

Fluid Inclusion Microthermometry and Dating of The Quartz Cement within The Upper Nubian Sandstone Reservoir, SE Sirte Bain, Libya

Sadeg M. Ghnia^{1*}, Ali M. Karboub², and Abdulsalam M. Almishkhi³

¹Department of Geophysics, Faculty of Arts and Science, Al-Mergheb University, Mesallata, Libya.

²Exploration Department, Waha Oil Company, Tripoli, Libya.

³Department of Marine Geology, Faculty of Marine Resources, Alasmarya Islamic University, Zliten, Libya.

*Corresponding author: sghnia@yahoo.com

ميكروثيرموميترى للموائع المحصورة داخل مادة الكوارتز اللاحمة مع تحديد عمرها
في مكمن الحجر النوبي العلوي، بجنوب شرق حوض سرت، ليبيا.

الصادق ميلود غنية^{1*}، علي محمد كربوب² و عبدالسلام مفتاح المشيخي³

¹ قسم الجيوفيزياء، كلية الآداب والعلوم، مسلاته، جامعة المرقب، ليبيا.

² إدارة التنقيب عن النفط، شركة الواحة للنفط، طرابلس، ليبيا.

³ قسم الجيولوجيا البحرية، كلية الموارد البحرية، الجامعة الأسمرية الإسلامية، زليتن، ليبيا.

Abstract

Quartz cement is the most important cementing agent in the Upper Nubian Sandstone reservoir within the Hameimat trough, SE Sirte Basin. Determining the timing and paleo-pore fluid compositions is important in order to understand the reservoir quality of the Upper Nubian succession. Fluid inclusions microthermometry study, using a modified U.S.G.S. gas flow heating/freezing stage system, together with basin modeling was carried out to determine the time of quartz cementation and paleo-pore fluid compositions. Fluid inclusions study results give a precipitation temperature range of silica cement as 115°C-128°C. Investigation of this temperature with burial history curves indicates the onset of quartz overgrowth cementation was in Late Palaeocene probably from through flow of silica-enriched pore fluid. Tice and Teu's measurements indicate that these were complex brines.

Keywords: Upper Nubian Sandstone, Quartz cement, Fluid inclusions microthermometry

الملخص

مادة الكوارتز اللاحمة تعتبر من اهم المواد اللاحمة المؤثرة في خزان الحجر الرملي النوبي العلوي بمنخفض الحميمات بجنوب شرق حوض سرت. إن معرفة العمر والتركيب الكيميائي للموائع القديمة يعتبر ذو اهمية من حيث تقييم جودة المكمن لتتابعات الحجر النوبي العلوي. إن دراسة الميكروثيرموميترى للموائع المحصورة داخل مادة الكوارتز اللاحمة باستخدام النظام المعدل (U.S.G.S) الذي يستخدم الغاز المتدفق للتدفقة مع نظام للتجميد واستخدام نمذجة الأحواض في منطقة الدراسة لتحديد وقت بداية تكون مادة الكوارتز اللاحمة والتركيب الكيميائي للموائع القديمة. نتائج الدراسة أظهرت ان درجة حرارة ترسيب مادة السيليكات تراوحت بين 115 و 128 °م وباستخدام منحني الازدحام الناتج من برنامج نمذجة الأحواض تبين أن بداية ترسيب مادة الكوارتز اللاحمة كان في وقت متأخر من عصر الباليوسين ربما من خلال تدفق سائل غني بالسيليكات. وتشير قياسات درجة التجمد للموائع بأن المحاليل المسؤولة عن ترسيب مادة الكوارتز اللاحمة كانت محاليل ملحية معقدة التركيب الكيميائي.

الكلمات الدلالية: ميكروثيرموميترى، الحجر الرملي النوبي العلوي، الإسمنت الكوارتزى، الموائع المحصورة داخل مادة الكوارتز.

1. Introduction

This paper presents the results of fluid inclusion study of primary quartz overgrowth on core samples from the Upper Sandstone reservoir in well A1-NC125. Knowledge of the diagenetic effect on the hydrocarbon reservoirs has long been important in hydrocarbon exploration and production. The objectives of this study are to determine the time of quartz cementation and paleo-pore fluid compositions. These were evaluated by measuring temperature at which phase transition occurs within primary fluid inclusions when heated and cooled. This includes: (a) Homogenisation temperature (T_h). Homogenisation temperature measurements can give an estimate of the temperature at which fluid inclusion become trapped during the precipitation of the authigenic quartz. (b) Final ice melting temperature (T_{ice}). The final ice melting temperature is proportional to the salinity of the fluid in the inclusions, depending on the amounts of salts and other solutes presents in the inclusions which depress the melting point of ice. (c) Eutectic temperature (T_e). This represents the temperature at which liquid first appears. If the studied inclusions are relatively small, as is generally the case with diagenetic environments, T_e is thus difficult to determine with great precision (± 5 °C).

