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Magnetic carriers and remanence mechanisms in magnetite-poor sediments of Pleistocene age, southern North Sea margin

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ABSTRACT: Pleistocene sedimentary sequences in the East Anglian region of Britain record both major and minor climatic oscillations, and the impact of isostatic and eustatic variations. Intensively studied in terms of their lithology and biostratigraphy, the sequences have been difficult to place in an absolute timeframe. Dating and correlation by magnetostratigraphy has been attempted over a number of years. However, these sediments are difficult to date by palaeomagnetic means because they are poor in detrital magnetite, are subject to post-depositional deformation and diagenesis, and have unknown rates of sedimentation. Determining whether their natural remanence magnetisation (NRM) directions are reliable thus requires information on the mode and timing of remanence acquisition. Here, we apply palaeomagnetic, rock magnetic and mineralogical analyses to identify the NRM carriers in these sediments and hence their palaeomagnetic reliability. Within oxidised fluvial sediments (the Kesgrave Formation), the magnetic carriers appear to be relict magnetic minerals (ferrian ilmenites, chromites, haematite and goethite), which sometimes carry a reliable primary depositional remanence (DRM) but often an overprinting viscous (time-varying) remanence (VRM). Within some reduced marine and intertidal sediments (within the Crag basin), the iron sulphide, greigite, has been found to carry a reliable, 'syn'-depositional chemical remanence (CRM). In all the sediments, magnetic inclusions within silicates are abundant, are significant for the mineral magnetic signal but contribute little to any recoverable palaeomagnetic information. Copyright © 2005 John Wiley & Sons, Ltd.

KEYWORDS: palaeomagnetism; remanence acquisition; British Pleistocene sequences.

Introduction

Palaeomagnetic analysis of freshwater and marginal-sea sediments, where sedimentation rates are often orders of magnitude higher than in deep-sea sediments, has the potential to provide detailed information not only on the timing and duration of changes in the Earth's magnetic field but also on changes in palaeoenvironments and climate. However, the reliability of the palaeomagnetic record acquired by such sediments depends on the timing of remanence acquisition (i.e. 'syn'-depositionally or significantly post-depositionally) and the stability through time of the recording media (i.e. the constituent magnetic minerals). Depending on the source of the detrital mineral grains, which controls the detrital magnetic mineralogy and magnetic grain size, and any subsequent diagenetic activity, the components of the NRM of such sediments may be of either primary or secondary origin and acquired as a (post-) depositional remanence (PDRM), a chemical remanence (CRM) or a viscous remanence (VRM). To assess the fidelity of the palaeomagnetic archives of freshwater and nearshore sediments, information on the nature of the magnetic carriers and their remanence mechanisms is needed. Here, we examine the palaeomagnetic behaviour of fluvial and nearshore sediments around the southern margins of the North Sea basin (specifically, the onshore suite of Early and Middle Pleistocene sediments of the East Anglian region of Britain). Intensive lithological and biological analyses of these discontinuous sedimentary sequences have shown that they record major and minor oscillations in Quaternary climate. Construction of a reliable magnetostratigraphic framework for the Plio-Pleistocene sediments of the region is increasingly important in deciphering tectonic, climatic and eustatic impacts, and in light of current controversies over the number of climate stages recorded by these sequences and their correlation with the marine oxygen isotope record (e.g. Hamblin *et al.*, 2000; Preece and Parfitt, 2000), and proposals (e.g. Pillans, 2004)

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to move the Plio-Pleistocene boundary to the Gauss/Matuyama transition at 2.6 Ma, from its presently accepted position at 1.65 Ma (i.e. close to the end of the Olduvai normal polarity Sub-chron). The deep-sea oxygen isotope record indicates that climatic oscillations during the Plio- Pleistocene interval were lower in amplitude and of shorter duration than in the later Pleistocene (post ~780 ka). Identification, correlation and dating of these numerous lower-amplitude climatic events by conventional litho- and biostratigraphy is difficult, especially given the fragmentary nature of the southern North Sea sequences. Palaeomagnetic analysis offers the sole means of absolutely dating these sequences and correlating them with the deep-sea oxygen isotope record.

Recent work has shown that some of the sedimentary units can be reliably dated using reversal magnetostratigraphy (e.g. Hallam and Maher, 1994; Maher and Hallam, 2005). However, compared with the more complete Pleistocene sediments in the Netherlands (van Montfrans, 1971), the East Anglian sediments display very few reversed polarity intervals. This suggests: (a) there are major hiatuses in the British record (Zagwijn, 1975) and/or (b) there has been post-depositional overprinting during the Brunhes normal Chron (Hallam, 1995). Using a range of palaeomagnetic, rock magnetic and complementary mineralogical techniques (X-ray diffraction, optical and scanning and transmission electron microscopy), we investigate here the mineralogy and remanence mechanisms of the magnetic carriers isolated by magnetic extraction from these magnetite-poor sediments, and hence assess the fidelity of the palaeomagnetic records they contain.

Geological setting

As described in Maher and Hallam (2005), the Pleistocene sequences of East Anglia consist of a series of discontinuous, unconsolidated sedimentary units, overlying ‘bedrock’ of Cretaceous chalk and Tertiary sands and clays. These pre-Anglian/Elsterian Pleistocene sediments fall into two main groups. First, sediments deposited at the western margin of the contemporary southern North Sea are predominantly shallow marine (‘crag’) and intertidal in origin. Thompson (1991) note that, with respect to palaeomagnetic data, shallow marine deposits are among the most persistently troublesome materials. In such sediments, with a rapid sedimentation rate, high organic input and anoxic conditions, iron- and sulphate-reducing conditions can both result in the dissolution of detrital magnetic iron oxides. Second, across the southern and eastern areas of East Anglia, fluvial sediments, deposited by an ancestor of the River Thames, are found (Fig. 1). The absence of non-durable clasts within these gravelly units suggests prolonged fluvial abrasion; any magnetic minerals present are far-travelled and mostly reworked.

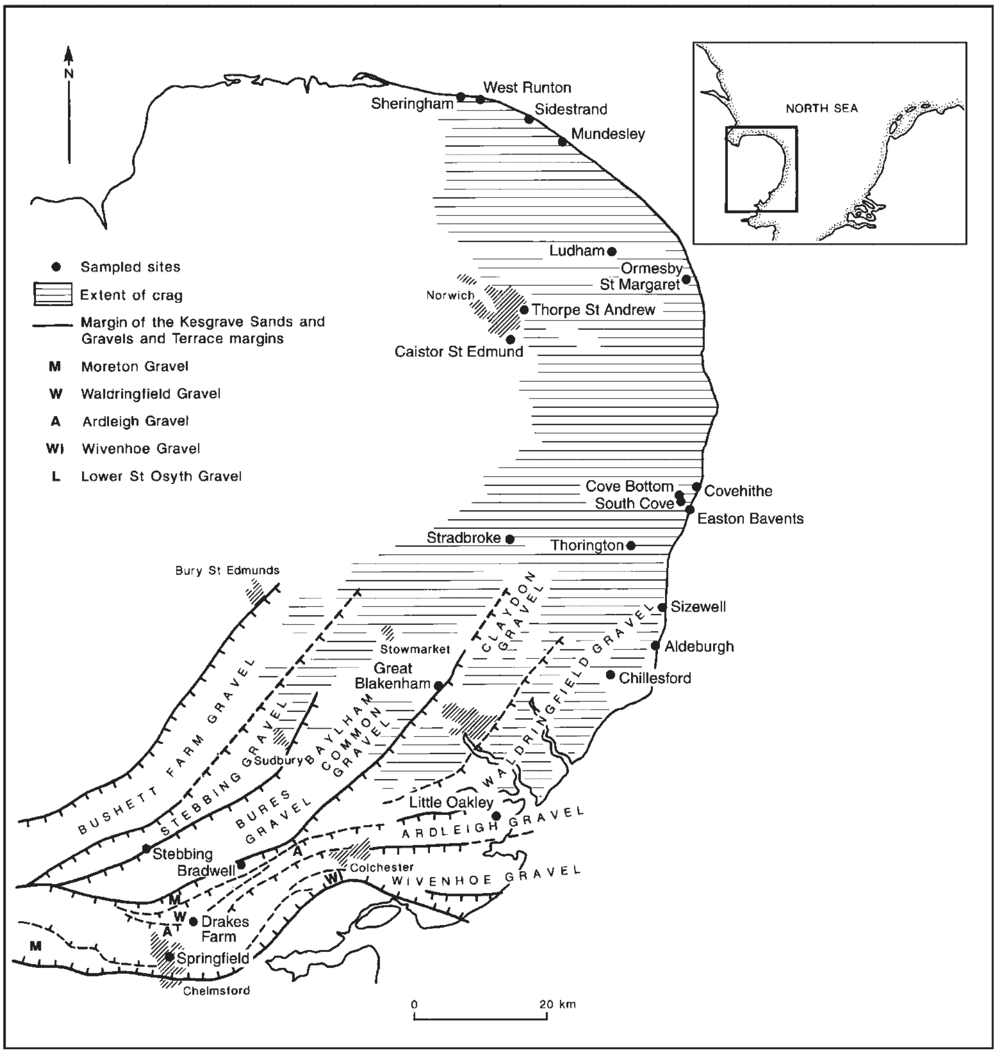

Figure 1 The distribution of Early and Middle Pleistocene sediments in East Anglia and location of sample sites

Stages have been defined, and correlations attempted, for these two major sediment groups on the basis of biostratigraphy (based on pollen, molluscs and vertebrates) and stratigraphic relations (e.g. Gibbard *et al.*, 1998).

