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Continuous production of nanosized magnetite through low grade burial

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[1] Geological processes, such as burial, can lead to remagnetization in rocks due to neofomed magnetic minerals that have passed a critical volume, called blocking volume. In this study, we designed a heating experiment for claystones obtained from the Paris Basin (France), in the 50–130°C temperature range, in order to simulate <4 km burial remagnetization. At a given temperature, remanence increased rapidly within a couple of days and stabilized afterward. There was a positive relation between the experimental temperature and the obtained remanence. Remanence was determined to be carried equally by stable chemical remanent magnetization and unstable thermo-viscous remanent magnetization. By assuming that magnetite formed during the experiment, we interpreted the increase of chemical remanent magnetization and the increase of thermo-viscous remanent magnetization as the continuous growth of the >20 nm and ~20 nm minerals respectively. This result led us to propose a conceptual model of nucleation-and-growth process of magnetite during low grade burial from ~2 to ~4 km depth. Ultrafine magnetite (≤ 20 nm) was predominant over single domain magnetite (>20 nm) for <4 km depth. Transposed to natural conditions, our heating steps experiment suggested that claystone-type rocks are remagnetized during burial. For temperatures higher than 200°C, the extrapolation of our results indicated that burial remagnetization, due to the chemical remanent magnetization, might be larger than the natural remanent magnetization.

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Theme: Magnetism From Atomic to Planetary Scales: Physical Principles and Interdisciplinary Applications in Geosciences and Planetary Sciences

1. Introduction

[2] During early diagenesis, in many anoxic sedimentary environments, sulfate reduction by bacterial activity and pyritization of detrital Fe-bearing minerals (e.g., iron oxides) lead to a decrease in magnetic properties of the sediments [e.g., Karlin, 1990; Leslie et al., 1990]. This is related to a dissolution process, with finest-grained magnetic minerals being preferentially dissolved [e.g., Bloemendal et al., 1993]. The iron sulfides formed [e.g., Roberts and Weaver, 2005] are then altered at higher depth/temperature to produce new magnetic minerals [e.g., Suk et al., 1990, 1993; Brothers et al., 1996].

[3] The neoformation of magnetic grains during burial promotes a new magnetization in rocks, called CRM (chemical remanent magnetization). CRMs are widely recognized in unmetamorphosed sedimentary rocks in basins and thrust-and-fold belts [e.g., McElhinny and Opdyke, 1973; McCabe and Elmore, 1989; Weil and Van der Voo, 2002]. However, the origin of these CRMs is still debated. Evoked mechanisms are early diagenesis [Roberts and Weaver, 2005; Rowan and Roberts, 2005], geochemical degradation along methane horizons [Larrasoana et al., 2007], smectite-to-illite transformation [Lu et al., 1990, 1991; Katz et al., 2000], pyrite alteration [Brothers et al., 1996; Gillett, 2003], fluid circulation [Oliver, 1986; Katz et al., 1998; Evans et al., 2000; Elmore et al., 2001], deformation [Lewchuk et al., 2003], and the maturation of organic matter [Banerjee et al., 1997]. Laboratory heating experiments from 95 to 250°C on claystones [Cairanne et al., 2004; Moreau et al., 2005; Aubourg et al., 2008; Aubourg and Pozzi, 2010; Aubourg et al., 2012] have demonstrated that temperature alone leads to the rapid formation of minute amounts of magnetic minerals. For experiments performed at 95°C, the thermal demagnetization of the laboratory CRM produced under heating of Dogger Opalinus claystones (Mont Terri, Jura, Switzerland) and Callovo-Oxfordian Bure claystones (Paris Basin, France) showed that the maximum unblocking temperature lies within 500–600°C. This indicates that CRM resides essentially in magnetite [Aubourg et al., 2008; Aubourg

and Pozzi, 2010]. In addition, the CRM thermal demagnetization of the Bure claystone displayed a break-in-slope at ~200–250°C corresponding to an iron sulfide which was identified as pyrrhotite [Aubourg and Pozzi, 2010]. For experiments carried out at 150°C and 250°C [Cairanne et al., 2004; Moreau et al., 2005], the CRM thermal demagnetization of Toarcian shales from Paris Basin indicated also that CRM resided essentially in magnetite. Some hematite was also detected, and it was attributed to the oxidation of magnetite. Moreau et al. [2005] have suggested that iron sulfides (greigite and others non identified) were also formed at 150°C. When extrapolating the results of these experiments to natural settings, it is likely that burial promotes the formation of magnetic minerals as a result of temperature elevation. Therefore, temperature elevation may be a simple explanation for the creation of CRMs.

[4] For this study, we repeated the experimental procedure proposed by Aubourg et al. [2008], from 50 to 130°C, in order to simulate the effect of stepwise burial on remanent magnetization in Bure claystones. We focused on the neoformation of magnetic minerals at temperature as low as 50°C. We aimed to elucidate the gap between subsurface conditions (<20 m) where iron sulfides are produced [Rowan et al., 2009] and ~100°C where magnetite is produced [Aubourg et al., 2008]. We showed that nanosized magnetite was continuously produced by 50°C. Eventually, we proposed a conceptual model of magnetite nucleation-and-growth during low grade burial from ~2 to ~4 km depth.

