VISUALIZATION OF INTERNAL DAMAGE OF ROCKS SUBJECTED TO THERMAL SHOCKS UP TO 800°C BY X-RAY CT IMAGING TECHNIQUE AND THEIR EFFECT ON MECHANICAL PROPERTIES

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Rocks may be subjected to thermal shocks in nature due to such as fires, explosions. There are some experimental studies on the effect of thermal shocks on the mechanical properties of rocks. These experimental results clearly showed that the mechanical properties such as strength, deformation modulus decreases while their porosity increases. The authors utilized X-Ray CT imaging technique to observed the internal damage caused by thermal shocks by varying temperatures from room temperature to 250, 500 and 800°C for about 6 hours and cooled down. Some physical characteristics were measured before and after thermal shocks. Furthermore, some mechanical properties of rocks were measured and compared with those of similar samples not subjected to thermal shocks. The authors report the outcomes of these experimental results and discuss their implications in rock mechanics and rock engineering.

Key Words : *Thermal shock, internal damage, X-Ray tomography, porosity, strength, deformation modulus*

1. INTRODUCTION

The behavior of rocks and rock masses under high temperature regimes is of great concern when the structures are subjected to fires, impacts of meteorites or missiles and geothermal exploitation. It is known that rocks become softer and ductile and their strength and deformation modulus decrease as temperature increases (e.g. Cooper and Simmons 1977; Simmons and Cooper, 1978; Homand-Etienne and Houpert, 1989; Gautam et al. 2016). Furthermore, they may expand in relation to temperature increases. It is known that rocks may suffer internal damage due to the difference of thermal expansion coefficient of minerals constituting rocks. Table 1 is a list of melting/decomposition and expansion coefficient of some common minerals relevant to this study. As noted from the table, temperature greater than 600 °C is a threshold value for internal structure changes leading to damage or decomposition of rocks. Therefore, the selected value of temperature for six hour long thermal shock tests were 250, 500 and 800 °C, and the state of samples at room temperature (20-25 °C) was selected as reference initial state. Rocks were granite, dolomite and tuff.

Table 1. Melting/decomposition and expansion coefficient of some common minerals.

Mineral	Melting/decomposition Temperature (°C)	Expansion Coefficient (10^{-6}/°C
Olivine, Pyroxene	1000	6.5-10.4
Quartz	573	7.5-13.7
K-Feldspar	600	8.9-15.6
Muscovite	600	9.9-13.8
Calcite	600-840	5.4-26
Dolomite	700-840	3.2-15.6

The initial and post thermal shock states of each sample were evaluated using the NIKON XT H320 X-Ray CT Scanning device in addition to the measurement of geometry and physical properties of samples. Geometry, geophysical properties and mechanical properties of samples subjected to thermal shocks are then measured and the internal damage situations are determined. The authors present the outcome of the investigations and discuss their implications in Rock Mechanics and Rock Engineering.

2. ROCK SAMPLING

Rocks are Kaore granite, Shakudani (Asuwayama) tuff and Daitojima dolomite (Figure 1) Kaore granite is pink-colored granite, which is quite rare in Japan, and obtained from a planned site of Kaore underground power house in Gifu Prefecture at a depth of about 500 m (Aydan and Kawamoto, 2001). Shakudani or Asuwayama tuff is a welded tuff in Fukui Prefecture. This tuff was used as a building stone and other purposes (Aydan 2016; Aydan et al. 2014). Dolomite samples were gathered from Kita-Daitojima in Okinawa Prefecture at a depth of 11 m in a port construction site (Aydan et al. 2018). Kita-Daitojima is located in the Pacific Ocean on a seamount rising to an elevation of 5000 m above sea-bottom. Dolomite of Daitojima is assumed to be as a result of chemical interaction of coral limestone with seawater. For each rock type, 4 samples, which could be tested in Brazilian tensile and uniaxial compression, were prepared..

3. THERMAL SHOCK APPARATUS AND THERMAL REGIMES

The kiln is capable of applying thermal shock up to 1000 ^{o}C. The temperature of the kiln was elevated to selected temperature levels before setting samples (Figure 2) In this study, samples were subjected to selected temperature levels of 250, 500 and 800 ^{o}C for a duration of 6 hours. Then, the samples were taken out of the kiln and cooled down at room temperature.

