STRENGTHENING COUNTERMEASURES AND QUALITY ESTIMATION OF BEDROCKS IN MASTABA IDOUT, EGYPT

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Abstract

As part of the conservation and restoration work of the Center for the Global Study of Cultural Heritage and Culture (CHC), this study explores the strengthening countermeasures and the quality estimation of rock at Mastaba Idout in Saqqara, Egypt.

The Mastaba Idout was built around 2360BC. The bedrock of this unique underground burial chamber is known to be weak and, as such, an investigation of its condition and quality and an examination of various strengthening methods are reported herein. The results of quality testing for four kinds of strengthening agents for stone in Japan are also examined. The results of quality testing and repairs for the bedrock in Egypt and the results of the quality testing of strengthening agents for dirt in Japan are reported. Quality testing measures the frequency of the accelerating wave generated by striking a rock surface with an impact hammer. The repair methods involve strengthening by coating with a strengthening agent for stone and injecting non-shrink cement slurry into cracks.

This research identified that the site's bedrock was of very low quality and that the strengthening methods were effective. Quality testing of the strengthening agents for dirt found that agent OH100 had good water permeability, good moisture permeability, and minimal negative effects on the compressive strength of rocks.

1. Introduction

As part of the conservation and restoration work of the Center for the Global Study of Cultural Heritage and Culture (CHC) of Kansai University, this study provides a quality estimation of the bedrock at Mastaba Idout in Saqqara in the Arab Republic of Egypt. CHC conducts ongoing research related to the conservation and preservation of cultural heritage at the burial chamber of Mastaba Idout.

The burial chamber was discovered in 1927 by Cecil Firth, and was first reported in the literature in 1935 by Macramallah[1)]. This site includes above-ground structures (including a

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chapel, storehouse, etc.) and an underground burial chamber. It is part of the pyramid complex of King Unis, located to the south of the Step Pyramid of King Djoser, which is the main monument at the Saqqara Necropolis archaeological site. It was built for Ihy around 2360BC (early in the sixth dynasty of the Old Kingdom), and was later used as the burial chamber of Mastaba Idout, who was the princess of King Teti or King Unis. The walls of the tomb and its burial chambers were decorated with brilliant colors, which have retained their original brilliant hues. There are some weak parts, called the marl layer, on the upper wall and ceiling of the burial chamber, and the separation and peeling of the wall paint from the bedrock surface is concerning. The main rocks in the chamber are marl and limestone and, on the whole, their weathering is proceeding[2].

We are researching the condition and quality of stonework blocks in two places in order to obtain effective data that will enable the development of technologies to preserve and restore the ruins in the future. First, the quality of the bedrock in Mastaba Idout was estimated. Next, reinforcement by application of a bedrock strengthening agent to a fragile part and the filling of a crack with cement slurry were tried. This ancient tomb is located underground a desert area, and the mother rock is thus weak and prone to collapse. As such, both restoration of the murals and reinforcement of the mother rock is needed. The main characteristics required for a restorative adhesive are high permeability, high strength, and solvent-free. In this paper, the high permeability and strength of the repaired rock were considered required qualities. We employed nondestructive examination to study the effect of the reinforcement measures, performed a quality evaluation test on the effect of the bedrock strengthening agent, and identified the special qualities of this agent. Lastly, we quantitatively and objectively evaluated the reinforcement measures and the quality evaluation technique applied herein.

2. Testing in Egypt

2.1 Materials

Bedrock reinforcement and crack repair were considered reinforcement measures. The bedrock was repaired and reinforced using the following two materials:

1) A reinforcement and preservation agent for dirt and stone (product of A company, hereafter referred to as "Agent A"). Ethyl alcohol is added to an alkoxide solution that contains silicon as an agent; it is then hydrolyzed and made into polycondensate.

2) Non-shrink cement (product of B company)

The cement slurry made using this is highly fluid, which enables it to fill gaps with complicated shapes.

