

Research Article

Ultrastructure of a Columbite-Tantalite Mineral from the Zhaojinggou Ta-Nb Deposit in the North China Craton: Direct Evidence of the Formation Mechanism of the Columbite-Group Minerals

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The formation of columbite-group mineral phases in peraluminous granite has not been demonstrated to date. Here, a nanoscale study of the columbite-tantalite mineral in the Zhaojinggou Nb-Ta deposit in North China Craton elucidated its formation mechanism and the role of fluids in Nb-Ta mineralization. Transmission electron microscopy (TEM) analysis of a focused ion beam cut of the columbite-tantalite mineral revealed a comparatively well-ordered mineral structure. Energy-dispersive X-ray spectroscopy (EDS) revealed the presence of Nb, Ta, Mn, W, Fe, Sn, and Pb in the columbite-tantalite mineral. Furthermore, detailed TEM images depicted the nanoscale hydrothermal fluid occurring within the columbite-group mineral grain as well as between columbite-tantalite mineral and quartz grains. K, Al, Si, and O were found to be enriched in the hydrothermal fluid that was present between the quartz grains and the columbite-tantalite mineral. It did not react with the mineral grains of the columbite group. The ultrastructure of the columbite-tantalite mineral suggested the columbite-group mineral in the Zhaojinggou Nb-Ta deposit formed during magmatic crystallization rather than from hydrothermal fluids. Furthermore, HR-TEM images provided the first nanoscale observations of the fluid-mediated mineral dissolution and amorphous phase formation. This study also revealed that the mineral dissolution, element transport, and reprecipitation were significantly influenced by the fluid amorphous phase in the Nb-Ta deposits.

1. Introduction

Tantalum and niobium have numerous applications due to their unique physical properties [1, 2]. Peraluminous granite is considered an important source of Ta and Nb [3–6]. There are two main theories regarding the formation of Nb- and Ta-rich phases. According to one view, experimental data showing a higher affinity of Nb and Ta for silicate melts than hydrothermal fluids supports the idea that magmatic crystallization is the source of Nb-Ta mineralization [7–10]. However, another view suggests the formation of Nb- and Ta-rich phases from late exsolved, hydrothermal fluids, or fluid-rich melts owing to the textural relationship and com-

position of columbite-group minerals [11–17]. Various researchers have deduced the specific roles of magmatic and hydrothermal processes in the formation of magmatic rare elements. The current research methods focused on geochemistry (e.g., trace elements of whole rocks) and mineralogy (e.g., composition characteristics of minerals (usually $>10\ \mu\text{m}$)) in the Nb-Ta deposit. However, the nanoscale research has emerged into the ore mineralogy and geochemistry field, and these researches have revealed some unknown parameters via the nanoscale ore processes [18, 19]. For example, the old-standing problem of “invisible Au” and of other elements (e.g., Ag, Pb, and Te) could finally be resolved by applying nanomineralogical and

