Effect of Temperature on Stiffness of Sandstones from the Deep North Sea Basin

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Abstract

Development of high-pressure, high-temperature (HPHT) petroleum reservoirs situated at depths exceeding 5 km and in situ temperature of 170 °C increases the demand for theories and supporting experimental data capable of describing temperature effects on rock stiffness. With the intention of experimentally investigating temperature effects on stiffness properties, we investigated three sandstones from the deep North Sea Basin. As the North Sea Basin is presently undergoing substantial subsidence, we assumed that studied reservoir sandstones have never experienced higher temperature than in situ. We measured ultrasonic velocities in a low- and high-stress regime, and used mass density and stress–strain curves to derive, respectively, dynamic and static elastic moduli. We found that in both regimes, the dry sandstones stiffens with increasing testing temperature and assign expansion of minerals as a controlling mechanism. In the low-stress regime with only partial microcrack closure, we propose closure of microcracks as the stiffening mechanism. In the high-stress regime, we propose that thermal expansion of constituting minerals increases stress in grain contacts when the applied stress is high enough for conversion of thermal strain to thermal stress, thus leading to higher stiffness at in situ temperature. We then applied an extension of Biot’s effective stress equation including a non-isothermal term from thermoelastic theory and explain test results by adding boundary conditions to the equations.

Keyword Sandstones · Rock stiffness · Temperature · Effective stress

List of Symbols

α	Biot’s coefficient	σ_A	Axial stress
β	Volumetric thermal expansion coefficient	σ_R	Radial stress
η	Linear thermal expansion coefficient	P	Pore pressure
ρ_d	Dry density	T	Temperature
ρ_m	Grain density	ϵ_{Biot}	Strain resulting from Biot’s effective stress
ϕ	Porosity	ϵ_{dra}	Strain resulting from total stress
k	Permeability	ϵ_P	Strain resulting from pore pressure
σ_{Biot}	Biot’s effective stress	ϵ_T	Strain resulting from temperature
σ_{dra}	Total stress	ϵ	Strain resulting from the non-isothermal effective stress
σ_P	Pore pressure reduced by Biot’s coefficient	δ	Strain resulting in volume expansion
σ_{eff}	Non-isothermal effective stress	ζ	Potential strain
σ_H	Hydrostatic stress	Δ	Change
		Γ	$\Delta\alpha/\Delta T$
		t_P	First arrival time of P-wave
		t_S	First arrival time of S-wave
		V_P	P-wave velocity
		V_S	Shear wave velocity
		E_{sta}	Static Young’s modulus
		E_{dyn}	Dynamic Young’s modulus
		K_s	Solid bulk modulus
		K_Q	Quartz bulk modulus

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K_C	Calcite bulk modulus
K_{dra}	Drained bulk modulus
M_{dra}	Drained uniaxial compaction modulus
G	Shear modulus

1 Introduction

Development of high-pressure, high-temperature (HPHT) petroleum reservoirs as well as increasing interest in exploration for geothermal energy give relevance to properties of sedimentary rocks at elevated temperature. Consequently, experimental data that include temperature effects on intact rock samples becomes essential as direct or indirect input in estimates of subsurface behaviour. Especially for reservoirs situated in areas of the North Sea Basin with high regional overpressure and temperature above 150 °C, correct estimates of the effective stress field and elastic moduli may be crucial for safe and successful drilling operations. The effective stress is primarily a function of the overburden load and the pore pressure, where the contribution from the latter is limited by the elastic stiffness properties of the rock as expressed in Biot's coefficient. Accordingly, the effective stress field and elastic moduli of the rock are linked, and in order to provide reliable estimates, we need to understand how rock stiffness of given lithology depends on temperature and stress.

We sampled three sandstones from the Mesozoic of the deep North Sea Basin, and determined the mineralogical composition and measured mass density of the cleaned dry samples. In a dry state, we measured ultrasonic P- and S-wave velocities at temperatures from ambient to in situ (150–170 °C) under hydrostatic and triaxial stress symmetry in a low-stress regime (≤ 15 MPa). To quantify the possibility of thermal micro-crack closure, we then estimated stiffness parameters, including Biot's coefficient as a function of temperature and stress symmetry. In a triaxial high-stress regime (≥ 25 MPa) and at in situ temperature, we derived stiffness properties from both dynamic and static data to estimate effect of temperature on stiffness moduli in a stress state with closed micro-cracks. We assume that the sampled sandstones have never experienced higher overburden load and temperature than the in situ conditions because the North Sea Basin is presently undergoing significant subsidence (Kooi et al. 1991). Hence, present in situ stress and temperature conditions represent a physical boundary closely associated with the rock properties. However, stress and temperature may not affect rock properties in identical ways or to the same extent, and so we designed the testing program to investigate the effects of stress and temperature separately.

With respect to Biot's coefficient, we acknowledge that sediment burial inevitably involves increased load and

temperature and assume that the resulting elastic effects on a cemented rock frame can be quantified by a superposition of poro- and thermoelastic effects. We link the proposed consequences of the combined elastic theory to the test results and describe governing mechanisms while emphasizing the temperature and stress history of the sandstones as equally important.

2 Theory

2.1 Temperature Effects on the Effective Stress

Originally introduced by Terzaghi in the 1920s for soils, the concept of effective stress brought a theoretical formulation of the relation between total stress, pore pressure and the strain in a soil volume undergoing deformation. In the classical paper “*General theory for three-dimensional consolidation*”, Biot (1941) formulated the generalized poroelastic theory, and later Biot and Willis (1957) introduced the coefficient, α , now often denoted simply as Biot's coefficient. With the convention of positive stress for compression, Biot's effective stress equation can be formulated as follows:

$$\sigma_{\text{Biot}} = \sigma_{\text{dra}} - \sigma_P, \quad \sigma_P = \alpha P, \quad (1)$$

where σ_{dra} is the total stress, P is the pore pressure, and σ_P is pore pressure reduced by Biot's coefficient. The effective stress is thus a function of the total load (σ_{dra}) as well as the magnitude of load carried internally in the rock volume by the pore fluid, αP . The equation is valid for linear elastic, isotropic rocks, hydrostatic stress and isothermal conditions. Referring to the latter, we also denote σ_{Biot} as isothermal effective stress. As in the original paper by Biot (1941), we prefer to formulate Eq. 1 in terms of the resulting strain on a representative volume because it allows us to add strain from mechanisms with no definite coupling between strain and stress. If we follow the sign convention so that compression is positive as well as assume a linear relation between stress and deformation the resulting isothermal strain, ϵ_{Biot} , from Eq. 1 becomes:

$$\epsilon_{\text{Biot}} = (\sigma_{\text{dra}} - \alpha P) K_{\text{dra}}^{-1} \quad (2)$$

which equates to

$$\epsilon_{\text{Biot}} = \epsilon_{\text{dra}} + \epsilon_P, \quad \epsilon_P = -\alpha P K_{\text{dra}}^{-1}, \quad (3)$$

where K_{dra} is the bulk modulus of the rock frame (drained), ϵ_{dra} is the theoretical volumetric strain from changes in the total stress, and ϵ_P is the volumetric strain due to changes in pore pressure. The isothermal effective stress is thus equivalent to the strain a rock volume experiences when stressed at isothermal conditions.

Biot's coefficient is formulated as $\alpha = 1 - K_{\text{dra}}/K_s$ (Biot and Willis 1957), where K_s is the bulk modulus of the solid phase and α conceptually represents the area on which the pore pressure can counteract load from the total stress. The residual $(1 - \alpha)$ is inaccessible to the pore pressure and represents the area of cemented grain contacts formed during burial as discussed by, e.g. Alam et al. (2012). Normalized with respect to the stiffness of the solid phase, Biot's coefficient is as such independent of mineralogy but not of temperature, and temperature dependency of, respectively, K_s and K_{dra} are not necessarily equal or proportional.

Theoretical extensions to Biot's original isothermal formulation of poroelasticity accounting for thermal expansion/contraction of fluid and solid constituents have been undertaken by a series of authors setting forth governing equations (e.g. Geertsma 1957; Brownell et al. 1977; Bear and Corapcioglu 1981; Palciauskas and Domenico 1982; Noorishad et al. 1984; McTigue 1986; Zhou et al. 1998; Ghabezloo and Sulem 2009; Khalili et al. 2010; Suvorov and Selvadurai 2010; Belotserkovets and Prevost 2011; Zimmerman 2017). As it is expressed in Palciauskas and Domenico (1982), McTigue (1986) and Zimmerman (2017), we adopt the formulation of resulting strain from combined poro- and thermoelastic effects. This non-isothermal constitutive equation is an extension of Biot's original equation, and by defining contraction from thermoelastic effects as positive strain, the resulting volumetric strain, ϵ , on the representative isotropic rock volume can for hydrostatic stress be expressed as follows:

$$\epsilon = \epsilon_{\text{dra}} + \epsilon_P + \epsilon_T, \quad \epsilon_{\text{dra}} = \sigma_{\text{tot}} K_{\text{dra}}^{-1}, \quad \epsilon_P = -\alpha P K_{\text{dra}}^{-1}, \quad \epsilon_T = -\beta T, \quad (4)$$

where $\epsilon_{\text{dra}} + \epsilon_P$ is the isothermal strain from Eq. 3, ϵ_T is the thermoelastic strain due to a temperature change, T . In this equation a linear relation is assumed between temperature change and deformation controlled by the volumetric thermal expansion coefficient β of the constituting solid phase.

