# The magnetism of a glacial aeolianite sequence from Lanzarote (Canary Islands): coupling between luvic calcisol formation and Saharan dust trapping processes during wet deposition events off northwestern Sahara

David Williamson,<sup>1</sup> Mike Jackson,<sup>2</sup> Subir K. Banerjee<sup>2</sup> and Nicole Petit-Maire<sup>3</sup>

<sup>1</sup>Centre National de la Recherche Scientifique, CEREGE, BP 80, 13 345 Aix-en-Provence, Cedex 4, France. E-mail: davwill@cerege.fr <sup>2</sup>Department of Geology and Geophysics and Institute for Rock Magnetism, University of Minnesota, Minneapolis MN 55 455, USA <sup>3</sup>Centre National de la Recherche Scientifique, MMSH, BP 647, 13 094 Aix-en-Provence, Cedex 2, France

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# SUMMARY

In order to better document the climatic origin of pedogenized loess deposits west of Sahara, rock-magnetic measurements were performed on a Last Glacial coastal sand sequence from La Mala (LM) quarry (Lanzarote, Canary Islands) containing six interbedded loess–palaeosol units. Hysteresis and susceptibility data point to a coarse-grained magnetic enhancement in the coastal sand, which contrasts with the superparamagnetic (SP) to pseudo-single domain (PSD) behaviour of the Saharan loess and palaeosol. High- and low-temperature experiments show that oxidized titanomagnetite contributions dominate in the sand, while fine-grained (SP to PSD) iron oxidation products such as maghemite and goethite are evidenced in the Saharan loess/palaeosol units. At room temperature, the detrital PSD–multidomain titanomagnetic susceptibility. Surface oxidation of detrital spinel grains and authigenesis of fine-grained iron oxides (including SP goethite) are proposed to explain the magnetic properties of the primarily fine, Saharan-dust-bearing material.

The dry, local climate of the present-day and Late Holocene means that loess deposits are not preserved. The six pedogenized loess units, however, point to dust trapping under semi-arid, wetter conditions, probably illustrating periods of reduced latitudinal temperature gradients and climate variability of the North Atlantic climate, respectively. These findings suggest that both the Canary palaeosol and its content of (ultra)fine iron oxide might be constrained by (wet) deposition and trapping of fine Saharan dust.

**Key words:** glacial climate, loess magnetism, low-temperature magnetic behaviour, magnetic susceptibility, Saharan dust, titanomagnetite.

#### **1 INTRODUCTION**

The magnetic properties of loess/palaeosol deposits are strongly controlled by the composition and deposition rate of aeolian dust, and by the biogeochemical cycles of iron and carbonate in soils, respectively (Heller & Liu 1984; Kukla *et al.* 1988; Maher & Taylor 1988). In many loess/palaeosol sections from Eurasia, a characteristic pedogenic magnetic enhancement in superparamagnetic (SP) to pseudo-single domain (PSD) iron oxides (Zhou *et al.* 1990; Maher & Thompson 1991; Oches & Banerjee 1996) has been evidenced in interglacial palaeosol layers. Although there are various origins, the climate-dependent magnetic enhancement in soils often corresponds to a strengthened authigenesis of ultrafine (or SP) magnetite and maghemite under warm and wet conditions (Banerjee *et al.* 1993; Verosub *et al.* 1993).

So far, however, very few rock-magnetic investigations have been carried out on aeolian deposits located along the margins of subtropical deserts such as the Sahara. In the Matmata loess sequence (Tunisia), Dearing *et al.* (1996a) observed a contribution of SP grains in modern soils and palaeosols that apparently developed under an annual rainfall higher than ~250 mm yr<sup>-1</sup>. In this paper, we investigate the magnetic properties of the Late Pleistocene aeolianite/palaeosol sequence from La Mala quarry (29°06' N, 9°49' W), on the eastern coast of Lanzarote (Canary Islands) (Fig. 1).

The climatic and geological contexts of loess deposition in the Canary archipelago differ from those in most other loess regions. Unlike the case for periglacial mid-latitude regions, where pedogenesis developed under precipitations higher than  $\sim$ 500 mm yr<sup>-1</sup> (Liu *et al.* 1995), in the eastern Canary Islands relatively semiarid conditions probably prevailed during interglacial/interstadial



Figure 1. Site location of La Mala Quarry, Lanzarote Island, and lithostratigraphy of the upper La Mala aeolianite sequence. The shaded area represents the general area of influence of Saharan dust events in the region.

periods (Petit-Maire *et al.* 1987). Our aim is to document the magnetic signature and the environmental and climatic significance of such aeolianite material, which is found in most archipelagos west of Africa, and which contains the early stages of calcisol formation under semi-arid conditions and in peridesertic subtropical areas.

# 2 GENERAL SETTING

Aeolianite deposits from the islands of West Africa (Cape Verde, Canary Islands) provide an accessible end-member of the Saharan aerosol (Coudé-Gaussen *et al.* 1987; Swap *et al.* 1996) as transported across the North Atlantic Ocean during dust outbreaks over Morocco and northwestern Africa (Robinson 1986; Grousset *et al.* 1992; Herwitz *et al.* 1996).

In Fuerteventura and Lanzarote islands (eastern Canary Islands), the present-day arid climate (mean annual rainfall <100 mm) favours the deflation of outcropping unconsolidated sediments by northeasterly winds, preventing the development of accretionary soils. However, Late Quaternary aeolianite deposits are found in the Canary Islands, for example the La Mala (LM) aeolianite sequence. At subtropical latitudes, these deposits are often associated with fossil coastal dunes that developed under stronger wind speeds during the Last Glacial period (Rognon & Coudé-Gaussen 1996). Loess and palaeosols interbedded in these aeolianites contain biological, mineralogical and geochemical evidence for relatively humid episodes since oxygen-isotope stage 3 (Petit-Maire *et al.* 1986; Hillaire-Marcel *et al.* 1995).

The 24-m thick Late Pleistocene aeolianite record from La Mala quarry is divided into eight superimposed sequences (A1, A2, B, C1 to C5) (Fig. 1). Except for the Bter loess/palaeosol, the B sequence consists of a  $\sim$ 15-m thick fossil dune. Six palaeosol horizons are defined for the upper 7 m of the sequence (Bter to C5). The uppermost C5 palaeosol is capped by a massive Early Holocene calcrete (Zöller *et al.* 2003). Laminar calcretes crop out in many areas of Lanzarote and Fuerteventura islands, and were formed during conditions more humid than today (Coudé-Gaussen & Rognon 1988; Meco *et al.* 1997; Alonso-Zarza & Silva 2002).

