Effects of humidity and temperature on subcritical crack growth in sandstone

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Abstract

In order to ensure long-term stability of structures in a rock mass, the study of time-dependent fracturing is essential. The influences of the surrounding environmental conditions and rock fabric on subcritical crack growth in sedimentary rocks in air are yet to be clarified, while the nature of subcritical crack growth in igneous rocks has been studied well. In this study, the influences of temperature and relative humidity on subcritical crack growth in Berea sandstone, Shirahama sandstone and Kushiro sandstone were investigated in air. The load relaxation method of Double Torsion (DT) testing method was used to measure both crack velocity and stress intensity factor under a controlled temperature and relative humidity.

Results show that the change of the crack velocity at a given stress intensity factor was unclear when the temperature increased under a constant relative humidity in air. On the other hand, we show that the crack velocity increased by several orders of magnitude when the relative humidity increased threefold or fourfold under a constant temperature at a given stress intensity factor. This increase is much larger than that expected from the conventional concept based on the theory of stress corrosion. It is therefore necessary to consider the additional mechanisms for subcritical crack growth in sandstone. The increase of the crack velocity was larger for sandstone which contained larger amount of clays. We conclude that subcritical crack growth in sandstone in air is affected remarkably by the relative humidity and the amount of clays in rock.

Keywords: Subcritical crack growth, Sandstone, Double Torsion method, Temperature, Relative humidity, Clay
1. Introduction

Long-term stability of the rock mass surrounding structures, such as repositories of radioactive wastes in underground, caverns to store liquid natural gas (LNG) or liquid petroleum gas (LPG), or underground power plants is paramount. For this purpose, it is necessary to estimate the long-term strength and time-to-failure of rock. To this end, a number of investigations have been conducted (Schmidtke and Lajtai, 1986; Wilkins, 1987; Jeong et al., 2007; Nara et al., 2010a). Additionally, it is considered that time-dependent fracturing of rock is related to the increase of seismicity seen prior to earthquake rupture and volcanic eruption (Kilburn and Voight, 1998). Therefore, study of subcritical crack growth is also important for seismology and volcanology. Chau and Shao (2006) reported that time-dependent crack growth in rock played an important role for the failure of rock panels on façade of buildings. It is considered that study of time-dependent crack growth in rock is essential to understand the failure of most structures made of rock.

Although the classical fracture mechanics has postulated that a crack propagates when the stress intensity factor reaches a critical value (fracture toughness), a crack can propagate even when the stress intensity factor below the fracture toughness of the material. This phenomenon is called subcritical crack growth (Atkinson, 1982, 1984; Atkinson and Meredith, 1987), one of the main mechanisms of time-dependent deformation in materials (Dascalu et al., 2010). To better understand the time-dependent behaviour of rock, a lot of studies of subcritical crack growth have been conducted especially in igneous rocks. It has long been known that subcritical crack growth in igneous rocks is affected by fabric of rock and surrounding environmental conditions. For granite, Sano and Kudo (1992) reported that the crack velocity was anisotropic and
affected by the preferred orientation of pre-existing microcracks. Nara and Kaneko (2006) reported that the anisotropy of the crack velocity in granite was decided by the crack propagation direction. According to Nara and Kaneko (2006), when the crack propagated parallel to Rift plane, along which the most pre-existing microcracks are distributed, the crack velocity was the highest. Nara et al. (2006) concluded that the crack propagated by connecting to microcracks ahead of the crack front, and that the crack path tended to be smoother when the crack propagated parallel to Rift plane.

Kudo et al. (1992) observed crack paths in granite and investigated the interaction between the crack paths and mineral grains. According to Kudo et al. (1992), the quartz grains played an important role as an obstacle; plagioclase could change the direction of the crack paths because of its cleavage plane; no definite tendency associated with the cleavage planes and pre-existing microcracks was found for potash feldspar; and biotite grains had a significant effect on the crack paths even when its constitutive ratio was very small.

It has been clarified that water has a significant effect on subcritical crack growth in igneous rocks. It has been reported that the crack velocity in water was much higher than that in air for andesite (Waza et al., 1980; Nara et al., 2009, 2010a), basalt (Waza et al., 1980; Sano and Kudo, 1992), granite (Atkinson and Rawlings, 1981; Lajtai and Bielus, 1986; Lajtai et al., 1987; Sano and Kudo, 1992; Kodama et al., 2003; Nara et al., 2009) and gabbro (Atkinson and Rawlings, 1981). In water, it was shown that the crack velocity increased when the temperature was higher for andesite (Nara et al., 2009, 2010a) and granite (Lajtai et al., 1987; Kodama et al., 2003). In air, it has been reported that the crack velocity increased with increasing the absolute humidity or water vapour pressure for andesite (Nara and Kaneko, 2005), granite (Meredith and Atkinson, 1985; Nara and Kaneko, 2006) and gabbro (Meredith and Atkinson, 1985). Nara et al. (2010a,
b) reported the increase of the crack velocity by 1 ~ 4 orders of magnitude when the relative humidity increased threefold or fourfold for andesite and granite.

For sedimentary rocks, the effect of water on subcritical crack growth has also been investigated. It was shown that the crack velocity in water was much higher than that in air for sandstone (Kodama et al., 2003), novaculite (Atkinson, 1980) and micrite at low stress intensity factors (Henry et al., 1977). In water, the crack velocity tended to be higher when the temperature was higher for sandstone (Kodama et al., 2003). Holder et al. (2001) reported that subcritical crack growth indices (slope of the relation between the crack velocity and the stress intensity factor plotted on a log-log scale) in water were lower than those in air for sandstone, chalk and dolomite. However, in air, the effects of environmental conditions such as the temperature and the relative humidity on subcritical crack growth in sedimentary rocks are yet to be clarified. Additionally, the influence of rock fabric is not clear for sedimentary rocks.

