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Fluid Evolution and Ore Genesis of the Songjianghe Au Deposit in Eastern Jilin Province, NE China: Constraints from Fluid Inclusions and H-O-S-Pb Isotope Systematics

Qi Yu, Keyong Wang, Xuebing Zhang, Qingfei Sun, Wenqiang Bai, Chao Ma and Yongchun Xiao

Abstract: The medium-sized Songjianghe Au deposit is located in the southeastern part of the Jiapigou-Haigou gold belt (JHGB) in central eastern Jilin Province, NE China. The gold mineralization is primarily characterized by disseminated-style ores and hosted in the low-/medium-grade metamorphic rocks of the Seluohe Group. The ore bodies are governed by NNW-striking brittle-ductile structures and spatially correlated with silicic and sericitic alterations. Four alteration/mineralization stages have been distinguished: (I) Quartz-pyrrhotite-pyrite, (II) quartz-polymetallic sulfides, (III) quartz-pyrite, and (IV) quartz-calcite. The fluid inclusion (FI) assemblage in quartz from Stage I comprises C1-type, C2-type, C3-type, and VL-type FIs, with total homogenization temperatures (Th-total) of 292.8 to 405.6 °C and salinities of 2.8 to 9.3 wt% NaCl eqv. Quartz from Stage II (main ore stage) developed C2-, C3-, and VL-type FIs, with a Th-total of 278.5 to 338.9 °C and salinities of 2.8 to 8.1 wt% NaCl eqv. Stage III is characterized by coexisting C3- and VL-type FIs in quartz, with a Th-total of 215.9 to 307.3 °C and salinities of 2.4 to 7.2 wt% NaCl eqv. Only VL-type FIs are observed in Stage IV, with a Th-total of 189.5 to 240.4 °C and salinities of 3.7 to 5.7 wt% NaCl eqv. The Laser Raman spectroscopic results demonstrated minor CH4 in the C-type FIs from Stages I and II. The results suggest that ore fluids may have evolved from a medium-high temperature, low-salinity immiscible CO2-NaCl-H2O ± CH4 system to a low temperature, low-salinity homogeneous NaCl-H2O system. Fluid immiscibility caused by the rapid drop in pressure may have been the main trigger for gold-polymetallic sulfide precipitation. The Songjianghe Au deposit may have been formed under 352–448 °C and 850–1380 bar pressure, based on the isochore intersection for Stage II fluid inclusions. The H-O isotopic compositions (Stage I: δ18Ofluid = 5.6 to 5.8‰, δD = −96.2 to −95.7‰; Stage II: δ18Ofluid = 3.7 to 4.2‰, δD = −98.7 to −89.8‰; Stage III: δ18Ofluid = 1.2 to 1.4‰, δD = −103.5 to −101.2‰) indicate that the hydrothermal fluids are dominated by magmatic water in the early stages (Stages I and II) and mixed with meteoric water since Stage III. The pyrite S-Pb isotope data (δ34S: −2.91 to 3.40‰, 206Pb/204Pb: 16.3270 to 16.4874; 207Pb/204Pb: 15.2258 to 15.3489; 208Pb/204Pb: 36.6088 to 36.7174), combined with Pb isotopic compositions of the intrusive rocks and wall rocks (the Seluohe Group) in the ore district, indicate that the ore-forming materials at Songjianghe are predominantly from a magmatic source and may have been affected by the contamination of the Seluohe Group. In accordance with the features of ore geochemistry, ore-forming fluids and metals, and geodynamic setting, the Songjianghe Au deposit belongs to a mesothermal magmatic hydrothermal vein gold deposit, which formed in the intermittent stage of Paleo-Pacific plate subduction during the Late Jurassic.

Keywords: fluid evolution; ore genesis; H-O-S-Pb isotopes; Songjianghe Au deposit; NE China
1. Introduction

The Jiapigou-Haigou gold belt (JHGB), located in the tectonic transition between the northeastern North China Craton (NCC) margin and the eastern Central Asian Orogenic Belt (CAOB), is a key gold province with an explored gold reserve of >200 metric tonnes (t) in NE China [1–3]. The region has undergone prolonged tectonic evolution with the Paleo-Asian Ocean closure and the Paleo-Pacific subduction, forming a series of hydrothermal gold deposits related to CO₂-enriched fluids (e.g., Erdaogou, Xiaobeigou, Bajiazi, Xiaxitai, Sandaocha, and Haigou). Extensive studies have been undertaken to unravel the geological setting, metallogenic ages, and mineralization systematics of these gold deposits [4–11]. However, the origin of ore-forming fluids has long been a controversial topic: (1) Predominantly metamorphic water with later meteoric water infusion [6,12]; (2) a mixed source from magmatic water and meteoric water [13–15]; and (3) mantle-derived fluids [8,16,17]. Meanwhile, diverging insights also exist on the source of metals: (1) Extracted from metamorphic country rocks [18–20] or (2) a mixture of crust and mantle [17,21]. Consequently, in many cases, both the origin of ore fluids and metal source are indistinct or under dispute, which hinders the understanding of the ore-forming process and the mechanism of gold deposition. Considering the aforementioned distinctions, the genetic type of these gold deposits is a subject of continuous discussion and with conflicting conclusions: (1) A greenstone-type gold deposit [12,18,20], (2) a magmatic hydrothermal vein-type gold deposit [4,22,23], or (3) an orogenic gold deposit [6,21,24].

The Songjianghe Au deposit is sited in the southeastern JHGB, hosting a 9 metric tonne (t) gold reserve @ 3.02 g/t. Compared with other major gold deposits, the Songjianghe Au deposit has received less attention. Prior studies on the Songjianghe Au deposit primarily addressed ore geology [25,26], ore fluids [27–29], geochronology, and the geodynamic setting [30,31]. These studies proposed that the Songjianghe Au deposit was formed in the intermittent stage that followed the first subduction of the Paleo-Pacific plate during the Late Jurassic (157–156 Ma [31]) and gold precipitation was primarily caused by fluid immiscibility [27–29]. Nevertheless, the links between the evolution of ore-forming fluids and gold mineralization have not been convincing. Meanwhile, the sources of ore fluids and ore-forming materials are still obscure, resulting in unclear ore precipitation processes. In addition, the ores in the Songjianghe Au deposit are predominately of the disseminated style [25,26], in contrast to the quartz vein-type ores that describe the majority of gold deposits (e.g., Erdaogou, Xiaobeigou, Bajiazi, and Haigou) in the JHGB [17,20,32,33].

In this study, we give detailed descriptions of the geology and mineralogy, as well as a systematic study of the fluid inclusions and H-O stable isotopes, to provide distinct constraints on the property, origin, and evolution of the ore fluids and the mineralization mechanisms. Additionally, the S-Pb isotopic data are reported to reveal the source of the ore-forming materials. In accordance with the research results generated, together with the previous studies, the genetic type is classified, and a metallogenic model is established for the Songjianghe Au deposit. It is expected that these results can provide new insights into regional metallogeny in the Jiapigou-Haigou gold metallogenic belt.

2. Geological Setting

The Central Asian Orogenic Belt (CAOB), extending over 5500 km from west to east, evolved through complex tectonics of the Neoproterozoic-Middle Triassic Paleo-Asian Ocean closure, the Mesozoic Mongolia-Okhotsk Ocean closure, and the Paleo-Pacific subduction [34–42]. The CAOB separates the Siberia Craton in the north from the Tarim-North China Craton in the south (Figure 1a). NE China, the adjacent Russia Far East, and eastern Mongolia are composed primarily of micro-continental masses, including (from NW to SE) the Erguna, Xing’an, Songliao, and Jiamusi–Khanka blocks, and the Sikhote–Alin accretionary complex (Nadanhada terrane), bounded by the Xinlin-Xiguitu, Hegenshan-Heihe, Mudanjiang-Yanji, and Primoria faults, respectively [43,44] (Figure 1b).
Mongolia are composed primarily of micro-continental masses, including (from NW to SE) the Erguna, Xing'an, Songliao, and Jiamusi–Khanka blocks, and the Sikhote–Alin accretionary complex (Nadanhada terrane), bounded by the Xinlin-Xiguitu, Hegenshan-Heihe, Mudanjiang-Yanji, and Primoria faults, respectively [43,44] (Figure 1b).

Figure 1. (a) Tectonic map of the Central Asian Orogenic Belt, showing the location of NE China (modified after [45,46]). (b) Tectonic sub-divisions of NE China and its adjacent regions, showing the location of the Jiapigou-Haigou gold belt (modified after [47,48]). F1: Solonker-Xar Moron-Changchun zone; F2: Yanji Fault; F3: Mudanjiang Fault; F4: Hegenshan-Heihe Fault; F5: Xinlin-Xiguitu Fault; F6: Yilan-Yitong Fault; F7: Dunhua-Mishan Fault; F8: Primoria Fault. (c) Simplified regional geological map of the Jiapigou-Haigou gold belt, showing the locations of representative gold deposits (modified after [9,31]), including (number in the figure) 1. Songjianghe; 2. Haigou; 3. Liupiye; 4. Bajiazi; 5. Xiaixita; 6. Erdaogou; 7. Miaoeling; 8. Sidaocha; 9. Sidaocha; 10. Daxiangou; 11. Xiaobeigou; 12. Renaogou; 13. Caiqiangzi; 14. Banmiaozhi.

