

## Use of Synthetic Fluid Inclusions as a Simultaneous Temperature-Pressure Logging Tool in High-Temperature Geothermal Reservoirs

Kotaro Sekine<sup>\*1</sup>, Greg Bignall<sup>\*2</sup> and Noriyoshi Tsuchiya<sup>\*3</sup>

<sup>\*1</sup> Institute of Fluid Science, Tohoku University, Katahira 2-1-1, Aoba-ku, Sendai, 980-8577, Japan

ksekine@ifs.tohoku.ac.jp

<sup>\*2</sup> Institute of Geological and Nuclear Science Ltd, Wairakei Research Centre

State Highway 1, Private Bag 2000, Taupo, New Zealand

G.Bignall@gns.cri.nz

<sup>\*3</sup> Graduate School of Environmental Studies, Tohoku University, Aza-Aoba 20, Aramaki, Aoba-ku, Sendai, 980-8579, Japan

tsuchiya@mail.kankyo.tohoku.ac.jp

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### ABSTRACT

Batch autoclave experiments were conducted to evaluate the potential use of synthetic fluid inclusions as a simultaneous temperature-pressure logging tool in high-temperature (>350°C) geothermal systems. The application of synthetic fluid inclusions provides pressure-temperature conditions for deep-seated geothermal systems, which cannot be obtained by conventional tools because of extreme temperature conditions. Fluid inclusions, up to 50 µm long were readily synthesized during 5 day-long autoclave experiments conducted at 375 to 475°C, and 39 to 62 MPa in pre-fractured, inclusion/impurity-free artificial quartz. Inferred fluid inclusions trapping conditions are calculated by deducing the intersection of isochores derived from microthermometric data for three sets of simultaneously trapped synthetic fluid inclusions in healed microfractures. Synthetic fluid inclusion logging offers a precise borehole temperature measurement technique with an estimated error of only  $\pm 12^\circ\text{C}$  without need of any pressure correction. Pressure estimates are less precise, although the method may be improved by using a combination of H<sub>2</sub>O-NaCl and H<sub>2</sub>O-KCl solutions. Saline fluid/quartz/amorphous silica systems, which facilitate crack healing but trap fluids that are not homogenizing at near-critical conditions may also be advantageous.

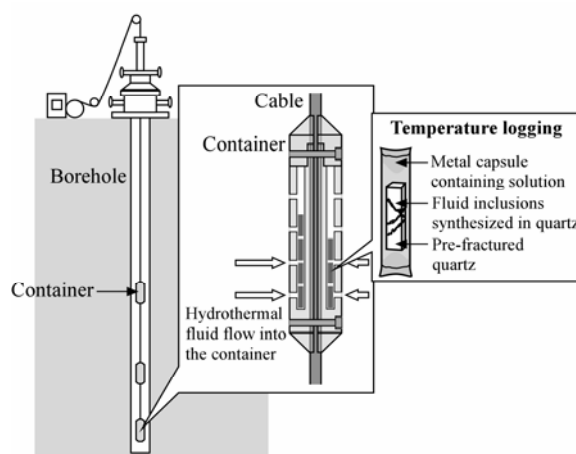
### 1. INTRODUCTION

Information about the thermal structure of a geothermal reservoir is important for optimum utilization of geothermal resources. Natural fluid inclusions trapped in hydrothermal mineral veins and wall rocks from coring samples yield insights into the thermal conditions of formation temperature. However, microthermometric characteristics of those fluid inclusions may not represent the current conditions of the geothermal reservoir due to thermal disequilibrium after drilling (Ruggieri and Bertini, 2000) and to the uncertainty of timing of fluid entrapment.

Synthetic fluid inclusion logging is an alternative method to directly determine the current formation temperature of geothermal well. This method involves synthesizing fluid inclusions in a pre-fractured mineral (e.g. quartz, calcite etc), which is enclosed with a fluid saturated with respect to that mineral in an appropriate capsule, then lowered into the well (Figure 1). Synthetic fluid inclusions trapped due to

microcrack healing are subsequently analyzed by standard microthermometric techniques. Our method is similar to the method of Bethke *et al.* (1990), who studied synthetic fluid inclusions in natural Brazilian quartz from Valles Caldera (USA). They demonstrated that trapping temperatures and fluid compositions were consistent with results obtained using other logging tools.

