

## A TEM Study on Fluid Inclusions in Coesite-bearing Jadeite Quartzite in Shuanghe in the Dabie Mountains

WU Xiuling, MENG Dawei and HAN Yujing

Testing Centre, China University of Geosciences, Wuhan 430074, Hubei;

E-mail: [dwmeng@cug.edu.cn](mailto:dwmeng@cug.edu.cn)

**Abstract** Fluid inclusions at a nano to sub-micron scale in quartz from jadeite quartzite at Shuanghe, Dabie Mountains, have been investigated by using the transmission electron microscopy (TEM). Most fluid inclusions are spherical or negative crystal shaped, forming wide swarm-like trails. The TEM reveals that the relationship between coesite and the host quartz is syntaxial and provides strong evidence of the occurrence of high-salty fluids at peak metamorphic conditions. The fluid inclusions are often connected to dislocations, which are undetected at the scale of optical microscopy. Non-decrepitation leakage of fluid inclusions may occur by pipe diffusion of molecule  $H_2O$  or  $CO_2$  along dislocations from the inclusions into the host quartz, thus leading to original inclusion density and composition changes. It should be taken into full account for the correct petrological interpretation of micro-thermometric results.

**Key words:** UHP jadeite-quartzite, quartz, nano-scale fluid inclusion, TEM, Dabie Mountains

### 1 Introduction

Fluid inclusions in coesite-bearing jadeite quartzite at Shuanghe, Dabie Mountains have been investigated by using optical microscopy and micro-thermometric measurements revealed that most inclusions were re-equilibrated at lower densities, with a wide spread of measured density data (You et al., 1996; Han et al., 1997; Fu et al., 2001). Because of the rapid drop of external pressure occurring in the uplift of the rocks, the fluid pressure exceeds the confining pressure and inclusions may decrepitate. But no evidence of decrepitation during re-equilibration was observed at the optical scale. The transmission electron microscopy (TEM with line resolution of 0.1 nm and point resolution of 0.2 nm) allows a detailed characterization of textural and chemical features of fluid inclusions at a resolution higher than that attainable with an optical microscope (Viti and Frezzotti, 2001). Therefore, TEM investigation of the fluid inclusions is necessary to understand the re-equilibration mechanism.

### 2 Samples and Analytical Methods

Fluid inclusions are found to occur notably in quartz based on petrography studies (You et al., 1996; Han et al., 1997; Fu et al., 2001). The samples for TEM studies were crystal grains of quartz from the jadeite quartzite at Shuanghe, Dabie Mountains. The geological background and mineral assemblages of the rocks have been reported (Cong et al., 1995; You et al., 1996; Dong and Guo, 1996;

Liou et al., 1997; Fu et al., 2001). The essential minerals are quartz (45%–60%) and jadeite (30%–50%) with subordinate garnet (5%). Peak metamorphic *P-T* conditions estimated by Liou et al. (1997) were  $>2.7$  GPa at  $660 \pm 40^\circ\text{C}$  and Okay (1993) suggested a minimum pressure of 2.2 GPa at  $800 \pm 50^\circ\text{C}$ .

The specimens for TEM studies were prepared using an Gatan-600 type ion-beam thinning device working at an accelerating voltage of 4 kV and an ion beam current of  $<0.8$  mA. The selected area electron diffraction (SAED) pattern and TEM bright-field images were taken at 200 kV on a H-8100 TEM ( $\pm 30^\circ$  tilt, spherical aberration  $C_s = 2.0$  mm) equipped with an energy-dispersive X-ray spectrometer (EDS), which has an ultra-thin-window detector.

### 3 Results and Discussions

TEM observations have revealed that fluid inclusions on a nano ( $<100$  nm) to a sub-micron ( $0.1$ – $1.0$   $\mu\text{m}$  or  $100$ – $1000$  nm) scale occur widely in quartz crystals, most inclusions ranging from  $0.01$  to  $0.30$   $\mu\text{m}$  in size. The inclusion shape is variable, frequently spherical or nearly negative crystal-shaped, especially in smaller inclusions. Commonly, these inclusions form regular trails or wide swarm-like trails (Fig. 1), and some of them are isolated in quartz. They are often empty or partially empty due to ion-thinning of the specimens. Figure 2 shows halite daughter crystals in the cavity of fluid inclusion hosted by quartz, and coesite at a nano scale ( $\sim 80$  nm or  $0.08$   $\mu\text{m}$ ) present in quartz. The SAED patterns in Fig. 2b and 2c confirm the

