

doi:10.1016/S0016-7037(03)00175-3

# Natural CaTi<sub>2</sub>O<sub>4</sub>-structured FeCr<sub>2</sub>O<sub>4</sub> polymorph in the Suizhou meteorite and its significance in mantle mineralogy

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(Received November 21, 2002; accepted in revised form February 27, 2003)

**Abstract**—The first natural occurrence of a high-pressure polymorph of chromite has been discovered in the shock-metamorphosed Suizhou meteorite. The composition of this high-pressure polymorph is identical to that of the precursor chromite. The Raman spectrum of this polymorph is distinct from that of chromite. Synchrotron X-ray diffraction analysis revealed that this polymorph has an orthorhombic CaTi<sub>2</sub>O<sub>4</sub>-type structure. The cell parameters are: a = 9.462(6) Å, b = 9.562(9) Å, c = 2.916(1) Å, V = 263.8(4) Å<sup>3</sup> (Z=4), space group = *Bbmm*, and the density = 5.63 g/cm<sup>3</sup> (the numbers in parentheses are standard deviations on the last significant digits). This polymorph is 11.5% denser than chromite. The P-T conditions for the phase transformation from chromite to the CaTi<sub>2</sub>O<sub>4</sub>-structured polymorph are estimated at 20–23 GPa and 1800 to 2000 °C, respectively. This dense CaTi<sub>2</sub>O<sub>4</sub>-structured FeCr<sub>2</sub>O<sub>4</sub> phase could be a host phase for Cr, Al, Fe, Mg and Mn and other metallic elements in the deep Earth. *Copyright* © 2003 Elsevier Ltd

## 1. INTRODUCTION

Spinel-type AB<sub>2</sub>O<sub>4</sub> compounds occur in many geological settings of the Earth's crust and mantle, as well as in lunar rocks and meteorites. High-pressure  $AB_2O_4$  compounds are of great importance for the understanding of the constituents of the deep Earth. High-pressure experiments revealed that AB<sub>2</sub>O<sub>4</sub> compounds at high-pressure may adopt CaFe<sub>2</sub>O<sub>4</sub>-, CaMn<sub>2</sub>O<sub>4</sub>- and CaTi<sub>2</sub>O<sub>4</sub>-type structures, whereby their structures attain atomic arrangements such that these structures become denser than spinel (Reid and Ringwood, 1970). Experimental investigations have also indicated that the spinel compounds Mn<sub>3</sub>O<sub>4</sub> and Fe<sub>3</sub>O<sub>4</sub>, and MgFe<sub>2</sub>O<sub>4</sub> transform to CaMn<sub>2</sub>O<sub>4</sub>-type structure at pressure above 25 GPa, that the CaAl<sub>2</sub>O<sub>4</sub> and MgAl<sub>2</sub>O<sub>4</sub> transform to a CaFe<sub>2</sub>O<sub>4</sub> structure at pressure above 26.5 GP, and that the MgAl<sub>2</sub>O<sub>4</sub> structure changes to a CaTi<sub>2</sub>O<sub>4</sub> structure at pressure above 40 GP (Reid and Ringwood, 1969; Mao et al., 1974; Irifune et al., 1991; Funamori et al., 1998; Akaogi et al., 1999; Fei et al., 1999; Andrault and Bolfan-Casanova, 2001). In addition, CaFe2O4-type NaAlSiO4 was experimentally shown to be stable at lower mantle conditions (Liu, 1978; Irifune et al., 1994; Yagi et al., 1994; Tutti et al., 2000). Dense CaFe2O4-, CaMn2O4- and CaTi2O4-structured high-pressure phases have so far not found in nature.

Chromite is an important member of the spinel group. In this paper, we report the discovery of a shock-produced high-pressure polymorph of chromite in a chondritic meteorite. This finding is important for the understanding of the structural characteristics of natural  $AB_2O_4$  compounds under high-pressure and temperature.