In recent years, synthetic fluid inclusion logging has proven to be an effective means of measuring in-situ borehole temperature, at sub to supercritical conditions in Enhanced Geothermal Systems (EGS) now being considered as potential heat resources (e.g. WD-1a, Kakkonda, Japan; Sawaki *et al.*, 1997). Because conventional logging tools that record temperature data electronically are only useful at temperatures below about 350°C, metal melting tablets are the only reliable means of inferring borehole temperatures of more than 400°C. (Sawaki *et al.*, 1997; Ikeuchi *et al.*, 1998). On the other hand, the synthetic fluid inclusion logging technique is effective up to the  $\alpha$ - $\beta$  quartz transition temperature (573°C at 0.1 MPa), if quartz is utilized as the host mineral.



**Figure 1: Schematic representation of synthetic fluid inclusion logging (modified after Sawaki *et al.*, 1997). Pre-fractured mineral is suspended in a borehole in order to synthesize fluid inclusions.**

As indicated, synthetic fluid inclusions have the potential to trap “in-situ” borehole fluids yielding borehole temperatures even at supercritical conditions. Conventional synthetic fluid inclusion logging however, requires pressure data to determine borehole temperatures.

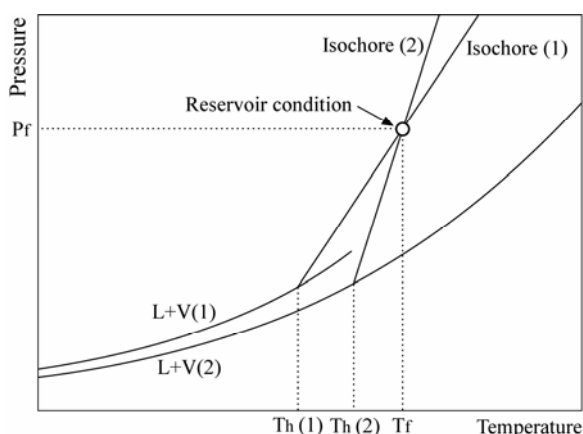
The aim of this study is to provide a new application of synthetic fluid inclusions for the determination of borehole temperature. The synthetic fluid inclusion logging method proposed here, offers not only borehole temperature in a geothermal reservoir without any pressure correction, but also yield insights into the pressure in the borehole. The accuracy of our method is confirmed through a series of batch autoclave experiments conducted at 375 to 475°C, and 39 to 62 MPa.

## 2. METHODOLOGY

### 2.1 Use of Synthetic Fluid Inclusions for Simultaneous Temperature-Pressure Measurement of Borehole

Figure 2 shows a  $P$ - $T$  projection of a phase diagram with corresponding isopleths for the system  $H_2O$ -NaCl. As shown, different salinities give a unique  $P$ - $T$  relationship of the boiling curve and isochores. It follows therefore, that measured fluid inclusion homogenization temperatures ( $T_h$ ) depend on the salinity of the trapped fluid, when fluid inclusions are trapped at the same  $P$ - $T$  conditions in a borehole or autoclave. Therefore, more than two sets of microthermometric data ( $T_h$  and salinity) of fluid inclusions, trapped simultaneously at the same  $P$ - $T$  conditions have the ability to provide the temperature and pressure condition in a geothermal reservoir.

In application of this synthetic fluid inclusion logging technique, three or more fluid-filled capsules are lowered to the same depth within a geothermal well to trap borehole fluids in fluid inclusions. Subsequently, reservoir/well  $P$ - $T$  conditions are inferred by extrapolating isochores based on the  $T_h$  data sets, which intersect at the inferred “trapping”  $P$ - $T$  condition.



**Figure 2: A conceptual diagram of a phase diagram demonstrating the potential of simultaneous  $P$ - $T$  logging using synthetic fluid inclusions. (Symbols; L+V: boiling curve,  $T_h$ : homogenization temperature,  $T_f$ : formation temperature,  $P_f$ : formation pressure)**

### 2.2 Experimental Procedure

The synthetic fluid inclusions technique has been developed by Sterner and Bodnar (1984) and Bodnar and Sterner (1985a; 1985b; 1987). The methods of trapping of borehole fluids in fluid inclusions used in this study are based on those of Bodnar and Sterner.

