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Formation mechanism of ferromagnetic minerals in loess of China: TEM investigation

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Abstract The results of TEM investigation indicate that magnetite and maghemite are the major ferromagnetic minerals in loess-paleosol sequences. Primary magnetite has the similar morphology and surface characteristics as eolian detrital particles. The magnetite can be classified into two categories, high-titanium and low-titanium, which may be the indicators of magmatic rocks and metamorphic rocks, respectively. TEM investigation at nanometer scale shows that primary detrital magnetite of micron scale had been partially weathered to maghemite of 5~20 nanometer during the pedogenic process, which maintain the pseudomorphism of the aeolian debris. Some chlorite particles were also weathered to nanometer scale magnetite or maghemite in the pedogenic process. So weathering of the two minerals leads to formation of superparamagnetism, which may be the important mechanism of magnetic-susceptibility increase in paleosols. The magnetite or maghemite resulting from the weathering of chlorite contains a small amount of P and S, which is the signal of microbe-mineral interaction, and indicates that microbes may play a certain role in chlorite weathering and formation of superparamagnetic particles.

Keywords: loess, paleosol, magnetite, maghemite, biogeochemistry, transmission electron microscope (TEM).

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In 1982, Heller et al.^[1] published a representative article about study on magnetism of loess-paleosols, and they reported increase of susceptibility in paleosols for the first time and found the correlation between the marine oxygen isotope change and susceptibility variation of loess-paleosol sequences. Ever since, magnetic study on loess-paleosols has been the focus of attention as a new breakthrough point to paleoclimate study. When discussing changes of paleomonsoon in East Asia, magnetic susceptibility has been used as a proxy indicator^[2-5] of intensity of the summer monsoon, to assess the change of paleopricipitation in Loess Plateau^[6-10].

In the last decade, numerous studies have contributed to discussion of the relativity between magnetic-susceptibility and paleoclimate in terms of rock magnetics. Magnetite and maghemite are the major two ferromagnetic minerals in loess-paleosols and the magnetic particles are included in singe-domain, polydomain or superparamagnetic^[11–13]. Some scholars believe that formation of ultrafine-grained magnetite in the pedogenic process is the major cause of magnetic-susceptibility increase in paleosols^[6,14–16], but others believe that maghemite plays a major role^[17–21]. Magnetic susceptibility variations in loesspaleosols are known well and progress has been made in study on categories and quantitative analysis of magnetic oxides^[6,22-24], but genesis of ferromagnetic minerals, which is relevant to magnetic-susceptibility, is controversial. There are a variety of theories about the mechanism of increase of susceptibility in paleosols, such as "sedimentary compaction and decalcification"^[1], "sedimentary dilution"^[25], "pedogensis"^[14,15,18], "original area difference"^[26], "natural fire"^[27], "degradation by plants"^[28], "magnetotactic bacteria interaction"^[29], so more and more scholars emphasize the important role of pedogenesis and microbe activities on increase of susceptibility. In order to figure out the essence, the categories and formation process of ferromagnetic minerals formed in the pedogenic process should be studied. One of the key points is to figure out the formation mechanism of superparamagnetic particles, which is still the problem unsolved in loesspaleosol studies.

Because of fine size and low content of ferromagnetic particles in loess, the resolution of SEM is not high enough to observe ultra-microstructure on nanometer scale, structural analysis and then mineral phase identification, so it is somewhat difficult to figure out the origin of ultrafine authigenetic ferromagnetic minerals. Such magnetic study such as thermodemagnetization can figure out the existence and content of maghemite, which could be evidence for the pedogenic origin^[24], but cannot provide direct information on formation mechanism. However, TEM has high resolution at atom scale and can carry out morphology, composition and structural analyses at one time, so it has been widely used to identify ultrafine-grained magnetite of biogenetic in such geological phases as soil and sediments^[30–36]. Maher and Thomson^[37] have studied the samples of the loess-paleosol in China with TEM and found out the biogenetic magnetite. TEM with high resolution was used in this study to observe and analyze the ferromagnetic minerals in the loess-paleosol. On the basis of some new results, the origin of ferromagnetic minerals and the mechanism of increase of susceptibility were discussed.

1 Background of samples and experimental methods

Experimental samples were obtained from a loess

layer L1 and two paleosol layers, S1 and S5 in the well-known Luochuan loess section, which represent Malan loess, upper Lishi loess and lower Lishi loess. The geological background of the Luochuan loess section has been elaborated^[38]. Sampling sites are shown in Fig. 1.



