Rb-Sr and U-Pb isotopic systematics of pyrite and granite in Liaodong gold province, North China: Implication for the age and genesis of a gold deposit

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The Liaodong gold province, along the eastern margin of the North China craton, is a large productive base of gold in China. The Wulong gold deposit hosted in granitoid is a famous Au-bearing quartz vein gold deposit in this region, and located at the northern side of Sanguliu granite. The ages of Sanguliu granite determined by the conventional Rb-Sr isochron and U-Pb zircon methods are 131 ± 4.5 Ma and 129 ± 2.9 Ma respectively, thus the comprehensive isotopic age of Sanguliu granite is 130 Ma. Lode gold deposits commonly consist of pyrite and lesser amounts of galena and sphalerit sulfides accompanied by quartz and calcite. Here we use direct Rb-Sr dating of pyrite from Wulong gold deposit, and determined the age of Wulong gold deposit. A positive correlation between present-day 87Sr/86Sr and 87Rb/86Sr ratios of pyrite from the Wulong gold deposit corresponds to an age of 120 ± 3 Ma, which dates the age of gold mineralization. The Sr initial ratios (0.714816 to 0.7148927 of Sanguliu granite and 0.715280 to 0.715504 of pyrites in Au-bearing quartz veins) and Pb isotopic compositions in pyrites and Sanguliu granite indicates that lead was derived partially from the similar magmatic source, and the linear array of Pb isotope composition in pyrite show a incorporation of two end members (may be incorporation of basic and acid wall rocks). The studies of H and O isotopes of fluid inclusions in auriferous quartz veins demonstrate that the magmatic water predominates in ore fluids, and also reflect a little formation waters.

INTRODUCTION

A lot of many gold deposits hosted in granodiorite are distributed in eastern part of China. Thus accurate timing of mineralization and diagenesis is fundamental in studying and contrasting the genesis of these gold deposits. In recent decade years or more, with development of determinable technology, many dating methods have been developed and widely used in age determination of gold deposit, e.g., 40Ar/39Ar and Rb-Sr dating of altered minerals such as sericite (Luo and Wu, 1987; Zhang et al., 1994), Rb-Sr dating of fluid inclusions in quartz (Böhlke and Kistler, 1986; Li et al., 1993), Rb-Sr, Sm-Nd, U-Pb, and Re-Os methods of scheelite (Frei et al., 1998; Hart et al., 2002), and dating of Rb-Sr isochron and U-Pb zircon methods of granitoid rocks (Li and Gui, 1990). However direct determination of the ages of hydrothermal mineral deposits has been hampered by the lack of minerals that are suitable conventional radiometric methods. Thus, most attempts to determine the age of lode gold deposits have been indirect.

The ideal way to determine the age of gold deposits is to analyze minerals that are thought to have formed coevally with the gold. One such mineral is pyrite (FeS2), which is commonly associated with gold in many mines. Recent analyses of trace-element abundances in pyrite (Chen et al., 1989) show relatively high levels of Rb, Sr, and rare earth elements (REEs). This characteris-
tic may suggest that Sr and Nd isotopes could be used to monitor the source of the ore-forming materials and fluids from which the pyrites were deposited. Variation in the concentrations of Rb and Sr and their ratios in pyrites show the potential for direct dating (Yang and Zhou, 2001). In this paper, the ages of Sanguliu granite and Wulong gold deposit in Liaodong region, eastern China have been determined by using Rb-Sr isochron and U-Pb zircon methods respectively, and the sources of the materials and fluids associated with gold mineralization have also been defined by multiple isotopic tracers including Sr, Pb, H, and O isotopic data.