In this study, the influences of relative humidity and temperature on subcritical crack growth were investigated experimentally on three sandstones by a fracture mechanics testing method. Especially, the relation between the stress intensity factor and the crack velocity in air was investigated under different temperatures at a constant relative humidity and under the different relative humidity at a constant temperature. Additionally, the influence of rock fabric on subcritical crack growth is discussed.
2. Rock samples

Berea sandstone (Ohio, USA, Mississippian period), Shirahama sandstone (Wakayama Prefecture, Japan, Neogene period) and sandstone obtained at Kushiro Coalmine (Kushiro sandstone, Hokkaido Prefecture, Japan, Paleogene period) were investigated.

In Fig. 1, results of X-ray diffraction (XRD) analysis are shown. From Fig. 1(a), it is recognized that XRD pattern of Berea sandstone shows the distinct peaks of quartz. It is considered that quartz is the main rock-forming mineral of Berea sandstone. The peaks of feldspars are also recognized and those of clay minerals are not remarkable for Berea sandstone. Fig. 1(b) and (c) show that the peaks of clay minerals for Shirahama and Kushiro sandstones are much more obvious than those for Berea sandstone. Especially, the peaks of smectite, kaolinite and illite are distinct.

Table 1 shows the results of X-ray fluorescence (XRF) analysis. It is shown that the main component for sandstones in this study is quartz. Especially, the percentage of quartz for Berea sandstone is high. On the other hand, the percentages of Fe$_2$O$_3$ and Al$_2$O$_3$ are higher for Shirahama sandstone and Kushiro sandstone.

Physical properties of sandstones are shown in Table 2. P-wave velocities were measured by ultrasonic transmission method in three orthogonal directions called axes-1, -2 and -3 in the order of increasing velocity. Axis-3 is normal to the bedding plane. From Table 2, it is shown that sandstones in this study all possess P-wave velocity anisotropies. However, the difference of P-wave velocity is smaller than that of Inada granite and Oshima granite (Nara and Kaneko, 2006). Young’s moduli and Poisson’s ratios were obtained by uniaxial compression tests with loading parallel to the bedding plane. Porosity was measured by water saturation.
In this study, all of specimens for fracture mechanics test were prepared so that the crack propagated parallel to the bedding plane. After cutting and grinding, we kept specimens under ambient air condition the temperature at 300 K and relative humidity at 20 ~ 30 % for at least 3 months in order to dry them enough.
3. Methodology

3.1 Outline of experimental method

The Double Torsion (DT) method was used in this study. DT method is a typical fracture mechanics testing method that is used to study subcritical crack growth. The loading configuration of DT method is shown in Fig. 2. Three different types of test can be performed using the DT arrangement, each using different loading conditions: the constant load (CL) method (Kies and Clark, 1969), the constant displacement rate (CDR) method (Evans, 1972), and the load relaxation (RLX) method (Evans, 1972; Williams and Evans, 1973). RLX method gives a wide range of data of the relation between the stress intensity factor, $K_I$, and the crack velocity, $da/dt$, ($K_I$-$da/dt$ relation), generally from $10^{-2}$ to $10^{-9}$ m/s by only a single experimental run, while CL and CDR methods give only one point of $K_I$-$da/dt$ relation. In this study, RLX method was adopted to obtain $K_I$-$da/dt$ relation.

In RLX method, the displacement of the loading points has to be kept constant during the experiment whilst the temporal decreasing (load relaxation) due to the crack propagation is measured. The stress intensity factor and the crack velocity are expressed as follows (Evans, 1973; Williams and Evans, 1973):

\[
K_I = P w_m \sqrt{\frac{3(1 + \nu)}{W d^3 d_n}} \tag{1}
\]

\[
\frac{da}{dt} = -\varphi_c \times \frac{W d^3 G S_i P_i}{3 W_m^2} \frac{dP}{P^2} \tag{2}
\]

where $P$ is the applied load, $w_m$ is the moment arm (23 mm in this study), $\nu$ is Poisson’s ratio, $W$ is the width of the specimen, $d$ is the thickness of the specimen, $d_n$ is the
reduced thickness of the specimen, \( P_1 \) is the initial value of the applied load, \( S_i \) is the compliance of the specimen at the initial crack length, \( dP/dt \) is the load relaxation rate, and \( G \) is the shear modulus. \( \varphi_c \) is a constant that is dependent on the shape of the crack front. Experiments using glass (Williams and Evans, 1973) and quartz (Atkinson, 1979a) suggested that \( \varphi_c = 0.2 \).

Eqs. (1) and (2) are approximate solutions based on a thin-plate assumption considering no thermal and hydro-mechanical phenomena (Williams and Evans, 1973). It is therefore important to consider their applicability. The size of the DT specimen has to satisfy the condition as follows (Evans et al., 1974; Atkinson, 1979b; Pletka et al., 1979):

\[
12d \leq W \leq L/2
\]

where \( L \) is the length of the specimen. According to Tait et al. (1987), Shyam and Lara-Curzio (2006) and Madjoubi et al. (2007), it seems to be better that the length of the specimen is larger than twofold of the width. By using a finite element analysis, Ciccotti and his co-workers expressed the corrective factor of the specimen compliance to consider the non-linear terms of the dependence of the compliance on the crack length in a DT specimen (Ciccotti, 2000; Ciccotti et al., 2000a). Based on their results, then they used thicker specimen (\( W : d = 8 : 1 \)) than that recommended by Evans et al. (1974) or Atkinson (1979b) (Ciccotti et al., 2000b, 2001). Sano (1988) showed the proportional relation between the compliance and the crack length in a DT specimen of soda-lime glass, basalt and quartz andesite. In general, the energy release rate \( g \) is related to the compliance \( S \) as follows:

\[
g = \frac{P^2}{2} \frac{dS}{dA_c}
\]

where \( A_c \) is the area of the crack. In the case of DT method, the shape of the crack front
is independent of the crack length (Evans, 1972; Williams and Evans, 1973; Atkinson, 1979a). Therefore, the following relationship can be obtained:

$$g \propto \frac{P^2}{2} \frac{dS}{da} \quad (5).$$

If the compliance $S$ is proportional to the crack length $a$, the energy release rate and thus stress intensity factor is independent of the crack length. Therefore, the result of Sano (1988) proves that the stress intensity factor of DT method is independent of the crack length.

