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Contributions to Mineralogy and Petrology

Implications of K – Ar ages of whole-rock and grain-size fractions of metapelites and intercalated metatuffs within an anchizonal terrane

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Abstract. White mica bearing fractions ranging in grain size from 0.4 μm to 6.3–20 μm were separated from metapelites and intercalated metatuffs of the eastern Rheinisches Schiefergebirge (FRG). The stratigraphic age of these rocks is Middle Devonian (Eifelian), and they contain detrital material of northwestern provenance (Old Red Continent, probably mainly derived from the Caledonian Orogen). Folding in the Carboniferous was associated with cleavage formation and an apparently synkinematic anchizonal metamorphism. Apparent K – Ar ages of metapelite fractions display a marked positive correlation with grain size illustrating the detrital influence which is diminished with decreasing grain size and increasing metamorphism (determined by illite "crystallinity"). Contrasting grain morphologies observed by SEM enable the interpretation of apparent age/grain size relationship for coarse fractions. The anticipated lack of detrital mica in metatuffs is confirmed by the fairly consistent apparent K – Ar ages determined for the coarser than 0.63 μm size fractions which date the anchizonal metamorphism at ca. 330 Ma. Comparison of metatuff and metapelite apparent ages suggests that the extent of rejuvenation in the latter was largely dependent on grain size. Rejuvenation was also somewhat controlled by the degree of anchizonal metamorphism as suggested by differences in K – Ar results of metapelites which were metamorphosed at variable anchizonal conditions. Fractions < 0.63 μm from upper anchizonal metapelites record ca. 330 Ma ages similar to those of the 0.63–20 μm sizes in metatuffs. Together those results confirm the limited applicability of conventional K – Ar dating on bulk clay fractions (< 2 μm) of very-low grade (anchizonal) metamorphic rocks in dating metamorphic events and concomitant cleavage formation.

Introduction

Several workers have applied K – Ar isotopic techniques in an attempt to date thermal (diagenesis or anchizonal metamorphism) or deformational (cleavage formation) events and/or to investigate mineralogical changes associated with evolution of clay minerals. Hurley et al. (1963) and Hower et al. (1963) clearly documented the problem of separating authigenic illite from detrital muscovite. Bailey et al. (1962), Weaver and Wampler (1970), Perry (1974), and Aronson and Hower (1976) presented results indicating that partial resetting of whole-rock argon systems is not related only to increasing temperature and induced argon diffusion, but is also closely dependent upon mineralogical changes during diagenesis. Of principal importance is the liberation of potassium during the breakdown of detrital feldspar and/or muscovite which is fixed within authigenically formed illite layers. This results in release of radiogenic argon from detrital phases and a decrease in the K – Ar age for the whole-rock. K – Ar ages of constituent fine fractions are not significantly affected because they do not contain detrital minerals, which are enriched in the coarser fractions. Hunziker et al. (1986) evaluated the transformation of 1Md to $2M_1$ illite polytypes during metamorphism ranging from diagenesis to lower greenschist facies. They showed that the affect on K – Ar systems initially involved volume diffusion loss of radiogenic ^{40}Ar from detrital 1Md grains. At slightly higher metamorphic grades, restructuring processes led to initial formation of $2M_1$ illite, but rejuvenation of argon systems was not complete until total elimination of the detrital 1Md component. From a combination of in situ borehole temperatures and variations in K – Ar ages within the diffusion realm, Hunziker et al. (1986) suggested that temperatures in excess of ca. $260 \pm 30°$ C would be needed to totally rejuvenate intracrystalline argon systems within < 2 μm, 1Md illite grains.

Dating of cleavage formation and synkinematic low grade metamorphism was first attempted by whole-rock K – Ar analysis of slate/phyllite samples (e.g., Harper 1967; Dodson and Rex 1971). Although the results were reasonable from a regional geologic perspective, complete rejuvenation of detrital argon systems was not demonstrated. In an effort to avoid problems of only partially rejuvenated detrital grains, e.g., Ahrendt et al. (1977), Clauer and Kröner (1979), and Bonhomme et al. (1980) separated clay-sized fractions from shales and/or slates. These were thought to be enriched in metamorphic white mica formed under anchizonal conditions, and their K – Ar ages were interpreted to closely date cleavage formation and/or very-low grade metamorphism. More recently, K – Ar ages have been determined for various grain-size fractions prepared from individual samples (e.g., Frank and Stettler 1979; Hammerschmidt 1982; Kralik 1983; Huon 1985). However, the geologic significance of those results has been difficult to evaluate because of the effects of stratigraphy, source of detrital material, degree of metamorphism and/or deformational history.

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The present investigation was undertaken to systematically evaluate the possibility of dating anchizonal metamorphism by comparing the conventional K – Ar data to lithology, cleavage and stage of evolution of white micas. This was approached by carefully controlled grain-size separations on slates (containing detrital white mica) and intercalated metatuffs (containing no detrital white mica). Microstructure, mineralogy, and metamorphic degree of these samples were investigated and apparent K – Ar ages of various size fractions determined. Because the stratigraphic age, detrital material, source area and lithology of the metapelite samples are similar, it was hoped that potential variations in apparent K – Ar ages could be directly correlated with microstructural variations (depending on the metamorphic temperatures maintained during deformation). This work formed the basis of a Ph.D. dissertation at the University of Göttingen, and a complete listing of all data is available therein (Reuter 1985).

Geologic setting

Introduction

The present study was carried out on samples of slate and metatuff collected in the eastern Rheinisches Schiefergebirge (FRG). It is located in the northern, external part of the Central European Variscides, where Devonian and Carboniferous sedimentary and volcanic rocks are exposed (Fig. 1).

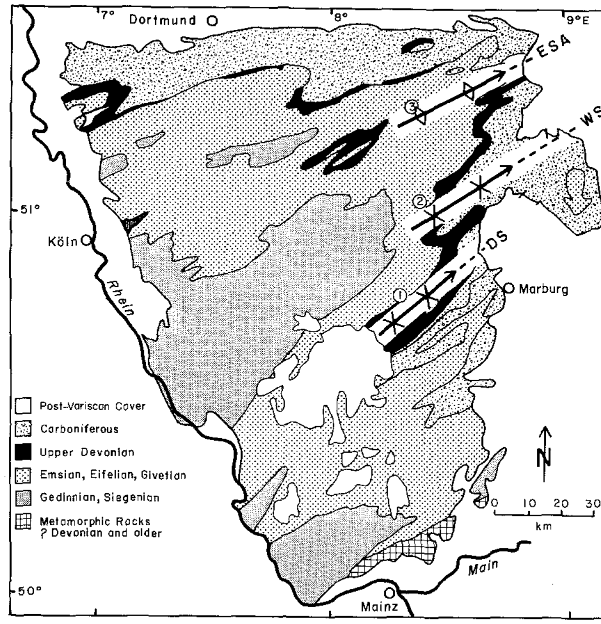

Fig. 1. Simplified geologic map of the eastern Rheinisches Schiefergebirge (after Schulz-Dobrick and Wedepohl (1983) Showing sample localities; *ESA* East Sauerland Anticline, *WS* Wittgenstein Syncline, *DS* Dill Syncline