An inclusion-free block of artificial quartz (3x3x7 mm) was pre-fractured by heating to 350°C, and then rapid cooling prior to our microcrack healing experiment. Microcrack healing experiments were conducted in gold capsules with an outer diameter of 5 mm, wall thickness of 0.15 mm and length of 35 mm. The end of each capsule was welded to enclose solution ( $\pm NaCl_{(s)}$ ), an amorphous silica, and pre-fractured artificial quartz.

Three such capsules containing fluid with different sodium chloride concentrations were loaded into the batch-type autoclave and held at a pre-determined temperature and pressure condition for 5 days. This procedure was repeated at several hydrothermal conditions, from 375°C to 475°C, and 39 MPa to 62 MPa (Table 1) to verify the experimental procedure, and to simulate the range of conditions that might be encountered in a high-temperature geothermal reservoir.

After synthesizing the fluid inclusions, ~150  $\mu$ m-thick sections were cut perpendicular to the  $c$ -axis of quartz, and doubly polished for fluid inclusion microthermometry using the Linkam THMS 600 heating-freezing stage. The stage was calibrated using metal melting (tin, 231.9°C; lead, 327.5°C; zinc, 419.6°C), ice melting temperature of pure water, and eutectic temperature of NaCl solution (-21.2°C). The calibration error of heating experiments ( $T_h$  and halite melting determinations) ranged less than 5°C, while Freezing measurement ( $T_m$  values for ice/hydrohalite melting) were accurate to  $\pm 1.0^\circ\text{C}$ . A 1°C/min heating rate was applied to all heating and cooling experiments.

## 3. OCCURRENCE AND THERMOMETRIC CHARACTERISTICS OF SYNTHETIC FLUID INCLUSIONS

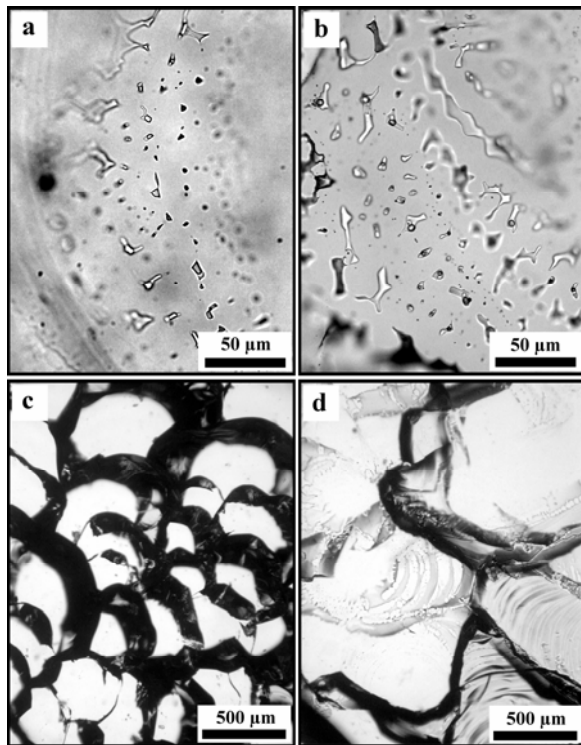
Figure 3 shows microphotographs of fluid inclusions synthesized in this study. Most fluid inclusions were trapped along conchoidal fractures formed by quenching prior to the microcrack healing experiment. Fluid inclusion shape differed with respect to the position of the fracture. Sub-rounded fluid inclusions were observed near the microcrack tip, but irregular, elongate inclusions occurred away from the tip, which is consistent with previous studies addressing crack healing mechanisms (e.g. Smith and Evans, 1984).

It is apparent that crack healing was most prevalent in our experiments containing highly saline fluid, where inclusions were abundant, larger (~50  $\mu$ m) and irregularly shaped. Petrographic fluid inclusion observation revealed no heterogeneous entrapment of fluid inclusions at elevated  $P$ - $T$  conditions.