Taking the above investigations into consideration, we set that the width $W$ was 55 mm and the length $L$ was 145 mm. For Berea sandstone and Shirahama sandstone, the thickness $d$ was 3.5 mm. It was impossible to make the thickness 3.5 mm for the specimen of Kushiro sandstone, because this sandstone was easily broken when it was immersed in water during making a DT specimen. Therefore, the thickness $d$ was 4 mm for Kushiro sandstone. The width and depth of the guide groove was 2 mm and 1 mm, respectively.

3.2 Experimental apparatus

A schematic illustration of the apparatus used in this study is shown in Fig. 3. This apparatus was same as that of Nara and Kaneko (2005, 2006) except for the controllable range of the environmental conditions. As shown in Fig. 3, this apparatus was set in a room where the temperature and the relative humidity can be controlled. Therefore, it is possible to conduct experiments under the controlled temperature and relative humidity in air. The controllable ranges of the temperature and the relative humidity were 278 ~
353 K and 40 ~ 90 %, respectively.

DT specimen is loaded by the electrical-powered cylinder via stainless steel balls, of diameter 4 mm. By using the digital microscope placed under the specimen, the crack propagation in the DT specimen can be monitored. The applied load is measured by the load cell with the accuracy of ± 0.04 N. Displacement of the loading point is measured by two displacement transducers with the accuracy of ± 0.5 μm.

3.3 Experimental condition

In order to investigate the effect of the temperature and the relative humidity separately, measurements of subcritical crack growth were conducted under different temperature at a given relative humidity and vice versa. We conducted experiments under low temperature (293 K, 53 ~ 56 %), intermediate temperature (328 ~ 329 K, 53 %) and high temperature (349 ~ 350 K, 50 ~ 54 %) conditions to investigate the influence of the temperature, and under low humidity (290 ~ 296 K, 18 ~ 29 %), intermediate humidity (293K, 53 ~ 56 %) and high humidity (293 K, 85 ~ 92 %) conditions to investigate the influence of the relative humidity. The measurements were conducted under controlled temperature and relative humidity. However, it was impossible to achieve the low humidity condition whilst controlling the temperature and the relative humidity. This is because of the limitation of the controllable range of the relative humidity. The low humidity condition was achieved by an ambient condition in the laboratory only in winter. Unfortunately, these measurements were thus carried out without controlling the temperature and relative humidity.
3.4 Experimental procedure

At first, precracking was conducted. For precracking, we applied the displacement at the loading point by 4 μm, and then fixed it in order to observe the surface of the specimen the digital microscope set under the specimen for the check of the crack length. We continued these operations until the crack length reached 30 mm, which is the minimum length required for the condition in which the stress intensity factor is independent of the crack length for a DT specimen (Trantina, 1977).

After precracking, the apparatus and specimen were exposed to the experimental environment, with the same temperature and relative humidity, for more than 20 hours. Following this period, the measurement of crack growth with RLX method was carried out.

3.5 Loading condition

For rock, it has been reported that hysteresis of the $K_t$-$da/dt$ relation was observed when conducting measurements by RLX method several times with one specimen (Swanson, 1984; Sano, 1988). Sano (1988) suggested that this hysteresis was due to the friction and locking on the crack surfaces, since no hysteresis was observed on soda-lime glass. When measurements by RLX method are conducted, it is necessary to minimize the effect of the friction and locking. Hence, it is important to open the crack as much as possible and not to repeat opening and closing the crack. It is also considered that the effect of the friction and locking can be larger when the initial length
of the crack is larger. Therefore, it is better to conduct only a single experimental run with one specimen to avoid the larger effect of the friction and locking. In order to ensure that a wide crack opened in the specimen, the displacement applied to the loading points was set so that the initial value of the applied load became large and close to the value corresponding to the fracture toughness as much as possible.

The fracture toughness was measured with CDR method in this study. According to Selçuk and Atkinson (2000), the measured stress intensity factor with CDR method increased with increasing displacement rate and was converged when the displacement rate was higher than 0.07 mm/s. It is considered that the displacement rate should be higher than 0.07 mm/s to measure the fracture toughness with CDR method. The displacement rate was 0.23 mm/s in this study. Measurements were conducted under low humidity condition. In Table 3, the measured fracture toughness is listed. In this table, the average and the standard deviation of 3 specimens for Berea sandstone and Shirahama sandstone and 2 specimens for Kushiro sandstone are shown.

