

STRUCTURAL VARIATIONS IN CHLORITE AND ILLITE IN A DIAGENETIC SEQUENCE FROM THE IMPERIAL VALLEY, CALIFORNIA

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Abstract—Samples of cuttings from the Borchard A-2 well, Imperial Valley, California, were collected over a measured borehole temperature interval 135° to 275°C. The <0.5- μm (e.s.d.) fraction was separated using high-gradient magnetic separation (HGMS) to create a nonmagnetic fraction rich in illite and a magnetic fraction rich in chlorite. Chlorite was less easily separated from illite in lower temperature samples (<200°C), presumably due to the presence of polymineralic grains of chlorite and illite. Grains in higher temperature samples were more nearly monomineralic and more easily separated.

The chlorite is the *IIb* polytype. The thickness of coherent scattering domains of chlorite increased until 220°C and then remained constant. The amount of 7-Å interstratified material increased downhole until 195°C and then decreased. Over the same temperature interval, the illite polytypes varied systematically from $1M_d$ (135° to 175°C) to $1M + 2M_1$ (230° to 275°C) and coherent scattering domains in the mineral became thicker to about 200°C and then remained constant in thickness. The percentage of illite in mixed-layer illite/smectite (*I/S*) increased from 40% at 135°C to 100% at temperature >205°C; ordering in the *I/S* changes from *R0* to *R1* between 135° and 155°C, and from *R1* to *R* ≥ 3 at temperatures >155°C.

The concurrent structural changes in chlorite and illite indicate a general improvement in the overall structural order of the clay minerals with increasing temperature. Differences between chlorite and illite suggest that the minerals may have reacted differently to changing conditions or that they may have formed by different mechanisms. The exclusive occurrence of *IIb* chlorite at temperatures as low as 135°C extends the limit of *IIb* chlorite stability to temperatures lower than previous estimates.

Key Words—Chlorite, Diagenesis, High-gradient magnetic separation, Illite, Interstratification, Polytype, X-ray powder diffraction.

INTRODUCTION

Various structural changes accompany clay mineral transformations with increasing temperature: polytypes change through a succession of metastable forms eventually to attain “stable” structures; crystals having defects and stacking faults recrystallize to create larger coherent diffracting domains; and the proportion of interstratification in mixed-layer clays decreases until only the stable layer type exists (see, e.g., Lee *et al.*, 1985). In diagenetic and metamorphic sequences, chlorite and illite commonly coexist, forming from the breakdown of such minerals as smectite and kaolinite. With increasing temperature, however, trioctahedral chlorite may react differently than dioctahedral illite.

Studies of chlorite polytypes (e.g., Bailey and Brown, 1963; Hayes, 1970) have been few in number, but have suggested that the *IIb* polytype is the stable structure >200°C. The change from the type-I to the type-II structure involves 60° rotation of the interlayer and is postulated to occur between 150° and 200°C (Hayes, 1970). The transition has rarely been documented in the field (Karpova, 1969; Walker, 1989), nor has it been investigated in the laboratory. Mica polytypes

(Yoder and Eugster, 1955), in contrast, have been studied extensively. Field occurrences have been reported in which $1M$ polytypes transform to, and coexist with, $2M_1$ illite (Reynolds, 1963; Maxwell and Hower, 1967). The polytype conversion has also been accomplished in the laboratory (Velde, 1965). These studies indicate that the change from $1M_d$ to $2M_1$ mica is progressive with increasing temperature. Samples containing both polytypes are common, although selected-area electron diffraction (SAD) photographs reported by Baxter and Peacor (1988) did not indicate the presence of the $1M$ structure; instead only $1M_d$ and $2M_1$ polytypes were identified. Illite $2M_1$ appears at about 200°C in field examples (Eslinger and Savin, 1973) and as low as 125°C in laboratory experiments (Velde, 1965).

