

**MINERALOGY  
GEOLOGY  
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# IRON MÖSSBAUER SPECTRA OF GEOLOGICAL AND ARCHEOLOGICAL SAMPLES FROM THE XINJIANG UIGHUR AUTONOMOUS REGION OF CHINA

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The chemical states of iron in sand and archeological sun-dried brick samples collected in the desert area around Tulupan in the Xinjiang Uighur Autonomous Region of China are studied. Hematite and another magnetically ordered species are found together with paramagnetic  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  ions. Magnetite is also identified in the fractions gathered with a magnet from the samples.

## 1. INTRODUCTION

The extremely dry climate and the alkaline nature of the ground constitute characteristic features of the Xinjiang Uighur Autonomous Region of China. In this work, the chemical states of iron in sand samples collected in the vicinity of Tulupan and along the highway between Tulupan and Urumqi are studied by Mössbauer spectroscopy. Sun-dried brick samples of city ruins of the Tang age are also studied.

## 2. EXPERIMENTAL

The sand samples were collected at the foot of Flaming Mountain, Tulupan Basin center, Chaivo, Hougou, Hougou riverside, and Dabancheng. The sand of Flaming mountain is brownish red, where the name of the mountain has its origin. The other samples are pale yellow or slightly brownish yellow. The sun-dried brick samples were gathered from the decayed walls of the Gaochan city ruin (the Tang age) and the Jiaohe city ruin (also the Tang age). Magnetic fractionation of the sand samples was performed by the use of a Sm-Co permanent magnet.

Measurement of Mössbauer spectra was made at room and liquid nitrogen temperature using a conventional spectrometer. The thickness of the samples was  $50 \text{ mg/cm}^2$  or less.

## 3. RESULTS AND DISCUSSION

The Mössbauer spectra of the red sand sample collected at the foot of Flaming Mountain are shown in Fig. 1. They consist of paramagnetic doublets of  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$ , along with magnetically split peaks of  $\text{Fe}^{3+}$ . The magnetic component is decomposed into two sextets with hyperfine magnetic fields of  $530 \pm 10 \text{ kOe}$  and  $490 \pm 10 \text{ kOe}$  at liquid nitrogen temperature, respectively. The electric quadrupole shift, defined as the splitting between the first and second peaks minus that between the fifth and sixth, is positive at both room and liquid nitrogen temperatures. We ascribe the sextet with the larger magnetic splitting to hematite (Sextet(1) in Fig. 1).

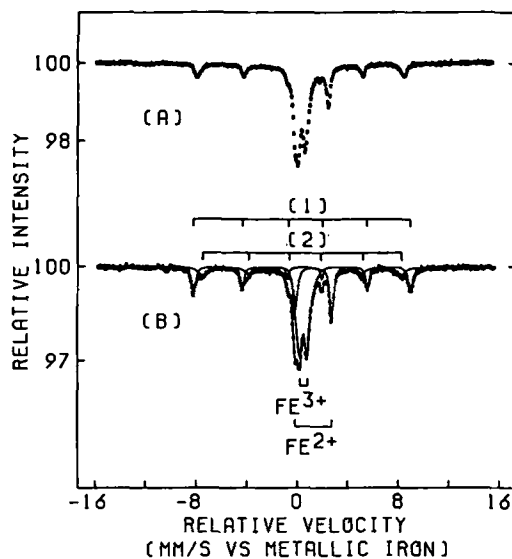


Fig. 1. Iron Mössbauer spectra of a red sand sample collected at the foot of Flaming mountain: (A). At room temperature and (B) at liquid nitrogen temperature.

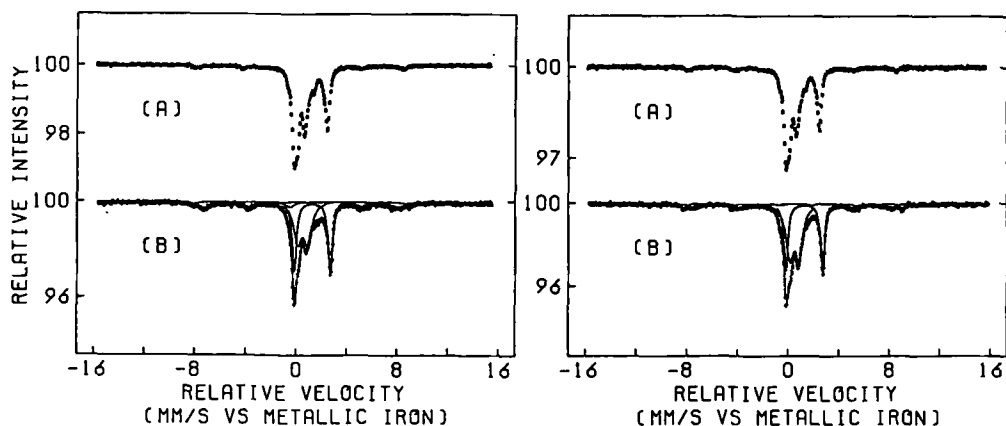


Fig. 2. Iron Mössbauer spectra of yellow sand samples collected at Hougou (left) and Dabancheng (right): (A) At room temperature and (B) at liquid nitrogen temperature.