2. Samples and Methods

2.1. Samples Description

[5] The samples used for this work are claystones, extensively studied in the context of their possible use for radioactive waste storage. The claystones came from the oblique core EST-211 (849.19–849.69 m MD, i.e., ~530 m vertical depth), drilled by the Agence Nationale pour la gestion des Déchets Radioactifs (the French National Radioactive Waste Management Agency), in the Jurassic

Table 1. Natural Remanent Magnetization, NRM_{AF80} Obtained After 80 mT AF Demagnetization, $\text{CRM}_{2\text{mT}}$ and $\text{TVRM}_{2\text{mT}}$ Created During the Heating Experiment Under a 2 mT Magnetic Field for Samples Used in This Study^a

Sample	Mass (g)	NRM ($\mu\text{Am}^2/\text{kg}$)	NRM_{AF80} ($\mu\text{Am}^2/\text{kg}$)	$\text{CRM}_{2\text{mT}}$ ($\mu\text{Am}^2/\text{kg}$)				$\text{TVRM}_{2\text{mT}}$ ($\mu\text{Am}^2/\text{kg}$)			
				50	70	80	130	50	70	80	130
1	1.097	9.75	0.12	0.69				0.65			
2	0.990	0.21	0.01	0.39				0.44			
3	1.144	0.14	0.01	0.39	0.63			0.47	0.76		
4	1.613	0.13	0.02	0.37	0.56			0.44	0.86		
5	1.197	0.52	0.01	0.4	0.62	0.73		0.45	0.87	0.79	
6	1.156	0.25	0.01	0.43	0.69	0.82		0.50	0.94	0.86	
7	1.791	0.18	0.01	0.40	0.64	0.72	1.15	0.35	0.85	0.8	1.51
8	1.262	0.01	0.01	0.38	0.62	0.74	2.27	0.48	0.94	0.77	1.33
9	1.038	0.13	0.01	0.34	0.58	0.68	2.17	0.46	0.79	0.76	1.97
10	1.370	4.39	0.05	0.74	1.11	1.08	1.93	0.43	1.34	0.13	1.38
11	0.892	0.2	0.01	0.67	1.02	1.01	1.59	0.33	1.17	0.92	1.66
12	1.200	0.22	0.01	0.38	0.53	0.71	2.05	0.44	0.91	0.77	1.43
13	0.955	0.24	0.02	0.59	1.14	1.35	1.11	0.76	1.41	0.73	0.58
14	1.022	0.15	0.01	0.59	1.02	1.07	2.92	0.56	1.08	1.09	1.43
Mean		1.18 (0.22)	0.02	0.48	0.76	0.89	1.90	0.48	1.0	0.88	1.41
SD		2.71 (0.11)	0.03	0.14	0.23	0.22	0.61	0.11	0.21	0.17	0.39

^aThe mean natural remanent magnetization (NRM) in italic corresponds to the mean NRM of the samples which do not present extreme values (1 and 10 show high values and 8 shows very low value). CRM = chemical remanent magnetization; TVRM = thermo-viscous remanent magnetization.

(Callovo-Oxfordian) Formation of the Paris Basin near the Bure locality, in France.

[6] The clay content of the core varies between 35 and 60% [Gaucher *et al.*, 2004]. Remaining core components are calcite and silt. The claystones contain less than 2% pyrite, and up to 0.9% TOC (Total Organic Carbon). The organic matter is associated with framboidal pyrite and bioclasts.

[7] In Bure claystones, the vitrinite reflectance (R_o) observation indicates R_o values range between 0.3 and 0.4% [Blaise *et al.*, 2011]. This suggests that Bure claystones underwent low burial temperatures near 40°C [Landais and Elie, 1999; Blaise *et al.*, 2011]. The magnetic properties of these claystones have also been studied. Low field magnetic susceptibility (χ) varies between 10 to 194 μSI , and natural remanent magnetization varies between ~ 0.02 and ~ 0.54 $\mu\text{Am}^2/\text{kg}$ [Esteban *et al.*, 2006]. Iron oxides ((titano)-maghemite or -magnetite) and iron sulfide (greigite, pyrrhotite, pyrite) occurrences have been documented [Esteban *et al.*, 2006; Aubourg and Pozzi, 2010]. In addition, Aubourg and Pozzi [2010] identified goethite and stoichiometric magnetite through the monitoring of magnetic properties below room temperature.

2.2. Methods

[8] Prior to the heating experiment, we measured the magnetic susceptibility χ and the natural remanent magnetization (NRM) of the claystones. Fragments

of Bure claystones ($\sim 1\text{--}2$ g) were glued into small glass flasks (one fragment per flask). The bottles were then filled with glass wool and plugged in order to create a quasi-confined atmosphere. A total of 14 bottles were prepared, and separated into two sets (A and B; odd and even samples in Table 1 respectively). We focused on the remanent magnetization created by newly formed grains. To highlight this phenomenon, the NRM of the samples was initially demagnetized using an 80 mT alternating field. We performed a heating experiment with progressively increasing temperatures of $T_{\text{exp}} = 50, 70, 80, 120,$ and 130°C in order to reproduce step-wise burial. The bottles were placed in an oven at a given temperature for approximately 25 days (20 days for 50°C , and ~ 10 days for 120 and 130°C). During heating, the Earth's magnetic field was removed and an upward magnetic field of 2 mT was applied. The resulting remanence, R , was measured repeatedly, after cooling down the samples to room temperature in a 2 mT field (~ 2 h long). The remanence R is expressed as follows: $R = \text{NRM}_{\text{AF80}} + \text{IRM}_{2\text{mT}} + \text{CRM}_{2\text{mT}} + \text{TVRM}_{2\text{mT}}$ where NRM_{AF80} is the NRM after 80 mT of AF demagnetization, $\text{IRM}_{2\text{mT}}$ is the isothermal remanent magnetization imparted at 2 mT, $\text{CRM}_{2\text{mT}}$ is the chemical remanent magnetization acquired by neofomed magnetic minerals above their blocking volume at 2 mT, and $\text{TVRM}_{2\text{mT}}$ is a thermo-viscous remanent magnetization carried by former and neofomed magnetic minerals at 2 mT, both SP-SD (superparamagnetic-single domain)