**GEOLOGICAL SETTING**

Liaodong area is situated in the eastern margin of the North China craton. It is bounded by the northeast-southwest trending Yalujiang fault zone in the east (Fig. 1), the Wulong gold deposit is limited by the northeast-southwest trending second order faults (fault F2~F6). The F2 and F4 faults are the border faults of west and east side of Wulong gold deposit, and faults controlling ore-bodies are the smallest north-south and/or northwest-southwest trending faults which are trending from 200 m to 2000 m in length (Fig. 1). Supracrustal rocks in the Liaodong district comprise both metamorphic Precambrian sequences and Mesozoic volcanic rocks and intrusions (Ni and Xu, 1993). The Precambrian sequences are composed of the low-grade metamorphic rocks of the Early Proterozoic Liaoh group. Rocks of the group such as metamorphic siltstone, phyllite, marble, graphic schist, which form the basement of the Liaodong gold mineralization area. The metamorphic siltstone in the Liaoh group was determined to be as old as 2200 Ma by conventional Rb-Sr isochron methods (Zhang, 1988). Plutonic rocks, which intruded into the early Proterozoic Liaoh group in the Liaodong area,
have been traditionally divided into two suits, the Dandong and Sanguliu. The Dandong suite consists of medium-grained and pehaluminous biotite granite with high content of SiO₂ (%), its age determined by conventional U-Pb zircon method is as old as 198 Ma (Mu et al., 1989). This suite (exposure area of 3800 km² in Liaodong region) is an area of predominantly low strain and transected by anastomosing shear and high-strain deformation zones. The Sanguliu suite consists of medium-coarse-grained porphyritic granite. Gold deposits (Wulong, Sidaogou gold mines etc.) are located at outside contact of the Sanguliu suite (Fig. 1). In gold deposits, there are well developed various kind of dykes, mainly including fine-grained diorite dykes and granite porphyritic dykes in pre ore-forming stage, and lamprophyre dykes and diorite dykes etc. in post ore forming stage. The former dykes are closely associated with orebodies in space and show intense alteration. The dykes and auriferous quartz stringers are found cutting through marginal phase of the Sanguliu granite (Liu, Y. D., 1987). Two main phases of deformation occurred in the region during the Mesozoic; the first stage, northwest-southeast oblique compression, produced prominent northeast-southwest trending brittle ductile shear zones with sinistral oblique reverse movements, and the second stage involved the development of brittle structure, accompanied by hydrothermal alteration and gold mineralization (Wei et al., 2001).

**GEOLOGICAL CHARACTERISTICS OF GOLD MINERALIZATION**

Historical production data indicate that the Liaodong region was a large repository of gold in China, and the details of the heterogeneous distribution of gold were stated by Liu (1994) and Peng (1994). The mineralization is mainly structurally controlled. Ore bodies are found mostly in third-order steeply inclined strike-slip brittle faults, which have two main orientations of Au-bearing brittle faults; (1) faults striking near S-N or 10°NE and controlling the greater part of the ore bodies of the main auriferous quartz vein, (2) faults striking N20°~40°W, and less controlling ore bodies. According to our field investigation, the above two group ore-bodies are not continuous along the strike, and composed of several en echelon ore bodies near to fined-grained diorite dykes.

Four stages of mineralization are recognized. Stage 1 is presented by non-metallogenic coarse-grained pyrite and quartz veins; Stage 2 shows a mineral assemblage of native gold, disseminated fine-grained pyrite, bismuthinite and lesser amount of galena and sphalerite; Native gold and very fine sulfide minerals, such as pyrite, galena, sphalerite, and chalcopyrite, and dark quartz veins formed stage 3; and stage 4 mainly involves the formation of minor amounts of gangue minerals, such as quartz, calcite, sericite and chlorite. Stage 2 contains the main gold source in the four metallogenic stages of Wulong gold deposit.

The gold is both free and sulfide hosted (usually pyrite, bismuthinite and galena). Bismuthinite is a common constitute in some Au-bearing quartz veins in this area, occurring as finely crystalline concentrations or scattered small grains. The relationship between gold abundance and bismuthinite concentration show a positive correlation.

**SAMPLING AND ANALYTICAL TECHNIQUES**

A total of 17 samples (7 samples for whole rock Rb-Sr isochron age, 4 samples for U-Pb zircon age, and 6 K-feldspar samples for Pb isotope data) of Sanguliu granite were collected from the surface different quarry. All samples are fresh, unaltered, and far away from fault zones. Whole rock samples for Rb-Sr isochron and separates of single minerals (K-feldspar) of Sanguliu granite accepted conventional disposal methods (Li et al., 1993; Li and Li, 1997). The transparent, colorless or light yellow zircon crystals from Sanguliu granite possess perfect crystal form of tetragonal bipyramid, with short or long column, showing principally combinatory forms of (100), (110), and (111), 0.15~0.35 mm long, 0.05~0.15 mm wide. Crackless zircon separates with perfect crystal form, varying shape, granularity, and color were
handpicked out under a binocular microscope, then they were analyzed by a single separate or multiple separates. Besides, a post-air-abrasion analysis was applied to bigger zircon separates.

