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Mineral magnetic record of environment in Late Pleistocene and Holocene sediments, Lake Manas, Xinjiang, China.

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Abstract. A study of magnetic parameters was carried out on a Late Pleistocene and Holocene sedimentary sequence (5 m) from Lake Manas (northern Xinjiang, China), that has already been analysed for bulk sediment mineralogy, stable isotopes from carbonates and biogenic material. Magnetic minerals confirm major changes in palaeoenvironmental and palaeohydrological conditions that were deduced from other methods. They also provide complementary information on changes in the lake system during the early and middle Holocene. The most important mineral magnetic change results from the authigenic formation of ferrimagnetic greigite, Fe_3S_4 during the lacustrine episodes dated from ~8.7 kyr B.P. to ~5.5 kyr B.P. (380-335 cm) and from ~4.5 kyr B.P. to ~3.5 kyr B.P. (320-285 cm) respectively. Our data also indicate an abrupt break in sedimentary conditions which coincides with a short-term return towards dry climatic conditions around 3.5 kyr B.P., which was only suggested from other proxy data.

Introduction

In 1991, Lake Manas (45°45'N, 86°00'E, 251 m.a.s.l.) was investigated as part of a Sino-French palaeoclimate and palaeoenvironmental program in northern Xinjiang (CNRS - Academia Sinica, and the International Atomic Energy Agency). This paper presents the results of a mineral magnetic study from a 5 m sediment core from Lake Manas. The core provides a Late Pleistocene and continuous Holocene record that has been already analysed for mineralogy, chronology, stable isotopes, carbonates, pollen, diatoms, and total organic matter [Rhodes et al., in press].

Lake Manas is the terminal lake of the Manas River (~400 km long) which flows from the Tian Shan mountain range and lies under desert conditions (Fig. 1). The lake drains a catchment area of ~11,000 km². The bedrock of the upper Manas River catchment consists of granites, sedimentary formations of Devonian and Carboniferous age, and Mesozoic limestone. At the city of Manas, the river enters the Zunggary plain which is covered by late Quaternary fluvial, aeolian and lacustrine deposits. To the north, the Manas basin is limited by a series of NNE-SSW trending normal faults which affect continental Neogene deposits and the underlying petroliferous Mesozoic formations of Karamai. Lake Manas was a salt lake (10-15 km x 50-52 km), which dried up in the 1960's. Today it is a large salt plain, exploited for halite.

Material and methods

Three cores, ~5 m in length (LM I, LM II, and LM III), were taken within ~1m of each other from near the centre of the Manas salt plain, using a hand auger to penetrate the salt crust, and then a Wright piston corer. Analyses presented here were performed on cores LM II and LM III.

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Chronology, lithology and climatic changes

The chronology is based on 9 AMS ¹⁴C dates measured on authigenic calcite and/or aragonite or total organic matter [Rhodes et al., in press]. Holocene ages of sediments are in sequence and range from 10 to 0 33 kyr B.P. between ~400 and 150 cm. Ages can thus be interpolated between levels with similar lithofacies.

Four major lithofacies are observed (Fig. 2). The pre-Holocene sediments (498-400 cm) were deposited in generally dry, oxidising conditions. They include, however, a Late Pleistocene lacustrine phase (498-460 cm) dated at ~32 kyr B.P. This wet phase is followed by an extremely arid episode (460-418 cm) that is attributed to the Last Glacial Maximum [Rhodes et al., in press]. A return to lacustrine conditions is registered by vaguely laminated to mottled brown and grey clay (418-403 cm). Assuming a sedimentation rate similar to that of the overlying sediments, this Late Glacial humid episode would begin around 12 kyr B.P. Several millimetric fine sand beds observed around 402 and 399 cm, precede the establishment of a permanent lake and the development of a steppe vegetation in the catchment area, which lasted ~4,500 years.