The mean size of coherent diffracting domains within a crystal is an important factor controlling the breadth of diffraction lines; larger domains produce sharper peaks. Observation of progressive sharpening of illite peaks with increasing temperature suggested the use of the breadth of the 001 peak of illite as an indicator of increasing diagenesis and low-grade metamorphism (Kubler, 1964). A similar analysis has been applied to chlorite to establish the relationship between changes in relative sizes of diffracting domains and increasing temperature during formation of slaty cleavage (Ishii, 1988). As long as interstratified material is present in

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a mineral (see below), the interstratified layers represent breaks in crystallographic continuity and β will be a function of both the percentage of interstratification and of the size of the scattering domains. In fact, β and percentage of interstratified material can be measures of the same phenomenon if the growth of coherent scattering domains is a direct result of the decrease in the percentage of interstratification.

Interstratification is common in clay minerals. The interlayering of 7-Å layers in 14-Å chlorite has been documented by transmission electron microscopy (TEM) (Ahn and Peacor, 1985; Yau *et al.*, 1988), but it has rarely been observed by X-ray powder diffraction (XRD) because the basal peaks of the 7-Å mineral superimpose on the even-order peaks of chlorite and are, therefore, difficult to detect. Calculated XRD profiles from such interstratified structures show systematic variations in the breadths of even- and odd-order peaks (Reynolds, 1988). The amount of interstratification visible in TEM images has not been quantified, but it has been observed to decrease at temperatures >220°C (Yau *et al.*, 1988). Variations in interstratification of illite and smectite have been studied extensively in burial diagenetic sequences (e.g., Hower *et al.*, 1976; Nadeau and Reynolds, 1981; Jennings and Thompson, 1986; Velde *et al.*, 1986). Important changes with increasing temperature include increase in the percentage of non-expandable (illite) layers in illite/smectite (I/S) and ordering of illite and smectite layers (Reynolds and Hower, 1970). In burial diagenetic sequences long-range ordering (ISII, $R \geq 3$) has been reported to commence between 150° and 175°C, and the percentage of illite in I/S increases to 100% at about 200°C (Jennings and Thompson, 1986; Velde *et al.*, 1986). The situation is complicated by the presence of discrete authigenic illite crystallites along with illite in layers in the I/S (Yau *et al.*, 1987; Thompson and McCarty, 1988).

The purpose of the present study was to document concurrent variations in polytypes, size of coherent scattering domains, and interstratification in chlorite and illite from a continuous sequence of samples. Samples were analyzed from the Borchard A-2 geothermal exploration well (Jennings and Thompson, 1986) drilled by the Geothermal Operations Division of Phillips Petroleum Company in Plio-Pleistocene sediments of the Colorado River delta near Brawley, California in the Imperial Valley. The actual depths of the samples collected are considered to be proprietary information by Phillips Petroleum Company; however, in the Imperial Valley, subsurface mineral assemblages occur at similar temperatures regardless of burial depth implying that temperature rather than pressure is the major factor controlling mineral reactions (Muffler and White, 1969; McDowell and Elders, 1980; Jennings and Thompson, 1986). Recent work in the Salton Sea Geothermal Field to the north of the Borchard A-2 well

indicates that measured temperatures may be 30° to 50°C below the maximum temperatures experienced by the rocks (Roedder and Howard, 1988; Sass *et al.*, 1988). For this reason the temperatures reported herein are used only to identify the samples and are not presumed to represent formation temperatures. The reported temperatures are, however, considered to measure relative changes in formation temperature, especially an increase in formation temperature with increasing depth.

ANALYTICAL METHODS

Well cutting samples from the Borchard A-2 well were selected at about 10°C intervals from the measured temperature range 135° to 275°C. Reported temperatures in the well were measured by Phillips Geothermal Operations Division four months after drilling (to allow for re-equilibration) and are assumed to approximate pre-drilling conditions. The samples were shales and fine-grained siltstones.