Methods

Palaeomagnetic samples were obtained from a range of lithofacies (from the sites listed in Appendix 1) by inserting plastic sample cubes ($2 \times 2 \times 2$ cm) into clean, vertical faces cut into the sediments. All the cubes were orientated and labelled in the field, before removal from the sediment face. Samples were sealed in airtight boxes prior to measurement. NRMs were measured using a GM400 cryogenic magnetometer, with a sensitivity level of 0.01×10^{-8} A m^2. To check on the stability of the NRMs, NRMs were often measured and re-measured following periods of sample storage and/or the removal of samples from their plastic cubes for thermal demagnetisation. Due to their organic content, most samples were demagnetised by AF means; some samples *were* subjected to stepwise thermal demagnetisation (following AF demagnetisation), to identify any high-coercivity phases. Components of vectors removed at each demagnetisation step were calculated using a LINEFIND program (Kent *et al.*, 1983).

A range of rock magnetic measurements was also applied to selected samples. Low-field susceptibility was measured at room temperature and after thermal demagnetisation steps. Isothermal remanence (IRM) acquisition behaviour was determined for selected samples. In addition, a high-gradient magnetic extraction procedure (Petersen *et al.*, 1986; Hounslow and Maher, 1996) was used to obtain magnetic extracts from individual samples. To calculate the extraction efficiency, magnetic susceptibility, ARM and IRM were measured on samples before extraction and then on the 'non-magnetic' residues resulting after extraction. The extracts were analysed by optical microscopy, scanning (Hitachi S450) and transmission (JEOL 100CX) electron microscopy (STEM), energy-dispersive X-ray analysis (EDXA, Link Systems), and X-ray diffraction (Philips PW1710 with monochromatic Cu radiation, automatic divergence slit and scan speed 0.005° 20 sec^{-1}). The sequence of analyses and the sediments treated are listed in the appendices.

Results

CRM carriers

Tables 1 and 2 summarise the palaeomagnetic and mineralogical data obtained for a number of the East Anglian sedimentary lithofacies. On the basis of these data, two main groups of NRM carriers can be identified. In unoxidised, blue-grey intertidal muds (silts and clays), the ferrimagnetic iron sulphide, greigite, is the major carrier, acquiring its remanence 'syn'-depositionally as a CRM. In oxidised counterparts of these muds, post-depositional CRMs have been produced from ongoing percolation of iron-bearing groundwater and subsequent precipitation of iron oxyhydroxides, especially goethite. In the oxidised, fluvial sediments of the Kesgrave Formation, a relict, detrital magnetic assemblage is found, comprising a range of minerals, carrying either a stable DRM/PDRM or an unstable VRM.

As shown in Table 1, blue-grey intertidal silty clays at two sites, South Cove and Sheringham, Norfolk (Fig. 1), display distinctive magnetic behaviour, characterised by clustered NRMs ($\theta_{95} = 13$–$19°$, >100 samples), with slightly shallow inclinations (5–10° less than the inclination, 68–69°, of a geocentric axial dipole (GAD) at the latitude of these sites), and major loss of remanence (up to 99%) upon drying of the sediment. They also display smooth AF demagnetisation trajectories (Fig. 2(a) and (b)), but thermally 'demagnetise' completely at around 450 °C (Fig. 2(c)). Colour changes in the sediments, from blue-grey to grey and reddish colours, are associated with significant decreases in remanence intensities. For example, at South Cove, samples from unoxidised blue clays mostly had NRM values between 10 and 100 mA m^{-1}; samples from overlying, lithologically continuous but lighter-coloured, grey clays had values of less than 1 mA m^{-1} (Fig. 3(a)). A similar, striking correlation between sediment colour (oxidation state) and NRM intensity was seen at Sheringham (Hallam and Maher, 1994); in this latter case, these variations were also associated with a change in magnetic polarity. Ratios of saturation remanence to magnetic susceptibility were also found to be high (30–80 kA m^{-1}) for the blue clays. Values of this ratio for magnetite are normally around $\sim$10 kA m^{-1} or less; elevated values have been found for samples containing significant concentrations of the ferrimagnetic iron sulphide, greigite (e.g. Snowball and Thompson, 1990; Roberts and Turner, 1993).

The mineralogy of the magnetic carriers in these blue intertidal clays was independently identified by X-ray diffraction, microscopy and elemental analysis of magnetic extracts. For fresh samples from South Cove, the edge of a rare-earth magnet was used to create a magnetic gradient at the side of a test-tube containing a dispersed sample suspension. Figure 4 shows the XRD spectra obtained from the extracts. Significant quantities of iron-rich clays are present (kaolinite, glauconite and illite), but greigite peaks are also clearly shown, and, less importantly, pyrite and quartz. Small peaks also indicate the minor presence of jacobsite ($MnFe_2O_4$) or titanomagnetite. For the Sheringham samples, XRD peaks corresponded mainly to clay minerals and greigite, with some quartz also present. Under optical microscopy, the quartz grains present in the magnetic extracts were found to contain sub-micrometre opaque inclusions. However, some 70% of the extracts were seen to consist of clustered, opaque minerals, with grain sizes beyond the resolution of both optical and scanning electron microscopy. Elemental analysis of the clustered opaques indicated the presence of significant abundances of Fe and S, and subordinate amounts of Si. Under TEM, the clusters were seen to consist of often-perfect rhombohedral, hexagonal and cubic crystals, with grain sizes between 0.1 and 1 micrometre (Fig. 5).

These data identify the importance of the metastable iron sulphide, greigite, in these reduced, blue-grey, intertidal sediments. The palaeomagnetic results indicate that this carrier is highly unstable under oxidation, both *in situ* and in laboratory and storage conditions. Under thermal demagnetisation, these samples were probably being oxidised (producing paramagnetic iron oxyhydroxides upon heating) rather than 'demagnetised'. However, AF demagnetisation of fresh samples was effective in removing a stable NRM vector over a range of fields.

DRM and VRM carriers

For the remaining marine sediments, and also for the fluvial sediments of the proto-Thames, the NRM appears to be of depositional, detrital origin, carried by a persistent, relict magnetic assemblage of ilmenites, chromites and magnetite (but only as inclusions within host silicate grains). The magnetic behaviour at many of the sites (Table 1) is characterised by

Table 1 Sampled locations, polarity and palaeomagnetic behaviour

Location, Sampled unit	Stratigraphic age or gravel aggradation	NRM polarity	Qualities of the NRM record N = no. of samples	AF demagnetisation	Thermal demagnetisation	Probable carrier(s)
CRM carriers:						
Sheringham: unit e	Pastonian Tidal marine	N	NRMs highly clustered, $\theta_{95} = 11.3°$; mean dec. = 33°, inc. = 61.4° (slightly shallow). Intensities variable but high (12–165 mA m^{-1}). Significant intensity loss with storage. $N = 12$.	Convex AF demag. profile. Linear trajectories, stable demag. to 50 mT.	NRM loss with drying. Demagnetisation complete at 400°C.	Greigite
Sheringham: unit g. sub-unit 2	Pastonian Tidal marine	R	NRMs high intensity (10 mA m^{-1}), very clustered, $\theta_{95} = 18.5°$, mean dec. = 193°, inc. = −61.9° (slightly shallow). $N = 32$.	Very stable AF demag. to 90 mT; mdfs 25–30 mT. Subunits 2–3 (boundary samples) display removal of normal component.	Thermal demag. of IRM—IRM virtually destroyed at 400°C. Maximum rates of demag. 0–80°C, 200–250°C.	Greigite
South Cove	Baventian Near-shore marine	N	NRMs clustered, $\theta_{95} = 15.2°$, dec. = 22°, inc. = 58.3°. Intensities high (15–30 mA m^{-1}). Significant loss with storage. $N = 80$.	Very stable demagnetisation up to 60 mT (large intensity loss in storage makes mdfs irrelevant).	Most of remanence lost on drying. Some demag. to 150°C.	Greigite
DRM and VRM carriers:						
Great Blakenham/ Creeting Sands	Bramertonian Tidal marine	N	NRMs clustered, $\theta_{95} = 19.5°$, mean dec. = 29°, inc. = 67.4°. Low intensities (0.25–0.8 mA m^{-1}). Storage results in decreased incs. but increased intensity. $N = 33$.	Low coercivity, mdfs 4.5–15 mT. Typically stable to 10 or 15 mT (max. 30 mT).	Demagnetisation on drying. Most of new remanence removed at 200°C.	Chromite, ferrian ilmenite
Great Blakenham/ College Farm Silty Clay	Bramertonian Freshwater lagoon	N	NRMs somewhat scattered, $\theta_{95} = 31°$, mean dec. = 32.9°, inc. = 67.9°. Intensities very varied (0.2–12 mA m^{-1}). $N = 38$.	Low coercivity, mdfs 8–15 mT. Above 20 mT spurious remanences. Storage following AF demag.: large effects on direction and intensity.	Following AF demag., large decrease in remanence with drying. Demag. to 100/200°C.	Ferrian ilmenite (chromite)
Thorpe St Andrew	Bramertonian Near-shore marine	N	NRMs scattered, $\theta_{95} = 40°$, average direction further from GAD when bed correction applied, mean dec. = 75.2°, inc. = 62.2°. Intensities low to moderately high (0.3–1.2 mA m^{-1}). $N = 13$.	Low coercivity, mdfs 8–15 mT. Stable to 20–30 mT. Demagnetisation trajectories further from the expected direction when bed correction applied.	Large effects of cutting and drying sediment on NRM direction and intensity. Stable demagnetisation to 200°C.	Ferrian ilmenite
Thorington	Baventian Fluvial channel fill	N	NRMs moderately clustered, $\theta_{95} = 21.3°$, mean dec. = 12.4°, inc. = 76.9°. Intensities moderately low (0.4–0.9 mA m^{-1}), some loss (one increased) with storage. $N = 40$.	Stable demag. Significant low coercivity component, mdfs 14–26 mT. Stable to 60 mT, with component not demagnetised.	Some demag. with drying. Stable demag. to 200°C, some demag. to 450°C.	Ferrian ilmenite, titano-haematite (chromite)
Great Blakenham/ Kesgrave Sands and Gravels	Baylham Common Gravel Fluvial	N	NRMs very scattered, $k = 4$, mean dec. = 23.4°, inc. = 64.7°. Intensities mostly low (0.07–0.4 mA m^{-1}). Large effects with storage on remanence direction and intensity. $N = 8$.	Unstable demagnetisation, low coercivity, mdfs 2.5–13 mT.	Large effects on drying of sediment. Some demagnetisation to 100°C.	Ferrian ilmenite, magnetite, chromite

