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## Fluid inclusion studies of the high-grade Ashuanipi Complex, Superior Province, Canada: retrograde P-T path and conditions of gold formation\*,\*\*

by Robert Moritz<sup>1</sup> and Serge Chev  <sup>2</sup>

### Abstract

Gold-bearing Algoma-type iron formations and their high-grade metamorphic host rocks of the Ashuanipi Complex, Superior Province, Canada, have been the subject of a microthermometric fluid inclusion study. Isochores calculated for early  $\text{CO}_2$ -rich fluid inclusions, in conjunction with published geothermobarometric data, indicate a clockwise retrograde P-T-t path for the Ashuanipi Complex. Late low salinity  $\text{H}_2\text{O}$  fluid inclusions are spatially associated with gold showings and yield isochores which suggest, together with arsenopyrite thermometry and metamorphic petrology, that gold was precipitated, at least partially, during post-peak metamorphic cooling and uplift. One of the gold occurrences is characterized by  $\text{CH}_4$ - $\text{N}_2$ -rich fluids which could be responsible for the main gold deposition event or for secondary gold enrichment processes in that showing.

**Keywords:** Fluid inclusions, gold mineralization, metamorphism, Ashuanipi Complex, Superior Province, Canada.

### Introduction

The Ashuanipi Complex is a high-grade metamorphic terrane of the eastern Superior Province in the Canadian Shield (CARD, 1991), and it is host to several sub-economic gold occurrences (CHEV   et al., 1989). The aim of the present microthermometric fluid inclusion investigation is twofold. First, this study seeks to constrain the retrograde pressure-temperature (P-T) path of the Ashuanipi Complex and compare this path to other high-grade metamorphic subprovinces of the Superior Province, such as the Kapuskasing structural zone. Second, in conjunction with other P-T indicators, this study aims at defining the P-T conditions and the relative timing of gold mineralization within the tectonic evolution of the Ashuanipi Complex.

### Geologic setting

The geology of the Ashuanipi Complex is dominated by metatexite with interlayered homogeneous and heterogeneous diatexite. Several stages of igneous activity have affected the Ashuanipi Complex, including tonalitic to granodioritic intrusions, nepheline syenites, and late mafic dikes. U-Pb ages indicate that the metasedimentary rocks and minor metavolcanic rocks were deposited prior to 2700 Ma. The main event of high-grade metamorphism (upper amphibolite and granulite facies), accompanied by intense migmatization, took place between 2650 and 2670 Ma. Cooling from the metamorphic peak and retrogression are interpreted to have taken place from 2650 to 2639 Ma (MORTENSEN and PERCIVAL, 1987; CHEV   and BROUILLETTE, 1991; PERCIVAL et al.,

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Tab. 1 Summary of microthermometric fluid inclusion data.

*Regional scale:*Type 1:  $\text{CO}_2$ -rich fluid inclusions

Tm  $\text{CO}_2$ :  $-64.5 - -56.6^\circ\text{C}$  (n = 109)  
 Th  $\text{CO}_2$ :  $-48.0 - +29.1^\circ\text{C}$  (n = 108)

Type 2: low-temperature/high-salinity aqueous fluid inclusions ( $\text{H}_2\text{O}-\text{NaCl}-\text{CaCl}_2$ )

Tm Ice:  $-35.1 - -12.0^\circ\text{C}$  (n = 23)  
 Tm hydrohalite:  $-24.3 - -23.6^\circ\text{C}$  (n = 3)  
 Th:  $+114 - +207^\circ\text{C}$  (n = 42)  
 Tm halite:  $+140 - +160^\circ\text{C}$  (n = 8)

*Gold occurrences, local scale:*Type 3: Subtype 3a:  $\text{CH}_4$ -rich fluid inclusions (Lac Lilois gold occurrence)

Tm  $\text{CH}_4$ :  $-181.4 - -158.0^\circ\text{C}$  (n = 9)  
 Th  $\text{CH}_4$ :  $-105.0 - -82.1^\circ\text{C}$  (n = 8)

Subtype 3b:  $\text{CH}_4-\text{N}_2$ -rich fluid inclusions (Arsène gold occurrence)