## 2. THE SUIZHOU METEORITE

The Suizhou meteorite is a fall that was recovered in Dayanpo in the southeast of Suizhou in Hubei, China, on April 15, 1986. This meteorite was classified as an L6 chondrite, and its shock classification stage as S3 to S4 (Xie et al., 2001, 2002). The meteorite is moderately shock-metamorphosed as expressed by weak mosaicism, planar fractures in olivine and partial isotropism of plagioclase (i.e., presence of maskelynite). The meteorite also contains thin shock-produced veins ranging from 20 to 90  $\mu$ m in width (Fig. 1). The host meteorite consists mainly of olivine, pyroxene, and plagioclase (partially maskelynite), and iron-nickel metal and troilite, in addition to accessory chromite, ilmenite, whitlockite (merrillite) and chlorapatite. As observed for many other shock vein-bearing meteorites (Putnis and Price, 1979; Rubin, 1985; Langenhorst et al., 1995; Chen et al., 1996; Tomioka and Fujino, 1997; Gillet et al., 2000; Langenhorst and Poirier, 2000), the shock veins of the Suizhou meteorite contain abundant high-pressure minerals thought to also occur in the Earth's mantle. The high-pressure mineral assemblages in the shock veins of Suizhou meteorite were previously investigated in detail by Xie et al. (2001, 2002). The high-pressure minerals identified are: (1) coarsegrained polycrystalline aggregates of ringwoodite, majorite, NaAlSi<sub>3</sub>O<sub>8</sub>-hollandite and a high-pressure polymorph of whitlockite formed from the precursor minerals olivine, pyroxene, plagioclase, and whitlockite, respectively; and (2) fine-grained majorite-pyrope garnet and ringwoodite crystallized from shock-induced dense melt. Metal and troilite in the shock veins were molten and occur as eutectic FeNi-FeS grains.

### **3. EXPERIMENTAL TECHNIQUES**

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Polished thin sections of the Suizhou meteorite, containing shock veins, were prepared for analysis of texture, mineral phases and their crystal structure, and their chemical compositions.

The mineral assemblages and textures in thin sections were investigated with a petrographic microscope and a Hitachi S-3500N scanning electron microscope in back-scattered electron (BSE) mode. Compo-



Fig. 1. Back-scattered electron (BSE) image showing a shock vein in the Suizhou meteorite. Olv = olivine; Pyx = pyroxene; Ms = maskelynite; Tr = troilite; M = FeNi-metal.

sitions of minerals were quantitatively determined by a Cameca SX-51 electron microprobe using wave-length dispersive analysis at 15 kV accelerating voltage and 10 nA sample current at the Institute of Geology, Chinese Academy of Sciences.

Raman spectra of minerals in the polished thin section were recorded with a Renishaw RM-1000 instrument at the China University of Geology. A microscope was used to focus the excitation beam (Ar<sup>+</sup> laser, 514 nm line) to 2- $\mu$ m-wide spots and to collect the Raman signal. Accumulations of the signal lasted from 120 to 150 s. The laser power was 26.8 mW.

For determining the crystal structure of micro-phases in a polished thin section, synchrotron X-ray diffraction analyses were conducted at the beamline X17C of the National Synchrotron Light Source of the Brookhaven National Laboratory, New York. The X-ray beam was collimated to a size of 8  $\mu$ m by 18  $\mu$ m. The thin section was rotated to collect diffraction lines. Analysis of diffracted X-rays was carried out with an intrinsic germanium detector. The X-ray diffraction data were acquired at fixed  $2\theta$  (the  $2\theta$  is the angle between the incident X-ray beam and the diffracted X-ray beam) settings of 8, 10, 13 and 15 degree, respectively.

## 4. RESULTS

The chromite grains in the less shocked portions of the meteorite usually contain abundant cracks and fractures (Fig. 2). In the regions adjacent to the shock veins and within the veins, we observed a new phase with only few fractures that has an identical composition to chromite (Figs. 3, 4). This new phase usually occurs as polycrystalline aggregates of 5 to 40  $\mu$ m size. In particular, many intergrowths between chromite crys-



Fig. 2. A chromite grain (Chr) in a, in comparison to the shock veins, less shock metamorphosed area of this meteorite contains abundant fractures that also penetrate the neighboring silicates, in which sets of fractures can be recognized. BSE image. Ms = maskelynite; Olv = olvine; Pyx = pyroxene.



Fig. 3. A BSE image showing an intergrowth of the new phase  $(CaTi_2O_4$ -structured FeCr<sub>2</sub>O<sub>4</sub>, CT) and relic of chromite (Chr). On the image, the new phase is brighter than chromite. The intergrowth area (CT + Chr) occurs between a pure area of chromite (Chr) and a pure area of new phase (CT). This grain was irradiated by the synchrotron beam in an area composed of the new phase (CT) and the intergrowth of two phases (CT + Chr).

tals and the new, relatively electron-brighter phase have been observed in a zone up to 40  $\mu$ m wide adjacent to shock veins.