Three dominant facies are observed, corresponding to the dominant contributions of coastal sands, Saharan dust (loess), and su-

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perimposed palaeosol end-members, respectively (Chamley *et al.* 1987; Coudé-Gaussen *et al.* 1987; Damnati *et al.* 1996). Coastal sands are the main component of LM deposits (>60 per cent of the bulk deposit). They typically consist of coarse calcitic and aragonitic bioclasts (63–400  $\mu$ m), and basaltic minerals (augite, olivine, chlorite, titanomagnetite, smectite, palygorskite) derived from the erosion and weathering of the Canary basalts.

Saharan dust-derived 'loess' has a unimodal grain size distribution (20 to 40  $\mu$ m), with characteristic mineral assemblages of Fe-coated quartz, micas, and carbonates. Illite, chlorite and kaolinite are the main clay minerals and represent more than 50 per cent of the clay fraction. The illite-kaolinite enhancement in the LM loess testifies to an African origin (Damnati *et al.* 1996; Caquineau *et al.* 1998). A comparison of mineralogical and Nd–Sr isotopic signatures of the Canary loess with present-day observations of dust outbreaks in southern Morocco suggests that the main part of the dust (>65 per cent) originates from northwestern Sahara (Coudé-Gaussen *et al.* 1987; Grousset *et al.* 1992; Rognon *et al.* 1996).

Palaeosols corresponding to pale-brown to yellowish brown (Munsell colour 10YR 6/3 to 6/4) luvic calcisols (Driessen & Deckers 2001) are observed in the uppermost parts of the loess sections of the LM profile. Strikingly, these palaeosol horizons systematically develop between underlying loess and overlying sand. Compared with the underlying weakly pedogenized loess horizons, the carbonate-dominated silt matrix of the palaeosols is enriched in authigenic clay minerals (montmorillonite, interstratified clays, palygorskite).

The palaeosols present a characteristic upward enhancement in fossil *Helicidae* shells and *Anthophora* nests, consolidated and cemented by silt, sand and calcium carbonate. Although the abundance of fossil *Hymenoptera* nests and land snails (mostly *Theba geminata, Rumina decollata*, and *Pomatias laevigatum*) attest to slightly wetter conditions (mean annual rainfall ~300 mm) than today at the time of palaeosol formation (Meco *et al.* 1997), the considerable concentrations of nests and snails in some palaeosols (more than 300 nests  $m^{-2}$ ) probably illustrate the differential erosion of soil particles by wind after soil formation. Therefore, the upper palaeosol units in La Mala quarry are, most probably, only partly preserved.

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Although the detailed chronology of the glacial, 'Erg Ogolien' (e.g. Zöller et al. 2003) low-sea-level dune of La Mala remains to be established, lithostratigraphies very similar to that of the LM section are found in the most preserved aeolianite deposits from Fuerteventura and Lanzarote. The chronology of the upper palaeosols relates to humid episodes of the Late Glacial-Early Holocene period (Petit-Maire et al. 1986, 1987; Damnati et al. 1996; Rognon & Coudé-Gaussen 1996; Zöller et al. 2003). In a neighbouring aeolianite section (~20 km west of La Mala), a comparison of radiocarbon, amino acid and U-series chronologies obtained from fossil land snails indicates that the six most recent palaeosols are interbedded in an aeolianite sequence deposited between ca. 50 and 30 kyr BP (Hillaire-Marcel et al. 1995). The coherent succession of lithostratigraphic units in both sites suggest that the La Mala loess/palaeosol record mostly represents a succession of six wet spells between Marine Isotope Stage 3 and the Early Holocene.

#### 3 MATERIALS AND METHODS

26 bulk sediment samples characteristic of the main lithostratigraphic units were collected for a variety of studies, including some in palaeobiology and sedimentology. Three additional samples from the catchment basalts close to La Mala (GU-3, Bruhnes age; GL16, Matuyama age, and SH5, Miocene age) were also taken for comparison.

The magnetic properties were measured on the fine (<63  $\mu$ m) sieved fraction to increase the contribution of Saharan dust at the expense of coastal sandy material. Additional pilot samples were prepared for thermomagnetic analysis: (i) mixtures of sand (SA), loess (L) and palaeosol (PSL); (ii) clay-sized fractions (<2  $\mu$ m) of palaeosol samples PSL (SMIX) and C5-180 (SC5), obtained by centrifugation; and (iii) a magnetic concentrate of palaeosol C2-70 (fraction >63  $\mu$ m), obtained with a static magnet (0.2 T) connected to a closed circuit containing the sample in solution.

The magnetic measurements were performed at the Institute for Rock Magnetism at the University of Minnesota. After drying at 50°C, the sediment was gently mixed and packed in non-magnetic gel caps containing ~220 mg of dry sediment. Additional sets of the fine fraction were kept for CaCO<sub>3</sub> analysis (coulometry), and high-temperature (HT) measurements. The magnetic values were normalized to the carbonate-free mass of the samples, after removal of the diamagnetic susceptibility of calcite and aragonite ( $\chi_{CaCO3} \sim -0.5 \times 10^{-9} \text{ m}^3 \text{ kg}^{-1}$ ). HT samples were prepared by mixing the dry sediment with a high-temperature diamagnetic cement (Omega CC).

Hysteresis loops and isothermal remanence curves were measured on a Micro-Mag Vibrating Sample Magnetometer (µVSM, Princeton Measurements). High-field susceptibility values  $(\chi_{hf})$ were obtained from the high-field slope between 0.7 and 1 T. Magnetic susceptibility measurements, including field dependence, frequency dependence, and low-temperature dependence of susceptibility, were performed on a Lakeshore model 7130 AC susceptometer. The susceptibility was measured at 21 frequencies between 40 and 6000 Hz (operating field: 80 A  $m^{-1}$ ). The resulting frequency dependence ( $\chi_{FD}$ ) between 470 and 4700 Hz was calculated from a best fit through the data points. The field dependence of AC susceptibility was measured at nine applied fields ranging from 30 to 2000 A  $m^{-1}$  (operating frequency: 40 Hz). The magnitude of the field dependence  $\chi_{ACD}$  ( $\chi_{ACD} = \chi_{1000 A m^{-1}} - \chi_{30 A m^{-1}}$ ) was also quantified by the (percentage) increase of susceptibility between 30 and 1000 A m<sup>-1</sup>:

# $\chi_{ACD \, per \, cent} = 100 (\chi_{1000 \, A \, m^{-1}} - \chi_{30 \, A \, m^{-1}}) / \chi_{30 \, A \, m^{-1}}$

Thermomagnetic curves were determined on the  $\mu$ VSM in an inert helium gas atmosphere, by measuring either the total magnetization in a 1.6-T field, or the HT hysteresis cycles in a few selected samples. HT susceptibility measurements (in argon gas) were performed on the Geofyzika kappabridge CS2 for the pedogenized loess samples Bt-50 and Bt-100, and for the GU-3 basalt.