2.2 Application areas

Fig. 1 shows the roof-ceiling assembly and the side wall part of the underground burial chamber of Mastaba Idout where the above-mentioned two kinds of materials were applied. A reinforcement preservation agent for dirt and stone was used for the roof-ceiling assembly, and cement slurry was used for a crack in the side wall.

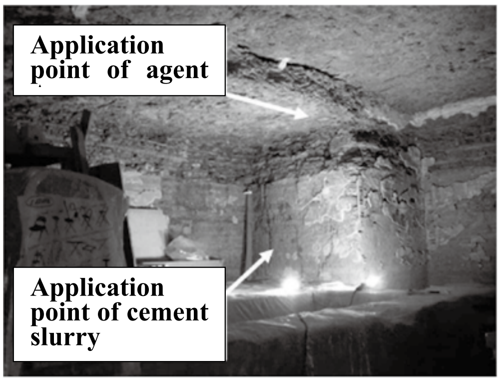


Fig. 1 Application point in burial chamber

Fig. 2 Construction status of Agent A

2.3 Method

1) Agent A

Agent A was applied repeatedly using a brush until the coating reached 3 kg/m^2 on the rock surface of the roof-ceiling assembly of the underground burial chamber (Fig. 2). The application shape was a square of 100 mm × 100 mm, as shown in Fig. 3.

2) Non-shrink cement slurry

A cement slurry water-binder ratio (W/B) of 18% was achieved by mixing 0.2 kg of non-shrink cement and 0.036 kg of water. The application area was the crack shown in Fig. 4, the width of which was, at most, approximately 10 mm. The day before the application, water was poured into the crack, and the part where water leaked (sealed part in Fig. 4) was sealed with cement slurry via a syringe on the day of application. The slurry was poured into a hose placed in the upper part of the crack (the filled part on Fig. 4), as shown in Fig. 5. When the cement slurry overflowed from the upper part of the cracked portion, the crack was deemed to have been filled with the cement slurry.

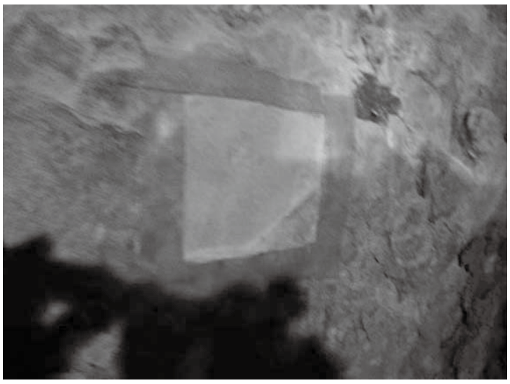


Fig. 3 Application point of Agent A

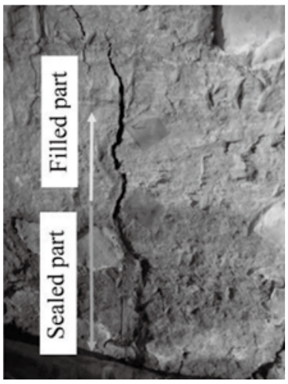


Fig. 4 Application point of cement slurry

Fig. 5 Construction status of cement slurry

2.4 Estimation methods

1) External observation

A change in color and spalling circumstances at the coated surface were confirmed at the application area for Agent A. At the cement slurry application area, differences between the hardened cement slurry and the surrounding bedrock, namely, the presence of drying shrinkage and peeling at the filling part, were confirmed.

2) Frequency measurement using an impact hammer

Fig. 6 shows the commercial measuring device for frequency and wave shape (product of C company) that was used to confirm the effect of the repair and reinforcement at the application areas for Agent A and cement slurry. By striking the target surface with the impact hammer and recording the wave with an acceleration sensor, this device captures the predominant frequency and wave shape at the main device. Fig. 7 shows an example of the location of the acceleration sensor and the striking points by the impact hammer in the main test. In the figure, "※" indicates the location of the acceleration sensor and "×" shows the striking points by the impact hammer. In the test, the distance between the acceleration sensor and the hitting points differed from the test conditions, and was almost 10 cm. A large frequency value indicates that the texture of the measured object is dense and of high quality, and a small frequency value indicates that the texture of the object is coarse and of poor quality.