Figure 3 shows an intergrowth consisting of the new phase and chromite in the matrix directly in contact with the shock vein. One can see on this back-scattered electron image that the new phase is electron-brighter than chromite (Fig. 3). Such intergrowth grains usually show that the amount of the new phase increases closer to the shock vein, whereby the intermediate zone contains both phases. In comparison with cracked chromite grains, the new phase has few fractures. As the shock veins are thought to have been formed due to shock-induced high-pressure and temperature excursions (Putnis and Price, 1979; Rubin, 1985; Stöffler et al., 1991; Langenhorst et al., 1995; Chen et al., 1996; Tomioka and Fujino, 1997; Langenhorst and Poirier, 2000; Xie et al., 2001, 2002), the formation of the new phase appears to be pressuredependent. It, thus, appears that this new phase formed due to shock transformation from precursor chromite.

Inside the shock vein, the grain sizes of the new phase are relatively small (less than 20  $\mu$ m) (Fig. 4). This could mean that the precursor chromite was fragmented first, and then transformed into the new phase. These small grains consist of the new phase only. Neither intergrowths of the new phase and



Fig. 4. A BSE image showing that the shock vein consists of ringwoodite (Rgt), majorite (Mjt), NaAlSi<sub>3</sub>O<sub>8</sub>-hollandite (Hlt), the new phase (CT) and a fine-grained matrix composed of majorite-pyrope garnet, ringwoodite, molten and solidified metal, and troilite.

Table 1. Average compositions of chromite and the new phase. All data in weight %; the number in parentheses is the number of analyses (n.d., not detected; STDEV, standard deviation).

	New phase		Chromite	
	(5)	STDEV	(5)	STDEV
MgO	2.62	0.12	2.62	0.11
FeO	29.70	0.28	29.53	0.25
MnO	0.81	0.08	0.85	0.14
CaO	n.d.		n.d.	
TiO <sub>2</sub>	2.59	0.16	2.95	0.16
$Cr_2O_3$	57.30	0.18	56.81	0.25
Al <sub>2</sub> O <sub>3</sub>	5.94	0.07	6.29	0.17
$V_2O_3$	0.97	0.06	0.91	0.04
	99.93		99.96	

chromite, nor single grains of chromite have been observed inside the shock veins.

Electron microprobe analyses of chromite and the new phase indicate that their chemical compositions are identical. The compositions are: 56-57 wt.% Cr<sub>2</sub>O<sub>3</sub>, 29 wt.% FeO, and small amounts of Al<sub>2</sub>O<sub>3</sub>, MgO, TiO<sub>2</sub>, MnO and V<sub>2</sub>O<sub>3</sub> (Table 1).

Raman spectroscopic analysis indicates that the spectrum of the new phase is distinct from that of chromite. We observed three Raman bands at wave numbers 601, 637 and 664 cm<sup>-1</sup> in comparison to four characteristic bands of chromite at 500,

595, 631 and 677 cm<sup>-1</sup> (Fig. 5). The strong band at 677 cm<sup>-1</sup> in chromite can be assigned the  $A_{1g}$  mode of the spinel structure (Gupta et al., 1993). The strong peak at 601 cm<sup>-1</sup> for the new phase in the Suizhou meteorite is suggestive of a phase with a crystal structure that is different from that of chromite.

We determined the structure of the new phase by synchrotron X-ray diffraction on the same intergrowth shown in Figure 3. The X-ray patterns obtained with the sample in fixed orientation indicate a polycrystalline nature. We collected 21 X-ray reflections from the new phase in addition to several reflections from chromite (Fig. 6 and Table 2), because the target area of the sample irradiated by the X-ray beam contains both the new phase and a small amount of chromite. The crystal structure of the new phase is stable during X-ray irradiation, although the sample was exposed to X-ray irradiation for over 60 h in total. The d spacings of 21 X-ray reflections from the new phase were indexed in the framework of an orthorhombic lattice with the cell parameters: a = 9.462(6) Å, b = 9.562(9) Å, c = 2.916(1)Å (numbers in parentheses are standard deviations for the last significant digits). The space group of this orthorhombic lattice is Bbmm. The d-spacings of the X-ray diffraction lines can be indexed in terms of the CaTi<sub>2</sub>O<sub>4</sub> structure (Bright et al., 1958). The calculated density of the new phase is  $5.63 \text{ g/cm}^3$ , which is 11.5% denser than the chromite structure. This is the first observation of a dense postspinel polymorph for AB<sub>2</sub>O<sub>4</sub> compounds.