and MD (multidomain) grains. NRM_{AF80} and $\text{IRM}_{2\text{mT}}$ are one to two orders in magnitude less than $\text{CRM}_{2\text{mT}}$ and $\text{TVRM}_{2\text{mT}}$, and are, therefore, neglected [Aubourg *et al.*, 2008]. Finally, the measured remanence can be expressed as: $R \sim \text{CRM}_{2\text{mT}} + \text{TVRM}_{2\text{mT}}$. The TVRM is by essence time-dependent and unstable. The initial TVRM may be carried by a set of preexisting magnetic minerals such as magnetite and greigite. Given the duration of the laboratory experiment (tens of days), it is likely that any increase of TVRM may be related to the input of ultrafine neofomed grains, with size below or near the blocking volume, where relaxation time changes abruptly from laboratory to geological times. At the end of the heating step, we left the samples at temperature in a null magnetic field for at least 2 days in order to remove $\text{TVRM}_{2\text{mT}}$. The samples were then cooled down in a null magnetic field (<100 nT). The procedure constitutes what we refer to as the TVRM test [Aubourg *et al.*, 2008]. It is important to note that previous studies never reported evidence of thermal remanent magnetization (TRM) that can be imprinted when cooling down our samples from T_{exp} to room temperature [Cairanne *et al.*, 2004; Aubourg and Pozzi, 2010]. After the TVRM test, the measured magnetization is a $\text{CRM}_{2\text{mT}}$ for a given T_{exp} temperature. It is possible that a small amount of the $\text{CRM}_{2\text{mT}}$ measured is actually a VRM (viscous remanent magnetization), since our samples were cooled down for a time less than the acquisition time [Dunlop, 1973; Moskowitz, 1985]. We repeated the same experimental procedure for higher temperatures. For each temperature step, two samples (one from set A and one from set B) were removed from the experiment after the TVRM test and left in a null field. The TVRM tests were performed for $T_{\text{exp}} = 50, 70, 80,$ and 130°C . At the end of the experiment, we obtained 4 $\text{CRM}_{2\text{mT}}$ values.

[9] The laboratory series of heating steps were different from natural conditions. Indeed, burial conditions were not satisfied. In our experiments, the heating rate ($1^\circ\text{C}/\text{min}$) was very different from the natural heating rate which is on the order of $5^\circ\text{C}/\text{My}$ [Sweeney and Burnham, 1990]. Pressure was not considered and the experiments were performed at atmospheric pressure (0.1 MPa).

[10] The sample remanence was measured with a 2G cryogenic SQUID magnetometer at Ecole Normale Supérieure, Paris, France. In order to identify the mineral responsible for the $\text{CRM}_{2\text{mT}}$, the set B samples were thermally demagnetized from $T_{\text{dem}} = 100^\circ\text{C}$ to 600°C using 20 or 50°C steps. Set A

samples were crushed and sealed in a gelcap and were measured with a Magnetic Properties Measurement System (MPMS) at the Institut de Physique du Globe de Paris, France. An initial low-temperature saturation isothermal remanent magnetization (LT-SIRM) was acquired at 10 K using a magnetic field of 2.5 T. We then monitored the LT-SIRM demagnetization curve from 10 to 300 K.

3. Results

[11] Natural Bure claystones studied have low magnetic susceptibility ($<200 \mu\text{SI}$) and low remanence at saturation ($\sim 60 \mu\text{Am}^2/\text{kg}$). This indicates a low concentration of ferromagnetic grains. The variability of magnetic susceptibility and saturation remanence is about tens of percent between adjacent samples. The NRM is generally weak ($\sim 1 \mu\text{Am}^2/\text{kg}$) (Table 1). However, one Bure sample presented higher NRM ($\sim 10 \mu\text{Am}^2/\text{kg}$). The NRM after demagnetization at 80 mT was two orders of magnitude less than the initial NRM ($\sim 0.02 \mu\text{Am}^2/\text{kg}$) (Table 1). However, one sample presented a higher value, by one order of magnitude ($\sim 0.1 \mu\text{Am}^2/\text{kg}$).

[12] Figure 1 represents the evolution of the mean remanence R for the different T_{exp} temperatures, for reproducing stepwise burial. At $T_{\text{exp}} = 50^\circ\text{C}$, we observed, in the first days of the experiment, that remanence increased rapidly by two orders of magnitude. The remanence then progressively reached a quasi-plateau with a mean moment of $1.2 \pm 0.2 \mu\text{Am}^2/\text{kg}$. No significant changes occurred in the plateau. The last measured remanence ($t = 20$ days) at $T_{\text{exp}} = 50^\circ\text{C}$ was less than expected. We do not have any explanation for this particular finding. Given the weak remanence measured, it is possible that some pollution has altered the measure. When the temperature was increased to $T_{\text{exp}} = 70^\circ\text{C}$, remanence again, very rapidly, reached a quasi-plateau at a higher mean value ($1.6 \pm 0.3 \mu\text{Am}^2/\text{kg}$) than that of the previous heating step. The general pattern was the same for the other heating steps. The mean moments were $1.7 \pm 0.1, 2.1 \pm 0.6,$ and $3.1 \pm 0.4 \mu\text{Am}^2/\text{kg}$ for $T_{\text{exp}} = 80, 120,$ and 130°C , respectively. The difference between the remanence acquired at 70 and at 80°C (a 10°C difference) was not very distinct (1.6 versus $1.8 \mu\text{Am}^2/\text{kg}$ for 70 and 80°C , respectively).