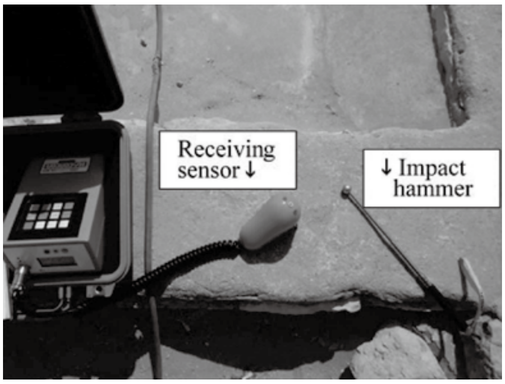


Fig. 6 Frequency measuring equipment

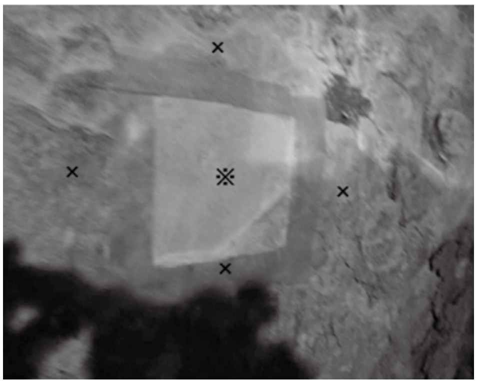


Fig. 7 Example of striking location in measuring frequency

3. Test Results and Discussion

3.1 Results of external observation

In the Agent A application, the color of the coated surface became slightly whitish, but no marked discoloration or detaching was observed. Cement slurry was used to fill a crack, and the crack was to subsequently be buried. The difference between the color of the surrounding bedrock and the cement slurry was slightly conspicuous, but no shrinkage cracks or detaching were observed in the area that was filled. When checked 5 months later, no abnormalities were observed.

3.2 Results of measuring frequency in the underground burial chamber

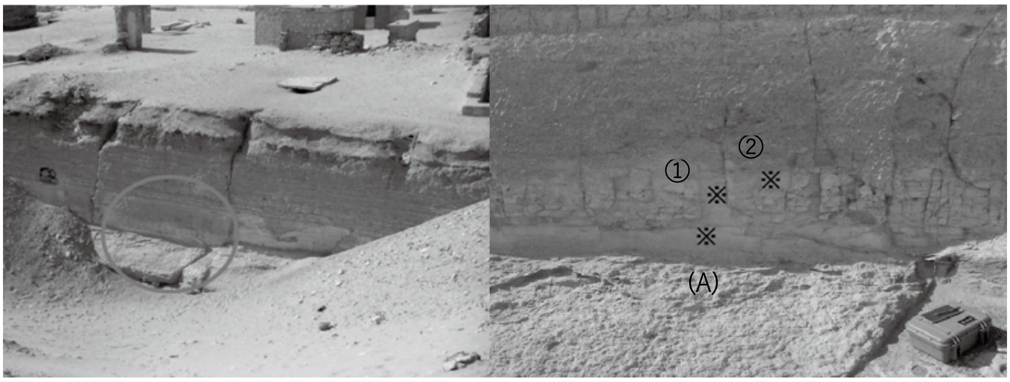


Fig. 8 Outdoor measurement point (Right picture: Enlargement of circle in left picture)

Table 1 Outdoor measurement results

	(A) High-quality part	①Slightly degraded part	②Vulnerable part
Measured predominant frequency (Hz)	3,984 3,633 4,336 3,516 3,867	3,652 3,144 2,956 2,820 2,507	1,349 1,505 576 1,281 2,562
Average values (Hz)	3,867	3,016	1,455
Standard deviation (Hz)	286	380	639

1) Outdoor bedrock around the burial chamber

Before conducting testing in the underground burial chamber, preliminary tests were performed on the surrounding aboveground bedrock. Fig. 8 indicates the area where we took measurements from—it was of similar height as the underground burial chamber. (A) was high-quality bedrock, ① was slightly deteriorated bedrock, and ② was very weak bedrock. A receiving sensor was set at "※", and measurement was conducted by striking with an impact hammer at a point 5 to 15 cm away from "※". Table 1 shows the measurement results. According to the table, the measured values grew smaller in the order of (A) high-quality area, ① slightly deteriorated area, and ② weak area. The tendency in which variations increase was determined from the standard deviation, and increased in the order (A), ①, and ②. This also indicates the quality deterioration in the bedrock.

