# **Phase Separation of Ore Forming Fluid Related to Gold Mineralization in Wynad Gold Field, Southern Granulite Terrain, India: Evidences from Fluid Inclusion Studies1**

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**Abstract**—Fluid inclusion studies were carried out on auriferous quartz veins of Wynad Gold Field, Southern Granulite Terrain of India. Three types of primary fluid inclusions have been observed; Type-I:  $H_2O-CO_2$ inclusions, Type-II: CO<sub>2</sub> inclusions and Type-III: aqueous inclusions. The Type-I and Type-II inclusions are more abundant than Type-III inclusions. The coexistence of Type-I and Type-II inclusions are common within quartz grains in most of the samples studied. Variation in phase ratio and broad range of total homog enization temperature of Type-I and Type-III inclusions (i.e. 194°C to 300°C and 189°C to 282°C, respectively) indicate the entrapment of heterogeneous fluid in inclusions. This heterogeneity could be due to phase separation of original low saline  $H_2O$ –CO<sub>2</sub> ore fluid in response to drop in pressure and temperature. Gold along with other constituents could have precipitated in response to phase separation of the ore fluid.

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## **INTRODUCTION**

Hydrothermal solutions (fluids) generally consist of water with volatiles  $(CO_2, CH_4, N_2,$  sulfur species and so on), dissolved components of metals and salts at ele vated temperature and pressure. These hydrothermal solutions have been considered to produce huge metal deposits of Cu, Pb, Zn, Au, Ag and so on in different geological setting. Such solutions with different compo nents might have come from either a single or multiple sources such as magma, mantle, rocks, meteoric water and sea water (Misra, 2000). Being less dense, they find suitable ways or make their own ways to come up. Dur ing upwelling they are subjected to changes in physico chemical conditions that will lead to deposition of the dissolved metals present in the fluids.

Lode gold deposits that are often categorized as orogenic gold deposits and characterized by: i—gold quartz ( $\pm$  calcite) veins hosted in metamorphic rocks irrespective of grade of metamorphism and age, ii spatial association with convergent tectonic set-up, iii—high enrichment of some of the rare metals like Au, Ag, As, W, Sb, B, Bi, Pd and Te with very low enrichments of Cu, Pb and Zn, and iv—hydrothermal alterations involving massive introduction of  $CO<sub>2</sub>$ , K, S and H<sub>2</sub>O (Fyfe and Kerrich, 1982; Groves, 1993; Christopher and Vanderhor, 1998; Zhang et al., 2012). Fluid inclusion studies of this type of gold deposits indicate that the hydrothermal fluids involved in min-

The Wynad Gold Field (WGF) of Southern Gran ulite Terrain (SGT), India, is known for ancient min ing activity for gold (Radhakrishna and Curtis, 1999) and the mining activity in this region dates back to 1875 (King, 1878). Several old workings along with high grade gold bearing quartz reefs (10–11 g/t of Au) were discovered by British geologists in the nineteenth century. In WGF, gold has been reported as: 1—pri mary gold associated with quartz and sulfide minerals within quartz veins and 2—secondary gold associated with laterite and placer (Binu Lal et al., 2003; Krish-

eralization are: 1—low to moderately high tempera ture (200–500°C), 2—H<sub>2</sub>O–CO<sub>2</sub> ± CH<sub>4</sub> rich, 3—low to moderate saline and 4—near-neutral to alkaline pH (Groves et al., 1998; Ho et al., 1985; Mikucki, 1998; Ridley et al., 1996). Experimental and thermo dynamic calculations indicate that gold is generally dissolved in hydrothermal solutions as sulfide and chloride complexes (Benning and Seward, 1996; Seward, 1973; Seward, 1984). Sulfide complexes are thought to be the major dissolved form of transport of gold in case of lode gold deposits (Groves et al., 1998; Mikucki, 1998). The reasons for the deposition of gold from high temperature fluids are change in P-T condi tions, phase separation, wall rock alteration and sur face chemistry-driven processes (Mikucki, 1998; Wil liams-Jones et al., 2009). The phase separation is one of the noteworthy processes for the deposition of gold in significant amount. This mechanism has been pro posed based on the fluid inclusion studies (Caulibaly et al., 2008; Zhang et al., 2012).