Th (liquid):  $-116.0 - -110.5^\circ\text{C}$  (n = 9)  
 Th (vapour):  $-114.4 - -98.3^\circ\text{C}$  (n = 8)  
 T<sub>sublimation</sub>:  $-64.4 - -59.6^\circ\text{C}$  (n = 5)

Type 4: high-temperature/low-salinity aqueous fluid inclusions ( $\text{H}_2\text{O}-\text{NaCl}$ )

Tm Ice:  $-12.2 - -4.4^\circ\text{C}$  (n = 20)  
 Th:  $+229 - +374^\circ\text{C}$  (n = 20)

Homogenization is to the liquid phase unless stated otherwise. Fluid inclusions were studied in quartz.

1991). Peak metamorphic conditions were constrained to  $700-850^\circ\text{C}$  and 3.5–6.5 kbar based on geothermobarometry of garnet-orthopyroxene-plagioclase-quartz assemblages (PERCIVAL and GIRARD, 1988; PERCIVAL et al., 1991).

Local, metamorphosed Algoma-type iron formations, interbedded with metatexite, host small, sub-economic gold occurrences (CHEVÉ et al., 1989). The iron formations consist of alternating quartz bands and layers of magnetite or pyrigarnite, that include clinopyroxene-orthopyroxene-garnet-quartz-fine grained and disseminated pyrrhotite  $\pm$  biotite  $\pm$  graphite. Local retrograde metamorphism affects the iron formations, with the appearance of amphibole, calcite, quartz, chlorite, epidote, sericite, pyrite, and magnetite replacing pyrrhotite. The gold-bearing mineralization is typically characterized by pyrrhotite, arsenopyrite and pyrite accompanied by subsidiary löllingite, chalcopyrite and pentlandite, and in some rare occurrences by native gold. The highest gold grades tend to be associated with high arsenopyrite contents.

### Fluid inclusion types and microthermometry

The fluid inclusion microthermometric data are summarized in table 1. Four types of fluid inclusions were recognized based on phase relationships at room temperature and heating and freez-

ing behaviour. All types of inclusions occur along trails or in clusters, therefore all types are considered to be secondary inclusions. Type 1 inclusions are  $\text{CO}_2$ -rich fluids. With the exception of four inclusions, melting temperatures of  $\text{CO}_2$  range between  $-58.8^\circ\text{C}$  and  $-56.6^\circ\text{C}$  which indicates a low content of other dissolved gases such as  $\text{CH}_4$  or  $\text{N}_2$ . Type 2 inclusions are low-temperature/high-salinity  $\text{H}_2\text{O}$  fluids. Some of them are two phases (liquid and gas) at room temperature and some contain a third phase (halite crystal). Ice starts to melt at temperatures below  $-40^\circ\text{C}$  which indicates that additional cations besides  $\text{Na}^+$  are in solution, such as  $\text{Ca}^{++}$  and  $\text{Mg}^{++}$ . Type 3 inclusions are  $\text{CH}_4-\text{N}_2$ -rich fluids. Two subtypes can be distinguished. The first subtype has melting and homogenization temperatures between  $-181.0^\circ\text{C}$  and  $-181.5^\circ\text{C}$ , and between  $-82.1^\circ\text{C}$  and  $-105.0^\circ\text{C}$ , respectively. Such melting behaviour is characteristic for  $\text{CH}_4$ -rich fluids. The second subtype shows homogenization to the liquid or vapour phase between  $-98.3^\circ\text{C}$  and  $-116.0^\circ\text{C}$ , and some inclusions show sublimation between  $-59.6^\circ\text{C}$  and  $-64.4^\circ\text{C}$ . Such microthermometric behaviour is diagnostic for  $\text{CH}_4$ - and/or  $\text{N}_2$ -rich fluids (VAN DEN KERKHOE, 1990). Type 4 inclusions are high-temperature/low-salinity  $\text{H}_2\text{O}$  fluids. Melting of ice starts around  $-21^\circ\text{C}$  which suggests that  $\text{Na}^+$  is the dominant cation in solution. Melting temperatures of ice indicates salinities between 7 and 16 weight %  $\text{NaCl}$  equivalent.