2) Burial chamber ceiling bedrock

Testing was carried out on the ceiling bedrock examined in Section 2.2. Measurements were conducted before and after coating with Agent A to examine its effects. The receiving sensor was set to "※", as shown in Fig. 7. Measurement was carried out by striking "×" using an impact hammer. Table 2 shows the results. The measured frequencies were increased by 1.4 times after coating. The quality of the bedrock surface improved slightly as a result of coating with Agent A, and a stiffening effect was observed.

Table 2 Measurement results for the ceiling of the underground burial chamber

	Before coating with Agent A	After coating with Agent A
Measured predominant frequency (Hz)	1,250	1,328
	820	2,070
	1,328	1,484
	1,172	1,875
	742	938
Average values (Hz)	1,062	1,539
Standard deviation (Hz)	236	401

3) Burial chamber side wall bedrock

The wall that was being restored was also tested, and the measured predominant frequency was 3,406 Hz, which was not low quality. This was judged from the relationship between the appearance of the bedrock and the predominant frequency of the high-quality area shown in Table 1. A repair with non-shrink cement slurry was performed, and its effect was examined by frequency measurement of the wall area where a large crack had generated (Fig. 1). The receiving sensor was placed in the left side of the crack, hitting five points on the right side of the crack, and the frequency was measured as shown in Fig. 9. "※" indicates the location of the receiving sensor, and "×" indicates the part that was struck in this figure. Table 3 shows the measured results, and Fig. 10 shows the crack that was filled with cement slurry. The table shows that the predominant frequency was changed before and after filling with cement slurry. It was also found that the surface was filled with cement slurry densely and the propagation of waves was improved. An increase in frequency was not, however, evident at the sealed area. The filling of the non-shrink cement slurry gradually moved to the upper part while small amounts were gradually poured in from the upper part of the sealed part. Therefore, it is considered that only the surface of the sealed portion was filled, and both the surface and the inside of the filled portion are filled with cement slurry.

Fig. 9 Status of measurement point

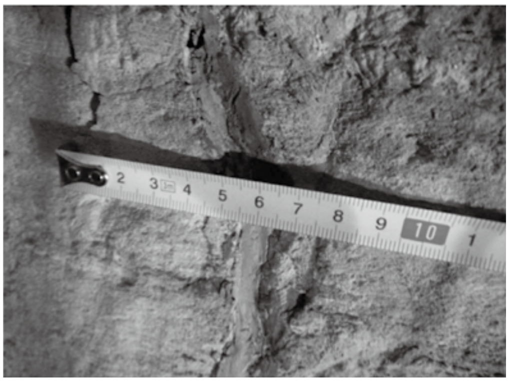


Fig. 10 Status of crack filled with cement slurry

Table 3 Measurement results on the wall of the underground burial chamber

	Filled part		Sealed part	
	Before filling	After filling	Before filling	After filling
Measured predominant frequency (Hz)	2,031	2,852	1,719	1,641
	1,602	2,305	1,328	1,250
	1,641	2,891	2,070	1,523
	1,680	3,594	1,719	1,602
	1,694	2,266	2,188	1,445
Average values (Hz)	1,730	2,782	1,805	1,492
Standard deviation (Hz)	154	484	303	139

4. Quality Testing of Reinforcement Preservation Agent for Dirt and Stone in Japan

In this chapter, quality testing is conducted on reinforcement preservation agents for dirt and stone, including TOT (Agent A, which was used in Chapters 2 and 3), and OM50, OM25, and OH100 (which Professor Nakamura previously used in Mastaba Idout)[3)4)].

