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UNIVERSITY OF OKLAHOMA

GRADUATE COLLEGE

WIDESPREAD CHEMICAL REMAGNETIZATION: OROGENIC FLUIDS OR BURIAL DIAGENESIS OF CLAYS?

A Dissertation

SUBMITTED TO THE GRADUATE FACULTY

in partial fulfillment of the requirements for the

degree of

Doctor of Philosophy

By

Bodo Katz Norman, Oklahoma

1998

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WIDESPREAD CHEMICAL REMAGNETIZATION: OROGENIC FLUIDS OR BURIAL DIAGENESIS OF CLAYS?

A Dissertation APPROVED FOR THE SCHOOL OF GEOLOGY AND GEOPHYSICS

BY

Pula El

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FOREWORD

Chemical remanent magnetizations (CRMs) are a common occurrence in sedimentary rocks (e.g., Elmore & McCabe, 1991). Understanding the processes that are responsible for chemically remagnetizing many rocks is important, especially since the CRMs can be dated paleomagnetically and therefore can provide information on the timing of the associated chemical event.

The most commonly invoked mechanism for the creation of CRMs is the so called "orogenic fluid hypothesis" (e.g., McCabe & Elmore, 1989; Oliver, 1992, and many others). Fluids that are expelled during orogeny are believed to migrate basinward and cause magnetite authigenesis, as well as ore mineralization, hydrocarbon pooling and a number of other geologically and economically important processes (e.g., Oliver, 1992). Several recent studies have identified inconsistencies between many widespread or pervasive CRMs and the "orogenic fluid hypothesis" (e.g., Elmore et al., 1993; Fruit et al., 1995; Banerjee et al., 1997). The presence of pervasive CRMs in relatively impermeable rocks or in units that show no evidence for alteration by externally derived fluids can be explained by a burial diagenetic mechanism (e.g., Banerjee et al., 1997). In fact, Katz et al. (1998) have shown that a CRM is created in the contact zone of a dike as a consequence of moderate heating by the intrusion, which can be considered a proxy for burial heating. The objective of this dissertation is to test one burial diagenetic process, the smectite-to-illite conversion, as a remagnetization mechanism.

In chapter one of the dissertation, paleomagnetic, rock magnetic, and geochemical tests for the orogenic fluid hypothesis as well as for a clay mineral diagenetic mechanism in carbonates of the Vocontian Trough of SE-France are presented. A number of studies have previously recognized the importance of burial diagenesis of clays in these rocks (e.g., Deconinck, 1987; Levert & Ferry, 1988).

Jurassic and Cretaceous carbonates have also been deformed by orogenic phases related to the Alpine orogeny and to thrusting of the Provence platform (Flandrin & Weber, 1966). Therefore, the area is appropriate to test for the presence of a CRM and an association to either orogenic fluids or burial diagenesis of clays. The paleomagnetic and rock magnetic results presented in chapter one provide evidence for an association of a widespread CRM in the central part of the Vocontian Trough with the conversion of smectite and geochemical tests argue against a connection to orogenic type fluids.

In chapter two, the extent of the CRM in the Vocontian trough and its possible association to clay diagenesis or orogenic type fluids is discussed. All locations and units are characterized by the CRM where smectite has been altered. The CRM is absent or weakly developed in partially altered units and a primary magnetization with no stable secondary remanence is observed in the only location where the clays are interpreted to show no sign of burial diagenesis. Evidence from rock magnetic studies confirm that authigenesis of magnetite has occurred with increasing degree of smectite alteration throughout the basin and additional geochemical evidence is presented that rules out possible connections to orogenic type fluids.

In chapter 3, evidence is presented indicating that burial diagenesis of smectite is also a viable remagnetization mechanism in other rock types such as sandstones and shales. The results suggest that magnetite authigenesis and the creation of a CRM might be commonly caused by burial diagenesis of smectite and that the changes in the magnetic signature with creation of a CRM as a result of burial diagenesis of clays, might be similar in carbonate and clastic settings.

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CHAPTER 1

Widespread chemical remagnetization: Orogenic fluids or burial diagenesis of clays?

ABSTRACT

Paleomagnetic, rock magnetic, and geochemical results from Mesozoic carbonates in the Vocontian trough in southeast France support a hypothesized acquisition of a chemical remanent magnetization (CRM) during burial diagenesis of smectite and are inconsistent with an orogenic fluid remagnetization mechanism. The geographic and vertical distribution of a widespread CRM, which resides in magnetite, is parallel to the degree of burial diagenesis of clay minerals. The CRM is absent only where there is no evidence for clay diagenesis. The CRM is poorly developed where significant smectite is still present and is well developed with higher natural and anhysteretic remanent intensities where smectite has completely altered to illite. Strontium isotope results do not support alteration by orogenic-type fluids, a commonly invoked mechanism for similar CRMs. The results suggest that the burial diagenesis of clays is a viable remagnetization mechanism for limestones in the Vocontian trough.

INTRODUCTION

Secondary magnetizations of chemical origin are common in sedimentary rocks (Elmore and McCabe, 1991). Although much progress has been made in the past 15 years in recognizing, describing, and determining the origin of chemical remanent magnetization (CRM), the mechanisms responsible for many CRMs are poorly understood and open to debate (Elmore and McCabe, 1991). Elucidating mechanisms of chemical remagnetization is therefore of significant interest to better understand the processes involved and because determining the time of secondary remanence acquisition by paleomagnetic methods can provide valuable data on the timing of diagenetic processes (e.g., McCabe and Elmore, 1989).

The temporal and spatial association between many CRMs and orogeny has led to the hypothesis that fluids expelled (Oliver, 1992) or derived (Garven, 1995) from orogenic belts are an important agent of chemical remagnetization. These fluids could potentially cause magnetite authigenesis and acquisition of a CRM on a large scale (McCabe and Elmore, 1989; Oliver, 1992; Stamatakos et al., 1996). Some previous studies have established a connection between CRMs and geochemical alteration caused by orogenic-type fluids around conduits for flow, but the evidence for a connection between pervasive and/or widespread CRMs in rocks that contain no geochemical evidence of alteration by such fluids is problematic (e.g., Elmore et al., 1993). Due to the pervasive nature of many of these CRMs, and their occurrence in relatively impermeable rocks, a burial diagenetic process is a likely remagnetization mechanism (Banerjee et al., 1997).

One potential burial mechanism is the alteration of smectite to illite. Smectite rapidly disappears at depths of 2 to 3.5 km (Chamley, 1989). Iron ions can be released during the conversion (Boles and Franks, 1979) and laboratory experiments confirm that magnetite authigenesis can result from clay diagenesis (Hirt et al., 1993). Smectite has been proposed as the source of the iron for magnetite authigenesis (Lu et al., 1991) and some studies have suggested a link between the degree of illitization and the amount of remanence carried by magnetite in remagnetized carbonates (Jackson et al., 1988b; McCabe et al., 1989). These studies, however, suggested that the potassium required for the clay transformation was provided by orogenic fluids.

In this study, the burial diagenetic conversion of smectite to illite and orogenic fluids were tested as possible remagnetization mechanisms in the Vocontian trough in southeast France. Results are reported for the Berriasian limestones at Berrias in the western part of the basin, where the clay mineralogy has been interpreted as showing little evidence for diagenetic alteration (Deconinck, 1993), and from limestones in the central part of the basin, where smectite has partly altered to illite in the younger units and completely disappears in older units (Deconinck et al., 1985; Levert and Ferry, 1988). The magnetization is characterized by paleomagnetic field tests and rock magnetic investigations. The ⁸⁷Sr/⁸⁶Sr values for different age rocks from different locations in the basin are presented to test for the presence of externally derived fluid signatures. Standard paleomagnetic, rock magnetic, and geochemical methods were used to analyze samples (e.g., Banerjee et al., 1997).

GEOLOGIC SETTING

The Vocontian trough in southeast France is bordered by the Massif Central to the west, the Provence platform to the south, the Vercors platform to the north, and the Alps to the east (Fig. 1). The pelagic and/or hemipelagic deposits of the basin are Jurassic and Cretaceous limestone-marl alternations and a few interbedded thick and resistant limestone breccias. The dominant structural features are large scale "pyreneoprovencal" folds caused by Eocene thrusting of the northward-moving Provence platform (Flandrin and Weber, 1966). Superimposed structural features include Miocene or younger north-south-trending faults and folds related to the Alpine orogeny. Several previous studies have documented the clay mineralogy in the basin and described burial diagenetic trends of the clays (Deconinck and Chamley, 1983; Deconinck et al., 1985; Levert and Ferry, 1988). Illitization was an important diagenetic process (e.g., Levert and Ferry, 1988) and the potassium needed for the conversion in these limestone-marl sequences could have been provided by the decomposition of potassium feldspar and mica (Chamley, 1989).



Figure 1-1. Map showing location of Vocontian trough (dashed line) and sampling locations (1, Sigottier-Serres; 2, Aulan; 3, Montclus; 4, Espreaux; 5, Chalancon). (Modified after Deconinck, 1993).

RESULTS AND INTERPRETATIONS

Stepwise demagnetization (thermal or alternating field) and subsequent data analysis confirm the presence of a primary normal or reversed polarity magnetization carried by magnetite at Berrias as determined in a previous study (Galbrun, 1985). Other than a modern viscous component, there is no secondary magnetization present at Berrias.

In the central part of the basin, the locations are characterized by a magnetization with northerly declinations and steep down inclinations after removal of a modern viscous overprint. At Montclus, the characteristic magnetization (ChRM) shows two distinct patterns based on stratigraphic position within the section. Most specimens (76%) in the older part of the section (Oxfordian to lower Tithonian), where all the smectite has been altered (Fig. 2D), contain the characteristic magnetization, which is removed below 580 °C and 120 mT (Fig. 2A; Table 1-1). The ChRM is well constrained; the average mean angular deviation (MAD; Kirschvink, 1980) is 5.1° (standard deviation = 3.1°). In contrast, 73% of specimens from the younger units (upper Tithonian to Hauterivian age), which still contain significant smectite, do not reveal a stable magnetization beyond 300 °C or 20 mT (Fig. 2A; Table 1-1). Only 18 specimens from these strata exhibit a mostly poorly defined ChRM directed northerly and down (average MAD of 10.5° , standard deviation = 4.4°).

Specimens from the older part of the section at Montclus also have higher natural remanent magnetization (NRM) intensities (Fig. 2B) and acquire more anhysteretic remanence (ARM) when exposed to a small magnetic field (0.1 mT) during alternating field treatment at 100 mT (Fig. 2C). At another section in the central area near Espreaux, an increase in NRM and ARM intensities also coincides with the disappearance of smectite, but in contrast to Montclus, the change occurs between Kimmeridgian and Oxfordian units. The average NRM intensity in Kimmeridgian



Figure 1-2. Results from central part of Vocontian trough near Montclus as a function of stratigraphic position (left column; limestone, hatchured; marl, blank; breccia, spotted) A: Orthogonal projection diagrams for representative specimens (tilt-corrected) from younger units (top) and from older units (bottom). Demagnetization usually isolates a modern viscous overprint at low unblocking temperatures or low alternating fields and characteristic magnetization with northerly declinations and down inclinations in most samples from older units at higher demagnetization steps. Closed symbols: horizontal projection; open symbols: vertical projection. **B:** Natural remanent magnetization (NRM) as a function of stratigraphic position. Intensities are higher in older units where characteristic magnetization is well developed. **C:** Anhysteretic remanent magnetization (ARM) as a function of stratigraphic position indicating that older units contain more remanence carrying magnetite. **D:** Percent smectite of total clay fraction (Deconinck et al., 1985).

limestones at Espreaux is $3.0 \times 10^{-8} \text{ Am}^2/\text{kg}$ (standard deviation = 1.8×10^{-8}), which is significantly lower than values in Oxfordian specimens ($1.1 \times 10^{-7} \text{ Am}^2/\text{kg}$; standard deviation = 2.5×10^{-8}). The ARM intensities at Espreaux are $3.4 \times 10^{-6} \text{ Am}^2/\text{kg}$ (standard deviation = 6.5×10^{-7}) for Oxfordian specimens and $6.6 \times 10^{-7} \text{ Am}^2/\text{kg}$ (standard deviation = 4.4×10^{-7}) for Kimmeridgian specimens. Therefore, the transition from high to low NRM and ARM intensities occurs at different biostratigraphic positions between Montclus and Espreaux but correlates with presence/absence of smectite.