This first Holocene lacustrine episode dated from 10 to ~5.5 kyr B.P. (400-335 cm) is recorded by soft lacustrine mud, rich in calcite and aragonite. Fluvial deposition of coarse sands occurred from 335 to 320 cm (~5.5 to ~4.5 kyr B.P.). This is most likely due to a return to arid conditions. A second Holocene lacustrine episode, dated from ~4.5 to ~2.5 kyr B.P. (320-240 cm) is documented by sediments very similar to those deposited in the 10-5.5 kyr interval. A dry spell is, however, suggested by both the mineralogy and pollen records around a depth of 280 cm (~3.5 kyr B.P.). Above 240 cm (~2.5 kyr B.P.), the diatom flora, mineralogy and stable isotopes indicate a stepwise establishment of shallow basin with hypersaline conditions in Lake Manas, attributed to a general trend toward arid climate.

Palaeomagnetic sampling and analysis

Standard sampling methods were used to obtain a semi-continuous coverage of each section of the core. About 300 oriented

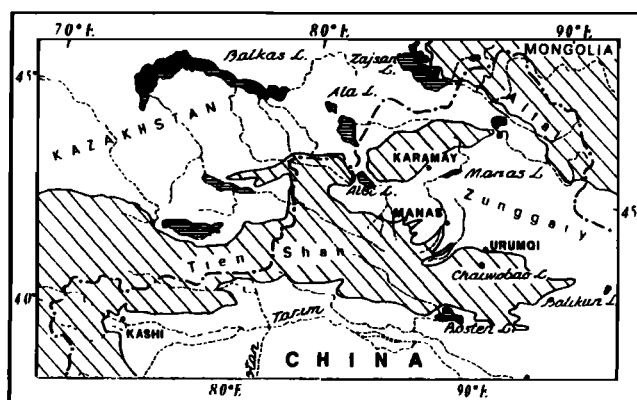


Figure 1. Location map of Lake Manas in northwest China.