4.1 Examined agents

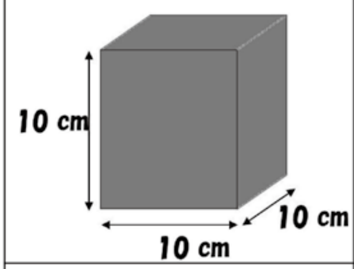
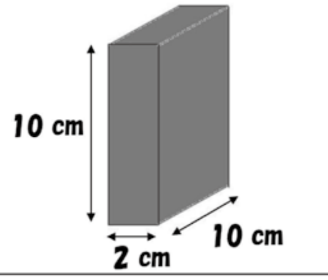
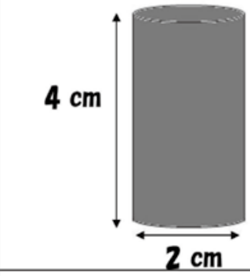
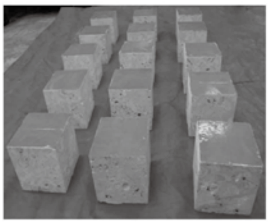
Four kinds of reinforcement agents were investigated, namely, OH100, TOT (Agent A), OM25, and OM50, all of which are alkoxide silane-type materials. OM25 and OM50 are high-strength types of OH100.

4.2 Specimen manufacture method

Here, specimen manufacture was carried out in accordance with JSCE-K571 2010, "Test method of surface penetrant materials," which addresses testing for surface protection methods for concrete structures. Three kinds of tests were conducted, namely, the water penetration test, moisture transmission test, and compressive strength test. Three kinds of test specimens were used for each test. The original specimens were Ryukyu limestone cubic specimens (100 mm × 100 mm × 100 mm) (Fig. 11), which were used as-is for the water penetration test. In the moisture transmission test, specimens were cut to 100 mm × 100 mm × 20 mm with a diamond cutter. For the compressive strength test, Φ20 × 40 mm of cores were extracted using a core drill from the cubic specimens. Table 4 shows the shape and size of specimens for each test. Ryukyu limestone was selected as the rock species closest to the target bedrock of Egypt.

Fig. 11 Ryukyu limestone specimens

Table 4 Dimensions and appearance of various test specimens

For water penetration test	For moisture transmission test	For compressive strength test
		
		

The flow of specimens for the water penetration test and the compressive strength test is as follows. First, specimens were cut into fixed sizes. Epoxy resin was used to seal them all except for the test face, and agents were coated on the specimens' test face. Next, specimens were cured for 7 days in air at 20°C and 60% relative humidity in a curing room. Testing commenced after curing. In the moisture transmission test, specimens were treated similarly to the above after coating with an agent. Three specimens per agent were tested.

The surface with fewest voids was selected as the coating surface and tested, because Ryukyu limestone was porous and had many surface voids. The amount of coating agent was not clarified except for in TOT, and was determined to be 200 g/m^2, which is a standard amount of coating used as a surface penetrant for concrete structures. The amount of coating for TOT was 2000 g/m^2 according to the coating manual. For the compressive strength test, every specimen surface was coated by every agent. The surface moisture content for test specimens was measured using a surface moisture meter for concrete and mortar, and it was measured before coating with JSCE-K 571. The range of surface moisture content was determined to be 1.3 to 2.1%.

4.3 Testing methods

4.3.1 Water penetration test

The equipment for this test consisted of an infundibulum and a 50 ml measuring pipet. The funnel and measuring pipet were glued together by underwater bonding, fixed by vinyl tape, and were then glued to specimens by underwater bonding (Fig. 12).