Several field tests were conducted in the central area to constrain the timing of the characteristic magnetization. A fold test (Fig. 3; Table 1-1) on units at Serres-Sigottier (near Montclus) indicates a prefolding origin for the characteristic magnetization (Table 1-1). The declination/inclination (D/I) at Serres-Sigottier $(15.4^{\circ}/50.8^{\circ})$ is different than at Montclus $(D/I = 337.0^{\circ}/53.7^{\circ})$, which may be due to vertical-axis rotations associated with thrusting. The Montclus section is in the hanging wall of a thrust sheet and is separated from the Serres-Sigottier fold by the thrust fault. Results from another fold test at Aulan also indicate the presence of a normal polarity prefolding magnetization in the Upper Jurassic-Lower Cretaceous limestones in the cental part of the Vocontian trough east of Berrias (Table 1-1). A negative conglomerate test at Chalancon (Fig. 3B; Table 1-1) and the lack of reversed polarities throughout the section at Montclus and several other locations (Table 1-1) in the central area indicate that the magnetization is secondary. The mean paleopole position for the secondary magnetization is lat 78.5° N and long 178.8° E (A_{os} = 13.5°, K = 26, N = 5 locations from Table 1-1). Similar paleopoles are reported for Late Cretaceous-early Tertiary magnetizations of stable Europe (Van der Voo, 1990; Besse and Courtillot, 1991).



Figure 1-3. Fold and conglomerate test results. A: Stereographic projections of characteristic directions before tilt (and plunge) correction from opposite limbs of a fold at Serres-Sigottier in central area (circles and squares). B: Stereographic projections of characteristic directions at Serres-Sigottier after tilt and plunge correction. Directions from both limbs of a fold at Serres-Sigottier pass the fold test; best grouping occurs at 100% tilt correction, large circle: α 95. Characteristic directions from five individual clasts in Tithonian limestone-breccia at Chalancon (pluses, tilt corrected) as compared to well-bedded limestones (stars, tilt corrected) are illustrated. Directions from clasts are similar, indicating that magnetization is younger than deposition of the breccia. Solid symbols - lower hemisphere projection; open symbols - upper hemisphere projections.

	N/N ₀ •	In situ				Tilted			
LOCATION (age)		Decl. [†]	Incl. ⁶	k*	œ95∙•	Deci. †	Incl. [†]	k*	a95**
Sigottier/									
Serres FOLD									
(Kimm-Tith)	28/31	5 5 .2	43.0	1.5	52.7	15.4	50.8	28.3	5.2
Aulan FOLD									
(Tith)	18/18	354.1	54.8	10.1	11.4	354.1	56.3	97.8	3.5
Manalua									
Montcius									
(Ox-Lower Tith)	38/50	20.1	41.1	34.7	4.1	337.0	53.7	32.5	4.1
(Upper Tith-Haut)	18/67	5.3	46.1	18.0	8.4	337.4	43.7	14.6	9.4
F									
Espreaux									
(Ox-Kimm)	11/18	281.5	66.9	35.3	7.4	11.3	41.1	50.8	6.2
Chalanaan									
Chalancon	-		<i>.</i>						
bed (Kimm)	7/8	45.5	61.0	91.0	5.9	0.0	57.0	62.4	7.1
clast (Tith)	5/6	43.8	56.9	33.8	11.9	16.3	51.5	33.7	11.9

TABLE 1-1. PALEOMAGNETIC DATA

* Ratio of specimens used in the statistical analysis to the number of specimens studied.

[†] Declination in degrees.

⁴ Inclination in degrees.

* Statistical parameter (Fisher, 1953) associated with the mean directions.

"Radius in degrees of the cone of 95% confidence of the mean.

Results from acquisition of isothermal remanent magnetization (IRM) and the thermal decay of a triaxial IRM identify magnetite as the dominant carrier of remanence in the central area. Hysteresis loops consistently close below 300 mT, confirming the dominance of a low-coercivity mineral such as magnetite. Wasp-waistedness, which is frequently associated with chemical remagnetization, is not observed.

Burial depths (< 2.5 km) and low inferred burial temperatures (< 100 °C) are not consistent with a thermoviscous origin for the magnetization. Maximum laboratory unblocking temperatures are higher than predicted by unblocking temperaturerelaxation time curves for magnetite (Pullaiah et al., 1975), and thus the magnetization is interpreted to be a CRM. This interpretation is supported by the increase in ARM in the older parts of the sections, which indicates additional remanence-carrying magnetite where smectite has been completely altered to illite (Fig. 2, C and D).

The limestone muds are relatively impermeable and there is little evidence for pervasive flow of fluids (e.g., veining, alteration) in the limestones. The ⁸⁷Sr/⁸⁶Sr values for carbonate mud in limestones from different parts of the basin plot within the range of coeval seawater for the corresponding time intervals (Fig. 4) as compared to reference curves (Koepnick et al., 1985; 1990). Ratios would be higher than the coeval values if the rocks were affected by deep basinal fluids (e.g., Land and Prezbindowski, 1981). Because the rocks were not altered by radiogenic, chemically active fluids, the CRM is not related to such fluids.



Figure 1-4. ⁸⁷Sr/⁸⁶Sr ratios from different locations in Vocontian trough (circles) compared to range of values for coeval seawater for corresponding time intervals (Koepnick et al., 1985, 1990). Results are also consistent with another reference curve from Jones et al. (1994). Solid symbols show data from sites with chemical remanent magnetization, whereas the open circle is a sample that contains the primary magnetization at Berrias.

DISCUSSION AND SUMMARY

The paleomagnetic, rock magnetic, and geochemical results are consistent with the hypothesized genetic association between burial diagenesis of smectite and the creation of a CRM residing in magnetite. In the western part of the Vocontian trough where there is no evidence of clay diagenesis, the limestones contain a primary magnetization in magnetite and the CRM is absent. The CRM is present in rocks in the central part of the basin where smectite has altered to illite and is best developed in older units, which were buried below the critical depth at which smectite altered completely. This trend, as well as the increase in ARM and NRM intensities in the older units, suggests an acquisition of a magnetization through the addition of magnetite with increasing clay diagenesis. It is unlikely that differences in these values reflect variations specific to the different lithologic units, because increases in ARM and NRM occur at different stratigraphic positions in the two test sections. Although there is no independent determination of the timing of the smectite-illite transformation, acquisition of the CRM before Eocene folding is consistent with maximum burial in the Cretaceous (Levert, 1991). There is no temporal connection with Alpine folding, because the magnetization was acquired before the older thrusting of the Provence platform. A potential link between the CRM and burial diagenetic processes such as hydrocarbon maturation (Banerjee et al., 1997) can probably be excluded, because the amount of total organic matter in the rocks is low (Levert, 1991). There is also no apparent connection with externally-derived fluids.

One potential problem with the hypothesized burial diagenesis-remagnetization connection is the apparent absence of a primary magnetization in the rocks in the central part of the basin. This could be due to subtle facies differences between Berrias and the more basinward localities in the central part of the Vocontian trough or to an unknown dissolution process. Another issue relates to the possibility that the characteristic magnetization is a viscous overprint that reset a preexisting magnetization during the Cretaceous long normal polarity interval. Long polarity viscous remanent magnetizations (VRMs) reside in pseudo-single domain (PSD) magnetite (Moon and Merrill, 1986). Rock magnetic studies such as acquisition of an ARM (e.g., Jackson et al., 1988a) suggest a broad range of sizes, probably including single domain (SD) magnetite. This SD magnetite is likely to carry some of the remanence which could not be a long polarity VRM. Even if some PSD grains were reset by a viscous mechanism, the stratigraphic distribution of the characteristic magnetization suggests that it was originally chemical in origin and related to clay diagenesis.

The pervasive magnetization in the Vocontian trough is interpreted to be a prefolding CRM that is related to burial diagenesis of smectite. If additional tests confirm the proposed CRM-clay-diagenesis relationship, the potential benefits will include a better understanding of remagnetization mechanisms and the development of a method to date the burial diagenesis of smectite and potentially associated processes such as hydrocarbon migration.

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CHAPTER 2

Associations between burial diagenesis of smectite, chemical remagnetization and magnetite authigenesis in the Vocontian trough of SE-France
ABSTRACT

Results of a comprehensive paleomagnetic, rock magnetic, geochemical and petrographic study on Jurassic and Cretaceous carbonates of the Vocontian trough support a hypothesized connection between burial diagenetic alteration of smectite and the widespread occurrence of a chemical remanent magnetization (CRM) carried by magnetite. Where smectite has altered to illite, limestones are characterized by a prefolding, secondary, normal polarity magnetization throughout the basin. The magnetization is interpreted to be a CRM based on low burial depths which can not cause thermoviscous resetting. Where significant smectite is still present due to insufficient burial to cause complete illitization, the CRM is absent/weakly developed and where the clays show no evidence for burial alteration, the units are characterized by a primary magnetization. Isothermal, anhysteretic, and natural remanent magnetizations (IRM, ARM, and NRM, respectively) increase with degree of smectite alteration, both, stratigraphically and geographically. This is interpreted to indicate magnetite authigenesis with illitization. Based on acquisition of ARM, the magnetite grain size spectrum shifts to coarser mean values as the degree of smectite diagenesis increases. Also, superparamagnetic magnetite is more dominant in highly altered units based on the results of low temperature experiments. All sections away from the Alps have ⁸⁷Sr/⁸⁶Sr values that are similar to coeval seawater and stable isotopes of carbon and oxygen show no sign of alteration. Orogenic-type fluids, therefore, are not a likely agent of remagnetization. Near the Alps, the rocks are characterized by an additional reversed polarity component which is interpreted at one location (near the town of Blegiers) to reflect acquisition of the CRM through a reversal. A postfolding remagnetization is also present there and strontium isotopic ratios at this location are higher than elsewhere in the basin and might indicate some alteration by orogenic-type fluids. We conclude that burial diagenesis of smectite is the likely cause for the

development of a widespread and stratigraphically pervasive CRM in the Vocontian trough and that it might explain pervasive chemical remagnetization elsewhere.

INTRODUCTION

Chemical remanent magnetizations (CRMs) are a common occurrence in sedimentary rocks [e.g., *McCabe and Elmore*, 1989]. Many of these CRMs are stratigraphically pervasive and occur on a basin-wide scale [e.g., *Van der Voo*, 1989, *Elmore and McCabe*, 1991]. Several mechanisms have been suggested to be capable of chemically remagnetizing rocks. One of the most commonly invoked agents of chemical remagnetization is orogenic-type fluids [e.g., *McCabe and Elmore*, 1989, and numerous others]. Fluids expelled during orogenic phases are believed to be capable of causing magnetite authigenesis as well as other processes such as mineralization and potassium metasomatism [e.g., *Oliver*, 1992]. Although there is little doubt that these kinds of fluids can cause remagnetization in permeable rocks along fluid conduits [e.g., *Elmore et al.*, 1993], the common and pervasive CRMs which occur in rocks that contain no geochemical evidence of alteration by orogenic-type fluids need to be explained by another widespread diagenetic mechanism [e.g., *Elmore et al.*, 1995].

One alternative mechanism that could cause chemical remagnetization on a large scale is burial diagenesis of clays [*Katz et al.*, 1998]. Illite can form at the expense of smectitic minerals during burial to depths generally greater than 2 km [e.g., *Chamley*, 1989]. Smectites can release iron ions during the conversion to illite [*Boles and Franks*, 1979] and laboratory studies have shown that magnetite authigenesis can result from the conversion [*Hirt et al.*, 1993]. Potassium, which is needed for illitization, can be derived from the decomposition of potassium feldspars [e.g. *Chamley*, 1989]. Previous paleomagnetic studies have recognized the possibility that

smectite provides the source for the iron during widespread chemical remagnetization [e.g., Lu et al., 1990, Suk et al., 1990] and other studies recognize a connection between magnetite authigenesis and degree of illitization on a large scale [Jackson et al., 1988b, McCabe et al., 1989]. However, these studies suggest that potassium was provided by orogenic-type fluids which trigger the chemical remagnetization event.

Many examples of burial diagenesis of smectite have been described in the scientific literature [e.g., *Hower et al.*, 1976; *Chamley*, 1989] and one area for which several previous studies have recognized the importance of burial diagenetic processes on the clay mineralogy is the Vocontian trough of southeast France [e.g., *Deconinck*, 1987; *Levert and Ferry*, 1988; *Levert*, 1991]. The basin has also been affected by orogenic activity and several previous studies have investigated paleocirculation patterns of fluids [e.g., *Guilhaumou et al.*, 1996]. The location is therefore appropriate to test for an association between the magnetization and clay diagenesis, as well as orogenic-type fluids.