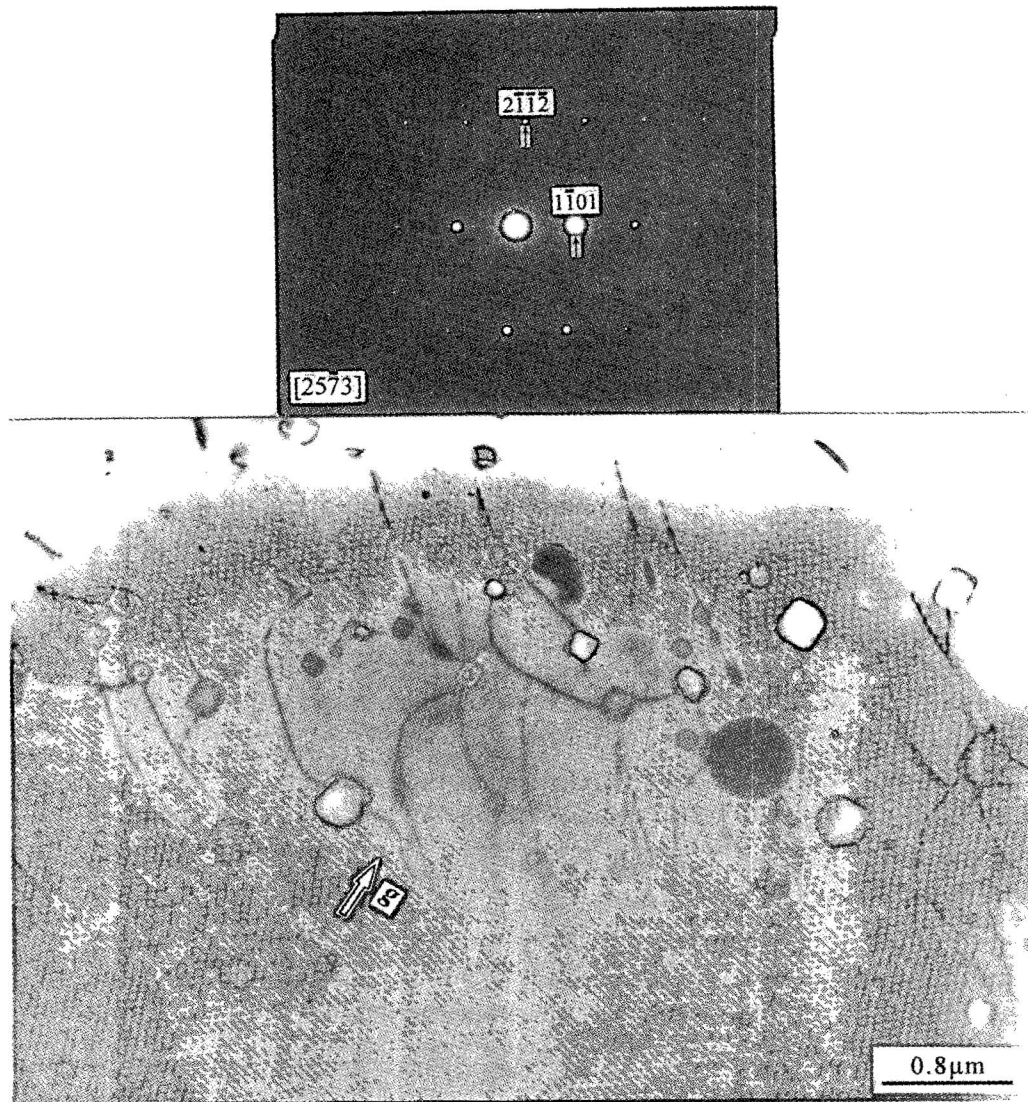


Fig. 1. Bright-field TEM micrograph ( $g = 1\bar{1}01$ ) of quartz showing small strain-free bubbles at dislocations and isolated between dislocations, and the associated SAED pattern of the hosted quartz crystal corresponding to crystal zone  $[2573]$  (Scale bar:  $0.8\mu\text{m}$ ).