Sedimentary history

The sedimentary history of the eastern Rheinisches Schiefergebirge is summarized in e.g., Meischner (1971), Franke et al. (1978) and Engel et al. (1983). A thick clastic, early Devonian shallow marine sequence (up to 4000 m) was deposited, the source area being the Old Red Continent in the northwest which in turn probably derived its detritus from the Caledonian Orogen. Main depocenters were initially situated in the southern part of the Rheinisches Schiefergebirge, and subsequently retreated northwestward during the Devonian (Langenstrassen 1983). These neritic clastics are overlain by Devonian pelagic sediments (dark shales). The transition from neritic to pelagic facies occurs in progressively younger strata towards the northwest indicating the retreat of the shelf margin. Beginning in the late Devonian, becoming increasingly important in Lower Carboniferous, and dominant in Upper Carboniferous, a Variscan flysch was deposited. This was derived from a southern source area („Mitteldeutsche Schwelle").

This palaeogeography provides information about the Caledonian provenance of the detritus (during the first two phases mentioned above) which is also supported by investigation of heavy mineral assemblages (Press 1982, 1986). K – Ar and $^{40}Ar/^{39}Ar$ cooling ages of muscovites from the Scandinavian Caledonides range between 410 and 384 Ma (Sturt et al. 1975; Dallmeyer et al. 1985). Ziegler (1978) reported whole-rock K – Ar ages of 445 – 407 Ma for slates and gneisses beneath the North Sea. These Caledonian cooling ages provide a likely age bracket for the detritus-rich size fractions encountered in the present study.

Volcanic activity during the Devonian in the sedimentary basin of the Rheinisches Schiefergebirge was characterized by spilites and keratophyres (Wedepohl et al. 1983). These are intercalated with the pelagic shales but are probably not contaminated with detrital white mica, and were therefore considered to be suitable for comparative investigation.

Structural style

The Rheinisches Schiefergebirge is characterized by large-scale, northeast trending folds and listric overthrusts. Weber (1981 a) distinguished two types of listric overthrusts: 1) those formed simultaneously with folding and which die out in higher structural levels where the displacement can be compensated by folding; and, 2) younger overthrusts which cut preexisting northwestward facing fold structures. Folding was associated with the development of a variably penetrative foliation which generally parallels axial surfaces. Weber (1981 b) described two types of foliations: 1) a disjunctive cleavage with syntectonic phyllosilicate recrystallization, and 2) a crenulation cleavage defined solely by the rotation of preexisting phyllosilicate grains. The first cleavage is generally southeast dipping but was locally rotated antithetically above younger listric overthrusts into steeper or even northwest dipping position (Weber 1981 a). Where the first cleavage was rotated to dips lower than ca. 20°, further compression led to the formation of a second, overprinting crenulation cleavage (Weber 1978).

Metamorphism

Illite "crystallinity" measurements (Weber 1972 a; Kasig and Spaeth 1975), coalification data (Wolf 1972, 1978; Paproth and Wolf 1973; Kalkreuth 1976; Teichmüller et al. 1979), facies critical minerals in spilitic rocks (Weber 1972 a), and fluid inclusion measurements on pre- to late-kinematic quartz mineralizations (Koschinski 1979) have shown that the metamorphic temperatures in the Devonian and Carboniferous rocks of the Rheinisches Schiefergebirge generally did not exceed ca. 350° C. In most cases they range between 200 and 300° C. The metamorphic grade corresponds to the very-low grade of Winkler (1976) or the anchizone of Kubler (1967). Higher temperatures (up to 400–500° C) were reached only in the southern part of the Rheinisches Schiefergebirge. According to Winkler (1976) those rocks belong to the albite-actinolite-clinozoisite(epidote)-zone of low grade metamorphism, i.e., to the lowest greenschist metamorphism (Meisl 1970; Meisl et al. 1982). Weber (1976) indicates that metamorphism is syntectonic with respect to folding and formation of first cleavage. This is suggested for fine-grained slates by a positive correlation between increasing anchizonal metamorphism (illite crystallinity) and an increasing portion of phyllosilicates oriented parallel to cleavage. Folding and syntectonic metamorphism appear to have migrated from southeast to northwest through the Rheinisches Schiefergebirge. This is documented by the migration of the flysch facies