Fig. 1. Sampling sites in loess-paleosol sequences of Luchuan section.

Samples for TEM investigation were prepared with water-suspension, embedding method or magnetic selection. The former two methods are conventional for TEM samples and need not go into details. Magnetic selection method was carried out as follows according to Hounslow and Maher^[39]: 5 g loess sample was put into a beaker of 500 mL, 200 mL distilled water was added, into which 2-cm-long white magnetic rotor coated with PTEF was put, and stirred with 100 r/m for 2 h using a magnetic stirrer. Then the rotor was taken out with a nonmagnetic tweezer and rinsed with distilled water for 3 times to remove the clay minerals adhered. Then the relative content of ferromagnetic minerals can be roughly predicted according to the rotor weight discrepancy before and after magnetic separation. A small amount of magnetic mineral powder was taken from the surface of the rotor for preparing water suspension. A drop of suspension was deposited on a copper grid coated with holey carbon. After the water was evaporated, it was fixed on the TEM sample holder for further analysis with JEOL JEM-2010 High-Resolution Transmission Electron Microscope (HRTEM with a lattice resolution of 0.14 nm). If necessary, nanometer scale morphology analysis, X-ray energy-dispersive spectroscope (EDS), selected-area electron diffraction (SAED) and high-resolution lattice image analyses can be carried out. All the analytic work was carried out in Department of Earth and Planetary Sciences of the University of New Mexico, USA.

2 Results and discussion

The oxides and hydroxides of iron in nature include magnetite, maghemite, goethite, hematite, lepidocrocite and iron hydroxide. In loess and paleosols, magnetic substances include magnetite (90 $\text{Am}^2 \text{ kg}^{-1}$), maghemite (70 $\text{Am}^2 \text{ kg}^{-1}$) and hematite (0.4 $\text{Am}^2 \text{ kg}^{-1}$). Compared with magnetite or maghemite, contribution of hematite to susceptibility can be ignored^[17] because of its weak magnetism. Accordingly, magnetite and maghemite are the only two ferromagnetic iron oxides. The former is black and the latter rufous to maroon. According to the color difference, the ferromagnetic minerals after magnetic separation were supposed to be the mixture of magnetite and maghemite. Figs. 2—5 show the TEM micrographs of the ferromagnetic minerals in loess-paleosol sequences.

(i) Morphology and composition of primary magnetite. Morphological characteristic of primary magnetite of eolian sediments is shown in Fig. 2, which indicates that magnetite particles of eolian sediments present micron to sub-micron scale and angular (Fig. 2(a) and (b)), subangular (Fig. 2(b) and (c)) or spheroidal (Fig. 2(d)) morphology. The information obtained from SAED suggests that the magnetite of above features is monocrystal. The spheroidal particles are occasionally found to intergrow with rutile (Fig. 2(c) and (f)) and not disaggregated into monocrystals completely. So we can make sure that those angular to spheroidal magnetite belong to the pri



Fig. 2. Bright field TEM images, SAEDs, EDSs of detrital magnetite. (a) Angular morphology of magnetite, left corner inserts SAED, sampling from L1 lay; (b) angular or subangular morphology of magnetite, left corner inserts SAED, sampling from L1 lay; (c) subangular morphology of magnetite, half weathering and intergrowth with rutile, right corner inserts EDS of rutile, sampling from S1 lay; (d) spheroidal morphology of magnetite, SAED is same as in Fig. 2(b) and omit, sampling from L1 lay; (e) SAED of magnetite in Fig. 2(c), which indicates two series diffracting spots of magnetite and magnetite; (f) EDS of rutile in Fig. 2(c); (g) EDS of magnetite in Fig. 2(a), which indicates high content of TiO₂; (h) EDS of magnetite in Fig. 2(b), which indicates low content of TiO₂. All the scales in the figures are 300 nm.



Fig. 3. Bright field TEM images showing transition process of magnetite weathering into maghemite (L1 lay). (a) Primary detrital magnetite



(Mt)-weathering half into maghemite (Mh); (b) magnetite SAED overlapped maghemite SAED; (c) maghemite SAED.