[13] While increasing temperature T_{exp} , the $\text{CRM}_{2\text{mT}}$ displayed a consistent increase from 0.5 ± 0.1 to $1.9 \pm 0.6 \mu\text{Am}^2/\text{kg}$ (Figure 2 and Table 1). The $\text{TVRM}_{2\text{mT}}$ was on the same order of magnitude as

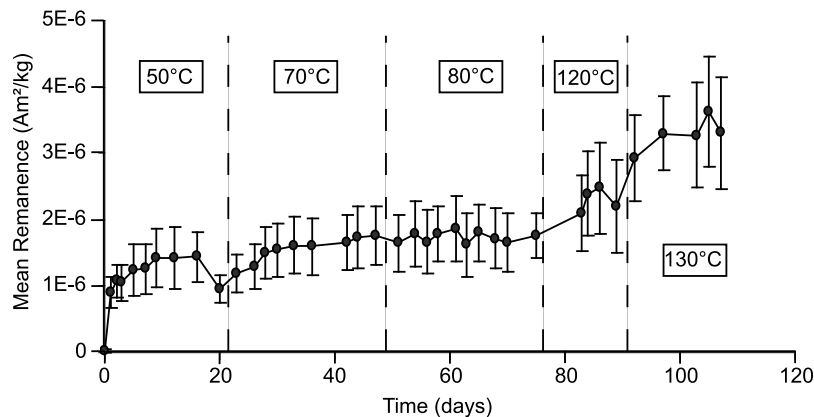


Figure 1. Evolution of the mean remanent magnetization R during the heating steps experiment from $T_{\text{exp}} = 50$ to 130°C . Standard deviation ($\pm 1\sigma$) is also reported.

the $\text{CRM}_{2\text{mT}}$, and increased from 0.5 ± 0.1 to $1.4 \pm 0.4 \mu\text{Am}^2/\text{kg}$ (Figure 2 and Table 1). At the latter temperature, the $\text{CRM}_{2\text{mT}}$ portion was predominant in the total remanence of the samples.

[14] To characterize the unblocking temperature spectrum of $\text{CRM}_{2\text{mT}}$, samples from set B were then heated from $T_{\text{dem}} = 100$ to 650°C (Figure 3a). Remanent magnetization values from $T_{\text{dem}} > 400^\circ\text{C}$ are not represented on the figure, as a result of remagnetization effect due to heating. These remagnetizations are common in Bure claystones and are likely due to alteration of iron sulfides [e.g., *Aubourg and Pozzi, 2010*]. Distinct unblocking spectra of $\text{CRM}_{2\text{mT}}$ were observed. We focused on the following two parameters: (1) the percentage of $\text{CRM}_{2\text{mT}}$ loss at the thermal demagnetization temperature of $T_{\text{dem}} = 150^\circ\text{C}$ (higher than the maximum heating step temperature T_{exp} utilized) and (2) the maximum unblocking temperature (T_{UB}). At the demagnetization temperature $T_{\text{dem}} = 150^\circ\text{C}$, we observed a variable decrease of the CRM depending on the temperature of the experiment T_{exp} that simulates burial (Figure 3a). When the temperature of experiment T_{exp} is 50°C , there was a fall of 80% of the $\text{CRM}_{2\text{mT}}$. When the temperature of experiment T_{exp} is 130°C , the $\text{CRM}_{2\text{mT}}$ shows almost no decrease (-1%). The higher CRM acquisition temperature, the more resistant it is to thermal demagnetization. Note the plateau of the demagnetization curve up to $T_{\text{dem}} = 150^\circ\text{C}$ for the sample heated at 130°C . This indicates that the remanence is essentially a CRM, and that residual TVRM after the TVRM test is negligible.

[15] T_{UB} evolved toward higher values from 280 to $>400^\circ\text{C}$ for $T_{\text{exp}} = 50$ to 130°C , respectively (Figure 3b). At $T_{\text{dem}} = 400^\circ\text{C}$, 12% of the $\text{CRM}_{2\text{mT}}$ acquired at $T_{\text{exp}} = 130^\circ\text{C}$ remained. By extrapolating

the thermal demagnetization curve for heated samples at $T_{\text{exp}} = 130^\circ\text{C}$, a maximum T_{UB} near 500°C was found (Figure 3a).

[16] We measured the Low Temperature SIRM for set A samples from 10 to 300 K. We observed a decrease of LT-SIRM from 10 to 150 K, with a more abrupt decrease from 10 to 50 K (Figure 4a). LT-SIRM curves were shifted downward with an increasing heating step temperature. Here, again, we could use a parameter to characterize the evolution of LT-SIRM, with PM expressed as $\text{PM} = (\text{LT-SIRM}_{10\text{K}} - \text{LT-SIRM}_{35\text{K}}) / \text{LT-SIRM}_{10\text{K}}$. This parameter was first defined by *Aubourg and Pozzi [2010]* as a mean to represent the relative contribution of pyrrhotite and magnetite. The PM parameter may also integrate grain size (SP and SD

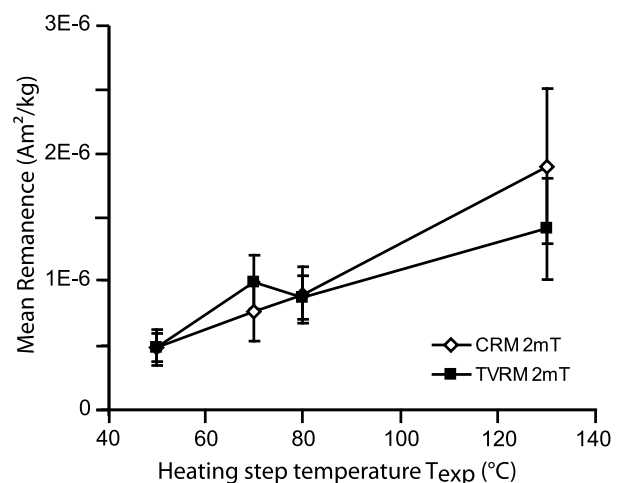


Figure 2. Evolution of the mean $\text{CRM}_{2\text{mT}}$ and mean $\text{TVRM}_{2\text{mT}}$ created during the heating experiment from $T_{\text{exp}} = 50$ to 130°C . Standard deviation ($\pm 1\sigma$) is also reported.