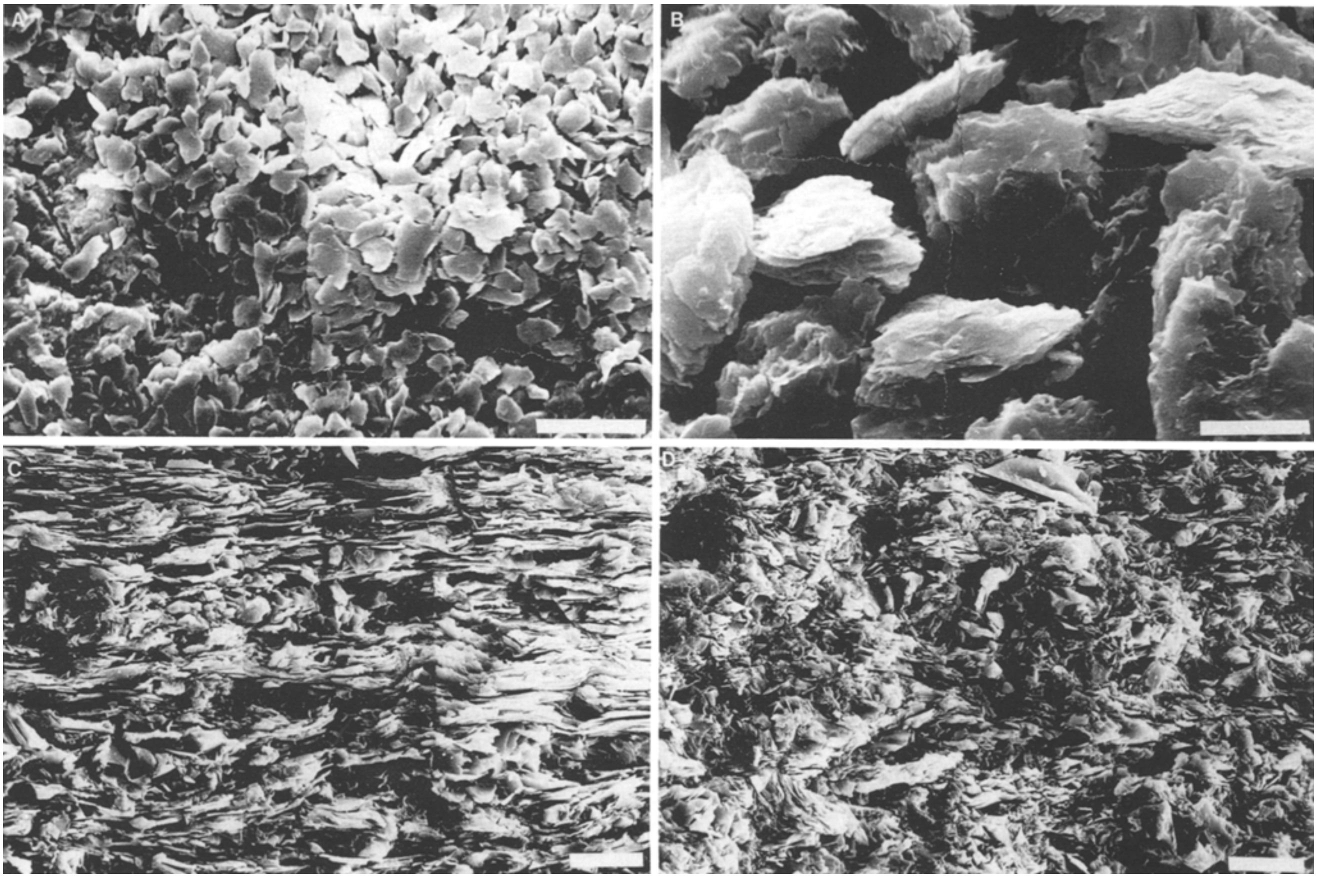

Fig. 2. A-D. A SEM microphotograph of a grain-size fraction consisting of individual mineral grains (metatuff sample of middle anchizone; size fraction 0.63–1 μm). Bar size is 5 μm. **B** SEM microphotograph of a grain-size fraction consisting of rock fragments (metatuff sample of middle anchizone; size fraction 6.3–20 μm). Bar size is 5 μm. **C** SEM microphotograph of upper anchizonal metatuff; macroscopic bedding and cleavage run horizontally. Bar size is 20 μm. **D** SEM microphotograph of middle anchizonal metapelite; the trace of cleavage runs vertically, macroscopic bedding from the *upper left to the lower right*, angle between cleavage and bedding ca. 20°. Bar size is 20 μm

into the same direction (Wunderlich 1964) and is supported by K−Ar and Rb−Sr isotopic ages on clay-size fractions from metatuffs (Ahrendt et al. 1978, 1983) which display a continuous cleavage fabric of synmetamorphic origin and therefore are likely to closely date the anchizonal metamorphism. These ages range from 327±10 Ma (K−Ar) to 312±7 Ma (Rb−Sr) in the south and from 302±10 Ma (K−Ar) to 290±8 Ma (Rb−Sr) in the north (East Sauerland Anticline). These ranges correspond to the upper Visean and uppermost Westphalian-Stephanian according to the time scale of Odin et al. (1982).

Samples investigated

In order to investigate the influence of different synkinematic metamorphic temperatures on the resetting of detrital mica in slates, samples from two different illite crystallinity ranges were selected for the grain-size separation (Reuter 1985). Intercalated metatuffs were collected to control the detrital influence in the metapelites. A sample suite was collected from middle anchizonal terranes of the Middle Devonian (Eifelian, Wissenbacher Schiefer) of the Dill Syncline (sample locality 1 in Fig. 1) and the Wittgenstein Syncline (sample locality 2 in Fig. 1), as well as of upper anchizonal slates in the middle Devonian (Eifelian, Fredeburger Schiefer) of the East Sauerland Anticline (sample locality 3 in Fig. 1).

The metapelites are dark grey slates and display a smooth disjunctive cleavage defined largely by the preferred orientation of very fine-grained phyllosilicate minerals (Fig. 2D). The spacing between the cleavage planes is ca. 25–30 μm. Detrital muscovite, which can be recognized in the handsample, and quartz grains are up to 50–100 μm in diameter. The (001)-planes of detrital micas form a large angle with the cleavage planes and often show rotation by slip on (001)-planes. These detrital micas are epitactically overgrown by metamorphic chlorite in upper anchizonal slates. X-ray textural analysis shows that the finer-grained chlorite and white mica in the matrix is aligned parallel to cleavage. The metatuffs are very fine-grained, light yellow to greenish sericite slates, displaying a continuous cleavage (Fig. 2C). They consist of metamorphic white mica and quartz and both minerals are aligned parallel to cleavage. The upper anchizonal samples are slightly coarser grained than those of the middle anchizone.

Sample preparation and analytical techniques

Details of the techniques used during sample preparation are listed in Reuter (1985). After removal of weathered surfaces (wire brush) the samples were crushed (chipmunk crusher) and ground in a shatter box (Scheibenschwingmühle) for 20 s. Three different disaggregation techniques (shatter box; H_2O_2; liquid nitrogen and subsequent ultrasonic treatment) were initially compared to evaluate the possible production of artificial grain sizes. Neither the K−Ar ages nor the illite crystallinity of these size fractions displayed significant differences (Reuter 1985, Table 9 and 12), and it was therefore concluded that none of the disaggregation techniques led to an artificial grain-size distribution.

A grain-size separation by settling methods (Atterberg settling cylinders and centrifuge) was used to isolate up to six fractions

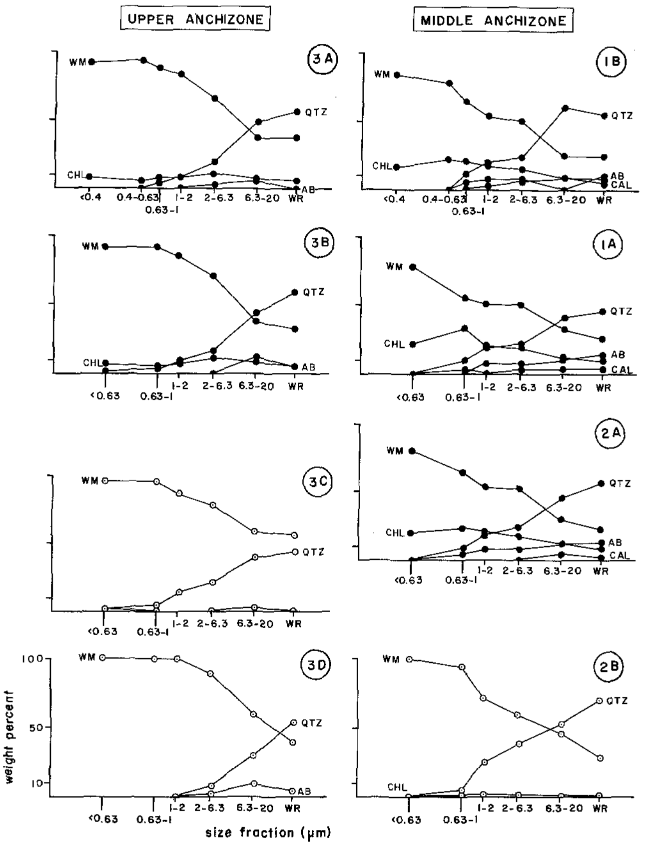

Fig. 3. Mineralogic variations among the various grain-size fractions (determined by infrared spectroscopy); *WM* white mica, *CHL* chlorite, *QTZ* quartz, *AB* albite, *CAL* calcite, *WR* whole rock, *dots* metapelites, *circles* metatuffs