Fig. 4. Bright field TEM images, SAED, EDS of detrital magnetite magnetite weathered (S5 lay). (a) Showing the ultra-microstructure of nanometer scale polycrystalline aggregate and porous of maghemite which formed from weathering of magnetite and maintains the profile of the primary detrital magnetite; (b) high resolution lattice fringe; (c) the SAED pattern showing a polycrystalline ring; (d) EDS of maghemite.

mary detrital minerals of eolian sediments. This kind of magnetite exists in loess and paleosol samples, but there is more primary detrital magnetite in loess than in paleosols according to the rough statistic of observation results. SAED analysis also indicates that the diffraction pattern of magnetite is overlapped by weak diffraction spots of maghemite such as (110) and (106) plane meshes (Fig. 2(b) and (e)), which reveals that primary magnetite had been slightly oxidized and transformed partly into maghemite. The results suggest maghemite film coating on the primary magnetite particle. Generally, eolian dust is abrased violently in the process of transport and thus the maghemite film formed by oxidation before loess sediment is difficult to be preserved, so it is inferred that the oxide film of maghemite on the surface of primary magnetite may be formed after loess packing but it is also possible for it to form before loess packing. Barrn and Torrent^[40] have pointed out that there are three major mechanisms in maghemite forming in soils: (1) heating of goethite (α -FeOOH) in presence of organics; (2) oxidizing of magnetite when weathering; and (3) dehydroxylating of lepidocrocite (γ -FeOOH). So the results of TEM investigation indicate that maghemite in loess can be formed directly from oxidation of magnetite.

EDS analyses indicate that primary detrital magnetite particles have two categories, high-Ti and low-Ti, which represent different sources zone of detrital magnetite. Magnetite containing Ti is the signal of the endogenic geological action and was formed in magmatic rocks and metamorphic rocks. The Ti content in magmatic rocks is often higher than that in metamorphic rocks. So composition analysis of magnetite and the ratio of magnetite containing Ti may provide some information about the loess sources zone. Variation regulation in different regions of Ti content magnetite should be further studied systematically. (ii) Formation of nanometer scale maghemite from magnetite weathering. Fig. 3 shows a transition relation-

ship between magnetite and maghemite in one detrital

particle, which suggests that the former is gradually



Fig. 5. Bright field TEM images showing the relationship of chlorite and ferromagnetic minerals (S5 lay). (a) Chlorite in edges transformed into I/S mixlayer minerals and ferromagnetic minerals; (b) ferromagnetic minerals distribution on the surface of chlorite; (c) ferromagnetic minerals on the surface of I/S mixtlayer minerals; (d) ferromagnetic minerals in porous of paleosol, showing authigenetic characteristics; (e) EDS of authigenetic ferromagnetic minerals, showing that they contain a small amount of P and S.

transformed into the latter. The TEM image (Fig. 3(a)) shows that the weathering part has been polycrystallized, but SAED present feature of monocrystal, which indicates that the formed maghemite maintains the same crystal orientation as the magnetite in the transition state.

Fig. 4 shows the ultra-microstructure of nanometer scale polycrystalline aggregate of ferromagnetic iron oxides. Based on the morphology features (Fig. 4(a)), the whole particle is a nanometer scale polycrystal while monocrystal particles of 5-20 nm (Fig. 4(b)) constitute the porous aggregate of nanometer scale iron oxide. But the aggregate maintains the profile of the subangular to spheriodal primary detrital magnetite. The SAED pattern (Fig. 4(c)) is a polycrystalline ring, and presents characteristic of superstructural diffraction of maghemite, which proves the existence of maghemite. But the diffraction ring is dispersive, which is in agreement with the fine crystal shown in the image. EDS analysis proved that the Ti content is high. This result has not been reported in the past. After careful observation with TEM, this kind of phenomena widely presents in the samples of S1 and S5. The transformation of micron-grained minerals into

nanometer scale polycrystalline aggregate is a common phenomenon in microstructure observation of synthesis materials, such as transformation of micron-grained gibbsite into nanometer scale y-Al₂O₃ and micron-grained pyrite into nanometer scale hematite, and it is characteristic of solid phase transformation. The theoretic density of magnetite is 5.197, and that of maghemite 5.074, so the volume change is the main driving force for transformation from monocrystals to polycrystals. Figs. 2-4 show a continuous process of oxidation of magnetite and structural and morphology change. TEM investigation not only provides the microstructure evidence for chemical weathering from magnetite to maghemite, but also proves that maghemite can be directly formed by oxidation of magnetite and nanocrystallized because of crystal structural adjustment in the process of transformation. Micron-grained eolian magnetite can be transformed into nanometer maghemite in the pedogenic process and change from ferromagnetic to superparamagnetic. Maher^[41] pointed out that susceptibility of magnetite about 10 nm is 4 times higher than that of magnetite about several microns. Even if there is no increase in ferromagnetic particle content in paleosols, susceptibility can be amplified by several times because of nanocrystallization of magnetite when weathering. In general, this forming process of maghemite and superparamagnetism may be the major cause for the increase of susceptibility in paleosols.