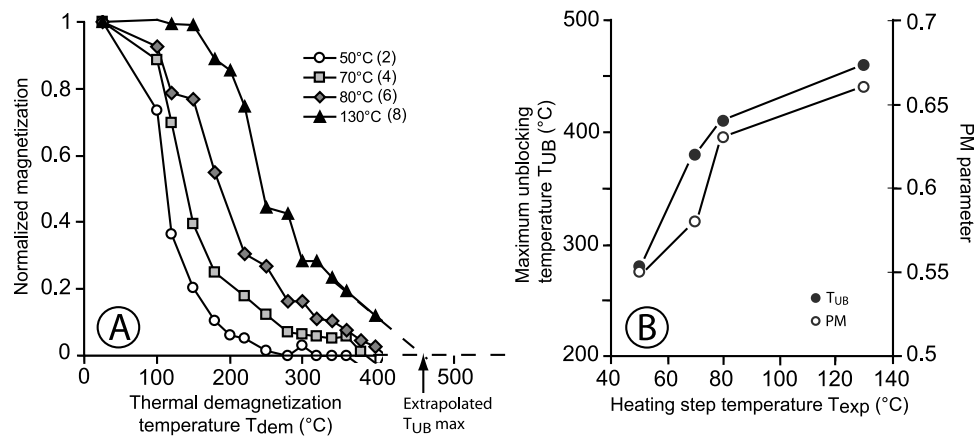


Figure 3. (a) Thermal demagnetization curves of the CRM_{2mT} created during the heating steps experiment from $T_{exp} = 50$ to 130°C . The extrapolated maximum unblocking temperature T_{UB} is also shown. Sample names are reported in brackets. (b) Evolution of the maximum unblocking temperature T_{UB} and PM parameter as a function of the heating step temperature T_{exp} .

sizes). The PM values increased from 0.55 to 0.66 for $T_{exp} = 50$ to 130°C in heated samples, respectively, and PM was 0.34 for natural samples (Figure 3b).

4. Discussion

4.1. The Nature of Neoformed Magnetic Minerals

[17] Our study demonstrates the formation of magnetic minerals in Bure claystones for heating temperatures as low as 50°C , which corresponds to

the burial temperature encountered at approximately 2 km. Stepwise burial-like heating experiment revealed the following: (1) an increase in the CRM_{2mT} and the TVRM_{2mT} consistent with an increasing T_{exp} temperature; and (2) an evolution of rock magnetism parameters.

[18] The process behind neoformed magnetic grains is likely fast on the geological time scale since the remanence acquired during heating claystones rapidly attained a quasi-plateau (Figure 1). Our heating steps experiment was not long-lasting enough to determine the long-term trend of the plateau [e.g., *Aubourg et al.*, 2008]. However, we assumed that

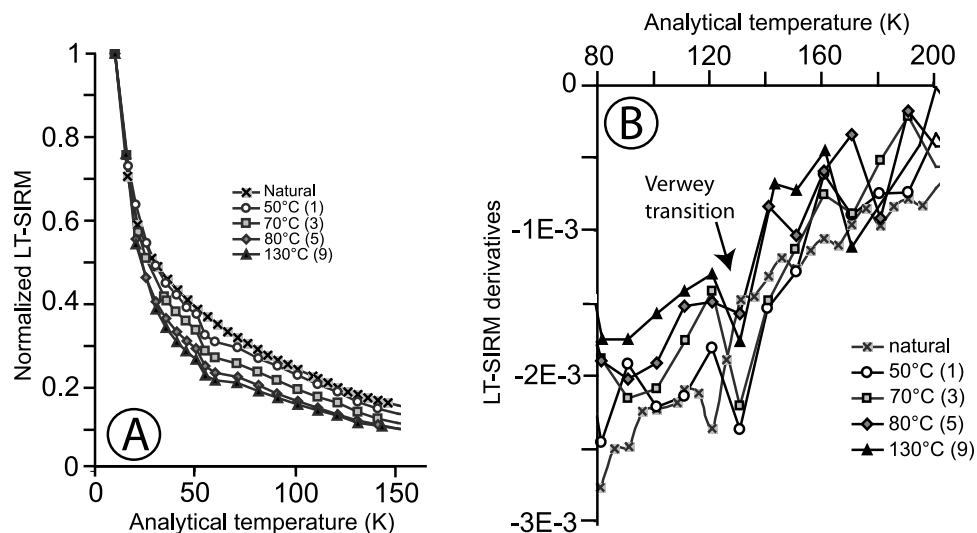


Figure 4. (a) LT-SIRM evolution for the set A samples from 10 to 150 K. Natural sample LT-SIRM is represented on the figure for reference. (b) LT-SIRM first derivatives from 80 to 200 K displaying a break-in-slope at about 130 K. Samples names are reported in brackets.

most of the magnetic minerals were rapidly formed upon heating. One motivation of our work was to simulate the effect of an increasing burial temperature. In this respect, we observed different plateaus for stepwise heating from $T_{\text{exp}} = 50$ to 130°C (Figure 1). Extending this result to burial suggests that magnetic minerals form continuously and rapidly attain a steady state when the burial temperature increases.