The MPMS magnetometer (Quantum Corp.) was used to measure the low-temperature behaviour of remanences imparted in a strong DC field (2.5 T). The zero-field-cooled remanence ( $Mr_{ZFC}$ ) was acquired in a 2.5-T field at 10 K, after cooling the sample in zero-field. The field-cooled remanence ( $Mr_{FC}$ ) was acquired by re-cooling from 300 to 10 K in a 2.5-T field. Both  $Mr_{ZFC}$  and  $Mr_{FC}$  curves were measured during warming from 10 to 300 K in zero-field. Additional low-temperature susceptibility curves were measured on the Lakeshore susceptometer between 30 and 300 K. Susceptibility was measured during warming at variable AC field strengths and frequencies.

# 4 STRATIGRAPHIC CHANGES AND MAGNETIC PROPERTIES AT ROOM TEMPERATURE

As shown in Fig. 2, the fine fraction percentage ( $<63 \mu m$ ) of the sediment reflects the lithology, with minimum values in the coastal sand and maximum values in loess and palaeosol horizons. In contrast, the high carbonate content (>40 per cent) of the fine fraction is poorly correlated with the lithology. This is not surprising, because (biogenic) calcium carbonate is a significant fraction of coastal sand (marine shell debris), Saharan dust, and palaeosol (cemented nests, snail debris, authigenic carbonates).

Given the very low diamagnetic susceptibility of CaCO<sub>3</sub>, the magnetizations and the susceptibilities were corrected for the CaCO3 'dilution' effect. The bulk and carbonate-free records of saturation magnetization  $M_s$ , low- and high-field magnetic susceptibilities ( $\chi_{lf}$ and  $\chi_{\rm hf}$ , respectively) show very similar features, indicating that the magnetic signature of the fine fraction does not primarily depend on dilution by calcium carbonate. Magnetic 'concentration' proxies such as  $M_{\rm s}$ ,  $M_{\rm rs}$ , HIRM,  $\chi_{\rm lf}$ , and  $\chi_{\rm hf}$ , show maximum values in the coastal sand horizons, pointing to the dominant contribution of local, strongly magnetic minerals in this material enriched in local basaltic products. In addition, magnetizations and susceptibilities in units Bter, C1, C2, C3 and C4 also tend to increase upwards from the weakly pedogenized loess to the palaeosol horizons. As suggested for the Tunisian loess (Dearing et al. 2001), the latter observation indicates that a pedogenic magnetic signature could occur in peri-Saharan areas under relatively semi-arid conditions (rainfall  $\leq$  300 mm yr<sup>-1</sup>).

Hysteresis parameter ratios  $M_{\rm rs}/M_{\rm s}$  and  $H_{\rm cr}/H_{\rm c}$  display little variation through the LM sequence (Figs 1 and 2). The relationship between these ratios and the stratigraphy is not straightforward, although the loess and palaeosol horizons tend to show higher  $H_{\rm cr}/H_{\rm c}$  values than coastal sands. According to the Day plot (Day *et al.* 1977), a pseudo-single domain (PSD) behaviour is inferred for LM aeolianites, with relatively constant  $M_{\rm rs}/M_{\rm s}$  values (0.19 to 0.24), and, more significantly, mostly  $H_{\rm cr}$ -controlled changes in  $H_{\rm cr}/H_{\rm c}$  (1.3 to 2.6) (Fig. 4). High  $H_{\rm cr}$  and  $H_{\rm cr}/H_{\rm c}$  values are associated with a slight wasp-waistedness of hysteresis loops, suggesting a multicomponent magnetization with variable contributions of hard and soft coercivity components (Jackson 1990; Roberts *et al.* 1995).



Figure 2. The La Mala aeolianite stratigraphic record: (i) lithology (sand: spaced dots; loess: dense dots; palaeosol: hatched area); (ii) labelled stratigraphic subunits; (iii) carbonate content; (iv) silt content; (v) bulk (closed circles) or carbonate-free (open circles) changes in saturation magnetization, low-field susceptibility, high-field susceptibility; (vi) magnetization and coercivity ratios.

In aerial continental deposits such as loess/palaeosol sequences, the potential influence of hard magnetic minerals such as haematite and goethite on  $H_{\rm cr}$  should not be excluded. However, significant contributions of such minerals should also affect the *S* ratio. In La Mala samples, *S* values (S > 0.93) as calculated following the formula of Bloemendal *et al.* (1992) are remarkably high and stable, and do not clearly co-vary with  $H_{\rm cr}$  (Fig. 3), suggesting little control of stable goethite or haematite components on hysteresis at room temperature.

Coercivity changes are therefore probably driven by the compositional variation of spinel assemblages, which may present variable oxidation state, Ti substitution, and/or variable blocking volume close to the superparamagnetic (SP)/single-domain (SD) boundary (Tauxe *et al.* 1996). Potential contributions of viscous SP–SD haematite or goethite grains should not be excluded; neither should oxidized grains of (Ti-)magnetite. Indeed, it has been shown for a variety of loess sequences (van Velzen & Dekkers 1999) that partial, low-temperature oxidation of fine magnetite is responsible for considerable changes in  $H_{cr}$  due to internal strain and compositional boundaries.