Springfield SP2	Fluvial	N Shallow	NRMs clustered, $\theta_{95}=15.4°$, mean dec. = 27.8°, inc 37.2°. Intensities moderately low to moderately high (0.8–1.6 mA m^{-1}), small loss of intensity with storage. $N=18$.	Very shallow, very low coercivity component. Normal polarity, higher coercivity component stable to 50 mT. Large changes to demagnetised remanence on storage.	Normal polarity component removed 150°C. Very shallow component 150–300°C. No hard component.	Ferrian ilmenite, chromite.
Bradwell	Moreton Gravel Fluvial	N	NRMs somewhat scattered, $k=24.5$, mean dec. = 295.2, inc. = 62.8°. Intensities moderately high (1.1–5.7 mA m^{-1}). Some loss of intensity with storage. $N=6$.	Significant low coercivity component, but stable up to 80 mT (mdfs 13–60 mT). Up to 40% of NRM remained after AF demagnetisation.	[NRM]—demag. 40–700°C, particularly <200°C and 500–600°C. [Post-AF demag.]—significant demag. 400–700°C.	Ilmeno-haematite, titanomagnetite (chromite).
Springfield SP1	Gerrards Cross Gravel Fluvial	N Very shallow	NRMs clustered, $\theta_{95}=15.4°$, mean dec. = 42.7°, inc. = 6°. Intensities high (4.7–7 mA m^{-1}), little effect with storage. $N=19$.	Large, very shallow, very low coercivity component; small, normal polarity, higher coercivity component stable to 90 mT.	Very shallow component removed 0–700°C, hard normal polarity removed below 250°C, hard shallow remanence removed to 475/700°C.	Haematite, ferrian ilmenite, chromite (goethite).

unstable magnetic behaviour, evidenced by very low or low coercivity components (2–15 mT) and viscous (time-varying) changes in the NRM during storage (samples were stored in a cold store, at 6 °C, in the Earth's magnetic field). The instability of specimens upon AF demagnetisation, as shown by large variations in remanence direction (e.g. Fig. 6, from a sample of lagoonal silts/clays), reflects acquisition of spurious magnetisations during the demagnetisation process (VRMs, whilst exposed to the Earth's field between demagnetisation and measurement). Upon thermal demagnetisation, most remanence was lost at low temperatures, around 200–300 °C (e.g. Fig. 7); significant loss of remanence was also often observed on drying of sediment at 50 °C.

Rock magnetic measurements for these sediments indicate that most of the remanence-carriers are ferrimagnetic, with 90–97% of the total remanence acquired in fields up to 300 mT. Susceptibility values are low, around $5 \times 10^{-8} m^3 kg^{-1}$. Median destructive fields of another artificial remanence, the anhysteretic remanence (ARM), are high (19–22 mT) in comparison with the NRM$_{mdfs}$ (2–15 mT), which indicates that the sediments contain a total ferrimagnetic assemblage with higher coercivity than the sub-population of grains responsible for the NRM signal. It is likely that the artificial remanence (the ARM) is contributed to mostly by the magnetite inclusions, which, conversely, contribute little to the NRM, as the diamagnetic nature of the host silicate grains have precluded efficient orientation of the grain after deposition. SIRM/susceptibility ratios are low, around 1.5 kA m^{-1}.

Mineralogical analysis of magnetic extracts from these VRM-dominated sediments reveals the presence of various ferrimagnetic spinels, but particularly ilmenites and chromites. Figure 8(a) and (b), for example, show the XRD spectra for samples from estuarine/fluvial silts/clays (sampled from Thorington and the Creeting Sands at Great Blakenham, Suffolk, respectively). The main ilmenite peaks are typically significantly offset to the right, i.e. towards the positions of haematite peaks (Fig. 8(b)). Possible explanations for this observed offset include: (a) the coexistence of poorly crystalline ilmenite and haematite, with resultant broad and overlapping peaks; (b) intergrowth between ilmenite and haematite; or (c) the presence of a mineral intermediate in composition between ilmenite and haematite. Other minerals identified by XRD in the extract are clay minerals, quartz, feldspars, haematite and rutile. Figure 9 shows an SEM image of a cluster of magnetic grains extracted from the Creeting Sands, together with the elemental maps from EDXA. In addition to the four dominant elements of Fe, Si, Cr and Ti, minor abundances of K were identified, probably associated with the feldspars or the clay minerals seen by XRD.

Albeit rather rarely among this collection of sediments, high-coercivity components of remanence, carried by minerals such as haematite and goethite, were identified from both palaeomagnetic and mineralogical evidence. For instance, fluvial sediments are exposed at Bradwell, which have been attributed to the lowest stratigraphic member of the Sudbury division of the Kesgrave Formation (Whiteman and Rose, 1992). Within these coarse-grained sediments, occasional finer materials occur, often in the form of clay drapes. Upon AF demagnetisation of specimens from these fine-grained sediments, up to 40% of the NRM remained at fields up to 80 mT. Subsequent thermal demagnetisation produced little effect on the remaining NRM up to 400 °C, but between 400 and 700 °C, a stable, normal polarity component was removed (Fig. 10). After the 250 °C demagnetisation step, the magnetic susceptibility showed a sharp increase, indicating the formation of new magnetic phases. This was followed by a gradual decrease in susceptibility above 450 °C. However, there was no evidence from the

Table 2 Sampled locations, XRD peaks and EDXA elemental abundances

Location sampled unit	Stratigraphic age or gravel aggradation	XRD	Elemental abundances, EDXA
CRM carriers			
Sheringham: unit e	Pastonian Tidal marine	D: Quartz I: Feldspar M: Rutile, Pyrite, Ilmenite, Dolomite, Clays	V. common: Fe + Cr, Fe + Ti Common: Si Rare: Ti; Si + K + Ca
Sheringham: unit g, sub-unit 2	Tidal marine	D: Quartz I: Clays M: Greigite	Common: Fe + S, Fe + Cr Minor: S; Fe + Ti
South Cove Brickworks	Baventian Near-shore marine	D: Quartz, Greigite, Clays M: Pyrite, Spinel	V. common: Fe + S Common: Si
DRM and VRM carriers			
Great Blakenham/Creeting Sands	Bramertonian Tidal marine	D: Quartz I: Feldspar, Anatase, Rutile, Clays M: Spinel, Ilmenite M	V. common: Si Common: Fe + Cr and Fe + Ti Minor: Ti Rare: Si + K
Great Blakenham/College Farm Silty Clay	Bramertonian Freshwater lagoon	D: Quartz, Ilmenite I: Anatase, Rutile, Brookite, Clays M: Feldspar, Spinel, Pyrite	V. common: Si Common: Ti + Fe Minor: Ti Rare: Fe + Cr, S + Fe, Si + K
Thorpe St Andrew	Bramertonian Near-shore marine	D: Quartz I: Anatase, Ilmenite, Feldspar, Clays M: Spinel	Common: Fe + Ti, Si + K + Al + Fe Rare: Fe + Cr, Ti
Thorington	Baventian Fluvial channel fill	D: Quartz, Ilmenite/Haematite I: Rutile, Anatase, Feldspar M: Clays, Spinels	V. common: Fe + Ti Minor: Fe + Cr, Ti, Si
Great Blakenham/Kesgrave Sands and Gravels	Baylham Common Gravel Fluvial	D: Quartz M: Anatase, Rutile, Ilmenite, Haematite, Spinel, Feldspar, Clays	Common: Fe + Ti Minor: Si, Ti, Fe, Si + Al, Fe + Cr
Springfield SP2	Fluvial	D: Quartz M: Ilmenite, Rutile, Anatase, Spinel, Feldspar, Clays M	V. common: Si Common: Ti + Fe Minor: Ti, Fe + Cr Rare: Si + K
Bradwell	Moreton Gravel Fluvial	D: Quartz I: Rutile, Ilmenite, Haematite M: Feldspar, Spinel, Mica	Common: Fe + Ti Minor: Si, Ti, Fe, Fe + Cr
Springfield SP1	Gerrards Cross Gravel Fluvial	D: Quartz, Haematite I: Spinel, Clays M: Anatase, Rutile, Goethite	V. common: Fe, Fe + Cr Minor: Si, Ti Rare: Si + Al

Peak size: XRD; D = dominant; I = intermediate; M = minor.
Elemental abundances: >40%, v. common; 20–40%, common; 5–20%, minor; 1–5%, rare; <1%, v. rare.