[19] During burial, neofomed magnetic minerals acquire a remanence. Our heating experiment indicated that remanence (R) was partly carried by a TVRM_{2mT} and by a CRM_{2mT} for ten days of heating. During heating in a magnetic field, magnetic minerals that have passed the blocking volume will acquire a CRM. Conversely, when testing TVRM, where heating took place under a zero magnetic field, magnetic minerals that have passed the blocking volume did not acquire a CRM. When further heated in a magnetic field, these newly formed magnetic minerals will not acquire CRM, but only a TVRM. It is concluded that the CRM underestimates the newly formed magnetic minerals above the blocking volume. In addition, the CRM_{2mT} has an unknown fraction of the viscous magnetization as the TVRM test does not last long enough to remove all TVRM. Examination of the demagnetization curves of CRM_{2mT} (Figure 3a) shows that the decrease in the remanence is less than 30% at 100°C . This indicates that the contribution of viscous magnetization, if any, is marginal.

[20] The strength of the CRM_{2mT} obtained between $T_{\text{exp}} = 50$ and 130°C increased from 0.5 to $1.9 \mu\text{Am}^2/\text{kg}$, which is the strength of the NRM (Figure 2 and Table 1). The CRM_{2mT} increase suggests either a higher concentration of one kind of neofomed magnetic mineral at 130°C , or an assemblage of different magnetic species. To discriminate between these two hypotheses, we will use the results from the thermal demagnetization of CRM_{2mT}.

[21] When thermally demagnetizing the CRM_{2mT}, we did not observe any clear break-in-slope in the curves (Figure 3a). Thus, in this study, there is not net evidence of an assemblage of two magnetic mineral species. This differs from the result of Aubourg and Pozzi [2010] which showed a more pronounced break-in-slope at $\sim 200\text{--}250^{\circ}\text{C}$ when demagnetizing a laboratory CRM imparted at 95°C . They further suggested that this break-in-slope is due to neofomed pyrrhotite.

[22] In addition, we observed a regular shift in the maximum unblocking temperature (T_{UB}); the lowest burial-like T_{exp} temperature, with the lowest

being T_{UB} (Figure 3b). The shift can be attributed to the grain size distribution of magnetic minerals; the coarser the grain, the higher the T_{UB} [Stokking and Tauxe, 1987; Dunlop and Özdemir, 1997]. Magnetic grains become harder to thermally demagnetize as the T_{exp} increases. For simplicity, we consider in the following that magnetite is formed during the heating experiment. The reality is probably more complex and other magnetic minerals could be formed simultaneously. Aubourg and Pozzi [2010] suggested the formation of magnetite and pyrrhotite during heating at 95°C . It is also likely that other iron oxides such as maghemite or goethite are formed following the oxidation of magnetite or alteration of iron sulfides. Nevertheless, the extrapolated maximum unblocking temperature (T_{UB}) near 500°C supports the occurrence of fine magnetite (Figure 3a). The presence of neofomed magnetite is found in accordance with conclusions from other heating experiments in claystones at temperature from 95 to 250°C [Cairanne et al., 2004; Moreau et al., 2005; Aubourg et al., 2008]. The ZFC curves of heated samples and natural sample display a tiny break-in-slope at ~ 130 K illustrating by first derivative (Figure 4b). This may indicate the presence of the Verwey transition of magnetite, though the transition temperature is quite higher [e.g., Muxworthy and McClelland, 2000]. If some oxidized magnetite was present, then the temperature of the Verwey transition would be lower and the transition amplitude would be affected by the oxidation [e.g., Özdemir et al., 1993]. Maghemite does not display any transition at low temperature and thus it is unlikely that maghemite is present in our samples [Özdemir and Dunlop, 2010]. Therefore, the most robust indication of neofomed magnetite is the observed range of unblocking temperature $T_{\text{UB}} < 500^{\circ}\text{C}$ of the acquired CRM. We assume that magnetite formed continuously during our heating experiments. The boundary between superparamagnetic (SP) and single domain (SD) magnetite grains has been estimated to be 20 nm for $T = 300$ K [Dunlop and Özdemir, 1997]. The CRM_{2mT} may be a proxy for grains > 20 nm, while the TVRM_{2mT} essentially marks the contribution of ultrafine grains from SP-SD boundary (~ 20 nm). The evolution of CRM_{2mT} demagnetization curves (Figure 3a) suggests that grain size distribution evolves to larger grains. Following Stokking and Tauxe [1987], we used the Néel equation, which links grain size and the blocking temperature as follows:

$$V = (kT/K) \ln(C\tau)$$

where V is the grain volume (cm^3), k is the Boltzmann's constant (1.38×10^{-16} erg/K), T is the blocking temperature (Kelvin), K is the anisotropy constant of the magnetic mineral of interest, C is a frequency factor (1×10^9), and τ is the relaxation time (the duration of sample demagnetization). The grain diameter (d) can be expressed as $d(\text{cm}) = [\ln(C\tau)(6k/\pi K)T]^{1/3}$. Using $K = -1.35 \times 10^5$ erg/ cm^3 (the magnetocrystalline anisotropy of magnetite), and $\tau = 3600$ s, we obtained maximum grain sizes for magnetites ranging from 30 to 35 nanometers, for T_{UB} from 250 (523 K) to 500°C (773 K).