(iii) Chlorite weathering and formation of ferromagnetic minerals. Fig. 5 shows the relationship between magnetic iron oxides and clay minerals. Fig. 5(a) shows that marginal chlorite was weathered into illite-montomorillonite and iron oxides. Fig. 5(b) shows the distribution of nanometer scale iron oxide particles on the surface of weathered chlorite. Fig. 5(c) shows the iron oxide aggregate film adhered on the surface on illite-montomorillonite. Fig. 5(d) shows fine iron oxide particles in the porous of paleosol particles. These phenomena are found in paleosols and the iron oxides are authigenetic according to their morphology and distribution, and relevant to chlorite weathering Through the EDS, SAED and High-Resolution Lattice Image analysis, iron oxide minerals adhered on the surface of chlorite and illite-montomorillonlite are magnetite and/or maghemite. The diffraction rays of these two minerals are similar and overlapped partly. Because of fine size of iron oxide particles and their adhesion on the surface of clay minerals, the diffraction ring is not clear and thus the superstructural diffraction ring of maghemite is difficult to be identified. So it is difficult to identify these two ferromagnetic iron oxides. But according to the iron oxide minerals morphology, distribution and relation with clay minerals, these iron oxide minerals were formed from chlorite weathering. Chlorite is a mineral of easy weathering on the Earth's surface. Chlorite weathering action releases iron, which forms iron oxides while it is transformed illite-montomorillonite^[42-44]. Chlorite is a major Fe-containing silicate mineral and meanwhile the one of most unstable in silicate minerals in loess. Authigenetic maghemite and/or magnetite can be formed in the pedogenic process of chlorite, and thus the content of ferromagnetic minerals increases. So the pedogenic process is also one of the major mechanisms of susceptibility increase in paleosols. Ji et al.^[45] also came to a conclusion that chlorite weathering is relevant to increase of susceptibility based on the relation of chlorite/illite ratio and susceptibility in the Luochuan loess section. In general, TEM investigation provides the direct evidence for the relativity between chlorite weathering and formation of ferromagnetic iron oxides and lays a mineralogical foundation for explaining the phenomena about susceptibility.

It has been believed for a long time that magnetite and maghemite are products of endogenic geological processes and the two minerals in soils come from weathering of rocks. When magnetite was found widely in microbes, especially when ordered euhedral particulate magnetite was found in magnetotactic bacteria^[46,47], origin of magnetite in soils cannot be explained only by the weathering mechanism. But nanometer scale maghemite and magnetite of biogenetic origin are rare^[36,48]. The distribution of ferromagnetic minerals on the surface of chlorite and illite-montomorillonite in paleosols is in agreement with the intracellular iron minerals found by Glasauer et al.^[48] and EDS analyses prove that there is a small amount of P and S (Fig. 4(d)), which is also identical with Glasauer's results. It suggests that biochemical weathering should not be ignored in the process of chlorite weathering. Chlorite is unstable mineral under acid condition, so microbe-chlorite interaction may accelerate the weathering rate of chlorite^[49]. Meanwhile, biological activity depends on paleoclimate, so it is important to reconstruction of paleoclimate and pedogenic process in loess-paleosol sequences through further study on chlorite-microbe interaction using TEM investigation into iron oxides.

3 Conclusion

Magnetite is the major detrital ferromagnetic mineral in loess-paleosol sequences. It can be classified into two categories, high-titanium and low-titanium, which can be the indicators of different original rocks such as magmatic rocks and metamorphic rocks, respectively. Micron-grained primary detrital magnetite can be gradually weathered into maghemite of 5—20 nm in the pedogenic process. Transformation of magnetite into maghemite may be one of the major mechanisms for formation of superparamagnetism and increase of supspectibilty in paleosols.

Magnetite or maghemite can be formed by iron ion releasing from weathered chlorite and adhered on the surface of the clay minerals in the form of nanometer scale particles, which may be another one of the major mechanisms for increase of susceptibility in paleosols.

The magnetite or maghemite obtained from weathered chlorite contains a small amount of P and S, which is the signal of microbe-mineral interaction. It suggests that microbes may play a certain role in chlorite weathering. So it is necessary to put more effort on the study on chlorite-microbe interaction and its relation with paleoclimate.

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