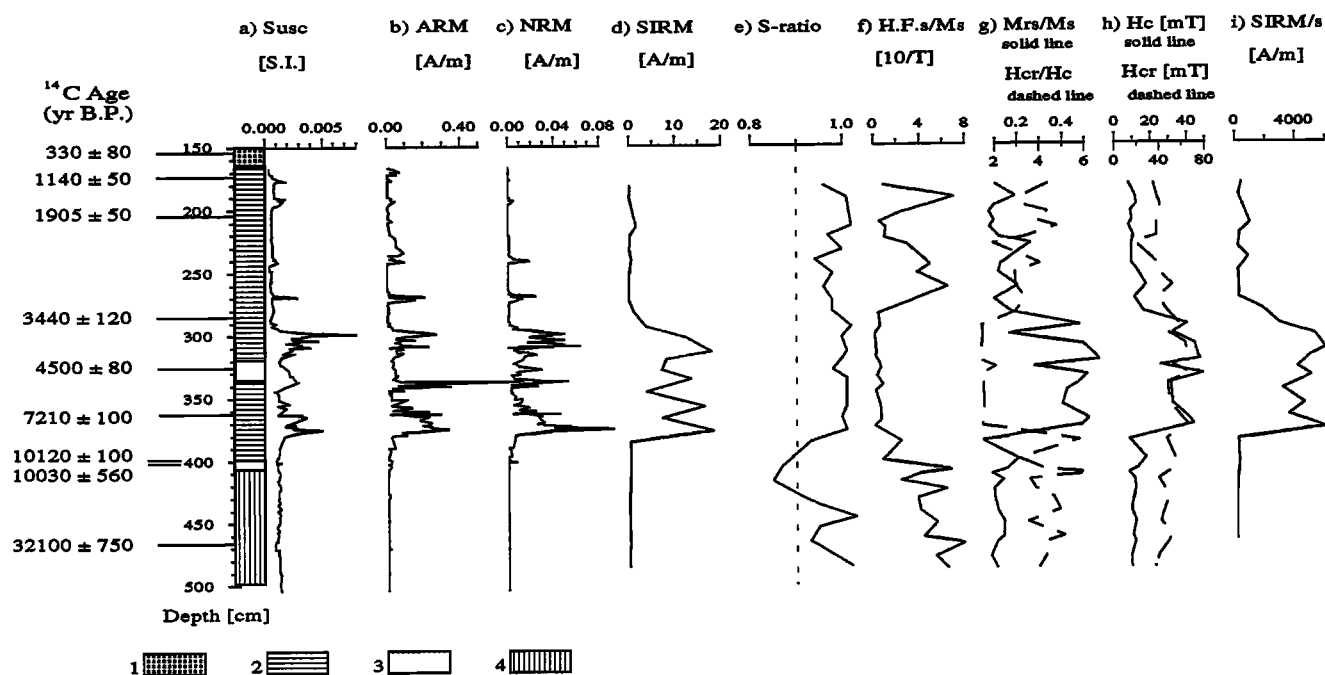


Figure 2. Down-core variation in a) magnetic susceptibility, b) intensity of ARM, c) intensity of NRM after a.f. de-magnetisation at 20 mT, d) SIRM, e) S-ratio, f) high field susceptibility/Ms, g) Mrs/Ms -upper legend; Hcr/Hc - lower legend, h) Hc -upper legend, Hcr -lower legend, i) SIRM/susceptibility a). b) & c) for cores LM II and LM III on common depth scale; from d) to i) core LM III ^{14}C ages measured in LM II Simplified lithofacies: 1) salt crust, 2) marls, muds olive grey to dark, 3) fluvial sands, 4) clay with salt crystals and several indurated red layers.

samples of 8 cm^3 perspex cubes were obtained from the two cores.

Natural remanent magnetisation (NRM) was measured with a CTF 3-axis SQUID magnetometer. Anhysteretic remanent magnetisations (ARM) were applied by superimposition of a $50\text{ }\mu\text{T}$ bias field on an alternating field that decreases smoothly from a peak of 50 mT (higher values of a.f. were impossible to obtain due to the limits of the used apparatus). Low field susceptibility (χ) was measured with a Bartington Instruments MS-2 susceptibility bridge. Isothermal remanent magnetisations (IRM) were applied to samples taken from 10 cm intervals in core LM III up to fields of 0.5 T . Although saturation was generally obtained in applied fields smaller than 0.3 T , a field of 0.5 T was used to introduce the saturation IRM (SIRM). All of these measurements were undertaken at the IPG in Paris.

An Alternating Gradient Magnetometer (Micromag) was used to measure the hysteresis properties: saturation magnetisation (Ms), saturation remanent magnetisation (Mrs), coercivity (Hc), remanent coercivity (Hcr) and also high field susceptibility (χ_{HF}). Samples ($10\text{--}40\text{ mg}$ of dry, bulk sediment) were taken at 10 cm intervals from core LM III. Curie balance analyses and X-ray diffraction (XRD) measurements were made on magnetic extracts obtained with a method similar to that of Papamarinopoulos et al., [1982]. These measurements were undertaken at the CFR du Laboratoire Mixte CNRS/CEA, in Gif-sur-Yvette.

NRM, ARM, and χ records from cores LM II and LM III record the same changes except for a 16 cm gap in core LM III due to slippage of the sediment within the coring tube. After removal of this gap, the records correlate perfectly.

The Lake Manas mineral magnetic record

Variations of bulk magnetic parameters χ , ARM, SIRM and NRM versus depth (Fig. 2 a-d) clearly define three different zones

of magnetic properties. These zones are also observed in the behaviour of hysteresis parameters (Fig. 2 f-h). Ms, Mrs, and χ_{HF} values depend on the dry mass of the sample, for which they have been normalised. In the Lake Manas sequence, however, the presence of relatively large diamagnetic salt crystals which have no, or negligible influence on magnetic parameters, might in a random way influence the mass of the small samples. Accordingly mass normalised values of these 3 parameters show large incoherent variations. Mass-independent quotients such as Mrs/Ms and $\chi_{\text{HF}}/\text{Ms}$ are therefore used to investigate the variation in magnetic properties. Mrs/Ms allows investigation of the size of grains in the ferrimagnetic fraction and $\chi_{\text{HF}}/\text{Ms}$ gives information on the relation between paramagnetic and ferrimagnetic fractions. Three zones are evident for these parameters: (i) $498\text{--}380\text{ cm}$, (ii) $380\text{--}285\text{ cm}$ and (iii) $285\text{--}164\text{ cm}$ (Fig. 2).