Soil contributions in SP and viscous SD (secondary-) ferrimagnetic oxides would be inferred from relatively high values of  $\chi_{\text{FDpercent}}$  and  $\chi_{\text{ferro}}/M_s$ , respectively (Banerjee *et al.* 1993; Forster *et al.* 1994; Hunt *et al.* 1995a; Dearing *et al.* 1996b). In the La Mala aeolianites,  $\chi_{\text{FDpercent}}$  and  $\chi_{\text{ferro}}/M_s$  values are slightly higher in the loess/palaeosol units (2–2.5 per cent and 1–1.2 × 10<sup>-5</sup> m A<sup>-1</sup>, respectively) than in the coastal sand units (~1 per cent and 0.7–1 × 10<sup>-5</sup> m A<sup>-1</sup>, respectively), possibly indicating a relative enhancement in SP to PSD grains in the former horizons (Fig. 3). Unlike in the case of Chinese loess (Forster *et al.* 1994), however, the mass-normalized frequency dependence  $\chi_{\text{FD}}$ , which co-varies with most (ferri-)magnetic-concentration proxies, shows high values in coastal sands, and maximum values in Quaternary basalts GU-3 and GL-16 ( $\chi_{FD} > 1.8 \times 10^{-7} \text{ m}^3 \text{ kg}^{-1}$ ). This further suggests that the frequency dependence of aeolianites might be partly constrained by local, basalt-originated material. Moreover, given the occurrence of titanomagnetite (with variable Ti content) in the Canary basalt (Carrecedo & Soler 1995; Quidelleur & Valet 1996; Szeremeta *et al.* 1999; Valet *et al.* 1999; Leonhardt *et al.* 2000), the potential effect of titanomagnetite composition on the frequency and field dependence of susceptibility of LM aeolianites should be considered (Jackson *et al.* 1998; de Wall 2000).

Improvements in determining the magnetic mineralogy and grain size of the aeolianites were made on pilot samples characteristic of the main lithological end-members; that is, palaeosol (samples PSL, GC2-70, FC2-70, Bt-100, GC5-180), loess (L, Bt-50), coastal sand (SA, GC1-20, C3-120) and Lanzarote basalt (GU-3, SH5) end-members. Additional measurements were performed on the clay-sized fraction (<2  $\mu$ m) of palaeosol samples (SMIX and SC5-180).

# 5 THERMAL DEMAGNETIZATION OF IRM

A thermal demagnetization of IRM in air was applied to sand (S), palaeosol (PSL) and loess (L) mixtures. Three orthogonal IRMs were tentatively imparted at 1.4, 0.3, and 0.05 T. The resulting 'hard'  $IRM_{0.3-1.4T}$ , 'medium'  $IRM_{0.05-0.3T}$  and 'soft'  $IRM_{0.05T}$  demagnetization curves present similar unblocking temperatures (Fig. 5), probably attributable to the imperfectly orthogonal acquisition of magnetization in a dominantly soft material. Major IRM unblocking occurs around 200–350°C, 450–500°C and 650°C, with slightly lower unblocking temperatures for the sand compared with the finer



Figure 3. Coercivity changes and magnetic mineralogy/grain size proxies: (i) lithology; (ii) stratigraphic subunits; (iii) coercivity ratio; (iv) *S* parameter; (v) frequency dependence of susceptibility; (vi) ferromagnetic susceptibility/saturation magnetization ratio (comments in the text).



Figure 4. Day–Fuller plot of LM aeolianite hysteresis properties.

loess/palaeosol material. The 200–350°C drop suggests unblocking of a viscous spinel phase, while the 450–500°C drop is consistent with a Ti-poor spinel phase. Finally, the  $\sim$ 600–650°C unblocking illustrates the presence of maghemite or haematite.

# **6 CURIE TEMPERATURE**

Thermomagnetic curves in a 1.6-T field and in argon were obtained for sand (SA), loess(L) and palaeosol (PSL) mixtures, and for the clay-sized fraction (<2 µm) of the palaeosol mixture (SMIX). In addition, HT hysteresis yielded changes in high-field slope-corrected magnetization  $(M_{sc})$  at 0.3 T for a larger set of samples, including the GU-3 basalt and a magnetic extract from palaeosol C2-70. M-T or  $M_{sc}-T$  curves are observed to be similar to each other. The final Curie points (Fig. 6) range between 550°C (SA) and 590-600°C (L, PSL, FC270), consistent with Ti-poor magnetite/maghemite. As observed in (Ti-)maghemite (Özdemir & Banerjee 1984; Özdemir 1987),  $T_{\rm C}$  values tend to decrease during the cooling cycle (in our case, by 10 to 40°C), and/or during longer high-temperature treatment (hysteresis). Such behaviour can also be observed on numerous thermomagnetic curves from Lanzarote basalts (Carrecedo & Soler 1995). Unlike the case for IRM thermal unblocking curves obtained in air, no Curie point of 600-650°C is found after heating in argon. Most probably, the alteration of (Ti-)maghemite and/or iron-bearing clays or oxy-hydroxides in argon tends to favour the precipitation of near-stoichiometric magnetite, which overwhelms any residual contribution of the original high  $T_{\rm C}$  mineral.

In contrast, the GU-3 basalt shows a  $T_{\rm C}$  of 195°C, pointing to the significant contribution of a Ti-rich iron oxide such as titanomagnetite ( $x \sim 0.6$  or TM60), or ferrimagnetic hemoilmenite ( $y \sim 0.55$ ). Additional magnetization drops at 440 and 550°C are consistent with the behaviour of a highly oxidized TM60 ( $z \sim 0.6$ – 0.8), which inverts above 400°C into a spinel phase near magnetite in



Figure 5. IRM thermal demagnetization curves of lithological endmembers. Open circles: 'hard' component (0.3–1.4 T); closed circles: soft component (0–50 mT).

composition (Özdemir 1987). Thus, (oxidized) TM60 is probably a well represented mineral in the local basalt, together with previously reported (oxidized) magnetite (Carrecedo & Soler 1995).

Unlike the IRM thermal demagnetization, where oxidizing conditions probably prevailed during the experiment, high-temperature measurements in argon probably favoured the reduction of iron oxides, resulting in a dominant magnetite-like component. Despite the magnetite-like component, the behaviour of relatively weakly magnetized phases during heating is suggested from M-T curves. Magnetization drops at 80°C (GU-3 basalt) or 110°C (pedogenized loess GC2-70 and sand GC1-20), followed by slight increases at 90°C or 130–140°C, which might be respectively associated with the increased ferromagnetism of goethite close to its Néel temperature (de Boer & Dekkers 1998; Mathé *et al.* 1999), or to the release of strain in partly oxidized (Ti-)magnetite (van Velzen & Dekkers 1999).