Experimental conditions and microthermometric data for synthetic fluid inclusions in this study are listed in Table 1. Sample labels “a”, “b” and “c” in each microcrack healing experiment represent three starting fluids of different salinities at the same microcrack healing experiment. The “a” capsule contains pure water, “b” contains 25 wt.% NaCl aqueous solution (unsaturated at room temperature), and “c” is a fluid of unknown salinity, which is supersaturated with NaCl at room temperature. In a few experiments, seal breaks resulted in fluid leaking from the capsule. These samples are not useful for determining the conditions that the fluid inclusions were trapped, although microthermometric data are recorded in Table 1 (labeled as “leaked”).

**Table. 1: Experimental conditions and microthermometric data of synthetic fluid inclusions. In case of sample 2a, healing behavior is weak and no measurable fluid inclusions for ice melting temperature were trapped. Note that  $T_f$ : formation temperature ( $^{\circ}\text{C}$ ),  $P_f$ : formation pressure (bars),  $T_h$ : homogenization temperature ( $^{\circ}\text{C}$ ),  $T_{m(\text{ice})}$ : ice melting temperature ( $^{\circ}\text{C}$ ),  $T_{m(\text{HH})}$ : hydrohalite melting temperature ( $^{\circ}\text{C}$ ),  $T_{m(\text{halite})}$ : halite melting temperature ( $^{\circ}\text{C}$ ).**

Sample	$T_f$ ( $^{\circ}\text{C}$ )	$P_f$ (bars)	$\lambda\text{NaCl}$	$T_h$ [median]	$T_h$ [range]	$n$	$T_{m(\text{ice})}$ [median]	$T_{m(\text{ice})}$ [range]	$T_{m(\text{HH})}$ [median]	$T_{m(\text{HH})}$ [range]	$T_{m(\text{halite})}$ [median]	$T_{m(\text{halite})}$ [range]	Salinity [median]	Salinity [range]	$n$	Remark
1a	470	469	0wt.%	371.5	370.0 ~ 378.8	10	-0.3	-0.4 ~ 0.0	—	—	—	—	0.5	0.0 ~ 0.7	11	
1b	470	469	25wt.%	453.3	436.3 ~ 458.0	10	—	—	-14.2	-15.6 ~ -13.9	—	—	24.2	24.0 ~ 24.3	9	
1c	470	469	>25wt.%	455.4	442.3 ~ 467.5	43	—	—	—	—	161.4	158.9 ~ 164.2	30.1	30.0 ~ 30.2	10	
2a	380	524	0wt.%	321.8	308.3 ~ 327.1	7	0.0	—	—	—	—	—	0.0	—	0	
2b	380	524	25wt.%	328.3	326.7 ~ 329.3	6	-2.9	-3.3 ~ -2.8	—	—	—	—	4.8	4.6 ~ 5.4	3	leaked
2c	380	524	>25wt.%	357.9	352.6 ~ 374.2	9	—	—	—	—	323.1	319.4 ~ 324.8	40.0	39.7 ~ 40.2	9	
3a	430	565	0wt.%	358.6	357.2 ~ 359.9	10	-0.2	-0.4 ~ -0.1	—	—	—	—	0.4	0.2 ~ 0.7	11	
3b	430	565	25wt.%	379.4	377.2 ~ 384.4	14	-8.5	-15.2 ~ -8.2	—	—	—	—	12.3	11.9 ~ 18.8	13	leaked
3c	430	565	>25wt.%	403.1	402.0 ~ 404.0	11	—	—	—	—	345.5	344.6 ~ 346.4	42.0	41.9 ~ 42.1	10	
4a	475	617	0wt.%	376.1	376.1 ~ 376.1	2	-0.6	-0.6 ~ -0.4	—	—	—	—	1.1	0.7 ~ 1.1	4	
4b	475	617	25wt.%	438.2	436.6 ~ 440.9	13	—	—	-8.6	-8.8 ~ -8.6	—	—	25.0	25.0 ~ 25.0	6	
4c	475	617	>25wt.%	456.7	455.5 ~ 466.8	10	—	—	—	—	440.2	439.0 ~ 441.1	52.1	51.9 ~ 52.2	10	
5a	400	391	0wt.%	357.6	355.6 ~ 360.9	8	-0.6	-0.7 ~ -0.6	—	—	—	—	1.1	1.1 ~ 1.2	7	
5b	400	391	25wt.%	377.7	371.9 ~ 391.9	13	—	—	-17.8	-18.5 ~ -17.3	—	—	23.7	23.6 ~ 23.8	10	
5c	400	391	>25wt.%	389.3	385.3 ~ 408.0	12	—	—	—	—	391.3	375.4 ~ 396.4	46.5	44.9 ~ 47.0	17	
6a	375	388	0wt.%	337.4	336.9 ~ 337.7	6	-0.4	-0.4	—	—	—	—	0.7	0.7 ~ 0.7	5	
6b	375	388	25wt.%	339.5	321.1 ~ 358.5	6	—	—	-21.4	-22.2 ~ -18.1	—	—	23.2	23.1 ~ 23.7	7	
6c	375	388	>25wt.%	355.6	346.0 ~ 366.3	11	—	—	—	—	364.1	362.7 ~ 377.1	43.7	43.6 ~ 45.0	6	