Temperature change is physically linked to change in pore fluid density, and depending on boundary conditions (drained or undrained) this may lead to changes in pore pressure. For a fluid-saturated rock one can imagine two conditions where temperature increase is coupled, respectively, uncoupled with increase in pore pressure: (1) in undrained conditions, due to difference in expansion of fluid and solid constituents, the temperature increase will induce pore pressure increase resulting in a coupling; (2) in drained conditions with a constant pore pressure ($\Delta P = 0$), temperature and pore pressure are uncoupled. However, constant pore pressure does not imply a constant pore pressure effect (αP), because α is coupled to temperature effects on the rock frame as well as the stiffness of the solid phase. Further, one can imagine the uncoupled case where increased pore pressure due to overpressure balances thermal fluid expansion from

temperature increase, resulting in constant fluid density and no pressure increase due to thermal expansion.

The thermal expansion coefficient of the constituting solid phase equals that of the rock frame (McTigue 1986). However, simple inversion of Eq. 4 to give strains as functions of the stresses is not straightforward. The thermal strain, ϵ_T , induced on a rock frame can physically only convert to stress when a volume constraining boundary condition is resulting in zero thermally induced deformation, e.g. that, ϵ_T is only potential and do not result in physical volume change. An un-stresses and un-constrained rock frame exposed to a positive or negative temperature change will inevitably experience a change in both pore and bulk volume. When the frame is free to expand, it will remain un-stressed, and similarly, the ratio of pore- to bulk volume change remains constant, leaving an unchanged porosity (Cheng 2016). On the other hand, a constrained rock frame exposed to a temperature increase will undergo zero volume change, but suffer a conversion of thermal strain to stress. Correspondingly, a subsequent temperature decrease will result in thermal stress release until initial temperature, at which point continued temperature decrease will result in contraction on the rock frame; however, not a decrease in porosity. For a constrained rock frame, inversion of Eq. 4, to the non-isothermal effective stress can be formulated as follows:

$$\sigma_{\text{eff}} = \sigma_{\text{dra}} - \sigma_P - \sigma_T, \quad \sigma_{\text{dra}} = K_{\text{dra}} \epsilon_{\text{dra}}, \quad \sigma_P = \alpha P, \quad \sigma_T = \beta T K_{\text{dra}}, \quad (5)$$

where σ_P and σ_T are the reduced pore pressure, and thermoelastic stress, respectively, and both are assumed to act isotropically. In order to apply Eq. 5, we must establish arguments for boundary conditions justifying thermal strain to stress conversion: presence of a constrained rock frame. Accordingly, we first rewrite Eq. 4 and express the resulting strain as follows:

$$\epsilon = K_{\text{dra}}^{-1} (\sigma_{\text{dra}} - \alpha P - \beta T K_{\text{dra}}), \quad (6)$$

where it becomes clear that for a positive resulting strain (ϵ , overall compaction), $(\sigma_{\text{dra}} - \alpha P - \beta T K_{\text{dra}})$ must also be positive, and we will use this in our establishment of arguments for conditions leading to a constrained rock frame. Second, we envisage a representative rock volume in a temperature-controlled laboratory experiment or during burial. The rock volume is simultaneously exposed to volumetric strains from pore pressure ($\alpha P K_{\text{dra}}^{-1}$) and temperature (βT). Keeping uncoupled preconditions in mind ($\Delta P = 0$) we observe from Eq. 6 that changes in $(\alpha P K_{\text{dra}}^{-1})$ from temperature is controlled by α , which by definition controls fluid added to storage per change in bulk volume at constant pore pressure. Hence, even with conditions of constant pore pressure ($\Delta P = 0$) the term cannot be ignored, because α is related to the temperature-dependent elastic modulus of the constituting mineral. Further, we observe that both pore pressure

and temperature counteract σ_{dra} (Eq. 6). Consequently, we define $\varepsilon_{\text{dra}} = K_{\text{dra}}^{-1} \sigma_{\text{dra}}$ (Eq. 5 and 6) as the strain for which a rock volume can be considered constrained, meaning that if ε_{dra} exceeds counteracting strain ($\alpha PK_{\text{dra}}^{-1} + \beta T$), the resulting strain is positive (overall compaction). Further, this describes conditions of a constrained rock frame with thermal strain to stress conversion, and we write

$$\varepsilon_{\text{dra}} \geq \alpha PK_{\text{dra}}^{-1} + \beta T \wedge \varepsilon \geq 0, \quad \text{Thermal strain/stress conversion,} \quad (7)$$

and the opposite where the rock frame is free to expand as

$$\varepsilon_{\text{dra}} < \alpha PK_{\text{dra}}^{-1} + \beta T \wedge \varepsilon < 0, \quad \text{Expansion.} \quad (8)$$

The second relation in principle describes volume expansion (negative resulting strain). It is, however, more complex as it potentially involves both volume expansion as well as thermal strain to stress conversion. In order to separate thermal expansion from thermal strain–stress conversion, we seek support in a conceptual illustration of a rock frame (Fig. 1).

We define the portion of $\alpha PK_{\text{dra}}^{-1} + \beta T = -(\varepsilon_p + \varepsilon_T)$ leading to $\varepsilon_{\text{dra}} < 0$ as $-\delta$ and the only strain physically resulting in volume expansion from a temperature increase (Fig. 1). The remaining portion of $-(\varepsilon_p + \varepsilon_T) + \delta$ we denote as $-\zeta$, resulting in $-\zeta = -(\varepsilon_p + \varepsilon_T) + \delta$ and that the sums must equal as $-(\zeta + \delta) = -(\varepsilon_p + \varepsilon_T)$. Because ζ arises as strain to stress conversion and not as physical volume change,

Fig. 1 Conceptual illustration of a rock volume exposed to strain $-(\varepsilon_p + \varepsilon_T)$ from pore pressure and temperature simultaneously, $-\delta$ is the portion of $-(\varepsilon_p + \varepsilon_T)$ resulting in expansion of the rock volume, whereas $-\zeta$ results in thermal strain to stress conversion. ε_{dra} is the theoretical strain from changes in total stress

we define it as a *potential* strain (Fig. 1). Hence, thermal strain to stress conversion can only occur when ζ is negative, implying that thermal stress (σ_T) only evolves when $-(\varepsilon_p + \varepsilon_T) > \delta$. In order to illustrate and quantify when $-(\varepsilon_p + \varepsilon_T) > \delta$ (thermal strain to stress conversion) we invert our definition of constraining strain in Eqs. 7 and 8 to terms of stresses as

$$\sigma_{\text{dra}} \geq \sigma_p + \sigma_T \wedge \sigma_{\text{eff}} \geq 0, \quad \text{Thermal strain/stress conversion,} \quad (9)$$

$$\sigma_{\text{dra}} < \sigma_p + \sigma_T \wedge \sigma_{\text{eff}} < 0, \quad \text{Expansion.} \quad (10)$$

Thus we have defined the total stress (σ_{dra}) as the constraining stress as well as the maximum applied stress at laboratory conditions or, e.g. the overburden stress in the subsurface. This gives a bound to Eq. 9, because it is impossible for the combined pore pressure and temperature to generate stresses larger than σ_{dra} , and we deduce that the non-isothermal effective stress (Eq. 9) at all times is neutral or positive ($\sigma_{\text{eff}} \geq 0$). We see that $\sigma_{\text{eff}} = 0$ may be used as the limit defining when counteracting strains ($-\varepsilon_p$ or $-\varepsilon_T$) result in thermal strain–stress conversion or volume expansion. Accordingly, to allow thermal strain to stress conversion, we include Eq. 9 in formulation of boundary conditions to Eq. 5:

$$\begin{aligned} \sigma_T > 0 \text{ when } \sigma_{\text{dra}} \geq \sigma_p + \sigma_T \wedge \sigma_{\text{eff}} > 0 \\ \sigma_{\text{eff}} = \sigma_{\text{dra}} - \sigma_p - \sigma_T, \quad \sigma_p = \alpha P, \quad \sigma_T = \beta TK_{\text{dra}}. \end{aligned} \quad (11)$$

The non-isothermal effective stress is directly influenced by the magnitude of experimental or in situ conditions (σ_{dra} , P and T) and rock properties (K_{dra} , K_s and β) (Eqs. 5 and 11), but the magnitude of rock properties may be of less significance than their respective ratios because changes due to pore pressure and temperature are not necessarily proportional. As an example, first consider the rock properties K_s and β , which combined with K_{dra} , control σ_p and σ_T and thus control the boundary condition of conversion from thermal strain to stress (Eq. 11). For a quartz dominated sandstone with K_s and β of, respectively, 37 GPa and $34.7 \cdot 10^{-6} \text{ K}^{-1}$ (Table 1) the ratio between K_s and β approximates 10^6 GPa/K . By contrast, a calcite-dominated limestone formation with K_s and β of, respectively, 74 GPa and $14.6 \cdot 10^{-6} \text{ K}^{-1}$ is represented by a ratio of around $5 \cdot 10^6 \text{ GPa/K}$ (Table 1). There is thus a factor 5 difference between quartz and calcite, and by imagining the two rocks at equal σ_{dra} and assuming equal frame stiffness (K_{dra}), the resulting effective stress (σ_{eff}) must be different, because of the different ratio.