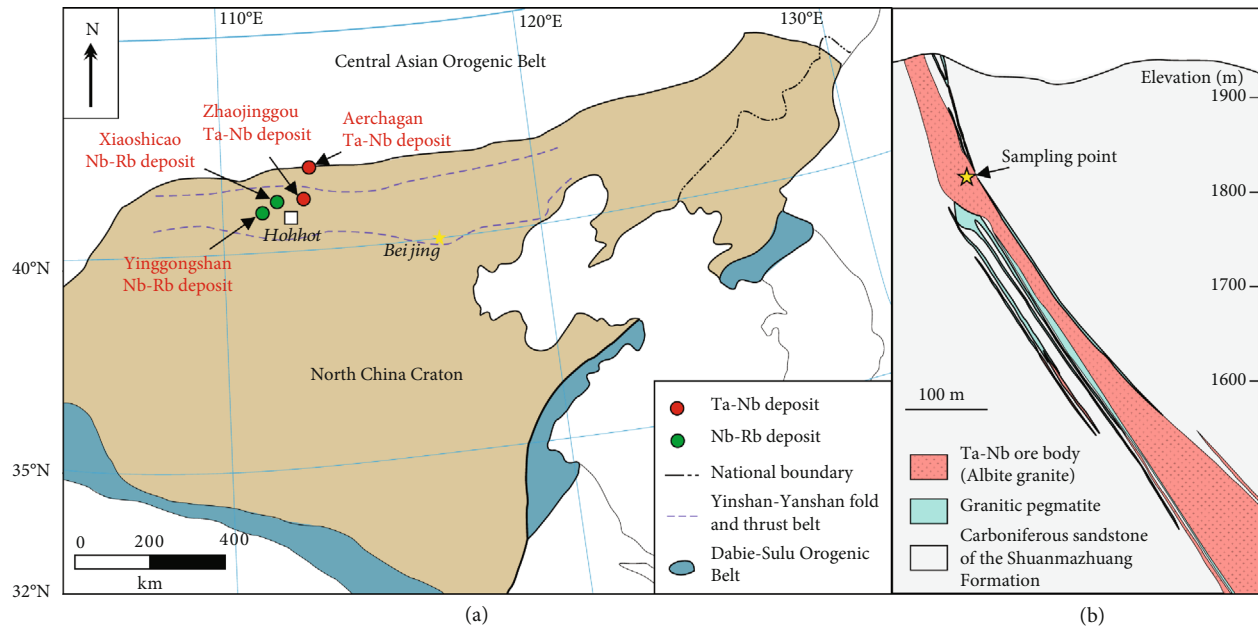


FIGURE 1: (a) Simplified geological map of the Zhaojinggou Nb-Ta deposit at the northern margin of the North China Craton (modified from Zhu true [27]). (b) Cross section of the Zhaojinggou Ta-Nb deposit (modified from Chai and Wu [31]).



FIGURE 2: Ore types of the Zhaojinggou Ta-Nb deposit, showing that the ore mainly are massive ore including quartz (purple in color) and amazonite (blue in color).

nanogeochemical methodologies [20, 21]; the mineral nanosized studies of minerals reflect the formation stages of ore-forming systems [22, 23]. In addition, transmission electron microscopy (TEM) has been widely used to reveal nanoscale mineral interactions with fluids. Therefore, direct evidence for columbite-group mineral formation can be ascertained from studying the ultrastructure of these minerals.

This study focused on the ultrastructure and nanoscale chemical compositions of the columbite-group mineral in the Zhaojinggou Ta-Nb deposit, North China Craton. In addition, the hydrothermal effects on columbite-group mineral crystallization were also discussed, and the observed findings support the processes that lead to Ta-Nb mineralization.

2. Geological Background

The Zhaojinggou Ta-Nb deposit is located in the northwest North China Craton, which consists of an Archean to Paleoproterozoic metamorphic basement and a Mesoproterozoic

to Permian platform cover (e.g., [24]) (Figure 1(a)). The predominant constituents of the metamorphic basement are serpentinized marble and biotite quartz schist. Additionally, the platform cover is primarily composed of feldspathic quartz sandstone and feldspar sandstone interbedded with marble. Large-scale tectonic thermal events in the early Cretaceous (130–120 Ma) [25] were triggered by the early Mesozoic thinning and destruction of the lithospheric crust [26, 27]. Significant early Cretaceous magmatism occurred in this region simultaneously [28, 29], along with significant gold [30], W, Fe, and Ta-Nb mineralization [31, 32]. This region experienced intense tectonic activity. In general, it has undergone multistage ductile shear deformation, nappe structure, and fold structure since the Archean and is distinguished by an early-stage ductile shear zone and brittle fracture and a nappe structure in the later stage [28, 29].

The Zhaojinggou Ta-Nb can be found in the easternmost part of the Daqingshan metallogenic belt, which also produced numerous gold and REE deposits, such as the

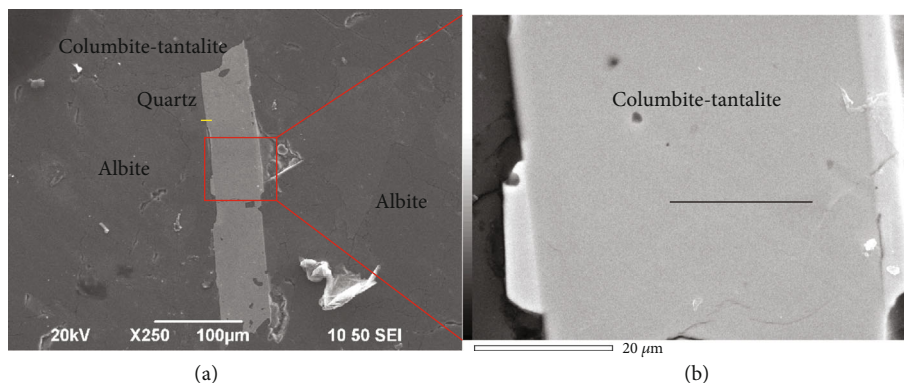


FIGURE 3: SEM images of the columbite-tantalite mineral. The yellow and black lines indicate the positions of the FIB section and SEM line analysis. Those results are illustrated in Figures 6 and 4, respectively.