**Figure. 3: Microphotograph of cracked artificial quartz sample after crack healing experiment. (a) Conchoidal healed crack treated for 400 $^{\circ}\text{C}$ , 511bars experiment with 1.4 wt.% NaCl solution (sample not listed in Table. 1). (b) High salinity fluid inclusions synthesized at 430 $^{\circ}\text{C}$ , 565bars in 42wt.% NaCl solution (sample 3c). (c) Unhealed microcracks treated in pure water (sample 2a). (d) Healed microcracks with saline fluid (sample 2c).**

Homogenization temperature and salinity data demonstrate a slight variation of  $\pm 10^{\circ}\text{C}$ , and  $< 2$  wt.% NaCl variation respectively, which indicates that homogeneous fluid was trapped at each elevated  $P$ - $T$  condition. Measurable fluid inclusions were produced in experiments lasting only 5 days over a wide range of  $P$ - $T$  conditions. This is a positive attribute for utilization of the synthetic fluid inclusion technique as a temperature-pressure logging tool (presumably also fluid sampling tool) in sub to supercritical hydrothermal reservoirs.

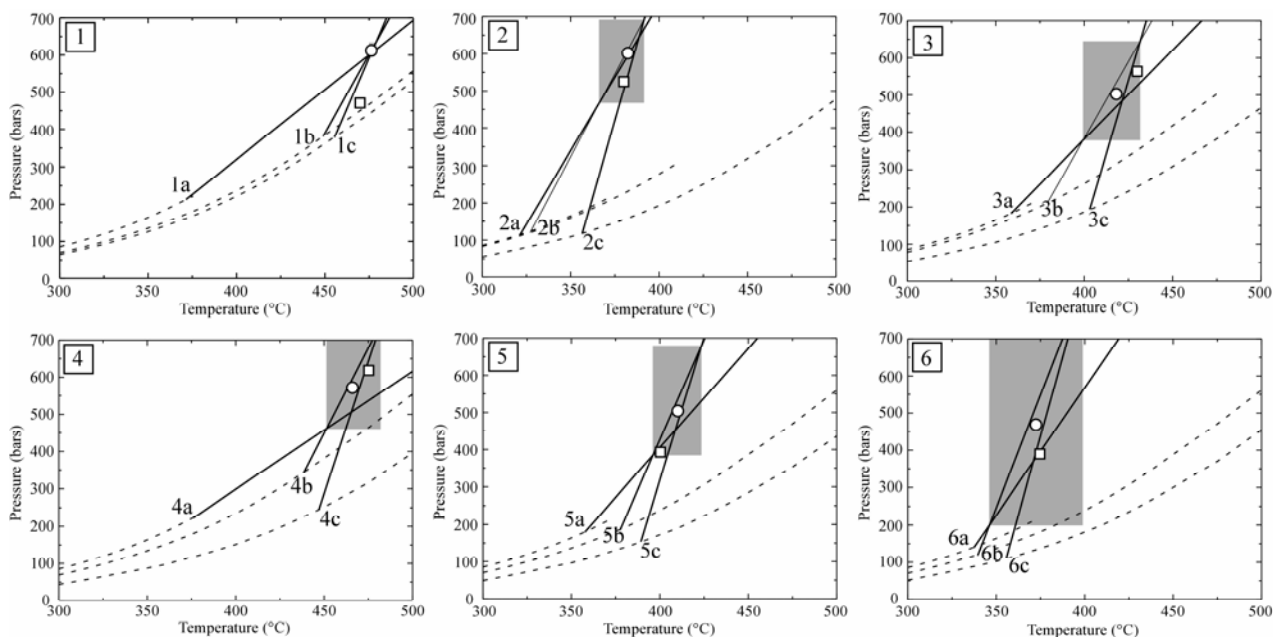
#### 4. TEMPERATURE/PRESSURE EVALUATION USING SYTHETIC FLUID INCLUSIONS

Experimental  $P$ - $T$  conditions (i.e. borehole conditions) were deduced by calculating the intersection of three isochores, derived from microthermometric data for three sets of simultaneously trapped synthetic fluid inclusions. In this study, the internal pressure of low salinity fluid inclusions at homogenization ( $< 1.1$  wt.%  $\text{NaCl}_{\text{eq}}$ ) was calculated following Haas (1971), whereas the phase diagram of Bischoff and Pitzer (1989) was used to obtain the internal pressure of high salinity inclusions at homogenization ( $> 1.1$  wt.%  $\text{NaCl}_{\text{eq}}$ ). The slope of isochore drawn for  $< 40$  wt.%  $\text{NaCl}_{\text{eq}}$  fluid inclusions was calculated following Bodnar and Vityk (1994), while the isochores for high salinity inclusions ( $> 40$  wt.%  $\text{NaCl}_{\text{eq}}$ ) are inferred using the equation of state described by Anderko and Pitzer (1993).

Figure 4 illustrates the  $P$ - $T$  relationships between *actual* experimental condition and the *inferred* “borehole” condition based on synthetic fluid inclusion microthermometry. Median values of  $T_h$  and salinity data were chosen for delineation of isochores. In the figure, dashed lines indicate boiling curves and solid lines represent isochores (the isochore for fluid inclusions, synthesized in a capsule where the seal break occurred, is represented in narrow solid line). Inferred experimental conditions are indicated by shaded area based on intersections of isochores.