Based on the above consideration, after applying the preload of 4 ~5 N (27 ~ 30 % of the maximum load) in order to avoid hitting the specimen, we applied 0.52 mm of the displacement at the loading points on the specimens of Berea sandstone in all environmental conditions. On the specimens of Shirahama sandstone, we applied 0.44 mm of the displacement at the loading points after applying the preload of 4.5 ~ 5.5 N (15 ~ 38 % of the maximum load). On the specimens of Kushiro sandstone, we applied 0.44 mm of the displacement at the loading points after applying the preload of 7.5 N (13 ~ 20 % of the maximum load). The above displacements were applied quickly within 0.3 seconds and kept constant for 90 ~ 120 minutes as conducted in previous studies (Nara and Kaneko, 2005, 2006; Nara et al., 2009, 2010a, 2010b). In Fig. 4, the examples of temporal changes of the applied load are shown. The stress intensity factor
and the crack velocity were estimated from the data in Fig. 4 using Eqs. (1) and (2), respectively. With the above loading conditions, we obtained the maximum loads corresponding to more than 85% of the fracture toughness under the low humidity condition.
4. Results

The crack velocity $\frac{da}{dt}$ is related to the stress intensity factor empirically as follows (Charles, 1958; Wiederhorn and Bolz, 1970):

$$\frac{da}{dt} = AK_1^n \exp \left( \frac{-E^\ddagger}{RT} \right)$$  \hspace{1cm} (6)

$$\frac{da}{dt} = v_0 \exp \left( \frac{-E^\ddagger + bK_1}{RT} \right)$$  \hspace{1cm} (7)

where $E^\ddagger$ is the stress-free activation energy, $R$ is the gas constant, $T$ is the absolute temperature, and others are constants determined experimentally. Especially, $n$ is called subcritical crack growth index (Atkinson and Meredith, 1987). In this study, Eq. (6) was used to describe $K_1$-$da/dt$ relations.

$K_1$-$da/dt$ relations for Berea sandstone, Shirahama sandstone and Kushiro sandstone obtained under different relative humidities at a constant temperature are shown in Figs. 5, 6 and 7, respectively. From these figures, it is recognized that the reproducibility of the results is very high under the same environmental conditions. In addition, it is shown that the crack velocity increased with increasing the relative humidity. For Shirahama sandstone and Kushiro sandstone, the increase of the crack velocity was much more remarkable than that for Berea sandstone.

In Tables 4, 5 and 6, the results of subcritical crack growth measurements for Berea sandstone, Shirahama sandstone and Kushiro sandstone under different relative humidities are summarized, respectively. In these tables, the stress intensity factor at $da/dt = 10^{-5}$ [m/s], $K_1(10^{-5})$, is listed to provide a quantitative comparison of the stress intensity factor, because the range of the crack velocity was $10^{-2} \sim 10^{-8}$ m/s. Since the range of the stress intensity factor was $0.2 \sim 0.4$ MN/m$^{3/2}$ for Berea sandstone, the value
of the crack velocity at $K_I = 0.3$ [MN/m$^{3/2}$], $da/dt(0.3)$, is listed in Table 4. For Shirahama sandstone, since the range of the stress intensity factor was 0.2 ~ 0.6 MN/m$^{3/2}$, the value of the crack velocity at $K_I = 0.4$ [MN/m$^{3/2}$], $da/dt(0.4)$, is listed in Table 5. For Kushiro sandstone, since the range of the stress intensity factor was 0.3 ~ 0.9 MN/m$^{3/2}$, the value of the crack velocity at $K_I = 0.6$ [MN/m$^{3/2}$], $da/dt(0.6)$, is listed in Table 6. In Tables 4 ~ 6, the values of average and standard deviation (logarithmic average and standard deviation for the crack velocity) from 2 ~ 3 specimens are listed.

Tables 4 ~ 6 show that the crack velocity increased for all sandstones when the relative humidity increased. However, the extent of the increase of the crack velocity for Berea sandstone was smaller obviously than that for Shirahama sandstone and Kushiro sandstone. For Berea sandstone, the crack velocity increased by 5 ~ 6 orders of magnitude when the relative humidity increased threefold or fourfold. On the other hand, the crack velocities for Shirahama sandstone and Kushiro sandstone increased by 9 ~ 10 orders of magnitude.

Figs. 8 and 9 show $K_I$-$da/dt$ relations under different temperatures at constant relative humidity for Berea sandstone and Shirahama sandstone, respectively. From these figures, it is recognized that the changes of the crack velocity and the stress intensity factor are unclear.

In Tables 7 and 8, the results of subcritical crack growth measurements for Berea sandstone and Shirahama sandstone under different temperatures are summarized, respectively. In Table 7, $da/dt(0.27)$ indicates the value of the crack velocity at $K_I = 0.27$ [MN/m$^{3/2}$]. This is listed because the range of the stress intensity factor for Berea sandstone was 0.22 ~ 0.32 MN/m$^{3/2}$ (see Fig. 8). Similarly, we also list $da/dt(0.35)$ in Table 8, which indicates the crack velocity at $K_I = 0.35$ [MN/m$^{3/2}$], because the range of the stress intensity factor for Shirahama sandstone was 0.28 ~ 0.42 [MN/m$^{3/2}$] (see Fig.
9). Additionally, $n$ and $da/dt(10^5)$ are also listed in these tables. It is recognized that the changes of the stress intensity factor and the crack velocity are unremarkable, even though the temperature increases.

It is shown that subcritical crack growth index $n$ for Kushiro sandstone tends to be smaller when the relative humidity is higher (see Table 6). On the other hand, dependence of $n$ on the temperature and relative humidity for Berea sandstone and Shirahama sandstone is not clear (see Tables 4, 5, 7 and 8). Additionally, it is recognized that the values of subcritical crack growth index $n$ for Berea sandstone is larger than those for other sandstones. In summary, the reproducibility of the evaluation of $n$ is high.
5. Discussion

In order to estimate the stress intensity factor and the crack velocity, we used Eqs. (1) and (2) which consider no thermal and hydro-mechanical phenomena. Experimental results therefore show that the relative humidity affects subcritical crack growth in sandstone significantly. An increase of the crack velocity by several orders of magnitude was observed when the relative humidity increased threefold or fourfold.