existence of halite and coesite respectively. The SAED patterns in Fig. 2c shows the syntaxic relationship between coesite and quartz is, that is, the crystal zone  $[021]_C$  of coesite is parallel to the crystal zone  $[12\bar{3}1]_Q$  of quartz, and  $\{11\bar{2}\}_C$  (or  $\{hh\bar{2}h\}_C$ ,  $h = 0, \pm 1, \pm 2, \dots$ ) of coesite and  $\{1\bar{1}01\}_Q$  (or  $\{h\bar{h}0h\}_Q$ ,  $h = 0, \pm 1, \pm 2, \dots$ ) of quartz share a crystal face. It indicates that the quartz crystals have been transformed from coesite and that the existent coesite is a residual. The occurrence of high-salty fluids at peak metamorphic conditions is supported by the TEM observations. Besides, carbonic inclusions at a sub-micron scale were also detected in quartz. Fig. 3 shows the SAED pattern of quartz and carbonic inclusions with a shape of the negative crystal hosted by quartz. The EDS analyses demonstrate that the dark phase indicated with "A" in the inclusion in Fig. 3 corresponds to  $\text{CO}_2$  (Fig. 4).

A very important feature revealed by the TEM is that fluid inclusions are always connected to dislocations which are undetected at the optical microscopic scale. In Fig. 1, small strain-free bubbles are at a dislocation or isolated between dislocations, indicating that recovery processes may have operated (Bakker and Jansen, 1994; Joreau et al., 1997). In Fig. 5, small negative crystal-shaped inclusions are arranged in a trail and connected with a dislocation network. These dislocations play a major role in molecular  $\text{H}_2\text{O}$  diffusion through quartz crystals. They are formed by fluid overpressure in the inclusion and provided many routes for non-decrepitation preferential  $\text{H}_2\text{O}$  leakage (Bakker and Jansen, 1994). In addition,  $\text{CO}_2$  inclusions connected to dislocations are also detected (Figs. 3 and 4), which could have determined a fluid phase leakage. The dislocations represent a favorable path for  $\text{CO}_2$  diffusion.

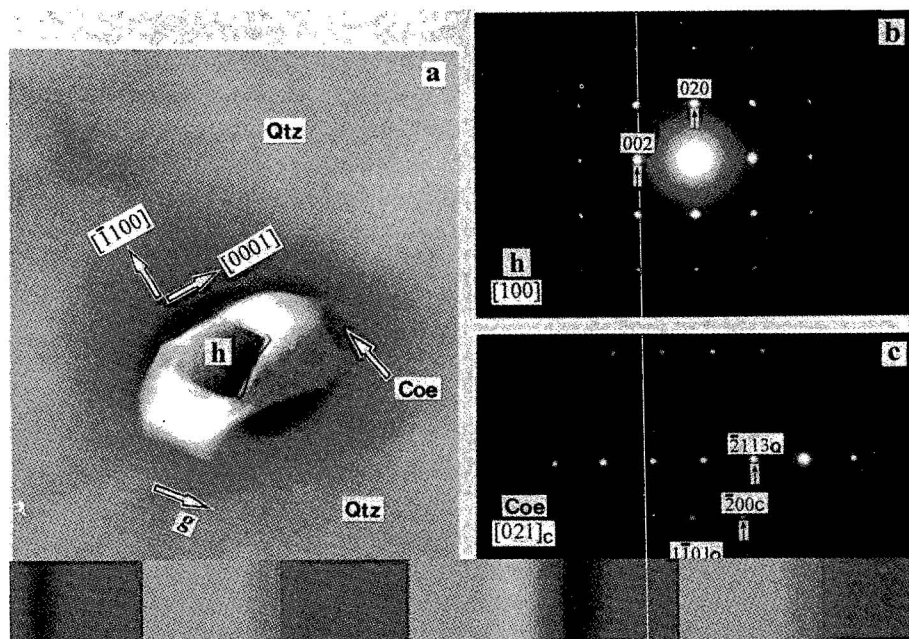


Fig. 2. Bright-field TEM micrograph ( $g = 1101$ ) of quartz (Qtz).

(a) Appearance of coesite (Coe) in quartz and halite daughter crystal (h) in a cavity of Qtz fluid inclusion (scale bar:  $0.1 \mu\text{m}$ ); (b) The SAED pattern of halite corresponding to crystal zone  $[100]$ ; (c) The SAED patterns of Qtz (strong diffraction spots) and Coe (weak diffraction spots), corresponding to crystal zone  $[12\bar{3}1]_Q$  and  $[021]_C$  respectively.