2.2 Stiffness at Experimentally Reestablished in Situ Stress and/or Temperature

Several researchers have identified and documented factors causing frame stiffness measured in the laboratory to differ

Table 1 Bulk modulus and thermal expansion coefficient of common minerals

Mineral	Bulk modulus, $K_{\min}$, (GPa)	Linear thermal expansion coeff., η , (10^{-6} K^{-1})	Volumetric thermal expansion coeff., β , (10^{-6} K^{-1})
Quartz	37 ^a	7.7 ^{b,1} , 13.5 ^{b,2}	34.7 ^c
Calcite	72 ^a	-4.4 ^{b,1} , 23 ^{b,2}	14.6 ^c
Feldspar	75.6 ^a	1.3 ^{b,3} , 13.5 ^{b,4}	19.8 ^c
Clay/shale	25 ^a		31 ^d

^aCitation in Mavko et al. (2009), ^bJohnson and Parsons (1944), ^cDerived as the sum of linear thermal expansion coeff., ^dEstimate based on work by McKinstry (1965), ¹Parallel to c-axis, ²Perpendicular to c-axis, ³Parallel to b-axis, ⁴Parallel to a-axis

from in situ stiffness: (1) stress-closure of micro-cracks and changes in pore pressure/effective stress (e.g. Banthia et al. 1965; King 1966; Nur and Simmons 1969; Todd and Simmons 1972; Christensen and Wang 1985; Wu et al. 1991; Prasad and Manghnani 1997; Siggins and Dewhurst 2003; Frempong et al. 2007; Ma 2019), (2) frequency dependence on elastic wave velocities (e.g. Biot 1956a, b; Jones and Nur 1983; Winkler 1986; Batzle et al. 2006; Mavko and Vanorio 2010), (3) effects from saturating fluid (e.g. Hadizadeh and Law 1991; Dyke and Dobereiner 1991; Wu et al. 1991; Zang et al. 1996; Pimienta et al. 2017; Heap et al. 2019). Hence, a general understanding of factors influencing rock stiffness at ambient temperature exists.

By contrast, published studies on rock stiffness at elevated temperature are few in number. Publications on experimentally derived mechanical properties of rocks as a function of temperature are written in the context of (1) petroleum and geothermal industries, (2) nuclear waste deposits, (3) fire damaged buildings and monuments, and (4) volcanology. Experiments with application in petroleum technology, geothermal energy and nuclear waste disposal typically focus on temperatures below 300 °C (e.g. Hughes and Cross 1951; Hughes and Kelly 1952; Handin and Hager 1958; Mobarak and Somerton 1971; Timur 1977; Jones and Nur 1983; Rao et al. 2007; Hassanzadegan et al. 2011), whereas studies related to fire damage and volcanology include temperatures as high as 1200 °C (e.g. Hajpál 2002; Mao et al. 2009; Ranjith et al. 2012; Zhang et al. 2009; Wu et al. 2013); however, the latter typically concern igneous rocks (e.g. Smith et al. 2009; Bakker et al. 2015, 2016; Schaefer et al. 2015; Heap et al. 2018). Experimental rock properties for application within petroleum, geothermal industries and nuclear waste disposal fall into one category, typically derived from samples “tested at” a given temperature, whereas with respect to fire damage and volcanology experimental data fall in another category where samples have undergone “heat treatment” because of inherent challenges of data collections at temperatures above 300 °C. Independent of application and testing category, rock properties are typically evaluated with reference to ambient temperature. However, ambient temperature is as foreign to the rock as temperature above

in situ, so for applications in the petroleum and geothermal industry, choice of temperature is central for proper evaluation of properties.

One aspect is the temperature dependence of mineral properties. Ohno (1995) and Lakshtanov et al. (2007) derived quartz mineral bulk modulus (K_Q) from measurements of stiffness as function of crystallographic orientation of a quartz crystal (quartz typically dominates in sandstones) as function of temperature, and their results show a distinct softening with increasing temperature especially around the transition from α - to β -quartz at approximately 570 °C. For application to sandstones within the petroleum and geothermal industry and thus temperatures below 300 °C only α -quartz is a concern. Results by Ohno (1995) and Lakshtanov et al. (2007) indicate a decrease in K_Q in the order of 2% per 100 K temperature increase with a trend closely approximated by a second-order polynomial (Fig. 2). In the same temperature range, they found that the shear modulus decreases in the order of 0.3% per 100 K with an approximately linear trend.

Another aspect is fracturing of rocks. Sampling of subsurface rock material inevitably involves equilibration to

Fig. 2 Average bulk modulus of calcite and quartz minerals versus temperature up to 300 °C. Calcite data are from Dandekar and Ruoff (1968). Quartz data are from Ohno (1995) and Lakshtanov et al. (2007) with circles and squares, respectively. The temperature trends are fitted with second-order polynomial

atmospheric conditions. Grains strained due to overburden and temperature would equilibrate to conditions prior to burial, whereas the contact cement bridging grains would also equilibrate to surface conditions (Holt 1994), but this equilibrium differs from the stress and temperature conditions where the cement precipitated. This diverse equilibrium would induce tensional forces leading to ruptures of the weakest contact cement, thereby causing a reduction in rock stiffness (Holt 1994). Ruptures of contact cement are in the rock physical society denoted as micro-cracks. They include ruptures due to stress release and ruptures due to cooling. These ruptures are different in physical principle and presumably also in order of magnitude. Ruptures originating from cooling would be homogeneously distributed but related to the mineralogical composition, whereas ruptures from stress release may have distinct orientation caused by anisotropy in the subsurface stress field at the time of contact cementation. The term “micro-crack” indicates that ruptures must extend at least one grain-contact and be limited in extension such that a rock volume still sticks together. Because reestablishing contact cement requires a chemical process, ruptures may close but not heal during the reestablishment of stress and temperature in the laboratory, so sampled rock material will rarely be fully representative of in situ rock.

Separating temperature effects on rock properties from other known effects (e.g. stress level, stress symmetry, saturation degree, fluid type) adds complexity to the experimental program. In sequential publications by Handin and Hager (1957, 1958) and Handin et al. (1963), the authors systematically added degrees of freedom (temperature and single fluid) to the experimental program. Experimental quantification of rock properties such as stiffness and strength requires a minimum of three samples with identical petrophysical properties, and the simplest test program hence requires approx. ten individually tested samples. As the number of required samples increases, and core samples are sparse, outcrop rocks can be the only option for testing material. Difficulties arise when outcrop rocks are assumed analogue to in situ reservoir without taking the geological temperature history of the outcrop rock into account. If exposed to temperature above or below maximal burial, characteristics and properties of the strained rock frame may lead to misguided use of temperature trends in evaluation of in situ reservoir properties.

To address the effect of in situ temperature on the rock frame, we measured static and dynamic elastic moduli as function of temperature on dry sandstones from the North Sea Basin. By not including fluid saturated rocks we acknowledge that some probably significant effects were not studied. In order to ensure that no mineral transformations take place, the maximum applied testing temperature is equal to the in situ. Anticipated mechanisms controlling

changes in rock stiffness with temperature include the following:

- Mechanism 1: in line with Tamuzs et al. (1994), Balme et al. (2004) and Fertig and Nickerson (2016) consider a rock sample with a constant applied total stress and pore pressure and with effective stress below the stress leading to full micro-crack closure. Restitution of temperature to previous equilibrium causes closure of contact cement ruptures (i.e. of micro-cracks) by thermal expansion of the constituting rock minerals, hence leading to stiffening of the rock frame. Because the constitutive formulations in Eq. 11 assume a homogeneous and un-fractured rock volume, it is incapable of describing this mechanism. Further, at a constant temperature, increase in applied stress may enlarge contact area in ruptures possibly created by thermal shrinkage and continued stress increase will add to the number of contacts or increased contact area of existing contacts, thereby leading to stiffening.
- Mechanism 2: consider the same rock sample, but with constant applied total stress and pore pressure equivalent to full micro-crack closure and with the resulting strain $\varepsilon_{\text{Biot}}$ in accordance with Eq. 2. Restitution of in situ temperature will inevitably cause thermal expansion of the rock minerals, and the resulting strain may be in accordance with conversion of Eq. 11 if applied stress and pore pressure respect the boundary conditions of a constrained volume/rock frame ($\sigma_{\text{dra}} \geq \sigma_P + \sigma_T$). Assuming so, the rock frame might behave stiffer at in situ temperature, depending on the proportion between the petrophysical properties, K_S and β . Physically, the cause of stiffening is the conversion of the thermally induced strain to thermal stresses internally imposed on the rock frame. Thus, stiffening of the rock frame from thermal stress occurs only when $\sigma_{\text{dra}} \geq \sigma_P + \sigma_T$.