Five specimens of auriferous quartz veins for ore-forming age were collected from the mineralization stage 2 in the Wulong gold deposit underground. Pure pyrite separates were hand-picked under a binocular microscope to select grains similar in appearance from auriferous quartz. Every specimen from different locations (several to tens of meters apart) were analyzed.

Chemical treatment and U-Pb analysis of zircons were carried out in an ultra-clean laboratory in Tainjun institute of geology and mineral resources. Pb blank values of reagents were as follows: HCl and HF 2~5 pg/ml, ultrapure water 1~3 pg/ml, 7N HNO₃ 10~20 pg/ml. Mean blanks of total process were: Pb 40 pg and U 2 pg. Details of zircon dissolution and U-Pb separation and determination were given elsewhere (Lu and Li, 1991). U and Pb isotope data were determined with a VG-354 mass spectrometer on single Re filaments at the temperature of 1200~1300°C and 1300~1450°C, respectively. The raw data were corrected for instrumental mass fractionation using factors of 0.12%/amu (atomic mass unit) for U and 0.3%/amu for Pb.

Sr and Pb isotope analyses are as follows: Rock or mineral samples weighing ~550 mg were transferred into Teflon vessels, after being washed ultrasonically in Millipore water, and dissolved using a mixture of HCl and HNO₃ in a ratio of 1:3. Subsample solutions were dried and redissolved in HCl for Rb, Sr, and in HBr for Pb, and loaded on quartz columns. Separation of the required elements for isotope analysis was made by cation exchange. Rb, Sr concentrations were obtained by isotopic dilution. Isotopic ratio measurements were made on a multicollector MAT-261 mass spectrometer as described by Ling et al. (1998). Sr isotope data were normalized to ⁸⁶Sr/⁸⁸Sr = 0.1194 and the ⁸⁷Sr/⁸⁶Sr of the Sr standard NBS-607 during this study was 1.200395 ± 8 (2σ, n = 8). Errors are quoted throughout as two standard deviations from measured or calculated values.

Analytical uncertainties are estimated to be <0.5% for ⁸⁷Rb/⁸⁶Sr. The decay constant used in the age calculation is λ⁸⁷Rb = 1.42 × 10⁻¹¹/yr. For Pb isotopes, in-run precision for typical individual analysis was better than 0.1% at the 2σ level. A fractionation correction of 0.1%/amu was applied on the basis of analysis of standard NBS981. Blanks during the course of this study averaged 0.4 ng for Rb, 0.2 ng for Sr, and 0.3 ng for Pb. The Rb-Sr isochron ages and U-Pb zircon age were calculated by using the ISOPLOT (2.9 version) program (Ludwig, 1996).

Nine Au-bearing quartz vein samples from the levels between +80 and –40 m (Table 5) were systematically analyzed for oxygen and hydrogen isotope data, at Yichang Institute of Geology and Mineral Resources using a Nuclide 3-inch 600 ratio mass spectrometer. Separation of quartz from ores (auriferous quartz vein) was accomplished by handpicking without any chemical treatment. The quartz separates were analyzed for δ¹⁸O and extracted fluid inclusion waters were analyzed for δD. To ensure adequate fluid for analyses, 5–8 g of quartz separate were crushed in copper tubes under vacuum. The analytical techniques used are the same as those described in Li et al. (1993) and involve the reaction of ground quartz powders with BrF₅. Results were reported in delta notation relative to SMOW. The overall analytical errors are on the order of 0.1 and 1‰ for oxygen and hydrogen isotopes, respectively.