The system used to conduct this analysis is a modified U.S.G.S. gas flow heating/freezing stage mounted on an optical petrographic microscope with UV fluorescence and photographic facilities. The sample is placed in the middle window of a metal chamber which contains 6 other windows separated by glass cover slips. The system is able to measure a temperature range between -196 and +700 °C by using preheated element present within the stage. During this analysis the system was operated at a temperature range of -65 to +140 °C.

Fluid inclusion studies go back to 1858 when Sorby explained that the gas bubbles in fluid inclusions were the result of differential shrinkage when cooled to the temperature of observation. The formational temperature can thus be estimated by heating the sample until the bubble disappears. Since this original observation, fluid inclusions techniques have been successfully used in numerous mineralogical studies. Fluid inclusions can be defined as fluids (petroleum, water, or gas) once present in the subsurface and trapped in vacuoles sealed within the crystals of the host mineral. Most of these fluids (more than 99%) were trapped from homogenous fluids that were saturated with regard to the host crystal (Roedder, 1984). They may contain one fluid phase, a gas phase and a liquid phase, two liquid phases, or a gas phase or two liquid phases. Crystalline phases may occur with any combination of liquid and gas phases. When the inclusions, which formed at higher temperature during deep burial, are brought to the surface they generate small vapor bubbles (less than 15 volumes %) (Goldstein and Reynolds, 1994). Consequently, all fluid inclusions that formed under the same physical and chemical conditions have the same liquid to vapor ratio and would homogenise at the same temperature when heated in the lab. Thus the consistency in L:V ratio is the diagnostic feature underpinning the validity of individual fluid inclusions assemblage (FIA) measurements.

Fluid inclusions may be primary or secondary (formed at later stage from different pore fluids). Primary fluid inclusions are vacuoles of pore fluid that were trapped during mineral growth and can be used to estimate the physical and chemical conditions at the time the host mineral was precipitated (Roedder, 1984). In the case of quartz overgrowth cements, the fluid inclusion presents at the boundary between the detrital grain and the quartz overgrowth represents the initiations of quartz cementation and thus the microthermometric data will give temperature and composition of the pore fluid from which precipitation took place providing that the fluid inclusions developed in a predictable way.

2. Nubian Successional the South-East Sirte Basin (Hameimat Trough)

The term Nubian Sandstone is widely applied in North Africa and the Middle East to denote a clastic lithofacies that unconformably overlies Palaeozoic rocks or Precambrian basement, depending on the geologic/geographic setting, and is capped by Upper Cretaceous marine transgressive sediments. In the Hameimat Trough (concession 82 and 100) Figure (1), the Nubian lithofacies consists of sandstone and shale and is subdivided into three units currently named, in ascending order, as; Lower Nubian Sandstone, Varicoloured Shale and Upper Nubian Sandstone. The Upper and Lower Nubian Sandstones form the main hydrocarbon reservoirs in the area. The current study focuses in the Upper Nubian Sandstone.

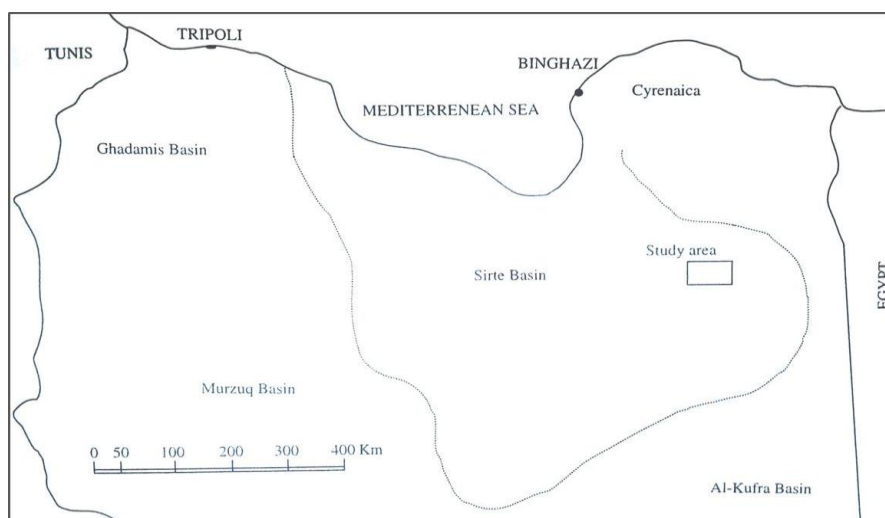


Figure 1. Location map of the study area.