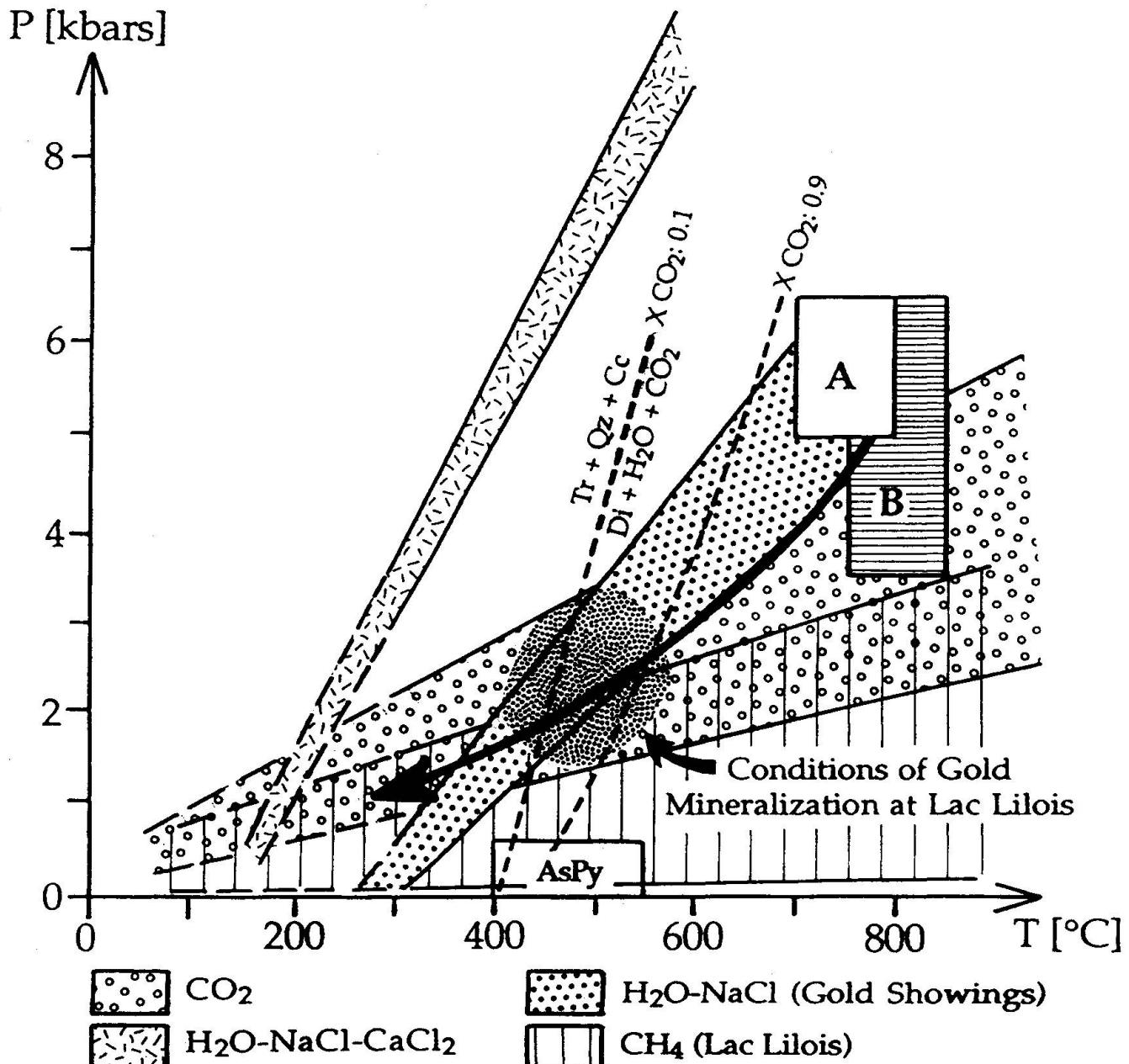


Fig. 1 Uplift and cooling path of the Ashuanipi Complex and conditions of gold mineralization inferred at the Lac Lilois occurrence. Isochores are plotted for the different fluid inclusion types described in the present study. Conditions of gold mineralization at the Lac Lilois occurrence are shown by the highly densely dotted area. The inferred uplift and cooling path is shown by the long arrow. The diopside (Di) +  $H_2O$  +  $CO_2$  = tremolite (Tr) + quartz (Qz) + calcite (Cc) reaction lines are from SLAUGHTER et al. (1975) and have been traced for fluids with different  $CO_2$ -contents, i.e.  $XCO_2$ : 0.1 and  $XCO_2$ : 0.9. A and B are peak metamorphic conditions according to PERCIVAL and GIRARD (1988) and PERCIVAL et al. (1991), respectively. AsPy: preliminary temperature range based on arsenopyrite thermometry.

#### Distribution and origin of the fluids

Type 1 ( $CO_2$ -rich) fluids and type 2 (low-temperature/high-salinity  $H_2O$ ) fluids occur regionally, whereas type 3 ( $CH_4$ - $N_2$ -rich) fluids and type 4 (high-temperature/low-salinity  $H_2O$ ) fluids have only been observed in gold occurrences. Type 1

( $CO_2$ -rich) inclusions are the earliest trapped fluids. Such  $CO_2$ -rich fluids are characteristic of granulite facies terrains (TOURET, 1981). Since migmatization is widespread in the Ashuanipi Complex, a likely origin of the  $CO_2$ -rich fluids is by melting of the high-grade metamorphic rocks whereby the more soluble  $H_2O$  is dissolved in the