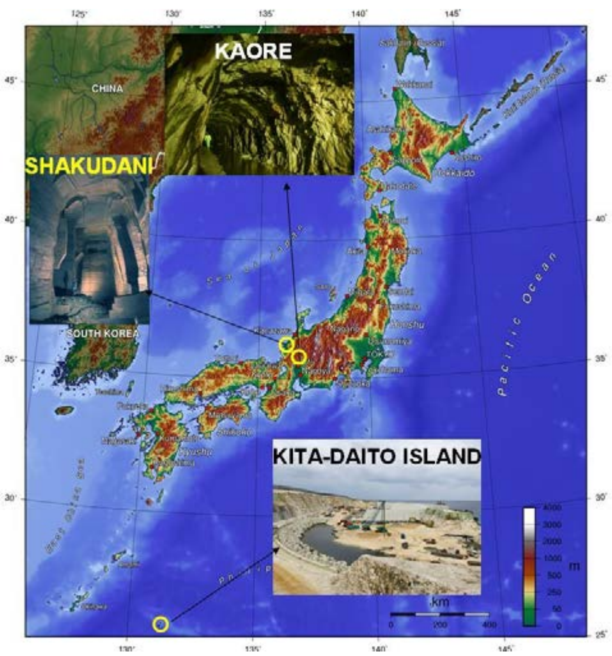


Figure 1. Locations of samples.

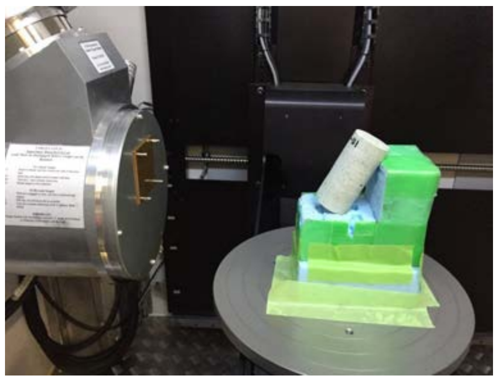
(a) Overall view of the kiln (b) Heated inside view

Figure 2. An overall and inside view of the kiln used.

4. X-RAY CT DEVICE AND OBSERVATIONS

X-ray Computed Tomography (CT) device was produced by NIKON and its type is XT H320. The maximum size of the objects is 270x183x225 cm with a minimum focus diameter of 3 μ m. Samples before and after thermal shocks were scanned by XT-H320 device and images were compared in order to see the internal damage (Figure 3).

(a) XT-H320 X-Ray CT Scanning Device



(b) Sample inside the XT-H320

Figure 3. A view of sample in XT H320.

Figures 4, 5 and 6 show the view of samples before and after thermal shocks. As noted from Figure 3, visual cracks appeared in Sample KG-U4 of Kaore granite, which was subjected to a temperature shock of Figure 4. Views of Kaore granite samples after thermal shocks of 800 ^{o}C. These cracks are assumed to be inherent planes of granite such as flow, grain and hardway planes. However, no visible external damage was observed at other samples subjected to temperature 500 ^{o}C or less. As for Shakudani tuff, there was no visible crack on the outer surface of samples while color changes were observed (Figure 5). The Kita-Daitojima dolomite sample subjected to 800 °C lost its integrity and it became fragmented or powder as seen in Figure 6.

Figure 4. Views of Kaore granite samples after thermal shocks except KG-U1 sample.

Figure 5. Views of Shakudani tuff samples after thermal shocks except S-U1 sample.

Figures 7 and 8 show the X-Ray CT images of KG-U4 and S-U4 samples before and after thermal shocks. The cracks visible on the outer surface of the KG-U4 sample were visible in X-Ray CT images and they were very extensive. The cracks in KG-U4 coincided with flow and grain planes. As for S-U4 sample, there were internal cracks which could not be observed on the outer surface of the sample.

Figure 6. Views of Daitojima dolomite samples after thermal shocks except KDD-U1 sample.

Figures 9 and 10 show 3D views of the damage intensity in KG-U4 and S-U4 samples, respectively. The intensity scale shows the aperture of cracks. The volumetric crack opening was quite large in KG-U4 compared with that of S-U4.