#### 7 HIGH-TEMPERATURE SUSCEPTIBILITY

Thermomagnetic  $\chi_{1f}$ -*T* runs on pedogenized loess samples (Bt-50 and Bt-100) show irreversible susceptibility changes (Fig. 7), as generally observed in loess deposits. Such changes differ little when running the experiment in argon or in air, suggesting that the air flow in the kappabridge furnace is too low to ensure an additional oxidation of aeolianites. Both samples show a reversible  $\chi_{1f}$  increase between 20°C and ~100°C, followed by a first irreversible increases between 100°C and 200°C. The major irreversible  $\chi_{1f}$  increases, however, occur at ~250–300°C and above 350°C.

The two latter temperature intervals correspond to drops in remanent magnetization, indicating that the thermomagnetic behaviours of the (pedogenized) aeolianites mostly result from structural transformations of the original minerals.

Given the occurrence of goethite and montmorillonite in the pedogenized aeolianites from the Canary Islands, the irreversible thermomagnetic changes above respectively ~180°C and ~350°C result at least partly from the surface alteration of iron-bearing clays and iron oxy(hydr)oxide coatings (Hirt *et al.* 1993; van Velzen & Dekkers 1999). Indeed, the adsorbed iron at the surface of montmorillonite tends to transform between 200°C and 500°C into magnetite/maghemite particles, which further transform into haematite (Hirt *et al.* 1993). Lepidocrocite ( $\gamma$ -FeOOH) dehydrates into SD maghemite above 250°C, which in turn inverts to haematite above 400°C (Özdemir & Dunlop 1993). In the same way, the dehydration of goethite ( $\alpha$ -FeOOH) into haematite can be associated with the production of metastable magnetite above 240°C, the latter finally oxidizing into haematite above 350°C (Lima-de-Faria 1967; Özdemir & Dunlop 2000).

The production of magnetite in reducing conditions for palaeosol samples (SMIX, C5-180) would be in agreement with the alteration of iron-loaded smectite and/or the dehydration of FeOOH. However, the production of magnetite-like spinel above  $400^{\circ}$ C is not restricted to the fine, clayey samples: it also characterizes the sandy fraction of aeolianites. Moreover, the original high (> $600^{\circ}$ C) IRM unblocking temperatures of dominantly soft material tends to disappear during treatment in neutral (reducing) atmosphere. Therefore, the contribution of titanomaghemite to the observed irreversible changes above  $400^{\circ}$ C is probably significant.

#### 8 LOW-TEMPERATURE REMANENCE

Field-cooled ( $Mr_{FC}$ ) and zero-field-cooled ( $Mr_{ZFC}$ ) remanence warming curves are shown in Fig. 8 for (i) local basalt (GU3), (ii) sand (C3-120), (iii) coarse (C2-70, 150–63 µm), fine (Bt-75, <63 µm) and clay-sized fractions (SMIX) of pedogenized loess. A synthetic goethite powder (GTS3) available at the Institute for Rock Magnetism was also measured for comparison.

A characteristic feature of aeolianite  $Mr_{ZFC}-T$  curves is the strong (>50 per cent), gradual decrease of magnetization between 20 K and 300 K, pointing to a progressive unblocking of remanence upon warming. This is especially noticeable in the finer samples such as the clay-sized pedogenic fraction SMIX, suggesting the SP



**Figure 6.** Thermomagnetic *M*–*T* (mostly warming) curves of characteristic LM samples.

behaviour of fine particles upon warming, or the progressive, linear decrease of the defect moment of goethite below its Néel temperature (Rochette & Fillion 1989). However, similar behaviour also characterizes relatively coarse (C2-70) and non-pedogenized sand (C3-120) samples, where significant (ultra)fine particle contributions and/or goethite enhancements are not expected. The gradual  $Mr_{\rm ZFC}$  decrease in such coarse samples rather indicates the thermal unblocking of MD titanomagnetite grains, where the considerable decrease in anisotropy and magnetostriction with increasing temperature cause the initial remanence to decrease remarkably (Moskowitz *et al.* 1998).

Further additional magnetization drops (of a small percentage) are identified in aeolianite samples below ~50 K and between ~80 and 120 K, respectively. The ~50 K change is also observed on the basalt, and locates close to the Néel temperature of ilmenite (Hunt *et al.* 1995b), or to the phase transition in Ti-poor titanomagnetite (x < 0.4) (Moskowitz *et al.* 1998). The blurred 80–110 K drop is a slight indication of the Verwey crystallographic phase transition of magnetite, which occurs at  $T_V \sim 120$  K for stoichiometric magnetite (Hunt *et al.* 1995b). However, the large temperature interval of this transition in our samples clearly indicates a departure

from stoichiometry, which may be ascribed to titanium substitution (Moskowitz *et al.* 1998), or surface oxidation of magnetite grains into a shell of (SP) maghemite (Özdemir *et al.* 1993).

The field-cooled curves are consistently higher than zero-fieldcooled curves, indicating the efficiency of cooling in the field: at 20 K,  $Mr_{\rm FC}$  values are up to 20 per cent higher than  $Mr_{\rm ZFC}$  values (sample Bt-75). As observed for the synthetic goethite sample GTS3, the divergence ( $\Delta = Mr_{\rm FC} - Mr_{\rm ZFC}$ ) gradually decreases upon warming for the finer pedogenized loess (SMIX and Bt-75), and remains positive until room temperature. In the coarser samples, the divergence primarily drops abruptly below ~50 K, and then shows a second gradual decrease from  $T_{\rm V}$  (100–120 K) up to 270 K at least.

The maximum divergence below 50 K is consistent with the high coercive force of a weakly ferromagnetic hemo-ilmenite below the Néel temperature  $T_N$ , and/or a considerable increase in anisotropy of Ti-poor magnetite (x < 0.4) below its isotropic point (Moskowitz *et al.* 1998). Similarly, relatively high  $\Delta$  values until ~100–120 K (samples Bt-75, C2-70, C3-120) are probably caused by the strong coercivity and anisotropy of monoclinic magnetite-like SD grains below  $T_V$ .



**Figure 7.** Thermomagnetic  $\chi$ -*T* curves (in argon) of pedogenized loess samples Bt-50 and Bt-100, showing irreversible magnetic transformations.

The persisting divergence at temperatures higher than  $T_V$  was not observed in magnetite grain assemblages of various origins (Moskowitz *et al.* 1998). In the finest pedogenized samples (SMIX), the unblocking of fine, hard antiferromagnetic goethite grains becoming superparamagnetic upon warming probably produces such divergence, this further explaining the soft signature of the aeolianite at room temperature.