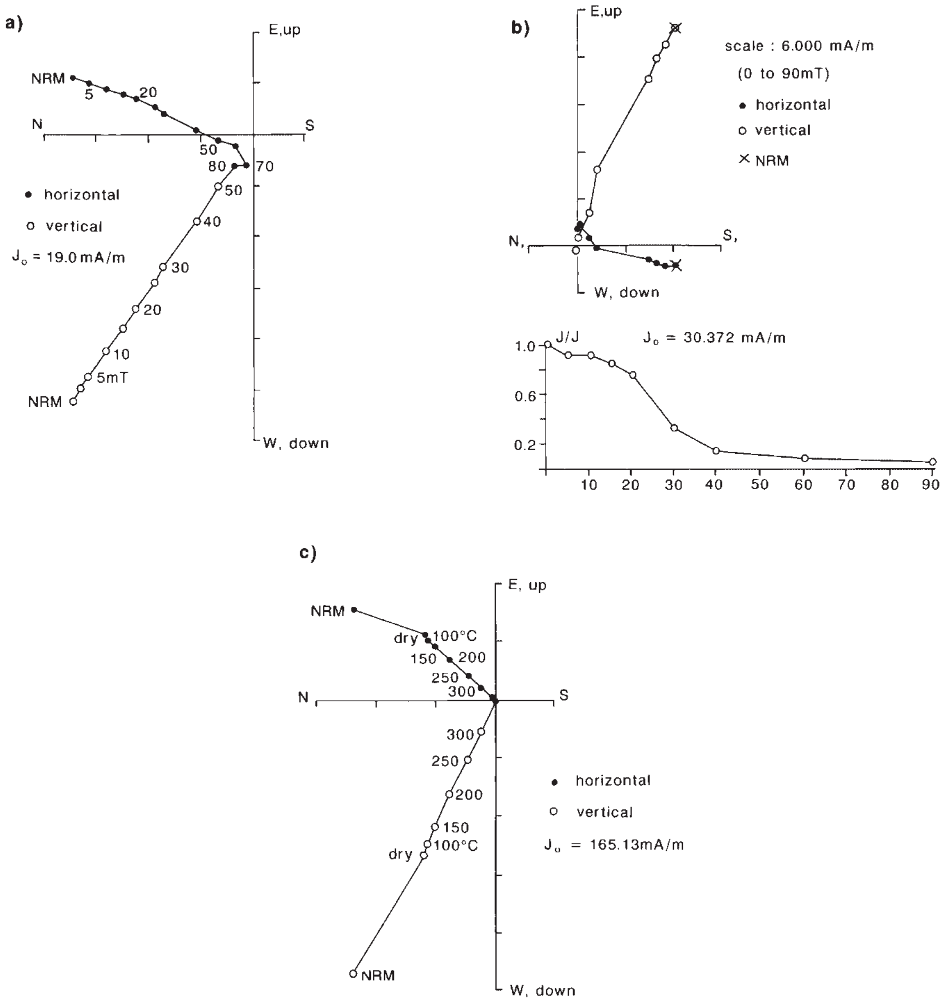

Figure 2 (a) AF demagnetisation, sample CB106, South Cove: linear demagnetisation trajectories towards the origin indicate a stable palaeomagnetic direction carried by magnetic minerals with a range of coercivities. (b) AF demagnetisation, sample SH07, Sheringham sub-unit 2: linear demagnetisation trajectories, as above. (c) Thermal demagnetisation, sample SH309, Sheringham unit e: linear demagnetisation trajectories upon heating, with all remanence 'demagnetised' by 350 °C

demagnetisation data of spurious or new remanences accompanying these susceptibility changes. In IRM acquisition experiments, 29–34% of the IRM was acquired in fields between 0.3 and 1 T, confirming the presence of significant numbers of magnetically hard, high-coercivity grains. Magnetic extracts from the Bradwell samples were found by XRD to contain quartz, ilmenite and haematite, with minor amounts of rutile, anatase, feldspar, spinel and mica. The ilmenite peaks were offset towards haematite peak positions as described above. Elemental analysis showed that Fe was the most abundant element, often associated with Ti, and occasionally with Cr.

Discussion

Magnetic minerals and remanence acquisition

Detrital (titano)magnetite dominates the magnetic assemblages of most rocks and sediments (due to its high saturation magnetisation, $\sim$480 kA m^{-1}). However, in the Early and Middle Pleistocene sediments of the southern North Sea basin, discrete grains of magnetite are rarely found; rather, it occurs as ferrimagnetic inclusions within silicate host grains, especially quartz and feldspars. The major remanence-carriers in these sediments appear to be the 'less common' magnetic minerals, greigite, ferrian ilmenite and ferrimagnetic chromites.

Greigite

Iron sulphide formation in sulphate-rich sediments is a bacterially mediated process. Sulphate-reducing bacteria generate high concentrations of H_2S, oxidised by bacteria to produce elemental sulphur, which then combines with iron in the extracellular environment. Under most conditions, pyrite forms as the diagenetic end product of a series of intermediate iron sulphide minerals, which include greigite. Greigite will be preserved if diagenesis is arrested, for example if insufficient elemental sulphur is available. Ultimately, pyrite formation may be limited by the availability of organic matter and/or sulphate, with greigite being preserved under specific conditions of Eh and pH (Demitrack, 1983). Given the identity of greigite as the major NRM-carrier in the blue-grey intertidal muds at two sites, the NRM was probably acquired as a CRM soon (i.e. within $\sim$100 years) after sediment deposition and subsequent sulphide formation (Demitrack, 1983; Tric *et al.*, 1991; Roberts and Turner, 1993). Greigite is chemically unstable during erosion and transport and, when detected in sediments,

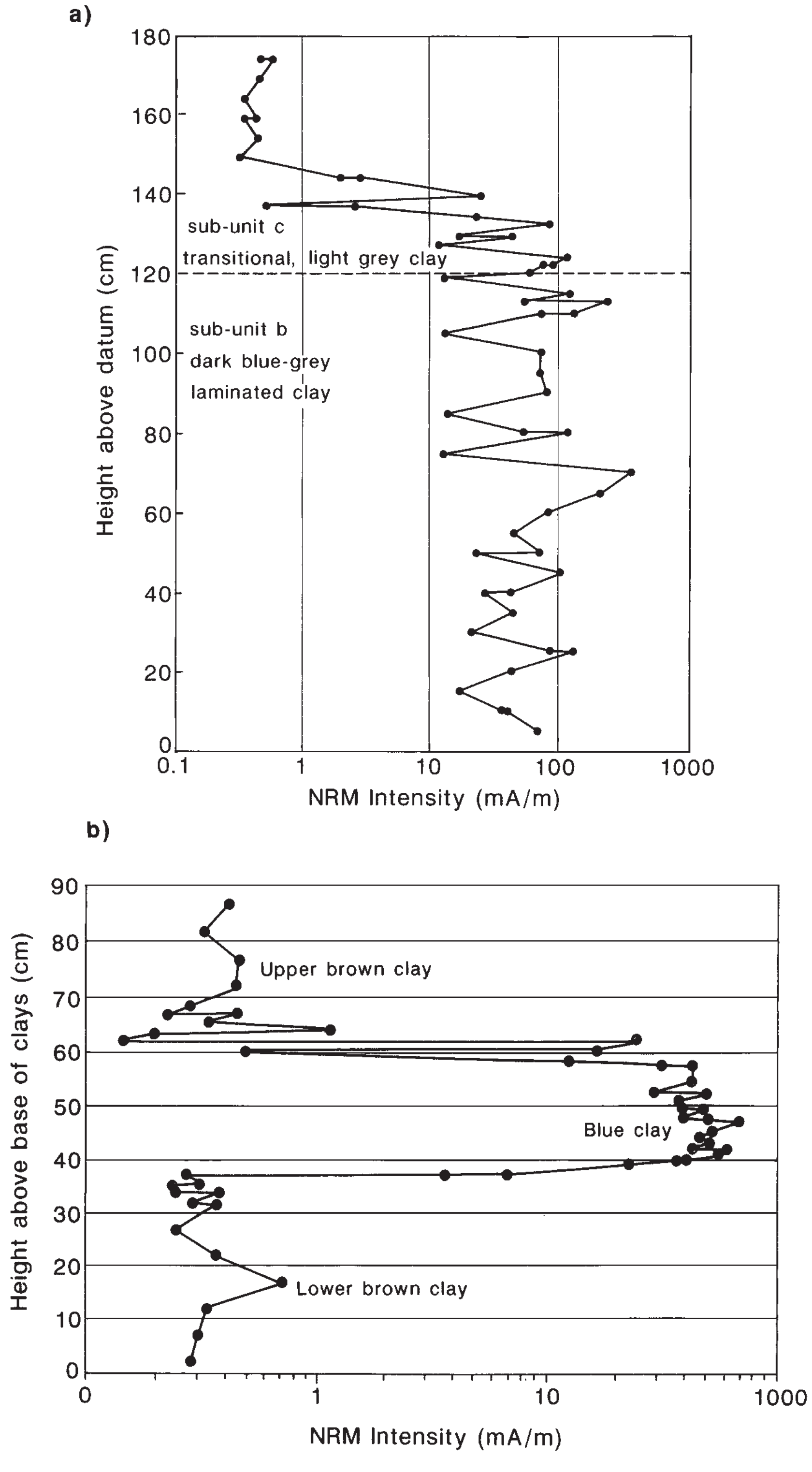

Figure 3 Variation of NRM intensity and sediment colour. (a) South Cove. (b) Sheringham (from Hallam and Maher, 1994)

must have an authigenic origin. Its formation results in a CRM, as individual grains grow past the superparamagnetic/stable single domain boundary. Alternatively, as proposed by Stanjek *et al.* (1994), close aggregation of superparamagnetic grains results in single domain-like behaviour.