It has been considered that the main mechanism of subcritical crack growth is stress corrosion in the Earth’s upper brittle crust (Atkinson, 1982). In a silica-quartz system, this is considered to be the chemical reaction between the siloxane bond and water at the crack tip under tension (Anderson and Grew, 1977; Michalske and Freiman, 1982; Atkinson and Meredith, 1987). According to Wiederhorn et al. (1980) and Freiman (1984), if stress corrosion is the main mechanism of subcritical crack growth, the crack velocity should be proportional to the relative humidity when the temperature is constant. Wiederhorn (1967) and Soga et al. (1979) showed experimentally the proportional relation between the crack velocity and relative humidity for soda-lime glass at a constant temperature. According to their measurements, the crack velocity at a given stress intensity factor increased proportionally with the increase of the relative humidity at a constant temperature. Specifically, Wiederhorn (1967) reported that this proportional relationship was satisfied if the relative humidity was higher than 1%.

Therefore, within the range of the relative humidity in this study, it is confirmed that the increase of the crack velocity in soda-lime glass is proportional to the increase of the relative humidity if the temperature is constant.

As mentioned in Section 1, however, for igneous rocks, the crack velocity was not proportional to the relative humidity and the increase of the crack velocity was 1 ~ 4
orders of magnitude when the increase of the relative humidity at a constant temperature was threefold or fourfold (Nara et al., 2010b). The increases of the crack velocity in sandstones were also much larger than those expected from the concept of stress corrosion (Freiman, 1984) and larger than those in igneous rocks (Nara et al., 2010b). In addition, the decreases of the stress intensity factor were 20 % for Berea sandstone and 50 % for Shirahama sandstone and Kushiro sandstone. For Shirahama sandstone and Kushiro sandstone, the decreases are larger than those for igneous rocks (10 ~ 14 %) (Nara et al., 2010b). Therefore, it is necessary to consider the mechanisms which control subcritical crack growth in sandstone.

Fujita et al. (2000) reported that a significant decrease of the strength and the stress intensity factor attributed to the decrease of the strength of illite contained in Shirahama sandstone by conducting uniaxial compression test, Brazilian tensile test and bending test with Chevron notched specimen. Funatsu et al. (2004) reported that dehydration of interlayer and absorptive water from smectite contained in Kimachi sandstone caused the increase of fracture toughness. Thus, interactions between water and clay minerals such as illite and smectite seem to be important factors of the crack growth in sandstone.

In Figs. 10, 11 and 12, photomicrographs of the crack path observed with thin sections of 0.03 mm thickness for Berea sandstone, Shirahama sandstone and Kushiro sandstone are shown, respectively. In these figures, (a), (b) and (c) are obtained under ultraviolet light by a fluorescent method (Ali and Weiss, 1968; Gardner and Pincus, 1968; Nishiyama and Kusuda, 1994, 1996, Nishiyama et al., 2002), open nicol and crossed nicols, respectively. For each sample, all photos were taken at a same position. From these figures, it is recognized that the crack propagated mainly along the grain boundaries. In addition, the crack paths through clays were observed for Shirahama
sandstone (see Fig. 11) and Kushiro sandstone (see Fig. 12). Subcritical crack growth in sandstone may be dependent on the characteristics of clays and grain boundaries mainly.

For the crack growth along the grain boundary of hard mineral grains such as quartz and feldspar without clays, it is considered that the crack propagated in similar way to igneous rocks related to the mechanical properties of these minerals (Nara et al., 2010b).

It is necessary to consider the effect of water on clays. If the content of water in smectite increases, the basal spacing increases remarkably (Sato et al., 1992), and the strength of sandstone containing smectite decreases (Young et al., 2009). Shirahama sandstone and Kushiro sandstone include smectite (see Fig. 1). Therefore, it is considered that smectite caused the remarkable decrease of the stress intensity factor for these sandstones. Additionally, the strength of sediment containing illite tended to decrease with increasing water content according to Francisca et al. (2005). It is thus considered that the resistance to crack growth decreases in clays such as illite and smectite when the relative humidity is higher and the water content increases. Consequently, this leads to a remarkable increase the crack velocity in Shirahama sandstone and Kushiro sandstone. On the other hand, it is considered that the change of the crack velocity and the stress intensity factor in Berea sandstone were smaller because the content of clays was much less than that for the other sandstones.

Although the crack paths through clays were observed for Shirahama sandstone and Kushiro sandstone (see Figs. 11 and 12), apparently the crack propagated mainly along grain boundaries. It is considered that clays existing along grain boundaries as cements were weakened by water as suggested by Dhakal et al. (2002), and then the crack propagated along the weakened grain boundaries.

It is worth considering the reason why the temperature had little effect on subcritical crack growth in sandstone in air. This is apparently inconsistent with the theory of stress
corrosion. After measuring subcritical crack growth, we determined the water content of DT specimens used in our experiments. In Fig. 13, the relation between the water content and temperature are shown. The water content was calculated by the following equation:

\[ w_c = \frac{W_e - W_d}{W_d} \times 100 \]  

(8)

where \( w_c \) is the water content [%], \( W_e \) is the weight of the specimen under the experimental condition, and \( W_d \) is the weight of the specimen after dried in oven at 363 K. Fig. 13 shows that the water content tends to decrease with increasing temperature. In this case, thermally-activated processes such as stress corrosion were inhibited due to the decrease of water in the rock. Therefore, it is considered that the acceleration of crack growth, due to the increase of the temperature, and the suppression of the crack growth, due to the decrease of water, in sandstone occurred simultaneously, and then the changes of the crack velocity and the stress intensity factor became indistinct.

