni-filtered Cu-$K\alpha$ radiation at a tube voltage of 40 kV and current 20 mA; divergence and scatter slits were 1° and receiving slit 0.1 mm. The illite diffraction pattern was determined

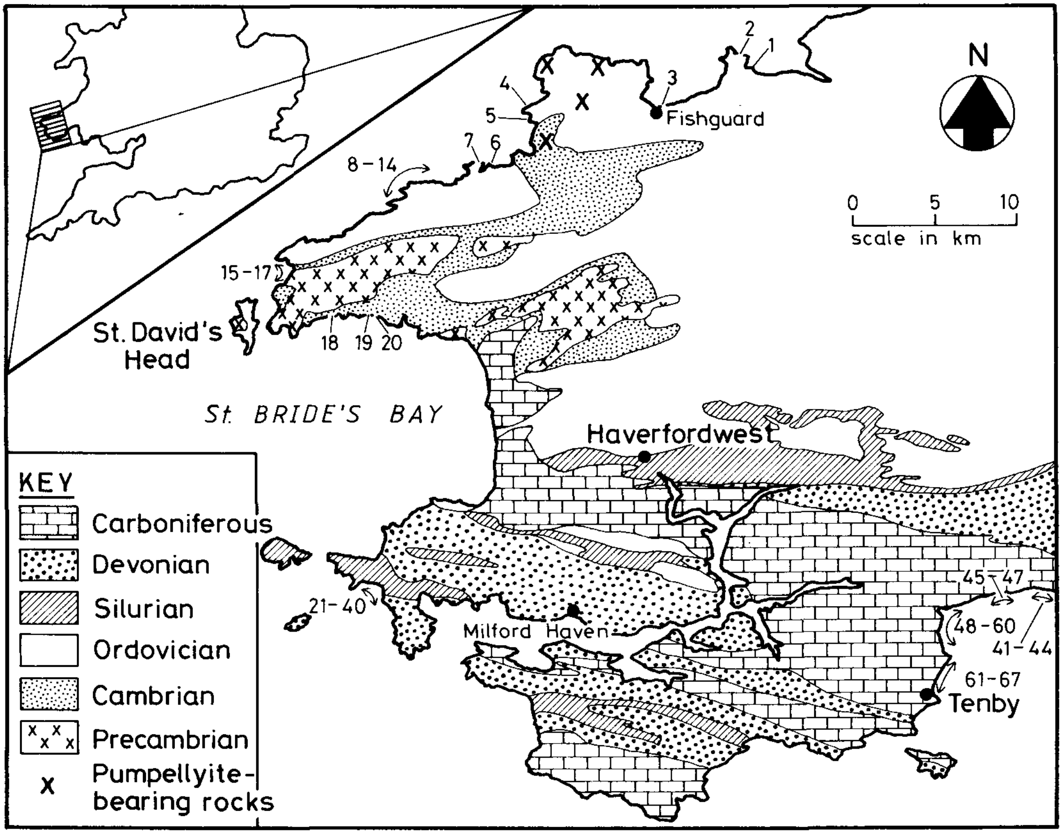

FIG. 1. Simplified geological map of south-western Dyfed, showing sample locations.

from hand-picked material using a Debye-Scherrer powder camera of 11.46 cm diameter.

Illite crystallinity. A numer of indices have been proposed to characterize illite crystallinity and distinguish diagenesis from low-grade metamorphism. In this work, two indices were determined on each sample:

A. Weaver index: the ratio of the peak minus background heights at 10 Å and 10.5 Å (Weaver, 1960). This is an index particularly suited to the study of poorly crystalline illites encountered at diagenetic stages (Kubler, 1968). Values < 2.3 are taken to indicate the diagenetic zone, 2.3 to 12.1 anchizone, and > 12.1 the greenschist facies (Weaver, 1960; Kubler, 1968).
B. Kubler index: the peak-width of the 10 Å illite peak measured at half-peak height above background (Kubler, 1967; Dunoyer de Segonzac *et al.*, 1968; Kubler, 1968).