The JHGB is located in the tectonic transition between the northeastern NCC margin (locally named the Longgang terrane) and the eastern CAOB (Figure 1b,c). The formation, accretion, and solidification of the continental nucleus, as well as its cracking and subsequent cratonization, are primary tectonic evolution processes during Precambrian [2,49]. The Early Paleozoic witnessed the beginning of the closure of the Paleo-Asian Ocean, which had completely closed before the Middle Triassic [43,50]. The subduction of the
Paleo-Pacific plate beneath Eurasia is the most significant tectonic event in the JHGB during the Mesozoic [7,17].

The regional strata comprise Archean medium-/high-grade metamorphic rocks, Proterozoic low-/medium-grade rocks, Paleozoic low metamorphic/sedimentary rocks, Mesozoic volcanic/sedimentary rocks, and Cenozoic sediments (Figure 1c). The Archean metamorphic basement composed of the Longgang and Jiapigou Groups is exposed in the Longgang terrane on the southwestern side of the JHGB (Figure 1c). The Mesoarchean Longgang Group may have experienced medium-/high-grade metamorphism and primarily consists of felsic gneiss, granulite, plagioclase amphibolite, and supracrustal rocks [51]. The Neoarchean Jiapigou Group comprises characteristically biotite plagioclase gneiss, plagioclase amphibolite, hornblende plagioclase gneiss, and minor magnetite quartzite [21,52], which is the main host rock of many gold deposits (e.g., Bajiazi, Xiaxitai, and Sandaocoa). The Paleoproterozoic Ji’an Group composed of plagioclase amphibolite, biotite granulite, biotite schist, and marble was formed in the early rifting stage [53]. The Seluohe Group is discontinuously exposed along the Jinyinbie and Fuerhe faults (Figure 1c), which experienced low-/medium-grade metamorphism. As the host rock of the Songjianghe Au deposit, the Seluohe Group has long been considered a Meso-Neoproterozoic metamorphic suite [54,55], yet more recent studies have shown that the Seluohe Group may actually represent a complex of large tectonic slices that primarily comprise metamorphic strata and mylonitized granites of different ages and origins [31,56–58]. The Paleozoic strata consist primarily of the Qinglongcun Group and the Hulan Group, which are characterized by low metamorphic volcano-sedimentary rocks. The Late Triassic greenschist facies retrograde metamorphism of the Hulan Group represents a post-collisional extension setting after the closure of the Paleo-Asian Ocean [43,59]. The Mesozoic strata crop out in the northern and southeastern JHGB (Figure 1c), primarily comprising terrestrial sedimentary and intermediate-acid pyroclastic rocks.

The oldest granitoid, a TTG (tonalite-trondhjemite-granodiorite) suite, and a set of potassium-rich granitoids are exposed in the central and northern Longgang terrane, respectively (Figure 1c), which are characterized by sodic to potassic compositional evolution [60]. These two granitoid suites are likely the product of late Neoarchean to early Paleoproterozoic magmatic events under an active continental margin environment [52,61]. The Paleozoic intrusive rocks are exposed along NW-striking faults (Figure 1c), primarily comprising late Hercynian biotite granite and granodiorite, followed by monzogranite and quartz diorite, which were formed in the process of the Paleo-Asian Ocean closure [44,62]. The Mesozoic intrusive rocks are characteristically composed of large batholiths of granodiorite, monzogranite, and quartz diorite. These intrusive rocks were considered to be formed in the tectonic settings of post-collisional extension after the Paleo-Asian Ocean closure and/or the multiple subductions of the Paleo-Pacific plate [14,63,64].

Several fault zones developed in the region, including the NE-striking Dunhua-Mishan and Ji’an-Songjiang faults, as well as the NW-striking Fuerhe, Jinyinbie, and Jiapigou faults (Figure 1c). The Dunhua-Mishan fault is considered to be a branch of the Tancheng-Lujiang fault zone [10,65,66], whilst the NW-striking Jiapigou, Jinyinbie, and Fuerhe faults are the subsidiary structures of the Dunhua-Mishan fault [21,67], which experienced brittle deformation superimposed on brittle-ductile deformation [68]. These subsidiary faults, characterized by dextral shear [17,69], control the distribution of gold deposits in the study area [7,17] (Figure 1c). The ultra-crustal Ji’an-Songjiang fault locally controls the Mesozoic magma activities and the distribution of Early Jurassic and Cretaceous strata [1,70].

3. Deposit Geology

The Songjianghe Au deposit is located in the southeastern part of the Jiapigou-Haigou gold belt (JHGB). The only exposed stratigraphic unit at Songjianghe is the Seluohe Group (NE-dipping with 40–55° dip angle), which is also the ore host (Figure 2a). The Seluohe Group at Songjianghe was formed in the Late Neoarchean, primarily composed of chlorite-epidote-actinolite schist, biotite-plagioclase schist, amphibolite-plagioclase schist,
muscovite-biotite schist, granitic gneiss, meta-sandstone, and marble, and experienced stages of multistage metamorphism and deformation. The protoliths of the Seluohe Group may have primarily consisted of intermediate-basic and acidic volcanic and sedimentary rocks [31].

Figure 2. (a) Simplified geological map of the Songjianghe Au deposit, showing the Pb isotope sample locations in the pluton and wallrocks; (b) geologic profile along Exploration Line 0, showing No. II-1 orebody; (c) geological plan of mining level (584 m), showing the sampling locations for ores of the various mineralization stages.

There are three sets of major structures, which are (from old to young) EW-, NNW-, and NE-striking fractures (Figure 2a). The EW-striking (N-dipping with 40–80° dip angle) fractures are cut by the NNW- and NE-striking fractures, exposed on the north side of the ore district. The orebodies are controlled by the NNW-striking fractures (E or NE-dipping at 40 to 50°), which are characterized by the early-stage ductile shearing superimposed by the late-stage brittle-ductile fractures. The NE-striking fractures marked by SE-dipping with a 60–70° dip angle are characteristically transtensional, which crosscut the NNW-striking ore-controlling structures.

The local intrusive rocks consist primarily of plagiogranite and granodiorite (171 ± 5 Ma [71]), K-feldspar granite (169.6–173 Ma [25,30]), and biotite monzogranite (168.2 ± 2 Ma [72]) (Figure 2a). These Middle Jurassic igneous rocks were considered to have formed in the tectonic setting of the island arc or active continental margin under the subduction of the Paleo-Pacific plate [30,71,72].

A total of 10 orebodies have been delineated at Songjianghe, with a gold reserve of 9 t (@ 3.02 g/t). The largest orebody (II-1: 240 m long, 600 m deep, and 0.8–21.6 m thick) is lensoidal and developed along the NNW-striking structures (Figure 2b,c). The ores are primarily characterized by disseminated or veinlet types and minor quartz vein types. The ore textures primarily include anhedral granular, metasomatism, metasomatic relict, and minor euhedral-subhedral granular. Metallic minerals include characteristically pyrite, pyrrhotite, chalcopyrite, galena, sphalerite, and native gold. Gangue minerals primarily include quartz, calcite, mica, chlorite, and epidote.
Based on field observations, ore texture/structure, mineral assemblage, paragenetic sequence, and vein crosscutting relations, four alteration/mineralization stages were recognized (Figure 3). Stage I (quartz-pyrrhotite-pyrite) is characterized by medium-fine-grained milky white quartz with a euhedral-subhedral texture, and euhedral-subhedral fine-medium-grained pyrrhotite and pyrite (Figure 3a,e,f). Stage II (quartz-polymetallic sulfides) is the main Au ore stage, characterized by pyrite, chalcopyrite, galena, sphalerite, native gold, and quartz (Figure 3a–c,g–k). The quartz is usually smoke gray in color with subhedral-anhedral grains. The metal sulfides are mostly anhedral grains, and a few are euhedral-subhedral (Figure 3g–k). Pyrite, the predominant metal sulfide (~90 vol%), can be locally observed in coexistence with native gold, yet most native gold is characteristically hosted in the intergranular spaces and fissures of quartz (Figure 3j,k). Stage III is marked by the coexistence of quartz and pyrite (Figure 3c,l). It is visible that Stage II quartz-polymetallic sulfides were cut by Stage III quartz-pyrite veins (Figure 3c). Stage IV represents the end of hydrothermal activity, characterized by quartz-calcite veins (Figure 3d). The ore and gangue mineral paragenetic sequence is summarized in Figure 4. The Wall rock alteration zoning (from orebody to wallrock) includes sericite-silicic → weak potassic-biotite → epidote-chlorite → carbonate [31].