The bottom of the sequence ($498\text{--}380\text{ cm}$, Zone I) is characterised by low values of χ , ARM, NRM and SIRM, which are suggestive of low concentrations of ferrimagnetic grains. Low values of Hc ($\sim 10\text{ mT}$) and the hysteresis loop forms observed here suggest the presence of magnetite [Thompson and Oldfield, 1986; Peters and Thompson, 1994]. Diagrams of Mrs/Ms versus Hcr/Hc, useful for estimating relative grain size variation for magnetite [Day et al., 1977], indicate the presence of PSD and MD particles (squares in Fig. 3a). Therefore S-ratio ($\text{IRM}_{0.3}/\text{SIRM}$) smaller than 1 (Fig. 2e) and Hcr values that are higher than expected for MD magnetite [Thompson and Oldfield, 1986], especially between 430 and 390 cm , indicate the presence of oxidised magnetite or minor amounts of minerals with higher coercivities such as goethite or haematite. High $\chi_{\text{HF}}/\text{Ms}$ values reflect relatively high quantities of paramagnetic grains (Fig. 2f).

Zone II ($380\text{--}285\text{ cm}$) is characterised by high values of χ , ARM, NRM and SIRM with their drops at 350 and 325 cm . High ARM values are due to the high concentrations of magnetic grains when

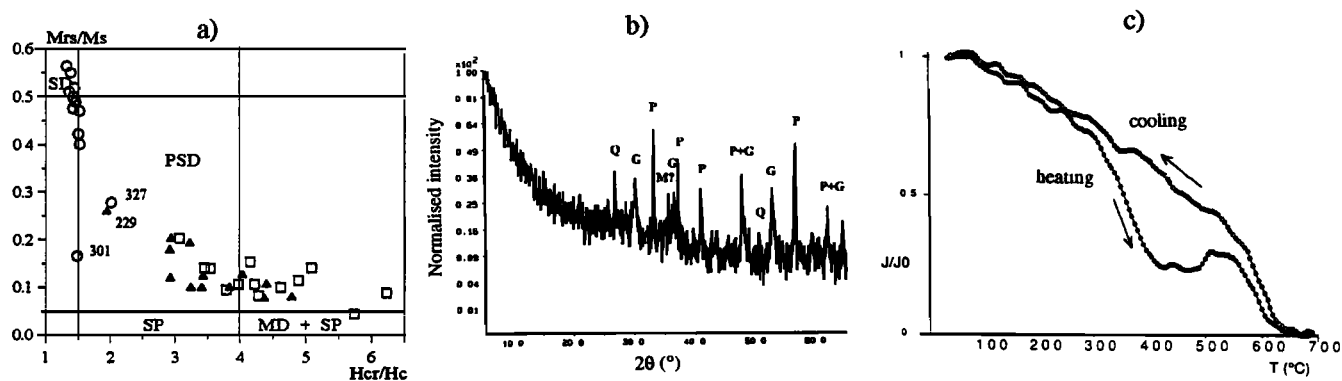


Figure 3. a) Classification of magnetic minerals in terms of magnetisation and coercivity ratios. Squares - zone I, circles - zone II, triangles - zone III. SD -single-domain grains, MD -multidomain grains, PSD -pseudo-single-domain grains and SP-superparamagnetic grains b) X-ray diffraction pattern obtained for magnetic extract from core LM III at a depth 380-360 cm. Pyrite (P) and greigite (G) dominate with some quartz (Q) and probably magnetite (M?) c) Thermomagnetic curve of the same magnetic extract heated in argon in magnetic field of 2.5 T.