3 Sandstone Samples

Sandstone cores experimentally investigated in this study originate from three North Sea HPHT wells denoted as, respectively, O, H and C. The sandstones originate from depths of 4.5 to 5.5 km and in situ temperatures of approximately 150 °C, 165 °C and 170 °C for, respectively, O, H and C.

Eight vertical plugs from each core were drilled within a depth interval of 2–3 m with the aim of achieving maximum sample similarity. Each plug has an approximate diameter of 37.6 mm. End surfaces were trimmed so that the plugs obtained a length approximately twice the diameter, paralleled and polished within 0.05 mm. All dimensions were measured within 0.05 mm. Samples were methanol and toluene cleaned by the soxhlet method (Dean 1998) and

subsequently, oven-dried (60 °C) before being equilibrated to derive dry density from measurement of dry mass and sample dimensions as well as gas-porosity by N_2 expansion. Each plug was placed in a core holder designed for flow-through experiments and equipped with up- and downstream discharge and pressure transducers. At confining stress of 2.75 MPa, the gas-permeability was derived from flow of N_2 at steady state of minimum three differential up- and down-stream pressure steps and Darcy's equation corrected to account for gas compressibility by the ideal gas law (Dullien 1979). We estimated liquid permeability, using Klinkenberg correction. In order to identify significant fractures prior to applying mechanical stress, all samples were scanned by a medical CT-scanner (Fig. 3). The resolution of collected CT-scans does not allow for detailed analysis of mineralogy (Fig. 3). Soxhlet cleaned side-trims were used for determining mineralogy by X-Ray Diffraction (XRD). Backscatter Electron Micrographs (BSEM) were recorded from polished thin sections, and the identification of solid phases corroborated by Energy Dispersive X-ray Spectroscopy (EDS). Additionally, sidetrims were used for

measurement of the specific surface by N_2 adsorption (BET-method, Brunauer et al. 1938). After mechanical testing to failure, the carbonate content was determined from core plug pieces by HCl dissolution and NaOH titration.

With the exception of sample O2, CT-scans of samples from O show no consistent layering, and on sample scale, the sandstone appears undamaged from downhole sample extraction, but with presence of both less and more dense rock material distributed in clusters and thin streaks (Fig. 3). Sample O2 has a distinct layering of less dense material, and sample O4 and O8 appear denser compared to the remaining samples (Fig. 3). CT-scans of samples from H show the samples to be undamaged (Fig. 3). On the sample scale, the samples appear to have a high degree of homogeneity; however, sample H1 and H5 show local discrepancies by the presence of more dense material. Samples from well C have an overall homogeneity on the sample scale, although clusters of more dense material are seen in C2, C5 and C7, and a faint layering can be noticed in C3, C5, C6 and C7 (Fig. 3).

Fig. 3 CT-scans of sandstone plugs. 1st row shows samples from well O. 2nd row shows samples from well H and 3rd row shows samples from well C. Typical plug size is 37.6 mm and 76 mm in diameter and height, respectively

3.1 Mineralogical Composition and Sandstone Classification

The mineralogical composition of investigated side-trims from O, and H as derived from X-ray diffraction (XRD) and Energy Dispersive X-ray Spectroscopy (EDS) analysis shows presence of quartz, feldspar and calcite (Table 2). XRD and EDS detected no calcite in samples from C (Table 2), but the chemical analysis indicates carbonate (Table 3). Phyllosilicates were detected in all samples and include mica,

(illite) and chlorite. Pyrite and organic matter were found in samples from O (Table 2).

For samples from well O, BSEM-images show quartz and calcite as dominating minerals; however, the quantity ratio varies and separates the sample material in two sub-groups (Fig. 4). This is in accordance with carbonate content ranging from 5 to 37% (Table 3). Figure 4a shows an approximately equal quantity of quartz and calcite, whereas Fig. 4c shows a clear quartz dominance. The quantity of pore space appears inversely correlated to the quantity of calcite

Table 2 Mineralogical composition as detected by XRD and EDS

Well	Quartz	Kali-feldspar	Calcite	Phyllosilicate	Pyrite	Organic matter (coal)
O	X	X	X	X	X	X
H	X	X	X	X		
C	X	X		X		

X indicates detected presence

Table 3 Properties of sandstone samples

Sample ID	Well	Length, <i>l</i>	Diameter, <i>d</i>	Ini. Dry density, ρ_d	Grain density, ρ_m	N ₂ Porosity, ϕ	Klink. ^a permeability, <i>k</i>	Carbonate content (CaCO ₃)
		mm	mm	g/cm ³	g/cm ³	–	mD	%
O1	O	75.99	37.64	2.39	2.79	0.14	0.04	15
O2	O	75.70	37.62	2.47	2.85	0.13	<0.01	5.0
O3	O	76.13	37.70	2.48	2.79	0.11	0.01	13
O4	O	75.69	37.69	2.63	2.73	0.03	<0.01	34
O5	O	59.87	37.69	2.43	2.76	0.12	<0.01	12
O6	O	75.95	37.60	2.41	2.78	0.13	0.12	6.2
O7	O	75.37	37.69	2.62	2.76	0.05	<0.01	37
O8	O	75.75	37.73	2.56	2.73	0.06	0.01	25
H1	H	75.84	37.57	2.38	2.65	0.11	0.11	4.2
H2	H	72.21	37.68	2.33	2.65	0.13	0.13	1.7
H3	H	76.93	37.70	2.32	2.64	0.13	0.13	1.7
H4	H	75.72	37.73	2.36	2.64	0.11	0.11	2.5
H5	H	75.73	37.68	2.35	2.65	0.12	0.14	1.2
H6	H	73.83	37.57	2.33	2.65	0.13	0.18	1.1
H7	H	75.91	37.63	2.32	2.66	0.14	0.22	1.7
H8	H	70.60	37.56	2.34	2.64	0.13	0.21	2.3
C1	C	75.96	37.50	2.22	2.66	0.20	44.65	0.8
C2	C	75.52	37.56	2.14	2.68	0.20	20.27	0.6
C3	C	75.57	37.54	2.15	2.67	0.19	5.91	1.3
C4	C	75.71	37.55	2.13	2.66	0.20	37.69	1.7
C5	C	75.48	37.48	2.17	2.67	0.19	1.78	0.7
C6	C	68.37	37.55	2.10	2.68	0.22	5.26	0.9
C7	C	75.06	37.54	2.13	2.67	0.21	15.00	1.1
C8	C	76.02	37.54	2.01	2.66	0.21	104.60	1.4

^aKlinkenberg permeability

Fig. 4 BSEM-images of sandstone from well O, representing side-trim material from plugs. *Q* quartz, *C* calcite, *K* feldspar, *Ph* phyllosilicate, *P* pyrite, *PO* pyrite in organic matter. **b** Is high-resolution version of **a**

(Fig. 4a, c). Extensive cementation contacts between quartz and calcite are seen. Feldspar, phyllosilicates and pyrite are found as clusters, and the latter is additionally found in coal (Fig. 4b, d). O-samples is classified as a tight sandstone, with varying degree of quartz and calcite cementation.

For samples from well H, BSEM-images show quartz as dominating and cementing mineral (Fig. 5a, b). Cementation is extensive but less so than in samples from well O. There are single calcite and feldspar crystals along with clusters of phyllosilicate in the pore space (Fig. 5a, b). H-samples are classified as a quartz sandstone.

For samples from well C, BSEM-images show quartz as dominating mineral, but with a significant quantity of feldspar (Fig. 6). Both quartz and feldspar cementation are found, but the degree of cementation is less than in samples from O and H. Bands of phyllosilicate are found

Fig. 5 BSEM-images of side-trim material from H. *Q* quartz, *C* calcite, *K* feldspar, *Ph* phyllosilicate

Fig. 6 BSEM-images of side-trim material from C. *Q* quartz, *K* feldspar, *Ph* phyllosilicate. Figure **b** and **d** are high resolution versions of, respectively, **a** and **c**

corresponding to the layering seen on CT-scans (Figs. 3 and 6c, d). C-samples are classified as a quartz sandstone with a significant presence of feldspar and phyllosilicate bearing stylolites.