In this work, measurements of peak-width have been made in units of degrees 2θ. The Kubler index has been widely adopted in continental Europe but comparisons between different laboratories prove difficult because this index is an absolute measurement and variations in machine condition will obviously alter the peak-width values. The ranges of some peak-width values (mm) that have been used in demarcating boundaries are shown below:

Diagenesis	Anchizone	Greenschist	Source
> 4.0	4.0–2.5	< 2.5	Kubler (1968, fig. 2)
> 7.75	7.75–4.0	< 4.0	Kubler (1968, fig. 3)
> 8.5	8.5–5.0	< 5.0	Kubler (1968, fig. 4)
> 7.5	7.5–4.0	< 4.0	Frey (1970, fig. 3)
> 5.5	5.5–3.5	< 3.5	Dunoyer de Segonzac (1970, fig. 11)
> 4.5	4.5–2.5	< 2.5	Dunoyer de Segonzac (1970, fig. 12)
> 2.5	2.5–1.5	< 1.5	Stalder (1979, fig. 3)

The 67 samples examined for illite crystallinity range in age from Lower Cambrian to Westphalian. Although these are relatively small in number they do represent virtually the full stratigraphic range exposed in this part of Wales. Sample localities are as shown in fig. 1, the Weaver indices and peak-width values in Table I, and the two

TABLE I. *Sample locations, Weaver, and peak-width values*

Sample	G.R.	Weaver value	Peak-width degrees 2θ	Stratigraphic horizon
1	SN017400	8.5	0.20	Caradocian
2	SN015403	5.0	0.24	Llandovery
3	SM952376	6.1	0.24	Llandeilo
4	SM886364	8.0	0.22	Caradocian
5	SM887364	10.5	0.26	Caradocian
6	SM852337	8.1	0.19	Arenig
7	SM852337	7.6	0.21	Arenig
8	SM814327	8.5	0.21	Arenig
9	SM813328	9.4	0.23	Upper Cambrian
10	SM812325	3.8	0.31	Arenig
11	SM794315	4.9	0.21	Llanvirn
12	SM796312	6.5	0.21	Llandeilo
13	SM796310	9.1	0.20	Llanvirn
14	SM797307	4.0	0.29	Llanvirn
15	SM732276	9.8	0.18	Arenig
16	SM728261	20.9	0.18	Lower Cambrian
17	SM724262	15.8	0.17	Middle Cambrian
18	SM753243	12.5	0.19	Lower Cambrian
19	SM799240	8.9	0.22	Middle Cambrian
20	SM802238	6.5	0.24	Upper Cambrian
Marloes Sands section				
21	SM789067	3.8	0.37	U. Wenlock/L. Ludlow
22	SM789067	3.2	0.40	U. Wenlock/L. Ludlow
23	SM789067	4.4	0.30	U. Wenlock/L. Ludlow
24	SM789067	5.3	0.30	U. Wenlock/L. Ludlow
25	SM789067	5.0	0.31	U. Wenlock/L. Ludlow
26	SM790067	5.0	0.28	U. Wenlock/L. Ludlow
27	SM790067	4.3	0.28	U. Wenlock/L. Ludlow
28	SM790067	4.7	0.32	U. Wenlock/L. Ludlow
29	SM789068	4.7	0.34	U. Wenlock/L. Ludlow
30	SM789068	5.2	0.33	U. Wenlock/L. Ludlow
31	SM789068	4.7	0.32	U. Wenlock/L. Ludlow
32	SM790068	6 0	0.31	Wenlock
33	SM789068	3.7	0.38	Wenlock
34	SM789068	5.2	0.30	Wenlock
35	SM790068	4.0	0.40	Wenlock
36	SM790069	4.9	0.30	Wenlock
37	SM789070	3.9	0.34	Wenlock
38	SM789071	3.2	0.36	Wenlock
39	SM788071	4.6	0.29	U. Llandovery/L. Wenlock
40	SM788071	4.3	0.31	U. Llandovery/L. Wenlock
Carboniferous				
41	SN220071	4.5	0.19	Namurian
42	SN218072	2.7	0.39	Namurian
43	SN217072	1.6	0.44	Namurian
44	SN217072	2.5	0.52	Namurian
45	SN189073	2.2	0.36	Namurian
46	SN185072	2.6	0.42	Namurian
47	SN183073	2.4	0.44	Namurian
48	SN161069	3.8	0.21	Coal Measures
49	SN159063	3.1	0.34	Coal Measures
50	SN156066	2.2	0.46	Coal Measures
51	SN154064	2.2	0.48	Coal Measures
52	SN150063	2.9	0.33	Coal Measures
53	SN149062	2.3	0.49	Coal Measures
54	SN148062	2.4	0.51	Coal Measures
55	SN143055	2.7	0.39	Coal Measures
56	SN142053	2.8	0.45	Coal Measures
57	SN138046	3.0	0.36	Coal Measures
58	SN139044	2.7	0.38	Coal Measures
59	SN140041	2.8	0.38	Coal Measures
60	SN141040	2.8	0.34	Coal Measures
61	SN145031	2.8	0.36	Coal Measures
62	SN145030	3.1	0.40	Coal Measures
63	SN145029	2.6	0.33	Coal Measures
64	SN137020	3.4	0.29	Coal Measures
65	SN134013	3.6	0.24	Namurian
66	SN134011	3.0	0.38	Namurian
67	SN133010	3.0	0.37	Namurian