The Nubian Sandstone facies has historically been interpreted as fluvial in origin, the shales are generally interpreted as lacustrine deposits (e.g. Rossi *et al.*, 1991). The Lower Nubian Sandstone comprises sandstone and shale interbeds with frequent thin beds of siltstone. The sandstone is quartzose, white to light grey, fine to medium grained. The grains

are subangular and moderate to well sorted. The thickness of the formation is variable depending on the local tectonic setting. In the depocenter it exceeds 3,000 *ft*.

The Varicoloured Shale consists of shale with frequent thin levels of fine to very fine sandstone locally grading to siltstone. The shale is varicoloured with green grey, brown and reddish. The thickness of this unit exceeds 2,200 *ft* in the depocenter.

The Upper Nubian Sandstone comprises fine to coarse grained sandstone. The grains are subrounded to subangular and moderately to well sorted. Silica and koalinite cement are present within both the Lower and Upper Nubian Sandstones.

3. Results of Fluid Inclusions Microthermometry

Fluid Inclusions Microthermometry on samples from the upper Nubian Sandstone in well A1-NC125 were used to establish the characteristics and timing of the authigenic quartz cement in the reservoir interval. The Upper Nubian Sandstones contain reasonable number of measurable fluid inclusions. The homogenization temperatures (T_h) were measured to a precision of ± 1 °C.

Microthermometric data of quartz overgrowth were collected from the Upper Nubian Sandstone from samples at depths of: 14220.2; 14512; 14545; 14665; and 14710 *ft*. The mode of occurrence of the measured fluid inclusions in the Upper Nubian Sandstone is shown in Figures 2 (A, B and C). The sandstone from which fluid inclusions were chosen is clean, pale to light grey, medium to coarse grained with mainly quartz cement. The thickness of authigenic quartz ranges from 20 to 60 μm . The sample contain two phases; liquid rich inclusions trapped along the boundary between the detrital grains and the authigenic quartz cement and; some inclusions that are present within the body of the authigenic quartz. The size of the inclusions ranges from 5 to 10 μm with a degree of fill ranging from 90 to 95% liquid phase. Solid inclusions various from highly-irregular to subspherical. UV fluorescence shows no inclusions of hydrocarbon entrapment in the studied samples.

The microthermometric data collected from the detrital grain-authigenic quartz boundary show that the minimum temperature (T_h) of authigenic quartz precipitation ranges from 115 to 128 °C. The most common temperature ranges between 118 and 120 °C Figure (3a).

Measured T_m ice shows values from -5.2° C to -2.7° C Figure (3b) indicating the pore fluid responsible for the authigenic quartz precipitation had a salinity ranging from 4.49 to 8.14 *wt%* NaCl equivalent.