3.2 Physical Parameters

Table 3 lists physical properties measured on tested core samples. For O-samples, grain density above 2.7 g/cm^3 corresponds to presence of calcite and pyrite found in accordance with XRD and EDS analysis. Porosity falls into two groups around 0.05 and 0.12 corresponding to observations in BSEM-images (Fig. 4). Klinkenberg permeability is in general below 0.02 mD. The specific surface of O samples is around $6.5 \text{ m}^2/\text{g}$ presumably caused by the presence of highly porous organic matter and does consequently not represent the specific surface of the minerals.

For H-samples and in accordance with XRD and EDS analysis, grain density close to 2.65 g/cm^3 corresponds to dominance of quartz and a low quantity of carbonate (Table 3). In general, measured porosity, Klinkenberg permeability and the specific surface are, respectively, around 0.13, 0.13 mD and $0.9 \text{ m}^2/\text{g}$.

For C-samples, not only grain density close to 2.67 g/cm^3 corresponds well to the dominance of quartz minerals in accordance with XRD and EDS analysis (Table 3), but also a significant presence of slightly heavier presumably phyllosilicates as the carbonate content is relatively low and undetected by XRD. In general, measured porosity is around 0.2. Klinkenberg permeability ranges from 0.3 to 105 mD

and apparently is related to the extent and number of stylolites. The specific surface measured is around $1.7 \text{ m}^2/\text{g}$.

4 Rock Mechanical Procedures

The four samples with index 1–4 from each well (Fig. 3; Table 3) were in the dry state placed in a triaxial testing cell designed with temperature control. At each step of ambient temperature, 50 °C, 100 °C, 150 °C and in situ (≈ 170 °C), samples were subjected to cycles of stress; first, under hydrostatic stress from 2 to 15 MPa and back, then at constant confining stress of 2–15 MPa axial stress and back. We denoted this sequence *branch 1* (Fig. 7). To quantify influence on the frame stiffness from repeated loading and unloading alone, one selected sample from each well were at ambient temperature repeatedly loaded and unloaded with the same stress path as branch 1. Subsequently to branch 1 and hence at in situ temperature, samples with index 1–4 were hydrostatically loaded to, respectively, 0.7, 3, 10 or 20 MPa where the confining stress was held constant and followed by increased axial stress until mechanical failure. We denoted this sequence *branch 2* (Fig. 7). For reference, four additional samples with index 5–8 from each well (Fig. 3; Table 3) were tested at ambient temperature with a stress path identical to the stress path branch 1 and 2 (Fig. 7). Three linear variable differential transformers (LVDT) were calibrated to account for system self-deflection and temperature and then continuously measured axial deformation during all tests. The test setup does not allow for measurement of radial strain. Stress ramps were controlled by a deformation rate of 0.15% axial strain/h. At both ambient and in situ

temperature, combined data from stress and strain recordings from branch 2 were used to determine static Young's modulus (E_{sta}) from a running least squares best fit with a length of 5 MPa.

Piezoelectric transducers placed inside upper and lower piston heads were used to generate and receive an ultrasonic compressional wave (V_p) and two perpendicular shear waves (V_{S1} and V_{S2}) in the axial direction of the sample. Throughout testing, all wave signals were continuously recorded and first arrival times t_p and t_s , respectively, detected as well as corrected for system delay and temperature effects on the system delay within an accuracy of 0.1 μs . Central signal frequencies of ≈ 0.2 MHz and ≈ 0.5 MHz of V_p and V_s , respectively, were derived from the frequency spectrum using Fast Fourier Transformation. Wavelengths of V_p and V_s range from 5 to 9 mm and 8 to 12 mm, respectively, and are hence minimum 20 times greater than estimated average pore and grain size (Figs. 4, 5, 6).

The drained bulk modulus (K_{dra}) is determined from compressional and shear modulus in the dry state as $K_{\text{dra}} = K_{\text{dry}} = M_{\text{dry}} - 4/3G$ where $M_{\text{dry}} = \rho_{\text{dry}} V_{p,\text{dry}}^2$ and $G = \rho_{\text{dry}} V_{s,\text{dry}}^2$ are compressional, and shear moduli and ρ_{dry} , $V_{p,\text{dry}}$ and $V_{s,\text{dry}}$ are the dry density, compressional and shear wave velocity, respectively.

In order to determine if trendlines (slopes) of derived stiffness parameters (M_{dry} , G and K_{dra}) versus temperature are statistically different from zero thus indicating a correlation, we used Analysis Of Variance (ANOVA) to test the null hypothesis for linear regression with a confidence level of 0.05.

5 Results

5.1 Experimental Results of V_p and V_s at Temperature from Ambient to in Situ

For all studied sandstones, ultrasonic velocities, V_p and V_s , increase with stress and temperature irrespective of stress symmetry, but with distinct V_s – V_p ratios, so that for axial stress above 5 MPa, V_s is relatively high for hydrostatic stress symmetry as compared to triaxial stress symmetry (σ_s -trendline, Fig. 8 and Appendix A, Fig. 21); although for samples from well O, the effect of stress symmetry is undetectable (Fig. 8 and Appendix A, Fig. 21).

Independent of axial stress and temperature, V_s – V_p data from well O plot close to the trendline of a tight sandstone in a dry state defined by Jizba (1991) (cited in Mavko et al. 2009). Sample O4 has relatively high V_p and V_s corresponding to the highest carbonate content and high density as illustrated by CT-scans (Table 3; Fig. 3). When temperature reaches 150 °C, a distinct and immediate increase in V_p is

Fig. 7 Conceptual sketch of the stress path used for testing of sandstone samples as a function of temperature. Branch 1 consists of multiple looping with alternating hydrostatic and triaxial stress symmetry at increasing temperature levels. Branch 2 involves increasing axial stress towards mechanical failure at in situ temperature of 150 °C, 165 °C and 170 °C for sandstone samples from, respectively, O, H and C wells

Fig. 8 Cross plot of ultrasonic velocity data for the sandstones O, H and C measured at hydrostatic stress from 2.5 to 15 MPa and temperature ranging from ambient to in-situ. Thick ticks indicate corresponding stress or temperature for O- and C-samples shifted to increase readability. The dashed line in V_p – V_s cross plot shows the

trendline of a tight sandstone in the dry state (citation in Mavko et al. 2009), as well as the quartz line, defined after quartz values in Mavko et al. (2009). The solid lines (σ_s) show the trendlines of data obtained for well H and C under triaxial stress symmetry with radial stress of 2 MPa (Appendix A, Fig. 21)

observed. The increase is most pronounced at triaxial stress symmetry (Fig. 8 and Appendix A, Fig. 21).

Samples from well H and C have higher ultrasonic velocities for higher axial stress for both investigated stress symmetries (Fig. 8 and Appendix A, Fig. 21). For hydrostatic stress symmetry and independent of stress level and temperature, V_s – V_p is close to the quartz trendline (Fig. 8). At triaxial stress symmetry, V_s is relatively low, so the corresponding trendline (σ_s) plots with a shallower slope (Fig. 8 and Appendix A, Fig. 21).

5.2 Results of Elastic Moduli Derived from Reference Tests at Ambient Temperature

With respect to elastic moduli of the tested dry sandstone samples derived at ambient temperature (reference testing), the compressional (P-wave) modulus (M_{dra}) shows no significant dependence on stress symmetry (Fig. 9). A stiffness increase as reflected in M_{dra} and G , of 40–90% is seen when axial stress is increased from 2.5 to 15 MPa. At hydrostatic stress symmetry, a change in moduli is seen between the first

Fig. 9 Elastic moduli, M_{dra} and G of dry sandstone from wells O, H, and C as calculated from ultrasonic velocity and density data sampled at ambient temperature. Left column shows data from hydrostatic stress symmetry and the right triaxial stress symmetry with radial stress of 2 MPa. The stress path is repeated subsequently four or five times and “Cycle number” refers to the count. Data points assumed to be affected by bedding are disregarded in trendlines

and second cycle, whereas no significant change in moduli as function of cycle number is seen for triaxial test symmetry (Fig. 9). The observed initial change in moduli at hydrostatic stress is on the very first loading ascribed to the initial alignment of the sample, piston heads, connections pieces to load frame etc. These effects are denoted as bedding effects. The following load cycles do not show a significant effect on the stiffness properties (Fig. 9), also confirmed by ANOVA analysis.