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**Fig. 1.** Geological map of the Southern Granulite Terrain (Santosh and Sajeev, 2006). WGF—Wynad Gold Field; MSZ—Moyar Shear Zone; BSZ—Bhavani Shear Zone; PCSZ—Palghat Cauvery Shear Zone; CSZ—Cauvery Shear Zone; KKPT—Karur- Kambam-Painavu-Trichur shear zone; ACSZ—Achankovil Shear Zone.

namurthi, 2013). The deposition of primary gold within quartz veins was attributed to phase separation of original immiscible  $H_2O$ – $CO_2$  fluid (Binu Lal et al., 2003). But, concrete evidences for the phase separa tion mechanism were not given by Binu Lal et al. (2003). In this paper, we present certain new evidences such as presence of primary  $CO<sub>2</sub>$  fluid inclusions and coexistence of  $H_2O$ –CO<sub>2</sub> inclusions with CO<sub>2</sub> inclusions in auriferous quartz veins of WGF. Petrographic and microthermometric record of the inclusions related to gold-sulfide-quartz veins of the WGF indi-

cating phase separation of the ore fluid leading to dep osition of gold has been discussed in this paper.

## REGIONAL GEOLOGY

The SGT is one of the Proterozoic orogens in southern Peninsular India, comprising high grade metamorphic rocks like charnokite, khondalite and migmatite invaded by younger pegmatite intrusives (Fig. 1; Chetty and Santosh, 2013; Soman, 2002). It is a collage of crustal blocks which are separated by the



**Fig. 2.** Geological map of the Wynad Gold Field (after Binu-Lal et al., 2003).

Cauvery Shear Zone (CSZ) and the Achankovil Shear Zone (ACSZ) (Chetty and Santosh, 2013; Chetty et al., 2006). The CSZ marks the boundary of two major crustal blocks of SGT namely the Archean granulite block to the north and the Neoproterozoic granulite block to the south (Chetty and Santosh, 2013).

The CSZ trends E–W and consists of a set of par allel pervasive shear zones. The Moyar Shear Zone (MSZ), which hosts the mineralized quartz veins of WGF, is the northern most shear zone of CSZ (see Fig. 4 of Chetty and Santosh, 2013). The MSZ runs about 200 km from west to east and merges with Bhavani Shear Zone (BSZ) at the eastern periphery of the Nil giri Block. The shallow to steeply plunging stretching lineations characterize these shear zones of CSZ (Chetty and Santosh, 2013). Such multi-scale struc tural observations along the CSZ are suggestive of typ ical transpressional tectonics (Chetty and Rao, 1998). The CSZ has undergone two major phases of deforma tion. The first phase of deformation took place in the Neoarchean period and the second one in the Neoproterozoic period (Chetty and Santosh, 2013; Santosh et al., 2012).

## LOCAL GEOLOGY AND MINERALIZATION

WGF is located immediately south of the MSZ (Fig. 1). The area comprises mainly migmatitised hornblende gneiss, amphibolite, biotite gneiss, garnet biotite-sillimanite gneiss, pyroxene granulite, magne tite quartzite and talc-tremolite-actinolite schist (Binu Lal et al., 2003; Pruseth et al., 2011). The auriferous hydrothermal quartz veins are hosted in amphibolite and gneisses (Fig. 2). The veins generally strike along NE-SW direction and cut the regional trend of foliation. The discordant relationship of the auriferous quartz veins with respect to foliation of host rocks and age of emplacement of mineralized quartz veins (~450 Ma) determined by Pruseth et al. (2011) suggest that the veins were formed after the Neoprot erozoic tectonothermal event. Chloritization, sericiti zation, sulfidation and carbonatization represent the hydrothermal alteration of the host rocks in the area (Binu Lal et al., 2003; Pruseth et al., 2011). In the auriferous quartz veins, the dominant sulfide minerals are pyrite, pyrrhotite, arsenopyrite and chalcopyrite. Visible gold grains have been reported as native gold, inclusions in pyrite and, as micro-veinlets within pyrite and chalcopyrite (Binu Lal et al., 2003).