Katz et al. [1998] have previously presented evidence for a connection between a pervasive CRM in Mesozoic limestones around the town of Serres (Montclus, Sigottier, Espreaux) in the Vocontian trough (Figure 2-1) and the burial diagenetic alteration of smectite.

The objective of this study is to continue testing the clay hypothesis, as well as the orogenic fluid hypothesis, by correlating paleomagnetic, rock magnetic and geochemical results from throughout the basin. If the connection between burial diagenesis of smectite and acquisition of a CRM is correct, then a corresponding widespread CRM should be present throughout the basin where smectite has been altered. Paleomagnetic field tests are conducted to determine the time of remanence acquisition in different parts of the basin and to test for an association with fluids.



Figure 2-1: Location map of the Vocontian trough in SE-France. Sampling localities are indicated by closed circles.

Rock magnetic studies are performed to characterize the magnetization and to test for a correlation between authigenesis of magnetite and degree of smectite alteration. Geochemical studies are used to determine the potential influence of orogenic-type fluids.

GEOLOGIC SETTING

The Vocontian trough is a Mesozoic sedimentary basin that is located in SE-France (Figure 2-1). It is confined by the Provence platform to the south, the "Massif Central" to the west, the Vercors Platform to the north and the Alps to the east. The basin is filled with Jurassic and Cretaceous pelagic/hemipelagic limestone/marl alternations, except for a few thick and resistant limestone breccias that commonly occur in the Tithonian or occasionally in the Berriasian. Fold axes are generally oriented E-W and are doubly plunging. These kilometer-scale folds are mainly an effect of the northward thrusting of the Provence Platform during the Eocene [*Flandrin and Weber*, 1966]. Younger structural modifications include faulting associated with the Alpine orogeny. Modern tilting of the limestone/marl units caused by gravity induced slippage ("fauchage") frequently affects exposed beds. The investigated units were not buried significantly below 2 km anywhere in the basin based on estimates of the stratigraphic thickness [*Levert*, 1991].

Levert [1991] and Levert and Ferry [1988] have described the clay mineralogy in four isochroneous levels (in Upper Oxfordian, Kimmeridgian, Upper Valanginian and Clansayasian) in the Vocontian trough and concluded that the clay minerals generally reflect the degree of burial diagenesis. Smectite generally decreases and illite increases downsection throughout the Vocontian trough. They also note that the clay mineralogy in the eastern part of the basin (e.g., at Blegiers and Vergons/Angles) and in "isolated spots" (e.g., Kimmeridgian at La Roche sur Les Buis, Oxfordian at Col de Soubeyrand) in the basin center must reflect other alteration processes in addition to burial. In these locations, chloritization was an important mechanism. The authors suggest that the proximity of some isolated anomalies to major faults might indicate the influence of hydrothermal activity and the gradual increase in chlorite toward the Alps in the eastern part of the basin might be caused by a thermal effect related to the Alpine orogeny. In fact, *Guilhaumou et al.* [1996] described possible fluid migration events in underlying Callovian to Lower Oxfordian shales of the 'Terres noires' and along faults and salt domes.

Other studies have also observed the importance of burial diagenesis on the clay mineralogical signal (e.g., illitization and chloritization) and recognize the strong chlorite gradient toward the Alps as well [e.g., *Deconinck and Chamley*, 1983; *Deconinck*, 1987]. In another study, *Deconinck* [1993] interpreted the clay mineral assemblage in the Berriasian at Berrias in the western part of the basin to show no evidence for burial diagenetic alteration.

METHODS

Multiple cores were collected for paleomagnetic and rock magnetic analyses from sites at different localities throughout the Vocontian trough by utilizing a gasoline powered one inch drill. The samples were oriented with a clinometer and Brunton compass. Multiple cores were retrieved from opposing limbs of kilometer-scale anticlines and synclines for fold tests at 6 different locations (Aulan, Pradelle, Chavailles, Angles, and Blegiers, as well as Serres/Sigottier; *Katz et al.* [1998]). In the easternmost location of Blegiers, 3 sections from a syncline-anticline succession were sampled and at Angles opposing limbs of a fold that is separated by a series of faults were collected. At La Piarre and Blegiers, as well as Chalancon [*Katz et al.*, 1998], samples from individual clasts were retrieved for conglomerate tests. At Angles and Blegiers, as well as Montclus [*Katz et al.*, 1998], a large number of sites were collected covering substantial time intervals to test for the presence of a normal and reversed polarity pattern. At Cote Mare and near Blegiers, calcite veins and the surrounding rocks were sampled to test for a possible association of the magnetization to fluid alteration.

Samples were cut to standard lengths and weight normalized bulk magnetic susceptibility was measured on a Sapphire SI-2 Instrument for all samples (>1000 samples) prior to any other processing. The natural remanent magnetizations (NRMs) were measured on a 2G three-axes cryogenic magnetometer located in a magnetically shielded room. Specimens were subsequently stepwise demagnetized by alternating field (AF) demagnetization up to 160 mT in a 2G Automated Degaussing System or thermal demagnetization up to 700°C in a magnetically shielded Schonstedt TSD-1 oven. The resulting decay pattern was displayed in orthogonal projections [*Zijderfeld*, 1967] and line segments with mean angular deviations of less than 10° were identified prior to performing principal component analyses [*Kirschvink*, 1980].

Acquisition of isothermal remanent magnetizations (IRMs) produced by an impulse magnetizer and subsequent thermal demagnetization of three perpendicular IRMs with fields of 120, 400, and 1300 mT [*Lowrie*, 1990] were recorded for 36 samples from different localities and stratigraphic intervals to gain information on the magnetic mineralogy.

To investigate the total amount of remanence carried by magnetic minerals with low coercivity, the isothermal remanence acquired in a direct field of 200 mT was compared across the basin. The anhysteretic remanence (ARM) produced in an alternating field of 100 mT and a direct field of 0.1 mT was measured for previously AF-demagnetized samples. The ARM values are compared for individual beds for different sections with available clay data. Furthermore, the magnetic grain size distribution was estimated by ARM acquisition experiments. Selected specimens from various localities were subjected to the ARM field in increasing alternating fields (or the AF-decay of an ARM acquired at 100 mT) was recorded and the first derivative of the acquisition pattern was calculated. Alternatively, partial ARM (pARM, ARM acquired in AF windows; [*Jackson et al.*, 1988a]) was recorded on several specimens at the Institute for Rock Magnetism, University of Minnesota.

Hysteresis experiments and measurements of coercivity of remanence were performed on Micromag Alternating Gradient Force Magnetometers at the University of Utah and at the Institute for Rock Magnetism, University of Minnesota. The thermal decay of a low temperature saturation IRM (SIRM) was acquired on a Magnetic Property Measurement System (MPMS) at the Institute for Rock Magnetism, University of Minnesota, for 31 representative samples and cooling of SIRM acquired at room temperature was recorded for some samples.

Strontium isotopic ratios (87 Sr/ 86 Sr) were determined for samples from different localities and age intervals at the University of Texas, Austin. Homogenous limestones were crushed and micro-sampled for micrite. The rock powder was then washed three times in 0.2 reagent grade ammonium acetate and rinsed three times in ultrapure water (D3). Subsequently, the samples were dissolved in 1.4 N acetic acid for 5 minutes. Cation exchange columns with 2 N HCl concentrated the strontium. A MAT 261 mass spectrometer with a fractionation correction of 0.1194 was used for the analysis. The NBS 987 was measured frequently and the reported ratios were normalized to the standard. Stable carbon and oxygen isotope values were determined for multiple samples from a variety of locations and time intervals. Micritic limestones were crushed and digested in 100% phosphoric acid (2 hours, 50°C). The resultant CO₂ gas was analyzed using a Finnigan Delta E isotope ratio mass spectrometer at the University of Oklahoma. The δ^{13} C and δ^{18} O values are reported in

per mil relative to PDB. The are corrected to 25°C using the fractionation factors of Swart et al. (1991). Details of the method are reported elsewhere (Bixler et al., 1998). Thin sections of representative samples were also examined using a polarizing microscope in reflected and transmitted light.

PALEOMAGNETISM

Demagnetization of most specimens from all locations removes a northerly and down magnetization (e.g., Figure 2-2a, b) at low alternating fields (below 25 mT) or temperatures (below 250 to 320°C). This magnetization is interpreted to be a modern viscous remanent magnetization (VRM). Only one studied section in the basin allows for an identification of a primary magnetization. The Berriasian at Berrias is characterized by a normal and reverse polarity pattern that has been tied to the magnetic polarity time scale [*Galbrun*, 1985]. We have confirmed the existence of a primary magnetization at Berrias and observe no stable secondary magnetization at this location.

At most other locations, a characteristic remanent magnetization (ChRM) which is northerly and positive in tilt corrected coordinates is removed by thermal treatment to approximately 530°C or alternating field demagnetization to 100 mT (e.g., Figure 2-2a, b). The mean directions for this component differ between locations if no bedding correction is applied (Figure 2-2a, b, Table 2-1). The normal polarity component is the only ancient magnetization present in most locations, except at Blegiers and Angles in the eastern part of the basin, where the magnetization is more complex. The results from these locations are described separately from locations which only carry the normal polarity component.





Figure 2-2: Orthogonal projection diagrams [*Zijderfeld*, 1967] of representative stepwise demagnetizations of specimens from different locations. Open symbols: vertical projection, closed symbols: horizontal projection. a) Thermal demagnetization of a sample from one of the limbs of the Aulan fold, b) thermal demagnetization of a sample from Tithonian deposits at Pradelle, c) decay for a specimen from the Berriasian of Angles with the additional reversed polarity component at low alternating fields after heating to 250° C, d) thermal decay for a specimen from the western limb in the reversed polarity component at low temperatures, e) alternating field demagnetization of a specimen from the western limb in the Angles area, f) thermal decay pattern of a specimen from Blegiers with the two northerly and positive components, g) thermal demagnetization of a specimen from Blegiers with a third, high unblocking temperature component above 530° C, h) alternating field demagnetization of a specimen from Blegiers only isolates one apparent component, i) thermal cleaning to approximately 310° C and subsequent AF-demagnetization is the most successful method to isolate the 2 high stability components.

		IN SITU					П	LTED				
Location	n/n _o	DEC	INC K		α95	DEC	INC K		α95	POLE	dp/dm	
Aulan,Fold	26/26	354.6	54.9	11.3	8.8	354.2	56.3	99.9	2.9	81.4N 142.1W	3.0/4.1	
Pradelle,Fold	19/19	35.4	50.1	1.9	34.7	357.3	53.9	33.9	5.8	79.6N166.6W	5.7/8.2	
Chavailles, Fold	18/25	153.5	44.8	8.3	12.8	333.2	65.0	43.5	5,3	71.5N64.8W	6.9/8.6	
Vergons, Valangin	8/10	301.9	63.9	26.7	10.2	352.5	49.2	26.5	10.2	75N147.5W		
La Charce, Oxford	6/6	356.6	23.1	37.2	10.1	18.1	64.8	47.9	8.9	77,2N79.2E		
La Piarre,Kimm	5/6	302.9	75.8	11.8	20.6	314	56.3	12	20.4	54.8N80.3W		
La Piarre, clasts	6/6	286.4	77.9	130.9	5.4	326.1	44.9	130.6	5,4	57.5N106.2W		
La Piarre, Tithonian	10/30	254.1	75.1	28	8.8	321,8	51	30.3	8.4	57.8N93.4W		
Cote Mare, Ox-Kim	6/10	278.5	48.3	36.3	10.3	10.5	57.4	31.6	$\begin{bmatrix} \mathbf{n} \end{bmatrix}$	80.1N129.6E		
Crussol,Oxfordian	10/14	13.9	64.4	29	8.6	11.7	59.4	29.3	8;6	80.4N116.9E		
Aulan, Valanginian	7/8	8.3	32.1	29.6	10.4	12.5	61.8	_30	10.3	80.9N98.9E		
La Roche, Kimm	9/9	270.9	60.1	77.3	5.6	9.4	50.1	77	5.6	74.7N153.2E		
Col Soub.,Oxford	3/4	25.5	-10,6	18,4	23,9	22	56.1	18.5	23.8	71.7N112.2E		
ANGLES, Berr												
normal, fold-test	35/55	342.4	70,1	6.8	10.8	322.1	46.6	36.8	4.4	55.5N99.3W	3.6/5.7	
reversed, both limbs	13/55	158.1	-30.5	6.1	17.4	162.9	-1.4	4.9	19.7	44.1S31.1E		
BLEGIERS, Tith									_			
low T,T/AF,fold	39/60	358.1	59.6	57.6	3	322.8	73.4	3	16	86N153.1W*	3.9/5.2*	
AF,fold	14/17	351.4	54.3	55.8	5.2	322.3	59	2.8	27.6	78.5N136.7W*		
med. T, fold	36/60	340.4	28.8	2.7	17.6	355.5	39.8	17.4	5.9	68.1N163.5W	4.3/7.1	
med.T, CG-test	13/17	20.8	-14.7	19.2	9.3	20.3	43.1	19.1	9.3			
high T, only limb2	9/60	176.2	5.1	7.7	18.6	163.6	-45.4	7.7	18.5	68.2S48.1E		
high T, CG-test	8/17	173.2	6.5	7.4	20.2	161.3	-42.6	7.4	20.2			
near vein, older units	12/14	351.1	52.1	376.4	2.1	350.0	61.1	375.9	2.1			

Table 2-1: Paleomagnetic summary.