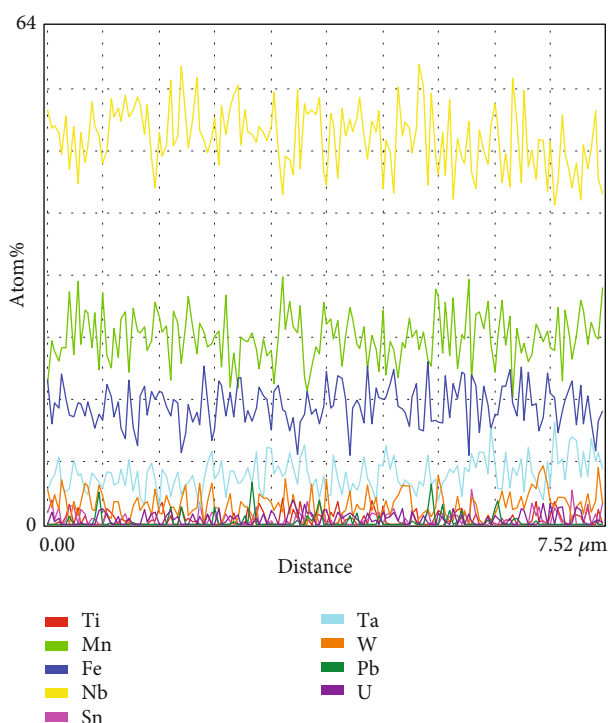


FIGURE 4: SEM line analysis of columbite-tantalite mineral grain indicating the homogeneously distributed elements.

Changfulonggou gold deposit, the Mouduqing gold deposit, the Luchang gold deposit, the Zhongdiyaozi gold deposit, the Motianling gold deposit, the Aerschagan REE deposit, and the Guangfengdexi polymetallic deposit. More than 38 Mt of ore is stored in the Zhaojinggou Ta-Nb deposit with an average Ta_2O_5 and Nb_2O_5 contents of 0.013 wt.% and 0.012 wt.%, respectively [31]. The Ta-Nb mineralization occurs within a ~400 m long and 20–100 m wide albite granite body [31]. The ore body invades the Shuanmazhuang Formation, a Carboniferous sandstone formation. Cross-sections revealed that the ore body extends to ~770 m in depth (Figure 1(b)). The ore mainly is composed by the quartz and amazonite (Figure 2). Ta and Nb are predominantly found in columbite-group minerals, which have a euhedral crystal-line structure and are distributed between albite and quartz.

3. Analytical Methods

On polished thin petrographic sections, standard microscopic analyses were performed using a Zeiss-Opton light microscope with transmitted light. SEM observations were performed on polished rock platelets using a Hitachi SU8220 instrument at 5 kV and 10 kV. Energy-dispersive X-ray spectrometry (EDS) mapping was used to determine the major elemental distribution. TEM (FEI Tecnai G2 F20) equipped with an EDS detector was used to acquire structural/chemical information about the columbite-tantalite. The maximum accelerating voltage was found to be 200 kV. The TEM foil was extracted for attachment to Cu grids via Pt welding and then trimmed to 50–70 nm thicknesses. High-angle annular dark field (HAADF) STEM imaging was performed using an ultrahigh resolution and a probe-corrected FEI Titan Themis TEM. Digital micrograph software from Gatan (version 3.7.4) was used to process high-resolution transmission electron microscopy (HRTEM) images, including fast Fourier transform (FFT). All analyses, including SEM and TEM, were carried out at the Sinoma Institute of Materials Research (Guangzhou) Co., Ltd.

4. Results

4.1. Grain-Scale Characterization for the Columbite-Group Mineral. This mineral occurs as a single euhedral grain that is exceedingly coarse ($>200 \mu m$) (Figure 3) and is intergrown with albite and quartz within albite granite. According to the SEM data, the columbite-group mineral is primarily composed of Nb, Ta, Mn, W, Fe, Sn, and Pb. These elements were uniformly distributed in the columbite-group mineral grain, as determined by SEM line and mapping (Figures 4 and 5). Focused ion beam- (FIB-) SEM nanoscale sampling targeted the columbite-group mineral and quartz boundary.