indices plotted against each other in fig. 2. The Weaver values show a range from 1.6 to 20.9, which extends from the diagenetic field (< 2.3) into greenschist facies (> 12.1) and is coupled with a greater than three-fold reduction in the peak-width. The results are noticeably grouped according to the stratigraphic position of the samples (Table I). The Lower Palaeozoic samples from the north of the area show the highest indices, those from the Marloes Sands section have intermediate values, while the lowest values recorded are from the Namurian and Coal Measures.

Lower Palaeozoic results. Weaver values for 40 samples range from 3.2 to 20.9 (Table I) which broadly indicates that anchimetamorphic grade has been attained in this general south-western region of Dyfed. North of the Variscan front, three of the Cambrian samples have crystallinities indicative of greenschist facies (Table I). Although these do come from a restricted zone on St. David's Peninsula, they are insufficient basis alone for identifying such a grade in this local area. The remaining 17 samples from this area have a mean Weaver index of 7.4 and a standard deviation of 2.05. These results, spanning the central portion of the anchizone, can be compared with the prehnite-pumpellyite facies established in the igneous rocks of the area (Bevins, 1978).

The 20 samples from the Marloes section, to the south of the Variscan front, show a range from 3.2 to 6.0 with a mean of 4.5 and a standard deviation of 0.72. The values all fall within the limits of the anchizone but are lower than those to the north (mean 7.4) and are restricted to the lower part of the anchizone. Evidence of metamorphic assemblages in the Skomer Volcanic Group, which outcrops in the Marloes Sands area, would thus help to elucidate whether the anchizone is equivalent to both the laumontite-prehnite and prehnite-pumpellyite facies (Frey, 1970), or just to the latter of these two facies (Weber, 1972; Stalder, 1979).

Small amounts of pyrophyllite have been recognized in diffraction traces from sedimentary rocks in the Marloes section (Nicholls, 1979) and this occurrence in the lower part of the anchizone is in accord with other recent records (Dunoyer de Segonzac, 1970; Frey, 1970, 1978).

The age of the metamorphic episode can simply be assigned to the Caledonian in the case of the Lower Palaeozoic rocks, with a small increase of grade from south to north toward the main part of the Welsh Basin. Marloes Sands rocks pose some

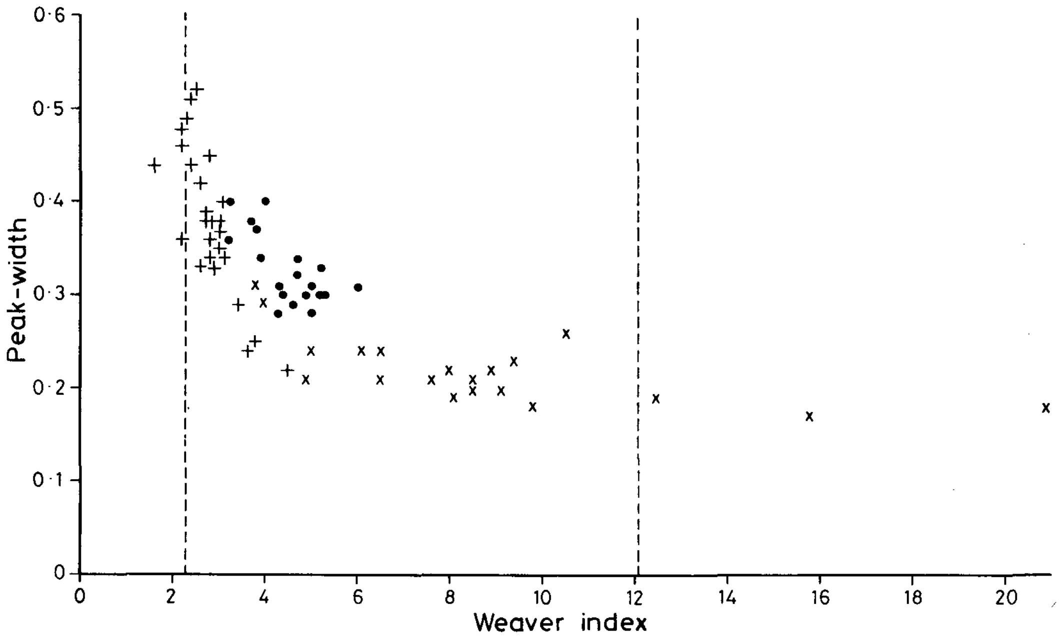

FIG. 2. Peak-width *v*. Weaver index
X Lower Palaeozoic samples north of the Variscan front
● Lower Palaeozoic samples south of the Variscan front
+ Upper Palaeozoic samples
The vertical dashed lines at Weaver indices 2.3 and 12.1 show boundaries between diagenetic zone and anchizone and between anchizone and greenschist facies respectively.

problems in that they show further features which can be assigned to the Variscan deformation and are considered below.