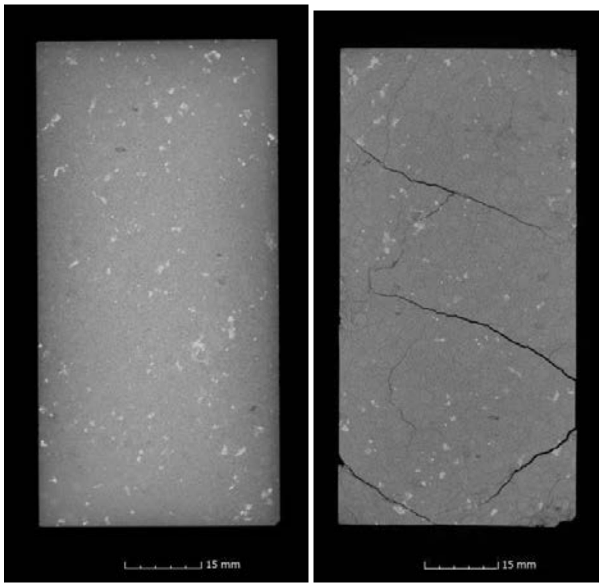


Figure 7. X-Ray images of KG-U4 sample before and after thermal shock.

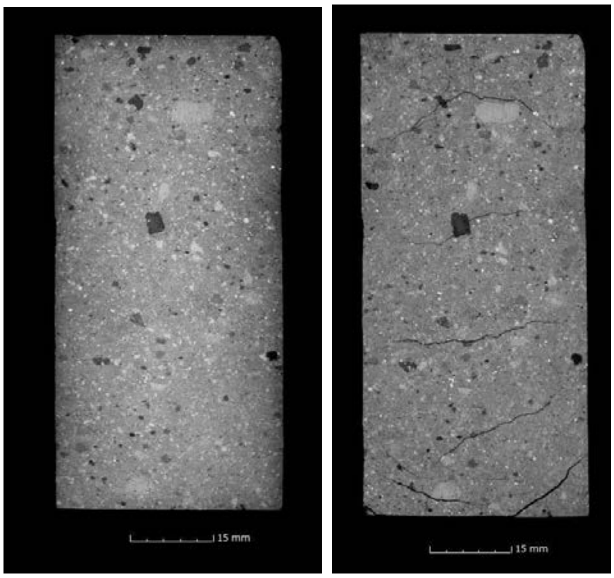


Figure 8. X-Ray images of S-U4 sample before and after thermal shock

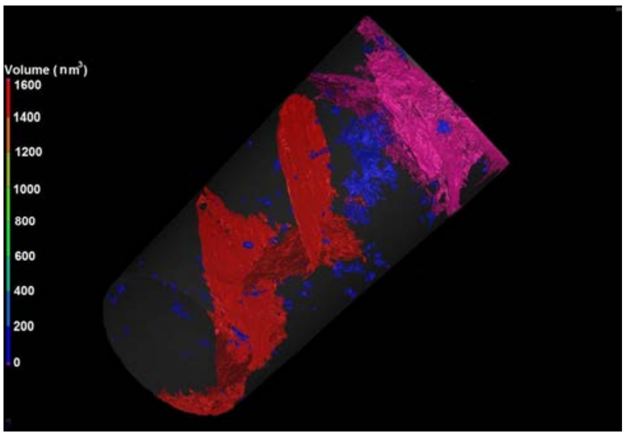


Figure 9. 3D view of damage in Sample KG-U4.

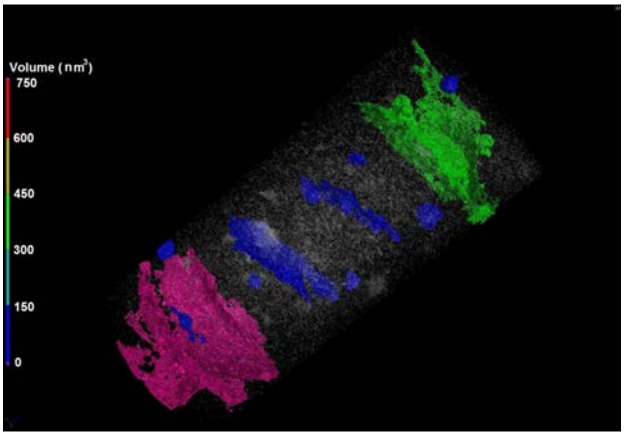


Figure 10. 3D view of damage in Sample S-U4.

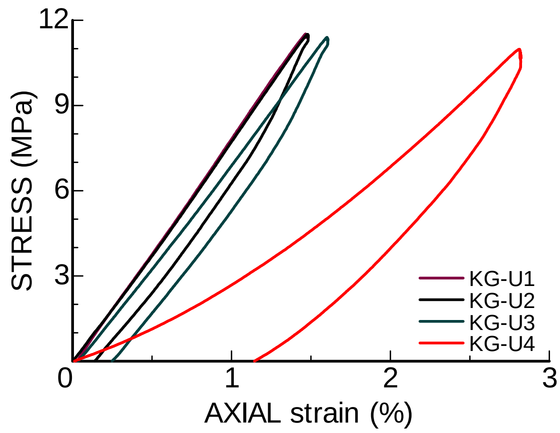


Figure 11. Strain-stress responses of Kaore granite samples

5. EFFECT OF THERMAL SHOCKS ON PHYSICO-MECHANICAL PROPERTIES OF ROCKS

Internal damage is likely to have some influence on physico-mechanical properties. The samples kept at the room temperature may be a reference to other samples.