The propensity for greigite's NRM to be destroyed upon oxidation does not necessarily cast doubt on its palaeomagnetic integrity; the greigite in these sediments appears to be stable as long as reduced conditions prevail. For marine sediments with a sedimentation rate of 1 m ka^{-1}, Canfield and Berner (1987) found the dissolved sulphide concentration reached a maximum within 1 m of the sediment surface. For modern freshwater sediments, sulphate was consumed within a few centimetres of the surface (Berner, 1984). Holocene sedimentation rates on the Norfolk barrier coast have been estimated (e.g. Funnell and Pearson, 1989) at 1 m ka^{-1}. Given similar rates in analogous sedimentary systems of the Early and Middle Pleistocene, greigite formation would be expected to occur within ~1 kyr of sedimentation. The CRM could thus reasonably be considered as 'syn'-depositional with respect to the resolution of the GPTS. However, greigite authigenesis may be incurred *late* in the depositional history of some sediments and thus overprint with its CRM an original palaeomagnetic signal. Thompson *et al.* (1992), for example, found anomalous

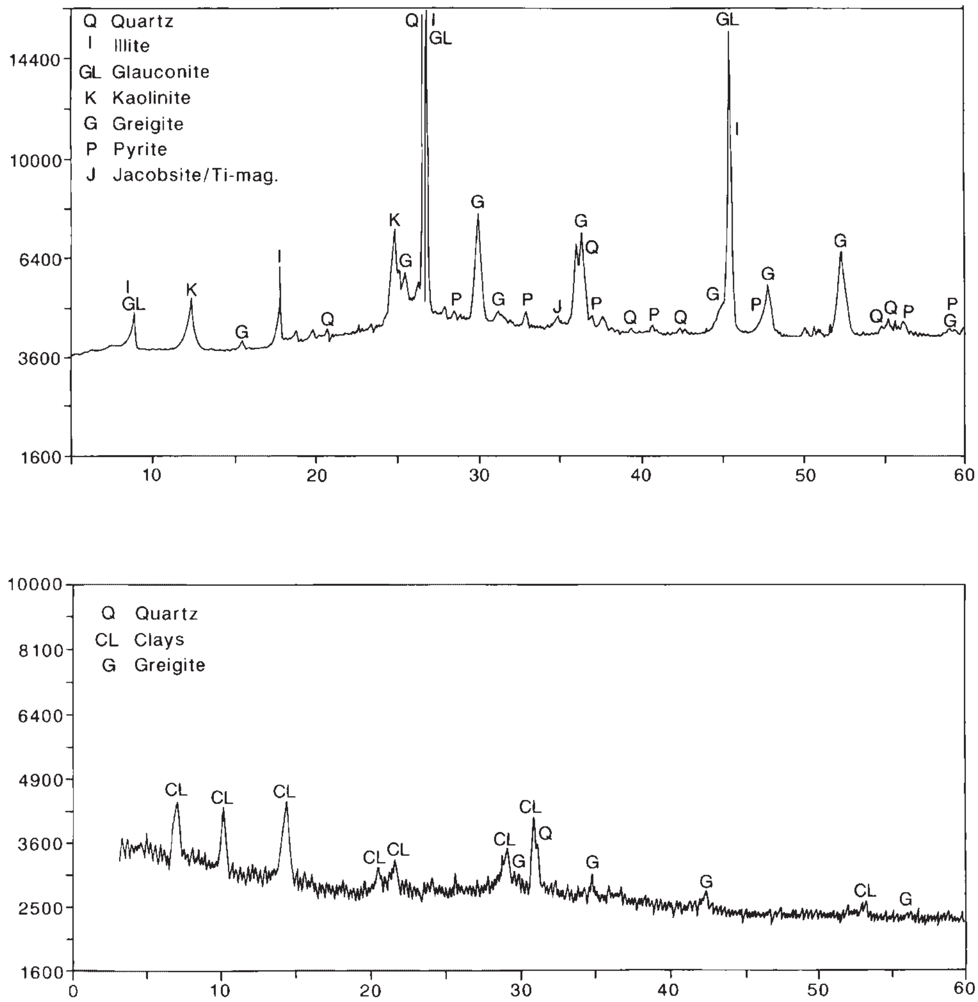

Figure 4 XRD spectra for magnetic extracts from. (a) South Cove. (b) Sheringham (from Hallam and Maher, 1994)

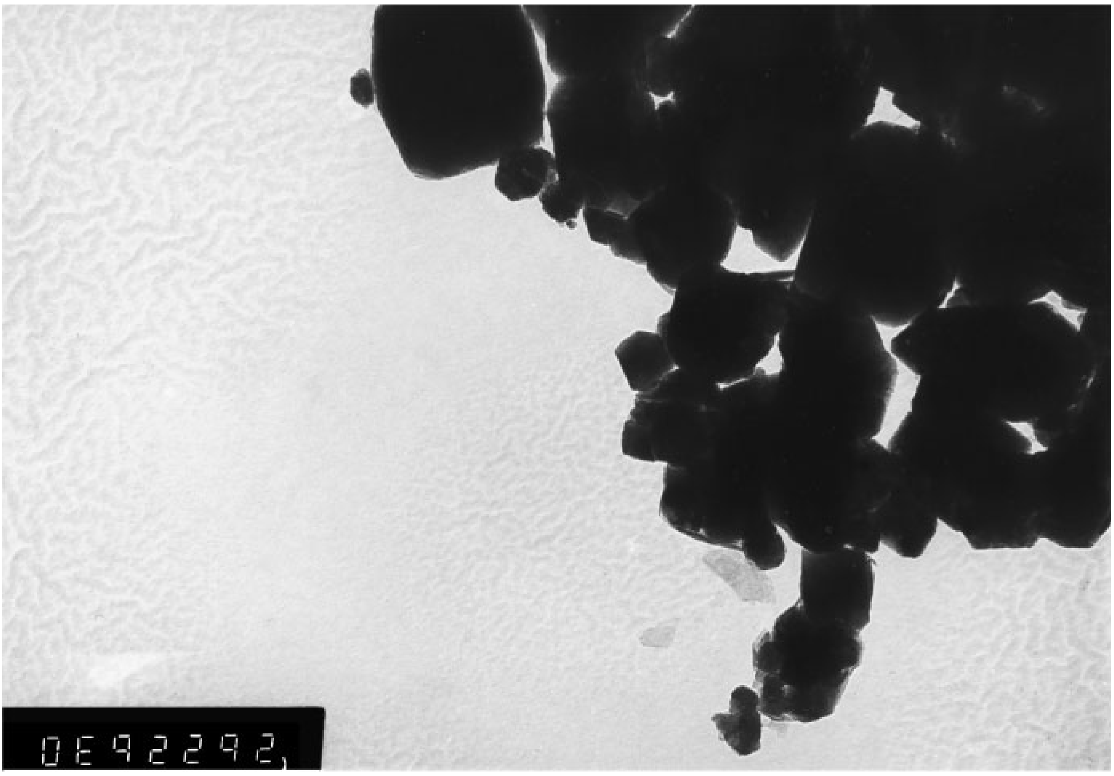

Figure 5 Transmission electron micrograph of iron sulphide crystals, magnetic extract from Sheringham

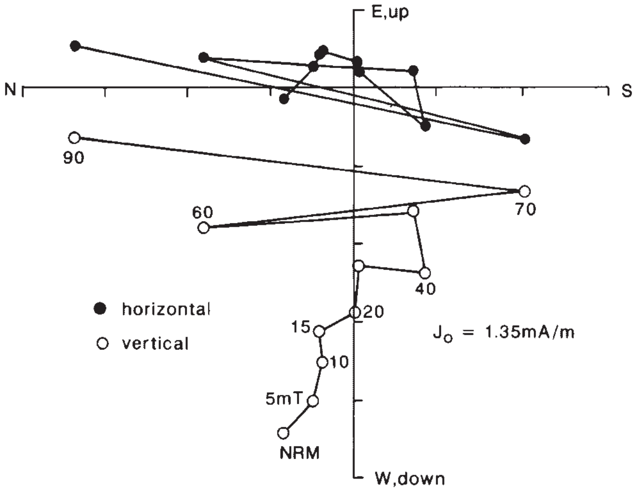

Figure 6 AF demagnetisation, sample GBA22, College Farm Silts: instability of remanence, probably reflecting VRM acquisition between demagnetisation and measurement (these instabilities were not removed upon correction of rotational remanence acquisition)

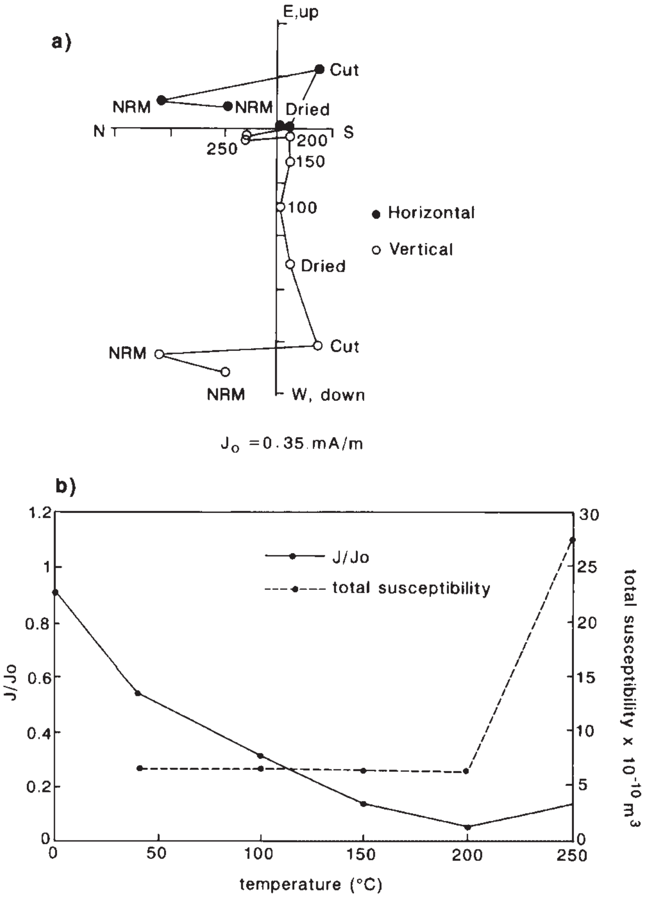

Figure 7 Thermal demagnetisation, sample CR09, Creeting Sands, Great Blakenham, ~95% of the NRM is lost upon heating to only 200 °C (a). Beyond 200 °C, a significant change in remanence direction was observed, together with a large increase in magnetic susceptibility (b), indicating formation during the experiment of a new magnetic phase and its probable acquisition of a laboratory CRM

normal polarity intervals within the Matuyama Chron in North Sea borehole samples, which they interpreted as possible Brunhes-age secondary magnetisations caused by late overgrowths of greigite in response to hydrocarbon seepage.