Figure 4 shows that (i) three isochores intersect close to the actual experimental conditions throughout all experimental conditions (e.g. experimental condition number 2, 3, 5 and 6); (ii) the isochore for high saline fluid inclusions tends to be most consistent with experimental condition; (iii) pressure estimation is less accurate due to the high angle of isochore; (iv) isochores starting from near the critical point of fluid inclusions, especially pure water fluid inclusions, are likely to deviate from actual experimental condition (e.g. experimental condition number 1 and 4).

These results demonstrate that  $P$ - $T$  conditions in a sub to supercritical geothermal reservoir can be estimated by the intersection of isochores derived from microthermometric



**Figure. 4:** *P-T* diagrams demonstrating actual experimental condition, and inferred “borehole” condition deduced by synthetic fluid inclusion logging. Actual experimental condition and inferred condition are represented by square and shaded area, respectively. Circle indicates averaged *P-T* values corresponding to the intersections of isochores for different saline fluid inclusions.

data for three sets of simultaneously trapped synthetic fluid inclusions. However, inferred “borehole” *P-T* conditions are less precise in some experiments (1 and 4).

The use of a narrow range of fluid salinities in the three-capsule autoclave experiments has a detrimental effect on a precise determination of trapping conditions and is likely to result in non-convergent isochores. A potential variation to our experimental procedure may be to use one or more capsules that do not contain  $\text{H}_2\text{O-NaCl}$  fluids, but instead contain fluids in the  $\text{H}_2\text{O-KCl}$  system.

In addition, fluids, which do not homogenize at near critical conditions, should be used. Knight and Bodnar (1989) showed fluid inclusions, which homogenize at the critical condition do so by a gradual fade and disappearance of the meniscus between the liquid and vapor. In this study, fluid inclusions that homogenized near the critical temperature invariably had erroneous  $T_h$  values (e.g. for “1a” and “4a” with median  $T_h$  values of 371.5°C and 376.1°C respectively). Small variations in measured  $T_h$  can seriously affect isochore slope and the occurrence of fluid inclusions with near critical density is the likely cause of apparent isochore deviation from the trapping condition.