C'vieux, Valan. (0/7), Tarandol, Clans. (0/6), La Charce, Hauteriv. (2/9), Sigottier, Clans. (1/6), Col de Paluet, Clans. (2/5),
*, calculated from in situ coordinates for low stability component at Blegiers,
() or n/no, number of demagnetized specimens versus number of specimens with direction,
Dec: declination, Inc: inclination, k: precision parameter, α95: cone of 95% confidence, dp/dm: semi minor and semi major

axis in degrees, respectively, of the oval of 95% confidence about the pole.

Locations with single component

A fold test and a negative conglomerate test in the central area of the Vocontian trough indicate a prefolding and secondary origin for the magnetization [Katz et al., 1998]. A negative reversal test is consistent with the interpretation that the magnetization is secondary [Katz et al., 1998]. Results from other areas, as described below, are also consistent with the preliminary studies.

An incremental fold test on Kimmeridgian beds from an anticline near Aulan indicates that specimen directions group best at 100% unfolding (Figure 2-3a, 2-4a) with a northerly and down direction. According to the *McElhinny* [1964] fold test, the result is significant at the 99% confidence level when compared to the postfolding direction. The McFadden and Jones [1981] fold test suggests that the two limbs do not have a common mean at the 95% confidence level. This result therefore, fails the test, which may be due to the fact that specimen directions were used instead of site means as suggested by *McFadden and Jones* [1981]. Directions of Tithonian specimens from an anticline at Pradelle also group best at 100% unfolding in an incremental fold test (Figure 2-4b), although the prefolding interpretation fails the McFadden and Jones [1981] test and specimen directions from both limbs never completely converge (Figure 2-3b). However, the McElhinny [1964] test is significant in the prefolding orientation at the 99% confidence level. Directions from another fold test on Barremian limestones at Chavailles also group at 100% unfolding (Figure 2-3c, 2-4c) with significance at the 99% confidence level after McElhinny [1964]. The McFadden and Jones [1981] test suggests that the two limbs have a common mean at the 95% confidence level after structural correction.





Figure 2-3: Stereographic projections of magnetic directions for *in situ* and tilt corrected coordinates from different folds. Closed circles: projections on the lower hemisphere, open circles: projections on the upper hemisphere. Alpha 95 indicated where applicable. a) Kimmeridgian at Aulan, b) Tithonian at Pradelle, c) Barremian at Chavailles, d) normal and reversed magnetization in Berriasian of Angles, e) Tithonian at Blegiers: low temperature component, f) Tithonian at Blegiers: medium and high temperature components.





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Figure 2-4: Incremental fold tests for normal polarity magnetization (a, b, c, d, f) which can be recognized at all locations where smectite has altered completely and e) for low temperature magnetization of the Tithonian at Blegiers. Closed symbols: precision parameter k at various steps of unfolding, F-values are indicated by open symbols, horizontal line is 95% confidence level [*McFadden and Jones*, 1981] for the common mean.

A secondary origin of the magnetization is indicated by a negative conglomerate test on clasts from Tithonian limestones at La Piarre (Table 2-1). In addition, no reversed polarity magnetizations were isolated in any of the sections, although multiple sites covering considerable stratigraphic intervals were collected at each location. The bedding corrected mean directions from other locations without fold tests (Valanginian at Vergons, Oxfordian at La Charce, Tithonian and Kimmeridgian at La Piarre, Oxfordian at Crussol, Valanginian at Aulan, Kimmeridgian at La Roche, Oxfordian at Col de Soubeyrand; Table 2-1) are similar to the tilt-corrected means from the fold test locations. The paleopole positions for this magnetization from the different locations (Table 2-1) are consistent with a Middle-Upper Cretaceous age of the magnetization [*Van der Voo*, 1993].

Limestones from 5 locations (Valanginian at Chateauvieux, Clansayasian at Tarandol, Hauterivian at La Charce, Clansayasian at Sigottier and Col de Paluet; Table 2-1) either contain a weakly developed normal polarity magnetization in a few specimens or do not allow for an identification of a stable remanence. The units of Upper Tithonian or younger age at Montclus also have a low percentage of specimens that carry a normal polarity magnetization as well [*Katz et al.*, 1998].

Locations with multiple components

ANGLES: Berriasian units from the Angles area carry two stable components. A southerly and up magnetization that is completely removed by approximately 400°C or 25 - 40 mT (Figure 2-2c, d, e) can be recognized in a few sites (9 of 32 specimens from 5 out of 22 sites). Alternating field demagnetization is less successful than thermal demagnetization in isolating this component (Figure 2-2e). Combining thermal demagnetization with AF-decay isolates this component at low alternating fields (Figure 2-2c, Table 2-1). A fold test on this southerly component shows a best

grouping at 0% unfolding. However, the postfolding result is not significant at the 95% confidence level of the *McFadden and Jones* [1981] and *McElhinny* [1964] tests. The inconclusive nature of the fold test may be due to overlapping unblocking temperature and coercivity spectra which cause a large scatter in specimen directions. Although not statistically valid, the results suggest a post-folding magnetization. We interpret this magnetization as postfolding and secondary in origin.

The ChRM (Figure 2-2c, d and Figure 2-3d, northern hemisphere) is generally present above 340°C and in excess of 20 mT. This component is isolated in most of the 22 sites that span the Berriasian. The exclusively normal polarity of this magnetization throughout the Berriasian suggests a secondary origin for this characteristic component. A fold test on the characteristic direction reveals a best grouping at 85% unfolding, but is not significantly different from a grouping in tilt-corrected coordinates (Figure 2-3d, 2-4d) and it is interpreted to have been acquired before folding. The prefolding, secondary nature of the component, the similarity in demagnetization characteristics, and the similarity in direction to the ChRM from other locations suggests that it is the same component. The area around Angles borders the Provence platform and intense thrusting may have caused vertical axes rotations of individual blocks. Therefore, a paleomagnetic pole from this location should be regarded with caution.

BLEGIERS: Specimens were collected from three limbs of an anticline-syncline system in the Tithonian near Blegiers. Tithonian limestones near the town of Blegiers carry up to three stable components.

Thermal treatment isolates a northerly magnetization that typically unblocks between 200 and 370°C but remains stable up to 460°C in a few specimens (Table 2-1, Figure 2-2f, g, low temperature). This component is isolated in alternating fields up to 140 mT (Figure 2-2h). The major part of the NRM in these specimens is carried by this magnetization. In some cases, the AF-demagnetization path is curved, which is indicative of overlapping coercivity spectra of different components. A fold test on this component indicates a best grouping at 0% unfolding (Figure 2-4e) for the thermally demagnetized specimens and the direction is northerly and down (Table 2-1, Figure 2-3e). Directional information from AF demagnetization is similar and also indicates a post folding origin of the magnetization (Table 2-1). However, the AF demagnetized specimens, which exhibit the postfolding direction, were not used for the fold test, since it is apparent that AF demagnetization results in an overlap with at least one other direction. The paleopole position for the low temperature component is consistent with a Tertiary age [*Van der Voo*, 1993].

An additional northerly and down component (Figure 2-2f, g, medium temperature) is removed between 375 and 520°C (21 of 38 specimens). This magnetization is also successfully isolated by alternating fields up to approximately 40 mT after initially heating the specimens to approximately 300°C (Figure 2-2i). The best grouping is at 100% unfolding (Figure 2-4f), although the data do not converge completely (Figure 2-3f). The direction is similar to the prefolding directions from other locations (medium temperature, conglomerate test; Table 2-1). A grouping of directions from clasts indicates a secondary origin of this magnetization (Table 2-1). The intermediate stability magnetization is interpreted to be the equivalent normal polarity, prefolding, secondary ChRM found elsewhere in the basin. The paleopole position for this magnetization is similar to the results from other locations (Table 2-1).

Some specimens from individual clasts (7 of 17) and one specimen of a bedded Tithonian limestone from the SW-dipping limb exhibit a southerly magnetization that unblocks at temperatures between 530 and 560°C (Figure 2-2g, high temperature). Deriving directional information for this component is hampered by viscous behavior at temperatures in excess of 500°C in some samples. The direction for specimens from

the SW-dipping limb is southerly in bedding corrected and uncorrected coordinates (Figure 2-3f). The inclination changes from shallow to steep down after correcting for the dip of the beds. Individual clasts group for this component and the conglomerate test is therefore negative. No specimens from the steep NE-dipping limb contain this reversed polarity magnetization and only one specimen contains this component on the third, shallow NE-dipping limb (square, Figure 2-3f). The high temperature component is a reversed polarity magnetization that was acquired after deposition and a primary origin can therefore probably be excluded. Although deriving directional information is hampered by partial overlap with the normal polarity magnetization and a fold test was not possible, the tilt corrected mean direction for this component is approximately antipodal to the prefolding normal polarity magnetization and we interpret the magnetization to reflect acquisition through a reversal. The one specimen from the shallow limb with the reversed polarity magnetization also plots closer to the mean of this magnetization in tilt corrected coordinates which supports this interpretation. In addition, it can be argued that the magnetization was acquired prior to folding since the shallow inclination in geographic (in situ) coordinates leads to a paleopole position that has no significance at any time interval between the Jurassic and the present.

MAGNETIC MINERALOGY AND ORIGIN OF THE MAGNETIZATIONS

All samples from Upper Jurassic/Lower Cretaceous limestones of the Vocontian trough show a rapid rise in remanent magnetization at low direct magnetic fields. The majority of samples analyzed (22 of 36) reach saturation below 300 mT, which indicates the dominance of a low coercivity mineral (Figure 2-5). Some samples

continue to acquire remanence at greater field strengths, indicating the presence of a high coercivity phase. The samples with both high and low coercivity phases are distributed throughout the sections and are not restricted to particular lithologies. The thermal decay of triaxial IRMs show that the low coercivity phase is removed by 580°C (Figure 2-5) in all samples, which is indicative of magnetite. Samples which also contain a high coercivity phase either lose their remanence on the 1.3 T axis by 680°C or show a significant drop below 150°C with subsequent complete decay by 680°C. Therefore, hematite and/or goethite, respectively, are present in these samples.



Figure 2-5: Representative acquisition of an IRM (normalized) and subsequent thermal demagnetization of a triaxial IRM (inset). Saturation remanence is acquired at low fields and heating to 580°C effectively removes the remanence, indicating that magnetite is the dominant carrier of the magnetization.

Magnetite is the dominant magnetic phase in limestones across the basin for all studied intervals. The presence of hernatite and/or goethite in some specimens is not related to the presence/absence of smectite and we interpret oxidation during surface exposure to be the likely cause for the presence of these minerals in some specimens. Neither goethite nor hematite, where present, carry a significant part of the NRM.

Since magnetite is the main carrier of remanence, the high maximum unblocking temperatures (530°C) of the prefolding, pervasive magnetization probably exclude a thermoviscous origin of the magnetization. The Upper Jurassic/Lower Cretaceous carbonates in the Vocontian trough were not buried significantly below 2 km based on estimates of the overlying stratigraphic section and, therefore, burial temperatures probably did not exceed 100°C anywhere in the basin. According to the time-temperature-unblocking temperature relationship of *Pullaiah et al.* [1975], the burial temperatures were insufficient to cause a thermoviscous resetting of the magnetization. The pervasive, normal polarity, prefolding magnetization which is present in all sections where smectite has altered is therefore interpreted to be a CRM. The reversed high temperature component at Blegiers is interpreted to represent acquisition of the CRM through a reversal.