4.2. Nanoscale Columbite-Group Mineral Characterization. A FIB-cut of our columbite-group mineral sample obtained from the area shown in Figure 4(a) was subjected to TEM imaging. The selected area electron diffraction (SAED) pattern of the domain (dotted rectangle) in Figure 6(c) exhibited a relatively well-ordered structure of columbite-group mineral with one of the d-spacings measured at 7.12 \AA

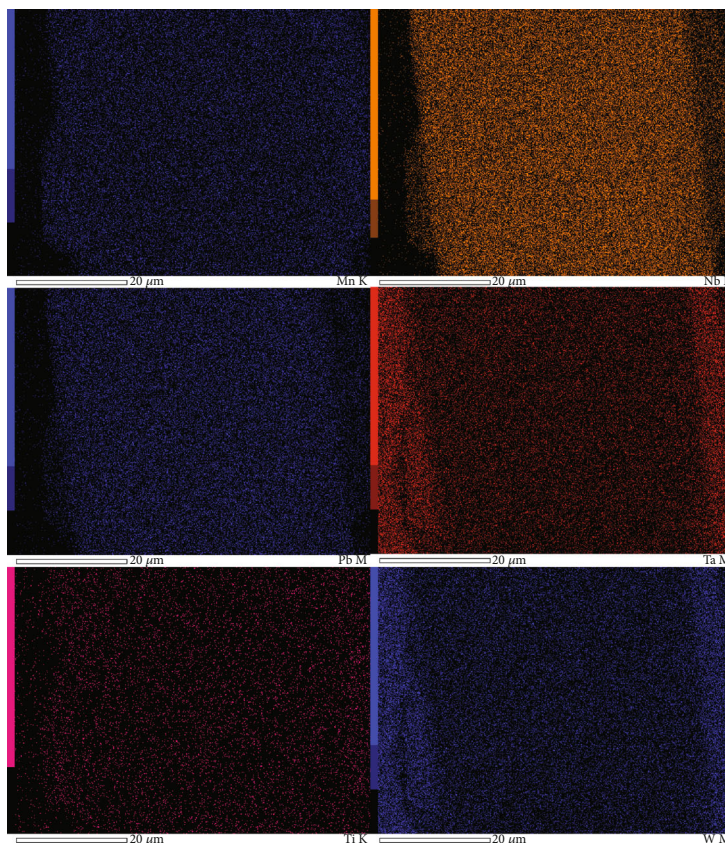


FIGURE 5: SEM mapping image of the columbite-tantalite mineral grain, indicating homogeneously distributed elements.

(Figures 6(c) and 6(d)). In contrast, a detailed TEM image of the domain (red lines) displayed the nanoscale hydrothermal dissolution behavior as shown in Figure 6(b). Furthermore, dissolution trails were prevalent in the grain. EDS spot analyses and mapping revealed that the columbite-group mineral contained Nb, Ta, Mn, W, Fe, Sn, and Pb (Figures 7–9), corroborating SEM findings.

Moreover, a nanoscale hydrothermal channel existed between the columbite-group mineral and quartz (Figure 6(a) and S1). Quartz possess a relatively well-ordered structure including the columbite-group mineral with one of the d-spacings measured at 6.6 Å (Figure 10). The hydrothermal channels are 200 nm wide, and numerous nanoparticles have been found along the trails (Figure 6(a)). The edge between the hydrothermal channel and the columbite-group mineral is straight, whereas the edge between the hydrothermal channel and quartz was variable. The composition of the hydrothermal fluid was enriched with K, Al, Si, and O but depleted Nb, Ta, Fe, Mn, Pb, U, W, and Sn (Figures 7–9). In addition, numerous flow traces were observed in the hydrothermal channel.

4.3. Fluid-Mediated Mineral Dissolution and Amorphous Phase Formation. HR-TEM images of the studied samples revealed the complex chemical and mineralogical changes that occur during mineral dissolution. The diffuse, cloudy, and patchy appearance along the grain boundaries was