Ten samples were selected for detailed XRD analysis. The samples were washed in deionized water to remove drilling mud contaminants and then crushed lightly in an iron mortar. The <0.5- μm equivalent spherical diameter (e.s.d.) size fraction was separated by timed centrifugation. The clay fractions were then separated by high-gradient magnetic separation (HGMS) (Tellier *et al.*, 1988) into a nonmagnetic fraction rich in illite and a magnetic fraction rich in chlorite. The same separation parameters (i.e., flow rate and magnetic field strength) were used to separate the magnetic fraction of all samples. A different, but also constant, set of parameters was used to separate the nonmagnetic fractions.

Oriented samples of the bulk <0.5- μm , magnetic and nonmagnetic fractions were prepared on glass slides by the filter-peel technique (Drever, 1973) and analyzed air-dry and after solvation in ethylene glycol vapor at about 40°C for at least 12 hr. XRD patterns were scanned from 3° to 33°2 θ with a Philips Norelco flat-specimen diffractometer using CuK α radiation (30 kV, 30 mA) and a graphite crystal monochromator. Digital data were collected using a Databox stepper motor controller (Radix Instruments, Inc.) and plotted with NEWMOD plotting software (Reynolds, 1985). Count times ranged from 10 to 40 sec per 0.1°2 θ step. Peak positions were located using NEWMOD and multiple analysis of the same peak indicated that the precision in d-values was on the order of 0.05 Å at 6°2 θ .

Randomly oriented samples produced by creating a spindle of powder with Duco cement were analyzed with a Gandolfi camera (114.6-mm diameter) using FeK α radiation. Total exposure times ranged from 36 to 48 hr. Illite samples used for polytype analysis were heated for 1 hr at 300°C prior to analysis to collapse expandable layers and improve resolution of the powder photographs.

Chlorite polytypes were identified from powder photographs by comparison with tabulated values of distinguishing diffraction lines (Bailey, 1984; Table 1.22). Illite polytypes were identified from tabulated values in Brindley (1984; Table 2.10) and in Bailey (1984; Table 1.15). The difficulties of illite polytype identification, especially in mixtures, are discussed in the next section.

Relative sizes of coherent scattering domains in crystals were estimated using the width at half height (β) of selected diffraction peaks from oriented preparations. Multiple analyses indicate that the precision of such measurements is about 0.01 Å. Factors, such as particle size distribution and preferred orientation of the grains in the sample, in addition to the size of coherent diffracting domains, contribute strongly to the character of low-angle reflections ($<15^\circ 2\theta$, $\text{CuK}\alpha$). The 003 reflection of chlorite was selected because the breadths of the odd-order chlorite reflections are very sensitive to interruptions in crystallographic continuity (see below) and, therefore, the size of the scattering domains, and the 003 peak is a strong odd-order reflection located at a relatively high angle. Likewise, the illite 005 peak was selected because it is located in an angular position where the effects of preferred orientation and particle size distribution are not as great. The width of the illite 005 peak was measured from glycolated preparations.

Relative percentages of 7-Å material interstratified in chlorite were estimated by comparison with patterns calculated by NEWMOD (Reynolds, 1985). Systematic differences in the width at half-height of odd- and even-order peaks in the basal series of chlorite result from the interstratification of 7- and 14-Å layers (Reynolds, 1988). As the proportion of interstratified 7-Å layers in chlorite increases, odd-order peaks become relatively weaker and broader compared to even-order peaks. A factor, Δ (measured in $^\circ 2\theta$), was defined as:

$$\Delta = \beta_{003} - (\beta_{002} + \beta_{004})/2,$$

and is a measure of the relative breadths of even- and odd-order peaks in the basal series of chlorite. As the amount of interstratification approaches zero, Δ also approaches zero. Δ can be converted to relative percentage of 7-Å material using a calibration curve derived from calculated diffraction patterns. This method provides only a *relative* estimate of the percentage of 7-Å interstratification. For the method to be rigorously quantitative, the effects of machine broadening and other experimental factors must first be separated from the peak in question by deconvolution (Reynolds, 1988). As this was not done in this study, the technique was used only to establish trends among the samples under investigation.