Upper Palaeozoic results. Twenty-seven samples of Namurian and Westphalian age from the eastern side of the Pembroke Coalfield (fig. 1) show a range of Weaver values from 1.6 to 4.5, with a mean of 2.8 and a standard deviation of 0.57, and therefore straddle the boundary of the anchizone with the diagenetic state. In the main South Wales coalfield, crystallinity studies by Gill *et al.* (1977) showed that Weaver values vary from 2.0 in the eastern part of the main coalfield to 6.0 in the north-west. Coal rank also increases from bituminous in the east to anthracite in the west with a decrease in volatile matter (VM) content from over 30% to under 6% respectively. Thus the Weaver values from the Pembroke coalfield are similar to those found in the lower grade eastern part of the South Wales coalfield where VM content is between 10 and 30%. However, the VM contents for coals from the Pembroke coalfield are lower and are similar to those reported from the anthracite region of the western part of the South Wales coalfield where Weaver values are between 4.0 and 6.0 (Gill *et al.*, 1979). Analyses of 145 coal samples from the Pembroke coalfield (Strahan and Pollard, 1915) show a range of VM content (dry, ash-free) from 10.1 to 5%, with a mean of 6.5% and standard deviation of 0.84%; these results are largely indicative of anthracite rank, with VM below 8% (Kisch, 1974).

Correlations of coal rank data with crystallinity indices and mineral facies are outlined below:

	Diagenetic	Anchizone	Greenschist	
VM%	> 28	28–4	< 4	Frey and Niggli (1971)
VM%	> 7.5	7.5–?	Not seen	Gill *et al.* (1979)
VM%	> 8.0	8.0–$\sim$4	< $\sim$4	Kisch (1974)
VM%	> 4.0	4.0–1.0	< 1.0	Stalder (1979)

A definitive correlation is not yet possible, and the major problem appears to be in correlation of the lower limit of the anchizone. Frey and Niggli (1971) tentatively correlate the lower part of the anchizone with the laumontite–prehnite facies and with VM contents of $\sim$ 28%, while Kisch (1974) and Stalder (1979) correlate the whole anchizone with the prehnite–pumpellyite facies. Although the results of the crystallinity indices and coal rank data from the Pembroke coalfield are not entirely compatible, they agree better with the proposal of Kisch (1974) in which the VM range for the anchizone is given as $\sim$ 4 to 8%. The cause of the discrepancies between the results for the main South Wales and the Pembroke coalfields is not yet known, but other examples have been reported and attributed to a variety of reasons (Kisch, 1974). These initial results from the Pembroke coalfield where the deformation is more intense than the main coalfield do, however, give support to the conclusion of Srodon (1979) that in tectonized areas clay diagenesis proceeds more slowly than coalification.

Evidence of elevated temperatures during the Variscan deformation is also available from fluid-inclusion studies of quartz veins which cut the Coal Measure rocks in the Pembroke coalfield. Homogenization temperatures of between 150 and 250 °C have been reported (Tringham, 1979), which can be used to give a guide to the possible temperatures during metamorphism. Tringham has suggested trapping temperatures in the region of 350 °C with an overburden thickness of 6.8 km, but these values seem somewhat high when compared with both the crystallinity and coal rank data. Temperatures in the 200 °C region are suggested for the lower anchizone (Winkler, 1976), but Gill *et al.* (1979) have proposed temperatures as low as 165 to 185 °C for their anchizone. Such low temperatures are difficult to reconcile with the occurrence of pyrophyllite (Frey, 1978).

The metamorphism affecting these Upper Palaeozoic rocks occurred after the end of the Westphalian deposition and prior to the Middle Triassic (Gill *et al.*, 1979), and was caused by a heat flux whose origin is as yet unknown.

The rocks of the Marloes sands sections lying to the south of the Variscan front must have suffered the effects of this Variscan metamorphic episode. Crystallinity values are lower anchizone and could, therefore, with regard to grade be equally ascribed to the Caledonian or Variscan metamorphic episode. To include them in the Caledonian episode would imply that a post-Caledonian molasse sequence has suffered metamorphism as a result of the parent orogenic event. A Variscan age of metamorphism would indicate that there was a time differential between the development of the illite crystallinity and the Variscan spaced cleavage (Graham *et al.*, 1977), as this cleavage cuts the fabric defined by the illite in these rocks.