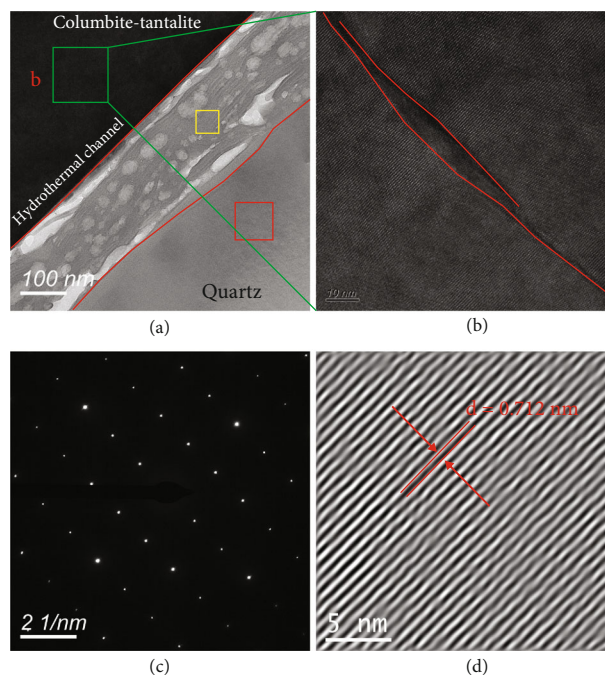


FIGURE 6: (a) Focused ion beam-cut of the columbite-tantalite mineral. (b) High-magnification transmission electron microscopy image obtained from the green square in (a). (c) Selected area electron diffraction pattern obtained from the green square in (a). (d) High-resolution transmission electron microscopy image obtained from the green square in (a).

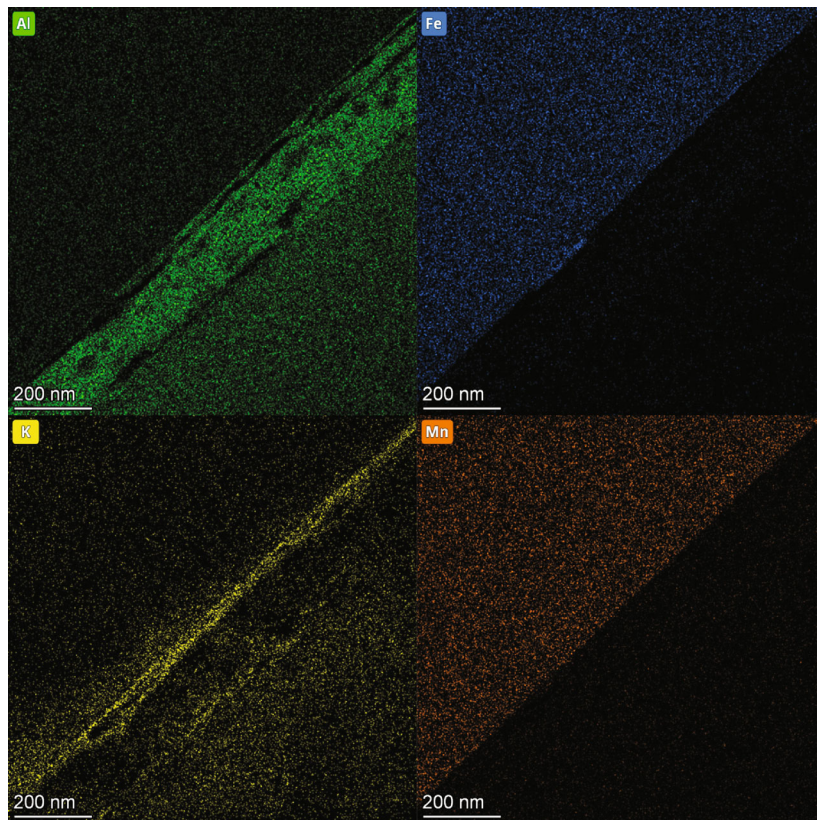


FIGURE 7: Al, Fe, K, and Mn maps of a FIB-cut of the columbite-tantalite mineral. The location of this FIB-cut is the same as in Figure 6.

observed as the first indicator of fluid-mediated mineral dissolution (Figure 6(a)). The second indicator was defect-rich areas within the columbite-group mineral grain that corresponded to cloudy and patchy compositional zoning. The lack of diffraction contrast in these cloudy materials is indicative of their amorphous nature (Figure 11). The surface of the reacting quartz was highly irregular, indicating its dissolution (Figure 6(a)). This material emerged from the dissolving crystal and reached the columbite-group mineral. Close inspection of a SAED image of the defect-rich regions of the crystal of a columbite-group mineral revealed amorphous material forming along the defect lines (Figure 6(b)).