[23] On the other hand, the intensity of $\text{TVRM}_{2\text{mT}}$ increased threefold from 50 to 130°C, respectively (Figure 2 and Table 1). The $\text{TVRM}_{2\text{mT}}$ is partly carried by neofomed ultrafine magnetic minerals and partly carried by former magnetic minerals. We assume that magnetite produced during the heating experiment contributes to a significant proportion of the $\text{TVRM}_{2\text{mT}}$. The increase of $\text{TVRM}_{2\text{mT}}$ is thus likely attributable to the increasing concentration of magnetite from SP-SD boundary. This observation is confirmed by the evolution of the PM parameter, as deduced from the low-temperature analysis (Figure 3a). This parameter may be a proxy for SP minerals that acquired a remanence at low temperature, but not at room temperature [Özdemir *et al.*, 1993]. The regular PM increased from $T_{\text{exp}} = 50$ to 130°C suggesting that SP magnetic grains, including SP magnetite, are formed continuously throughout the burial-like T_{exp} temperature elevation (Figure 3a). To identify the magnetic minerals responsible for the PM evolution, we performed measurements for field- dependence and frequency- dependence of AC susceptibility from 5 to 300 K. No difference was observed between the natural and heated samples, as a result of the high paramagnetic input of the samples (see auxiliary material).¹

4.2. Conceptual Model of Magnetite Nucleation-and-Growth During Burial

[24] In the following, we propose a nucleation-and-growth model based on our experimental heating and some natural observations (Figure 5). We consider in this scenario only the formation of magnetite upon burial.

[25] The heating steps experiment of Bure clays-tones showed the formation of nanosized magnetite

both above and below the blocking volume V_b , as it was suggested by the joint relationship of the $\text{CRM}_{2\text{mT}}$ and $\text{TVRM}_{2\text{mT}}$ (Figure 2). In addition, the evolution of the PM parameter suggested that the concentration of SP magnetite increased (Figure 3b). This trend is interpreted as a continuous nucleation process: the higher the burial temperature, the higher the amount of SP magnetite. On the other hand, the evolution of the maximum unblocking temperatures T_{UB} indicated that the average size of SD magnetite increased regularly with increasing temperature (Figure 3b). Transposing these results to burial, we propose that once magnetite is nucleated, it grows regularly under the influence of temperature elevation.

[26] By assuming a nucleation-and-growth process, we emphasize a burial model from surface to ~4 km depth. Nevertheless, the whole magnetic assemblage of the buried sedimentary rocks is not only constitutive of neofomed magnetic minerals. Some detrital magnetic grains may still be present. During sediment deposition, a large amount of detrital magnetic minerals are dissolved due to the bacterial activity [e.g., Bloemendal *et al.*, 1993]. This corresponds to a resetting of the detrital magnetic signal carried by iron oxides. Iron sulfides, mostly greigite, are then produced [Canfield and Berner, 1987; Roberts and Weaver, 2005; Rowan *et al.*, 2009]. Greigite, once formed, is very stable and preserved in the sediments [e.g., Roberts *et al.*, 2011].

[27] Below the “bacterial horizon,” up to ~2 km depth, we have no information on the processes of formation or alteration of magnetic minerals. Roberts *et al.* [2011] and references therein suggested that greigite is still present for temperature <280°C and thus it is likely that greigite is preserved from subsurface to ~4 km depth or more. It is probable that other magnetic minerals are present. Recently, Abdelmalak *et al.* [2012] reported the occurrence of the oxyhydroxide goethite in weakly buried clays-tones (<5 km depth) and subsequently proposed that this mineral is neofomed. Our conceptual model of magnetite nucleation-and-growth starts from ~2 km depth which corresponds to the first heating step temperature we performed (50°C).

[28] From ~2 km depth, magnetite grains are formed both SP and SD sizes. We define the PM parameter as a proxy of the ratio $C_{\text{SP}}/(C_{\text{SP}} + C_{\text{SD}})$ where C_{SP} and C_{SD} represent the concentrations of SP and SD grains respectively. In this case, a PM value near 1 means that C_{SP} overcomes C_{SD} . On the contrary, a PM value near 0 means that C_{SD} overcomes C_{SP} . As PM is defined as the loss of

¹Auxiliary materials are available in the HTML. doi:10.1029/2012GC004104.

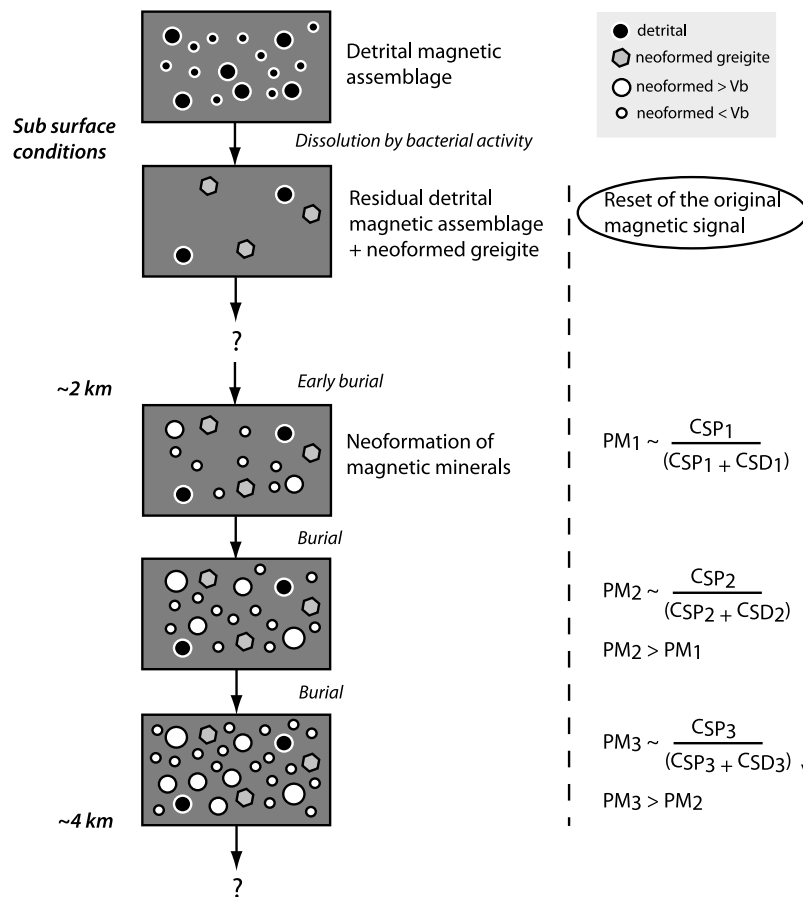


Figure 5. Simplified conceptual model of magnetite nucleation-and-growth during low grade burial <4 km. C_{SP} and C_{SD} represent the concentrations of SP and SD grains respectively. See text for discussion.