Proportions of illite layers in interstratified illite/smectite (I/S) and the nature of ordering of illite and smectite layers in I/S were determined by comparison

with patterns published by Reynolds and Hower (1970) and calculated with NEWMOD (Reynolds, 1985).

RESULTS

An example of the XRD patterns of typical magnetic and nonmagnetic fractions produced by HGMS separation is shown in Figure 1. In general, magnetic fractions consisted almost entirely of chlorite with different amounts of illite, I/S, and minor amounts of quartz. I/S, illite, and minor amounts of calcite and chlorite (or possibly kaolinite) comprised the nonmagnetic fractions. Chlorite was more easily separated from illite in the higher temperature samples ($>200^\circ\text{C}$), a phenomenon that has been observed elsewhere (Walker, 1987).

The results of XRD analysis of the Borchard samples are summarized in Figure 2 for chlorite and Figure 3 for illite. In general, the trends of the chlorite parameters are not smooth, although the values approach constants at elevated temperatures. In contrast, the trends of the illite parameters smoothly approach constant values with increasing temperature.

All of the chlorite analyzed was found to be the type-IIb polytype (Figure 2). The illite polytype results were more difficult to interpret. Figure 4 is a schematic diagram of the peaks between 2.0 and 4.3 Å in representative nonmagnetic fractions from the Borchard A-2 well. In sample 3 (175°C), the absence of the 112 peak at about 3.65 Å suggests the presence of $1M_d$ illite. In the 195° sample, the 3.65-Å peak indicates the presence of $1M$ illite; broadening of reflections between 2.0 and 2.5 Å indicates the possible presence of the $1M_d$ polytype (Brindley, 1984; Table 2.10). In sample 6 (215°C), the broad reflection at 2.42 Å is probably due to $1M$ reflections at 2.38 and 2.46 Å. The sample therefore probably contains only $1M$ illite. In sample 9 (250°C), reflections at 4.24, 4.04, and 2.19 Å indicate the presence of $2M_1$ illite, whereas the 3.67-Å peak indicates the $1M$ polytype, and the sample is probably a mixture of $1M$ and $2M_1$ structures. In general, illite polytypes range systematically from $1M_d$ (135° to 175°C) to $1M$ ($\pm 1M_d$) (175° to 200°C) to $1M$ (200° to 230°C) to $1M + 2M_1$ (230° to 275°C), demonstrating progressive ordering of the mineral with increasing temperature (Figure 3).

The appearance of the $2M_1$ mica polytype between 230° and 250°C is 50° to 70°C lower than the 300°C minimum temperature for the formation of the $2M_1$ structure determined by experimental studies (Yoder and Eugster, 1955; Velde, 1965). This discrepancy may be due to the possibility that this part of the Imperial Valley may have cooled by 50° to 70°C since attaining its maximum temperature. Alternatively, it may be due to the fact that the experimental work on the lower limit of mica stability was done under short-term laboratory conditions and was controlled more by reaction kinetics than equilibria, whereas the Imperial Valley samples have had much longer to react.