![Figure 3. Photographs and photomicrographs of the Songjianghe Au ores at various stages: (a) Stage I quartz-pyrrhotite-pyrite cut by Stage II quartz-polymetallic sulfides vein. (b) Stage II quartz-polymetallic sulfide veins. (c) Stage II quartz-polymetallic sulfides were cut by Stage III quartz-pyrite vein. (d) Stage IV quartz-calcite vein. (e,f) Stage I euhedral-subhedral pyrite and pyrrhotite in quartz. (g–i) Stage II pyrite-chalcopyrite-galena-sphalerite assemblage. (j,k) Stage II native gold in coexistence with pyrite and in intergranular spaces and fissures of quartz. (l) Stage III quartz and pyrite. Abbreviations: Cal—calcite; Ccp—chalcopyrite; Gl—native gold; Gn—galena; Po—pyrrhotite; Py—pyrite; Qz—quartz; Sp—sphalerite.](image_url)
The heating and cooling rates are 1–5 °C/min, which decreased to 0.1 °C/min when approaching phase transitions. Representative single FI compositions were analyzed on a Renishaw System-1000 Laser Raman Spectrometer (UK) with a 514 nm laser source. The spectral scanning range was set to between 1200 and 4000 cm\(^{-1}\), with a 60 s accumulation time for each scan.

4.2. Isotope Analysis

Nine quartz samples from representative ores (Stage I to III) were selected from the II-1 orebody of the 584 m mining level (Figure 2c) for hydrogen-oxygen isotope analyses to constrain the ore-fluid origin. The ore samples were examined and identified under a microscope to make sure they were representative of various hydrothermal stages. The quartz samples were crushed to 40–60 mesh, and then handpicked under a binocular microscope to obtain >98% purity. Hydrogen-oxygen isotope composition analyses were completed in the laboratory of the Beijing Research Institute of Uranium Geology and Beijing GeoAnalysis Co. Ltd. (Beijing, China). The quartz was reacted with BrF\(_5\) in a vacuum at a high temperature to extract oxygen, which was then converted to CO\(_2\) gas on a burning Pt-coated carbon rod [74]. The oxygen isotope composition of the obtained CO\(_2\) gas was analyzed on a MAT-253EM mass spectrometer. The same samples were heated at 150 °C for 3 h in a vacuum, and then heated to >500 °C in an induction furnace to release CO\(_2\) gas on a MAT-253EM mass spectrometer. The same samples were heated at 150 °C for 3 h in a vacuum, and then heated to >500 °C in an induction furnace to

<table>
<thead>
<tr>
<th>Stages</th>
<th>Hydrothermal mineralization period</th>
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<tbody>
<tr>
<td>Minerals</td>
<td>Stage I</td>
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<tr>
<td>Quartz</td>
<td>[ ]</td>
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<tr>
<td>pyrrhotite</td>
<td>[ ]</td>
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<tr>
<td>Pyrite</td>
<td>[ ]</td>
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<tr>
<td>Native gold</td>
<td>[ ]</td>
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<tr>
<td>Chalcopyrite</td>
<td>[ ]</td>
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<tr>
<td>Galena</td>
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<tr>
<td>Sphalerite</td>
<td>[ ]</td>
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<tr>
<td>Sericite</td>
<td>[ ]</td>
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<tr>
<td>Epidote</td>
<td>[ ]</td>
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<td>Chlorite</td>
<td>[ ]</td>
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<tr>
<td>Calcite</td>
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</table>

Figure 4. Mineral paragenetic sequence of the Songjianghe Au deposit.
release the inclusion water, which was converted to H₂ by reacting with zinc powder at 800 °C. Finally, the hydrogen isotope composition was analyzed on a MAT-253EM mass spectrometer. The oxygen and hydrogen isotope ratios are presented in standard δ notation (‰) relative to the Standard Mean Ocean Water (SMOW). The analysis accuracy is ±0.2‰ for oxygen isotope and ±2‰ for hydrogen isotope.

Twenty-two pyrite samples (Stage I and II) were selected from the II-1 orebody of the 584 m mining level (Figure 2c) for sulfur isotope analyses, which were conducted at Beijing GeoAnalysis Co., Ltd. Sulfur isotope compositions were determined by conventional combustion and LA-MC-ICP-MS in situ S isotope analysis. SO₂ was liberated for S isotope analysis using the conventional extraction technique of [75], and then measured with a Delta V Plus mass spectrometer. Results are reported as δ³⁴S relative to Vienna Canyon Diablo Troilite (V-CDT) with a precision of ±0.2‰. The in situ S isotope was analyzed using an NWR 193 laser ablation system connected to Neptune Plus MC-ICP-MS. The single-spot ablation mode was used with a large spot size (44 µm) and low pulse frequency (2 Hz). Approximately 100 laser pulses were completed in a single analysis [76]. Details of the S isotope analytical methods follow those described by [77,78].

Five pyrite samples (Stage II) were selected from the II-1 orebody of the 584 m mining level (Figure 2c) for lead isotope analyses. In addition, lead isotope analyses of the wallrock, K-feldspar granite, and monzogranite (Figure 2a) were also carried out. All these analyses were conducted at Beijing GeoAnalysis Co., Ltd. Furthermore, 10–50 mg powder samples were washed with acetone and distilled water to remove surface contamination before air drying at 60 °C. The cleaned samples were dissolved in HF + HNO₃ at 150 °C for 7 days. The conventional cation exchange technique was used to purify the lead, and then placed in 0.5 mol/L HBr-HCl solution for Pb separation. The lead isotope analyses were conducted on an ISOPROBE-T thermal ionization mass spectrometer (TIMS) with an analysis accuracy of better than 0.005%. In addition, Pb isotope ratios were calibrated by the NBS SRM 981 standard. For details on lead isotope analysis methods, refer to [79].

5. Results
5.1. Fluid Inclusion
5.1.1. Fluid Inclusion Petrography

On the basis of the criteria of [73], primary FIs in isolation or random clusters were selected for the analyses, whereas secondary FIs along micro-fractures or penetrating the crystal boundary were not selected [80]. Based on the FI phase composition at room temperature (25 °C) and the phase transition characteristics during heating or freezing, two types of primary FIs are identified: CO₂ (C-type) and liquid-rich two-phase (VL-type) FIs. C-type FIs usually have one to two components, i.e., CO₂ ± liquid H₂O. Depending on the CO₂/H₂O ratio, C-type FIs can be divided into three sub-types: CO₂-pure (C1-type, entirely CO₂), CO₂-rich (C2-type, CO₂/H₂O ratio > 1), and CO₂-bearing (C3-type, CO₂/H₂O ratio < 1).

C1-type FIs are usually composed of two phases (liquid CO₂ + vapor CO₂) or a single liquid CO₂ phase at room temperature (25 °C), and the latter would appear as vapor CO₂ when cooled to <10 °C. The volume range of vapor CO₂ is 5% to 20% at room temperature (25 °C). These FIs are usually rounded/elliptical with a size of 8–12 µm and primarily distributed in groups (Figure 5(a-4); Table 1).

C2-type FIs usually contain three phases (liquid CO₂ + vapor CO₂ + liquid H₂O). The volume range of the CO₂ phase (liquid CO₂ + vapor CO₂) is over 50% (primarily 70–90%, i.e., CO₂/H₂O ratio > 1), with the vapor CO₂ phase accounting for 85 to 90% (a few 30–35%). These FIs are commonly elliptical with a size of 6–15 µm. Most of these FIs are isolated, while a few are distributed in groups (Figure 5(a-5,b-1,b-2,b-4); Table 1).

C3-type FIs are composed of three phases (liquid CO₂ + vapor CO₂ + liquid H₂O). The volume range of the CO₂ phase (liquid CO₂ + vapor CO₂) is < 50% (primarily 35–40%, i.e., CO₂/H₂O ratio < 1), with the vapor CO₂ phase accounting for 65 to 90% (a few with 35–45%). These FIs are commonly elliptical or irregular-shaped with a size of 5–20 µm.
Most of these FIs are isolated, and a few are clustered (Figure 5(a-1–a-3,a-5,b-3,b-4,c-1,c-2); Table 1).

VL-type FIs have two phases (vapor H$_2$O + liquid H$_2$O) at room temperature (25 °C), with vapor H$_2$O accounting for <50 vol.% (primarily 15–30 vol.%, minor 5–10 vol.%). These FIs are commonly elliptical, irregular-, or occasionally negative-crystal-shaped, with a size of 5–22 µm, and most of them occur in clusters, with a few isolated FIs (Figure 5(a-1,a-5,b-1,b-4,c-1,c-3,d-1,d-2); Table 1).