A persisting divergence above  $T_{\rm V}$  might also reflect the domainwall mobility in PSD grains experiencing considerable thermaldependent changes in strain and anisotropy upon cooling, as is the case for fine oxidized magnetite (Smirnov & Tarduno 2000; Kosterov 2001, 2003). In such grains, the departure from stoichiometry causes the alignment of the easy axis of magnetization along the field direction upon cooling below  $\sim 80$  K, and the decreased mobility of domain walls at lower temperatures favours the stabilization of metastable, SD-like behaviour of saturated phases. A similar offset between field-cooled and zero-field-cooled SIRM demagnetization curves could also be invoked for Ti-rich magnetite (x > 0.4) particles, where the low domain-wall displacement energy at room temperature (Jackson et al. 1998) considerably increases at low temperatures, together with large and steep increases in magnetocrystalline anisotropy, magnetostriction, and coercivity (Moskowitz et al. 1998).

Therefore, the low-temperature remanence experiments point to a dominant contribution of titanomagnetite in the Canary aeolianites, with contributions of SP goethite and SP to PSD oxidized magnetite in the finest (pedogenized) dust fraction.



**Figure 8.** Low-temperature remanence curves (see explanations in the text). Closed circles: zero-field-cooled remanence  $Mr_{ZFC}$ ; open circles: field-cooled remanence  $Mr_{FC}$ ; crosses: divergence  $\Delta$ .

#### 9 LOW-TEMPERATURE SUSCEPTIBILITY

The in-phase susceptibility ( $\chi'$ ) of characteristic aeolianites was measured at variable frequency and AC amplitude to identify the



Figure 9. Low-temperature in-phase susceptibility curves ( $\chi'$ ) of aeolianite samples: (a) frequency-dependent measurements; (b) AC-field-dependent measurements.

potential contributions of titanomagnetite and/or superparamagnetic particle assemblages (Fig. 9). The general susceptibility increase upon warming from 30 to 300 K indicates little contribution of paramagnetic phases. As previously suggested from Mr-T

curves, this  $\chi'$  increase might be caused by the progressive unblocking of fine particles, which become SP upon warming. Unlike SP effects, however, measurements at variable frequency (Fig. 11a) do not show any significant increase in  $\chi_{FD}$  upon warming. Instead, the



**Figure 10.** FORC diagram of the calcisol fine fraction SMIX. The horizontal axis represents  $\mu_0 H_c$  (coercivity) and the vertical axis represents  $\mu_0 H_u$  (indicative of magnetic interaction). For each diagram, the saturation field was 1 T, magnetizations were measured at 1 s intervals, and a set of 99 FORCs was measured. The field increment was (a) 1.8 mT for the normal-resolution diagram, and (b) 0.8 mT for the high-resolution diagram. In both diagrams, a high smoothing factor (SF = 7) was used to remove the noise (see comments in the text).



**Figure 11.** Binary plot of the frequency dependence versus AC-field dependence. Open squares: sand; open circles: loess; solid circles: palaeosol end-member. Maximum  $\chi_{FD}$  per cent and minimum  $\chi_{ACD}$  per cent values are observed in the fine clayey fractions SC5-180 and SMIX.

susceptibility shows a slightly higher frequency dependence below 150 K, especially in the coarse samples. Strikingly, the  $\chi'$  increase above ~150 K is associated with a significant AC dependence in fields above ~100 A m<sup>-1</sup>, pointing to irreversible domain-wall displacements in low fields, as observed in titanomagnetite at room

temperature (Jackson *et al.* 1998; de Wall 2000). Such  $\chi'$  and ACdependent behaviours are also observed in the local basalt GU-3, and probably result from the thermal dependence of magnetocrystalline anisotropy (and magnetostriction) in multidomain grains of Ti-rich magnetite (x > 0.5), which causes the anisotropy to decrease up to room temperature (Moskowitz *et al.* 1998; de Wall 2000). In this context, the observed frequency-dependent susceptibility below 150 K probably also results from the low-temperature behaviour of titanomagnetite (e.g. Radhakrishnamurthy & Likhite 1993; Moskowitz *et al.* 1998).

In addition to the general  $\chi'-T$  increase upon warming, the sand (C3-120) and the coarse pedogenic fraction (PSLME) present a sharp  $\chi'$  increase until 50–60 K, followed by a peak at 100–115 K. The 50–60 K  $\chi'$  maximum is consistent with the magnetic ordering of weakly ferromagnetic hemo-ilmenite at  $T_N \sim 50$  K. However, the relatively high frequency dependence in this temperature range, together with the shift of the  $\chi'$  increase towards higher temperature with increasing frequency, suggests instead a relaxation process similar to those observed in Ti-poor magnetites (x < 0.4) (Radhakrishnamurthy & Likhite 1993; Moskowitz *et al.* 1998).

The small  $\chi'$  increase at 100–115 K occurs at a lower temperature than the isotropic temperature of cubic magnetite ( $T_i \sim 130$  K), where the first magnetocrystalline anisotropy constant  $K_1$  changes sign and vanishes (O'Reilly 1984). Unlike the case for stoichiometric magnetite, the observed change might result from Ti impurities in magnetite (Moskowitz *et al.* 1998).

#### **10 DISCUSSION**

#### 10.1 Magnetism and surface oxidation

Room-, high- and low-temperature magnetic measurements as performed on Lanzarote (pedogenized) aeolianites indicate heterogeneous magnetic mixtures, mostly controlled by the variable content and oxidation of primary titanomagnetite and magnetite assemblages, and by the probable formation of SP goethite-like coatings in the fine pedogenized dust. In this context, the slight  $H_{\rm cr}$ -controlled changes in hysteresis shape are driven by the combined effects of Ti-substitution and SD–PSD content, and by oxidation processes. High-Ti content and dominant MD behaviour of sands result in relatively lower  $H_{\rm cr}$  values, while low-temperature oxidation and smaller grain sizes in pedogenized loess result in relatively higher  $H_{\rm cr}$  values.

Our data also strongly suggest that the oxidation of local basaltic titanomagnetite controls the properties of coastal sands. The occurrence of multidomain (oxidized) Ti-rich magnetite in the coarse aeolianite fraction is especially evidenced from the strong increase in (AC-dependent) susceptibility above 150 K.