### MATERIALS AND METHODS

Auriferous quartz vein samples were collected from WGF (around Devala, Maruda and Pandalur area; see Fig. 2) and twenty doubly polished wafers were pre pared at Wadia Institute of Himalayan Geology, Dehradun. A Nikon ECLIPSE E200 petrological microscope was used for petrography of fluid inclu sions and microthermometry was carried out using a Linkam THMSG 600 heating-freezing stage equipped on a Nikon LV 100 Pol microscope at the Department of Earth Sciences, Indian Institute of Technology, Roorkee. The unit operates in the temperature range of  $-196^{\circ}$ C to + 600°C. The stage was periodically cal-

50 μm Trans-granular trail Intra-Intra-granular trail

Cluster of inclusions

**Fig. 3.** Photomicrograph showing the distribution pattern of fluid inclusions.

ibrated by synthetic pure  $CO<sub>2</sub>$  inclusions (triple point =  $-56.6$ °C) in quartz. The samples containing suitable fluid inclusions were observed before the microther mometry. Salinity and density of fluids in inclusions were calculated by FLINCOR software (Brown, 1989) using appropriate equations.

#### RESULTS

#### *Fluid Inclusion Petrography*

In the samples of WGF, abundant inclusions were observed in doubly polished wafers prepared from mineralized vein quartz. The inclusions occur as clus ters, along intra-granular trails as well as trans-granu lar trails and as isolated occurrence (Fig. 3). Criteria given by Roedder (1984) and Shepherd et al. (1985) were followed to distinguish primary, secondary and pseudo-secondary inclusions. The isolated inclusions and inclusions in clusters were considered as primary with respect to the formation of quartz vein. The inclusions in intra-granular trails and trans-granular trails were designated as pseudo-secondary and sec ondary, respectively. Only the primary inclusions (5– 15 µm) were studied in detail. At room temperature two types of primary inclusions were observed, biphase liquid-vapour and monophase liquid inclusions (Fig. 4b).

The inclusions were further classified based on micro thermometry as Type-I:  $H_2O$ – $CO_2$  inclusions, Type-II:  $CO<sub>2</sub>$  inclusions and Type-III: aqueous inclusions (Fig. 5). The coexistence of Type-I and Type-II inclu sions is very common (Fig. 4).

**Type-I: H<sub>2</sub>O–CO<sub>2</sub> inclusions.** At room temperature these inclusions consist of two phases with a bub ble in aqueous phase (Fig. 5a i), but during freezing a new phase appears exhibiting liquid  $H_2O$ , liquid  $CO_2$ and vapour  $CO<sub>2</sub>$  (Fig. 5a ii). These inclusions range in size from 3 to 15 µm having different shapes such as rounded, tubular and irregular. The phase proportion  $(H<sub>2</sub>O: CO<sub>2</sub>)$  within such inclusions varies from 90 : 10 to 10 : 90 (table). These are the most abundant variety of primary inclusions observed in the doubly polished wafers.

**Type-II:** CO<sub>2</sub> inclusions. At room temperature these inclusions appear to be monophase (Fig. 5b i), but vapour phase appears during freezing (Fig. 5b ii). These inclusions homogenise to a single phase below room temperature during heating runs. The size of the inclusions ranges from 2 to10 µm having different shapes such as rounded, tubular, elongated and irreg ular. This type of inclusions are as abundant as Type-I inclusions.



Fig. 4. Photomicrographs showing the co-existance of Type-I and Type-II inclusions (a) in plane polarized light and (b) magnified part of inset shown in (a).

**Type-III: aqueous inclusions.** At room temperature these inclusions appear as biphase with liquid  $H_2O$  and  $H<sub>2</sub>O$  vapour (Fig. 5c i). Size of these inclusions range from 4 to12 µm with different shapes such as oblate and spherical. The liquid to vapour ratio in these inclusions range from 80 : 20 to 50 : 50 (table).