4.1 Kaore Granite

Table 2 gives unit weight, p and s-wave velocity before (BTS) and after thermal shock (ATS) for Kaore granite samples. As noted from the table, the thermal shock has a pronounced effect on the physical properties. Generally, it can be said physical properties decreases as the temperature increases. Furthermore, thermal shock causes some permanent straining, which could be a measure of internal damage. The amount of the permanent strain increases as the temperature level increases.The unit-weight change also implies the macroscopic porosity increase due to internal cracking as well as relative slip among grains. Figure 11 shows strain-strain relation for each respective samples. As noted from the figure, the deformation modulus of KG-U4, which is subjected to 800 °C, is greately reduced. The deformation modulus of KG-U3 sample subjected to 500 °C also becomes smaller while the deformation modulus of KG-U2 sample subjected to 250 °C is almost the same. The results also imply that igneous rocks like granite are vulnerable to be influence by thermal shocks 500 °C or higher temperature levels.

Table 2. Physical properties of Kaore granite samples.

No	γ (kN/m³)		V_p (km/s)		V_s (km/s)		ε_r (%)
	BTS	ATS	BTS	ATS	BTS	ATS	
KG-U1	25.6	25.6	4.75	4.75	2.53	2.53	0
KG-U2	25.7	25.4	4.80	3.48	2.70	2.26	0.074
KG-U3	25.2	25.2	4.56	2.85	2.49	1.96	0.168
KG-U4	25.4	25.4	4.82	0.69	2.68	0.22	2.00

4.2 Shakudani (Asuwayama) Tuff

Table 3 presents unit weight, p and s-wave velocity before (BTS) and after thermal shock (ATS) for Shakudani tuff samples. As noted from the table, the thermal shock has a pronounced effect on the physical properties. The reduction of s-wave velocity is much higher than that of p-wave velocity of samples. Similarly, it can be said physical properties decreases as the temperature increases. Furthermore, thermal shock causes some permanent straining in case of Shakudani tuff, particularly the sample S-U4 indicates great amount of permanent straining. Figure 12 shows strain-strain relation for each respective samples. It is interesting to note that defromation responses of samples S-U2 and S-U3 subjected to 500 °C or lower temperature levels.imply that the increase of temperature increases result in stiffer response.

Table 3. Physical properties of Shakudani tuff compression samples.

No	γ (kN/m³)		V_p(km/s)		V_s(km/s)		ε_r (%)
	BTS	ATS	BTS	ATS	BTS	ATS	
S-U1	20.3	19.8	2.97	2.97	2.29	2.29	0
S-U2	19.9	19.9	3.04	2.97	2.30	2.13	0.15
S-U3	19.9	19.9	3.06	2.83	2.22	2.01	0.04
S-U4	19.9	19.4	3.06	1.51	2.21	0.89	1.63

Table 4 gives the physico-mechanical properties of Shakudani tuff samples for Brazilian tensile strength experiments. The overall tendency regarding the effect of thermal shocks are quite similar to those of compression experiments (Figure 13). As noted from Table 4, some shrinkage type volumetric strain occurs for S-U2 sample subjected to 25 °C. Nevertheless, tuff samples, which are originally ash-fall deposits, become much stiffer and stronger for temperature levels less than 500 °C.

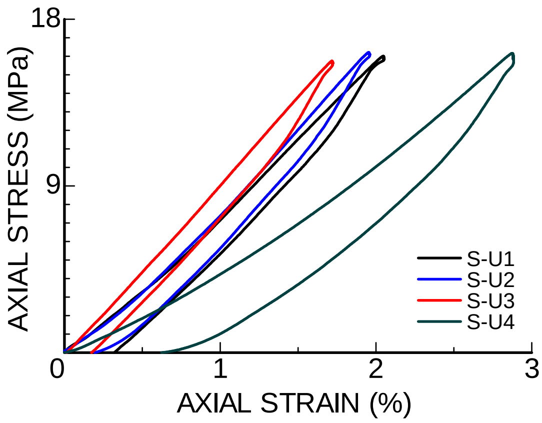


Figure 12. Strain-stress responses of Shakudani tuff samples subjected to compression.