The normal polarity postfolding magnetization at Blegiers could be a thermoviscous remanent magnetization (TVRM) or a CRM. Low burial depths (approximately 2 km) and relatively high maximum laboratory unblocking temperatures argue against a thermoviscous origin of the magnetization. However, other heat sources have to be considered at Blegiers, since evidence from the clay mineralogy indicates anomalous degrees of diagenesis. In fact, estimates from fluid inclusion and vitrinite reflectance studies derive paleotemperatures as high as 250°C in the underlying 'Terres noires' shales near Blegiers [e.g., *Guilhaumou et al.*, 1996]. These temperatures could be sufficient to explain the observed maximum laboratory

unblocking temperatures of the postfolding magnetization (460°C) by a thermoviscous mechanism.

The origin of the reversed polarity magnetization at Angles is unknown but could be a thermoviscous overprint because of the relatively low maximum unblocking temperatures. This magnetization was probably acquired after folding.

CLAY DISTRIBUTION AND ASSOCIATIONS TO THE MAGNETIZATION

The clay mineralogy in the Vocontian trough generally reflects a burial diagenetic signature with smectite being absent in the older and more deeply buried rocks [e.g., *Levert and Ferry*, 1988]. In younger, overlying units, the degree of smectite diagenesis is generally less and no sign of alteration of smectite exists in the Berriasian of Berrias (Figure 2-1, Table 2-2). Disappearance of smectite accompanied by an increase in illite downsection occurs in different stratigraphic intervals throughout the basin (Table 2-2). In proximity to the Alps, smectites are also altered in younger units (Table 2-2) and conversion to chlorite becomes more important [e.g., *Levert and Ferry*, 1988].

The NRM intensities are generally higher where smectite has been altered than in units where smectite is abundant (Table 2-2). These variations in NRM intensity with degree of smectite alteration suggest a connection between the magnetization and burial diagenesis of smectite. In addition, the widespread and stratigraphically pervasive normal polarity CRM is present at all locations where smectite has altered to illite (compare Tables 2-1 and 2-2).

LOCATION/ STRAT POS	BER	CR US	PRA	CH AL	LA CH	CS/ TAR	LA RO	AŬ	LA P	SIG	MC/ PAL	CV/ CM	ESP	AN/ VER	CH AV	BL EG
BARREMIAN CLANSAY						1.4 O				1.9 O	2.3 O	2.6 O			2.2 ●	•
HAUTERIVE					1.0						1.2 O					
VALANGIN			0			0		4.4		0	3.0 O	1.8 O		2.9 O		10.7
BERRIAS	1.8 ★								2.3		1.7 O	0.9		3.0 ●		
TITHONIAN			6.0					2.9	11	6.4	2.4 O 8.5 ●					58.5
KIMMERIDGE		6.9	•	8.2 O	11.2 O	0	4.3 ●	•	14	5.0 O	13.4 ●	0.5 O	3.1 O			•
OXFORD		3.9	•	•	20	8.3 ●			25 ●		16.5 ●	3.1 ●	8.5 ●			•

Table 2-2: Clay association with magnetization. Percent smectite and NRM data for the different locations and time intervals. Closed circles: smectite has altered completely, open symbols: significant smectite present, asterix: no burial diagenesis of clays, shaded circle: 15% (of total clay) smectite or less. Numbers indicate average NRM intensity in E-8 Am2/kg. Locations from west to east (approximately), BER: Berrias, CRUS: Crussol, PRA: Pradelle, CHAL: Chalancon, LA CH: La Charce, CS/TAR: Col de Soubeyrand/ Tarandol, LA RO: La Roche sur Les Buis, AU: Aulan, LA P: La Piarre, SIG: Sigottier, MC/PAL: Montclus/ Col de Paluet, CV/CM: Chateauvieux/ Cote Mare, ESP: Espreaux, AN/VER: Angles/ Vergons, CHAV: Chavailles, BLEG: Blegiers. Clay data compiled from Levert & Ferry (1988), Levert (1991), Deconinck (1987), Deconinck (1993).

The timing of burial diagenesis of smectite is not known, but the timing of maximum burial is estimated at 120 Ma [e.g., *Guilhaumou et al.*, 1996]. The poles from the fold tests are in good agreement with paleopoles of 60 to 120 million years before present [*Van der Voo*, 1993]. The age of this pre-Eocene CRM is therefore consistent with the likely timing of smectite alteration.

The only location where a primary magnetization can be identified and no ancient secondary magnetization exists (Berriasian at Berrias; [Galbrun, 1985]) shows no evidence for burial diagenesis of clays [Deconinck, 1993]. Units with low degrees of smectite alteration are either weakly magnetized or do not allow for an identification of a stable remanence. The stratigraphic positions in which smectite disappears vary across the basin, which is in accord with the paleomagnetic data.

ROCK MAGNETISM

IRM and magnetic susceptibility

The remanence per unit mass acquired in a direct field of 200 mT shows variations with locality and position in the different sections (Figure 2-6). Samples from Blegiers carry significantly more isothermal remanence than those from any other locations. Samples from Berrias, which contain the primary magnetization, are capable of carrying more IRM than samples from all other sections except Blegiers. At Montclus, all samples from units where smectite has altered completely carry more IRM at 200 mT than samples where smectite is present. Results from other sections in the basin are consistent with this observation, irrespective of where in the section the smectite disappears. Additionally, the magnitude of IRM is similar for the different locations where smectite has not altered completely and are also comparable for units



Figure 2-6: The IRM acquired at 200 mT for samples from different locations and ages. Open circles: abundant smectite present, closed circles: smectite has altered, crosses: samples from unaltered Berrias-section, squares: highly altered samples from Blegiers close to the Alps. Remagnetized samples where smectite is altered have generally higher values of IRM except at Berrias. The highest values can be observed at Blegiers where two remagnetization components were identified.

where the smectite was transformed during burial diagenesis. Magnetic susceptibility varies greatly within sites, sections and also between locations. No apparent pattern can be recognized.

In summary, the amount of remanence carrying magnetite as estimated by IRM acquired at 200 mT varies with the degree of smectite alteration. The increase in IRM is interpreted to reflect authigenesis of magnetite with increasing transformation of smectite. The variation also provides supporting evidence for the interpretation of a chemical remagnetization mechanism.

ARM and NRM

Values of ARM generally increase with decreasing amounts of smectite (Figure 2-7) in a Valanginian limestone bed that has been traced across the basin [*Levert and Ferry*, 1988]. The NRM follows a similar trend (Figure 2-7). Except for the differences in the degree of clay alteration, the rocks from the widely separated locations exhibit no apparent petrographic differences. Kimmeridgian results are consistent with the above observation, although the increases in ARM and NRM (Figure 2-7) are less pronounced and the variation within each section is greater, as expressed by the standard deviations.

The results from other time intervals are also consistent with such a correlation. For example, all 5 locations with Oxfordian units where smectite has disappeared have ARM values (in 10^{-6} Am²/kg: Espreaux: 2.38, La Charce: 4.25, Montclus: 2.52, Col de Soubeyrand: 2.0) that are higher than any values from units where smectite is still present (compare to Figure 2-7). Additionally, low ARM values in the Berriasian limestones of Montclus ($1.96 * 10^{-7}$ Am²/kg) which have approximately 45% smectite are comparable to higher ARM values in the Berriasian at Angles and Vergons (5.6 *



Figure 2-7: The ARM values as a function of smectite clay fraction for an Upper Valanginian limestone bed across the basin. Values are higher where smectite is more altered. On average, 5 samples were used to calculate the mean at each location. Inset: NRM (solid line) follows a similar trend for the same time interval. The dashed line indicates NRM variations in the Kimmeridgian across the basin, ARM of the Kimmeridgian is not shown but also generally decreases with increasing smectite.

 10^{-7} Am²/kg and 3.7 * 10^{-7} Am²/kg) where smectite exists only in traces. The unaltered Berriasian at Berrias, however, has ARM values (3.79 * 10^{-6} Am²/kg) that are higher than the Berriasian from remagnetized samples at Angles and Vergons.

These geographic trends in ARM intensities are interpreted to reflect varying degrees of magnetite authigenesis. The correlation to the amount of smectite present in the rocks from the different locations suggests an association between burial diagenesis of clays and formation of magnetite. The geographic trends in ARM (and NRM) are consistent with the previously identified vertical correlation to the degree of smectite alteration at Montclus and Espreaux [*Katz et al.*, 1998] and with increasing IRM intensities (at 200 mT) downsection. This lateral trend also argues against a possible vertical lithologic control on the magnetite content, at least for locations east of Berrias. The large values of ARM and IRM at Blegiers could reflect the two recognized remagnetization events.

ARM acquisition and pARM

Samples from all locations acquire most of the ARM at low fields (Figure 2-8), which is indicative of relatively coarse multi domain (MD) to pseudo-single domain (PSD) magnetite [e.g., *Jackson et al.*, 1988a] However, part of the ARM is acquired at high fields, which is interpreted to reflect the presence of some fine, more stable single domain (SD) magnetite.

Mass normalized changes in anhysteretic remanence with increasing alternating fields are more pronounced in samples from older units where smectite has altered (example: Montclus, Figure 2-8a). This observation is consistent with higher ARMs where smectite has altered in older units as described previously [*Katz et al.*, 1998],





Figure 2-8: Changes in anhysteretic remanence with increasing alternating fields. a) Mass normalized results from Montclus. Smectite bearing units (dashed lines) carry less magnetite in all size ranges than older units without smectite (solid line), b) Normalization to maximum change indicates shift to coarser mean size for samples from units with CRM and without smectite (solid lines), c) pARM also shows the shift to coarser magnetite at Montclus for altered samples (solid line), d) decay of an ARM acquired at 100 mT in Berriasian limestones shows a slight shift to higher fields for unaltered samples with the primary magnetization (Berrias) compared to samples where smectite is partially altered (Montclus) and samples where smectite has altered (Angles), e) Tithonian samples are consistent with a shift to coarse magnetite for inceasingly altered samples (Upper Tithonian at Montclus with smectite, Lower Tithonian at Montclus without smectite, Tithonian at Blegiers highly altered), f) the local Mg-chlorite anomalies (solid lines) do not indicate any differences in grain size spectrum to time equivalent rocks from Montclus (dashed lines).

and also with lateral variations across the basin. More anhysteretic remanence is acquired at all AF increments in samples with a high degree of smectite alteration, which is interpreted to reflect additional contribution to the remanence from a range of magnetite grain sizes.

When normalized to the value of maximum change, the samples that lack smectite and are strongly magnetized show a shift of their ARM-spectra to lower alternating fields (example: Montclus, Figure 2-8b) as expressed by a shift in the peak value and by relatively less acquisition of remanence at higher fields. Correspondingly, acquisition of pARM shows that samples without smectite also acquire relatively less remanence at higher alternating fields (Figure 2-8c). A shift in the peak value, however, is not observed, possibly due to the large pARM-window width at low alternating fields.

When comparing the ARM spectra from corresponding time intervals at different locations, samples with a higher degree of clay alteration and a well developed CRM are also shifted to lower alternating fields. For example, the Berriasian units from Angles (well developed CRM) indicate less pronounced changes at high alternating fields than Berriasian samples from Montclus (weakly developed CRM; Figure 2-8d), which is also consistent with the described vertical trends. The AF-decay of an ARM was used for analysis of the Berriasian and the significant noise in this method prevents any clear resolution of the peak value for altered samples. However, peak values of the two samples from Berrias (clays unaltered, primary magnetization) are at higher field strengths (Figure 2-8d). Tithonian units confirm the association between degree of alteration and shift to lower fields (Figure 2-8e); the sample from the Upper Tithonian of Montclus (abundant smectite, weak CRM) has its peak value at approximately 25 mT, whereas the lower Tithonian sample from Montclus (smectite altered, well developed CRM) peaks at 10 mT and the Tithonian

from Blegiers (high degree of clay alteration, well developed CRM) is shifted to an even lower field of 5 mT. Not shown are 2 samples from Espreaux, where the Oxfordian (altered) peaks at 10 mT and the Kimmeridgian (with smectite) at 20 mT. The two chlorite anomalies in the basin center exhibit no recognizable differences in ARM-spectra to their corresponding units at Montclus (Figure 2-8f).

The acquisition of ARM indicates the presence of a range of magnetite grain sizes. The differences in ARM-spectra of altered samples as compared to smectite-rich samples are interpreted as evidence for increased authigenesis of magnetite with illitization. The general shift to lower alternating fields with disappearance of smectite is interpreted to reflect addition of magnetite, especially of coarser sizes.