5. Discussion

5.1. Tracing the Nanoscale Genesis of the Columbite-Group Mineral. Observations encompassing the micron to nanoscale revealed that the columbite gains in the granite were euhedral and that the crystal structure was relatively regular. There were no crystalline flaws in the ordered lattices. In addition, there were no sudden changes in ion species composition observed in the columbite gain. The subsequent fluid-rich stage resulted in the formation of anhedral, replacement-textured columbite-group minerals [33]. Nonetheless, this research found that the columbite-group mineral textures in the Zhaojinggou Nb-Ta deposit were distinct from those formed in the fluid stage, suggesting that the columbite-group mineral may form in the deposit via magmatic fractional crystallization. Additionally, nanoscale crystal dissolutions in the mineral boundary may

have resulted from mineral replacement. In contrast, no crystallization dissolutions were observed in the columbite-group mineral boundary, indicating that it may not have formed during the hydrothermal fluid stage. On the contrary, there is hydrothermal dissolution occurring within the columbite-group mineral (Figure S2), indicating that the columbite-group mineral is formed before the hydrothermal fluid stage. The current findings suggest that the columbite-group mineral in the Zhaojinggou Nb-Ta deposit was formed during the magmatic stage and not the hydrothermal fluid stage.

5.2. First Discovery of a Nanoscale Hydrothermal Fluid in the Nb-Ta Deposit. During the final phase of highly evolved magma, large quantities of fluids are expelled through melting. Hydrothermal fluids derived from magma are typically enriched in numerous elements and have a close relationship with diagenesis and mineralization. However, hydrothermal fluid research focuses primarily on the hydrothermal vein body on a macroscopical scale (>cm) and mineral texture characteristics (e.g., replacement texture and zonal structure) on a micro-scale ($\sim\mu\text{m}$). Furthermore, the nanoscale hydrothermal fluid, which may represent the initial fluid characteristic, has never been directly observed in any previous research [33].

The hydrothermal fluid was first observed on a nanoscale between the columbite-group mineral and quartz. This fluid contained K, Al, Si, and O, and there was obvious interaction with quartz but no interaction with a columbite-group mineral. A nanoscale fluid was discovered within the columbite-group mineral. These results may provide a direct

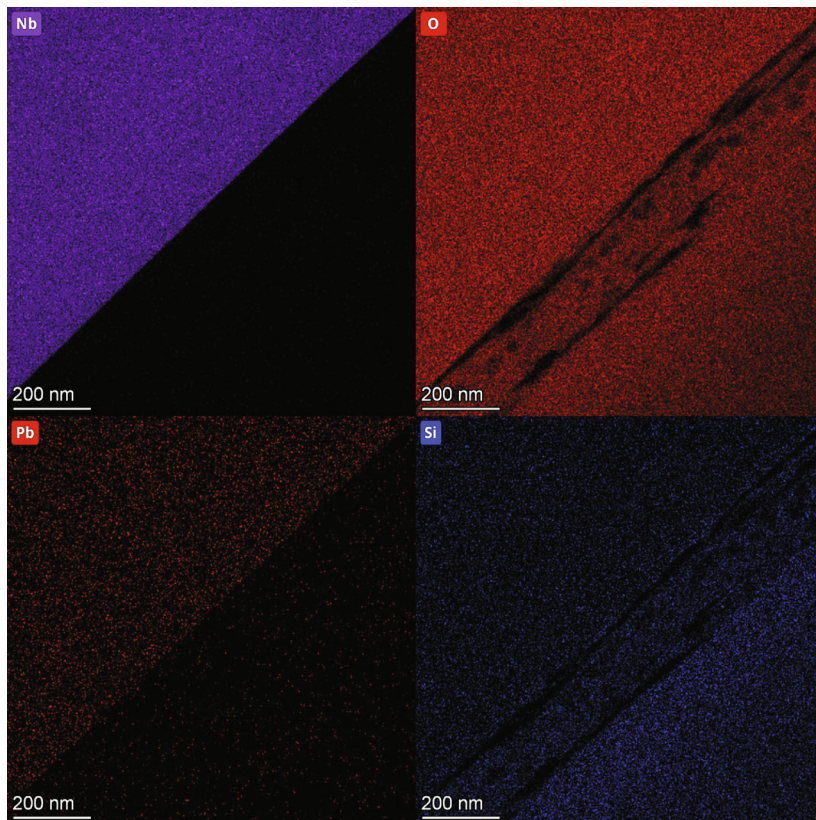


FIGURE 8: Nb, O, Pb, and Si maps of a FIB-cut of the columbite-tantalite mineral. The location of this FIB-cut is the same as in Figure 6.