per sample (<0.4 µm to 6.3–20 µm). All size fractions and the whole-rock were analyzed for their bulk mineralogical composition (X-ray diffraction) and K–Ar apparent age. Grain morphology and grain-size distribution in the various fractions were evaluated by SEM. Thin sections and polished rock slabs were investigated by optical and scanning electron microscopy.

Results

Sample mineralogy

The metapelites are composed largely of $2M_1$ phengitic white mica (according to the classification of the 'Clay Minerals Society Committee'; Bailey et al. 1979); 1Md polytypes were not detected by X-ray diffraction. Quartz, chlorite (ripidolite), albite, traces of K-feldspar (ca. 2%), calcite, and opaque components are also common constituents.

The metatuffs contain phengitic white mica and quartz. No K-feldspar or calcite was observed or detected by X-ray diffractometry. The samples from the East Sauerland Anticline (upper anchizone) contain less than 5% albite. The predominant white mica polytype depends on the degree of metamorphism, with middle anchizone samples consisting of 1Md polytype and samples from the upper anchizone made up largely of $2M_1$ polytype.

Mineralogy of the grain-size fractions

All size fractions and whole-rock samples selected for conventional K–Ar dating were analyzed by infrared spectros-

Table 1. Mineralogical composition of whole-rock and constituent grain-size fractions (analytical results of infrared spectroscopy)

	WM	CHL	QTZ	AB	CAL
Sample 1A (metapelite, middle anchizone)					
Whole-rock	26	10	46	14	4
<0.63 µm	78	22	–	–	–
0.61–1 µm	55	33	10	–	2
1–2 µm	51	21	20	8	–
2–6.3 µm	50	19	21	8	3
6.3–20 µm	32	12	41	11	4
Sample 1B (metapelite, middle anchizone)					
Whole-rock	24	7	55	10	4
<0.4 µm	84	16	–	–	–
0.4–0.63 µm	78	22	–	–	–
0.63–1 µm	64	20	11	5	–
1–2 µm	54	17	20	7	2
2–6.3 µm	50	14	23	7	6
6.3–20 µm	24	8	60	–	8
Sample 1C (metatuff, middle anchizone)					
Whole-rock	100	tr	tr	–	–
<0.63 µm	100	tr	–	–	–
0.63–1 µm	100	tr	–	–	–
1–2 µm	100	tr	tr	–	–
2–6.3 µm	100	tr	tr	–	–
6.3–20 µm	100	tr	tr	–	–
Sample 2A (metapelite, middle anchizone)					
Whole-rock	22	8	56	12	2
<0.63 µm	80	20	–	–	–
0.63–1 µm	64	23	9	4	–
1–2 µm	53	21	18	8	–
2–6.3 µm	51	17	24	8	–
6.3–20 µm	29	11	45	11	4
Sample 2B (metatuff, middle anchizone)					
Whole-rock	28	1	71	–	–
<0.63 µm	100	tr	–	–	–
0.63–1 µm	94	1	5	–	–
1–2 µm	72	2	26	–	–
2–6.3 µm	60	1	39	–	–
6.3–20 µm	46	1	53	–	–
Sample 3A (metapelite, upper anchizone)					
Whole-rock	37	6	57	–	tr
<0.4 µm	92	8	–	–	–
0.4–0.63 µm	94	6	–	–	–
0.63–1 µm	88	8	4	–	–
1–2 µm	83	8	9	–	–
2–6.3 µm	66	11	20	3	–
6.3–20 µm	37	8	49	6	–
Sample 3B (metapelite, upper anchizone)					
Whole-rock	32	5	58	5	–
<0.63 µm	91	7	2	–	–
0.63–1 µm	91	5	4	–	–
1–2 µm	84	7	9	–	–
2–6.3 µm	70	11	19	–	–
6.3–20 µm	37	8	43	12	–
Sample 3C (metatuff, upper anchizone)					
Whole-rock	56	tr	44	–	–
<0.63 µm	96	tr	2	2	–
0.63–1 µm	95	tr	5	–	–
1–2 µm	86	tr	14	–	–
2–6.3 µm	78	tr	22	–	–
6.3–20 µm	58	tr	40	2	–

Table 1 (continued)

	WM	CHL	QTZ	AB	CAL
Sample 3D (metatuff, upper anchizone)					
Whole-rock	40	tr	55	5	–
<0.63 µm	100	tr	–	–	–
0.63–1 µm	100	tr	–	–	–
1–2 µm	100	tr	–	–	–
2–6.3 µm	89	tr	8	3	–
6.3–20 µm	60	tr	30	10	–

WM White mica, *CHL* chlorite, *QTZ* quartz, *AB* albite, *CAL* calcite, *tr* traces

Table 2. Morphological grain types observed by SEM in the various size fractions

Fraction	Middle anchizone samples				
	Metapelites			Metatuffs	
	1A	1B	2A	1C	2B
0.63–1 µm	M	M	M	M	M(F)
1–2 µm	M(F)	M(F)	M(F)	M(F)	M(F)
2–6.3 µm	F	–	F(M)	F	F
6.3–20 µm	F	–	F	F	F

Fraction	Upper anchizone samples			
	Metapelites		Metatuffs	
	3A	3B	3C	3D
0.4–0.63 µm	M	–	–	–
0.63–1 µm	M	M	M	M
1–2 µm	M	M(F)	M	M
2–6.3 µm	M(F)	M(F)	M(F)	M(F)
6.3–20 µm	F(M)	F(M)	F(M)	M(F)

F rock fragments, *M* individual mineral grains, *F(M)* predominantly rock fragments, *M(F)* predominantly individual mineral grains

copy to quantitatively determine the distribution of the major mineralogical phases (Flehmig and Kurze 1973). White mica is enriched in the finer grain-size fractions from both metapelites and metatuffs (Fig. 3, Table 1). Quartz content varies inversely, with the finer fractions containing relatively less quartz. The average chlorite content is ca. 9 wt% for the 6.3–20 µm fraction and 14 wt% for the finest ones (<0.4 µm to <0.63 µm). Where present, albite and calcite are enriched in coarse fractions. The maximum albite contents are 10–20 wt% and occur in the fractions 6.3–20 µm and/or the whole-rock. The maximum calcite content detected is 8 wt% for fraction 6.3–20 µm. Fractions <1 µm generally do not contain albite or calcite.