5.3 Results of Elastic Moduli Derived at Temperatures from Ambient to in Situ

Presumably related to inhomogeneity, elastic moduli derived for O-samples show significant variation between samples (Fig. 3 and Appendix B, Figs. 22, 23). In general, the

compressional modulus of O-samples increases with temperature and does not significantly differ between hydrostatic and triaxial stress symmetry (Figs. 10, 11). Shear modulus for O-samples also increases with temperature but is 4% lower for the triaxial stress symmetry as compared to hydrostatic stress (Figs. 10, 11). For O-samples the resulting K_{dra} is consequently 8% higher for triaxial stress symmetry, and increases with temperature for low stress, whereas it is temperature independent or even decreases with temperature for higher stress levels (Figs. 10, 11).

For samples from well H, M_{dra} show independence of temperature for the higher levels of hydrostatic stress, whereas it is 1–2% smaller and tends to decrease with temperature at triaxial stress symmetry (Figs. 10, 11). The derived shear modulus is stable or tends to increase with temperature for both stress symmetries, however, is around

Fig. 10 Elastic moduli M_{dra} , G and K_{dra} as function of temperature at hydrostatic stress symmetry for one selected sample of well O, H and C. M_{dra} and G are calculated from dry density and ultrasonic veloci-

ties. Data points assumed to be affected by bedding are disregarded in trendlines. Corresponding data from the remaining samples are found in Appendix B

4% lower for triaxial stress symmetry (Figs. 10, 11). Consequently, K_{dra} is 8% higher for triaxial stress symmetry and tends to vary with temperature, especially at triaxial stress symmetry (Figs. 10, 11). Although the H-samples appear homogeneous from CT-scans (Fig. 3), both stress symmetries show that sample H3 and H4 are approximately 5% stiffer than samples H1 and H2 (Appendix B, Figs. 24, 25).

For sandstones from well C, all three moduli increase with temperature irrespective of stress symmetry (Figs. 10, 11). For M_{dra} no significant effect of stress symmetry is noted, whereas G is 10% lower at triaxial stress symmetry (Figs. 10, 11). Layering observed from CT-scan in sample C3 apparently has no or small effect in derived moduli, as moduli across samples are practically identical (Appendix B, Figs. 26, 27). Assuming homogeneity, K_{dra} is 12% higher

for triaxial stress symmetry than for hydrostatic stress symmetry (Figs. 10, 11).

In order to generalize the observations on effect of stress and temperature on elastic moduli, the slope of modulus versus temperature was plotted versus axial stress for each stress symmetry (Fig. 12). For O-samples data are scattered between samples and stress levels but in general show no change in K_{dra} moduli with temperature (Fig. 12, 1st row). We find a marked moduli increase from 100 to 150 °C which causes trendlines to have slopes different from zero (Fig. 10), whereas the corresponding ANOVA analysis shows no general and significant rate of modulus change as function of temperature. For H-samples, rate of change in stiffness with temperature are seen for M_{dra} and G at all stress levels, however, for M_{dra} predominantly for the triaxial stress symmetry (Fig. 12, 2nd row). For K_{dra} the resulting general tendency

Fig. 11 Elastic moduli M_{dra} , G and K_{dra} as function of temperature at triaxial stress symmetry for one selected sample of well O, H and C. M_{dra} and G are calculated from dry density and ultrasonic velocities. Data from the remaining samples are found in Appendix B

is zero change in rate with temperature (Fig. 12, 2nd row). For samples from well C, a change in rate of stiffness change with temperature is seen for M_{dra} and G at all stress levels and both stress symmetries. For K_{dra} changes are smaller and in several cases, approach zero (Fig. 12, 3rd row).

Biot's coefficient can be derived from K_{dra} , by assuming quartz as constituting mineral and a temperature corrected formulation of the mineral bulk modulus (Fig. 2). In general, all sandstones show decreasing Biot's coefficient with both increasing temperature and increasing stress (Figs. 13, 14). For H- and C-sandstone, Biot's coefficient ranges from 0.45 to 0.7 and 0.65 to 0.90, respectively, but generally with lower values for triaxial stress symmetry. Biot's coefficient corresponds to the observed cementation degree from BSEM images (Fig. 5, 6) where higher cementation is found in H-sandstone as compared to C-sandstone. For

sample O1, O2 and O3, Biot's coefficient are close to that of C-sandstone, whereas for sample O4 it is close to that of the H-sandstone. According to BSEM images, samples from well O are well cemented, not corresponding to the relatively high Biot's coefficient (Figs. 4, 13, 14). Presence of load-bearing soft organic material is presumably the cause.

Statistical analysis of the temperature effect on Biot's coefficient ($\Gamma = \Delta\alpha/\Delta T$) indicates that variation in temperature is close to insignificant for O-sandstone because values different from zero are scattered (Fig. 15). For H-sandstone, Γ values are significantly different from zero at the highest and lowest stress levels but are scattered between samples and stress symmetries (Fig. 15). For C-sandstone, Γ is centred around 0 or $-2 \cdot 10^{-5} \text{ K}^{-1}$, with slightly decreasing trend for increased stress (Fig. 15).

Fig. 12 Rate of change in elastic moduli, M_{dra} , G and K_{dra} for dry sandstones relative to change function of temperature versus axial stress level. Thick ticks indicate corresponding stress for O- and C-samples to increase the readability of the difference between stress

symmetries. Stress dependency of slopes of modulus versus temperature (Appendix B, Figs. 22, 23, 24, 25, 26, 27) that are not significantly different from zero as determined from ANOVA analysis are plotted as zero

5.4 Experimental Results of Ultrasonic Velocities and Elastic Moduli for Ambient and in Situ Temperature and Stress Conditions Approaching Rock Failure

For failure testing data (Branch 2, Fig. 7) ultrasonic velocities were selected at axial stress intervals below sample failure. For samples from well O higher velocities were found at ambient temperature than at in situ temperature for confining stress (σ_R) of 0.7, 3 and 10 MPa and oppositely for σ_R of 20 MPa (Fig. 16). For H- and C-sandstone, velocities are higher at in situ temperature for σ_R of 3, 10 and 20 MPa and practically identical for 0.7 MPa (Figs. 17, 18). Data

from O-sandstone plot close to the trendline of a tight sandstone in a dry state defined by Jizba (1991) (cited in Mavko et al. 2009) whereas data for H and C in general have lower V_S/V_P . For increasing axial stress, all samples show increasing velocities followed by a decrease (Figs. 16, 17, 18).

For samples from well O, stress–strain curves collected as part of Branch 2 at both ambient and in situ temperature in general show a linear deformation behaviour (brittle behaviour) before failure at peak stress. This applies to all confining stress levels (σ_R) (Fig. 19). The distinct layers of soft organic matter found in sample O2 tested at in situ temperature and confining stress of 3 MPa result in a significant axial strain more considerable than expected for

Fig. 13 Biot's coefficient as function of temperature at hydrostatic stress symmetry. Values are derived from K_{dra} by assuming a quartz-bearing rock frame, taking the temperature dependence of bulk

modulus for quartz into account (Fig. 2). Data points assumed to be affected by bedding are disregarded in trendlines

the corresponding level of confining stress (Figs. 3, 19). For samples from H and C, smoothly curving deformation curves indicate ductile behaviour before failure at confining stress of 10 and 20 MPa, whereas for 0.7 and 3 MPa the deformation behaviour is closer to brittle (Fig. 20). We do not find a clear difference in deformation behaviour (ductile versus brittle) for samples tested at ambient, respectively, in situ temperature (Figs. 19, 20).

With respect to static Young's modulus (E_{sta}) for O-sandstones, we find that it is lower at in situ temperature than at ambient temperature for confining stress of 3 and 10 MPa. Further, E_{sta} is practically similar for the two temperatures for confining stress of 0.7 MPa, whereas it is higher at in situ temperature than at ambient temperature for confining stress of 20 MPa (Fig. 19).

Fig. 14 Biot's coefficient as a function of temperature and triaxial stress symmetry with radial stress of 2 MPa. Values are derived from K_{dra} by assuming a quartz-dominated rock frame, where quartz bulk modulus depends on temperature (Fig. 2)

For samples from well H we find that E_{sta} is slightly higher at ambient temperature and confining stress of 0.7 MPa and 3 MPa, whereas it is higher at in situ temperature than at ambient temperature for confining stress of 10 and 20 MPa (Fig. 20). For samples from well C we find that E_{sta} is higher at in situ temperature than at ambient temperature for all samples (Fig. 20). For samples from all of the three wells, E_{sta} increases with increasing confining

stress, and also the difference between E_{sta} at ambient and in situ temperature increases (Figs. 19, 20). For each sample E_{sta} shows increase followed by decrease for increasing axial stress (Figs. 19, 20).