In this context, the additional low-temperature features of highfield remanence and (frequency-dependent) susceptibility do not clearly differentiate between the potential contributions of weakly ferromagnetic hemo-ilmenite and fine magnetite grains. First, a variable Ti content in stoichiometric titanomagnetite may result in the same magnetic changes near 50 K or 100–120 K as those of ilmenite or fine-grained magnetite, respectively (Moskowitz *et al.* 1998). Second, the fine grains of the two latter phases generally coexist with titanium-rich magnetite in natural basalts (Zhou *et al.* 1997).

The surface oxidation of primary (coarse titanomagnetite  $\pm$  fine magnetite) assemblages can be inferred from the observation, in coarse and fine fractions, of high-temperature irreversible changes, the smoothed and blurred shape of the low-temperature remanence and susceptibility changes, and the divergence between field-cooled and zero-field-cooled remanences below room temperature. In the coarse fraction, isolated SP-SD phases are unlikely to occur as single isolated particles: this indicates that the primary detrital fraction probably contains a non-negligible component of fine, partly oxidized magnetite.

Considering that surface processes are more effective in small particles due to their higher surface-area-to-volume ratio, the absence of low-temperature transitions and the continuous  $\Delta$  decrease from 150 K up to room temperature in the fine fraction is not surprising, because SP to PSD grains of maghemite and goethite would dominate in such a material.

In order to further observe potential contributions from stable, hard goethite particles, first-order reversal curve (FORC) diagrams (Pike et al. 1999), which allow distinct evaluations of magnetic hardness ( $H_c$ , horizontal axis) and interaction ( $H_u$ , vertical axis), were experimentally measured on the fine fraction (SMIX). The low-resolution diagram (Fig. 10a) shows that the density of contour lines is highest for  $H_c$  values lower than 40 mT, precluding a significant contribution of stable goethite (Roberts et al. 2000). The triangular shape of the contour lines, a characteristic of interactions in MD-like particles (Roberts et al. 2000), was not confirmed at higher resolution (Fig. 10b), where rather elongated ( $H_u$  < +5 mT), SD-like FORC distributions centred around  $H_c$  values of 10-18 mT are observed. In FORC diagrams, the thermal relaxation of SP and small SD particles yields vertical contour lines with maximum contour density at  $H_c = 0$  (Pike *et al.* 2001). Unfortunately, the high smoothing factor (SF = 7) as used for our weakly magnetized sample precluded such evaluation. Although such FORC diagrams confirm the minimal contribution of stable goethite in the Canary aeolianite, it is also obvious that the strongly smoothed FORC distribution of our weakly magnetized sample does not elucidate its mixture of soft magnetic particles, as previously observed by van Oorschot et al. (2002) for similar samples. In contrast, the lowtemperature behaviour of the Canary pedogenized aeolianite points to a specific contribution of SP goethite and SP to PSD maghemite, as evidenced in other palaeosol horizons with low-temperature techniques (Bógalo *et al.* 2001).

Based on surface oxidation, the average original size of the aeolianite material would be the primary factor in the determination of its magnetic mineralogy. This is further supported by the comparison, at room temperature, of the frequency and AC-field dependences of magnetic susceptibility ( $\chi_{FD}$  per cent and  $\chi_{ACD}$  per cent, respectively) (Fig. 11). Strikingly, minimum frequency dependences correspond to maximum AC-field dependences, and vice versa. In PSD and MD titanomagnetite grains, AC fields above 30 A m<sup>-1</sup> are sufficient to push the domain walls towards the equilibrium displacement. Despite the multidomain-state viscosity, such grains do not display a significant frequency dependence at the operating frequencies of the susceptometer (Jackson et al. 1998). Conversely, in the finest (Saharan dust) end-members, the stoichiometry poorly constrains the magnetic properties, which are controlled by changes in blocking volume of dominantly (ultra)fine-grained oxidation products, as shown from the higher frequency dependence of samples SMIX and SC5-180. In such samples, near-zero AC-field dependences are observed, or even negative values. As further suggested from the shape of low-field hysteresis cycles (Fig. 12), such behaviour is consistent with the saturation, in fields lower than  $10^3$ Am<sup>-1</sup>, of SP phases.

As mentioned in Section 4, some basalt samples present high frequency dependence and high AC dependence, indicating that SP particles and MD titanomagnetite grains coexist in the same rock. This supports the idea that the deposition of (aeolian) components having variable grain size distributions is a major process for controlling the rock-magnetic changes of Canary aeolianites.

#### 10.2 Deposition and early pedogenesis

Assuming that subaerial reworking and surface oxidation probably affected most aeolianite detrital components before deposition in suboxic conditions, the magnetic changes—and their palaeoclimatic significance—are primarily driven by the grain size of detrital end-members, i.e. coastal sand and Saharan dust. Periods of fine Saharan dust trapping are favourable to the recovery of SP–PSD grains of dominantly maghemite composition, and also to the remobilization and hydration of iron in the clay-sized fraction, resulting in an increased SP goethite component. In contrast, periods of strong wind erosion favour the recovery of titanomagnetite and magnetite components, which persist in the unoxidized core of coarse grains.

Therefore, the contribution of fine maghemite and goethite in the pedogenized aeolianite is primarily connected with (i) the input of Saharan dust, in which Fe-Al oxides are commonly represented (Bergametti *et al.* 1989; Balsam *et al.* 1995), and potentially also with (ii) the intrinsic grain size distribution of the dust, which contains an ultrafine (<0.1  $\mu$ m) fraction (Torres-Padron *et al.* 2002). This is further suggested by (i) the stratigraphy of Canary aeolianites, where palaeosols systematically develop on dust-enriched deposits, and (ii) several clay and isotope studies evidencing Saharan dust enhancements in soils of archipelagos west of Sahara, from the Canary and Cape Verde islands (Mizota & Matsuhisa 1995; Caquineau *et al.* 1998) to the distant Bermuda archipelago (Herwitz *et al.* 1996).