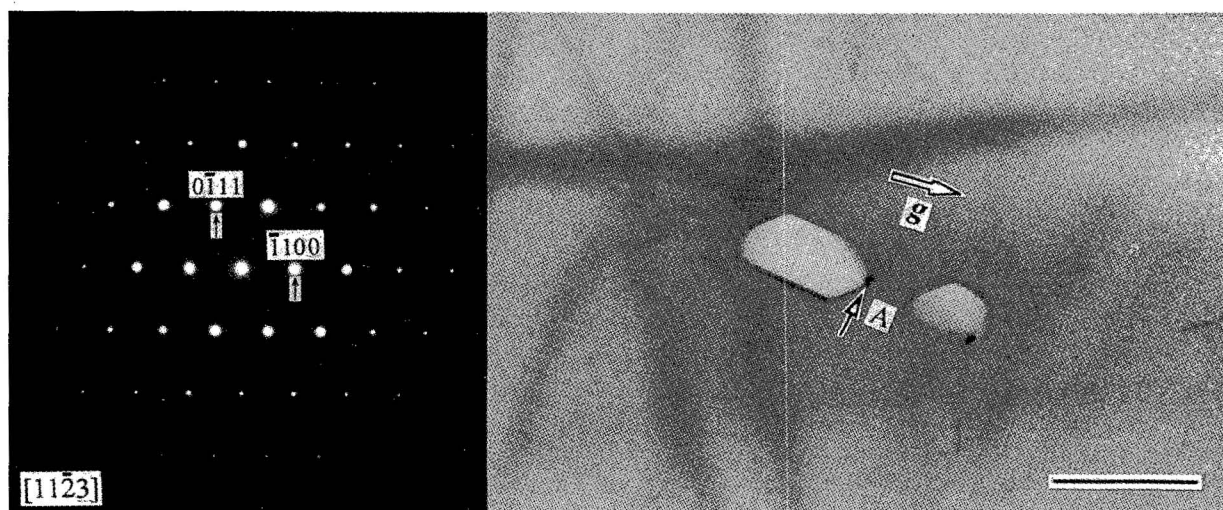


Fig.3. Bright-field TEM micrograph ( $g = 1\bar{1}00$ ) of quartz showing negative crystal-shaped inclusions containing  $\text{CO}_2$  connected to dislocations, and the associated SAED pattern of the host quartz corresponding to crystal zone  $[11\bar{2}3]$ , scale bar representing  $0.4 \mu\text{m}$ .

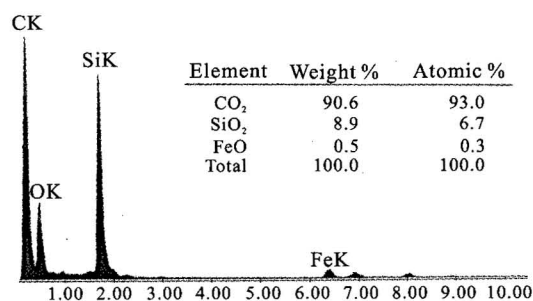


Fig. 4. EDS spectrum of "A" in Fig. 3.

The variability on measured  $\text{CO}_2$ -fluid densities, and the appearance of very low-density values by micro-thermometric methods (Han et al., 1997; Fu et al., 2001) may be due to the  $\text{CO}_2$  leakage. Consequently, the mechanism of re-equilibration could be attributed to a fluid leakage by pipe diffusion along dislocations (Viti and Frezzotti, 2000, 2001; Van den Kerkhof et al., 2001).