In water, it is possible to investigate the effect of the temperature on subcritical crack growth in sandstone at constant water content. In Fig. 14, \( K_1-da/dt \) relationship for sandstone in water with different temperatures is shown. Figs. 14(a) and (b) show the relations for Berea sandstone and Shirahama sandstone, respectively. It is shown that the crack velocity tends to be higher at higher temperatures even though the temperature increased only by 30 °C. This result is consistent with the concept of stress corrosion. The same tendency for Shirahama sandstone was observed by Kodama et al. (2003). This is also consistent with the result of Heap et al. (2009) about the temperature dependence of brittle creep in sandstone.

It is necessary to discuss the values of subcritical crack growth index \( n \). Since \( n \) relates to the estimate of the long-term strength and time-to-failure (Wilkins, 1987;
Jeong et al., 2007; Nara et al., 2010a), precise evaluation of $n$ is necessary. If the load relaxation due to the causes except for the crack growth is included, $n$ will be underestimated (Pletka et al., 1979). It is shown that the values of subcritical crack growth index $n$ for Shirahama sandstone and Kushiro sandstone are larger than that obtained by Atkinson (1984) (17 for Tennessee sandstone) and similar to those obtained by Kodama et al. (2003) (45 for Shirahama sandstone), Seto et al. (2001) (33 for Shirahama sandstone and 46 for Kimachi sandstone) and Holder et al. (2001) (35 for Scioto sandstone “block 1”) in air (see Tables 5, 6 and 8). The values of $n$ for Berea sandstone is similar to those of Scioto sandstone (52 for “block 2”) (Holder et al., 2001) and igneous rocks such as Kumamoto andesite (43 ~ 61) (Nara et al., 2010b) and granite (49 ~ 88 for Oshima granite, 53 ~ 75 for Inada granite and 47 ~ 77 for Westerly granite) (Nara and Kaneko, 2006) (see Tables 4 and 7). From these comparisons, it is considered that the underestimation of $n$ is not considerable and the effect of the load relaxation except for the crack growth was little.

Since the number of the studies of subcritical crack growth in sandstone (Atkinson, 1984; Holder et al., 2001; Kodama et al., 2003; Ponson, 2009) is much less than those in igneous rocks, it is essential to obtain more information in order to understand the crack growth in sandstone. In addition, it is necessary to obtain results by other experimental method and to compare with those by RLX method. For example, it will be worth conducting measurement by CDR method as well as RLX method as suggested by Sano (1988) to improve the reliability of experimental results. In order to understand the influence of clay on subcritical crack growth in sandstone, it is necessary to investigate the crack growth in clay. Additionally, it will be important to consider the detailed influences of water condensate (Thomson, 1871; Wondraczek et al., 2006; Grimaldi et al., 2008) and the existence of fracture process zone (Sano, 1981; Swanson, 1987)
around a crack tip as well as the influence of clay. These will be the goal of future studies and provide useful information for the long-term stability of structures in a rock mass.
6. Conclusion

In this study, the changes of the crack velocity and the stress intensity factor were investigated with DT testing method under controlled temperature and relative humidity.

It was shown that the crack velocity increased and the stress intensity factor decreased when the relative humidity was higher at a constant temperature. The decrease of the stress intensity factor was around 50% for Shirahama sandstone and Kushiro sandstone which contained relatively large amount of clays. On the other hand, the decrease was around 20% for Berea sandstone, which contained few clays. The increases of the crack velocity were 5 ~ 6 orders of magnitude for Berea sandstone and 9 ~ 10 orders of magnitude for Shirahama sandstone and Kushiro sandstone, respectively, when the increase of the relative humidity was threefold or fourfold at constant temperature. These increases in the crack velocity were much larger than that expected from the concept of stress corrosion. Since the changes of the crack velocity and the stress intensity factor were larger for sandstone in which the content of clays was larger, it is concluded that subcritical crack growth in sandstone in air is affected remarkably by the relative humidity and the amount of clays in sandstone.

It was shown that the changes of the crack velocity and the stress intensity factor were unremarkable for sandstone when the temperature increased at a constant relative humidity. This result was obtained due to the decrease of water content in sandstone with increasing the temperature. In this case, it is considered that the acceleration of the crack growth due to the increase of the temperature and the suppression of the crack growth due to the decrease of water in sandstone occurred at the same time and then the changes of the crack velocity and the stress intensity factor became indistinct.
The values of subcritical crack growth index for Shirahama sandstone and Kushiro sandstone agreed with those obtained for sandstones in air by other researchers. On the other hand, subcritical crack growth index for Berea sandstone was similar to those for igneous rocks.

This study reveals that the effects of the humidity and the clay in rock affect subcritical crack growth and the long-term stability of structures in sandstone remarkably. For the long-term stability, it is therefore effective to control the relative humidity of surrounding environment. Consequently, weakening of structures in a rock mass can be restrained by retarding subcritical crack growth.
Acknowledgement

The authors appreciate the support of Research Fellowships of the Japan Society for the Promotion of Science for Young Scientists. The sample of Kushiro sandstone was donated by Prof. Yoshiaki Fujii at Hokkaido University and Kushiro Coalmine Co., LTD. We also appreciate Dr. Michael Heap at Ludwig Maximilians Universität München for useful discussions and comments on this study.
References


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27, 14-22.


**Figure captions**

Fig. 1  X-ray diffraction patterns for sandstones. “c.p.s.” means “counts per second”.

(a): Berea sandstone, (b): Shirahama sandstone, (c): Kushiro sandstone

Fig. 2  Double Torsion specimen and loading configuration. The loading forces are shown by four thick arrows.

Fig. 3  Schematic illustration of Double Torsion testing apparatus.