Tensile strength of samples S-U2 and S-U3 were higher than than S-U1 sample, which was not subjected to any thermal shocks. However, higher temperature starts to induce some internal damage so that overall physico-mechanical properties tends to decrease. The deformation modulus and tensile strength increases may be related to this fact. However, the pysico-mechanical properties of the sample subjected to a temperature shock of 800 °C starts to decrease, drastically.

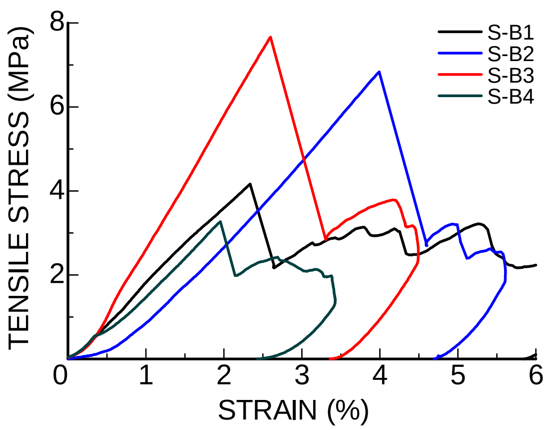


Figure 13. Strain-stress responses of Shakudani tuff samples in Brazilian tests.

Table 4. Physico-mechanical properties of Shakudani tuff Brazilian test samples

No	γ (kN/m³)		V_p (km/s)		V_s (km/s)		σ_t MPa	ε_r (%)
	BTS	ATS	BTS	ATS	BTS	ATS		
S-B1	19.2	19.2	3.21	3.21	2.32	2.32	4.16	0
S-B2	19.9	19.9	3.27	3.20	2.41	1.15	6.84	-0.83
S-B3	19.9	19.9	3.12	2.48	2.40	0.68	7.66	0.71
S-B4	19.9	18.9	3.01	1.71	2.36	0.66	3.37	2.66

4.3 Daitojima Dolomite

Daitojima sample subjected to a temperature shock of 800 °C decomposed and no further experiments could be carried out. Table 5 presents unit weight, p and s-wave velocity before (BTS) and after thermal shock (ATS) for Daitojima dolomite. The reduction of s-wave velocity is much higher than that of p-wave velocity of samples. As noted from the table, the thermal shock greater 250 °C than has a pronounced effect on the physico-mechanical properties. Physical properties also decrease as the temperature become greater than 250 °C. Similarly, thermal shock causes some permanent straining in dolomite samples. Figure 14 shows strain-strain relation for each respective samples subjected to compression. Again it is noted that deformation modulus decreases as the temperature level becomes greater than 250 °C.

Table 5. Physical properties of Daitojima dolomite compression samples

Sample No	γ (kN/m³)		V_p (km/s)		V_s (km/s)		ε_r (%)
	BTS	ATS	BTS	ATS	BTS	ATS	
KDD-U1	24.9	24.9	5.48	5.48	2.50	2.50	0
KDD-U2	25.6	25.4	5.96	5.79	3.12	2.46	0.35
KDD-U3	25.4	25.1	5.93	2.83	3.57	1.97	0.53
KDD-U4	25.4	-	6.10	-	3.22	-	-

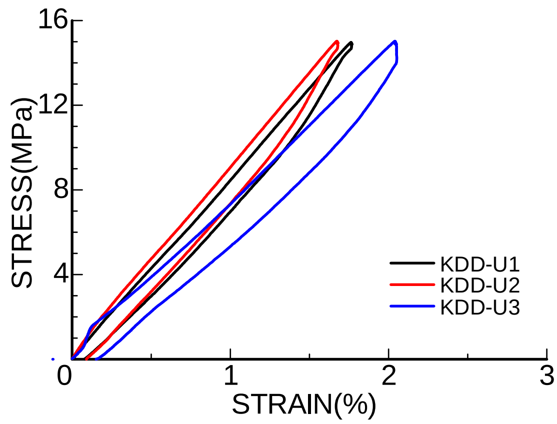


Figure 14. Strain-stress responses of Daitojima dolomite samples subjected to compression

Table 6 gives the physico-mechanical properties of Daitojima dolomite samples for Brazilian tensile strength experiments. The overall tendency regarding the effect of thermal shocks are quite similar to those of compression experiments. Although the sample for 500 °C was accidentally fracture before any recording, the pyhsico-mechanical properties of the sample subjected to a temperature shock of 800 °C was drastically decreased as seen in Figure 15.