occurring in phase with peaks in susceptibility (360-380 cm, 300 cm), and to the presence of SD particles at 338 cm. Hysteresis parameters (M_{rs}/M_s and H_{cr}/H_c) indicate small particle sizes throughout this zone (circles in Fig. 3a), with grains lying near the SD/PSD boundary, except for two points at 301 and 327 cm. The latter corresponds to a sand layer and the former indicates an important contribution from superparamagnetic (SP) grains. S-ratio indicates that all of the grains are saturated before 0.3 T (Fig. 2e), suggesting the presence of a ferrimagnetic mineral. However, values of H_c (~40 mT) are too high for SD to PSD magnetite [Thompson and Oldfield, 1986]. X-ray diffraction data from the separated material (Fig. 3b) indicates the presence of pyrite (FeS_2) and greigite (Fe_3S_4). The thermomagnetic behaviour of the magnetic extract in an argon atmosphere indicates the presence of a mineral that undergoes a major decrease in magnetisation between 300-400°C, which afterwards is transformed to a ferrimagnetic iron oxide (Fig. 3c). Such behaviour is similar to that observed for greigite if heated in air [Snowball and Thompson, 1990; Snowball, 1991; Hoffman, 1992; Roberts and Turner, 1993; Reynolds et al., 1994]. Considering the very small quantity of magnetic material in other zones, Curie Balance and XRD experiments were possible only in this lithological unit. Hysteresis parameters of greigite in Lake Manas sediments, such as high values of H_c and small particles sizes (SD), confirm observations of Snowball [1991] and Roberts [1994], concerning the behaviour of natural greigite. These properties are consistent with high values of $SIRM/\chi$ (Fig. 2i), which are commonly observed in natural greigite samples [Roberts and Turner, 1993; Reynolds et al., 1994]. Hysteresis loops are characterised by wide, open central sections as it is also observed in natural greigite samples [Snowball, 1991; Peters and Thompson, 1994]. Low χ_{HF}/M_s suggests relatively low contributions from paramagnetic grains in this zone.

In zone III (285-170 cm), a dramatic drop of ARM, SIRM, χ and NRM indicates a decrease in the concentration of magnetic grains. The hysteresis parameters and S-ratio correspond to those expected for PSD magnetite (triangles in Fig. 3a). The lack of direct evidence (as cited above) for greigite suggests that greigite is absent from this part of the sequence. Small increases of ARM and χ at 270, 240, and 190 cm are due to relatively higher concentrations of ferrimagnetic grains. High χ_{HF}/M_s values in this zone reflect higher contributions of the paramagnetic clays.

The Lake Manas sediments thus show large variations in the size, concentration and mineralogy of magnetic grains. These changes

are primarily due to authigenic formation of the ferrimagnetic iron sulphide, greigite (Fe_3S_4).

The formation of authigenic iron sulphides is possible in alkaline, anoxic and sulphate-rich environments with sufficient quantity of dissolved iron. During early diagenesis, iron sulphides are formed by the reaction of H_2S with iron ions [Berner, 1980; Roberts and Turner, 1993]. These components derive from the reduction of dissolved sulphate by bacteria which use oxygen (combined in sulphate) as a source of energy and detrital iron bearing minerals present in sediments. Significant sulphur can also be liberated as H_2S during bacterial degradation of organic matter.

According to Jones and Bowser [1978], greigite is an intermediate mineral in the pyritization reactions which occur during sulphurization of an initially amorphous precipitate of FeS. The transformation of iron monosulphides to pyrite will not occur in the absence of sulphur. In the absence of sulphur (e.g. in many freshwater environments) iron sulphide formation will be limited to cryptocrystalline FeS or greigite. Jones and Bowser suggest that the role of organisms affects only the sulphate reduction process. Therefore, besides this possibility of inorganic greigite formation (organic mediated), it is known that several species of magnetotactic bacteria synthesise intracellular magnetic inclusions of single domain greigite. These magnetotactic bacteria are common in brackish, sulphide-rich water and sediments [Farina et al., 1990].