Ferrian ilmenites

For the oxic sediments at a number of the East Anglian sites, the major remanence carrier displayed notably low coercivities upon AF demagnetisation and also demagnetised thermally at low (<400 °C) temperatures. Mineralogical data indicate the presence of significant concentrations of ilmenite or an ilmenite/haematite intermediary. Given the relative size of the ilmenite XRD peaks (compared to the spinels), and their degree of offset towards haematite peak positions, ferrimagnetic forms of haemo-ilmenite (ferrian ilmenite) appear to be significant carriers of the AF-demagnetised remanence in the East Anglian sediments. Appendix 3 summarises the changes in magnetic behaviour exhibited by these minerals with changes in the degree of ionic substitution. Briefly, minerals within the middle compositional range of the solid solution series between haematite and ilmenite are ferrimagnetic (like magnetite) and can retain a stable NRM (as a DRM) over geological timescales—especially if, as here, the sediments are held at surface environmental temperature. However, their blocking temperatures (i.e. the temperature below which magnetic ordering overcomes thermal agitation) are relatively close to surface environmental temperatures and so they are prone to being overprinted by late, viscous remanences.

Although ferrian ilmenites are common as igneous detrital particles in sedimentary rocks, the *ferrimagnetic* intermediate compositions are considered rare. For example, Basu and Molinaroli (1989) found such minerals comprised only 3% of detrital Fe–Ti oxide grains derived from igneous source rocks, and none were found where sources were metamorphic. Given the paucity of ferrimagnetic ilmenite in most source rocks and the mixed sources of the sediments under consideration here, magnetic dominance by this mineral implies that virtually all the titanomagnetite, which would dominate most primary sedimentary assemblages, must have been selectively removed.

Iron–chromium spinels

Significant numbers of grains containing iron and chromium were detected in many of the magnetic extracts from the East Anglian sediments, and these grains may account for many of the spinel peaks identified by XRD analysis. Pure chromite ($FeCr_2O_4$) is paramagnetic (i.e. with no remanence-carrying capability) at room temperature, only becoming ferrimagnetic below −183 °C (Schieber, 1967). However, a solid solution exists for Fe–Cr spinels (Appendix 3), with end members chromite and magnetite. Chromite is a common detrital heavy mineral, with origins in basic igneous rocks (Freeman, 1986), and is highly stable, persisting into deep burial (Milliken and Mack, 1990). Deer *et al.* (1966) note that chromites are often slightly magnetic, and that Cr may substitute for Fe^{3+} in magnetite, often in appreciable amounts. Hounslow *et al.* (1995) found that chromites represented a significant proportion of the relict magnetic assemblage in Triassic North Sea sediments.

As for the ferrian ilmenites, chromites with blocking temperatures close to environmental temperatures will be highly susceptible to VRM acquisition. Sediments containing such grains are thus likely to carry a late, secondary magnetic overprint.

Magnetic inclusions in silicate minerals

For all the East Anglian sites, quartz comprised an important part of the magnetic extract, and was often the most common mineral in the relict detrital assemblages. Opaque inclusions

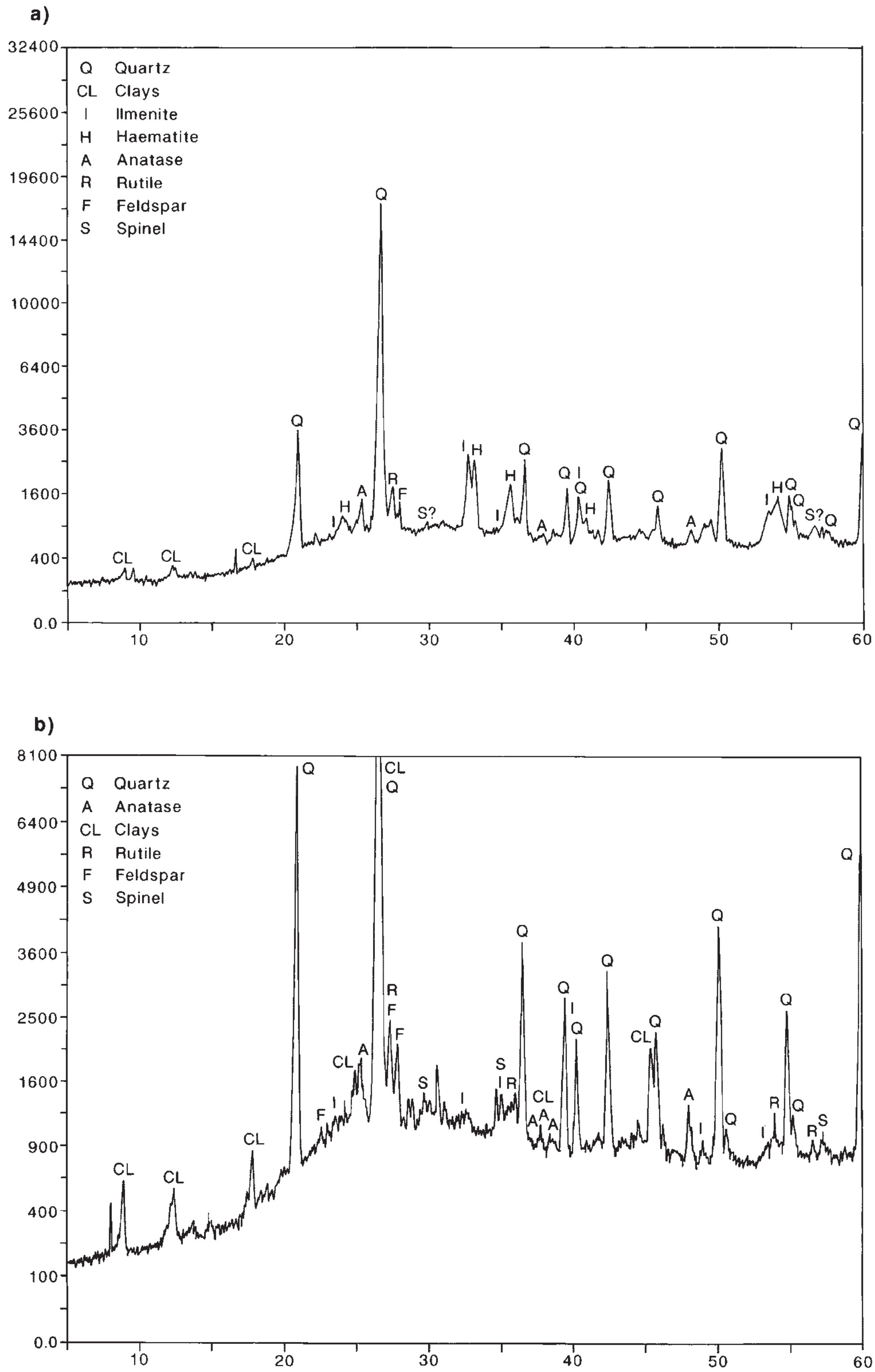

Figure 8 XRD spectra for magnetic extracts from (a) Creeting Sands, Great Blakenham and (b) Thorington. Ilmenite and haematite, together with quartz, are the major components of the extract mineral assemblage. The ubiquitous presence of quartz (a diamagnetic mineral) in the extracts reflects the presence of magnetic inclusions

were visible by optical microscopy in some (but not all) grains. A high-gradient/low intensity magnetic field (~40 mT) was used in the magnetic extraction procedure, preferentially to extract ferrimagnetic grains. Given the ubiquity of quartz grains in the extracts, we infer that magnetic inclusions, either above or below microscopic resolution, occur in all these extracted grains. Clearly, the host silicate particles protect the included magnetic material from diagenetic attack and dissolution, to which discrete magnetic grains will be vulnerable. Whether or not these inclusions are of palaeomagnetic importance might be gauged from the observed differences between the artificial and natural remanences. For instance, for the oxic sediments, the stability of the artificial remanence, the anhysteretic remanence (ARM) is often much higher than that of the