Positive quadrupole shift below the Morin temperature is attributed to the small size of hematite particles in the sample /1/. The other sextet with the smaller magnetic splitting and a larger line width (designated as Sextet(2) in Fig. 1) is assignable to hematite with much smaller particle sizes than that of (1) /1/. An

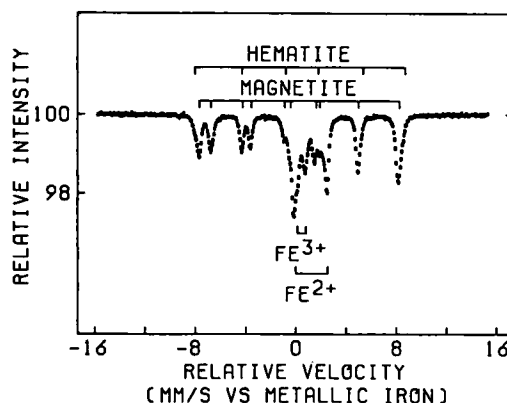


Fig. 3. Iron Mössbauer spectra of the fraction extracted with a Sm-Co magnet from the yellow sand sample collected at Chaivo; measurement at room temperature.

alternative interpretation of Sextet(2) is that it represents hematite with a certain diamagnetic impurity or goethite.

Typical spectra of yellow sand samples are given in Fig. 2. They also consist of paramagnetic doublets of  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  and two magnetically split components of  $\text{Fe}^{3+}$  with essentially the same hyperfine magnetic and electric quadrupolar parameters as the red sample from Flaming Mountain. It is seen from Fig. 2 that the sextet with smaller magnetic splitting is dominant over the other one in the yellow sand samples.

In case of the red sand sample from Flaming Mountain, the fraction attracted by the Sm-Co magnet is less than 0.1 % in weight. On the other hand, 2–12 % in weight is separated using the magnet from the yellow sand samples collected at the other sites. In some of the magnetically extracted fractions (those from Hougou, Hougou riverside, and Chaivo), absorption lines ascribed to magnetite are dominant. A typical example of Chaivo sample is shown in Fig. 3 with assignment of the peaks. Along with the lines of magnetite, there are observed in the spectrum those of paramagnetic  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  species, which are considered to have been gathered with magnetite sticking to it. Since the intensity of the lines apparently assigned to A site of magnetite is much larger than the half of those of B site, superposition of hematite sextet on the former lines is inferred as is indicated in Fig. 3. In the magnetically gathered fractions of samples from Tulupan Basin center and Dabancheng, the sticking paramagnetic species are dominant.

The paramagnetic ferric doublet of all the samples has an isomer shift of  $0.41 \pm 0.05$  mm/s and quadrupole splitting of  $0.63 \pm 0.05$  mm/s at room temperature. It is ascribed to high spin ferric ion with octahedrally coordinated oxide ions. The superparamagnetic fraction of hematite fine powder is considered to be included in the doublet.

The ferrous doublet has an isomer shift of  $1.17 \pm 0.05$  mm/s and quadrupole splitting of  $2.61 \pm 0.05$  mm/s at room temperature in all the sand samples studied. The line width is considerably small, suggesting that a single mineral species is dominant. The Mössbauer parameters are typical of ferrous silicate minerals /2/.

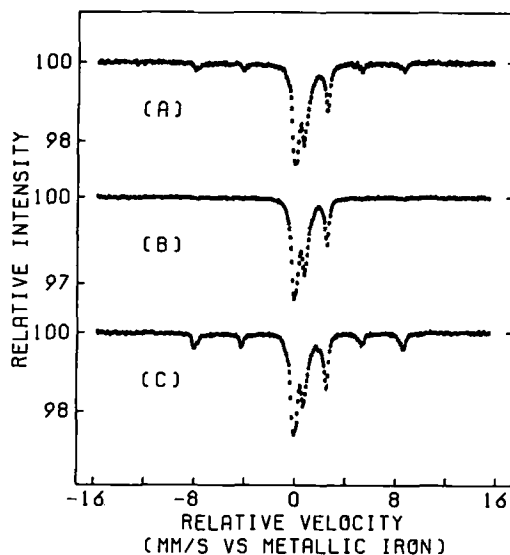


Fig. 4. Iron Mössbauer spectra of sun-dried brick samples collected at (A) Gaochang city ruin (the Tang age), (B) Jiaohe city ruin (the Tang age), and (C) modern Urumqi city; measurement at room temperature.

The sun-dried brick sample of Gaochang city ruin (Tang dynasty) gives a spectrum resembling that of Flaming-Mountain sand, while in that of Jiaohe city ruin (also Tang dynasty) the magnetically split component is scarce. The spectrum of the modern fire brick sample collected in Urumqi resembles the former (Fig. 4).

Further work is in progress with the aid of powder X-ray pattern measurement.

#### ACKNOWLEDGMENTS

Cooperation of Mr. Sui in sample collection and useful suggestion of Dr. Takeda are gratefully acknowledged.

#### References

- /1/ W. Kündig, H. Bömmel, G. Constabaris, and R. H. Lindquist, *Phys. Rev.* 142(1966)327.
- /2/ J.M.D. Coey, in "Mössbauer Spectroscopy Applied to Inorganic Chemistry", Vol. 1, G.J. Long ed., Plenum Press, New York, 1984, p. 443.