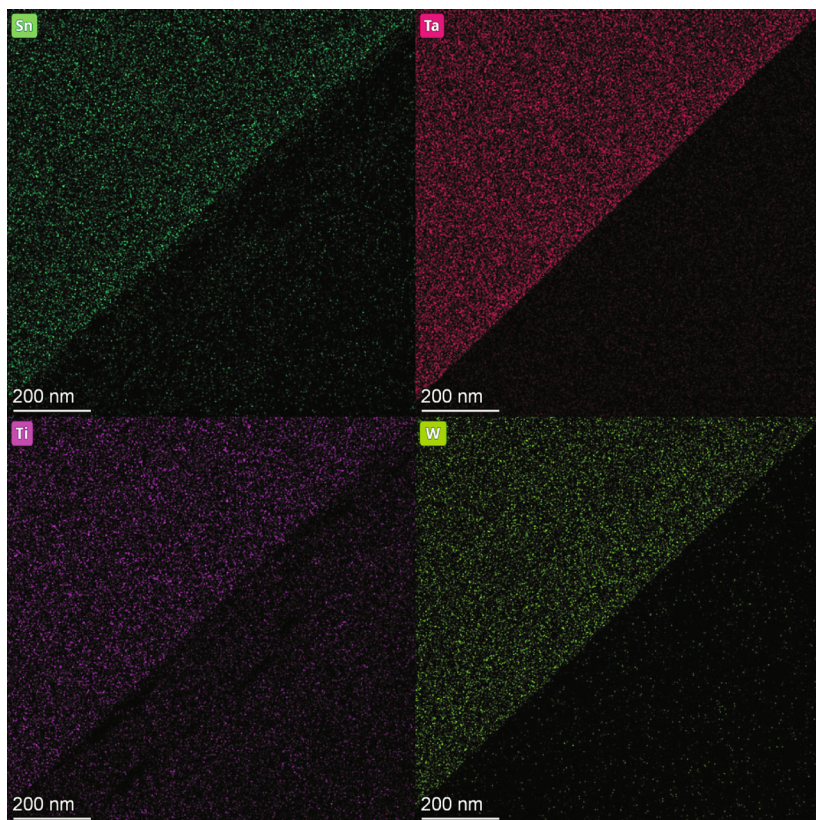


FIGURE 9: Sn, Ta, Ti, and W maps of a FIB-cut of the columbite-tantalite mineral. The location of this FIB-cut is the same as in Figure 6.

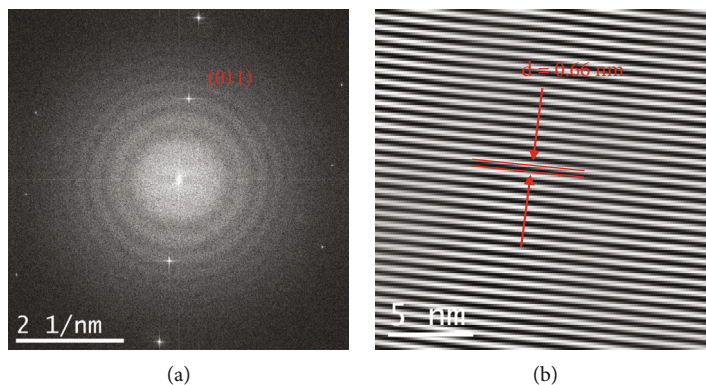


FIGURE 10: (a) SAED pattern of quartz obtained from the area enclosed within the red square in Figure 6(a). (b) HRTEM image of quartz obtained from the colored square in Figure 6(a).

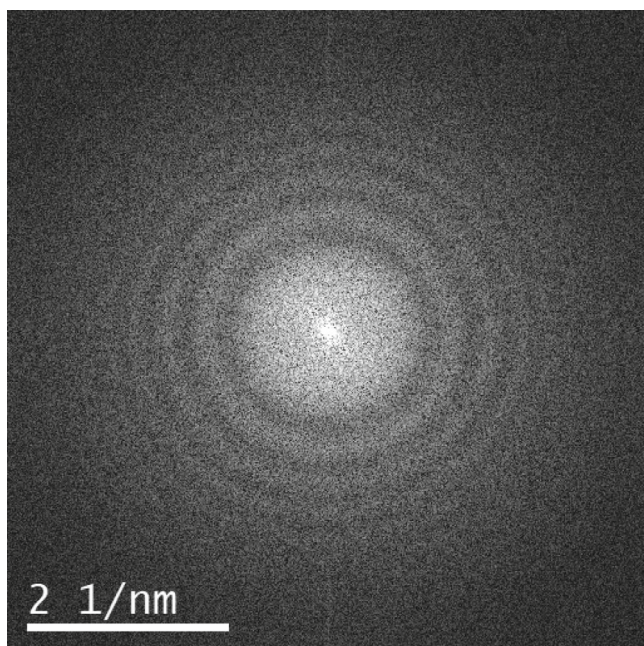


FIGURE 11: SAED pattern of hydrothermal channel obtained from the yellow square in Figure 6(a), indicating that the hydrothermal channel is amorphous phase.