Fig. 5. (A) Raman spectrum of chromite. (B) Raman spectrum of the  $CaTi_2O_4$ -structured polymorph of chromite (FeCr<sub>2</sub>O<sub>4</sub>).



Fig. 6. (A) X-ray diffraction pattern obtained on an intergrowth grain consisting of the new phase ( $CaTi_2O_4$ -structured FeCr<sub>2</sub>O<sub>4</sub>) and a relic of chromite, collected at fixed  $2\theta$ =14.9977. In total, 21 diffraction lines could be indexed for the CaTi<sub>2</sub>O<sub>4</sub> structure (CT). The remaining lines belong to chromite (Chr) and FeNi-metal (M).

## 5. DISCUSSION

Intensity

The CaTi<sub>2</sub>O<sub>4</sub>-structure is intimately related to the CaFe<sub>2</sub>O<sub>4</sub>and CaMn<sub>2</sub>O<sub>4</sub>-structures. The orthorhombic CaTi<sub>2</sub>O<sub>4</sub>-, CaFe<sub>2</sub>O<sub>4</sub>-, and CaMn<sub>2</sub>O<sub>4</sub> type structures are composed of dodecahedral (AO<sub>8</sub>) and octahedral sites (BO<sub>6</sub>), in which the differences among these structures lie in slight modifications of the polyhedral linkage (Reid and Ringwood, 1970; Andrault and Bolfan-Casanova, 2001). The only natural occurrence of minerals of any these three structural types is marokite (CaMn<sub>2</sub>O<sub>4</sub>) that has been found in two terrestrial rocks formed at low pressure (Gaudefroy et al., 1963; Villiers and Herbstein, 1968). A CaTi<sub>2</sub>O<sub>4</sub> crystal with space group *Bhmm* was synthesized experimentally (Bertaut and Blum, 1956; Bright et al., 1958). No natural occurrence of CaTi<sub>2</sub>O<sub>4</sub> and CaFe<sub>2</sub>O<sub>4</sub>-structured minerals has been reported to date.

The occurrence of the CaTi<sub>2</sub>O<sub>4</sub>-structured FeCr<sub>2</sub>O<sub>4</sub>-phase is related to a shock vein in the Suizhou meteorite, where peak shock pressure and temperature experienced by this meteorite were located. The P-T conditions for the formation of this CaTi<sub>2</sub>O<sub>4</sub>-structured FeCr<sub>2</sub>O<sub>4</sub> phase can be constrained according to the high-pressure mineral assemblage within the shock vein. The occurrence of ringwoodite, majorite, NaAlSi<sub>3</sub>O<sub>8</sub>hollandite and majorite-pyrope garnet in the shock vein constrains the peak pressure and temperature to 20–23 GPa and 1800–2000 °C, respectively (Agee et al., 1995; Chen et al.,

Table 2. Indexed peaks of the X-ray diffraction pattern and Miller indices for the natural CaTi<sub>2</sub>O<sub>4</sub>-structured FeCr<sub>2</sub>O<sub>4</sub> phase.  $d_{obs}$  and  $d_{cal}$  are observed and calculated d values, respectively. The cell parameters calculated from these data are: a = 9.462(6) Å, b = 9.562(9) Å, c = 2.916(1) Å, V = 263.8(4) Å<sup>-3</sup> (Z = 4), space group = Bbmm, calculated density = 5.63 g/cm<sup>3</sup>.

hkl	d <sub>obs</sub> , Å	d <sub>cal,</sub> Å
111	2.6747	2.6754
040	2.3890	2.3905
240	2.1306	2.1336
420	2.1220	2.1203
311	2.0887	2.0895
321	1.9526	1.9542
141	1.8138	1.8144
501	1.5881	1.5875
511	1.5661	1.5661
521	1.5060	1.5066
012	1.4394	1.4414
351	1.4247	1.4263
222	1.3373	1.3377
412	1.2292	1.2309
602	1.0717	1.0706
262	1.0502	1.0490
632	1.0140	1.0149
642	0.9775	0.9771
123	0.9479	0.9477
323	0.9138	0.9119
333	0.8900	0.8918