Fig. 2. Rb-Sr isochron of Sanguliu granite. Isr is initial ratio; MSWD is of mean square of weighed deviate.
Rb-Sr and U-Pb isotopic systematics of gold deposit, Liaodong province, China

**Dating of Sanguliu Granite and Pyrite Associated with Gold Mineralization**

Rb and Sr concentrations of seven samples from Sanguliu granite were listed in Table 1, the scatter observed reflect probably homogeneity of the initial Sr isotope compositions, and the homology of seven samples. Seven samples yielded the Rb-Sr age of 131.1 ± 4.5 Ma, with a low MSWD (mean square of weighted deviates) of 0.656 and an initial $^{87}\text{Sr}/^{86}\text{Sr}$ ratio of 0.71482 (Fig. 2).

Isotopic compositions and ages of U-Pb in zircons from Sanguliu granite were present in Table 2. It can be seen that the difference of the ages are in the order of $t_{206} < t_{207} < t_{206−207}$. The probable explanation is that radiogenic lead of zircons has been lost to a varying degree in the late geological events. The change scope of the four group U-Pb zircon ages ($^{206}\text{Pb}/^{238}\text{U}$ age) is 127.5~135.3 Ma, with a MSWD of 1.98, which also show a little lost of radiogenic lead. The scope of former three group ages and its weight average age are of 127.5~130.3 Ma and 129 ± 2.9 Ma, respectively (Fig. 3), above ages may be approximate to zircon crystal age and approach the emplacement age of Sanguliu granite. The No. 4 zircons in transparent would be reworked by later geological ther-

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**Table 1. Rb-Sr isotopic compositions of Sanguliu granite**

<table>
<thead>
<tr>
<th>Sample</th>
<th>Rock type</th>
<th>Rb (ppm)</th>
<th>Sr (ppm)</th>
<th>$^{87}\text{Rb}/^{86}\text{Sr}$</th>
<th>$^{87}\text{Sr}/^{86}\text{Sr}$ (±2σ)</th>
<th>$(^{87}\text{Sr}/^{86}\text{Sr})_i$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sgy-1</td>
<td>Sanguliu granite</td>
<td>145.2</td>
<td>285.4</td>
<td>1.5951</td>
<td>0.71779 ± 0.00003</td>
<td>0.714843</td>
</tr>
<tr>
<td>Sgy-2</td>
<td>Sanguliu granite</td>
<td>86.7</td>
<td>346.6</td>
<td>0.7204</td>
<td>0.71619 ± 0.00004</td>
<td>0.714859</td>
</tr>
<tr>
<td>Sgy-3</td>
<td>Sanguliu granite</td>
<td>154.2</td>
<td>386.9</td>
<td>1.1102</td>
<td>0.71688 ± 0.00002</td>
<td>0.714829</td>
</tr>
<tr>
<td>Sgy-4</td>
<td>Sanguliu granite</td>
<td>172.9</td>
<td>501.4</td>
<td>1.2958</td>
<td>0.71721 ± 0.00007</td>
<td>0.714816</td>
</tr>
<tr>
<td>Sgy-5</td>
<td>Sanguliu granite</td>
<td>169.4</td>
<td>254.8</td>
<td>1.9256</td>
<td>0.71845 ± 0.00004</td>
<td>0.714892</td>
</tr>
<tr>
<td>Sgy-6</td>
<td>Sanguliu granite</td>
<td>89.5</td>
<td>283.1</td>
<td>0.9088</td>
<td>0.71650 ± 0.00005</td>
<td>0.714821</td>
</tr>
<tr>
<td>Sgy-7</td>
<td>Sanguliu granite</td>
<td>188.1</td>
<td>558.3</td>
<td>1.1874</td>
<td>0.71702 ± 0.00003</td>
<td>0.714826</td>
</tr>
</tbody>
</table>

**Fig. 3. U-Pb zircon age of Sanguliu granite.**
mal event, its age of $^{206}\text{Pb}/^{238}\text{U}$ is 135.2 Ma, show a little older age. Perfect crystal form of analyzed zircons rules out the possibility of relict zircons from paleostrata. Thus comprehensive isotopic age of Sanguliu granite emplacement is 130 Ma.