#### *Microthermometry*

**Freezing data.** The melting of  $CO_2(T_m CO_2)$  in Type-I and Type-II inclusions occurred between –55.5°C and  $-60^{\circ}$ C (Fig. 6a; table). In most of the inclusions the values of  $T_{\text{m}}$  CO<sub>2</sub> fall in the range of  $-56$  to  $-57$ °C indicating the presence of pure  $CO<sub>2</sub>$ . However, a few

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Fig. 5. Photomicrographs of Type-I, Type-II and Type-III primary fluid inclusions at different temperature.

inclusions show  $T_m CO_2$  values around  $-60^{\circ}C$ , which can be due to the presence of other gases like  $CH<sub>4</sub>$  and  $N_2$  (Shepherd et al., 1985). The first melting of ice  $(T_{FM}$  ice) in most of the Type-I and Type-III inclusions occurred between  $-18^{\circ}$ C to  $-29^{\circ}$ C (table). But, in some inclusions it occurred between  $-37^{\circ}$ C to  $-40^{\circ}$ C (Fig. 6b). This data indicates the presence of different types of salts such as NaCl, KCl and MgCl<sub>2</sub> based on the eutectic temperature given in Shepherd et al. (1985). Final melting of ice  $(T<sub>m</sub>$  Ice) in Type-III inclusions was observed in a temperature range from  $-1$ <sup>o</sup>C to  $-5^{\circ}$ C (table) suggesting the salinities from 2 to 8 wt % NaCl equivalents (Fig. 6c, e). Melting of clathrate was observed in few larger Type-I inclusions. The temper atures of clathrate melting  $(T_m CL)$  are between  $2^{\circ}C$ and 8°C (Fig. 6d). Salinities deduced from the clath rate melting temperature are from 4 to 14 wt% NaCl equivalents (Fig. 6e).

**Heating data.** The homogenization of  $CO<sub>2</sub>$  (Th  $CO<sub>2</sub>$ ) in Type-I and Type-II occurred into liquid phase at temperature range of 4.7°C to 28.3°C and from –4°C to 28.3 $^{\circ}$ C, respectively (Fig. 7a). Density of CO<sub>2</sub> present in the inclusions estimated by  $Th CO<sub>2</sub>$  values ranges from 0.64 to 0.95 (Fig.7b). The total homoge nization of Type-I inclusions took place into both liq uid and vapour phase at the temperature (Th TOT) range 194°C to 300°C (Fig. 8a; table). The Type-III inclusions homogenized into liquid phase at tempera ture range from 189°C to 282°C (Fig. 8b; table).

#### DISCUSSION

The auriferous quartz veins of WGF are hosted in the shear induced fractures of MSZ (Krishnamurthi, 2013). The age of emplacement of the auriferous quartz veins is about 450 Ma (Pruseth et al., 2011). This geo-chronological information indicates a possi ble temporal linkage between the gold mineralization in WGF and the last phase of Neoproterozoic defor mation (Pan-African) event in MSZ. Based on the results of the present work, the condition of entrap ment of fluid inclusions and P-T conditions of fluid activity pertaining to gold mineralization have been discussed below.

#### *Conditions of Fluid Entrapment*

The cause of heterogeneity of an original homoge neous fluid is phase separation. A single generation of fluid inclusions trapped from a heterogeneous fluid due to phase separation would be of different types with different phase proportions and phases (Roedder, 1984; Diamond, 2003). Diamond (2003) illustrated the heterogeneous entrapment of a single fluid inclu sion assemblage (FIA) trapped within a growing crys tal from fluid experiencing phase separation (Fig. 9). Five inclusions (1 to 5 in Fig. 9) of a FIA would have different phase proportion at the time of trapping as shown in Fig. 9b. Upon cooling each inclusion of that assemblage behaves differently because of different bulk properties at the time of trapping. Hence, at room temperature all the five inclusions will have different