Fig. 4  Temporal changes of applied load.

Fig. 5  Relations between crack velocity and stress intensity factor for Berea sandstone in air with different relative humidities.

Fig. 6  Relations between crack velocity and stress intensity factor for Shirahama sandstone in air with different relative humidities.

Fig. 7  Relations between crack velocity and stress intensity factor for Kushiro sandstone in air with different relative humidities.

Fig. 8  Relations between crack velocity and stress intensity factor for Berea sandstone in air with different temperatures.

Fig. 9  Relations between crack velocity and stress intensity factor for Shirahama
sandstone in air with different temperatures.

Fig. 10  Images of crack path in Berea sandstone observed with polarizing microscope. The length of the images is 2.6mm. The crack propagates mainly along the grain boundaries.  (a): Image under ultraviolet light, (b): Image under open nicol, (c): Image under crossed nicols

Fig. 11  Images of crack path in Shirahama sandstone observed with polarizing microscope. The length of the images is 1.1mm. The crack propagates along the grain boundaries and through the clay.  (a): Image under ultraviolet light, (b): Image under open nicol, (c): Image under crossed nicols

Fig. 12  Images of crack path in Kushiro sandstone observed with polarizing microscope. The length of the images is 2.6mm. The crack propagates along the grain boundaries and through the clay.  (a): Image under ultraviolet light, (b): Image under open nicol, (c): Image under crossed nicols

Fig. 13  Relations between water content in sandstones and temperature.  (a): Berea sandstone, (b): Shirahama sandstone

Fig. 14  Relations between crack velocity and stress intensity factor for sandstone in distilled water with different temperatures.  (a): Berea sandstone, (b): Shirahama sandstone
Table 1  Chemical composition of sandstones obtained from X-ray fluorescence analysis in weight percent. “nd” means “not detected”.

<table>
<thead>
<tr>
<th></th>
<th>Berea sandstone</th>
<th>Shirahama sandstone</th>
<th>Kushiro sandstone</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO$_2$</td>
<td>92.24</td>
<td>74.31</td>
<td>55.57</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>4.39</td>
<td>12.47</td>
<td>14.87</td>
</tr>
<tr>
<td>K$_2$O</td>
<td>1.46</td>
<td>3.95</td>
<td>3.18</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>1.06</td>
<td>5.96</td>
<td>17.02</td>
</tr>
<tr>
<td>CaO</td>
<td>0.30</td>
<td>1.05</td>
<td>5.36</td>
</tr>
<tr>
<td>TiO$_2$</td>
<td>0.23</td>
<td>0.42</td>
<td>0.80</td>
</tr>
<tr>
<td>MgO</td>
<td>0.19</td>
<td>0.77</td>
<td>1.26</td>
</tr>
<tr>
<td>MnO$_2$</td>
<td>nd</td>
<td>nd</td>
<td>0.43</td>
</tr>
<tr>
<td>Na$_2$O</td>
<td>nd</td>
<td>0.90</td>
<td>1.30</td>
</tr>
</tbody>
</table>
Table 2  Physical properties of sandstones.

<table>
<thead>
<tr>
<th>Rock samples</th>
<th>P-wave velocities [km/s]</th>
<th>Young’s modulus [GPa]</th>
<th>Poisson’s ratio</th>
<th>Porosity [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Berea sandstone</td>
<td>2.3 (in axis-1)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.3 (in axis-2)</td>
<td>8.20</td>
<td>0.25</td>
<td>20.4</td>
</tr>
<tr>
<td></td>
<td>2.2 (in axis-3)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Shirahama sandstone</td>
<td>2.9 (in axis-1)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.8 (in axis-2)</td>
<td>6.23</td>
<td>0.33</td>
<td>12.9</td>
</tr>
<tr>
<td></td>
<td>2.6 (in axis-3)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Kushiro sandstone</td>
<td>2.9 (in axis-1)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.7 (in axis-2)</td>
<td>9.12</td>
<td>0.32</td>
<td>10.4</td>
</tr>
<tr>
<td></td>
<td>2.7 (in axis-3)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 3  Result of fracture toughness measurements for sandstones in air under low humidity condition (290 ~ 296 K, 18 ~ 29 %).

<table>
<thead>
<tr>
<th>Rock samples</th>
<th>Fracture toughness [MN/m$^{3/2}$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Berea sandstone</td>
<td>0.36±0.01</td>
</tr>
<tr>
<td>Shirahama sandstone</td>
<td>0.73±0.01</td>
</tr>
<tr>
<td>Kushiro sandstone</td>
<td>0.89±0.07</td>
</tr>
</tbody>
</table>
Table 4  Summary of subcritical crack growth measurement for Berea sandstone in air with different relative humidities. SD means the standard deviation.

<table>
<thead>
<tr>
<th>Condition</th>
<th>n</th>
<th>$K_I(10^{-5})$ [MN/m$^{3/2}$]</th>
<th>$da/dt(0.3)$ [m/s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low humidity (290-293K, 18-27%)</td>
<td>57±6</td>
<td>0.32±0.01</td>
<td>1.3×10$^{-7}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(SD = 1.2×10$^1$ in log)</td>
</tr>
<tr>
<td>Intermediate humidity (293K, 54-56%)</td>
<td>61±2</td>
<td>0.27±0.01</td>
<td>1.9×10$^{-3}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(SD = 4.8×10$^0$ in log)</td>
</tr>
<tr>
<td>High humidity (293K, 89-92%)</td>
<td>64±0</td>
<td>0.26±0.00</td>
<td>7.2×10$^{-2}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(SD = 1.3×10$^0$ in log)</td>
</tr>
</tbody>
</table>

Table 5  Summary of subcritical crack growth measurement for Shirahama sandstone in air with different relative humidities. SD means the standard deviation.