The test was carried out by following Section 7.12 of JIS A 6909. The infundibulum and the measuring pipette were placed on the test surface so that there was no gap between them. The diameter of the infundibulum was 75 mm, and the minimum scale of the measuring pipette was 0.05 ml. Distilled water was added to the apparatus, and it was kept at a constant temperature (20°C ± 2°C) and humidity (60% ± 5%) for the 7 days of the testing procedure. In the concrete test, the amount of water penetration over 7 days was minimal. As Ryukyu limestone has many voids and uneven surfaces, the water penetration speed is very fast and the test method was thus changed as follows. In the case of high water penetration, water leaked from the bottom of the specimens soon after starting the test. To address this, specimens were placed in a vat, and the amount of water penetration was measured by controlling the amount of leaked water and time. The many voids in Ryukyu limestone are connected to the interior of the specimens, and water started leaking from different voids as a result of changing the water in the fixed-height test equipment. Fig. 13 shows the water leakage. In this test, the test time was 1 h not 7 days.

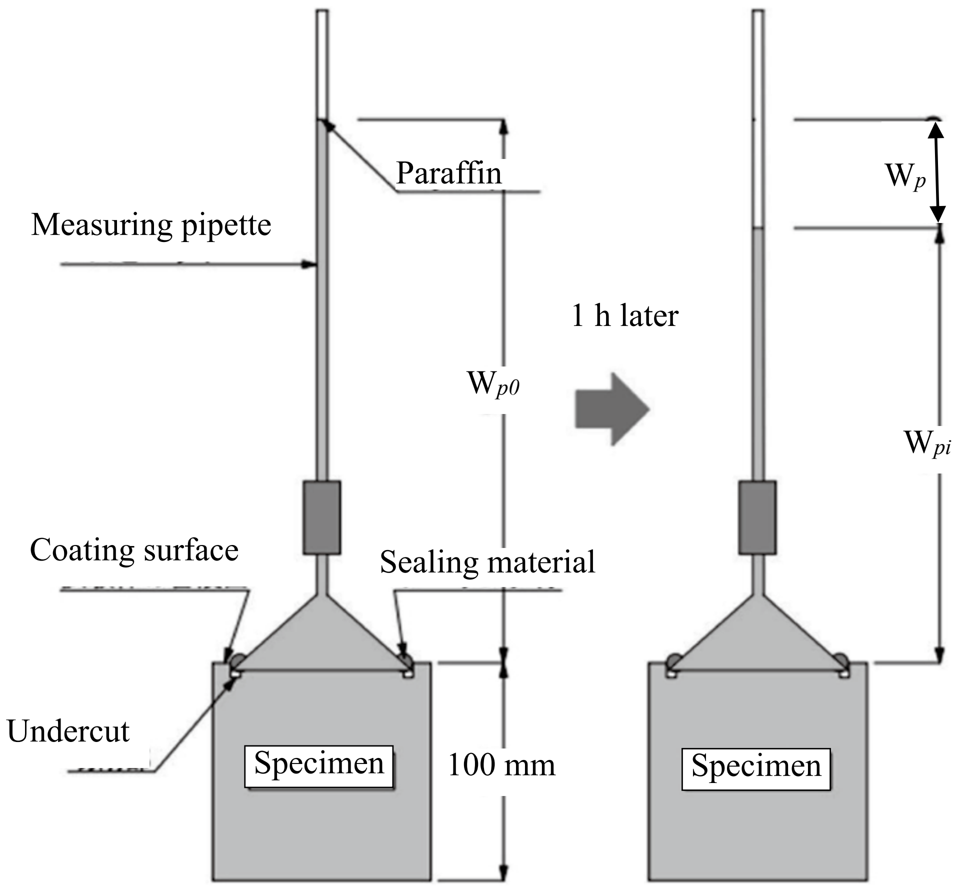


Fig. 12 Water penetration test device

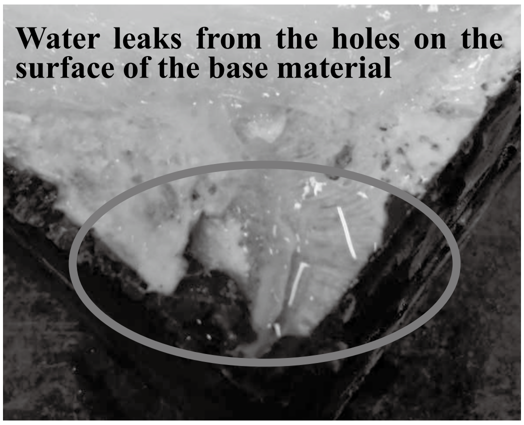


Fig. 13 Leakage

One hour after the start of the test, the height of the water head (W_{pi}) was measured, and the amount of water penetration (W_p) was calculated using equation (1), which determines the difference between the height of the water head at 1 h and 0 (W_{po}) as

$$W_p = W_{pi} - W_{p0} \quad . \qquad (1)$$

In addition, water permeability was estimated by this value.