Table 6. Physico-mechanical properties of Daitojima dolomite Brazilian test samples

Sample No	γ (kN/m^3)		V_p (km/s)		V_s (km/s)		σ_t MPa	ε_r %
	BTS	ATS	BTS	ATS	BTS	ATS		
KDD-B1	25.4	25.4	6.0	6.0	1.7	1.7	11.5	0.0
KDD-B2	25.8	25.2	5.8	4.7	1.7	1.4	No Data	0.9
KDD-B3	25.3	24.5	5.5	4.0	1.7	1.3	2.62	1.0
KDD-B4	25.5	-	4.9	-	1.9	-		-

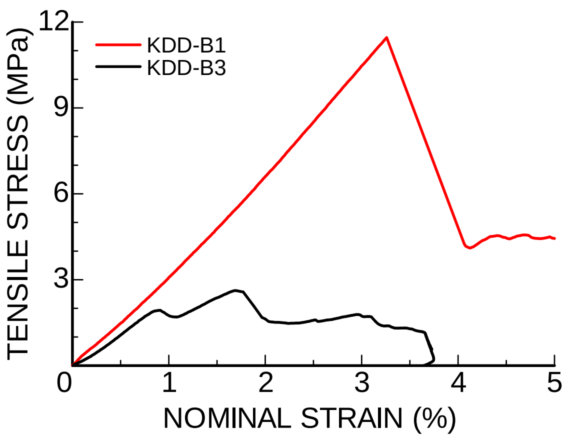


Figure 15. Strain-stress responses of Daitojima dolomite samples in Brazilian tests.

5. CONCLUSIONS

The authors have undertaken a study to investigate the effect of thermal shocks on physico-mechanical properties of granite, tuff and dolomite. The selected values of temperature were 250, 500 and 800 °C, for thermal shock tests for a duration of siz hours. The initial and post thermal shock states of each sample were evaluated using the NIKON XT H320 X-Ray CT Scanning device in addition to the measurement of geometry and physical properties of samples. X-Ray CT images clearly showed the internal damage in sample and likely locations of damage. Inherent weakness planes within the sample eventually lead to internal damage and cracking. These damages in turn result in the reduction of physico-mechanical properties of tested rocks. However, it is also noted that the temperature up to certain levels may result in healing effects as it happened in Shakudani tuff samples. Although further studies and experiments are necessary, it is shown that X-Ray CT technique is an effective non-destructive tool to investigate the internal damage without any disturbance to samples and the field of Rock mechanics and Rock Engineering should effectively use this technique to its further advances.

REFERENCES

Aydan, Ö. (2016). Time Dependency in Rock Mechanics and Rock Engineering, CRC Press, Taylor and Francis Group, 241p.

Aydan, Ö. and Kawamoto, T. (2001): The stability assessment of a large underground opening at great depth. 17th Int. Min. Congress and Exhibition of Turkey, IMCET 2001, Ankara, Vol.1, 277-288.

Aydan, Ö., Sato A., Yagi. M. (2014): The Inference of Geo-Mechanical Properties of Soft Rocks and their Degradation from Needle Penetration Tests. Rock Mechanics and Rock Engineering, 47:1867ï 1890.

Aydan, Ö., Watanabe, E., Tokashiki, N. (2018): The evaluation of crustal stresses in Ishigaki and Kitadaito Islands and their close vicinity (in Japanese). 45th Rock Mechanics Symposium of Japan, 190-195.

Cooper, H.W. and G. Simmons, 1977. The effect of cracks on the thermal expansion of rocks. Earth and Planetary Science Letters, vol. 36, pp. 404ï 412.

Gautam, P.K., A. K. Verma, M. K. Jha, K. Sarkar, T. N. Singh, and R. K. Bajpai, 2016. Study of strain rate and thermal damage of Dholpur sandstone at elevated temperature. Rock Mechanics and Rock Engineering, 49(9,) 3805ï 3815.

Homand-Etienne, F. and R. Houpert, 1989. Thermally induced microcracking in granites: characterization and analysis. International Journal of Rock Mechanics and Mining Sciences and Geomechanics Abstracts, 26,(2), 125ï 134.

Simmons, G. and H. W. Cooper, 1978. Thermal cycling cracks in three igneous rocks. International Journal of Rock Mechanics and Mining Sciences & Geomechanics Abstracts, 15(4), 145ï 148.