**Table 1.** Microthermometric data of fluid inclusion assemblages from different mineralization stages.

<table>
<thead>
<tr>
<th>Mineralization Stages</th>
<th>Host Minerals</th>
<th>Types (Number)</th>
<th>Size (µm)</th>
<th>Tm-CO$_2$ (°C)</th>
<th>Tm-Cla (°C)</th>
<th>Tm-Clathrate (°C)</th>
<th>Tm-Ice (°C)</th>
<th>Tm-Tot (°C)</th>
<th>Salinity (wt.% NaCl eqv)</th>
</tr>
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<tr>
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<td>quartz</td>
<td>C1-type (10)</td>
<td>8–12</td>
<td>−60.8 to −60.0</td>
<td>17.6 to 30.0</td>
<td>−6.1 to −3.9</td>
<td>305.0 to 386.4</td>
<td>2.8 to 6.6</td>
<td>1.0</td>
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<td></td>
<td></td>
<td>C2-type (22)</td>
<td>6–15</td>
<td>−61.0 to −56.9</td>
<td>17.5 to 30.2</td>
<td>−6.0 to −2.7</td>
<td>327.6 to 405.6</td>
<td>3.0 to 5.9</td>
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<td>C3-type (27)</td>
<td>6–20</td>
<td>−62.2 to −59.5</td>
<td>16.2 to 30.9</td>
<td>301.5 to 337.6</td>
<td>2.4 to 4.7</td>
<td>3.4 to 4.7</td>
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<td></td>
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<td>VL-type (31)</td>
<td>5–20</td>
<td>−60.1 to −57.5</td>
<td>25.4 to 30.5</td>
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<td>292.8 to 368.4</td>
<td>6.3 to 9.3</td>
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<td>Stage II</td>
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<td>C2-type (10)</td>
<td>8–12</td>
<td>−60.1 to −57.5</td>
<td>25.4 to 30.5</td>
<td>−5.2 to −3.6</td>
<td>301.5 to 337.6</td>
<td>2.8 to 5.1</td>
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<td>C3-type (21)</td>
<td>5–15</td>
<td>−60.0 to −56.9</td>
<td>21.8 to 30.7</td>
<td>301.9 to 338.6</td>
<td>2.8 to 5.1</td>
<td>2.8 to 5.1</td>
<td>1.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>VL-type (23)</td>
<td>6–22</td>
<td>7.4 to 8.6</td>
<td>30.5 to 30.7</td>
<td>301.9 to 338.6</td>
<td>2.8 to 5.1</td>
<td>3.4 to 4.7</td>
<td>1.0</td>
</tr>
<tr>
<td>Stage III</td>
<td>quartz</td>
<td>C3-type (20)</td>
<td>5–15</td>
<td>−57.7 to −56.7</td>
<td>11.8 to 28.0</td>
<td>−4.5 to −2.8</td>
<td>215.9 to 297.3</td>
<td>4.6 to 7.2</td>
<td>1.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>VL-type (24)</td>
<td>5–15</td>
<td>7.9 to 8.8</td>
<td>28.0 to 30.7</td>
<td>225.4 to 307.3</td>
<td>3.2 to 4.1</td>
<td>3.2 to 4.1</td>
<td>1.0</td>
</tr>
<tr>
<td>Stage IV</td>
<td>quartz</td>
<td>VL-type (19)</td>
<td>5–15</td>
<td>−3.5 to −2.2</td>
<td>240.0 to 240.4</td>
<td>189.5 to 240.4</td>
<td>3.7 to 5.7</td>
<td>3.7 to 5.7</td>
<td>1.0</td>
</tr>
</tbody>
</table>

Note: Tm-CO$_2$ = melting temperatures of solid CO$_2$; Tm-cla = melting temperatures of CO$_2$-H$_2$O clathrate; Th-CO$_2$ = homogenization temperatures of the CO$_2$ phase; Tm-ice = temperatures of ice melting; Th-total = total homogenization temperatures.

**Figure 5.** Photomicrographs of representative primary fluid inclusion assemblages from the Songjianghe Au deposit. (a-1–a-5) C1-, C2-, C3-, and VL-type FIA in Stage I quartz; (b-1–b-4) C2-, C3-, and VL-type FIA in Stage II quartz; (c-1–c-3) C3- and VL-type FIA in Stage III quartz; (d-1–d-2) VL-type FIs in Stage IV quartz. Abbreviations: L = liquid, V = vapor.

5.1.2. Fluid Inclusion Microthermometry

The FI microthermometric data are presented in Table 1 and illustrated in Figure 6. The FI salinities (expressed as wt.% NaCl eqv) were calculated according to the ice-melting...
temperature (Tm-ice) of VL-type FIs [81]. For C2- and C3-type FIs, the salinities were calculated based on the CO₂-H₂O clathrate melting temperatures (Tm-cla) [73].

Figure 6. Histogram of fluid inclusion total homogenization temperature (Th-total) and salinity of the different hydrothermal stages at Songjianghe.

Stage I: C- (C1-, C2-, and C3-) and VL-type FIs were identified (Figure 5(a-1–a-5)). C-type FIs have similar solid CO₂ (Tm-CO₂) melting temperatures (−62.2 to 56.9 °C) and homogenization temperatures to the CO₂ phase (Th-CO₂ = 16.2 to 30.9 °C). The CO₂ phase of C1- and C2-type FIs homogenized to the liquid CO₂ phase, while the CO₂ phase of C3-type FIs homogenized to the liquid or vapor CO₂ phase. The C2- and C3-type FIs yielded similar Tm-cla (6.5 to 8.6 °C), with corresponding salinities of 2.8 to 6.6 wt.% NaCl eqv (Figure 6). The total homogenization temperatures (Th-total) of C2- and C3-type type FIs are 305.0 to 405.6 °C (primarily 330–350 °C) during heating (Figure 6). It should be noted that the C2-type FIs are totally homogenized to the liquid CO₂ phase, while the C3-type FIs are totally homogenized to the liquid H₂O phase. VL-type FIs homogenized to the liquid H₂O phase with a Th-total of 292.8 to 368.4 °C (primarily 330 to 350 °C) (Figure 6).
Tm-ice of VL-type FIs is −6.1 to −3.9 °C, with calculated salinities of 6.3 to 9.3 wt.% NaCl eqv (Figure 6).

Stage II: FIs are predominantly C2-, C3-, and VL-type (Figure 5(b-1–b-4)). C2- and C3-type FIs have similar Tm-CO2 (−60.1 to −56.9 °C) and Th-CO2 (21.8 to 30.7 °C). The CO2 phase of C2-type FIs homogenized to the liquid CO2 phase, while the CO2 phase of C3-type FIs homogenized to the liquid or vapor CO2 phase. Tm-cla of C2- and C3-type FIs are 7.4 to 8.6, with corresponding salinities of 2.8 to 5.1 wt.% NaCl eqv (Figure 6). C2-type FIs are totally homogenized to the liquid CO2 phase at 301.5 to 337.6 °C, while C3-type FIs are totally homogenized to the liquid H2O phase at 301.9 to 338.6 °C. The Th-total of C2- and C3-type FIs cluster around 310 to 320 °C (Figure 6). VL-type FIs homogenized to the liquid H2O phase at 278.5 to 338.9 °C (primarily 310 to 320 °C) (Figure 6). Tm-ice of VL-type FIs is −5.2 to −3.6 °C, with corresponding salinities of 5.8 to 8.1 wt.% NaCl eqv (Figure 6).

Stage III: C3- and VL-type FIs were identified (Figure 5(c-1–c-3)). C3-type FIs have Tm-CO2 of −59.8 to −56.7 °C and Th-CO2 of 11.8 to 28.0 °C. The CO2 phase of C3-type FIs homogenized to the liquid or vapor CO2 phase. Tm-cla values of C3-type FIs are 7.9 to 8.8 °C, with corresponding salinities of 2.4 to 4.1 wt.% NaCl eqv (Figure 6). C3-type FIs are totally homogenized to the liquid H2O phase at 225.4 to 307.3 °C (primarily 250 to 260 °C) (Figure 6). VL-type FIs homogenized to liquid H2O phase at 215.9 to 297.3 °C (primarily 250 to 260 °C). These FIs have Tm-ice of −4.5 to −2.8 °C, with calculated salinities of 4.6 to 7.2 wt.% NaCl eqv (Figure 6).

Stage IV: Only VL-type FIs were identified (Figure 5(d-1,d-2)). The Tm-ice of these FIs is −3.5 to −2.2 °C, with corresponding salinities of 3.7 to 5.7 wt.% NaCl eqv (Figure 6). They homogenized into the liquid H2O phase at 189.5 to 240.4 °C (primarily 210 to 230 °C) (Figure 6).

5.1.3. Laser Raman Spectroscopy

Representative primary fluid inclusions were selected for Laser Raman analysis. Stage I to II C-type FIs are generally CO2-rich with minor CH4 (Figure 7a–d), whereas only CO2 was detected in Stage III C3-type FIs (Figure 7e). Stage IV VL-type FIs contain only H2O (Figure 7f).