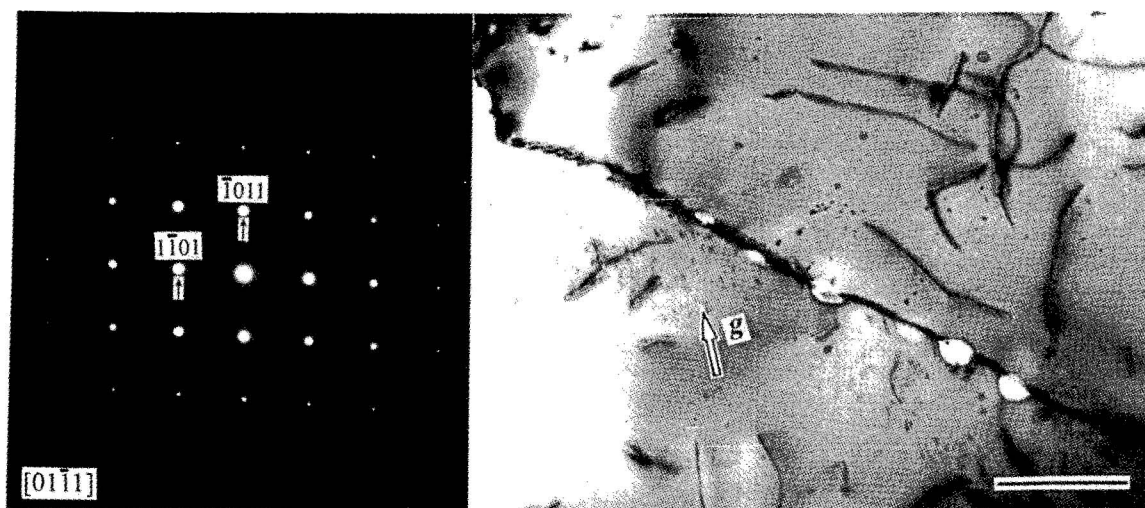


Fig. 5. Bright-field TEM micrograph ( $g = 10\bar{1}1$ ) of quartz showing a trail of fluid inclusions in a dislocation network, and the associated SAED pattern of the host quartz crystal corresponding to crystal zone  $[01\bar{1}1]$  (scale bar: 1.0  $\mu\text{m}$ ).

## 4 Conclusions

(1) The TEM reveals that fluid inclusions at a nano to sub-micron scale, with spherical or negative crystal shape, occur as groups or isolated crystals in quartz, most of which are aqueous inclusions with some containing  $\text{CO}_2$  fluids.

(2) TEM observations provide strong evidence of transformation from original coesite into quartz and the occurrence of high-salty fluids at peak metamorphic conditions.

(3) Fluid inclusions are commonly associated with numerous interlinked dislocations, which are undetected by optical microscopy. The dislocations represent a favorable path for non-decrepitative fluid phase leakage, leading to modification of the original density and composition of the inclusions. It is clear that further detailed petrographic studies and re-examination of samples that record peak densities are needed.

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## References

- Bakker, R. J., and Jansen, J.B.H., 1994. A mechanism for preferential  $\text{H}_2\text{O}$  leakage from fluid inclusions in quartz, based on TEM observations. *Contrib. Mineral. Petrol.*, 116: 7–20.
- Cong, B., Zhai, M., Carswell, D.A., Wilson, R.N., Wang, Q., Zhao, Z., and Windley, B.F., 1995. Petrogenesis of ultrahigh-pressure rocks and their country rocks at Shuanghe in Dabieshan, Central China. *Eur. J. Mineral.*, 7: 119–138.
- Dong, H., and Guo, Z., 1996. Structural aspects of ultrahigh-pressure metamorphic rocks at Shuanghe, Dabie Mountains, China. *Science in China (Series D) Supp.*, 39: 102–112.
- Fu, B., Touret, J.L.R., and Zheng, Y.-F., 2001. Fluid inclusions in coesite-bearing eclogites and jadeite quartzite at Shuanghe, Dabie Shan (China). *J. Metamorphic Geol.*, 19: 529–545.
- Han, Y., Zhang, Z., and Liu, R., 1997. Fluid inclusions in coesite-bearing jadeite quartzite from Shuanghe, Anhui Province. *Earth Science - J. China Univ. of Geosci.*, 22: 322–326 (in Chinese with English Abstract).
- Joreau, P., Reimold, W.U., Robb, L. J., and Doukhan, J.C., 1997. A TEM study of deformed quartz grains from volcanoclastic sediments associated with the Bushveld Complex, South Africa. *Eur. J. Mineral.*, 9: 393–401.
- Liou, J.G., Zhang, R.Y., and Jahn, B.M., 1997. Geochemistry and isotope data on an ultrahigh-pressure jadeite quartzite from Shuanghe, Dabie Mountains, Eastern China. *Lithos*, 41: 59–78.
- Okay, A.I., 1993. Petrology of a diamond and coesite-bearing metamorphic terrain: Dabie Shan, China. *Eur. J. Mineral.*, 5: 659–673.
- Van den Kerkof, A.M., and Hein, U.F., 2001. Fluid inclusion petrography. *Lithos*, 55: 27–47.
- Viti, C., and Frezzotti, M.L., 2000. Re-equilibration of glass and  $\text{CO}_2$  inclusions in xenoliths olivine: a TEM study. *Am. Mineral.*, 85: 1390–1396.
- Viti, C., and Frezzotti, M.L., 2001. Transmission electron microscopy applied to fluid inclusion investigations. *Lithos*, 55: 125–138.
- You, Z., Han, Y., Yang, W., Zhang, Z., Wei, B., and Liu, R., 1996. *The High-Pressure and Ultra-High-Pressure Metamorphic Belt in the East Qinling and Dabie Mountains, China*. Wuhan: China University of Geosciences Press, 150.