Magnetic hysteresis

Hysteresis loops close below magnetic fields of 300 mT (Figure 2-9a-d), which is consistent with magnetite being the dominant carrier of the magnetization in samples at all locations. Wasp-waistedness is not observed in any of the samples except at Blegiers (Figure 2-9d). The loops and measurements of backfield coercivities for many samples, especially those with significant smectite, are noisy. The majority of samples have ratios of saturation remanence (Mrs) to saturation magnetization (Ms) and coercivity of remanence (Hcr) to coercive force (Hc) within the PSD size range according to *Day et al.* [1977]. The samples from Berrias fall approximately along the line on a log/log plot (Figure 2-10) of coercivity ratios versus magnetization ratios which has previously been described to be indicative of primary magnetizations [*Channell and McCabe*, 1994]. Samples from the other areas plot above the primary magnetization line and some plot close to a line which has previously been interpreted to be characteristic of chemical remagnetization [e.g., *Channell and McCabe*, 1994].



Figure 2-9: Representative hysteresis loops of limestones from the Vocontian trough (corrected for paramagnetic slope). Loops a, b, and c close below 300 mT and do not exhibit wasp-waistedness and are representative for all samples except Blegiers, where the loops (d) also close below 300 mT but are wasp-waisted.



Figure 2-10: Representation of coercivity ratios versus magnetization ratios on double logarithmic axes. Lower diagonal: primary magnetizations from other studies fall along this line [*Channell and McCabe*, 1994]. Upper diagonal: chemically remagnetized rocks from other studies fall along this line [*Channell and McCabe*, 1994]. Mrs: saturation remanence, Ms: saturation magnetization, Hcr: coercivity of remanence, Hc: coercive force.
Among the altered samples, the strongly remagnetized samples have, on average, slightly higher magnetization ratios. These slight differences in hysteresis ratios with degree of smectite alteration could be indicative of variations in the magnetite content. Most samples have hysteresis ratios that are not in close proximity to the remagnetization line in the log/log plot. This might be caused by the general difficulty of determining reliable values. Alternatively, remagnetized samples from the Vocontian trough might not exhibit values comparable to other CRMs. The waspwaisted loops from Blegiers are interpreted to represent a mixture of magnetite grain sizes. The presence of a postfolding magnetization that could be a CRM might be responsible for the mixture.

Low temperature experiments

In all samples from Berrias, the thermal decay of SIRM acquired at 10 or 20 K shows a significant loss of remanence around 110 K (Figure 2-11a), i.e., the Verwey transition, which is indicative of the mineral magnetite. The Verwey transition is not apparent in samples from the eastern locations (e.g., Figure 2-11d), but is frequently observed in all other units where smectite has completely altered (e.g., Figure 2-11c). Samples from units where smectite has partially altered, however, show no Verwey transition (e.g., Figure 2-11b). Samples that exhibit the Verwey transition generally also display the magnetite isotropic point at around 120 K during cooling of an SIRM acquired at room temperature (Figure 2-11a,c).

Excluding the loss of remanence across the Verwey transition, the amount of remanence that relaxes to 300 K is a significant portion of the low temperature SIRM, and has been ascribed to relaxation of superparamagnetic (SP) magnetite [e.g., *Hunt et*



Figure 2-11: Representative thermal decay curves of SIRM acquired at 10 or 20 K and cooling of a room temperature SIRM. a) Berriasian of Berrias with Verwey transition and isotropic point for magnetite, b) sample with abundant smectite, c) sample with no smectite, and d) sample from highly altered eastern location.

al., 1995]. The relative proportion of total remanence lost between 10 and 300 K (excluding loss across the Verwey transition) ranges from 60% to 97%. Values for Berriasian samples from Berrias are the most consistent, with 66.7% of their magnetization carried by SP material (std. dev. 4.9%, N=3). Samples where the clays have partially altered are highly variable but have higher SP-proportions. For example, Berriasian samples contain, on average, 75% SP magnetite at Montclus and La Piarre (std. dev. 7.2%, N=3). Older units at Montclus have a mean value of 79.1% (std. dev. 8.9%, N=8). Samples from the eastern locations exhibit the highest values, with Berriasian samples generally carrying more than 90% of the low temperature remanence in SP grains (N=2).

Although the data are limited, the increase in relative abundance in SPmagnetite with degree of clay diagenesis is consistent with successive authigenesis of magnetite. The remanence at room temperature is also generally greater for altered samples, which is consistent with the other rock magnetic results.

GEOCHEMISTRY AND PETROGRAPHY

Strontium isotopes

Strontium isotopic ratios of micrite samples of different ages from various parts of the basin are generally consistent with previously reported data [*Katz et al.*, 1998]. The ⁸⁷Sr/⁸⁶Sr values are clearly within the range of coeval seawater [*Koepnick et al.*, 1985; 1990] for all limestones from representative times at all locations except at Blegiers. For example, Tithonian values of ⁸⁷Sr/⁸⁶Sr from four locations (mean = 0.70705) are within the range of coeval seawater (approximately 0.7069 to 0.70724). Two Tithonian samples from Blegiers, however, are higher with a mean of 0.70724 and fall at the upper limit of reference values for seawater.

The coeval ⁸⁷Sr/⁸⁶Sr values indicate that evolved, radiogenic fluids may have been present in veins but did not cause extensive alteration of the rocks. Values would be expected to be significantly more radiogenic than those of coeval seawater if the rocks had been altered by orogenic-type fluids [e.g., *Land and Prezbindowski*, 1981]. The fact that the strontium isotopic values at Blegiers are higher than other Tithonian values and are at the upper limit for coeval seawater could indicate some alteration by orogenic fluids.

Carbon and oxygen isotopes

Oxygen and carbon isotope values (Figure 2-12) are typical of carbonate sediment and marine limestone [*Hudson*, 1977] at all locations and time intervals. Limestones of older units from different sections where the CRM is well developed have values that are comparable to the respective overlying deposits. Oxygen values however, are slightly more negative for the eastern locations of Blegiers, Angles/Vergons and Chavailles.

Stable isotopes of carbon and oxygen are similar to unaltered limestones and therefore argue against extensive alteration by externally derived fluids. The slightly more negative oxygen isotopic values in the eastern sections could be connected to the higher degree of clay alteration [*Yeh and Savin*, 1977]. Extensive alteration by externally derived fluids, however, can not be postulated from this small variation.



Figure 2-12: Cross-plot of stable carbon and oxygen isotope values. Values are similar to reference numbers for marine limestones [Hudson, 1977].

Petrography

Observations of thin sections in plain and cross polarized transmitted light indicates that the lithology and diagenetic features vary little across the basin and across the Jurassic/ Cretaceous boundary. All units are characterized by abundant micrite with calpionnellids and spicules. Hairline fractures filled with sparry calcite are especially abundant in the Tithonian of Blegiers. Minor amounts of quartz, fossil fragments of various origins, and pyrite, some of which is altering to goethite, are also present.

The petrographic observations do not indicate significant differences in the diagenetic history between locations. The geochemical and petrographic observations do not support an interpretation of extensive alteration by orogenic-type fluids.

OROGENIC FLUIDS AND ASSOCIATIONS TO THE MAGNETIZATIONS

The Upper Oxfordian to Lower Cretaceous carbonates of the Vocontian trough are fractured and veins are not common, except in the Tithonian breccias at Blegiers. Field observations do not indicate extensive alteration of the rocks by fluids. Anomalous chloritization, which has been described for isolated areas and for the eastern locations in proximity to the Alps, could indicate some alteration by fluids [Levert and Ferry, 1988] around faults and above salt domes.

Two of the investigated sections in the basin center, where smectite has altered, are characterized by high amounts of chlorite (Kimmeridgian at La Roche and Oxfordian at Col de Soubeyrand). The paleomagnetic and geochemical results of these isolated chlorite anomalies do not differ from all other locations away from the Alps where smectite has altered. The specimens for the fold test at Pradelle come from a domal anticline that has been interpreted to be associated to salt tectonics [e.g., Levert and Ferry, 1988]. The paleomagnetic and geochemical results from this location are also similar to the other locations with the CRM.

When orogenic-type fluids migrate along fractures and faults and penetrate into the host rock, they can alter the magnetization and create a paleomagnetic halo [Elmore et al., 1993]. The NRM intensities as well as magnetic susceptibility do not vary away from a vein at Cote Mare (Figure 2-13a). The magnetic direction also remains constant with increasing distance from the vein, which indicates that the fluids that precipitated the calcite did not affect the magnetization (Table 2-1). The strontium isotope value for a sample from the calcite vein (0.70728) is higher than in the adjacent Oxfordian limestone (0.70691) at Cote Mare. The value of the limestone lies within the range of coeval seawater (range of coeval seawater: 0.7068 to 0.70715). Carbon and oxygen isotope values of the limestone are similar to unaltered limestones and to the other areas. Therefore, the host rock was not altered by the fluids that precipitated the calcite.

The gradual chlorite increase toward the Alps, which could indicate the action of orogenic-type fluids, is paralleled by the addition of postfolding magnetizations at Blegiers (normal polarity) and at Angles (reversed polarity). At Blegiers, a significant increase in NRM intensities is also observed and high paleotemperatures could have been caused by hot fluids. However, specimens from Angles and Chavailles, which are also located close to the Alps, do not have exceptional NRM values and specimens from Chavailles do not carry a stable postfolding magnetization. Also, the postfolding magnetizations at Blegiers and Angles could be TVRMs. A connection between the postfolding magnetizations and orogenic-type fluids, therefore, is problematic.



Figure 2-13: Various magnetic parameters as a function of distance from calcite veins. a) Neither NRM nor magnetic susceptibility vary in a systematic manner in an Oxfordian limestone in the vicinity of a vein near Cote Mare. b) Values of NRM, magnetic susceptibility, ARM, and IRM decrease toward a vein in samples from a Middle Jurassic limestone near Blegiers.

Additionally, oxygen, carbon, and strontium isotope values do not suggest pervasive alteration. The magnetic directions of specimens from Middle Jurassic limestones near Blegiers do not change in the vicinity of a meter scale calcite vein (Table 2-1). In fact, intensities of NRM, magnetic susceptibility, ARM, and IRM decrease towards the vein (Figure 2-13b) and it can be speculated that the fluids that caused calcite precipitation might have actually dissolved magnetite close to the vein (mineralogy based on IRM acquisition and thermal decay). The oxygen (and carbon) isotope values do not vary systematically in the vicinity of the vein, which argues against alteration by orogenic-type fluids.

DISCUSSION

A prefolding, secondary normal polarity magnetization is present throughout the Vocontian trough in all stratigraphic intervals where smectite has altered to illite. The direction of the magnetization is consistent with the Middle-Upper Cretaceous time for maximum burial in the Vocontian trough. A primary magnetization can not be identified away from Berrias, which is the only location where there is no evidence for burial alteration of the clays. The widespread, secondary magnetization is absent or weakly developed where smectite has not been completely altered. The paleomagnetic results of this study therefore indicate a connection between burial diagenetic alteration of smectite and the presence/absence of a widespread and stratigraphically pervasive secondary magnetization.

The magnetization is carried by magnetite and is interpreted to be a CRM based on low burial temperatures and high unblocking temperatures. Additional evidence for a chemical origin of the widespread magnetization comes from observed variations in the magnetite amount (ARM, IRM) and inferred grain-size spectrum (ARMacquisition) with the presence/absence of the secondary magnetization.

The results from the rock magnetic study are consistent with the proposed connection between authigenesis of magnetite and burial diagenesis of smectite. The amount of remanence carrying material, based on measurements of IRM, is less where smectite is abundant compared to altered parts of sections. The intensities of IRM of altered rocks are similar irrespective of stratigraphic age and location. The correlation between magnetite content, as estimated by ARM values, and alteration of smectite within the same time interval across the basin supports an interpretation of successive authigenesis of magnetite with degree of smectite conversion. Authigenesis of magnetite and the associated development of a CRM in the Vocontian trough are probably marked by contributions from a wide range of grain sizes, with an overall shift to coarser mean grain sizes in the remanence carrying fraction (based on ARM acquisition) and by higher SP abundance with increasing degree of clay diagenesis (based on low temperature results).

The strontium isotopic ratios of limestones are similar to coeval seawater. Carbon and oxygen isotope values are similar to unaltered limestones, and petrographic, as well as field observations, do not indicate extensive alteration by fluids. There is no association between the CRM and calcite veins and the geochemical signal in the limestones around the veins also does not support an interpretation of extensive alteration. The results suggest that the pervasive CRM was not caused by orogenic-type fluids.