1996; Gillet et al., 2000; Xie et al., 2002). There must be a sharp gradient of temperature from the shock veins to the surrounding less shocked material, as the new phase is not observed beyond 40  $\mu$ m from the edge of a shock vein. It indicates that the high-pressure phase transitions are not kinetically obstructed in hot regions, especially inside and close to the shock veins. In comparison to experimentally unquenchable CaMn<sub>2</sub>O<sub>4</sub>-structured Fe<sub>3</sub>O<sub>4</sub> (Fei et al., 1999) and CaFe<sub>2</sub>O<sub>4</sub> (or CaTi<sub>2</sub>O<sub>4</sub>)-structured ZnCr<sub>2</sub>O<sub>4</sub> polymorphs (Wang et al., 2002), this natural CaTi<sub>2</sub>O<sub>4</sub>-structured FeCr<sub>2</sub>O<sub>4</sub> phase is quenchable during decompression.

Experimental investigations indicate that, with increasing pressure, the MgAl<sub>2</sub>O<sub>4</sub> spinel dissociates first to Al<sub>2</sub>O<sub>3</sub> plus MgO above 25 GPa, then both oxides recombine to a CaFe<sub>2</sub>O<sub>4</sub>-type phase above 34 GPa, and finally to a CaTi<sub>2</sub>O<sub>4</sub>-structured phase above 40 GPa (Funamori et al., 1998). We did not find any evidence indicating that chromite was decomposed into Cr<sub>2</sub>O<sub>3</sub> plus FeO or recombined to a CaTi<sub>2</sub>O<sub>4</sub>-structured phase. Direct phase transition from FeCr<sub>2</sub>O<sub>4</sub> to CaTi<sub>2</sub>O<sub>4</sub>-structure phase is, therefore, inferred.

In the spinel group (AB<sub>2</sub>O<sub>4</sub>), the cationic substitutions between  $Al^{3+}$  and  $Cr^{3+}$ , and between  $Mg^{2+}$  and  $Fe^{2+}$  are extensive. Chromespinel (Mg,Fe)(Al,Cr)<sub>2</sub>O<sub>4</sub> is an important accessory mineral in the Earth's mantle, as for example seen in lherzolite from the upper mantle (Scarfe et al., 1979). If the compound (Mg,Fe)(Al,Cr)<sub>2</sub>O<sub>4</sub> or its analogues exist in the transition zone and the lower mantle, they might take the structure of CaTi<sub>2</sub>O<sub>4</sub>- or CaFe<sub>2</sub>O<sub>4</sub>-type compounds. Experiments have indeed demonstrated the existence of CaTi<sub>2</sub>O<sub>4</sub>- and CaFe<sub>2</sub>O<sub>4</sub>-structured MgAl<sub>2</sub>O<sub>4</sub> phases at P-T conditions of the lower mantle (Irifune et al., 1991; Funamori et al., 1998). The natural CaTi<sub>2</sub>O<sub>4</sub>-structured FeCr<sub>2</sub>O<sub>4</sub> phase in the Suizhou meteorite contains about 6 wt.% of Al<sub>2</sub>O<sub>3</sub> and 2.6 wt.% of MgO, whereby the cations  $Al^{3+}$  and  $Cr^{3+}$  occupy octahedral sites (BO<sub>6</sub>) and the Mg<sup>2+</sup> and Fe<sup>2+</sup> occupy the dodecahedral sites (AO<sub>8</sub>). Therefore, the natural occurrence of the CaTi<sub>2</sub>O<sub>4</sub>-structured FeCr<sub>2</sub>O<sub>4</sub> phase in the Suizhou meteorite indicates that the  $CaTi_2O_4$  structure could be an important host phase for  $Cr^{3+}$ ,  $Al^{3+}$  and other metal elements  $(Mg^{2+}, Fe^{2+}, Ni^{2+}, Mn^{2+})$  $Zn^{2+}$  and  $Mn^{3+}$  etc) in the deep Earth.

Acknowledgments—We are grateful to Z. H. Li for providing meteorite sample, and J. Z. Hu for assistance with synchrotron X-ray diffraction analysis. We thank A. El Goresy, F. Langenhorst, W. U. Reimold and P. C. Buchanan for constructive reviews. This project was supported by the Chinese Academy of Sciences under grants KZCX3-SW-123, KJCX2-SW-NO3 and the Program of Hundred Talents, and the National Science Foundation of China under grant 40272028.

Associate editor: W. U. Reimold

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