With respect to confining stress level, dynamic Young's modulus (E_{dyn}) derived from density, and ultrasonic velocities show trends similar to E_{sta} . Both E_{dyn} and E_{sta} initially increase with axial stress followed by a decrease and

Fig. 15 Rate of change in Biot's coefficient with change in temperature versus axial stress level for sandstone samples

absolute minimum at the maximum stress (Figs. 19, 20). The point of maximum E_{dyn} on each curve defines a condition where the difference in E_{dyn} for ambient and in situ temperature increases with increasing axial strain (Figs. 19, 20). At initial axial stress levels, the ratio of E_{sta} to E_{dyn} is 2–3, increasing to 10 toward the failure stress (Figs. 19, 20).

6 Discussion

6.1 Temperature Dependence of Frame Stiffness

Tested samples from well H show only negligible variation in petrophysical properties, and samples are hence practically identical (Table 3, Fig. 3). An identical conclusion can be made for well C. In contrast, petrophysical properties of O-samples show a significant variation making an assessment of temperature effects based on the conducted experiments too complex. The observed variation is presumably correlated to the different content of organic matter and calcite.

6.1.1 Effect of Temperature on Elasticity in a Low-Stress Regime—Thermal Micro-Crack Closure

Elastic moduli of the rock frame, M_{dra} and G of the investigated sandstones are unaffected by repeated loading (Fig. 9), and when discarding data associated with bedding effects, a general increase in moduli is observed for increasing temperature (Figs. 10, 11). In addition, independent of stress symmetry, the elastic moduli increase with increasing stress level but do not converge. Thermal closure of micro-cracks is thus only partial (Figs. 10, 11).

Shear waves cannot propagate unless contacts between individual grains exist, but both shear and compressional modulus show a positive temperature dependence (Fig. 12, H and C-samples). We interpret the stiffening as thermal

expansion of the rock minerals resulting in thermal closure of ruptured grain contacts (mechanism 1). The observed increase in shear modulus from increased temperature is equivalent to increased fracture toughness observed in igneous rocks by, e.g. Balme et al. (2004).

The dynamic K_{dra} modulus values show scatter and no general trend with temperature (Figs. 11 and 12). The temperature dependence of M_{dra} and G are thus counteracting and results in K_{dra} being unaffected by temperature. In the tested stress regime with partially closed micro-cracks, Biot's coefficient derived from K_{dra} and the temperature trend of quartz bulk modulus (Fig. 2) accordingly show only minor temperature dependence (Figs. 13, 14). Thus, if we acknowledge $(1 - \alpha)$ as a quantitative measure of the grain contact area, the closure of ruptured grain contacts by thermal expansion (mechanism 1) is minor. Consequently, the observed thermal stiffening is insignificant compared to closure of micro-cracks by increased stress level (Figs. 10, 11). Thus, in a low-stress regime ($\sigma_A \leq 15$ MPa, Branch 1) with partially closed micro-cracks, the rock frame is unconstrained and closure of micro-cracks by thermal expansion the sole cause of the observed stiffening on M_{dra} and G (Mechanism 1). If we disregard the constraints regarding the homogeneity of studied rocks (non-fractured, Eq. 11), we might for a typical dry H-sample tested in the hydrostatic stress regime be tempted to follow this logic: with zero pore pressure ($\sigma_p = 0$) and the applied stress (σ_{hyd}) equal to σ_{dra} ($\sigma_{\text{hyd}} = \sigma_{\text{dra}}$); then the boundary conditions of Eq. 11 reduces to the following:

$$\sigma_T > 0 \text{ when } \sigma_{\text{dra}} \geq \sigma_T, \quad \sigma_T = \beta T K_{\text{dra}}, \quad (12)$$

which we check by assuming a pure quartz frame ($K_s = K_Q$). For a temperature increase of 150 °C and K_{dra} of 17 GPa corresponding to a total stress levels of 15 MPa (Fig. 10), we obtain

Fig. 16 Cross plots of ultrasonic velocity and axial stress data for the O-samples collected as part of failure testing. The solid line in V_p – V_s plots shows the trendline of quartz whereas the dashed line shows a tight sandstone (sst) in the dry state as defined by Jizba (1991) (cited

in Mavko et al. 2009). Individual graphs show samples tested at identical confining stress at, respectively, ambient (grossed symbols) and in situ temperature (open symbols). CT-scans of the samples prior to testing are shown. *Amb.* ambient temperature

$$\sigma_{dra} \geq \beta TK_{dra}$$

$$15 \text{ MPa} \geq 34.7 \cdot 10^{-6} \text{ K}^{-1} \cdot 150 \text{ K} \cdot 17 \cdot 10^3 \text{ MPa} \Leftrightarrow \quad (13)$$

$$15 \text{ MPa} \geq 73.5 \text{ MPa},$$

which is false. The outcome indicates that the assumption of $\sigma_T > 0$ is incorrect and that the sample has an unconstrained

rock frame in the low-stress regime (branch 1). Thermal micro-crack closure is hence the sole cause of the stiffening observed at the lowest stress levels.

There are to the authors' knowledge no published data on ultrasonic velocities for the specific sandstones used in the present work. Further, there are only a limited number

Fig. 17 Cross plots of ultrasonic velocity and axial stress data for the H-samples collected as part of failure testing. The solid line in V_P – V_S plots shows the trendline of quartz whereas the dashed line shows a tight sandstone (sst) in the dry state as defined by Jizba (1991) (cited

in Mavko et al. 2009). Individual graphs show samples tested at identical confining stress at, respectively, ambient (crossed symbols) and in situ temperature (open symbols). CT-scans of the samples prior to testing are shown. *Amb.* ambient temperature

of ultrasonic velocity data recorded on dry sandstones comparable to the present, at elevated temperatures and low-stress levels suitable for demonstration of thermal microcrack closure. Regardless, dry state results from early work by Hughes and Cross (1951) as well as Hughes and Kelly (1952) on three US reservoir sandstones collected at depths

ranging from 2000 to 3000 m agree with the present finding. Tested at stress levels below 25 MPa, their results show a 50–250 m/s increase in V_P by temperature increase to 100 °C. However, in the same stress range, but for another reservoir sandstone, findings from Hughes and Cross (1951) show a 250 m/s decrease in V_P by temperature increase to

Fig. 18 Cross plots of ultrasonic velocity and axial stress data for the C-samples collected as part of failure testing. The solid line in V_P – V_S plots shows the trendline of quartz whereas the dashed line shows a tight sandstone (sst) in the dry state as defined by Jizba (1991) (cited

in Mavko et al. 2009). Individual graphs show samples tested at identical confining stress at, respectively, ambient (open symbols) and in situ temperature (closed symbols). CT-scans of the samples prior to testing are shown. *Amb.* ambient temperature

100 °C. Hence, results by Hughes and Cross (1951) and Hughes and Kelly (1952) show a noticeable disagreement. Having tested at low-stress levels comparable to the present work, Hughes and Cross (1951), as well as Hughes and Kelly (1952), tested uncleaned samples and did not state nor test at in-situ temperature, making it problematic to discuss the observed disagreement concerning stiffening by thermal micro-crack closure.

At varying pore- and confining pressure, and thus effective stress, Mobarak and Somerton (1971), Timur (1977) and

Jones and Nur (1983) tested a series of saturated US outcrop sandstones. Results show a general decrease in V_P and V_S by temperature increase to 200 °C and that the temperature-related decrease in V_P and V_S lowers with increasing effective stress. Ignoring that testing temperature may have extended maximum in-situ, the results indicate that at low effective stress levels, velocity decrease from fluid effects is more significant than velocity increase from thermal micro-crack closure.

Fig. 19 Stress–strain curves of O-samples tested at ambient and in situ temperature as part of failure testing (Branch 2). The stress–strain range of tested low-stress regime (Branch 1) is shown in grey. Static and dynamic E -moduli versus axial stress. E_{sta} is derived from stress–strain curves using a running least-squares best fit with a length of 5 MPa. E_{dyn} is derived from density and ultrasonic velocities

6.1.2 Effect on Elastic Properties at in Situ Temperature and High-Stress Levels

The high similarity between individual samples of well H and C (Fig. 3, Table 3) makes a direct comparison of stress and temperature effects on elasticity possible. For H and C samples, ultrasonic velocities, static and dynamic E -moduli, collected at the lowest confining stress is practically identical for ambient and in situ temperature. Increasing confining stress increases the magnitude of axial failure stress, but also the difference in stiffness observed between ambient and in situ temperature conditions (Figs. 17, 18, 20). The consistent stiffening of the dry rock frame from ambient to in situ temperature at confining stress above 3 MPa for H-samples and 0.7 MPa for C-samples illustrates the suggested stiffening from increased internal stress (mechanism 2). As an example, consider sample H1 tested at 20 MPa confining, axial stress approaching rock failure (branch 2) and in situ temperature. Because sample H1 is tested in a triaxial stress symmetry, we consider the stiffness in terms of the E -modulus (E_{dyn}) and only the longitudinal direction of the sample, which we denote z . For dry conditions, the applied axial stress in z (σ_A) is equal to $\sigma_{dra,z}$, and from the general assumption of a triaxial tests with zero stress change and non-zero strain perpendicular to σ_A , the boundary conditions of Eq. 11 reduce to the following:

$$\sigma_{T,z} > 0 \text{ when } \sigma_{dra,z} \geq \sigma_{T,z}, \quad \sigma_{T,z} = \eta T E_{dyn}. \quad (14)$$