Two of the eastern locations carry remanent magnetizations in addition to the normal polarity, prefolding CRM. The low stability reversed polarity magnetization at Angles is probably a postfolding magnetization, although other interpretations can not be excluded. The high stability reversed magnetization at Blegiers is secondary in origin and possibly was acquired before folding. It is approximately antipodal to the prefolding, secondary normal polarity magnetization and is interpreted to represent acquisition of the CRM through a reversal. The observed high values of NRM and the high strontium isotope ratios at Blegiers are accompanied by the presence of a strong postfolding, normal polarity magnetization. This component could be a CRM caused by orogenic-type fluids. The fact that there is a high degree of clay alteration that can not be explained by burial alone is consistent with such a connection. Oxygen and carbon isotope values, however, do not indicate extensive alteration by such fluids and the other eastern locations in this study do not carry the stable postfolding magnetization. Alternatively to a fluid related mechanism, increased diagenesis and high paleotemperatures in the east might be due to an unrecognized high geothermal gradient in the past or an increase in overburden as a result of thrust loading by a larger than expected areal extent of the alpine nappes. If correct, the postfolding component could be of thermoviscous origin.

Although a thermoviscous origin for the widespread, prefolding CRM has been excluded based on the time-temperature-unblocking relationship for single domain magnetite [*Pullaiah et al.*, 1975], a previous study has hypothesized that prolonged exposure to the earth's magnetic field during a long polarity interval might cause resetting of the magnetization if the magnetization is carried by PSD grains with certain lattice defects [*Moon and Merrill*, 1986]. This possibility has to be considered here, especially since hysteresis parameters are generally in the range of PSD magnetite. Additionally, the CRM is generally of normal polarity and might represent time-induced resetting during the Cretaceous normal polarity superchrone. However, the combined evidence from rock magnetic and paleomagnetic studies make this interpretation unlikely. Firstly, hysteresis ratios are probably a result of the effects of a mix of grain and domain sizes of magnetite. For example, ARM acquisition generally peaks at low fields but some remanence is acquired at high fields, which suggests the presence of a broad range of sizes from MD to SD magnetite. Also, a significant effect on hysteresis ratios must be expected from SP magnetite which is shown to be present by low temperature decay experiments. The ratios, therefore, might not represent true pseudo-single domain grains, but rather a mix of sizes. Secondly, a thermoviscous resetting during the Cretaceous normal polarity superchrone can not explain the possible acquisition of the CRM through a reversal at Blegiers. And thirdly, variations in magnetite content with degree of smectite diagenesis in the different sections argue for a chemical rather than a viscous origin of the magnetization.

The increase in magnetite amount in older units is interpreted to be associated to the degree of smectite alteration. Alternatively, the significant increase in IRM (or ARM as previously reported for Montclus and Espreaux; [*Katz et al.*, 1998]) downsection could theoretically be caused by unrecognized primary differences such as facies variations with age. In fact, *Hallam et al.* [1991] claim that smectite to illite variations might reflect climate differences across the Jurassic/Cretaceous boundary in Europe. This claim, however, would require drastic climate differences within the basin since smectite disappears in different stratigraphic intervals and the authors' view differs from diagenetic interpretations of all other clay studies of the Vocontian trough [e.g., *Deconinck*, 1987, *Levert and Ferry*, 1988]. The "climate interpretation" would also require a connection between magnetite content and climate, because magnetite abundance parallels lack of smectite.

The high values of ARM and IRM for rocks from Berrias are problematic as is the fact that none of the locations away from Berrias carry a primary magnetization. An unrecognized dissolution process prior to authigenesis of magnetite at all other locations or slight facies variations in the Berriasian between Berrias and the other locations might be responsible for the differences. The absence of the Verwey transition in partially altered limestones away from Berrias could support the suggested explanations. Although we could not recognize significant petrographic differences, Berrias was located closer to the basin margin during the Berriasian. Some of the magnetic differences in the Berriasian between Berrias and other locations could therefore be due to slight facies variations.

The paleomagnetic, rock magnetic, and geochemical results of this study support a connection between burial diagenesis of smectite to illite and the presence of a widespread CRM and rule out an orogenic fluid connection in the Vocontian trough of SE-France. The burial diagenetic conversion of smectite to illite could be responsible for the presence of widespread and pervasive CRMs elsewhere. Burial is probably the most common cause for illitization of smectite, although in some areas, mechanisms such as tectonic loading during intense deformation and migration of hydrothermal fluids during orogeny have also been described as potential agents for localized diagenesis of smectite [e.g., *Chamley*, 1989]. Although some CRMs may be related to fluid triggered conversion of smectite, many of the Late Paleozoic rocks that have previously been interpreted to be associated to orogenic-type fluids have probably been buried sufficiently to cause illitization without the presence of externally derived fluids. If illitization of smectite can cause magnetite authigenesis during burial, the origin of these widespread CRMs might need to be re-evaluated.

Maturation and migration of hydrocarbons has been linked to diagenesis of smectite [e.g., *Chamley*, 1989]. Smectite adsorbs organic compounds and the major stage of smectite-illite ordering occurs fairly shortly before oil generation and migration [e.g., *Chamley*, 1989]. Therefore, the empirical association between CRMs carried by magnetite and organic matter maturation [e.g., *Banerjee et al.*, 1997] or migration [e.g., *Katz et al.*, 1996] should be tested for a possible genetic connection with clay diagenesis.

An association between burial diagenesis of smectite and development of a CRM might also be used to determine the timing and duration of clay diagenesis. The hypothesis of a "punctuated", instantaneous clay diagenesis as envisioned by *Morton* [1985] might be tested with paleomagnetic studies. If illitization and magnetite authigenesis occur over long periods of time as hypothesized, for example, by *Yeh and Savin* [1977], the age of the magnetization (and polarity) should vary with burial history.

CONCLUSION

A widespread, stratigraphically pervasive CRM characterizes Jurassic and Cretaceous limestones of the Vocontian trough in SE-France where smectite has altered during burial. The CRM is absent or weakly developed where significant smectite is still present. The rock magnetic results are consistent with authigenesis of magnetite during burial diagenesis of smectite. Geochemical results rule out a connection between orogenic-type fluids and the widespread CRM. The results of this study suggest that burial diagenesis of smectite is a viable mechanism for the development of pervasive chemical remagnetization elsewhere.

If the connection between burial diagenesis of smectite and CRM can be confirmed, paleomagnetic methods could be used to date the diagenesis of clays and rock magnetic measurements could serve as rapid and inexpensive tools for the detection of smectite alteration and associated processes. Understanding the processes that are responsible for the common and widespread occurrences of CRMs could also be of importance to predicting the presence of primary magnetization and to paleomagnetic studies that are concerned with tectonic issues.

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CHAPTER 3

Connections between burial diagenesis of smectite and magnetization in clastic sediments of the Green River Basin, Wyoming

ABSTRACT

The objective of this study is to test the burial diagenetic conversion of smectite to illite as a mechanism for magnetite authigenesis in a core of clastic rocks from the Green River Basin in Wyoming. Changes in the demagnetization of the natural remanent magnetization (NRM) and in rock magnetic parameters that are related to the magnetic content occur across the depth interval where smectite alters to illite. The NRM is carried by magnetite in samples from all depth intervals. Magnetization ratios as determined from hysteresis experiments are higher where smectite has altered, which is consistent with previously reported trends for chemically remagnetized rocks. The magnetite grain size spectrum as estimated with partial anhysteretic remanence shows a slight shift to coarser grains downsection. Results from low temperature experiments indicate that the Verwey transition is suppressed and the contribution from superparamagnetic magnetite (SP) is higher in samples where smectite has altered. The intensity of NRM is slightly higher in specimens from greater depths. Magnetic susceptibility, on the other hand, has the opposite trend which is inconsistent with the other rock magnetic measurements. Specimens from smectite-rich units carry no stable remanence, but thermal and alternating field (AF) demagnetization removes a stable component in most specimens from greater depths where smectite has altered to illite. The magnetization in illite-rich specimens could be a chemical remanent magnetization (CRM). Most of the trends observed in this study are similar to those reported previously in limestone units of SE-France, where magnetite authigenesis occurs during the burial diagenetic conversion of smectite.

INTRODUCTION

Chemical remanent magnetizations (CRMs) are a common occurrence in sedimentary rocks. A frequently cited mechanism for pervasive CRMs is magnetite authigenesis caused by orogenic-type fluids (e.g., Oliver, 1992, and many others). Some recent studies, however have pointed out inconsistencies with the so called "orogenic fluid hypothesis" (e.g., Elmore et al., 1993, Fruit et al., 1995, Banerjee et al., 1997), such as a lack of geochemical evidence for a pervasive presence of externally derived fluids. We have proposed that burial diagenetic processes are an alternative mechanism that could explain a large number of widespread or pervasive CRMs (Katz et al., 1998a, b). For example, the diagenetic conversion of smectite to illite during burial can result in the release of iron ions and other elements (Boles & Franks, 1979) and laboratory studies show that conversion of smectite to illite can result in authigenesis of magnetite (Hirt et al., 1993). If magnetite authigenesis occurs during the burial diagenetic conversion of smectite, the newly formed magnetite might be capable of recording the magnetic field at the time of its formation.

We have recently demonstrated that burial diagenesis of smectite is a feasible mechanism for the creation of a CRM in carbonates of the Vocontian trough of SE-France (Katz et al., 1998a, b). Additional studies are needed to test whether this clay diagenesis association provides a potential mechanism for some of the frequently encountered pervasive CRMs found in different geologic settings. Of particular importance is to test whether burial diagenesis of smectite is a viable mechanism of chemical remagnetization in clastic sedimentary rocks. Burial diagenesis of smectite is a common process (Chamley, 1989) and if it can be shown that it is associated with the development of CRMs in different settings, current theories about the origin of widespread CRMs may have to be re-evaluated. Additionally, if the clay diagenesis/CRM connection is confirmed, magnetic measurements could be utilized to date diagenesis of clays and could also serve as rapid, inexpensive tools for the detection of diagenesis of clays and associated processes.

In this study, the magnetic signatures of Cretaceous/Tertiary clastic rocks from a core which was retrieved in the Green River Basin in Wyoming and contains the smectite-illite transition (Pollastro & Barker, 1986) are compared. It is tested whether differences in the degree of smectite diagenesis correlate to changes in the magnetic characteristics in the clastic sediments. The changes in magnetic signature are also compared to those that were previously observed in the carbonates of the Vocontian trough (Katz et al., 1998a, b).

BACKGROUND

Some of the early work on burial diagenesis of clays has described the rapid disappearance of smectite at depths in excess of 2 to 3.5 km in cores (e.g., Hower et al., 1976). A study in Wyoming on a core penetrating Upper Cretaceous and Tertiary sedimentary rocks of the Green River Basin (Wagon Wheel #1 well) shows a steep increase in illite-percentage in mixed-layer illite/smectite at depths between approximately 7000 and 7400 ft or 2100 and 2250 m (Pollastro & Barker, 1986). The deposits that cover the interval where smectite alters rapidly downsection are nonmarine sandstones and shales (Pollastro & Barker, 1986). These authors also note that calcite and quartz veins fill fractures caused by the Neogene uplift of the Pinedale Anticline. The authors observed hydrocarbon-bearing fluid inclusions in veins at depths from 7850 to 17000 ft.

METHODS

Samples were collected from multiple sites across the smectite alteration depth and were analyzed for their rock magnetic and paleomagnetic signatures. A continuous sampling was not possible, because of incomplete coring of the Wagon Wheel #1 well (sampled intervals: 5030-5075 feet, 7040-7138 feet, 7340-7400 feet, 8028-8142 feet, 8920-8944 feet). Samples were cut to standard lengths and weight normalized bulk magnetic susceptibility was measured on a Sapphire Instrument prior to further processing. The natural remanent magnetizations (NRMs) were measured on a 2G three-axes cryogenic magnetometer located in a magnetically shielded room. Specimens were subsequently stepwise demagnetized by alternating field (AF) demagnetization up to 160 mT in a 2G Automated Degaussing System or thermal demagnetization up to 700°C in a magnetically shielded Schonstedt TSD-1 oven. The resulting decay pattern was displayed in orthogonal projections (Zijderveld, 1967) and line segments with mean angular deviations of less than 10° were identified prior to performing principal component analyses (Kirschvink, 1980).

Acquisition of isothermal remanent magnetizations (IRMs) produced by an impulse magnetizer and subsequent thermal demagnetization of three perpendicular IRMs with fields of 120, 400, and 1300 mT (Lowrie, 1990) were recorded from different sites to gain information on the magnetic mineralogy.