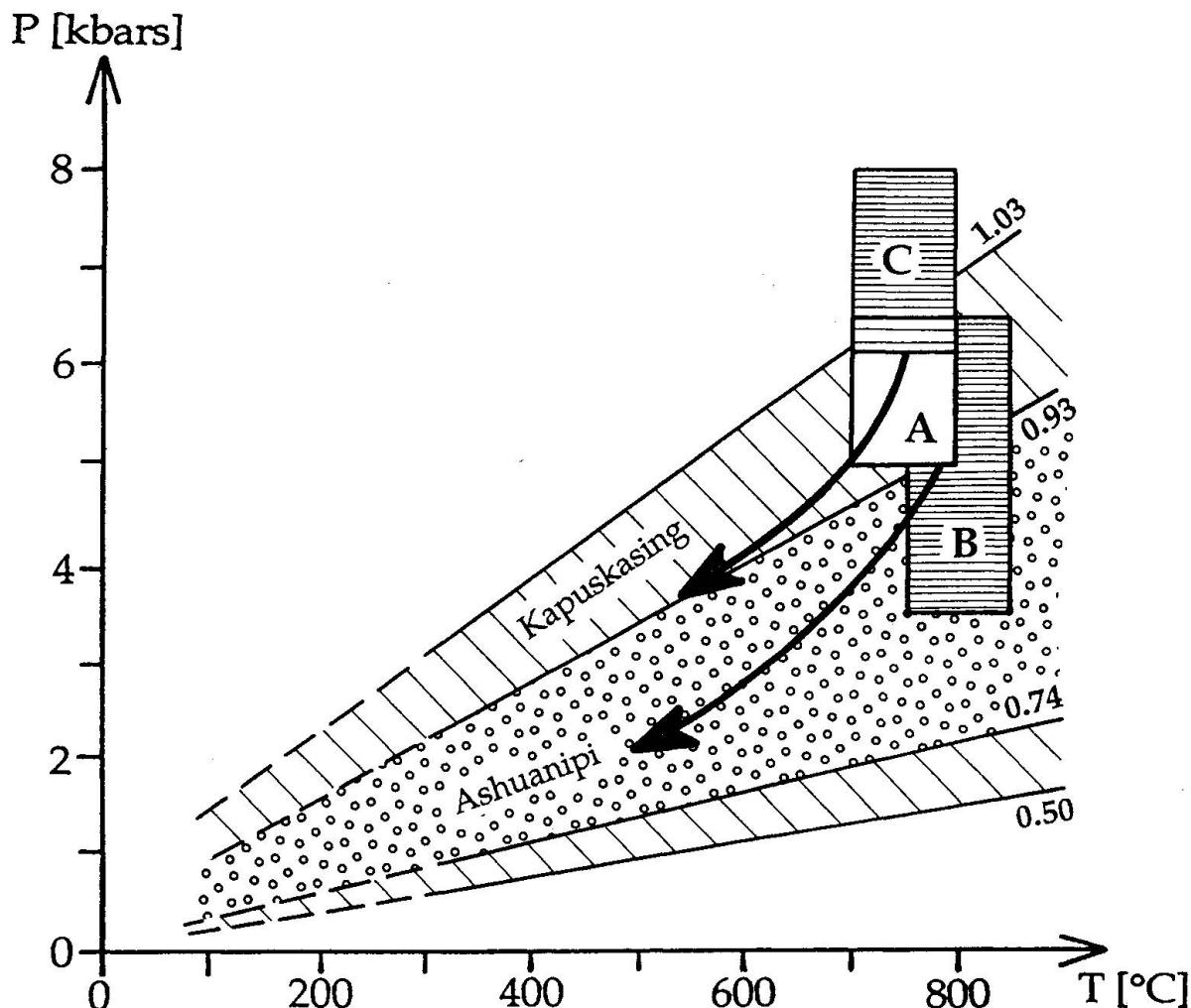


Fig. 2 Comparison of uplift and cooling paths of the Ashuanipi Complex and the Kapuskasing structural zone. Arrows show the uplift and cooling paths inferred from microthermometry on  $\text{CO}_2$ -rich fluid inclusions in the Kapuskasing structural zone (RUDNICK et al., 1984) and in the Ashuanipi Complex (present study). Isochores are in  $\text{g}/\text{cm}^3$ . A and B are peak metamorphic conditions in the Ashuanipi Complex according to PERCIVAL and GIRARD (1988) and PERCIVAL et al. (1991), respectively; and C are peak metamorphic conditions in the Kapuskasing structural zone according to PERCIVAL (1983).

molten fraction and then removed to shallower crustal levels, leaving  $\text{CO}_2$  as a residual component (TOURET, 1981). Additionally, some of the  $\text{CO}_2$  may originate from metacarbonates, the mantle,  $\text{CO}_2$ -bearing magmas or the oxidation of graphite. Type 2 (low-temperature/high-salinity  $\text{H}_2\text{O}$ ) inclusions are similar to saline inclusions found elsewhere in the Canadian Shield (GUHA and KANWAR, 1987; HAYNES, 1988) and are generally interpreted as trapped brines of Proterozoic or Palaeozoic age (FRAPE et al., 1984). However, it is possible that some type 2 inclusions are trapped fluids generated during retrograde metamorphism. Type 3 ( $\text{CH}_4 \pm \text{N}_2$ -rich) inclusions may represent a fluid that has equilibrated with local graphite-rich rocks, such as pyrigarnite. Some of the  $\text{CH}_4$  may also be the vapor component of an

$\text{H}_2\text{O}-\text{NaCl}-\text{CH}_4$  fluid that has undergone phase separation, and some of the  $\text{N}_2$  may be the product of mantle outgassing or breakdown of K-bearing minerals in which  $\text{NH}_4^+$  substitutes for K. Finally, type 4 (high-temperature/low-salinity  $\text{H}_2\text{O}$ ) inclusions may reflect decreasing P-T conditions in the Ashuanipi Complex from granulite to amphibolite-greenschist facies conditions, where  $\text{H}_2\text{O}$  predominates over  $\text{CO}_2$  (TOURET, 1981).