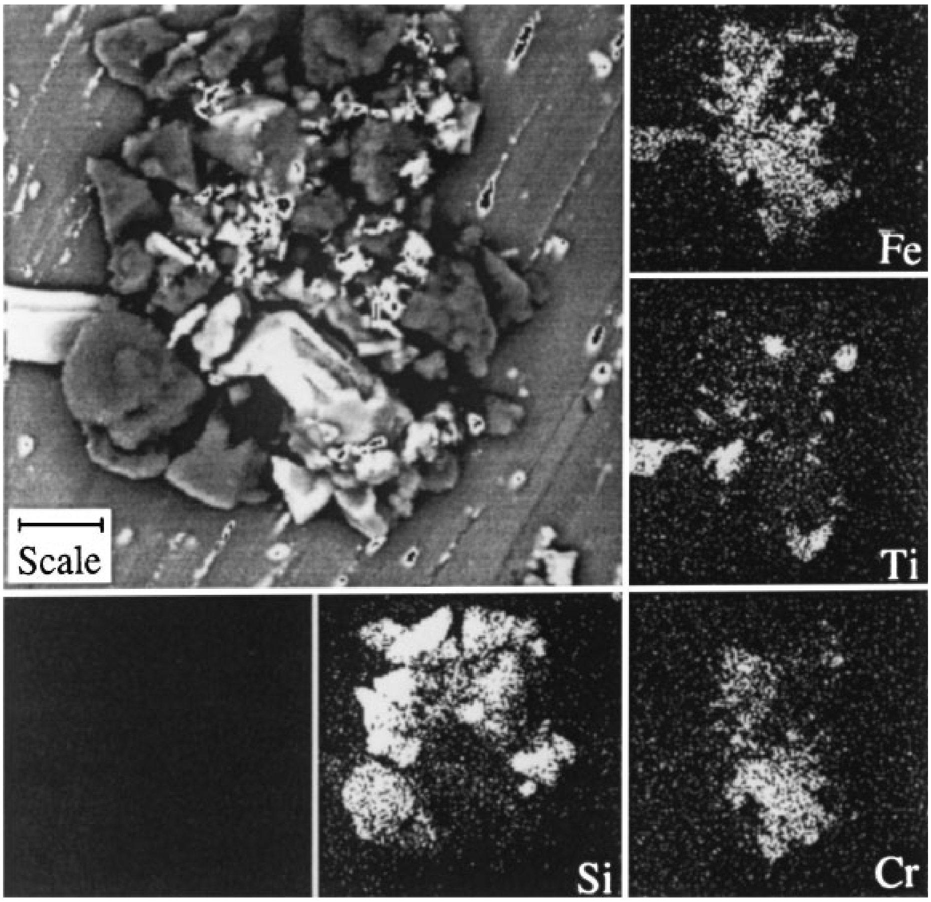

Figure 9 SEM image and EDXA elemental mapping for magnetic extract from the Creeting Sands, Great Blakenham, identifying the dominance of Fe, Si, Ti and Cr in the magnetically extracted grains. Scale bar = 10 μm

NRM; fine-grained magnetic inclusions can may efficiently acquire a stable ARM, but be less efficient as NRM carriers, being enclosed within relatively large, diamagnetic host grains. For inclusions to carry a DRM, their net magnetic moment must be sufficiently high that the magnetic torque overcomes other forces acting on the quartz grains during deposition. The larger the mass and volume of the host grain compared to the ferrimagnetic inclusions, the less efficient will be magnetic orientation.

The presence of both magnetic inclusions and discrete magnetic grains makes it difficult to assess how representative are the magnetic extracts of the NRM-carrying grain population. ARM extraction efficiencies for six sets of the oxic sediments vary between 10% and 66% ($x = 42\%$); susceptibility extraction efficiencies between 0% and 64% ($x = 25\%$). Susceptibility extraction efficiencies are strongly dependent on the origin, paramagnetic or ferrimagnetic, of the susceptibility. During the magnetic extraction process, the whole magnetic mineral population, not just the NRM carriers, is subjected to extraction. This is particularly important to note when, as suspected here, the magnetic assemblage contains many grains contributing less to the NRM than to the artificial remanences.

The extraction process may also incur loss by oxidation of magnetic components in those sediments containing metastable greigite. Upon oxidation of greigite, paramagnetic iron oxyhydroxides have been observed (Hallam and Maher, 1994). Extraction efficiencies for the sediments at Ardleigh, for example, were 88% for the ARM and 94% for the IRM. These high values either suggest very efficient magnetic extraction of the ARM- and IRM-acquiring grains, or they may be spurious, reflecting greigite loss by oxidation during the extraction process.

Despite these caveats, independent mineralogical analysis, in tandem with the standard palaeomagnetic and rock magnetic methods, can provide more information on likely remanence mechanisms than palaeomagnetic analysis alone.

Post-depositional iron oxyhydroxides, including goethite

A further palaeomagnetic complication at many of the East Anglian sites arises from groundwater percolation and ‘late’ precipitation of iron oxyhydroxides, including goethite. Seepage of oxidising groundwater is particularly enhanced in those sediments that have been subjected to deformation and micro- and macro-faulting, as is the case for many of the pre-Anglian/Elsterian sequences in the region. A post-depositional, overprinting CRM can result from this process, especially in sediments deficient in ferrimagnets.

Sediment transport and diagenesis

For the East Anglian Early and Middle Pleistocene sediments, ilmenite and chromite appear to be the most important detrital carriers of NRM. This is the case not only in the marine sediments, which are likely to have been subjected to

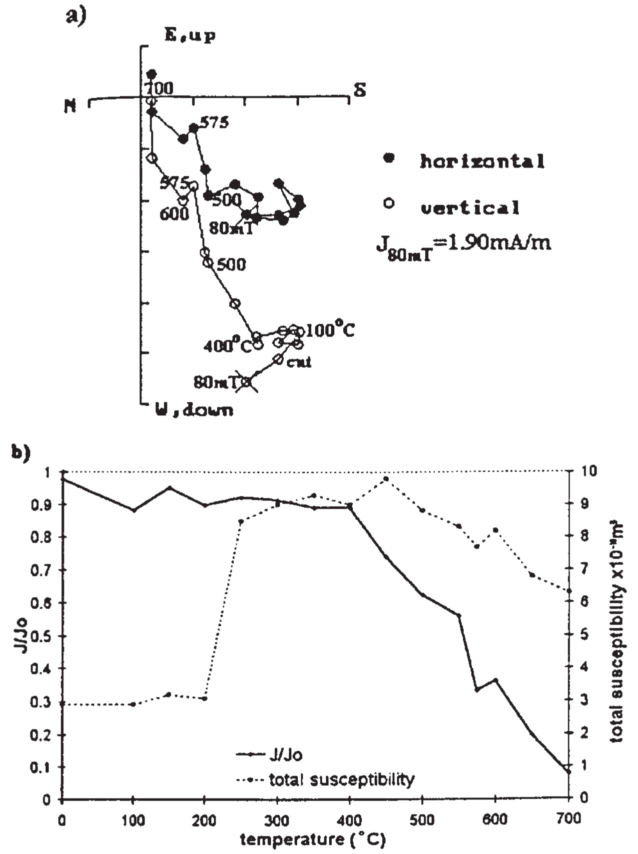

Figure 10 Thermal demagnetisation, sample BRD5, Bradwell, Suffolk: a magnetically very hard magnetic component (haematite probably) is evident, with >80% of the remanence remaining at 400 °C, and a stable, normal polarity component being removed between 400 and 700 °C

post-depositional magnetite dissolution, but also in the fluvial sediments of the Kesgrave Formation.

Under mildly oxidising conditions, e.g. during sediment transport, magnetite is delivered unaltered to most depositional environments. Most alteration occurs when sediment is buried and reducing conditions are established; magnetite, buried in anoxic conditions, is subject to dissolution and replacement. In highly sulphidic sediments, it is replaced by pyrite, whereas in mildly reducing environments, it is dissolved. The estimated half-life of magnetite in anoxic marine sediments varies between 50 and 1000 years, with magnetite preferentially dissolved with respect to more titanium-rich oxides (Karlin and Levi, 1985).

This initial diagenesis occurs at shallow depths and is likely to have affected many of the marine sediments discussed here, but not the fluvial sediments. However, most of the detrital material within the Kesgrave Formation is itself reworked (Rose and Allen, 1977). Further, it is likely that all the material laid down in the Crag Basin during the Early Pleistocene is also reworked, there being no local sources of 'primary' sediment. Thus, these sediments are likely to have resided in a variety of sedimentary burial environments, for long periods of geological time, prior to their transport to East Anglia. At shallow depths, anoxic conditions may have prevailed in some cases, and resulted in the dissolution of titanomagnetite.

Under deeper burial conditions (>500 m), many heavy minerals are unstable (Milliken and Mack, 1990). With depth, reducing conditions result in the leaching of iron, including Fe-rich lamellae, and its partial replacement with titanium oxide and pyrite (Milliken and Mack, 1990). This dissolution is progressive and selective, affecting the more reactive iron minerals first (Canfield *et al.*, 1992); magnetite is much more reactive than ilmenite. Thus, unless it is isolated from pore waters, the survivability of magnetite is limited. Although it will eventually alter (to TiO_2 or leucoxene, Dimanche and Bartholome, 1976), ferrian ilmenite persists in conditions which deplete magnetite (Reynolds *et al.*, 1994; Braun and Raith, 1985; Hounslow *et al.*, 1995). Following deep burial, therefore, a characteristic, relict, opaque mineral suite remains, consisting of only the most resistant magnetic minerals, including ferrian ilmenite and chromite.

During their journey from igneous and metamorphic source rocks, it is possible that the East Anglian sediments have been reworked several times, with each burial episode subjecting grains to dissolution and replacement. Following subsequent erosion, the most fragile grains would have subsequently been

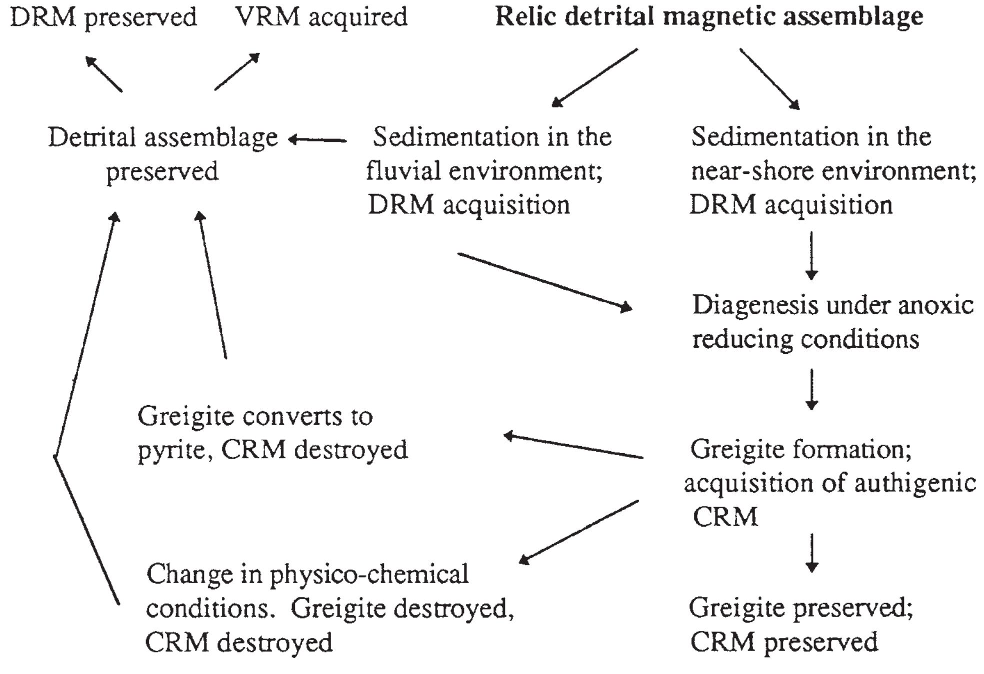

Figure 11 A model of remanence acquisition in the Early and Middle Pleistocene sediments of East Anglia

weathered and abraded in the mildly oxidising conditions of transport, removing, for example, skeletal titanium oxides. The detrital ferrimagnetic assemblage deposited by the proto-Thames as the Kesgrave Formation therefore consists mainly of silicates (with magnetic inclusions), with small quantities of ferrian ilmenites, chromites and titanium oxides.