For a temperature increase of 150 °C and with an upper dynamic E -modulus (E_{dyn}) of 50 GPa (Fig. 20) we calculate the theoretical thermal stress in the z -direction from the linear thermal expansion coefficient η ($\beta = 3\eta$) as follows:

$$\sigma_{T,z} = \eta T E_{dyn} = 11.5 \cdot 10^{-6} \text{K}^{-1} \cdot 150 \text{K} \cdot 50 \cdot 10^3 \text{MPa} = 86.3 \text{MPa}, \quad (15)$$

so that if inserted in the boundary conditions of Eq. 11, the sample is theoretically constrained at applied axial stress around 86 MPa. Hence, in this particular example, only for σ_A above 86 MPa is thermal strain fully counteracted by $\sigma_{dra,z}$ meaning that $\varepsilon_{T,z}$ has not resulted in physical expansion. In other words, σ_A of 86 MPa represents the threshold of $\delta = 0$ (Fig. 1), below which the sample is presumably only partly constrained and only part of the thermal strain is converted to thermal stress. Compared to data from ambient, the thermal strain to stress conversion at in situ temperature has resulted in a stiffening of the rock frame (Fig. 20, vertical shift in dashed to solid lines). The maximum E_{dyn} for sample H1 is recorded at σ_A of 86 MPa, which also represents the point where deviation of E_{dyn} from, respectively, ambient and in situ temperature starts to increase significantly

Fig. 20 Stress-strain curves of H- and C-samples tested at ambient and in situ temperature as part of failure testing (Branch 2). The stress-strain range of tested low-stress regime (Branch 1) is shown in grey. Static and dynamic E -moduli versus axial stress. E_{sta} is derived

from stress-strain curves using a running least-squares best fit with a length of 5 MPa. E_{dyn} is derived from density and ultrasonic velocities

(Fig. 20). Hence, at this point, the stiffening effect on the rock frame from thermal stress terminate because $\epsilon_{T,z}$ is fully counteracted by $\sigma_{dra,z}$. A continued increase in applied axial stress ($\sigma_A = \sigma_{dra,z}$) above 86 MPa results in breaking of contact cement inducing a stiffness decrease seen as a downward trend in E_{dyn} for both ambient and in situ temperature (Fig. 20). However, the breakage of contact cement

is delayed at in situ temperature as compared to ambient. This may be because a contribution from the thermal stress reduce the effective stress on the rock frame leading to the contact breakage (Fig. 20). Existence of a physical reduction in the effective stress from thermal stress may be deducted by examination of Eq. 11 which in the case of a dry test in the z -direction reduces to

$$\sigma_{\text{eff},z} = \sigma_{\text{dra},z} - \sigma_{T,z}. \quad (16)$$

This equation resembles Biot's effective stress equation (Eq. 1), but as opposed to pore pressure in Eq. 1, the thermal stress in Eq. 13 carry some of the load from $\sigma_{\text{dra},z}$.

In other words, pore pressure and the thermal stress both tend to reduce $\sigma_{\text{dra},z}$, and Eq. 16 shows that the effective stress leading to cement breakage in our example will decrease as the thermal stress increases. Thus, to match the effective stress at ambient temperature a higher applied axial stress ($\sigma_A = \sigma_{\text{dra},z}$) is needed at in situ temperature, and we observe this as a delayed stiffness reduction from contact breakage at in situ temperature (Fig. 20, horizontal shift in dashed to solid lines). Identical calculations and rationale can be made for remaining H-samples as well as the C-samples where observed axial failure stress is significantly larger than calculated thermal stress. This includes confining stress of 10 and 20 MPa for H-samples as well as 3, 10 and 20 MPa for C-samples, where the difference between the two is a result of the difference in E_{dyn} and thus in σ_T .

The studied O-samples have a mixed mineralogy and a significant heterogeneity on the sample scale. Layers of organic matter found in O-samples (Fig. 3; Table 3) influence the comparison of ultrasonic velocities for samples tested at identical confining stress both for ambient and in situ temperature. Together with the distinct difference between calcite and quartz with respect to stiffness change as function of temperature (Fig. 2), effects of temperature thus are uncertain for O samples, and discrepancies are found in results of both static and dynamic E -moduli adding to an incomplete picture for O-sandstone (Figs. 16, 19).

The ratio of dynamic to static E -moduli is in the order of 2 (Figs. 19, 20) corresponding well to ratios noted for various sedimentary rocks including sandstones (e.g. King 1969; Cheng and Johnston 1981; Yale et al. 1995; Gommessen and Fabricius 2001; Fjær 2009; Li and Fjær 2012; Fjær et al. 2013; Holt et al. 2013; Sone and Zoback 2013; Asef and Farrokhrout 2017). Both static and dynamic moduli may vary significantly with stress path as well as stress history (Walsh 1965; Dvorkin et al. 1996); however, for the tested sandstones, the ratio is practically constant for increasing confining stress (Figs. 19, 20).

7 Conclusion

At different stress levels and symmetries, we studied effects of testing temperature on dry stiffness moduli of three sandstones from the deep North Sea basin. From experimental results in a low-stress regime, we conclude that for the lowest stress levels, increased testing temperature increases dry dynamic compressional and shear modulus, thus stiffening

the dry rock frame. In contrast, the dynamic bulk modulus does not show temperature dependency. We propose closure of micro-crack by thermal expansion of constituting minerals as the mechanism governing increased compressional and shear modulus. Further, we conclude that stiffening of the dry rock frame by thermal closure of micro-cracks is only possible at stress levels with only partial micro-crack closure from stress. Further, stiffening of the rock frame from micro-crack closure from stress increase is orders of magnitude larger than that from thermal micro-crack closure.

In a high-stress regime with full micro-crack closure, experimental results show an increase in static and dynamic Young's modulus with temperature increase from ambient to in-situ. Observed stiffening at in-situ temperature is attributed to thermal expansion of constituting minerals, which induce thermal stress, leading to an overall stiffening of the dry rock frame. Stiffening by thermally induced stresses is, however, only possible at stress levels where the sample volume is fully or partly constrained. The magnitude of thermally induced stiffening is different for the two proposed mechanisms, but in principle, both are governed by the applied stress level.

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Compliance with Ethical Standards

Conflict of Interest The authors declare that they have no conflict of interest.

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Appendix A

See Fig. 21.

Fig. 21 Cross plot of ultrasonic velocity data for the sandstones O, H and C measured at triaxial stress condition with radial stress of 2 MPa, and temperature ranging from ambient to in-situ. Thick ticks indicate corresponding stress or temperature for O- and C-samples shifted to increase readability. The dashed line in V_p - V_s cross shows

the trendline of a tight sandstone in the dry state (citation in Mavko et al. 2009), as well as the quartz line, defined after quartz values in Mavko et al. (2009). The solid lines (σ_s) show trendlines of data obtained for wells H and C

Appendix B

See Figs. 22, 23, 24, 25, 26 and 27.

Fig. 22 Elastic moduli, M_{dra} , G and K_{dra} for dry sandstone as function of temperature at hydrostatic stress symmetry for samples of well O. Elastic moduli are calculated from dry density and ultrasonic velocities. Data points assumed to be affected by bedding are disregarded in trendlines

Fig. 23 Elastic moduli, M_{dra} , G and K_{dra} for dry sandstone as function of temperature and triaxial stress symmetry with radial stress of 2 MPa for sampels of well O. Elastic moduli are calculated from dry density and ultrasonic velocities

Fig. 24 Elastic moduli, M_{dra} , G and K_{dra} for dry sandstone as function of temperature at hydrostatic stress symmetry for samples of well H. Elastic moduli are calculated from dry density and ultrasonic velocities. Data points assumed to be affected by bedding are disregarded in trendlines

Fig. 25 Elastic moduli, M_{dra} , G and K_{dra} for dry sandstone as function of temperature and triaxial stress symmetry with radial stress of 2 MPa for samples of well H. Elastic moduli are calculated from dry density and ultrasonic velocities

Fig. 26 Elastic moduli, M_{dra} , G and K_{dra} for dry sandstone as function of temperature at hydrostatic stress symmetry for samples of well C. Elastic moduli are calculated from dry density and ultrasonic velocities. Data points assumed to be affected by bedding are disregarded in trendlines

Fig. 27 Elastic moduli, M_{dra} , G and K_{dra} for dry sandstone as function of temperature and triaxial stress symmetry with radial stress of 2 MPa for samples of well C. Elastic moduli are calculated from dry density and ultrasonic velocities

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