Grain morphology

The different grain-size fractions display contrasting-grain morphologies. Grains with straight edges and smooth surfaces are interpreted as individual mineral grains (Fig. 2A), whereas grains with rough surfaces and edges are considered rock fragments (Fig. 2B). Rock fragments dominate the 2–6.3 µm and 6.3–20 µm fractions of the Wittgenstein and Dill Syncline (middle anchizone), but only the

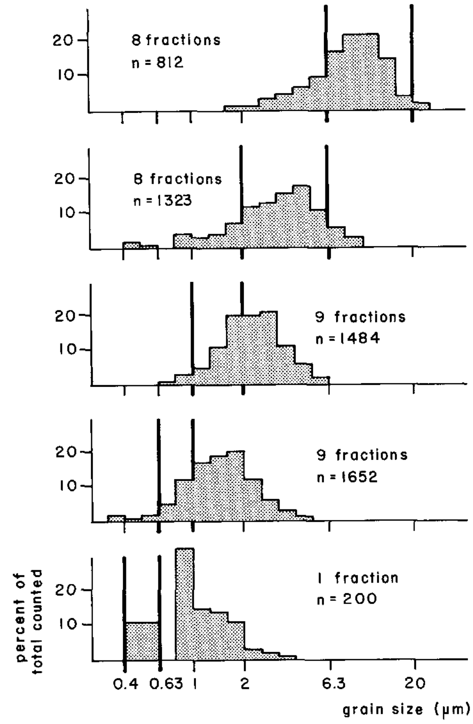

Fig. 4. Grain-size distribution within the various size fractions evaluated by measurements on SEM microphotographs. The theoretical grain diameter is indicated by two broad vertical lines in each diagram

6.3–20 µm fractions from the East Sauerland Anticline (upper anchizone) (Table 2). All fractions <6.3 µm from the East Sauerland Anticline are composed predominantly of individual mineral grains. These relationships suggest that the average grain-size of the samples from the East Sauerland Anticline (upper anchizone) is larger than of those from Wittgenstein and Dill Syncline (middle anchizone).

Grain-size distribution in the fractions

To evaluate the grain-size separations, the grain-size distribution of 35 different fractions was controlled by measuring the largest diameter of the constituent grains on SEM photomicrographs. A synoptic diagram (Fig. 4) shows that a grain-size separation was effectively achieved; however, the histograms only fit the equivalent (theoretical) diameter intervall (broad vertical lines in diagrams of Fig. 4) for the 6.3–20 µm and 2–6.3 µm fractions. Finer fractions do not correspond with their direct equivalent diameter interval, suggesting that the measured diameters are larger than the equivalent ones. This is considered to reflect increased settling times for phyllosilicates enriched in the finer fractions (cf., Fig. 3). As a result the platy grains occur in fractions finer than anticipated. For ease of discussion the size fractions are consistently referred to by their equivalent diameter interval (e.g., 1–2 µm).

Illite crystallinity

Variations in the degree of metamorphism of the samples were evaluated by measurements of illite crystallinity on oriented sedimentation slides prepared from <2 µm fractions, following procedures described by Weber (1972b). These involved comparison of the width-at-half-height of the first white mica basal reflection (10 Å on X-ray diffrac-

Table 3. Illite "crystallinity" (Hb_{rel} = quartz-normalized width at half height of 10 Å peak of white mica) of various size fractions determined according to Weber (1972b)

Fraction	Middle anchizone samples				
	Metapelites			Metatuffs	
	1A	1B	2A	1C	2B
<0.4 μm	–	334	–	–	–
<0.63 μm	262	–	242	403	464
0.4–0.63 μm	–	219	–	–	–
0.63–1 μm	210	185	223	357	399
1–2 μm	164	157	151	369	386
<2 μm	218	212	224	415	406
2–6.3 μm	167	151	169	384	377
6.3–20 μm	160	120	160	373	379

Fraction	Upper anchizone samples			
	Metapelites		Metatuffs	
	3A	3B	3C	3D
<0.4 μm	215	–	–	–
<0.63 μm	–	183	159	152
0.4–0.63 μm	172	–	–	–
0.63–1 μm	143	144	137	146
1–2 μm	131	135	130	123
<2 μm	144	144	137	138
2–6.3 μm	111	155	135	106
6.3–20 μm	114	141	103	–

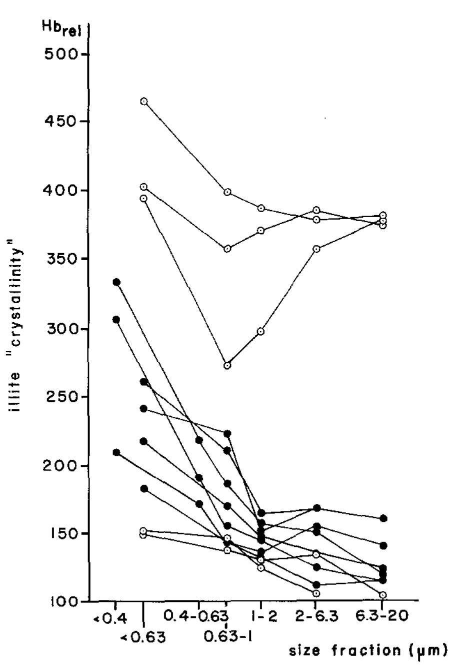

Fig. 5. Grain-size dependency of illite crystallinity (Hb_{rel}); *dots* metapelites, *circles* metatuffs

tograms) with the (100) reflection of an external quartz standard to yield a relative half width (Hb_{rel}). Minimum values for the bulk fraction <2 μm of this quartz-normalized illite crystallinity (Hb_{rel}) of ca. 120 are considered to characterize anchizone-epizone transition (Teichmüller et al. 1979). The transition between diagenesis and the anchizone is reflected by Hb_{rel} of 350–500. The relative half-width values of the present samples determined for <2 μm size fractions may be divided into middle anchizonal and upper anchizonal groups. The <2 μm fractions of middle anchizonal metapelites display Hb_{rel} of ca. 220, whereas those of the upper anchizone metapelites are ca. 150 (Table 3). This difference is even more pronounced in the metatuffs, where middle anchizonal samples yield values between 349 and 415, while those of the upper anchizone are of ca. 140.

To further evaluate the significance of illite crystallinity, Hb_{rel} was determined for all grain-size fractions, knowing that it depends upon the size of the very fine particles and the increasing difficulty to obtain best-oriented specimens with those particles (Fig. 5; Table 3). A striking grain-size dependency is displayed with the finest fractions displaying the broadest peaks; fractions >2 μm do not show this correlation.