Hysteresis experiments and measurements of coercivity of remanence were performed on Micromag Alternating Gradient Force Magnetometers at the University of Utah and at the Institute for Rock Magnetism, University of Minnesota. The magnetic grain size distribution was estimated (Jackson et al., 1988) by acquisition experiments of partial anhysteretic remanence (pARM) and the thermal decay of a low temperature saturation IRM (SIRM) acquired at 10 K was measured on a Magnetic Property Measurement System (MPMS) for representative samples at the Institute for Rock Magnetism in Minnesota. Total anhysteretic remanence (ARM) was acquired for multiple samples in a direct magnetic field of 0.1 mT and an alternating field of 100 mT. Subsequently, the samples were exposed to a direct magnetic field of 300 mT and the IRM was measured.

Thin sections of representative samples from various depth intervals were examined using a polarizing microscope in reflected and transmitted light.

ROCK MAGNETIC RESULTS AND

INTERPRETATIONS

The sedimentary rocks that were examined in this study are mainly light gray, fine to medium sandstones and siltstones. No significant differences in the nonmarine clastic units at depths between 7000 and 7400 feet can be recognized petrographically for the investigated intervals.

Smectite-rich samples (Figure 3-1A, B, left), and samples where smectite has altered (Figure 3-1C, D, left) all exhibit a rapid rise in the acquisition of an isothermal remanence below 300 mT which is indicative of a low coercivity mineral such as magnetite. At direct magnetic fields in excess of 300 mT, the magnetization of the samples does not increase significantly, which indicates that high coercivity phases are probably not contributing to the magnetization. The thermal decay of IRM imposed along three perpendicular axes in each sample confirms that the major part of the remanence is carried by a low coercivity phase. The magnetization on the "low coercivity axis" is removed at approximately 580°C in all samples (Figure 3-1), which is indicative of magnetite. The IRM acquisition and subsequent thermal decay of the triaxial IRM indicate that magnetite is the dominant magnetic mineral in all samples and the magnetic mineralogy does not change across the clay transition.





Figure 3-1: Acquisition of IRM (left) and subsequent thermal decay of triaxial IRM (right) for representative samples. Samples from sites with abundant smectite (A, B) and from sites where smectite has altered (C, D) acquire most of their remanence below 300 mT. The low coercivity contributions (diamonds) decay by approximately 580°C, which is indicative of magnetite, and the medium (squares) and high coercivity (triangles) contributions are low. Numbers above figures indicate depth below surface.

Pronounced differences, on the other hand, exist in rock magnetic characteristics between specimens from depths where abundant smectite is present and those where smectite has largely altered to illite. Magnetic hysteresis properties were acquired for several samples across the depth interval where smectite has altered (Figure 3-2). Although the data are generally noisy, the shapes of magnetic hysteresis loops do not drastically differ. However, the following observations can be made: the ratios of saturation remanence to saturation magnetization are distinct between the two clay zones, with ratios being generally higher for samples from greater depths (Figure 3-2). Elevated magnetization ratios at greater depths could be related to previously observed high ratios that have been related to chemical remagnetization (Channell &



Figure 3-2: Hysteresis parameters for samples with abundant smectite (crosses) and for samples where smectite has altered (circles). Magnetization ratios are generally higher where smectite has altered. Mrs: saturation remanence, Ms: saturation magnetization, Hcr: coercivity of remanence, Hc: coercive force.

McCabe, 1994). Magnetization ratios from other studies with primary magnetizations have been found to be generally lower than for locations with a CRM (Channell & McCabe, 1994). Coercivity ratios are between 5 and 30 (with one exception) and paramagnetic slopes do not vary in a systematic manner.

The acquisition of pARM also shows differences between smectite-rich and smectite-poor samples (Figure 3-3). The samples from greater depths acquire more anhysteretic remanence at low alternating fields and appear slightly shifted to lower fields compared to smectite-rich samples. A shift to lower alternating fields is interpreted to indicate more relative abundance of coarse magnetite grains (e.g., Jackson et al., 1988).

Results of thermal decay of SIRMs acquired at low temperatures demonstrate a less pronounced Verwey transition at greater depths (Figure 3-4). These results may indicate oxidation of magnetite (Özdemir et al., 1993) which suppresses the Verwey transition in samples where smectite has altered.

Intensities of ARM and IRM also show a different pattern above and below the smectite alteration depth (Figure 3-5). Values of ARM and IRM vary less between sites at greater depths. The IRM intensities do not differ significantly between the two depth zones, but ARM appears slightly higher in the shallower sites. One possible interpretation is that the shallower sites carry, on average, more fine magnetite in the single domain size range and the deeper sites have a stronger contribution to the total remanence from coarse magnetite.

Bulk magnetic susceptibility is, on average, lower at greater depth, the major decrease occurs over a narrow depth interval where smectite rapidly disappears (Figure 3-6). Lower values of magnetic susceptibility at greater depth could indicate differences in the relative contributions from different magnetite grain sizes or lower magnetite concentrations.

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Figure 3-3: Acquisition of pARM for samples with abundant smectite (closed circles) and for samples where smectite has altered (open circles). Samples from greater depths are slightly shifted to lower alternating fields which is interpreted to indicate a shift to coarser magnetite.



Figure 3-4: Thermal decay curves of a low temperature SIRM acquired at 10 K for samples with abundant smectite (line) and for samples where smectite has altered (lines with symbols). Samples from greater depths show a suppressed Verwey transition and more relative abundance of SP magnetite.



Figure 3-5: Intensities of ARM and IRM as a function of depth in the core. The IRM intensities (solid circles) are similar for samples with abundant smectite and for samples where smectite has altered, but ARM intensities (crosses) are slightly higher at shallower depths.



Figure 3-6: Bulk magnetic susceptibility as a function of depth in the core. Lower values of magnetic susceptibility at greater depth could indicate differences in the relative contributions from different magnetite grain sizes or lower magnetite concentrations.

PALEOMAGNETIC RESULTS AND INTERPRETATIONS

The intensities of the natural remanent magnetization (NRM) vary slightly across the depth where smectite disappears downsection (Figure 3-7). The values are, on average, higher at depths in excess of 7300 ft (approximately 2200 m) but the differences may not be significant. However, results of alternating field and thermal demagnetization of the NRM exhibit significant differences across the smectite/illite boundary. Only two of the samples from the shallower sites contain a stable magnetic direction (N=2/22) at alternating fields exceeding 20 mT or temperatures above 280°C (Figure 3-8A, B). In contrast, those from altered sites at greater depths (N=16/22) commonly display a stable magnetization that decays to 120 mT or 580°C (Figure 3-8C, D). The declination is variable due to rotations induced during drilling of the core and the inclination is mostly steep positive (Figure 3-9). Five of the specimens from three sites where the up and down directions.

A thermoviscous origin of the magnetization can be excluded since high laboratory unblocking temperatures (Pullaiah et al., 1975) can not be explained by low burial temperatures below 150°C at 2 to 3 km (Pollastro & Barker, 1986) and the magnetization could be primary in origin or a CRM. The magnetization which is removed at low alternating fields and temperatures is interpreted to be a viscous remanent magnetization.



Figure 3-7: Intensity of NRM as a function of depth in the core. Values are, on average, slightly higher at greater depths where smectite has altered.


Figure 3-8: Representative orthogonal projections of alternating field and thermal demagnetization of the NRM. Closed symbols: horizontal projection, open symbols: vertical projection, number in feet indicates depth in the core. Specimens with abundant smectite contain no stable magnetic direction (N=2/22) at alternating fields exceeding 20 mT or temperatures above 280°C (A, B). In contrast, those from altered sites at greater depths commonly display a stable magnetization (N=16/22) that decays to 120 mT or 580°C (C, D).



Figure 3-9: Stereographic projection of component directions. The declination is variable due to rotations induced during drilling of the core and the inclination is mostly steep positive (closed symbols). Five of the specimens from two sites where the up and down directions of the core pieces might have been confused, have apparent steep negative directions (open symbols).

DISCUSSION AND CONCLUSIONS

Magnetite is the only magnetic phase present at all depth intervals across the clay transition. Variations in magnetization ratios as determined by magnetic hysteresis experiments indicate differences in the magnetite content. As previously stated, the elevated magnetization ratios where smectite has altered could be related to previously observed variations with chemical remagnetization (Channell & McCabe, 1994). The results of pARM acquisition are also consistent with the clay diagenesis/magnetite authigenesis connection but are more subtle. The pARM spectrum is generally shifted to lower fields for remagnetized samples, which is interpreted to reflect addition of magnetite mainly of coarser grain sizes. The differences in the thermal decay behavior of SIRM acquired at low temperature are consistent with an onset of magnetite authigenesis downsection, as well. Samples with low smectite content have poorly developed Verwey transitions. A possible decrease in single domain (SD) magnetite downsection (as interpreted from ARM intensities) which is accompanied by a possible increase in coarse magnetite (IRM does not decrease) could reflect the growth of magnetite. This interpretation is also consistent with the observed shift to coarser magnetite with depth from pARM spectra.

The decrease in magnetic susceptibility downsection also suggests changes in the magnetite content. However, magnetic susceptibility is primarily a measure of ultrafine grained magnetite and the interpretation of a stronger contribution from SPmagnetite in deeper samples as derived from low temperature experiments is therefore not consistent with the susceptibility data. Alternatively, magnetic susceptibility could be dominated by coarser magnetite if little SP-magnetite is present. However, the postulated shift to coarser magnetite (e.g., pARM spectra) and the observed abundance of SP-magnetite (low temperature measurements) argue against such an interpretation. Therefore, the magnetic susceptibility data is in contradiction to all other measurements.

Except for magnetic susceptibility values, the rock magnetic data are consistent with authigenesis of magnetite during smectite alteration. The paleomagnetic results also support an association with clay diagenesis. A stable magnetization occurs downsection with the disappearance of smectite. The onset of the stable magnetization could indicate the creation of a CRM carried by magnetite with alteration of smectite.

The results of this study on clastic rocks from the Green River Basin are similar to previously reported observations from Mesozoic carbonates of the Vocontian trough of SE-France (Katz et al., 1998a, b). Magnetite is the dominant magnetic phase above and below the depth where smectite disappears in both studies. Magnetization ratios are higher, the Verwey transition is suppressed, and the magnetic grain size spectrum is shifted to coarser sizes in samples from greater depths where smectite has altered. Also, specimens from sites where smectite is abundant are weakly magnetized whereas those from greater depths are characterized by a stable remanence in this study and in limestones from SE-France.

Although the results from this study support a connection between burial diagenesis of clays and magnetite authigenesis, the following issues can not be resolved. A CRM origin is open to question because no tests that would constrain the timing of acquisition of the magnetization can be performed. However, rock magnetic differences between samples from smectite-rich sites that carry no stable remanence and from sites where smectite has mostly altered and the magnetization is well developed support the interpretation of a CRM. Alternatively, the differences in the magnetic signature with smectite abundance could be caused by facies variations with depth, although obvious petrographic differences can not be identified between the

sampled intervals. Also, the decrease in magnetic susceptibility downsection is enigmatic and does not conform with other rock magnetic observations.

In summary, paleomagnetic and rock magnetic evidence in clastic rocks of the Green River Basin supports an association between degree of burial diagenesis of smectite and a remagnetization that appears to be chemical in origin. The paleomagnetic and rock magnetic changes that are observed in this study are generally parallel to those from carbonates of SE-France (Katz et al., 1998a, b). The association between burial diagenesis of smectite and magnetite authigenesis in carbonates, as well as clastic sedimentary rocks of different time intervals and from different locations might suggest that such a connection occurs frequently and could be responsible for some of the widespread, pervasive CRMs in other rocks.

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SYNTHESIS

The results of this study indicate that widespread or pervasive chemical remanent magnetizations (CRMs) can form as a consequence of the burial diagenetic conversion of smectite to illite. Paleomagnetic, rock magnetic, and geochemical evidence supports the CRM-clay diagenesis connection in carbonates of the Vocontian trough in SE-France and rules out another commonly cited mechanism, i.e. orogenictype fluids. A widespread and pervasive CRM is present throughout the basin where smectite has converted during burial. The CRM is weakly developed or absent where smectite conversion is incomplete, and the only location with a primary magnetization is identified where the clays were not altered during burial. The magnetite content and mean grain size increase downsection and within time intervals across the basin with the degree of smectite alteration. Also, based on geochemical evidence, the widespread CRM is not associated to orogenic-type fluids. Paleomagnetic and rock magnetic trends with clay diagenesis from clastics of the Green River Basin in Wyoming are generally similar to those of the Vocontian trough and are also consistent with magnetite authigenesis and the development of a CRM. Among the benefits in understanding the mechanisms for the development of widespread CRMs are the potential to date diagenetic events paleomagnetically.







IMAGE EVALUATION TEST TARGET (QA-3)







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Rochester, NY 14609 USA Phone: 716/482-0300 Fax: 716/288-5989