4.3.2 Moisture transmission test

This test was carried out by the method shown in Fig. 14. Test specimens were soaked in water, and water was absorbed from the bottom surface for 72 h. After finishing the water absorption, test specimens were wiped with a dry towel, and the water-absorbed surface was sealed with adhesive tape. The mass of the sealed specimens was measured to the nearest 0.01 g, and this value (W_{v0}) was considered to be the mass of specimens before the moisture transmission test. Next, the specimens were placed on a test surface for up to 7 days under constant test surface conditions (temperature of 20°C, relative humidity of 60%). After 7 days, the mass of the specimens (W_{vi}) was measured and the moisture transmission (W_v) value was calculated. Wv was determined as the average of three test values and was rounded to two decimal points.

$$W_v=W_{v0}-W_{vi} \qquad (2)$$

The moisture permeability was calculated by equation (3) from W_v of the test specimens and the non-coated specimens, and was rounded to an integer.

The moisture permeability (%) = W_v of test specimens / W_v of non-coated specimens×100 (3)

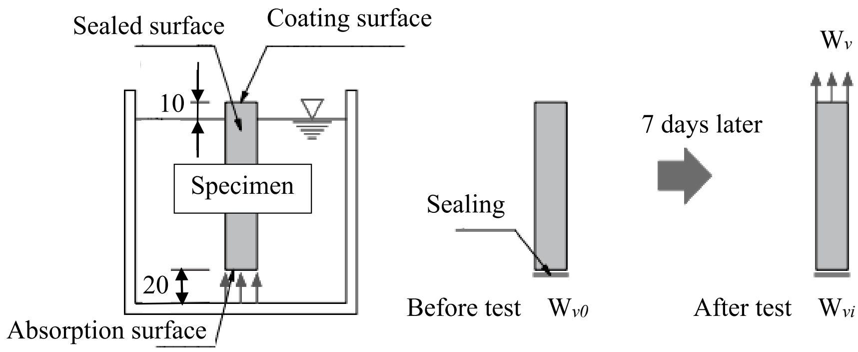


Fig. 14 Moisture transmission test flow

4.3.3 Compressive strength test

A compressive strength test was carried out following JIS A 1107, and the core specimens were extracted with a core drill from the cubic rock of Ryukyu limestone. The core specimens had a diameter of 20 mm and were coated by some of the agents. The compressive strength was calculated by equation (4) and rounded to three digits:

$$fc = P / \pi / (d/2)^2 \qquad (4)$$

fc : compressive strength (N/mm^2)

P : Maximum load (N)

d : Diameter of specimens (mm)

4.4 Test results

4.4.1 Water permeability test

Fig. 15 shows the results of the water penetration test. The average values are shown by a bar chart, and maximum and minimum values are shown by black lines. Fig. 16 shows the most effective results among all four cases. The results reveal that the test data varies widely. This is because Ryukyu limestone has a highly porous character and many voids. The average values of coated specimens were bigger than that of non-coated specimens, and OH100 or OM25 showed the best resistance for water permeability. Aiming at the lowest values, TOT showed the most excellent water permeability resistance.