Table 4. K–Ar analytical data of whole-rock anchizonal metapelites and constituent grain-size fractions

Sample	Fraction (μm)	K_2O (%)	$^{40}Ar^*$ (10^{-6} cm^3/g) STP	$^{40}Ar^*/^{40}Ar$ (%)	Apparent age (Ma ± s)
Middle anchizone metapelites					
1A	<0.63	5.87	74.65	95.01	357 ± 10
	0.63–1	5.28	68.98	97.19	366 ± 11
	1–2	4.36	62.03	98.30	395 ± 11
	<2	5.30	68.99	96.69	364 ± 10
	2–6.3	3.76	53.83	96.11	397 ± 11
	6.3–20	3.36	48.57	97.39	401 ± 11
	WR	3.69	50.62	94.82	382 ± 11
	WR	3.69	50.47	95.15	381 ± 11
1B	<0.4	6.33	77.57	96.03	345 ± 10
	0.4–0.63	5.59	74.65	96.02	373 ± 11
	0.63–1	5.01	70.85	95.73	393 ± 11
	1–2	4.22	61.44	95.14	403 ± 12
	2–6.3	3.58	52.11	93.65	403 ± 12
	6.3–20	2.95	41.14	95.19	388 ± 11
2A	<0.63	6.59	80.62	97.75	345 ± 10
	0.63–1	5.55	74.65	97.62	375 ± 11
	1–2	4.84	68.24	98.65	392 ± 11
	<2	5.83	73.57	95.29	354 ± 10
	2–6.3	3.97	57.42	98.52	401 ± 11
	6.3–20	3.65	51.42	98.52	391 ± 11
	WR	4.05	55.33	97.50	381 ± 11
Upper anchizone metapelites					
3A	<0.4	6.77	74.81	94.70	314 ± 9
	<0.4	6.77	75.21	94.30	315 ± 9
	0.4–0.63	7.22	87.19	97.12	340 ± 10
	0.63–1	7.07	85.70	97.47	342 ± 10
	1–2	6.32	78.10	97.75	348 ± 10
	2–6.3	4.67	61.08	97.30	366 ± 10
	6.3–20	3.62	50.80	96.69	390 ± 11
3B	<0.63	6.94	80.28	96.69	327 ± 9
	0.63–1	7.09	84.53	98.15	337 ± 10
	1–2	6.75	84.54	99.41	352 ± 10
	<2	7.05	81.95	98.08	335 ± 10
	2–6.3	4.85	61.76	97.71	357 ± 10
	6.3–20	3.53	47.80	98.95	378 ± 11
	WR	4.17	52.43	96.62	353 ± 10
	WR	4.17	52.96	95.85	356 ± 10

Table 5. K – Ar analytical data of whole-rock anchizonal metatuffs and constituent grain-size fractions

Sample	Fraction (μm)	K_2O (%)	$^{40}Ar^*$ (10^{-6} cm^3/g) STP	$^{40}Ar^*/^{40}Ar$ (%)	Apparent age (Ma ± s)
Middle anchizone metatuffs					
1C	<0.63	8.30	93.75	98.50	320 ± 9
	0.63–1	8.33	97.54	98.00	331 ± 9
	1–2	7.87	91.93	97.17	330 ± 9
	<2	8.28	93.65	99.30	321 ± 9
	2–6.3	7.59	90.43	97.65	336 ± 10
	6.3–20	7.49	89.46	97.48	337 ± 10
	WR	8.05	91.84	98.73	323 ± 9
2B	<0.63	7.97	90.85	98.24	322 ± 9
	0.63–1	7.24	86.24	98.40	336 ± 10
	1–2	5.72	68.12	98.18	336 ± 10
	<2	7.48	87.06	98.70	329 ± 9
	2–6.3	4.53	51.88	97.39	324 ± 9
	6.3–20	5.13	60.88	97.79	335 ± 10
	WR	5.77	67.73	98.65	332 ± 9
Upper anchizone metatuffs					
3C	<0.63	8.33	89.00	96.58	304 ± 9
	0.63–1	8.48	95.40	97.58	319 ± 9
	1–2	7.72	86.77	97.66	319 ± 9
	<2	7.98	89.04	97.46	317 ± 9
	2–6.3	6.24	72.43	98.00	328 ± 9
	6.3–20	6.05	70.40	97.79	329 ± 9
	WR	6.51	75.13	97.74	327 ± 9
3D	<0.63	7.31	80.45	94.53	313 ± 9
	0.63–1	8.16	93.10	96.57	323 ± 9
	1–2	8.06	93.84	96.63	329 ± 9
	2–6.3	7.33	86.74	96.27	334 ± 10
	6.3–20	6.32	75.17	95.40	336 ± 10

K – Ar apparent ages

K – Ar apparent ages have been determined on 30 size fractions separated from five metapelite samples, and 3 whole-rock samples (Table 4). 23 fractions (4 samples) and 3 whole-rock samples were analyzed from metatuff samples (Table 5). The analyses were carried out following methods explained in detail by Bonhomme et al. (1975). K concentrations were measured by flame spectrometry with a ±2% (1 σ) precision calculated from 27 duplicate, one triplicate and one quintuplicate analyses. Ar analyses were made after gas extraction and purification using a spike from Clusius (Zürich; 99.8635% ^{38}Ar) calibrated against a value of 24.80×10^{-6} cm^3 $^{40}Ar^*/g$ STP for the standard GLO (Flisch 1982). The Ar analyses were carried out during 3 periods. For each of these periods duplicate or triplicate measurements of the standard GLO average at 25.03 ± 0.17, 24.83 ± 0.21 and $24.90 \pm 0.34 \times 10^{-6}$ cm^3 $^{40}Ar^*/g$ STP respectively. The ages were calculated using the constants listed by Steiger and Jäger (1977), and have an error of ±2.7 to ±3 percent (1 σ) depending upon the $^{40}Ar^*/^{40}Ar$ ratio.

K – Ar apparent ages of size fractions from metapelites (Fig. 6) display the following variation: 1) There is a grain-size dependency, with finer size fractions generally recording younger apparent ages except for >2 μm size fractions within middle anchizonal samples. Their age values level or decrease for coarser sizes; 2) the data form two groups consistent with those defined by the illite crystallinity.

The results of the metatuffs do not show a significant grain size/age dependency for fractions >0.63 μm. Those <0.63 μm do display slightly younger apparent ages. In middle and upper anchizone metatuffs the apparent ages recorded by the >0.63 μm fractions are similar to those obtained by the finest fractions (<0.4 μm, <0.63 μm) of the associated metapelites (Fig. 6).