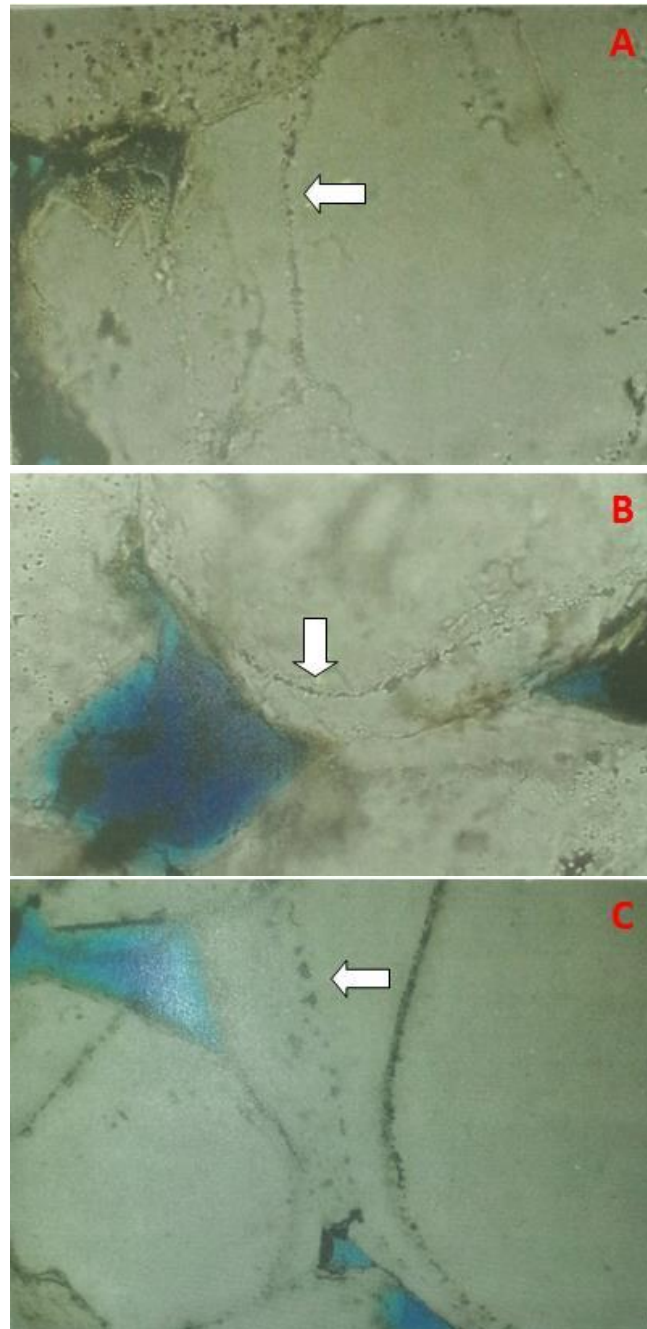


Figure 2. (A & B) Fluid inclusions present around the overgrowth-detrital grain contact in the Upper Nubian Sandstone (depth 14545, X400), and (C) Fluid inclusion present within the quartz cement of the Upper Nubian Sandstone (Depth 14334, X400).

The eutectic temperature of the inclusions ranges between -25 to -35.5 °C which broadly corresponds to the following salt system:

$H_2O-MgCl_2$ (-33.6 °C); $H_2O-NaCl-KCl$; $H_2ONaCl-MgOCl_2$ (-35 °C); and $H_2O-FeCl_2$ (-35 °C).

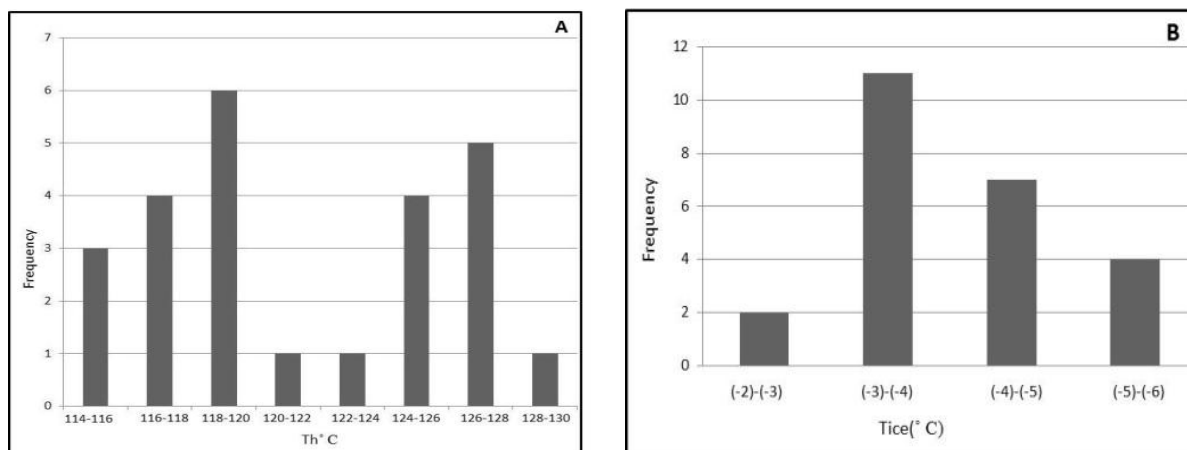


Figure 3. (A) Homogenisation temperature of the fluid inclusions present along the detrital – authigenic quartz cement in the Upper Nubian Sandstone, and (B) Tice range of the boundary inclusions in the Upper Nubian Sandstone.