As previously shown (Fig. 2), the palaeosol and loess samples are discriminated neither by silt concentration nor saturation magnetization, nor by  $\chi_{FD}$  per cent, a proxy for pedogenic SP enhancement (Fig. 11). In all cases, similar values are obtained for loess



Figure 12. Low-field hysteresis cycles of (i) sand (B3–1600) and (ii) claysized fraction of palaeosol (SC5-180). The Rayleigh behaviour of the sand is observed as soon as 500 A m<sup>-1</sup> In contrast, early saturation is observed in the clay-sized sample at  $\sim$ 500 A m<sup>-1</sup>, explaining its negative  $\chi_{ACD}$ .

and palaeosol end-members. The content in the loess of SP-like goethite and oxidized magnetite is unlikely to result from the exclusive translocation and trapping of pedogenic material into the underlying fine (loess) material, because the coarse fraction is also affected by surface oxidation. Thus, a potential climatic connection between fine dust deposition and early pedogenesis processes must be addressed.

#### 10.3 Palaeoclimatic inferences

In numerous loess-palaeosol magnetic studies, including some on Saharan loess from Tunisia (Dearing *et al.* 1996a, 2001), the pedogenic SP enhancement is ascribed to a strengthened authigenesis of iron minerals during periods of increased precipitation over the loess deposition area, while the pristine loess itself relates to arid (glacial) periods of strengthened loess deposition (Maher & Thompson 1999).

Additional observations on the early stages of soil formation under semi-arid conditions are provided by the Canary aeolianite. The systematic association of *in situ* pedogenesis indicators (bees' nests, roots, clay minerals) with Saharan dust enhancements, and the probable coupling between dust deposition and early pedogenesis through magnetic grain size changes and surface oxidation on the other hand, point to a causal relationship between dust input and soil formation.

According to stratigraphic observations at La Mala quarry and in the nearby preserved outcrops, the loess-palaeosol units as interbedded in coastal sand during the Last Glacial, low-sea-level period probably represent millennial periods of strengthened Saharan dust input and trapping, followed by wetter-than-today precipitation and palaeosol formation (Petit-Maire et al. 1986; Hillaire-Marcel et al. 1995; Meco et al. 1997). The average individual thickness of the six LM dust-palaeosol units ranges between 30 and 100 cm, while the silt fraction in such horizons ranges between 20 and 40 per cent in weight. Based on a present-day maximum dust deposition of  $\sim$ 30 g<sup>2</sup> yr<sup>-1</sup> in the area (Torres-Padron *et al.* 2002), and assuming a mean dry density of  $\sim 1.1$  for the aeolianite (Carmichael 1989), an apparent duration of individual pedogenized loess horizons at Lanzarote would range from  $\sim 2$  to  $\sim 15$  kyr. Thus, the deposition of La Mala dust during the Last Glacial period is consistent with present-day dust deposition.

However, recent or Late Holocene loess deposits are not found in the Canary Islands, like in many other peri-Saharan regions. The lack of such deposits is currently explained by the arid subtropical climate, where a precipitation of less than 100 mm yr<sup>-1</sup> prevents the development of vegetation traps, while strong wind speeds and tropical storms favour both erosion and the long-distance transportation of fine silt towards remote areas (Coudé-Gaussen *et al.* 1987; Goudie & Middleton 2001). According to such observations, the preservation of glacial Canary loess units probably needed wetter conditions and weaker seasonal to decadal climatic variability than today. Finally, (pedogenized) loess probably reflects transient conditions between (i) dry conditions favourable to the accumulation of sand and to the remote transportation of dust, and (ii) humid conditions favourable to a dominant authigenesis of secondary iron oxides.

Today, changes in precipitation over North Africa and Southern Europe, and Saharan dust loadings over the tropical Atlantic are strongly controlled by the strength of the Azores anticyclone, which induces a northward shift of the North Atlantic westerlies (the major source of atmospheric moisture to Northern Africa and Europe), for instance at seasonal to decadal scales during strong (positive) North Atlantic Oscillation (NAO)-year winters. Such conditions result in dry conditions and considerable dust loadings of the atmosphere, transported by trade winds over the subtropical Atlantic (Hurrel 1995; Moulin et al. 1997; Torres-Padron et al. 2002). Conversely, when the Azores anticyclone is weakened, westerlies are weaker and shifted southwards, resulting in higher winter precipitation and lower dust loadings over Northern Africa and Southern Europe, and in warmer air temperatures over Central Sahara (Bond et al. 2001). According to such features, the glacial Lanzarote pedogenized loess units would be consistent with a weaker Azores anticyclone, and a reduced latitudinal temperature gradient.

Despite the striking occurrence of six calcisols interbedded in the Last Glacial, low-sea-level coastal dune of La Mala, the poor chronology of the LM aeolianite and the low sampling resolution of our record does not testify to a direct correlation with the glacial, millennial climate variability in the region (Cayre *et al.* 1999; Sanchez Goni *et al.* 2000; Thouveny *et al.* 2000; Roucoux *et al.* 2001), nor with the so-called Dansgaard–Oeschger cycles and Heinrich events (Bond *et al.* 1993; Dansgaard *et al.* 1993). Nevertheless, the deposition of coastal sand under dry and windy conditions, followed by millennial-scale inputs of Saharan dust under increasing moisture and calcisol formation in semi-arid conditions, is consistent with the modelled onset of an interstadial period following a 'canonical' Heinrich event, where the reduced latitudinal temperature gradient causes relatively wetter conditions in the subtropical region (Rind 1998; Hostetler *et al.* 1999).

#### 11 CONCLUSIONS

The magnetic signature of aeolianite-Saharan loess deposits west of Africa is mostly controlled by (i) local coarse titanomagnetite and magnetite inputs, which dominate in the sand fraction, and (ii) SP to PSD (Ti)maghemite and SP goethite oxidation products, which dominate in fine Saharan dust-bearing material.

Owing to the instability of the aeolianite magnetization at high temperatures, and to complex magnetic mixtures, the magnetic mineral contributions are better identified from the low-temperature behaviour of aeolianite sample remanence, and from the AC dependence of magnetic susceptibility at room temperature.

The systematic development of palaeosols on underlying loessbearing material, and the association between the magnetic properties of pedogenized loess and the grain size of the parent material suggest that loess formation in the Canary Islands occurred during periods of lower glacial climate variability and wetter conditions in the subtropical region, possibly associated with the millennial weakening of the Azores anticyclone.

This study strongly suggests that the magnetic properties of soils, including any potential enhancement in (ultrafine) grained iron oxides, primarily depend on the grain size of the matrix, independently of rainfall. Further work is needed to discriminate more efficiently the respective controls of surface oxidation and authigenesis in pedogenized loess along rainfall and dust-size gradients in (semi-)arid areas.

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