Discussion

Variation in grain size and mineralogical characteristics

The mineralogical composition of the metapelites does not significantly change between the middle and upper anchizone. However, there is a more pronounced enrichment of white mica in the finest fractions of samples from upper anchizone. Significant variations in white mica polytypes occur within the metatuff samples. Those from the middle anchizone are composed predominantly of the 1Md and those of the upper anchizone display mostly a $2M_1$ polytype. This is not the case for the metapelites which consist only of $2M_1$ white mica. This difference suggests a primary lithological control on polytype formation because similar metamorphic conditions were likely experienced by both rock types. Hunziker et al. (1986) documented that the ratio $2M_1/(2M_1 + 1Md)$ increases with increasing degree of incipient metamorphism, but noted that it cannot be used as

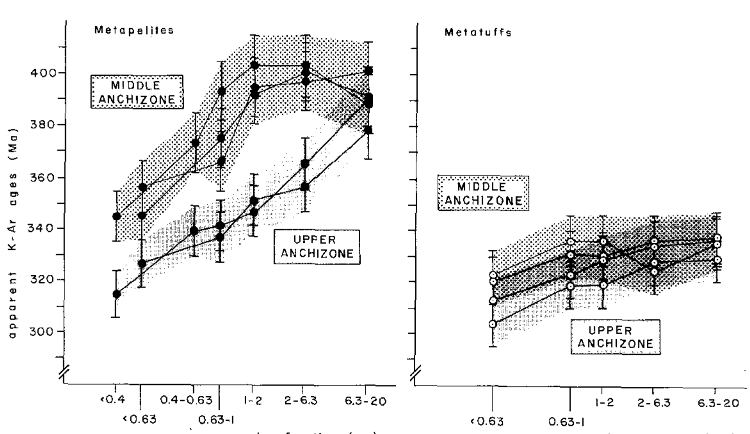

Fig. 6. Relationship of apparent K – Ar ages and grain size

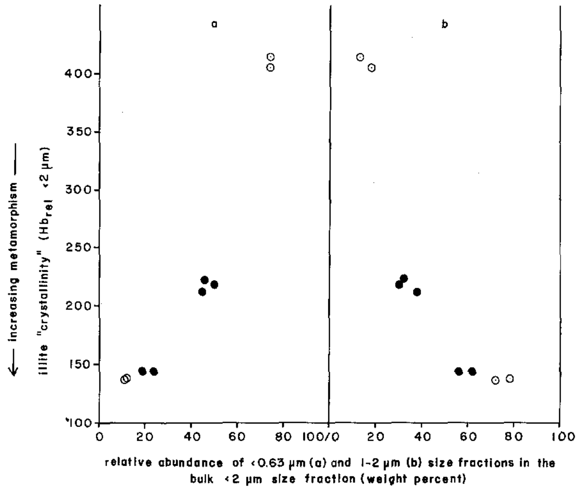

Fig. 7. Relationship of illite crystallinity (metamorphic grade) and grain-size distribution within the bulk <2 µm fraction; *dots* metapelites, *circles* metatuffs

Table 6. Relative abundance (weight percent) of size fractions comprising the bulk <2 µm fraction

Fraction	Metapelites			Metatuffs	
	1A	1B	2A	1C	2B
Middle anchizone samples					
<0.4 µm	–	24	–	–	–
0.4–0.63 µm	–	21	–	–	–
<0.63 µm	50	–	46	74	74
0.63–1 µm	20	17	22	13	8
1–2 µm	30	38	32	13	18
Fraction	Metapelites			Metatuffs	
	3A	3B		3C	3D
Upper anchizone samples					
<0.4 µm	10	–		–	–
0.4–0.63 µm	9	–		–	–
<0.63 µm	–	24		11	12
0.63–1 µm	19	20		17	10
1–2 µm	62	56		72	78

a measure of metamorphic grade because varying lithology and/or chemical composition of the white mica influence the extent of this transition. Since 1Md polytype white mica (illite) is a common constituent of argillaceous sediments (e.g., Dunoyer de Segonzac 1970; Foscolos and Stott 1975; Jonas 1975), it is likely that this polytype was present prior to metamorphism. If so, the transition from 1Md to $2M_1$ in the metapelites has to have taken place at lower metamorphic conditions than in the metatuffs. On the other hand, delayed transition in the metatuffs could be due to the lack of any detrital mica precursor in these rocks.

An increase of grain size from middle to upper anchizone is observable for both the metatuffs and the metapelites. It is observable by scanning electron microscopy on polished rock slabs (Reuter 1985) and can be concluded from variations in grain morphology, and the grain-size distribution within <2 µm size fractions. The 1–2 µm size fractions of the middle anchizonal samples consist predominantly of individual mineral grains, whereas >2 µm size fractions display a significant number of rock fragments (Table 2). Thus, individual mineral grains are larger in upper anchizone than in middle anchizone samples. The grain-size distribution within the fraction <2 µm changes significantly with varying illite crystallinity (Fig. 7, Table 6), an increase of metamorphic degree produces a decrease in the relative amount of <0.63 µm material and a relative increase in the amount of 1–2 µm material. A similar relationship has been documented by Huon (1985).

Implications for illite crystallinity

The degree of very-low grade metamorphism is commonly determined by means of illite crystallinity on <2 µm fractions separated from argillaceous sedimentary rocks (e.g., Weaver 1960; Kubler 1967; Dunoyer de Segonzac 1969; Weber 1972a, b). The illite "crystallinity" is among other factors dependent on grain size (Fig. 5) and therefore grain-size distribution within these <2 µm fractions. Because the relative abundance of the grain sizes changes to coarser grains with increasing metamorphic grade, it contributes significantly to the narrowing of the 10 Å peak in <2 µm fractions.

Apparent K – Ar ages

Although not experimentally calibrated, empirical comparison of variations in K – Ar ages recorded by various minerals suggest that the temperature required for the retention of intracrystalline argon in muscovite and phengite is ca. $350 \pm 50°$ C (Jäger 1979). Similar temperatures are not likely to control diffusive argon mobility during thermal overprint of K – Ar isotopic systems of detrital white mica in very-low grade metasedimentary rocks, and Hunziker et al. (1986) have suggested that temperatures of approximately $260 \pm 30°$ C are required to totally reset argon systems in less than 2 µm detrital 1Md illites. The extent of rejuvenation in very fine grain-size fractions may be evaluated by the comparison of metapelite and metatuff grain size/age relationships observed in the present study. Such partial rejuvenation of the K – Ar isotopic system in fine-grained white mica populations at very-low grade metamorphic temperatures (anchizonal to epizonal) is due to: 1) metamorphic temperatures (diffusion and/or migration of dislocations), 2) deformation during metamorphism (neoformation and/or recrystallization) and, 3) changing mineralogic characteristics (1Md to $2M_1$ polytype transition).