Study of the fluid inclusions within the authigenic quartz cement shows them to have Th values that are generally very similar to those of fluid inclusions present along the detrital clasts-quartz overgrowth boundary. Values range between 114.5 °C with most falling between 117.5-119.6 °C as shown in Figure (4.a).

The Tm ice is very low when compared to that of the population present along detrital-quartz cement boundary. The Tm ice records values ranging between -22.2 to -14.8 °C (Figure 4.b), corresponding to a pore fluid salinity range between 18.47 to 23.8 wt.% NaCl equivalent.

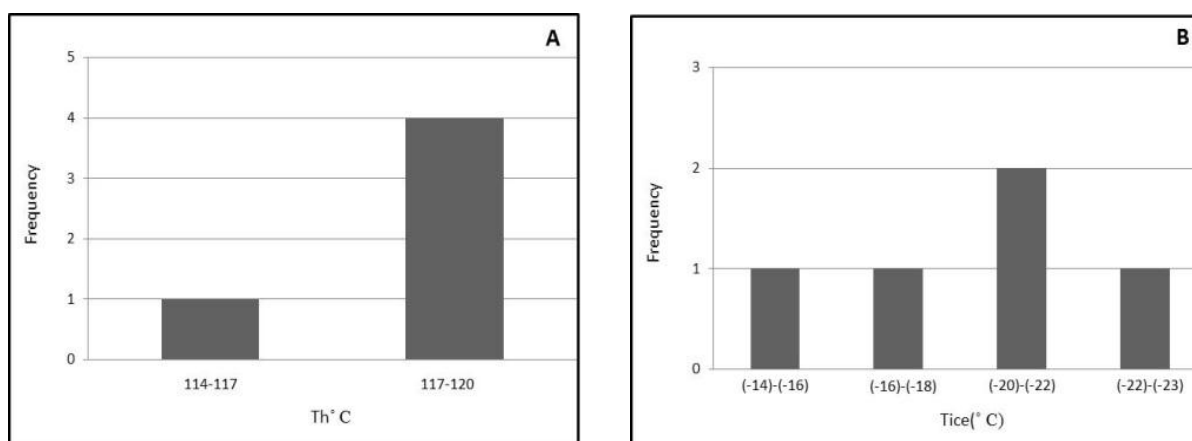


Figure 4. (A) Homogenisation temperature of the fluid inclusions present within authigenic quartz cement in the Upper Nubian Sandstone, and (B) Tice range of the fluid inclusions present within authigenic quartz in the Upper Nubian Sandstone.

The eutectic temperature ranges between -32 to -37 °C. These values give an eutectic composition generally corresponding to the following salt systems:

$H_2O-MgCl_2$ (-33.6 °C); $H_2O-NaCl-MgCl_2$ (-35 °C); $H_2O-FeCl_2$ (-35 °C); and $H_2O-MgCl_2-FeCl_2$ (-37 °C).

The trapping temperature (Tt) can be estimated from homogenization temperature (Th) by using the appropriate P-T phase diagram. Estimation of the trapping temperature was carried out using MacFlinCor computer software package (Brown, 1989) which enables the user to create a database of suitable fluid inclusion system that might be present. In this study the system is assumed to be an H_2O -mixed salts. The temperature range used started at 0 and rose up to 200 °C in 5 °C increments. The results were plotted in the P-T diagram containing both hydrostatic and lithostatic pressures calculated on the basis of the present day geothermal gradient of Upper Nubian Sandstone in well A1-NC125. The hydrostatic and lithostatic gradients were defined using a geothermal gradient of $22.784\text{ }^\circ\text{C.km}^{-1}$ in well A1-NC125. The result of the isochore calculations, however, gives trapping temperature (Tt) values above the present day reservoir temperature.

Recent studies of diagenetic fluid inclusions (Hanor, 1980; Burley *et al.*, 1989; and Haszeldine and Osborne, 1993) show that they can be very complex and may contain organic components, particularly methane. Hanor (1980) stated that the breakdown of dissolved acetate present in sedimentary waters at temperature above 80 °C will produce methane and bicarbonate as represented by the following reaction:



These studies clearly indicate that dissolved methane is usually present in pore water of sedimentary rocks, especially in organic –rich sediment or near hydrocarbon accumulations. In the Hameimat Trough both possibilities exist.