Quantitative evaluation of our method was undertaken by comparing averaged *P-T* values corresponding to the intersection of fluid inclusion-derived isochores (i.e. inferred “borehole” conditions) and actual experimental conditions (Table. 2). There is good agreement between inferred temperatures and experimental temperatures ( $\pm 12^\circ\text{C}$ ) but pressure estimations are less reliable (up to 143 bars discrepancy for experiment “1”), as shown in Table 2. The pressure deviation is a consequence of the isochores having a steep gradient, which limits the use of synthetic fluid inclusion as a pressure estimator, although the method is clearly effective in indicating precise (e.g. borehole) temperatures without need of pressure correction.

**Table. 2:** Comparison of inferred *P-T* conditions based on our method and actual experimental conditions.

Sample	Actual experimental condition		Inferred “borehole” condition	
	Temperature (°C)	Pressure (bars)	Temperature (°C)	Pressure (bars)
1	470	469	476	612
2	380	524	382	602
3	430	565	418	503
4	475	617	466	570
5	400	391	410	506
6	375	388	372	467

In conclusion, if a method can be developed to successfully lower and retrieve a three-capsule micro-autoclave system into/from a high-temperature geothermal well, then synthetic fluid inclusions offer an easy-to-use technique of determining the temperature-pressure regime within deep, high-temperature geothermal reservoirs. This technique avoids operating limitations of conventional temperature logging tools. The time required to heal microcracks and trap the fluid inclusions may be as much as 5 days (but possibly less), which means the tool would be unlikely to be used during drilling stages, although a set of pre-prepared quartz - amorphous silica - fluid filled capsules could be kept in-site and used as required, which means the tool would most likely be used after a period of “shut-in” following completion of the drilling program.

## 6. CONCLUSIONS

A series of batch autoclave experiments were conducted to test the application of synthetic fluid inclusions for measuring temperature-pressure conditions in geothermal boreholes. We show that the use of synthetic fluid inclusions in enhanced geothermal systems up to supercritical conditions, can overcome problems associated with conventional temperature logging tools, which commonly operate at conditions of no more than  $\sim 350^\circ\text{C}$ .

Our method involves synthesizing fluid inclusions in pre-fractured, inclusion and impurity-free quartz that is loaded into sealed gold capsules with amorphous silica powder and fluids of varying salinity. Autoclave experiments were conducted at hydrothermal conditions from 375 to 475°C and 39 to 62 MPa, which correspond to the range of possible conditions within high-temperature geothermal reservoirs. After 5 days, the artificial quartz was examined and revealed microcrack healing had trapped liquid-rich fluid inclusions up to 50  $\mu\text{m}$  long. Crack healing was more advanced in experiments using saline fluids rather than pure water, and tended to form large, irregularly-shaped fluid inclusions.

Temperature and pressure conditions of trapping were calculated from the intersection of isochores derived from microthermometric data for three sets of simultaneously trapped synthetic fluid inclusions. Our work shows that synthetic fluid inclusion logging is a precise borehole temperature measurement technique with an error of  $\pm 12^\circ\text{C}$  at supercritical conditions without need of any pressure correction. Our pressure estimates were less reliable due to the steep slope of isochores derived from our fluid inclusion microthermometry.

The narrow range of fluid salinities in some three-capsule autoclave experiments also had a detrimental effect on our ability to accurately deduce the trapping condition, as sub-parallel isochores were indicated. Furthermore, it can be difficult to resolve the disappearance of the liquid-vapor meniscus for fluid inclusions that homogenize near the critical point, and this may lead to errors in measuring some  $T_h$  values, and also slope of isochores is strongly dependent on  $T_h$  in this  $P$ - $T$  range. For these reasons, we advocate using a range of salinities, as well as one or more capsules that contain  $\text{H}_2\text{O}$ -KCl or other species, instead of only  $\text{H}_2\text{O}$ -NaCl.

There are technical limitations to the use of synthetic fluid inclusions as a temperature-pressure indicator, and some issues remain unresolved such as how to place a three-capsule micro-autoclave into a deep, high temperature geothermal well for 5 days – but this study has formulated a means of utilizing synthetic fluid inclusions to obtain information about high-temperature geothermal reservoir systems, that could not be obtained by conventional logging tools.

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