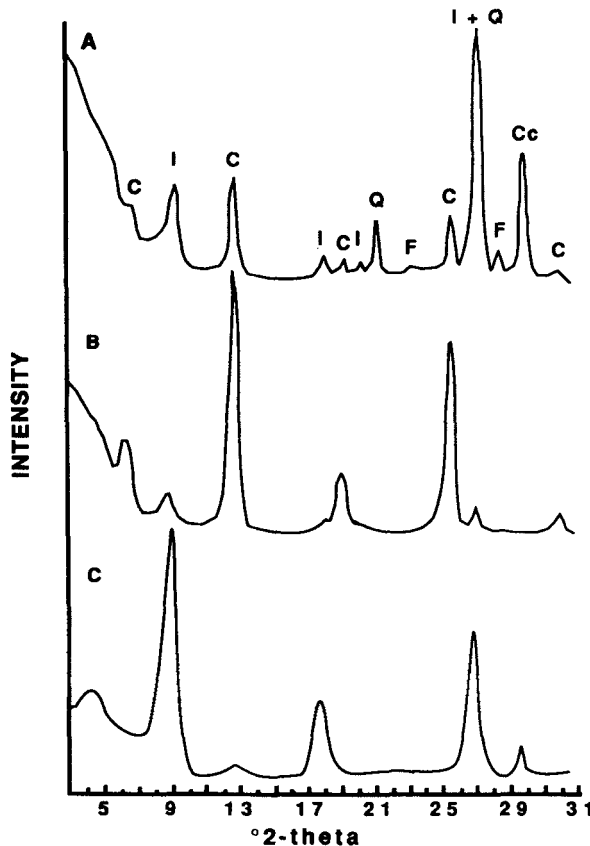


Figure 1. Representative X-ray powder diffraction patterns from oriented preparations of sample 6 (215°C), Borchard A-2 well, Imperial Valley, California, separated using high-gradient magnetic separation (Tellier *et al.*, 1988). (A) unseparated, <0.5- μm (e.s.d.) fraction containing chlorite, illite, quartz, feldspar, and calcite; (B) magnetic fraction containing primarily chlorite and minor illite and quartz; (C) nonmagnetic fraction containing primarily illite and minor chlorite or kaolinite, feldspar, and calcite (CuK α radiation).

β_{003} for the chlorite decreased from $0.62^{\circ}2\theta$ at 135°C to $0.45^{\circ}2\theta$ at temperatures >225°C (Figure 2). β_{005} for illite decreased from $2.24^{\circ}2\theta$ at 135°C to $1.0^{\circ}2\theta$ >205°C (Figure 3). These observations suggest that coherent diffracting domains within chlorite and illite grew to maximum sizes at about 200°–225°C and did not change at higher temperatures.

The percentage of 7-Å material in chlorite increased from 2% at 135°C to a maximum of 4.75% at 195°C; it then decreased to <1% >225°C (Figure 2). Yau *et al.* (1988) reported the presence of 7-Å layers (bertherine) in 14-Å material in lattice-fringe images of minerals formed in shales from the Salton Sea geothermal field at temperatures <200°C and no interstratification above that temperature. The percentage of illite in I/S increased from 40% at 135°C to 100% >205°C. Ordering in I/S changed from R0 to R1 between 135° and 155°C, and from R1 to $R \geq 3$ between 155° and 175°C

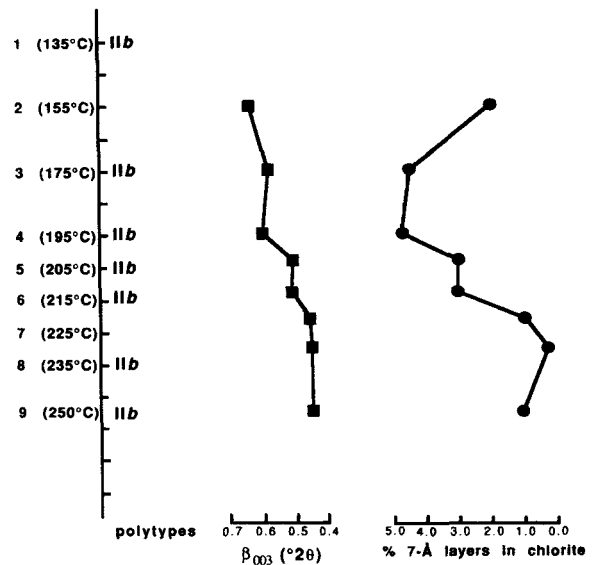


Figure 2. Downhole variations in chlorite polytype (symbols after Bailey and Brown, 1963), β_{003} , and relative percentage of 7-Å layers, Borchard A-2 well, Imperial Valley, California. Sample numbers are shown on the vertical axis with measured borehole temperature in parentheses.