The microthermometric data of the Upper Nubian Sandstone show that the Th range in the fluid inclusions present in the detrital grain-quartz cement boundary is 115 to 128 °C giving a 13 °C difference. The difference in the Th values in the fluid inclusions present within the body of the quartz cement in the Upper Nubian Sandstone is much lower at about 5 °C. Most literature on fluid inclusions studies agrees that Th values obtained from FIA of known origin that fall within a range of 10-15 °C are consistent (e.g. Golden and Reynolds, 1994), and therefore interpretation of such data is valid. In the present instance petrographic examination of the fluid inclusions population shows that the slight difference in Th values is probably due to different growth rate (i.e. slight different size and orientation) of the authigenic quartz. It has been noted that authigenic quartz can grow at different rates in different clasts and crystal faces (James *et al.*, 1986).

The values obtained from fluid inclusions present along the dust rim of the detrital quartz grains shows that entrapment of inclusions in quartz overgrowth started at a temperature of about 115 °C. This indicate that the onset of quartz cementation in the Upper Nubian

Sandstone reservoir (in the well A1-NC125 area) took place at a relatively high temperature during significant burial depth. A plot of the homogenization temperature against salinity Figure (5) shows a slight increase of salinity with increasing Th. This trend suggests that the inclusions have not been thermally reset but rather represent a simple burial depth model where both salinity and temperature increased with increasing depth of burial. The slight increase of the Th range within the population probably results from differences in growth depth rates as larger inclusions require more time to seal (Walderhaug, 1990).

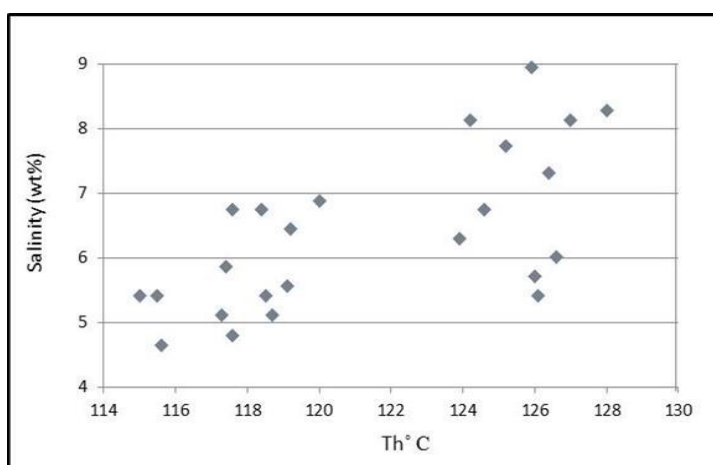


Figure 5. Relationship between salinity and homogenisation temperature of the boundary fluid inclusions in the Upper Nubian Sandstone.

The fluid inclusions present within the body of the quartz overgrowth have Th values ranges from 115.1 to 119.6 °C. Although the number of measured inclusions is relatively low the small range is valid and each fluid inclusion has been petrographically examined against the basic principles.

Graphic representation of Th values against salinity of the fluid inclusions present within the quartz cement show significant correlation. This further proves that none of these inclusions has undergone thermal re-equilibration.

The similarity, relatively speaking, between Th values of the fluid inclusions present along the detrital grain authigenic quartz cement and those which are present within the authigenic quartz, suggests that, at least in this area, quartz cementation occurred over a relatively short period of time.

Dating of quartz cement can be achieved by relating homogenization temperature, obtained from microthermometric study of primary fluid inclusions to a modeled burial and thermal history curve. The burial history curves were reconstructed for the wells A1-NC125 by using Basin-Mod software package.

The minimum homogenization temperature of the fluid inclusions presents along the boundary between the detrital grain and the quartz cement is 115 °C for the Upper Nubian Sandstone. Transferred of this value to the burial history curve gives a palaeo-burial depth of about 7431 ft (2.25 km) which correspond to a geologic time of 62 Ma BP. This implies that quartz cementation of the Upper Nubian Sandstone was initiated in Palaeocene time as shown in Figure (6).