In the Wulong gold deposit, compositions of Rb-Sr isotope of five pyrite samples are listed in the Table 3. Rb (0.823~1.988 ppm) and Sr (1.923~3.024 ppm) concentrations are variable. Five pyrite samples (Table 3) yielded the Rb-Sr age of 120 ± 3 Ma, with a lower MSWD of 3.62 and an initial $^{87}\text{Sr}/^{86}\text{Sr}$ ratio of 0.715402 (Fig. 4).

Previous efforts on determining the age of Sanguliu granite, based on K-Ar method used on biotite and K-feldspar in Sanguliu granite, gave the age range of 91~136 Ma (Peng, 1994). The age of granite porphyry dykes mineralized is 78.6 Ma, i.e., ore-forming age of Wulong gold deposit is later than the age of emplacement of granite porphyry dykes, whereas the age of altered sericite in the ores is 134 Ma (Liu, 1987), thus previous researches provide different metallogenic ages of Wulong gold deposit. Field geological reconnaissance show that the Au-bearing quartz veins cut the magmatic phase of the Sanguliu granite, i.e., the mineralization is later than the emplacement of Sanguliu granite. Therefore this study provides that the ages of Sanguliu granite and Wulong gold deposit are 130 Ma and 120 ± 3 Ma respectively.

The Liaodong and Jiaodong gold concentration regions are located in the same metallogenic
It is bounded by the north-northeast-trending Tanlu fault belt in the west, and the Jiaodong gold province is at south side of the Bohai Sea. The ages of SHRIMP U-Pb method for Mesozoic intrusion in this region is about 160~126 Ma (Qiu et al., 2002; Wang et al., 2002), and the age of lode gold deposits in this area are from 122~123 Ma (Yang and Zhou, 2001). This effort in Liaodong area interprets the identity on diagenetic and metallogenic ages between Liaodong and Jiaodong region.

### SOURCE OF METELLOGENIC MATERIALS AND FLUIDS

Pb isotope compositions of 23 samples were listed in Table 4. Several K-feldspars in the Sanguliu granite were examined for Pb isotope data in this study. Pyrites in gold-bearing quartz veins contain visible gold or have a high gold content, and their characteristics provide convincing evidence that gold and pyrite are genetically associated (Peng, 1994). The Pb isotope data from
Dandong biotite granite, Sanguliu granite, and pyrites in the auriferous quartz veins of Wulong gold deposit can be used to shed light on the source of ore-forming materials, and their compositions are within rather narrow ranges of 17.193~17.467, 17.496~17.717, 17.487~17.793 for $^{206}\text{Pb}/^{204}\text{Pb}$, 15.464~15.586, 15.552~15.621, 15.514~15.860 for $^{207}\text{Pb}/^{204}\text{Pb}$, 38.220~38.694, 38.521~38.749, 38.269~39.521 for $^{208}\text{Pb}/^{204}\text{Pb}$, respectively. The $^{207}\text{Pb}/^{204}\text{Pb}$ versus $^{206}\text{Pb}/^{204}\text{Pb}$ values (Fig. 5) of Dandong biotite granite, Sanguliu granite, and pyrites in Au-bearing quartz veins also show that pyrites plot within the partial area of the Sanguliu granite, which indicates that the lead in pyrites and Sanguliu granite was derived partially from the similar magmatic source, and the linear array of Pb isotope composition in pyrite show a incorporation of two end member (may be incorporation of basic and acid rocks), but there are obvi-

Table 5. Oxygen and hydrogen isotope data of fluid inclusions in quartz from Au-bearing quartz vein