(Figure 5). These downhole variations in I/S are consistent with the results of Jennings and Thompson (1986). Variations in percentage interstratification in both chlorite and illite correlate well with increasing

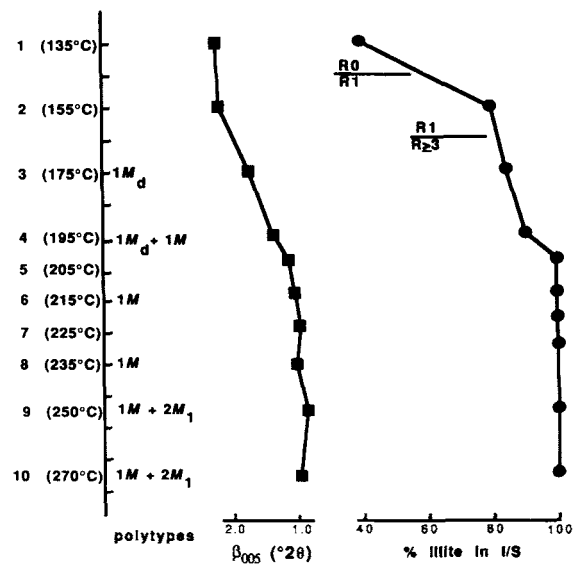


Figure 3. Downhole variations in illite polytype (symbols after Yoder and Eugster, 1955), β_{005} , percentage illite interstratification and ordering (R0 = random ordering, R1 = nearest neighbor ordering, R3 = long range ordering), Borchard A-2 well, Imperial Valley, California. Sample numbers are shown on the vertical axis with measured borehole temperature in parentheses.

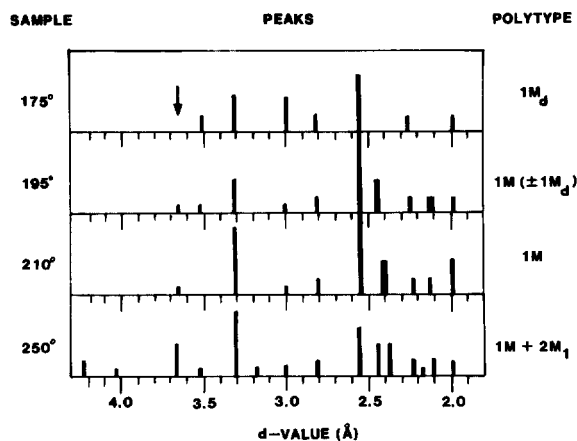


Figure 4. Schematic diagram of X-ray powder diffraction peaks observed between 2.0 and 4.3 Å in representative non-magnetic fractions heated to 300°C for 1 hr, Borchard A-2 well, Imperial Valley, California (114.6-mm Gandolfi camera, FeK α radiation).

size of coherent diffracting domains, suggesting that elimination of interstratified material was an important mechanism in the formation of larger diffracting domains.

DISCUSSION AND INTERPRETATION

The progressive ease with which chlorite was separated from illite in increasingly higher temperature samples is probably due to the presence of polyminerals grains containing both chlorite and illite in the <0.5- μ m size fraction of rocks formed at temperatures <200°C. TEM images of shales in the Salton Sea show interleaving of 200-Å packets of discrete illite and chlorite at 170°C, whereas at 300°C chlorite grains are >1000 Å thick (Yau *et al.*, 1988). Therefore, individual minerals grew larger with increasing temperature so that particles in the <0.5- μ m size fraction became more monomineralic downhole. The net result was cleaner separates of the higher temperature samples. This interpretation supports the hypothesis that chlorite and illite in the <0.5- μ m fraction were not detrital, but instead transformed from pre-existing material or neoformed. Qualitative comparison of the relative intensities of the 00 l peaks in the chlorite XRD patterns indicated no significant change in total Fe content with depth, which might have affected the character of the separations. The dissolution of iron oxide cements during diagenesis may have caused similar differences in separations; however, the system under investigation has been reported to be isochemical (Jennings and Thompson, 1986), and no evidence for selective dissolution has been reported.