The mineral assemblage deposited in the Crag Basin, on the palaeo-margins of East Anglia, has probably followed a longer and more complex pathway. Other than the proto-Thames, there is no evidence of deposition of fluvial sediment in this area during the time of Crag deposition, nor of pre-Anglian glacial transport of 'primary' sediment directly to the area. Subsequent to deposition, many of these marine sediments would have been subjected to yet more dissolution of iron minerals and hence the mineral assemblage is less diverse than that of the Kesgrave Formation, as shown by the mineralogical data here. XRD analysis also indicated that haematite, probably mostly occurring as inter-growths with ilmenite, was a relatively more important member of the magnetic assemblage for the Kesgrave Formation, than is the case for extracts from the marine sediments.

A model for remanence acquisition in these sediments, based on the evidence from this investigation and the information above, is summarised in Fig. 11.

Conclusions

1. A range of relatively unusual, relict and authigenic magnetic minerals occurs in the Early and Middle Pleistocene sediments of East Anglia and have acquired their NRMs via a variety of mechanisms. This information is key to assessing the fidelity of the palaeomagnetic record contained by such magnetite-poor sediments. Detrital iron oxides, including magnetite, have been largely removed from sediment during previous burial episodes, prior to reworking and final deposition in East Anglia.
2. The detrital carriers of NRM in the fluvial Kesgrave Formation consist mainly of ferrian ilmenites, iron–chromium spinels and (titano-)haematite. Some magnetic material (magnetite?) occurs as inclusions within host silicate minerals, especially quartz. Some magnetite possibly also occurs as intergrowths with ilmenite.
3. Probably as a result of their longer sediment transport pathway, and further dissolution of iron oxides during final burial, the detrital NRM carriers in the marine sediments investigated are dominated by ferrian ilmenite and chromite.
4. Unreliable polarity determinations for these sediments are common and attributable either to inefficient recording of the geomagnetic field by the magnetic carriers, or, more commonly, the acquisition of normal polarities where the timing of remanence acquisition is uncertain. In the latter case, the remanences are often VRMs, carried by low-blocking temperature, low-coercivity grains of ferrian ilmenite and iron–chromium spinels.
5. For those sites where reliable polarities were obtained, their NRM was acquired, at least in part, as a DRM. Important DRM carriers were ferrian ilmenite, titano-haematite, and, possibly, iron–chromium spinels.
6. The ferrimagnetic iron sulphide, greigite, is also an important carrier of NRM, especially in intertidal sediments. If greigite formation occurs 'contemporaneously' with sedimentation, then the CRM can be a reliable recorder of the geomagnetic field, acquired 'syn'-depositionally with respect to the GPTS.

Appendix 1: Summary of the sample investigation procedure

NRM measurement
↓↑ ↓↑
Stepwise AF demagnetisation Stepwise thermal demagnetisation
(sample unusable for further analysis)
↓
low-field susceptibility measurement
↓
frequency-dependence of susceptibility
↓
ARM acquisition (NRM destroyed)
↓
ARM demagnetisation
↓
Stepwise IRM acquisition
↓
Sample dried, weighed
Magnetic extraction → X-ray diffractometry
Optical microscopy
Electron microscopy
Energy dispersive X-ray analysis

Appendix 2: Analyses used for investigation of different sediment units. (GB = Great Blakenham)

Site/unit	No. samples	AF demag	Thermal demag. of:	Mineral mag.	Magnetic extract	Mineral mag. on extract	XRD	Optical micro-scopy	SEM EDXA	TEM EDXA
GB—Creeting Sands	33	•	NRM	•	•		•	•	•	
GB—College Farm Silty Clay	38	•	NRM (after AF demag.). 3-axis IRMs	•	•		•	•	•	
Thorpe St Andrew	13	•	NRM	•	•	•	•	•	•	•
South Cove Brickworks	76	•	NRM	•	• (2)		•	•	•	
Thorington	22	•	NRM	•	• (2)		•	•	•	
Caistor Pit	59	•	NRM	•	•		•	•	•	
Sheringham unit e	12	•	NRM	•	•		•	•	•	
Sheringham unit g	58	•	IRM	•	• (3)		•	•	•	
Stebbing	8	•	NRM (after AF demag.) 3-axis IRMs	•	•	•	•	•	•	
GB—Baylham Common Gravels	8	•	NRM	•	•	•	•	•		•
Drakes Farm	5	•	NRM (after AF demag.) 3-axis IRMs	•	•	•	•	•		•
Bradwell	6	•	NRM, NRM and AF demag.	•	•	•	•	•	•	
Springfield	37	•	NRM, NRM and AF demag.	•	• (2)	•	•	•	•	•
Ardleigh inorganic clay (upper gravel)	6	•	NRM (after AF demag.) 3-axis IRMs	•	•	•	•	•	•	
Ardleigh organic silts	20	•	NRM	•	•	•	•	•	•	
Ormesby Borehole	62	•	NRM	•	•		•			

Appendix 3: Additional information on unusual remanence carriers identified in East Anglian Plio/Pleistocene sequences

(a) Ferrian ilmenites

Ionic substitution results in a solid solution series between ilmenite and haematite, with a general formula $Fe_{2-X}Ti_XO_3$ ($0 < X < 1$). Ti^{4+} ions substitute for Fe^{3+} ions of the haematite end member, and the remaining cation changes valence to Fe^{2+}. For $0 < X < 0.45$, compositions are imperfectly antiferromagnetic, with low saturation magnetisations and higher coercivity (Nagata, 1961). For $0.45 < X < 1$, the Ti^{4+} cations are preferentially in alternate cation layers. This results in an antiparallel coupling of two unequal sublattices and a corresponding ferrimagnetism (as occurs in magnetite, for example). Coercivity is lowered and saturation magnetisation can be as high as 20–30 A m^2 kg^{-1} (Akimoto, 1955). The Curie temperature (T_c , i.e. the temperature at which magnetisation becomes zero) lies above room temperature for those compositions with X less than ~0.75. The maximum T_c for ferrimagnetic compositions (i.e. when $X > 0.45$) is approximately 270 °C. Critical in the context of NRM-carrying capacity is the blocking temperature of these minerals. (The blocking temperature is that at which a magnetic grain's behaviour changes from unstable superparamagnetic—with thermal agitation overcoming magnetic order—to stable, single domain.) Even though the T_c of these ferrian ilmenites may be relatively low (e.g. compared with magnetite, which has a T_c of 580 °C), if the blocking temperature is close to T_c, then these minerals can retain a stable NRM over geological timescales—especially if, as here, the sediments are held at surface environmental temperature. It is thus possible, given the presence in a detrital assemblage of sufficient quantities of ferrian ilmenite with blocking temperatures above environmental temperatures and high saturation magnetisation (i.e. with compositions in the range $0.45 < X < 0.75$), that those grains might acquire a stable DRM.

Ilmenite can also occur as inter-growths with magnetite. Basu and Molinaroli (1989) found that between 30% and 50% of detrital, opaque iron–titanium oxide minerals were polymineralic, being inter-growths of two or more phases. It is thus also possible that ferrimagnetism associated with ilmenite grains might often be due to intergrown magnetite lamellae. However, such lamellae would be expected to be of small effective grain size and have blocking temperatures close to those of magnetite (e.g. Dunlop and West, 1969).

(b) Iron–chromium spinels

A solid solution exists for Fe–Cr spinels (with the general formula $Fe_{3-X}Cr_XO_4 (0 \leq X \leq 2)$), with end members chromite and magnetite. Chromites representing a significant proportion of the relict magnetic assemblage in Triassic North Sea sediments, had a range of compositions, between chromite and magnetite, with the $CrFe_2O_4$ component comprising between 30% and 100% of grains (Hounslow *et al.*, 1995). No grain structure was observed at magnifications up to 1000 ×, suggesting the intermediate compositions were either the result of substitution or sub-micrometre exsolution. Freeman (1986) investigated magnetic extracts from pelagic limestones and found that chromites contributed significantly to the 1–20 µm grain size interval, with 44% of grains containing iron and chromium, in association with nickel or titanium.

Schmidbauer (1971) reported T_cs for the chromite–magnetite solid-solution series varying between 580 °C for the Fe_3O_4 end-member ($X = 0$), to 200 °C for $X = 1$, −30 °C for $X = 1.3$ and −193 °C for $X = 2$. Hounslow *et al.* (1995) assumed a linear decrease in T_c from 580 to −193 °C. Their observed chromite compositions correspond to T_cs below 250 °C, in accordance with their low blocking temperature NRMs.

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