Figure 7. Laser Raman spectra for representative fluid inclusions (FIs) from the different hydrothermal stages at Songjianghe: (a,b) C1-type and C2-type FIs in Stage I quartz. (c,d) C3-type and C2-type FIs in Stage II quartz. (e) C3-type FIs in Stage III quartz. (f) VL-type FIs in Stage IV quartz.
5.2. Hydrogen-Oxygen Isotope Compositions

The H-O isotope data are presented in Table 2 and illustrated in Figure 8. Stage I $\delta^{18}O_{\text{quartz}}$ values are $-96.2$ to $-95.7\%$ and $11.1$ to $11.3\%$, respectively. Stage II $\delta^{18}O_{\text{quartz}}$ values are $-98.7$ to $-89.8\%$ and $10.1$ to $10.6\%$, respectively, whilst Stage III $\delta^{18}O_{\text{quartz}}$ values are $-103.5$ to $-101.2\%$ and $9.8$ to $9.9\%$, respectively. Based on the host mineral-fluid fractionation equations [82] and the FI Th-total of Stages I to III, the calculated $\delta^{18}O_{\text{fluid}}$ values are $5.6$ to $5.8\%$ (Stage I), $3.7$ to $4.2\%$ (Stage II), and $1.2$ to $1.4\%$ (Stage III).

Table 2. Hydrogen and oxygen isotope data of fluid inclusions in quartz from different mineralization stages.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Mineralization Stages</th>
<th>Mineral</th>
<th>$\delta^{18}O_{\text{quartz}}$ (%)</th>
<th>$\delta^{18}O_{\text{fluid}}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SJH-HO-1</td>
<td>Stage I</td>
<td>quartz</td>
<td>$-95.7$</td>
<td>11.1</td>
</tr>
<tr>
<td>SJH-HO-2</td>
<td></td>
<td>quartz</td>
<td>$-96.2$</td>
<td>11.3</td>
</tr>
<tr>
<td>SJH-HO-3</td>
<td>Stage II</td>
<td>quartz</td>
<td>$-98.7$</td>
<td>10.6</td>
</tr>
<tr>
<td>SJH-HO-4</td>
<td></td>
<td>quartz</td>
<td>$-95.5$</td>
<td>10.5</td>
</tr>
<tr>
<td>SJH-HO-5</td>
<td></td>
<td>quartz</td>
<td>$-93.9$</td>
<td>10.1</td>
</tr>
<tr>
<td>SJH-HO-6</td>
<td></td>
<td>quartz</td>
<td>$-89.8$</td>
<td>10.6</td>
</tr>
<tr>
<td>SJH-HO-7</td>
<td>Stage III</td>
<td>quartz</td>
<td>$-101.2$</td>
<td>9.8</td>
</tr>
<tr>
<td>SJH-HO-8</td>
<td></td>
<td>quartz</td>
<td>$-103.5$</td>
<td>9.9</td>
</tr>
<tr>
<td>SJH-HO-9</td>
<td></td>
<td>quartz</td>
<td>$-102.1$</td>
<td>9.9</td>
</tr>
</tbody>
</table>

Note: $\delta^{18}O_{\text{fluid}} = \delta^{18}O_{\text{SMOW}} - 3.38 \times 10^6/(T + 273)^2 + 3.40$ [82], $T =$ average total homogenization temperatures (Th-total) of fluid inclusions.

Figure 8. $\delta^{18}O_{\text{fluid}}$ diagram for the different-stage hydrothermal fluids at Songjianghe (base map after [83]). The field of mantle juvenile water is from [84]. The field of Au, Cu (Mo) series of magmatic water is from [85]. The volcanic vapor and felsic magma fields are from [86]. The published data of $\delta^{18}O_{\text{SMOW}}$ and $\delta^{18}O_{\text{fluid}}$ compositions for the Erdaogou, Xiaobeigou, Bajiazi, and Haigou gold deposits in the JHGB are from [6,8,10,17,20,33,87].

5.3. Sulfur-Lead Isotope Compositions

Sulfur isotope compositions of Stages I and II (main-ore stage) pyrites are presented in Table 3 and illustrated in Figure 9. Seven pyrite samples from Stage I show $\delta^{34}S$ values
varying from $-2.91$ to $0.42\%$ (average $= -1.04\%$) and fifteen pyrite samples from Stage II have $\delta^{34}S$ values varying from $-0.96$ to $3.40\%$ (average $= 1.19\%$). The samples as a whole yielded a relatively narrow $\delta^{34}S$ range ($-2.91$ to $3.40\%$, mean $0.48\%$) with a cluster around $-1$ to $1\%$ (Figure 9a).

### Table 3. Sulfur isotope compositions of the Songjianghe Au deposit.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Mineralization Stages</th>
<th>Mineral</th>
<th>$\delta^{34}S_{\text{V-CDT}}$ (‰)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SJH-S-1</td>
<td>Stage I</td>
<td>pyrite</td>
<td>-2.91</td>
</tr>
<tr>
<td>SJH-S-2</td>
<td>Stage I</td>
<td>pyrite</td>
<td>-1.87</td>
</tr>
<tr>
<td>SJH-S-3</td>
<td>Stage I</td>
<td>pyrite</td>
<td>-1.33</td>
</tr>
<tr>
<td>SJH-S-4</td>
<td>Stage I</td>
<td>pyrite</td>
<td>-1.65</td>
</tr>
<tr>
<td>SJH-S-5</td>
<td>Stage I</td>
<td>pyrite</td>
<td>0.42</td>
</tr>
<tr>
<td>SJH-S-6</td>
<td>Stage I</td>
<td>pyrite</td>
<td>-0.15</td>
</tr>
<tr>
<td>SJH-S-7</td>
<td>Stage I</td>
<td>pyrite</td>
<td>0.22</td>
</tr>
<tr>
<td>SJH-S-8</td>
<td>Stage II</td>
<td>pyrite</td>
<td>1.39</td>
</tr>
<tr>
<td>SJH-S-9</td>
<td>Stage II</td>
<td>pyrite</td>
<td>-0.84</td>
</tr>
<tr>
<td>SJH-S-10</td>
<td>Stage II</td>
<td>pyrite</td>
<td>0.76</td>
</tr>
<tr>
<td>SJH-S-11</td>
<td>Stage II</td>
<td>pyrite</td>
<td>-0.96</td>
</tr>
<tr>
<td>SJH-S-12</td>
<td>Stage II</td>
<td>pyrite</td>
<td>3.40</td>
</tr>
<tr>
<td>SJH-S-13</td>
<td>Stage II</td>
<td>pyrite</td>
<td>-0.33</td>
</tr>
<tr>
<td>SJH-S-14</td>
<td>Stage II</td>
<td>pyrite</td>
<td>3.08</td>
</tr>
<tr>
<td>SJH-S-15</td>
<td>Stage II</td>
<td>pyrite</td>
<td>1.55</td>
</tr>
<tr>
<td>SJH-S-16</td>
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<td>0.92</td>
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<td>SJH-S-17</td>
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<td>2.42</td>
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<td>SJH-S-18</td>
<td>Stage II</td>
<td>pyrite</td>
<td>2.38</td>
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<tr>
<td>SJH-S-19</td>
<td>Stage II</td>
<td>pyrite</td>
<td>2.76</td>
</tr>
<tr>
<td>SJH-S-20</td>
<td>Stage II</td>
<td>pyrite</td>
<td>1.04</td>
</tr>
<tr>
<td>SJH-S-21</td>
<td>Stage II</td>
<td>pyrite</td>
<td>-0.60</td>
</tr>
<tr>
<td>SJH-S-22</td>
<td>Stage II</td>
<td>pyrite</td>
<td>0.90</td>
</tr>
</tbody>
</table>

**Figure 9.** (a) $\delta^{34}S$ histogram from main-ore Stage II pyrite for the Songjianghe Au deposit; (b) $\delta^{34}S$ ranges for major Au deposits in the Jiapigou-Haigou gold belt (modified after [2,8,10,17,20,88–90]).

Lead isotope compositions of stage II pyrite, Middle Jurassic monzogranite and K-feldspar granite, and the Seluohe Group biotite-plagioclase schist are presented in Table 4 and illustrated in Figure 10. The monzogranite ($^{206}\text{Pb}/^{204}\text{Pb} = 17.1720$ to $17.3080$, $^{207}\text{Pb}/^{204}\text{Pb} = 15.3720$ to $15.4060$, $^{208}\text{Pb}/^{204}\text{Pb} = 37.2280$ to $37.3910$) and K-feldspar granite ($^{206}\text{Pb}/^{204}\text{Pb} = 17.4630$ to $17.5480$, $^{207}\text{Pb}/^{204}\text{Pb} = 15.4080$ to $15.4440$, $^{208}\text{Pb}/^{204}\text{Pb} = 37.5280$...
to 37.6810) have relatively high Pb isotopic ratios, whilst the biotite-plagioclase schist (\( {206}^{\text{Pb}}/{204}^{\text{Pb}} = 16.4550 \) to 16.8380, \( {207}^{\text{Pb}}/{204}^{\text{Pb}} = 15.2050 \) to 15.2920, \( {208}^{\text{Pb}}/{204}^{\text{Pb}} = 36.5910 \) to 36.9210) and Stage II pyrite (\( {206}^{\text{Pb}}/{204}^{\text{Pb}} = 16.3270 \) to 16.4874, \( {207}^{\text{Pb}}/{204}^{\text{Pb}} = 15.2258 \) to 15.3489, \( {208}^{\text{Pb}}/{204}^{\text{Pb}} = 36.6088 \) to 36.7174) display low Pb isotopic ratios.