The lack of detrital illite and chlorite among the mineral phases studied in this investigation is consis-

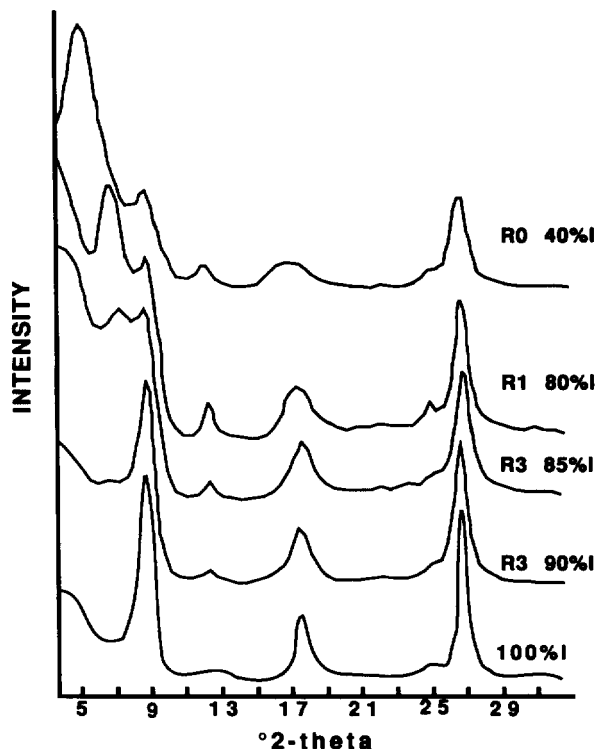


Figure 5. Representative X-ray powder diffraction patterns of ethylene glycol-saturated, oriented preparations of non-magnetic fractions, Borchard A-2 well, Imperial Valley, California. From top to bottom the samples were collected at measured borehole temperatures of: 135°, 155°, 175°, 195°, and 205°C (CuK α radiation).

tent with previous XRD studies (Muffler and Doe, 1968; Jennings and Thompson, 1986). It is also consistent with TEM observations that show that detrital illite grains in sediments of the Salton Trough are usually >2 μ m in diameter and that detrital chlorite is not present (Yau *et al.*, 1987; Yau *et al.*, 1988). In fact, fine-grained chlorite and illite have been observed intergrown as fillings in pore space on a micrometer scale, and based on their texture and composition, were interpreted to be authigenic (Yau *et al.*, 1988).

Structural changes in chlorite and illite occurred simultaneously with increasing temperature in the Borchard A-2 well. The nature of the structural changes in both minerals is consistent with an approach to equilibrium, inasmuch as heterogeneous (mixed-layer), metastable mineral phases alter to homogeneous, defect-free, stable phases (Lee *et al.*, 1985). Although the progress of illite and chlorite mineral reactions were similar, important differences existed. Chlorite did not differ in terms of polytype, whereas illite went through a regular series of polytype transformations. The progression of chlorite toward its final stable state was not continuous (Figure 2), whereas illite changed contin-

uously (Figure 3). The chlorite structural parameter β_{003} and the percentage of interstratification of 7-Å material did not change $>220^\circ\text{C}$ (Figure 2). For illite (Figure 3), β_{005} and the percentage of interstratification of smectite did not vary $>200^\circ\text{C}$, whereas polytype changes continued to at least 275°C .

If the minerals did, in fact, approach equilibrium, why do these differences exist? The Borchard A-2 system has been interpreted to be isochemical with respect to the major elements, the detrital mineral composition remaining the same throughout the section (Jennings and Thompson, 1986; McDowell and Elders, 1980). Observed differences between chlorite and illite may, therefore, be related to structural differences between the two minerals or to the mechanisms, such as neoformation or transformation, by which the minerals formed. The relationships between mineral structures and formation mechanisms can be understood by considering the possible mechanisms by which chlorite and illite may nucleate and grow in diagenetic sequences.