Neo-formed illite. In the lower part of the Marloes Sands section the Variscan spaced cleavage cuts a volcaniclastic pebble conglomerate within which a greenish, fine-grained phyllosilicate mineral has developed. This suggests that the metamorphic process was continuing during the pressure solution activity, to give rise to the new mineral (Beach, 1979).

The mean of twelve electron microprobe analyses, the structural formula, and the X-ray diffraction pattern are shown in Table II.

Pretreatment by glycolation and heating

TABLE II. *Illite microprobe analysis, representing the mean of 12 determinations with 1 sigma value and X-ray diffraction results*

	Wt%	1σ	d	I
SiO_2	52.94	1.20	10.14 Å	s
Al_2O_3	27.31	0.96	5.02	w
TiO_2	nd		4.56	m
*FeO	3.25	0.59	4.35	mw
MgO	1.90	0.10	4.29	mw
CaO	nd		4.18	vw
Na_2O	nd		3.94	vw
K_2O	9.41	0.42	3.72	vvw
MnO	nd		3.43	vvs
Total	94.81		3.16	w
			2.99	vw
Ions on the basis of 11(O, OH)			2.78	w
Si	3.51	4.0	2.70	s
Al	0.49		2.56	mw
Al	1.65	2.02	2.52	mw
Fe^{2+}	0.18		2.41	w
Mg	0.19		2.38	w
K	0.80		2.15	mw
			2.00	mw
			1.90	w
			1.87	mw

* Total iron given as FeO.
nd-not detected.

revealed the presence of a small amount of expandable inter-layer material. The glycolation generated a small shoulder on the low 2θ side of the 10 Å peak, but did not lead to the resolution of any regular or irregular reflections. This, plus comparisons with the data of Weaver (1956) and of Hower and Mowatt (1966), suggests that the expandable content is at a minimum. The substitution of Al by (Mg, Fe) has occurred in the octahedral position with a corresponding increase in the Si occupancy of the tetrahedral site—a sequence similar to that seen in the muscovite-celadonite series (Velde, 1977). However, the inter-layer occupancy of 0.80 falls short of the 1.0 ± 0.10 per $O_{10}(OH)_2$ limit proposed by Velde. The mineral can be most readily linked to illite with a formula $M^+_{0.6-0.9}$ $(R^{2+}, R^{3+})_{2.00}(Si, Al)_4O_{10}(OH)_2$ where Si occupancy is in excess of 3.0 (Velde, 1977). The 060 spacing of the illite occurs at 1.503 Å, which corresponds to an octahedral content of (Mg+Fe) per $O_{10}(OH)_2$ of 0.41 (Maxwell and Hower, 1967), and corresponds well with the content of 0.37 determined by microprobe.

Comparison of the illite diffraction pattern (Table II) with the data of Yoder and Eugster (1955) and of Maxwell and Hower (1967) suggests that the illite is a $2M$ variety. Records and analyses of neo-formed illite, as distinct from material formed by recrystallization in pelitic rocks, are infrequent but Eslinger and Savin (1973) suggest good agreement between experimental and natural systems where $2M$ illite is restricted to environments in which temperatures have been in excess of 200 °C, although at high pressure (4.5 kb) $2M$ illite is stable as low as 125 °C (Velde, 1965). The lower limit of 200 °C would correspond well with fluid-inclusion data (Tringham, 1979) and low anchizone crystallinity evidence. Pressure at the time of illite origin is difficult to estimate but evidence from stratigraphic thicknesses of Silurian, ORS, and Carboniferous sequences (Walmsley and Bassett, 1976; Allen and Williams, 1978; Tringham, 1979) suggest that an overburden in the region of 8 km may have been present.

Conclusions. Lower Palaeozoic rocks to the north of the Variscan front in Dyfed have illite crystallinity values indicative of the anchizone. Lower Palaeozoic rocks to the south of the Variscan front have a lower crystallinity but are still anchizone and this is corroborated by the presence of pyrophyllite in some of these rocks. The presence of a Variscan-spaced cleavage cutting these later rocks allows the provisional interpretation that the illite crystallinity reflects the Caledonian episode.

A Variscan metamorphic episode has also affected rocks to the south of the Variscan front where a neo-formed $2M$ illite within the Variscan-spaced cleavage and anthracite coal rank in the Pembroke coalfield also suggest low anchizone.

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