**Table 4.** Lead isotope compositions of ores and wallrocks from the Songjianghe Au deposit.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Sample Description</th>
<th>Test Object</th>
<th>( {208}^{\text{Pb}}/{204}^{\text{Pb}} )</th>
<th>( {207}^{\text{Pb}}/{204}^{\text{Pb}} )</th>
<th>( {206}^{\text{Pb}}/{204}^{\text{Pb}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>SJH-Pb-1</td>
<td>monzogranite whole rock</td>
<td></td>
<td>37.2820</td>
<td>15.3930</td>
<td>17.1720</td>
</tr>
<tr>
<td>SJH-Pb-2</td>
<td>monzogranite whole rock</td>
<td></td>
<td>37.2280</td>
<td>15.3720</td>
<td>17.1760</td>
</tr>
<tr>
<td>SJH-Pb-3</td>
<td>monzogranite whole rock</td>
<td></td>
<td>37.3910</td>
<td>15.4060</td>
<td>17.3080</td>
</tr>
<tr>
<td>SJH-Pb-4</td>
<td>K-feldspar granite whole rock</td>
<td></td>
<td>37.5280</td>
<td>15.4080</td>
<td>17.4630</td>
</tr>
<tr>
<td>SJH-Pb-5</td>
<td>K-feldspar granite whole rock</td>
<td></td>
<td>37.5980</td>
<td>15.4250</td>
<td>17.4970</td>
</tr>
<tr>
<td>SJH-Pb-6</td>
<td>K-feldspar granite whole rock</td>
<td></td>
<td>37.6810</td>
<td>15.4440</td>
<td>17.5480</td>
</tr>
<tr>
<td>SJH-Pb-7</td>
<td>biotite-plagioclase schist whole rock</td>
<td></td>
<td>36.9210</td>
<td>15.2920</td>
<td>16.8380</td>
</tr>
<tr>
<td>SJH-Pb-8</td>
<td>biotite-plagioclase schist whole rock</td>
<td></td>
<td>36.5910</td>
<td>15.2140</td>
<td>16.4550</td>
</tr>
<tr>
<td>SJH-Pb-9</td>
<td>biotite-plagioclase schist whole rock</td>
<td></td>
<td>36.6000</td>
<td>15.2050</td>
<td>16.4980</td>
</tr>
<tr>
<td>SJH-Pb-10</td>
<td>Stage II pyrite</td>
<td></td>
<td>36.6265</td>
<td>15.2258</td>
<td>16.4874</td>
</tr>
<tr>
<td>SJH-Pb-11</td>
<td>Stage II pyrite</td>
<td></td>
<td>36.6088</td>
<td>15.2655</td>
<td>16.4205</td>
</tr>
<tr>
<td>SJH-Pb-12</td>
<td>Stage II pyrite</td>
<td></td>
<td>36.6153</td>
<td>15.2250</td>
<td>16.3270</td>
</tr>
<tr>
<td>SJH-Pb-13</td>
<td>Stage II pyrite</td>
<td></td>
<td>36.6878</td>
<td>15.3489</td>
<td>16.4388</td>
</tr>
<tr>
<td>SJH-Pb-14</td>
<td>Stage II pyrite</td>
<td></td>
<td>36.7174</td>
<td>15.3300</td>
<td>16.4230</td>
</tr>
</tbody>
</table>

**Figure 10.** (a,b) Lead isotope compositions of the monzogranite, K-feldspar granite, Seluohe Group biotite-plagioclase schist, and the main ore-stage pyrite at Songjianghe. The average growth lines for the mantle, orogen, upper crust, and lower crust are from [91]. (c,d) The published data of Lead isotope compositions for the Erdaogou, Xiaxitai, Sandaocha, Sidaocha, Bajiazi, and Haigou gold deposits in the JHGB are from [20,89,92,93].
6. Discussion

6.1. Property and Origin of the Ore-Forming Fluids

Stage I: There are four types of primary FIs (C1-, C2-, C3-, and VL-type) coexisting in the quartz crystals (Figure 5(a1–a5)). These FIs have similar total homogenization temperatures (Th-total, 292.8 to 405.6 °C; Figure 6a) but diversified salinity (2.8 to 9.3 wt% NaCl eqv; Figure 6b) and different homogenization paths (in Section 5.1.2). The C- (C1-, C2-, and C3-) type FIs exhibit various CO2/H2O ratios (in Section 5.1.1). Moreover, the C-type FIs have similar homogenization temperatures of the CO2 phase (Th-CO2 = 16.2 to 30.9 °C). Thus, there has been considerable evidence that the ore fluids from Stage I were immiscible when the FIs were captured [94–96]. The melting temperatures of solid CO2 (Tm-CO2) of C-type FIs (−62.2 to 56.9 °C) are lower than the CO2 triple point (−56.6 °C), implying the presence of a certain amount of CH4 and/or N2. The Laser Raman spectroscopic results demonstrated minor CH4 in the C-type FIs, which is in accordance with the above inference (Figure 7a,b). The Stage I δDv-SMOW and δ18Ofluid values have similar distribution ranges and plot into the magmatic domain (Figure 8). Additionally, the oxygen isotopic data (δ18Ofluid = 5.6 to 5.8‰) are also in accordance with those of magmatic water (5.5‰ to 9.5‰), as proposed by [97]. The hydrogen-oxygen isotopic composition from the early stage at Songjianghe is comparable to other gold deposits (e.g., Erdaogou, Xiaobeigou, Bajiazi, and Haigou) in the JHGB (Figure 8), indicating a potentially similar system, which was sourced from the mixing of magmatic water and meteoric water. In combination, Stage I ore fluids belong to a medium-low-temperature, low-salinity homogeneous CO2 system, which was sourced from the mixing of magmatic water and meteoric water. Meanwhile, in contrast to the quartz vein-type ore, the ore fluids of the altered type are characteristically dominated by magmatic fluids and less admixed meteoric water [99], as supported by the hydrogen-oxygen isotope data of quartz vein-type gold deposits (e.g., Erdaogou, Xiaobeigou, Bajiazi, and Haigou) in the JHGB (Figure 8). Therefore, the slight variation in δ18Ofluid values is interpreted as an exchange of material between the ore fluids and wallrocks, rather than being caused by the input of meteoric water. In combination, Stage I ore fluids belong to a moderate-temperature, low-salinity immiscible CO2-NaCl-H2O ± CH4 system and are primarily derived from magmatic water.

Stage II: The coexisting C2-, C3-, and VL-type FIs have similar Th-totals (278.5 to 338.9 °C; Figure 6c), yet they have different salinity values (2.8 to 8.1 wt% NaCl eqv; Figure 6d) and homogenization paths (in Section 5.1.2). The C2- and C3-type FIs are characterized by various CO2/H2O ratios (in Section 5.1.1). In addition, the Th-CO2 of C2- and C3-types have similar distribution ranges (21.8 to 30.7 °C). These features suggest that the ore fluids from Stage II are immiscible [94]. Tm-CO2 of C2-type (C2- and C3-type) FIs (−60.1 to −56.9 °C) and Laser Raman spectroscopic results (Figure 7c,d) demonstrated minor CH4 in the fluid inclusions. Although δ18Ofluid values have slightly drifted toward meteoric water relative to Stage I, δDv-SMOW values remain almost unchanged (Figure 8). In general, the fluid–rock interaction is a common process at temperatures of 280–380 °C in hydrothermal gold deposits [98]. Meanwhile, in contrast to the quartz vein-type ore, the ore fluids of the altered type are characteristically dominated by magmatic fluids and less admixed meteoric water [99], as supported by the hydrogen-oxygen isotope data of quartz vein-type gold deposits (e.g., Erdaogou, Xiaobeigou, Bajiazi, and Haigou) in the JHGB (Figure 8). Therefore, the slight variation in δ18Ofluid values is interpreted as an exchange of material between the ore fluids and wallrocks, rather than being caused by the input of meteoric water. In combination, Stage II ore fluids belong to a moderate-temperature, low-salinity immiscible CO2-NaCl-H2O ± CH4 system derived from magmatic water.

Stage III: The identified C3- and VL-type FIs commonly occur in groups in the same quartz grain and have a similar Th-total (215.9 to 307.3 °C; Figure 6e), suggesting that they were captured almost simultaneously. These FIs do not constitute an immiscible FIA and cannot represent a fluid-immiscible assemblage. This is because C3-type FIs have the same CO2/H2O ratio (in Section 5.1.1), and the vapor/liquid ratios of VL-type FIs are similar. The δDv-SMOW and δ18Ofluid values fall between the fields of meteoric water and magmatic water (Figure 8), indicating meteoric water incursion. The volatiles (CO2 and CH4) of FIs from Stage I to III decreased apparently, which may have been attributed to decompression, crystallization-induced degassing, and/or meteoric water incursion [100]. Hence, Stage III ore fluids belong to a medium-low-temperature, low-salinity homogeneous CO2-NaCl-H2O system, which was sourced from the mixing of magmatic water and meteoric water.