and important method to elaborate a study of fluids in a geological body, as they may indicate the characteristics of the initial nanoscale hydrothermal fluid. Additionally, the fluid and mineral ultrastructure revealed valuable insights into the resulting mineral. In this study, the columbite-group mineral was found to be crystallized before the hydrothermal fluid owing to the occurrence of nanoscale fluid within the columbite-group mineral. This result also suggested that the columbite-group mineral may not form during the hydrothermal fluid stage, which was consistent with the conclusion attained in Section 5.1.

5.3. The Amorphous Phase Role in the Nb-Ta Deposit Fluid. Fluid-mediated mineral dissolution and reprecipitation processes are the most prevalent mineral reaction mechanisms in the solid Earth and fundamental to internal dynamics including the release of carbon dioxide in subduction zones

[34, 35] and the precipitation of ore minerals from percolating fluids [36]. In addition, a comprehensive understanding of mineral reactions is essential for quantifying associated elemental transport. During these mineral reactions, element exchange occurs through aqueous solutions with mineral solubility in the coexisting fluid. Minerals in contact with a fluid phase dissolve, according to the classical theory of fluid-mediated dissolution and reprecipitation, until the chemical potential of the solid phase is equal to that of the dissolved material [37]. Several experimental examples demonstrate that dissolution-reprecipitation processes produce a silica-rich amorphous phase. However, the role of these amorphous phases as element transport agents and prenucleation products remains unclear. Several recent publications have demonstrated that amorphous and nanocrystalline precursor phases such as calcium carbonates [38] and oxides [39] from seawater play critical roles in natural precipitation mechanisms. A complete reaction mechanism for a mineral, including amorphous phase formation and elemental transport, has not been reported.

The aforementioned observations demonstrate exhaustively an unprecedented dissolution-reprecipitation process involving an amorphous phase in which Al and Si transport effectively between the reactant and product phases. The current findings also revealed various mineral reaction processes. First, solids react directly to form an amorphous material, thermodynamically decoupling mineral dissolution from aqueous solutions. The amorphous material effectively transports Al and Si, reflecting the composition of the parent mineral. Second, the amorphous material is present in a reaction-induced syn-metamorphic porosity, where it efficiently transferred elements required to form multioxide silicate complexes. Third, short-range atomic order effects in the amorphous material likely facilitate the direct formation of products via repolymerization of the amorphous phase. The combination of these mechanisms has crucial implications for all major processes occurring during mineral dissolution, element transport, and reprecipitation within solid materials.

6. Conclusions

- (1) This study investigated the ultrastructure of the columbite-group mineral within a peraluminous granite of the Zhaojingou Nb-Ta deposit in the

North China Craton. A FIB slice revealed that the mineral structure was relatively well-ordered by TEM and the columbite-tantalite mineral contained Nb, Ta, Mn, W, Fe, Sn, and Pb examined using EDS. The hydrothermal fluid was enriched in K, Al, Si, and O, which is different from the composition of the columbite-tantalite mineral. In addition, there is no reaction phenomenon occurring in the columbite-tantalite mineral boundary. These results in this deposit provided strong evidence for the formation of columbite-group minerals during the magmatic stage

- (2) HR-TEM images revealed the nanoscale fluid channel characteristics as well. The fluid channels contain Al and Si in composition and, and they are diffuse, cloudy, and patchy in appearance. These cloudy materials showed no diffraction contrast, as seen in SAED images. The amorphous material composition and the reaction-induced syn-metamorphic porosity provided an effective transfer of elements. The mineral dissolution and repolymerization from the amorphous phase exhibited dissolution-reprecipitation in the Nb-Ta deposit

Data Availability

Data can be requested from Rui Liu, whose e-mail is liurui35@mail.sysu.edu.cn.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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Supplementary Materials

Figure S1: a nanoscale hydrothermal channel existed between the columbite-group mineral and quartz. Figure S2: nanoscale hydrothermal channel within columbite-group mineral. (*Supplementary Materials*)

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