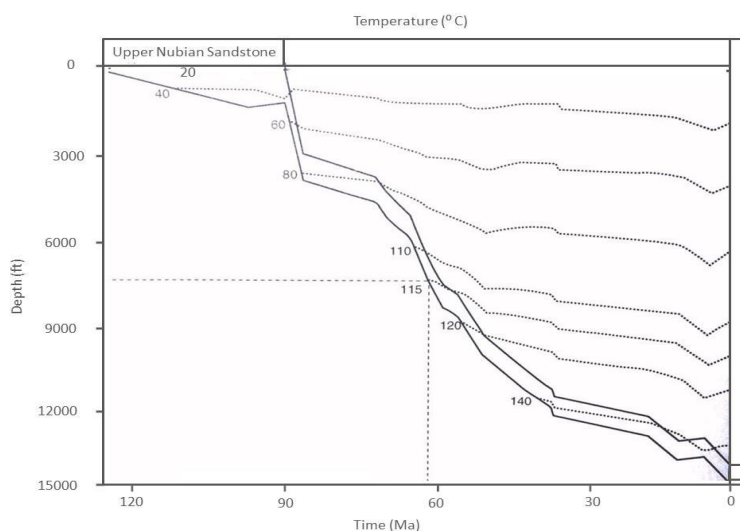


Figure 6. Burial history curve for the Upper Nubian Sandstone with estimated age for the onset of quartz cementation.

4. Conclusion

Fluid inclusions data from core samples of the Upper Nubian Sandstone indicate that the precipitation temperature of the silica cement ranges between 115 and 128 °C. The range probably reflects different growth rate since larger inclusions take longer to seal. The solubility of quartz decreases with decreasing temperature so cooling fluids will precipitate quartz cement. This being the case the pore fluid must have been hotter than 115 °C before reaching the Upper Nubian Sandstone in the area under study. Integration of precipitation temperature with the burial history curve for the well A1-NC125 indicates that onset of quartz cement was about 62 Ma BP (late Palaeocene) for the Upper Nubian Sandstone. Tice and Teu measurements indicate at the onset of quartz cementation the pore fluids from which the quartz precipitated were complex brines containing $MgCl_2$ and $FeCl_2$ as well as $NaCl$ and KCl .

References

Brown P.E. (1989). FLINCOR; a microcomputer program for the reduction and investigation of fluid-inclusion data. *American Mineralogist*, 74(11-12): 1390-1393.

- Burley S.D., Mullis J., and Matter A. (1989). Timing diagenesis in the Tartan reservoir (UK North Sea): Constraints from combined cathodoluminescence microscopy and fluid inclusion studies. *Mar. and Petrol. Geol.*, 6: 98-120.
- Goldstein R.H., and Reynolds T.J. (1994). *Systematics of fluid inclusions in diagenetic minerals: SEPM (Society for Sedimentary Geology) Short Course 31*. Tulsa Oklahoma, USA.
- Hanor J.S. (1980). Dissolved methane in sedimentary brines: potential effect on the PVT properties of fluid inclusions. *Econ. Geol.*, 75: 603-609.
- Haszeldine R.S., and Osborne M. (1993). *Fluid Inclusion Temperatures in Diagenetic Quartz Reset by Burial: Implications for Oil Field Cementation In: Diagenesis and Basin Development*. (Ed. Horbury A.D., and Robertson A.G.), *Bll. Am. Ass. Petrol. Geol.*, Tulsa, 35-46.
- James W.C., Wilmar G.C., and Davidson B.G. (1986). Role of Quartz type and grain size in silica diagenesis, Nugget Sandstone, south central Wyoming. *J. Sed. Petrol.*, 56: 657-662.
- Roedder E. (1984). *Fluid inclusions*. Ribbe P.H. (Ed.), (Vol. 12, pp. 12-45). Mineralogical Society of America, Washington DC.
- Rossi M.E., Tonna M., and Larbash M. (1991). *Latest Jurassic-Early Cretaceous Deposits in the Subsurface of the Eastern Sirte Basin (Libya): Facies and Relationships with Tectonics and Sea-Level Changes*. In: *The Geology of Libya* (Ed. By M. J. Salem and A. M. Sbeta and M. R. Bakbak), Elsevier, Amsterdam, 5: 2211-2225.
- Sorby H.C. (1858). On the microscopical, structure of crystals, indicating the origin of minerals and rocks. *Quarterly Journal of the Geological Society*, 14(1-2): 453-500.
- Walderhaug O. (1990) A fluid inclusion study of quartz-cemented sandstones from offshore mid-Norway: possible evidence for continued quartz cementation during oil emplacement. *Jur. Sed. Petrol.*, 60: 203-210.