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sample description</th>
<th>$\delta^{18}\text{O}_{\text{Quartz}}$ (%)</th>
<th>$\delta^{18}\text{O}_{\text{H,O}}$ (%)</th>
<th>$\delta^{18}\text{D}_{\text{H,O}}$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Vein 76-1</td>
<td>Auriferous quartz vein in Stage 2 at 80 m level</td>
<td>12.69</td>
<td>5.33</td>
<td>-67.54</td>
</tr>
<tr>
<td>Vein 76-2</td>
<td>Auriferous quartz vein in Stage 2 at 80 m level</td>
<td>13.44</td>
<td>6.08</td>
<td>-72.13</td>
</tr>
<tr>
<td>Vein 76-3</td>
<td>Auriferous quartz vein in Stage 2 at 80 m level</td>
<td>11.98</td>
<td>4.62</td>
<td>-62.32</td>
</tr>
<tr>
<td>Vein 76-4</td>
<td>Auriferous quartz vein in Stage 2 at 80 m level</td>
<td>11.56</td>
<td>4.20</td>
<td>-75.85</td>
</tr>
<tr>
<td>Vein 76-5</td>
<td>Auriferous quartz vein in Stage 2 at 40 m level</td>
<td>13.69</td>
<td>6.33</td>
<td>-82.32</td>
</tr>
<tr>
<td>Vein 76-6</td>
<td>Auriferous quartz vein in Stage 2 at 40 m level</td>
<td>12.98</td>
<td>5.62</td>
<td>-67.73</td>
</tr>
<tr>
<td>Vein 76-7</td>
<td>Auriferous quartz vein in Stage 2 at -40 m level</td>
<td>11.36</td>
<td>4.00</td>
<td>-82.73</td>
</tr>
<tr>
<td>Vein 76-8</td>
<td>Auriferous quartz vein in Stage 2 at -40 m level</td>
<td>14.45</td>
<td>7.09</td>
<td>-82.12</td>
</tr>
<tr>
<td>Vein 76-9</td>
<td>Auriferous quartz vein in Stage 2 at -40 m level</td>
<td>11.24</td>
<td>3.88</td>
<td>-89.58</td>
</tr>
</tbody>
</table>

Quartz-water fractionation equation: $1000\ln \alpha_{\text{H,O}} = 3.306 \times 10^{-6} T^{-2} - 2.71$ (Zhang, 1989), temperature used is 300°C in ore stage 2.
ous difference in Pb isotopic characteristics between pyrites and Dandong biotite granite. The initial $^{87}\text{Sr}/^{86}\text{Sr}$ ratios of Sanguliu granite (0.714816 to 0.714892) and pyrites (0.715280 to 0.715504) in gold-bearing quartz veins, also suggest a possibility that a little higher $^{87}\text{Sr}/^{86}\text{Sr}$ ratios of pyrites reflect the incorporation of Sr from surrounding metamorphic wall rocks (Fig. 6).

The analytical results of H and O isotope compositions are presented in Table 5. The $\delta^D$ values of the inclusion fluids range from $-89.58$ to $-62.32\%e$. These values are similar to those of magmatic fluid (Taylor, 1974). The $\delta^{18}\text{O}$ values of the ore fluid can be calculated using the quartz-water fraction equation (Zhang, 1989). Nine quartz samples yielded $\delta^{18}\text{O}$ values from 11.24 to 14.45\%e (Table 5). Based on the homogenization temperature (average temperature of main stage 2 is 300°C) from microthermometric study of the fluid inclusions, the calculated average $\delta^{18}\text{O}$ values of water in equilibrium with the quartz would be 3.88 to 7.09\%e, near to the magmatic water area (Taylor, 1974) (Fig. 7), and also reflect a little formation waters.

**CONCLUSIONS**

Rb-Sr isochron age (131.1 ± 4.5 Ma) and U-Pb zircon age (129 ± 2.9 Ma) of Sanguliu granite reported in this paper are reliable, thus the comprehensive isotopic age of Sanguliu granite is 130 Ma.

Rb-Sr isochron age (120 ± 3 Ma) of pyrites in Au-bearing quartz veins from the Wulong gold deposit represent the direct dating of a lode gold deposit using pyrites as the Rb-Sr geochronometer. Results for pyrite document not only the feasibility of using pyrite as the Rb-Sr geochronometer, but also the applicability of Rb-Sr geochronology to various types of hydrothermal pyrite-rich orebodies and rocks elsewhere.

Combined Sr and Pb isotopic data of pyrites, Dandong biotite granite, and Sanguliu granite, this study can be used to interpret that the ore-forming and diageneric materials of Sanguliu granite come near the magmatic source, the liner array of Pb isotope composition in pyrite show a incorporation of two end member of basic and acid rocks, but there are obvious difference in Pb isotopic
characteristics between Pb in pyrites and in Dandong biotite granite.

Oxygen and hydrogen isotope characteristics suggest that the ore-forming fluid came near magmatic water, and also reflect a little formation waters.

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