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Type-II primary fluid inclusions		
S.No.	$T_{\rm m}$ CO <sub>2</sub> (°C)	T <sub>h</sub> CO <sub>2</sub> (°C)
$\mathbf{1}$	$-57.3$	$+8.2(L)$
	$-56.8$	$+25.4(L)$
$\frac{2}{3}$	$-57$	$+21(L)$
$\overline{4}$	$-57.4$	$+28.3(L)$
5	$-56.9$	$-3(L)$
6	$-56.7$	$-2(L)$
$\overline{7}$	$-56.8$	$+7(L)$
8	$-56.9$	$+11(L)$
9	$-56.7$	$+26.3(L)$
10	$-56.7$	$-4(L)$
11	$-56.7$	$+1.7(L)$
12	$-56.6$	$+5(L)$
13	$-56.8$	$+9.3(L)$
14	$-56.8$	$+11(L)$
15	$-56.8$	$+12(L)$
16	$-57.1$	$+9.2(L)$
17	$-56.6$	$+16.6(L)$
18	$-56.6$	$+12.7(L)$
19	$-57$	$+5(L)$
20	$-60$	$+25(L)$
21	$-56.8$	$+28.2(L)$
22	$-56.6$	$+14(L)$
23	$-56.9$	$+15.8(L)$
24	$-57.1$	$+9(L)$
25	$-56.5$	$+5.6(L)$
26	$-57.1$	$+7(L)$
27	$-56.7$	$+12.8(L)$
28	$-56.6$	$+24(L)$
29	$-56.6$	$+21.9(L)$
30	$-56.8$	$+6(L)$
31	$-57.2$	$+14.6(L)$
32	$-56.6$	$+8.9(L)$
33	$-56.9$	$+26(L)$

phase proportions and bulk properties as shown in (Fig. 9c). As a result the isochores, temperature of homogenization and state of homogenization will be different for each inclusion of that FIA.

From the results of fluid inclusion studies of WGF, the following inferences have been outlined and used for detailed discussion:

i. Three types of primary inclusions have been observed; Type-I:  $H_2O$ – $CO_2$  inclusions, Type-II:  $CO_2$ inclusions and Type-III: Aqueous inclusions. The co existence of Type-I and Type-II primary inclusions in a single quartz grain is very common.

ii. The phase proportions at room temperature in Type-I  $(H_2O: CO_2)$  and Type-III (liquid to vapour) inclusions range from 90 : 10 to 10 : 90 and 80 : 20 to 50 : 50, respectively (table). The range of total homog enization temperature (Th TOT) is very broad i.e. 194°C to 300°C for Type-I inclusions and 189°C to 282°C for Type-III inclusions. The Type-I inclusions homogenizes into both liquid and vapour state (table).

iii. The fluid involved in the mineralization of gold in WGF was  $H_2O$ – $CO_2$  dominant low saline (calculated average value of 8 wt % NaCl equivalents) with minor amount of other gases like  $N_2$  and CH<sub>4</sub> as inferred from microthermometric analysis.

iv. The  $T<sub>h</sub>$  TOT v/s salinity plot shows (Fig. 10) that there is a considerable variation of  $T<sub>h</sub>$  TOT within a narrow range of salinity. This indicates the fluid immiscibility as reported in other areas such as Piaba gold deposit, São Luís cratonic fragment of Brazil and Xincheng deposit, Jiaodong Peninsula, China (de Freitas and Klein, 2013 and Wang et al., 2014).



\*—Not observed/Not calculated.

Type-III primary fluid inclusions

S.No—Serial Number.

*T*h TOT—Temperature of total homogenization.

(L)—Homogenization into liquid state.

(V)—Homogenization into vapour state.

 $T<sub>h</sub> CO<sub>2</sub>$ —Temperature of homogenization of CO<sub>2</sub>.

 $T_{\rm m}$  CO<sub>2</sub>—Temperature of first melting of CO<sub>2</sub>.

*T*m CL—Temperature of melting of clathrate.

 $T_{\text{FM}}$  Ice—Temperature of first melting of ice.

*T*m Ice—Temperature of final melting of ice.

L : V—Liquid to vapour ratio.



**Fig. 6.** Histograms of different parameters of primary inclusions obtained during freezing. (a)  $T_m CO_2$  v/s frequency, (b)  $T_{FM}$  Ice v/s frequency, (c) *T*m Ice v/s frequency, (d) *T*m CL v/s frequency (e) Salinity v/s frequency.