#### Retrograde P-T path

Type 1 ( $\text{CO}_2$ -rich) fluid inclusions yield isochores that pass through and below peak metamorphic conditions in the Ashuanipi Complex determined by PERCIVAL and GIRARD (1988) and PERCIVAL et

al. (1991) (Fig. 1). This suggests that these inclusions record syn- to post-peak metamorphic P-T conditions and indicates a clockwise retrograde P-T-time path, as is common in most granulite terranes (TOURET, 1981). According to PERCIVAL (1989), granulites from the Superior Province can be separated into those having low- to moderate-pressure metamorphic assemblages (3.5–6.5 kbar) (e.g. Ashuanipi Complex) and those having high pressure assemblages (7.5–9 kbar) (e.g. Kapuskasing structural zone). In figure 2 we compare the microthermometric data obtained on  $\text{CO}_2$ -rich fluids from the Kapuskasing high-pressure granulites by RUDNICK et al. (1984) and from the Ashuanipi Complex (this study). The  $\text{CO}_2$ -rich fluids in both terranes yield isochores passing through and below peak metamorphic P-T conditions with similar clockwise P-T-time paths. Figure 2 also shows that our fluid inclusion investigation and that of RUDNICK et al. (1984) support the conclusion of PERCIVAL (1989) that the Kapuskasing zone represents a high-pressure granulite terrane and the Ashuanipi Complex is a low-pressure granulite terrane, in all probability reflecting two distinctive tectonic environments of granulite formation.

### Implications for gold metallogeny

In the Ashuanipi Complex, the best gold grades tend to be associated with high arsenopyrite abundances. In addition, at one of the richest gold occurrences (Lac Lilois), some of highest grade zones are characterized by small quartz segregations or quartz veinlets. The clinopyroxene in proximity to such segregations/veinlets is partially replaced by amphibole, calcite and quartz. As mentioned above, type 4 (high-temperature/low salinity  $\text{H}_2\text{O}$ ) inclusions are restricted to gold occurrences and yield isochores that pass through metamorphic reaction lines that apply to mineralogical transformations observed at the Lac Lilois occurrence, in a temperature range compatible with preliminary arsenopyrite thermometry at the same occurrence (Fig. 1). This led MORITZ and CHEVÉ (1991) to suggest that part of the gold in the Ashuanipi Complex was deposited in a P-T range of 1.5–3 kbar and 400–550 °C, that is at the greenschist-lower amphibolite facies transition.

Another gold occurrence (Arsène) is characterized by  $\text{CH}_4\pm\text{N}_2$ -rich fluids. The association of gold occurrences with such fluids has been reported elsewhere (e.g. GUHA and KANWAR, 1987; BOTTRELL and MILLER, 1990; DE ALVARENGA et al., 1990). According to GUHA and KANWAR

(1987) such  $\text{CH}_4$ -rich fluids could be responsible for late, secondary gold enrichment, whereas BOTTRELL and MILLER (1990) propose that leaching of  $\text{NH}_4^+$  from local wall rocks and subsequent reaction with graphite could provide a reducing mechanism whereby gold would be deposited.

### Conclusions

Four distinctive fluids have been recognized in the high-grade metamorphic rocks of the Ashuanipi Complex: (1)  $\text{CO}_2$ -rich fluids, (2) low-temperature/high-salinity aqueous fluids, (3)  $\text{CH}_4\text{-N}_2$ -rich fluids, and (4) high-temperature/low-salinity aqueous fluids. The two former are distributed regionally, whereas the two latter are restricted to local gold-bearing iron formations. The  $\text{CO}_2$ -rich fluids are the earliest trapped fluids, and they indicate a clockwise retrograde P-T-time path. In combination with the fluid inclusion of RUDNICK et al. (1984) in the Kapuskasing structural zone, the present study supports the suggestion by PERCIVAL (1989) that the Kapuskasing uplift contains high-pressure granulites whereas the Ashuanipi Complex contains low-pressure granulites. One of the gold occurrences, i.e. Arsène, was affected by late  $\text{CH}_4\text{-N}_2$ -rich fluids, that could have been involved in the main mineralization event, or that could have caused secondary gold enrichment. Fluid inclusion microthermometry, arsenopyrite thermometry and metamorphic petrology in another major gold occurrence, i.e. Lac Lilois, suggest that gold deposition took place, at least partially, during post-peak metamorphic cooling and uplift in a P-T range compatible with the amphibolite-greenschist transition, a setting similar to that of major mesothermal gold deposits (KERRICH and WYMAN, 1990).

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