1. Metapelites. The marked grain-size/age dependency displayed by most metapelite samples (Fig. 6) may be produced by one or a combination of the processes mentioned above. Cleavage formation could have involved only rotation of detrital grains (pressure-solution assisted grain-boundary sliding). If so, the observed grain-size dependency of apparent K – Ar age would likely reflect variable diffusive loss of radiogenic argon due to variable temperatures during anchizonal metamorphism, and the two groups defined by varying grain size/age relationships (Fig. 6) would likely reflect an enhanced diffusive loss from comparable grain-size fractions in the upper anchizone samples. On the other hand, the observed grain-size/age relationship could reflect variably completed synkinematic recrystallization of white mica during cleavage formation (depending on varying metamorphic temperature) as suggested by Weber (1976, 1981 b) for the Middle Devonian slates of the eastern Rheinisches Schiefergebirge. This would require an enrichment of synkinematically recrystallized relative to detrital white mica in fine vs. coarse fractions to produce the observed grain-size dependency of apparent K – Ar ages. The restructuration of 1Md to $2M_1$ white mica (Hunziker et al. 1986) cannot be responsible for the relationship found in the metapelites because the samples no longer contain detectable 1Md white mica.

The changing trend of the grain size/age dependency at ca. 2 μm (similar or slight decrease of apparent K – Ar ages towards coarser fractions) in samples from the middle anchizone (Fig. 6) is likely due to rock fragments consisting of coarse detrital and variably reset finer grains. These mixed ages are consistently higher than the comparable whole-rock ages (Table 4), indicating a relatively smaller portion of fine grains in these fragments than in the bulk samples. Similar relationships have been reported by other workers who interpreted them to reflect aggregates of smaller grains (Sedivy et al. 1984), or a rejuvenation of coarse detrital white mica by rotation and bending during cleavage formation (Huon 1985).

2. *Metatuffs.* Regardless of metamorphic grade, all metatuff size fractions < 0.63 μm consistently record younger K – Ar apparent ages than coarser size fractions within the same sample (Fig. 6). The lack of variation in K – Ar apparent ages between size fractions coarser than 0.63 μm confirms that there are no significant detrital components. The < 0.63 μm size fractions from samples in the middle anchizone consist largely of a 1Md polytype and record apparent ages of ca. 321 Ma. Similar size fractions in upper anchizone samples are represented by a $2M_1$ polytype and yield markedly younger ages of ca. 309 Ma. Hunziker et al. (1986) have documented that conversion of 1Md to $2M_1$ white mica in the Glarus alps involved restructuration processes which effected partial loss of radiogenic argon. Therefore, the relatively younger K – Ar apparent ages recorded by $2M_1$ white mica in the < 0.63 μm size fractions in the present metatuff samples could reflect such restructuration processes. However, there is no age difference between the coarser size fractions within these two sample groups although they are also composed of contrasting polytypes. The significance of the younger ages recorded by < 0.63 μm fractions remains unclear. It might possibly reflect a slight diffusive loss of radiogenic argon during decline of metamorphic temperatures. It is surprising that this loss appears to be more pronounced for the $2M_1$ than for the 1Md polytype. Because of this uncertainty, no geologic significance is affixed to the K – Ar ages recorded by the very fine metatuff size fractions.

3. *Comparison of the results of the metapelites and metatuffs.* The metapelite fractions generally display higher K – Ar apparent ages than equivalent size fractions in corresponding metatuffs. However, in both the middle and upper anchizone sample group apparent ages obtained from fine metapelite fractions (< 0.4 μm and < 0.63 μm) are similar to those recorded by the > 0.63 μm metatuff fractions (Fig. 6; Tables 4 and 5). This implies that, regardless of degree of anchizonal metamorphism, detrital memory in the finest metapelite fractions is apparently completely removed and that they therefore can be interpreted to closely date the metamorphic climax.

Geological significance

Results of this study suggest that apparent K – Ar ages of all but the very fine size fractions within metapelite samples from the anchizone reflect varying detrital memory and have no geological significance. The highest apparent ages obtained from coarse metapelite fractions (401 ± 11 Ma) are minimum detrital ages matching the before mentioned cooling ages reported from the Scandinavian Caledonides and the North Sea subsurface. Apparent ages yielded by very fine fractions may in this particular case (age control by metatuffs available), be interpreted to date cleavage formation and anchizonal metamorphism.

The ca. 330 Ma apparent ages recorded by the > 0.63 μm metatuff fractions are interpreted to closely date the metamorphic climax and concomitant cleavage formation. This is older than the 315 – 305 Ma age range proposed for metamorphism of these rocks based on Rb – Sr and K – Ar analyses of < 2 μm metatuff fractions (Ahrendt et al. 1978, 1983). The metatuffs appeared to these authors as a suitable material for isotopic dating because of the anticipated absence of any detrital component. Although the present study has confirmed a lack of detrital components, the fractions < 0.63 μm appear to have been affected by processes not yet understood, which result in a decrease of K – Ar ages.

The obvious problem with this interpretation (the 330 Ma apparent ages dating the cleavage formation) is the fact that the sedimentation in the northeastern Rheinisches Schiefergebirge persisted into the Westphalian. This lasted according to the time scale of Odin et al. (1982) from 310 to 300 Ma with an uncertainty of ca. ± 10 Ma, and according to the DNAG time scale (Palmer 1983) from 315 to 296 Ma with an uncertainty of ca. ± 20 Ma. It is beyond the scope of this paper to discuss the basis of the different time scales. However, comparison of the data presented here with these time scales suggests that Westphalian sediments must have been deformed immediately after their deposition.

Conclusions

Grain-size fractions and whole-rock samples of anchizonal metapelites and associated metatuffs from the northeastern Rheinisches Schiefergebirge were investigated for microstructural and mineralogical characteristics and were analyzed by means of conventional K – Ar techniques. Apparent K – Ar ages of the metapelites show a positive correlation with grain size demonstrating the decreasing detrital influence and increasing importance of partially or completely reset grains in finer fractions. Metatuff fractions > 0.63 μm record consistent apparent ages confirming the anticipated lack of detrital white mica. The K – Ar ages are therefore interpreted to date the age of synkinematic metamorphism and cleavage formation at ca. 330 Ma. Slight diffusive loss of radiogenic argon might be responsible for decreasing apparent ages of < 0.63 μm metatuff fractions. Comparison of the results obtained for metapelites and metatuffs suggests that only the finest metapelite fractions (< 0.4 μm; < 0.63 μm) are free of detrital influence and yield K – Ar ages of geologic significance. In geologic settings where similar controls (comparison of apparent ages obtained from metatuffs and metapelites) are not available the apparent K – Ar ages recorded by very fine metapelite fractions should only be interpreted to reflect maximum ages of anchizonal metamorphism and cleavage formation.

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