Stage IV: Only VL-type FIs are identified, which commonly occur in groups in the same quartz grain. These FIs have similar vapor–liquid ratios (5 to 15%), Th-totals (189.5...
to 240.4 °C), and salinity values (3.7 to 5.7 wt.% NaCl eqv), which decreased further in comparison to Stage III equivalents (Figure 6g,h). This suggests that the VL-type FIs are almost captured simultaneously and that the ore fluids were transformed into a low-temperature, low-salinity homogeneous NaCl-H₂O system accompanied by more meteoric water mixing.

### 6.2. Fluid Evolution and Gold Mineralization

The characteristics of FIs from Stages I and II demonstrate that fluid immiscibility occurred prior and synchronous to gold mineralization in the Songjianghe Au deposit, and the initial ore-forming fluids were derived from the exsolution of magma. Along with the continuous crystallization and evolution of the magma, the volatile-rich (CO₂, CH₄, and H₂O) magmatic fluids ascended to shallower depths owing to the fracturing and/or hydrofracturing of country rocks [73]. Subsequently, the rapid pressure drop caused by fault failure and fluid drainage triggered the fluid immiscibility and phase separation [101] (Stage I; Figure 11).

To capture the fluid immiscibility, various FIs have different vapor–liquid ratios (5 to 15%; Figure 11).

**Figure 11.** Total homogenization temperature (Th-total) vs. salinity plot for ore-stage fluid inclusions assemblages (FIAs) from the Songjianghe Au deposit.

In hydrothermal fluids, gold bisulfide complexes (Au(HS)²⁻ and/or Au(HS)⁰) are the primary ligands for gold migration at a pressure of 200 MPa and temperatures of 200–400 °C [102,103]. Hence, gold bisulfide was the most probable ligand for gold migration in the Songjianghe Au deposit. Additionally, the primitive ore fluids were enriched in CO₂, which may be conducive to the transport of gold bisulfide complexes by buffering the pH of the solution [104]. Stage II (main ore stage) FIs document the continuous occurrence of fluid immiscibility (Figure 11). Relative to Stage I, the CO₂ component of FIs in Stage II is significantly reduced, and salinity drops (Figures 6a–d and 11), revealing the escape of volatile components due to fluid immiscibility. The release of volatiles can result in a decrease in oxygen fugacity, an increase in pH, and a drop in the pressure of the ore fluids, which destabilized the gold bisulfide complexes and accelerated the gold precipitation [80,105]. Synchronously, the HS⁻ generated by the decomposition of gold bisulfide complexes was combined with metal cations to form polymetallic sulfides (e.g., pyrite, chalcopyrite, galena, and sphalerite) and released H⁺ [106].
Following a large amount of volatile components’ escape and metal precipitation, the hydrothermal fluid entered a relatively open condition and meteoric water began to admix (Stage III; Figures 8 and 11). C3-type FIs have the same proportion of aqueous solution and CO₂, and the vapor–liquid ratios of VL-type FIs are similar, revealing that fluid immiscibility essentially ended. As meteoric water was continuously introduced, the ore fluids gradually cooled, diluted, and tended to resemble meteoric water (Figure 11), which signaled the decline and conclusion of the gold mineralization process with the production of calcite and quartz (Stage IV; Figure 4d).

6.3. Fluid Trapping Temperature-Pressure Conditions

In general, it is difficult to accurately estimate the mineralization pressure, unless the mineralization occurred under the exact temperature or immiscible/boiling conditions when the FIs were trapped [94–96]. As end-members trapped in immiscible CO₂-NaCl-H₂O ± CH₄ system fluids, the trapping temperature–pressure conditions of C2- and VL-type FIs could approximately represent the mineralization temperature and pressure. For Stage I and II, the Th-total of some VL-type FIs is slightly lower than that of C2-type FIs, which can be interpreted as a result of multiple fluid immiscibilities with fluctuating pressure during mineralization [107,108]. Therefore, we applied the isochore intersection method to constrain the trapping temperature–pressure conditions of C2- and VL-type FIs (with similar Th-totals) for Stages I and II in the same thin sheet. The volume range of the CO₂ phase for C2-type FIs is over 50% (primarily 80–90%), and VL-type FIs have two phases (vapor H₂O + liquid H₂O) with vapor H₂O accounting for < 50 vol.% (primarily 20–30 vol.%). The isochores of C2- and VL-type FIs were plotted using MacFlincor software [95]. As shown in Figure 12, the trapping temperature and pressure for Stage I and II are 395–525 °C, 1046–1778 bar, and 352–448 °C, 850–1380 bar, respectively. The Songjianghe Au deposit was likely formed at a higher pressure (850–1380 bar) than other major gold deposits characterized by the quartz vein-type in the JHGB (520 to 1030 bar; e.g., Bajiazi, Xiaitai, and Sandaocha [2]), suggesting that the gold mineralization characterized by disseminated-style ores may have occurred at a greater mineralization depth with a small-scale and relatively close structural system. Meanwhile, field investigations also show that the ore-controlling structural system of the Songjianghe Au deposit is characterized by narrower brittle-ductile fractures, whilst other major quartz vein-type gold deposits in the JHGB (e.g., Jiapigoubenqu, Erdaogou, Sendaocha, Xiaobeigou, and Banmiaozhi [8,17]) are controlled primarily by relatively wider ductile-brittle fractures.

![Figure 12. Trapping temperature–pressure diagram for Stage II C2-type and VL-type fluid inclusions from the Songjianghe Au deposit.](image)
6.4. Source of Ore-Forming Materials

There are four stable isotopes of sulfur in nature, with a relative abundance of $^{32}\text{S}$ 95.02%, $^{33}\text{S}$ 0.75%, $^{34}\text{S}$ 4.21%, and $^{36}\text{S}$ 0.02% [109]. Geochemical studies generally consider only the most widely distributed and abundant $^{34}\text{S}/^{32}\text{S}$ ratio, expressed as $\delta^{34}\text{S}$. It is generally accepted that different $\delta^{34}\text{S}$ values can be derived from three common sulfur sources [110]: (1) Mantle or magmatic source ($\delta^{34}\text{S} = 0 \pm 3\%$); (2) seawater sulfates ($\delta^{34}\text{S} = \text{approximately} + 20\%$ but varies greatly); and (3) reduced sulfur from sedimentary rocks (negative $\delta^{34}\text{S}$, generally $<-15\%$). In hydrothermal systems, where $\text{H}_2\text{S}$ is the dominant sulfur species in the fluids or the fluid redox state is below the $\text{SO}_2/\text{H}_2\text{S}$ boundary, the following relation exists as $\delta^{34}\text{S}$ (sulfide) $= \delta^{34}\text{S}$ (fluid) [111]. The sulfur speciation at Songjianghe is dominated by sulfides (especially pyrite, $\sim 90\%$) with no obvious sulfate development. Thus, the $\delta^{34}\text{S}$ values of pyrite could represent the $\delta^{34}\text{S}$ values of the hydrothermal fluid system [112]. There are major differences in sulfur isotopes between the Songjianghe gold deposit and other major gold deposits in the JHGB (Figure 9b): The latter (e.g., the Erdaogou, Xiaobeigou, Bajiazi, Xiaixitai, and Sandaocha deposits; Figure 1c) have relatively wide-range and positive $\delta^{34}\text{S}$ values (1 to 10%), which clusters around 1 to 3% and 5 to 9% [2,87]. The low $\delta^{34}\text{S}$ values (1 to 3%) exhibit a magmatic sulfur isotope signature [110]. The high $\delta^{34}\text{S}$ values (5 to 9%) imply that the deep-sourced sulfur may have mixed with shallow crustal sulfur during the hydrothermal fluid ascent [17,21,87]. Sulfides from the Haigou gold deposit (southeast of Songjianghe; Figure 1c) have negative $\delta^{34}\text{S}$ values ($-10.9$ to $-2.3\%$ [90]), which may be caused by the following factors: (1) The early mineralization stage may have been oxidizing, causing the fractionation of $^{34}\text{S}$, as suggested by the extensive development of magnetite and hematite in the early stage [113]; (2) fluid reactions with the low-$\delta^{34}\text{S}$ wallrocks ($-10.9$ to $-10.5\%$ [90]). In this study, Stage II $\delta^{34}\text{S}$ values ($-0.96$ to $-3.40\%$) are slightly higher than those of Stage I ($-2.91$ to $-0.42\%$), which may be caused by crustal components added during hydrothermal fluid evolution [114]. However, the samples as a whole yielded a relatively narrow $\delta^{34}\text{S}$ range ($-2.91$ to $-3.40\%$, mean $0.48\%$; Figure 9), suggesting a typical magmatic sulfur source (0 ± 3% [112]). Compared to other major gold deposits (e.g., the Erdaogou, Xiaobeigou, Bajiazi, Xiaixitai, and Sandaocha deposits) in the JHGB, the Songjianghe Au deposit has a single sulfur source and relatively lower $\delta^{34}\text{S}$ values. Wang and Yan [115] and Wang et al. [116] proposed that pyrite $\delta^{34}\text{S}$ values are relatively higher in the gold deposits hosted in large and open structural systems, and lower in those hosted in small-scale and relatively close structural systems. Hence, the Songjianghe Au deposit may have been formed in a small-scale and relatively close structural system, as supported by the trapping temperature–pressure conditions and field investigations.