In burial diagenetic sequences, the breakdown of smectite has been reported to contribute the Mg and Fe necessary for the formation of chlorite (Hower *et al.*, 1976; Ahn and Peacor, 1985). Because of the difficulties inherent in transforming dioctahedral 10-Å layers into trioctahedral layers either 7 or 14 Å thick, chlorite probably does not grow directly from smectite, but instead crystallizes from solutions derived in part from smectite dissolution (neoformation). In this reaction, the 7-Å material interstratified in chlorite can be considered as "mistakes" that disappeared as the mineral recrystallized at higher temperatures (Ahn and Peacor, 1985). The uneven course by which chlorite progresses toward its stable state could result because nucleation and growth of a metastable phase is kinetically controlled and therefore not an equilibrium function of temperature.

Illite in burial diagenetic sequences may form by one of two mechanisms: by replacement of expandable layers in I/S (transformation) (Hower *et al.*, 1976) or by neoformation (Boles and Franks, 1979; Whitney and Northrop, 1988). Both mechanisms may operate simultaneously in the same rock (Yau *et al.*, 1987; Thompson and McCarty, 1988). The smooth progression by which illite transforms from $1M_d$ RO I/S containing 40% illite layers to $1M + 2M_1$ pure illite may result from the regular replacement of expandable layers in I/S by illite. The situation is no doubt complicated by the presence of discrete authigenic illite crystallizing over the same temperature interval as the I/S to illite transformation (Yau *et al.*, 1987). The occurrence of the $1M_d$ polytype exclusively with I/S of measurable expandability supports the suggestion that the $1M_d$ structure is present only in I/S and may not, in fact, be a polytype of pure illite (Brindley, 1984; Srodoń and Eberl, 1984).

Variations in chlorite polytypes are commonly assumed to depend on temperature, and the change from type-I to type-II polytypes is thought to occur at about 200°C (Bailey and Brown, 1963; Hayes, 1970). Field occurrences of a regular progression from type-I to type-II chlorite with increasing temperature, however, have rarely been documented (e.g., Karpova, 1969). To the authors' knowledge, coexisting authigenic type-I and type-II chlorite have been reported from only one locality (Tuscaloosa Formation of the Gulf Coast; Beskin, 1984). Occurrences of authigenic type-I chlorite in sedimentary rocks are limited to coarse-grained lithologies (sandstone, granular limestone, oolitic ironstones) and have not been described from fine-grained mudrocks (see Walker, 1989). The results of the present investigation suggest that authigenic chlorite crystallizing from solution in fine-grained rocks may form as the *I**b*** polytype at temperatures as low as 135°C , indicating that the lower limit of the temperature range of chlorite-*I**b*** formation could be $<135^\circ\text{C}$. The results further demonstrate that chlorite and illite polytypes behave differently under the same conditions.

SUMMARY AND CONCLUSIONS

In the Borchard A-2 well, chlorite was found to be uniformly the *I**b*** polytype. With increasing measured borehole temperature from 135°C , the size of coherent scattering domains increased until about 225°C , and the percentage of 7-Å interstratified material increased to a temperature of about 195°C , whereupon it decreased to about 225°C . Both parameters showed little variation in higher temperature samples. Over a similar temperature range, illite polytypes progressed from $1M_d$ to $1M$ to $1M + 2M_1$; however, even at 275°C , $1M$ illite coexisted with $2M_1$ illite, indicating that stability had not been achieved. The size of coherent scattering domains and the percentage of illite interstratification increased to maximum values at about 200°C and did not vary at higher temperatures.

The results suggest that chlorite and illite in the Imperial Valley may have formed by separate mechanisms. The exclusive occurrence of chlorite-*I**b*** at temperatures as low as 135°C and the appearance of $2M_1$ mica at temperatures as low as 250°C suggest that chlorite and illite polytypes behaved differently under the same conditions and that the formation temperatures of these "stable" polytypes may have been lower than previously suspected.

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