On the basis of the above inferences and comparing with the concept of heterogeneous entrapment of fluid inclusions given by Diamond (2003), we consider that gold bearing homogeneous fluid became heteroge neous due to phase separation (fluid immiscibility) in WGF. Due to the heterogeneous entrapment of aque ous carbonic fluid, different kinds of primary fluid inclusions such as aqueous (Type-III), carbonic

(Type-II) and aqueous-carbonic (Type-I) with differ ent phase proportion were trapped (point i, ii and iii as mentioned in the inferences and Fig. 11). In addition to this, the broad range of total homogenization tem perature, different state of homogenization (liquid and vapour) observed during thermometric runs (point ii as mentioned in the inferences and Fig. 8) and co existence of Type-I and Type-II primary inclusions

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**Fig. 7.** Histograms of Th CO<sub>2</sub> v/s frequency (a) and Density of CO<sub>2</sub> v/s frequency (b).



**Fig. 8.** Histograms of *T*h TOT of Type-I inclusions v/s frequency (a) and *T*h TOT of Type-III inclusions v/s frequency (b).

(point i as mentioned in the references and Fig. 4) are the important outcome of the current fluid inclusion studies on WGF samples. All these evidences support phase separation of original homogeneous ore forming fluid and consequent heterogeneous entrapment of aqueous carbonic gold bearing fluid in WGF. The results of studies by Caulibaly (2008), Yao et al. (2001) and Klemd (1998) related to lode gold deposits also indicated phase separation of ore forming fluid result ing in gold deposition.

## *Estimation of P-T conditions of Fluid Entrapment*

The principal methods described by Shepherd et al. (1985) to estimate fluid pressure at the time of trap-

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ping are (i) the vapour pressure of the fluid at temper ature of total homogenization, (ii) fluid isochores used in conjuction with independent geothermometers, (iii) intersecting isochores for coeval fluid and (iv) dis solution of daughter minerals (halite). The co existance of Type-I and Type-II inclusions in the sam ples during the present work allowed us to use the intersecting isochores method. Isochores of Type-I and Type-II inclusions were plotted using Bowers and Helgeson (1985) and Holloway (1981)'s equations of state, respectively. P-T diagrams were constructed for three suitable cases of co-existance of Type-I and Type-II (Fig. 12). The temperature and pressure val ues of the intersection points are: 328°C/2.41 kbar (IP-1), 289°C/1.93 kbar (IP-2), 253°C/1.49 kbar (IP-3),



**Fig. 9.** Illustration of heterogeneous entrapment of a fluid inclusion assemblage (Diamond, 2003).



Fig. 10.  $T<sub>h</sub>$  TOT v/s salinity plot of Type-I and Type-III inclusions.

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**Fig. 11.** Schematic diagram depicting the heterogeneous entrapment of original aqueous carbonic homogeneous fluid in WGF. Different kinds of primary inclusions (Type-I, Type-II and Type-III) have been shown at the bottom of the diagram. At room temperature the Type-III and Type-I inclusions appear bi-phase but were distinguished during microthermometric analysis (see section 5.1).

241°C/1.34 kbar (IP-4) and 237°C/1.42 kbar (IP-5). These estimated temperature and pressure values have been considered as P-T conditions of trapping of fluid inclusions in the auriferous quartz veins.

#### **CONCLUSION**

Our study indicates entrapment of immiscible flu ids (i.e. heterogeneous entrapment) in inclusions present in auriferous quartz veins of WGF. The original homogeneous fluid in WGF was low saline  $H_2O$ –CO<sub>2</sub>. The fluid was subjected to immiscibility (or phase sep aration) within temperature and pressure range of 240 to 330°C and 1.4 to 2.4 kbar. Gold along with other constituents could have precipitated in response to phase separation of the ore fluid. The phase separation of original homogeneous gold bearing ore fluid could be due to drop of pressure and temperature as it entered the fractures developed during the late phase of Neoproterozoic (Pan-African) deformation event.

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**Fig. 12.** Estimation of Pressure-Temperature conditions of fluid entrapment related to gold mineralization by inter secting isochores method using isochores of coexisting Type-I and Type-II inclusions.

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