The relative atomic mass of Pb is large, and the relative mass difference between different Pb isotopes is limited. As a result, Pb not only struggles to experience isotope fractionation when leaching from the ore source layer but also generally has no changes in composition during transfer into the ore-forming hydrothermal fluid and subsequent migration [117]. Therefore, lead isotope compositions of sulfides are widely used to constrain the source of lead [118,119]. The pyrite Pb isotope compositions from Stage II projected primarily toward the field between the mantle and lower crust evolution curve (Figure 10a). Meanwhile, they also exhibit a consistent range between $^{206}\text{Pb}/^{204}\text{Pb}$ and $^{208}\text{Pb}/^{204}\text{Pb}$ (Figure 10b). These characteristics indicate that the primitive Pb at Songjianghe may have sourced from a low-radiogenic lower crust and mantle lead reservoir. Although the Pb isotope data of monzogranite and K-feldspar granite in the Songjianghe Au deposit also fall between the field between the mantle and lower crust evolution curve, its relatively high-radiogenic member is distinctly different from those of Stage II pyrite (Figure 10a,b). The Pb isotope compositions from the Seluohe Group biotite-plagioclase schist fall close to the lower crust evolution curve (Figure 10a) and are similar to the ore Pb (Figure 10b), therefore the involvement of Pb from wallrock cannot be ruled out. The Pb isotope data from other major gold deposits (e.g., Erdaogou, Xiaixitai, Sandaocha, Sidoacha, Bajiazi, and Haigou) in the JHGB fall mostly between the mantle and lower crust (Figure 10c,d). Addi-
tionally, the Pb isotope compositions of these gold deposits show broad correlations, which suggest a two-component mixing of the lower crust and mantle components (Figure 10c,d), inconsistent with the Songjianghe Au deposit. These features indicate that the crustal-source Pb contributes significantly less to the Songjianghe Au deposit than other major gold deposits in JHGB. In conclusion, we propose that the Pb in the Songjianghe Au deposit was primarily derived from a magmatic source (mantle and lower crust) and may have been affected by the Seluohe Group wallrock contamination due to the fluid–rock interaction.

6.5. Ore Genesis

The Songjianghe Au mineralization characterized by disseminated-style ores is predominantly hosted in the metamorphic rocks of the Seluohe Group. The distribution of the ore body, spatially related to the sericitization-silicification zone, is primarily controlled by the NNW-striking structures, which were characterized by the early-stage ductile shearing superimposed by the late-stage brittle-ductile fractures.

Previous studies suggest that the Songjianghe Au deposit shares several characteristics with orogenic gold deposits, such as the mineralization type, composition of metallic minerals, characteristic of structural ore-control, and wallrock alteration [25,27], whereas there are apparent differences between the Songjianghe Au deposit and the typical orogenic gold deposits hosted in Precambrian medium-low grade metamorphic rocks [114]. The ore-forming fluids of orogenic gold deposits are usually derived from the progressive metamorphism process under the conditions of greenschist and amphibolite facies [120], whilst the ore fluids at Songjianghe are dominated by magmatic water with later meteoric water input. Additionally, the metamorphic age (2521–2506 Ma) of the ore-bearing wallrocks (the Seluohe Group) is much older than the mineralization age (157–156 Ma) [31]. In addition, there was no visible orogenic metamorphic event in the study region during the Late Jurassic [121,122].

The JHGB is located in the tectonic transition between the northeastern NCC margin and the eastern CAOB, witnessing the formation and extinction of the Paleo-Asian Ocean and the subduction of the Paleo-Pacific plate beneath Eurasia [17,50]. As mentioned in the geological setting, the greenschist facies retrograde metamorphism of the Hulan Group indicates that the Paleo-Asian Ocean closed before the Late Triassic [43,59]. In the Early-Middle Jurassic, the variation in composition polarity of igneous rocks from the continental margin to the intracontinental region revealed the beginning of subduction of the Paleo-Pacific plate in NE China [121–123]. In addition, the widely developed late Early Jurassic to early Middle Jurassic (178–168 Ma) medium-acid intrusions in the JHGB show characteristically geochemical signatures of island arcs or active continental margins, suggesting the occurrence of Paleo-Pacific plate subduction [2,30,49,71]. Abundant gold mineralization events occurred during this period in the JHGB (e.g., Xiaxitai, Sidaocha, Liupiye, and Xiaobeigou [2,4,7]). However, during the Late Middle Jurassic to Early Cretaceous (165–140 Ma), there has been a significant intermittence of magmatic activity in eastern Jilin Province and Songliao Basin, which is interpreted as the Paleo-Pacific plate experiencing an intermittent stage of subduction and/or a strike-slip tectonic regime existing between the continental margin of Northeast Asia and the Paleo-Pacific plate [121,122]. Simultaneously, under the effects of the Paleo-Pacific plate subduction in the Early-Middle Jurassic, the thickened lithospheric mantle from NE China was delaminated due to gravity instability, accompanied by lithospheric thinning [124–126] (Figure 13). According to the sericite $^{40}$Ar/$^{39}$Ar dating, the Au mineralization at Songjianghe took place during the Late Jurassic (157–156 Ma [31]). The delamination and lithospheric thinning may have led to asthenosphere upwelling, resulting in the partial melting of the upper mantle and lower crust to generate original magma carrying abundant metals and volatile components [125,126] (Figure 13). With the evolution and exsolution of the magma, the initial ore-forming fluids were generated (Figure 13). As the ore fluids ascended to the shallower zone, the rapid pressure drop triggered the fluid immiscibility and phase separation, which contributes to the decrease in oxygen fugacity, the increase in pH, and the decomposition of gold bisulfide
complexes. These processes led to significant gold-polymetallic sulfide precipitation (primarily in Stage II). The H-O isotope compositions suggest that the ore-forming fluids are dominated by magmatic water (Stage I and II) and mixed with meteoric water in the later stage (Stage III). As meteoric water was continuously introduced, the ore fluids gradually cooled, diluted, and tended to resemble meteoric water, which suggested the decline and termination of the gold mineralization process (Stage IV). The S-Pb isotope data indicate that ore-forming materials at Songjianghe are characterized by deep magmatic sources and may be affected by the contamination of Seluohe Group wallrocks due to the fluid–rock interaction. Consequently, the Songjianghe Au deposit belongs to a mesothermal magmatic hydrothermal vein gold deposit, which formed in the intermittent stage of Paleo-Pacific plate subduction during the Late Jurassic.

Figure 13. Schematic ore genetic model for the Songjianghe Au deposit.

7. Conclusions
1. The Songjianghe Au deposit is a magmatic hydrothermal gold deposit formed in the intermittent stage of Paleo-Pacific plate subduction during the Late Jurassic. Four alteration/mineralization stages were recognized in this gold deposit: (I) A quartz-pyrrhotite-pyrite stage; (II) a quartz-polymetallic sulfides stage; (III) a quartz-pyrite stage; and (IV) a quartz-calcite-minor pyrite stage.
2. The ore-forming fluids have evolved from a medium-high-temperature, low-salinity immiscible CO$_2$-NaCl-H$_2$O ± CH$_4$ system to a low-temperature, low-salinity homogeneous NaCl-H$_2$O system.
3. The H-O isotope data suggest that the ore-forming fluids were dominated by magmatic water (Stage I and II) and intruded by meteoric water since Stage III.
4. Fluid immiscibility caused by the rapid drop in pressure may have been the main trigger for gold-polymetallic sulfide precipitation in the Songjianghe Au deposit.
5. The S-Pb isotope data indicate that the ore-forming materials have predominantly derived from a magmatic source and may have been affected by the contamination of the Seluohe Group.

Author Contributions: Conceptualization, Q.Y. and K.W.; investigation, Q.Y., K.W., W.B., C.M. and Y.X.; funding acquisition, K.W.; project administration, K.W. and Q.S.; writing—original draft preparation, Q.Y.; writing—review and editing, Q.Y., K.W., X.Z. and Q.S. All authors have read and agreed to the published version of the manuscript.

Funding: This research was supported by the Opening Foundation of Key Laboratory of Mineral Resources Evaluation in Northeast Asia, Ministry of Land and Resources of China (DBY-ZZ-18-12, DBY-KY-18-08).

Data Availability Statement: The experimental data used to support the conclusions of this study are included within the article.

Acknowledgments: We would like to thank Gang Zhao and Hong Wei from the Songjianghe gold mine for helping with the field sampling. In addition, we wish to thank Shuo Zhao (Chinese Academy of Geological Sciences) for providing valuable advice on the manuscript, and to the anonymous reviewers and editors for their insightful comments.

Conflicts of Interest: The authors declare no conflict of interest.

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