remanence from 10 to 35 K, it represents the proportion of grains with size fine enough to be thermally unblocked at 35 K. According to our heating experiment results, the successive PM values will increase with burial (Figure 3b). Superparamagnetic minerals are continuously formed with burial, at a rate faster than they can grow to larger size (i.e., size large enough to unblock at >35 K).

[29] Interestingly, the increase of the PM values during burial/temperature has been previously observed by *Aubourg and Pozzi* [2010] for burial temperature ranging from 50 to ~90°C. They proposed a model of the PM evolution with temperature: a PM-Up branch characterized by an increase of the PM values from ~50°C until a maximum value PM max is reached at about ~90°C and then a PM-Down branch (decrease of the PM values) from 90 to 250°C. The PM-Up branch was obtained from natural observations and the PM-Down from heating experiments with gold capsules. Some discrepancies occurred between our study and the *Aubourg and Pozzi* [2010] paper. First, we observed

a regularly increase of PM values from $T_{exp} = 50$ to 130°C, while *Aubourg and Pozzi* [2010] reported an increase up to ~90°C. The PM max value at ~90°C and the PM decrease (PM-Down) are not observed in our study. Second, the range of PM values we obtained from our heating experiment (0.55 to 0.65) differs from the PM values reported by *Aubourg and Pozzi* [2010] for burial (~0.6 to ~1). These differences suggest that our experimental heating protocol does not recreate the natural conditions.

[30] Below ~4 km depth, we do not know about the nucleation-and-growth process of magnetite, but experimental heating results at 150°C [*Moreau et al.*, 2005] and at 250°C [*Cairanne et al.*, 2004] showed that magnetite is still forming.

4.3. Burial and Magnetic Overprint

[31] The chemical remanent magnetization produced by the neoformed SD magnetites may influence the

natural remanent magnetization of buried sedimentary rocks.

[32] The magnetic field imparted during the experiment was 2 mT, which is 40:1 of the Earth's magnetic field. Assuming that chemical remanent magnetization is proportional to the strength of the magnetic field [Stokking and Tauxe, 1990; Pick and Tauxe, 1991], we calculated the expected CRM_{EMF} obtained at 50 μ T, the strength of the Earth's magnetic field (EMF). Therefore, the CRM_{EMF} ranged from 0.01 to 0.05 μ Am²/kg, which is approximately 1:100 to 1:10 of the NRM of our samples (Table 1). Considering individual values, for example the CRM_{EMF} of 0.05 μ Am²/kg for T_{exp} = 130°C and a NRM of 0.2 μ Am²/kg, it appears that the ratio of CRM/NRM would be 1:4. Thus, for moderate burial conditions (~4 km and more), the CRM could be a significant fraction of the NRM.

[33] Assuming that the CRM_{EMF} evolution linearly increased with increasing temperature, as suggested from our experiments (Figure 2), that would lead to a temperature where CRM_{EMF} \geq NRM. When extrapolating our results (Figure 2), this temperature would be >220°C with a weak NRM (~0.1 μ Am²/kg). Beyond 220°C, the neoformed magnetic grain remanence would be predominant over the preexisting grain remanence. Our experimental data are corroborated by natural examples from mid-Jurassic claystones from the Vocontian trough in the occidental French Alps. In the French Alps a pervasive remagnetization has been documented [Aubourg and Chabert-Pelline, 1999; Katz et al., 2000; Cairanne et al., 2002]. Mid-Jurassic claystones witnessed burial temperatures between 200 and 250°C during the late Cretaceous [Guilhaumou et al., 1996]. Interestingly, this pervasive remagnetization, with a magnitude on the order of 1 μ Am²/kg, completely overlapped the primary NRM. This likely indicates that long-term burial heating promotes larger magnetic overprints in claystones than our experimental results do.

[34] Thus burial remagnetization could have important consequences for the interpretation of remanence in sedimentary rocks, and, in particular, for magnetostratigraphy as discussed recently by Aubourg et al. [2012].

5. Conclusion

[35] For the first time, we have shown that laboratory heating by 50°C triggers the formation of magnetic minerals. The rock magnetic analyses

indicated that a fraction of neoformed magnetic minerals are ultrafine magnetites both superparamagnetic (<20 nm) and single domain (>20 nm) sizes, reaching a maximum size of ~35 nm. We noticed an evolution of the magnetite sizes with increasing heating step temperature. We have hence proposed a conceptual model of magnetite nucleation-and-growth during low grade burial (<4 km depth). Ultrafine magnetites are continuously produced with burial. The concentrations of superparamagnetic and single domain neoformed magnetites change with burial, the ratio between them as well. By assuming a reset of the detrital magnetic signal and a continuous process throughout burial, the latter key result, if quantified, may be used to determine the maximum depth/temperature experienced by sedimentary rocks.

[36] On the other hand, the growth and neoformation of magnetic minerals in sedimentary rocks produces a secondary magnetization that may overprint the initial magnetization during low grade burial. Our results suggested that this overprinting may occur from ~220°C (~8 km depth).

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