Discussion and conclusions

Mineral magnetic zonation of the Lake Manas sedimentary sequence is generally in agreement with the palaeoenvironmental record defined by other proxy data [Rhodes et al., in press].

The pre-Holocene period is clearly characterised by a low concentration of magnetic particles which is dominated by PSD and MD detrital magnetite, with oxidised magnetic grains between 430 and 390 cm (~12.5-9.5 kyr B.P.). This is consistent with the predominance of detrital mineral and organic material in the sediments, with low ^{13}C and ^{18}O of carbonates reflecting short residence time of the water. These observations are interpreted as being indicative of low lake levels which is consistent with a desert type vegetation (*Chenopodiaceae* dominant). Maximum aridity, as suggested by several environmental indicators [Rhodes et al., in press], was recorded between 460 and 418 cm, and is tentatively attributed to the Last Glacial Maximum. The presence of oxidised magnetic grains in the Lake Manas record roughly coincides with

the upper part of this dry episode and the lower part of the following lacustrine period.

The magnetic properties of the sediments indicate a long term episode from ~8.7 to ~3.5 kyr B.P. (380–285 cm) which was characterised by the authigenic formation of greigite. This could suggest high biological activity which may favour authigenesis of iron sulphides (either biomediated or synthesised by bacteria) in stagnant waters with anoxic bottom conditions. The limits of this episode are sharply defined by most of the magnetic parameters analysed. The lower limit is shifted by ~1.3 kyr above the major climatic event deduced from the hydroclimatic record, which corresponds to a rapid transition from dry to wet conditions at ~10 kyr B.P. (400 cm). The abruptness of the onset of this magnetic interval indicates a sudden change in the lake metabolism. The magnetic parameters of the lower part of the lacustrine episode (10 to 8.7 kyr B.P.) indicate that a detrital influx dominated the lake during this time. The concentration of magnetic grains decreases during the very wet, warm interval (7.5 to ~6 kyr B.P.). This could be explained by the transformation of greigite to pyrite due to completion of the sulphurisation reactions. Such change could be due to a decrease of detrital iron influx into the lake or to increased availability of dissolved sulphate.

The sudden influx of fluvial material recorded around 5.5–4.5 kyr B.P. (335–320 cm), is clearly documented in the magnetic record by a sharp decrease in ARM, χ and other parameters. This likely reflects a short-term return to dry climate, as suggested by the pollen record. However, the hypothesis of a tectonic uplift in the Tian Shan mountains cannot be ruled out [Rhodes et al., in press].

Conditions similar to those of the early Holocene are recorded from ~4.5 to ~3.5 kyr B.P. (320–285). The abrupt change at ~3.5 kyr B.P. coincides with a short-term dry event suggested by a sudden input of detrital organic material to the lake. The carbonate content and the stable isotope records only document a general shift towards increasing aridity from that time [Rhodes et al., in press]. The magnetic data thus clearly indicate a major environmental change in the lake and the catchment area at ~3.5 kyr B.P.

The late Holocene (up to 285 cm: 3.5 kyr - recent) is similar to that of the pre-Holocene period, except for lack of evidence for oxidation of magnetite grains. The lake became shallow and better mixed, without evidence of greigite formation. The pollen record suggests instability in hydrology or climatic conditions in the catchment area which is not reflected in the magnetic record of the lake sediments.

The Lake Manas record is broadly consistent with previous palaeoclimatic data from western China. Different types of proxy data [e.g. Zhou et al., 1991; Fang, 1991; Lister et al., 1991; Gasse et al., 1991], indicate that conditions warmer and wetter than today prevailed in western China during the early-middle Holocene period. These data document the rapid establishment of monsoon precipitation around 10 kyr B.P., and a climatic optimum (wet and warm conditions) around 7–6 kyr B.P., followed by a general trend towards aridity which culminated at ~3.5 kyr B.P. The Lake Manas record also suggests that the summer monsoon penetrated westward over the Zunggary Basin during the early-middle Holocene.

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