<table>
<thead>
<tr>
<th>Condition</th>
<th>n</th>
<th>$K_I(10^{-5})$ [MN/m$^{3/2}$]</th>
<th>$da/dt(0.4)$ [m/s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low humidity (293-296K, 21-25%)</td>
<td>28±2</td>
<td>0.50±0.03</td>
<td>2.4×10$^{-8}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(SD = 5.9×10$^0$ in log)</td>
</tr>
<tr>
<td>Intermediate humidity (293K, 53-56%)</td>
<td>37±0</td>
<td>0.34±0.01</td>
<td>4.9×10$^{-3}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(SD = 1.9×10$^0$ in log)</td>
</tr>
<tr>
<td>High humidity (293K, 90-92%)</td>
<td>30±2</td>
<td>0.23±0.00</td>
<td>1.6×10$^{2}$</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>(SD = 2.9×10$^0$ in log)</td>
</tr>
</tbody>
</table>
Table 6  Summary of subcritical crack growth measurement for Kushiro sandstone in air with different relative humidities. SD means the standard deviation.  

<table>
<thead>
<tr>
<th>Condition</th>
<th>$n$</th>
<th>$K_I(10^{-5})$ [MN/m$^{3/2}$]</th>
<th>$da/dt(0.6)$ [m/s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low humidity (291K, 21-25%)</td>
<td>44±0</td>
<td>0.77±0.01</td>
<td>1.1×10^{-10} (SD = 1.7×10^{0} in log)</td>
</tr>
<tr>
<td>Intermediate humidity (293K, 54-55%)</td>
<td>35±2</td>
<td>0.53±0.03</td>
<td>8.7×10^{4} (SD = 7.4×10^{0} in log)</td>
</tr>
<tr>
<td>High humidity (293K, 85-88%)</td>
<td>26±1</td>
<td>0.41±0.02</td>
<td>1.8×10^{-1} (SD = 2.0×10^{0} in log)</td>
</tr>
</tbody>
</table>

Table 7  Summary of subcritical crack growth measurement for Berea sandstone in air with different temperatures. SD means the standard deviation.  

<table>
<thead>
<tr>
<th>Condition</th>
<th>$n$</th>
<th>$K_I(10^{-5})$ [MN/m$^{3/2}$]</th>
<th>$da/dt(0.27)$ [m/s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low temperature (293K, 54-56%)</td>
<td>61±2</td>
<td>0.27±0.01</td>
<td>3.0×10^{6} (SD = 3.9×10^{1} in log)</td>
</tr>
<tr>
<td>Intermediate temperature (328-329K, 53%)</td>
<td>51±1</td>
<td>0.26±0.01</td>
<td>5.0×10^{5} (SD = 4.8×10^{0} in log)</td>
</tr>
<tr>
<td>High temperature (349-350K, 51-54%)</td>
<td>52±2</td>
<td>0.27±0.02</td>
<td>1.5×10^{5} (SD = 1.1×10^{1} in log)</td>
</tr>
</tbody>
</table>
Table 8  Summary of subcritical crack growth measurement for Shirahama sandstone in air with different temperatures. SD means the standard deviation.

<table>
<thead>
<tr>
<th>Condition</th>
<th>n</th>
<th>$K_i(10^{-5})$</th>
<th>$\frac{da}{dt}(0.35)$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>[MN/m$^{3/2}$]</td>
<td>[m/s]</td>
</tr>
<tr>
<td>Low temperature</td>
<td>37±0</td>
<td>0.34±0.01</td>
<td>3.5×10$^5$</td>
</tr>
<tr>
<td>(293K, 53-56%)</td>
<td></td>
<td></td>
<td>(SD = 3.8×10$^0$ in log)</td>
</tr>
<tr>
<td>Intermediate temperature</td>
<td>32±2</td>
<td>0.37±0.01</td>
<td>1.6×10$^5$</td>
</tr>
<tr>
<td>(329K, 53%)</td>
<td></td>
<td></td>
<td>(SD = 1.9×10$^0$ in log)</td>
</tr>
<tr>
<td>High temperature</td>
<td>33±2</td>
<td>0.35±0.02</td>
<td>9.1×10$^6$</td>
</tr>
<tr>
<td>(349-350K, 50-51%)</td>
<td></td>
<td></td>
<td>(SD = 1.9×10$^0$ in log)</td>
</tr>
</tbody>
</table>
Berea sandstone

- Star: Quartz
- Circle: Plagioclase
- Triangle: Potash feldspar
- Inverted Triangle: Kaolinite

Intensity [c.p.s.]

2 θ [°] (CuKα)
Figure 4

Low humidity condition (290~296K, 18~29%)

Kushiro sandstone

Shirahama sandstone

Berea sandstone

Applied load [N]

Time [s]
Figure 7

Crack velocity [m/s] vs. Stress intensity factor [MN/m^{3/2}] for Kushiro sandstone.

- ○, △: 291K, 25-29%
- +, ×: 293K, 54-55%
- ●, ▲: 293K, 85-88%
Figure 13a

Berea sandstone (relative humidity: 50%)
Shirahama sandstone (relative humidity: 50%)
Figure 14a
Berea sandstone (in water)

- Open circles, triangles, and squares: 325K, pH=5
- Filled circles, triangles, and squares: 293K, pH=6

Crack velocity [m/s]

Stress intensity factor [MN/m^{3/2}]
Figure 14b

Crack velocity [m/s] vs. Stress intensity factor [MN/m$^{3/2}$] for Shirahama sandstone (in water).

- Open circles, triangles, and squares: 325K, pH = 5 - 6
- Filled circles, triangles, and squares: 293K, pH = 6