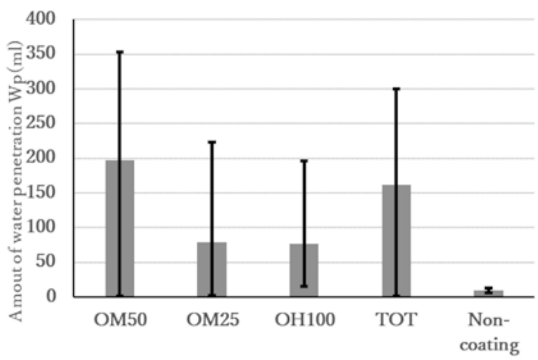


Fig. 15 Water penetration test results

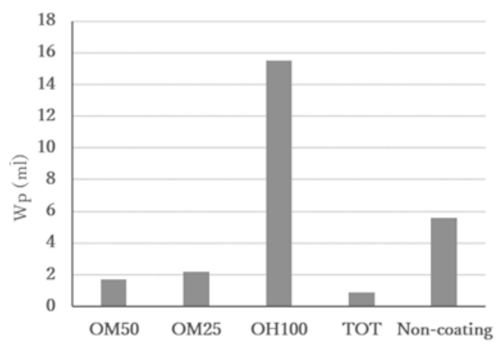


Fig. 16 Water penetration test results (best results)

4.4.2 Moisture transmission test

Fig. 17 shows the results of the moisture transmission test. The average values are shown by a bar chart, and the maximum and minimum values are shown by black lines. Fig. 18 shows the best data among the results of the moisture transmission test. In OH100 there is a large moisture transmission value for a non-coated specimen. TOT has the biggest resistance for moisture permeability.

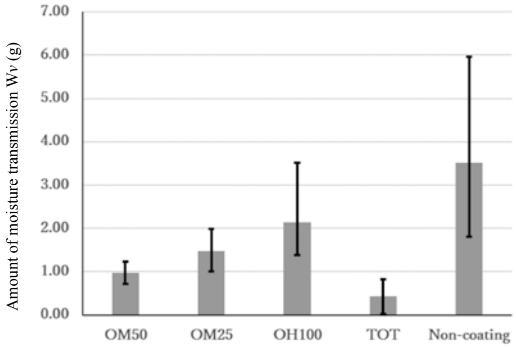


Fig. 17 Moisture transmission test results

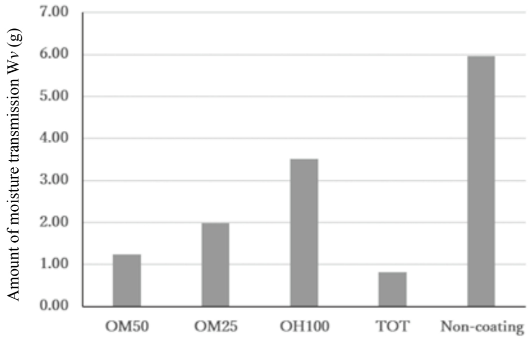


Fig. 18 Moisture transmission test results (best results)

4.4.3 Compressive strength test

Fig. 19 shows the results of the compressive strength test. The average values are shown by a bar chart, and the maximum and minimum values are shown by black lines. The results reveal that the strength of coated specimens is lower than that of non-coated specimens. This is thought to be the chemical effect of the agent, but the exact cause has not been identified. The decrease in the strength of OH100 was the lowest of all agents. The strength of coated specimens can be considered mostly equal, however, because it is dependent on the difficulty of making core specimens for Ryukyu limestone, the number of test specimens, and the large variations in strength. It is thus necessary to make a judgment in consideration of improved molding accuracy and an increased number of tests.

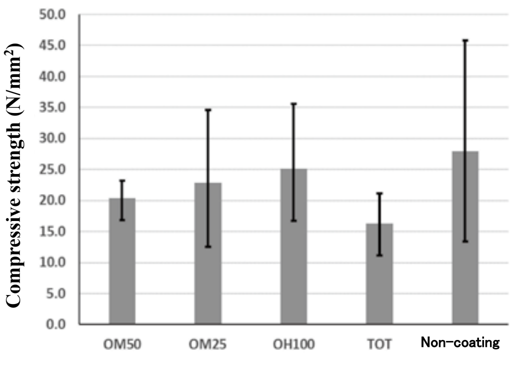


Fig. 19 Compressive strength results

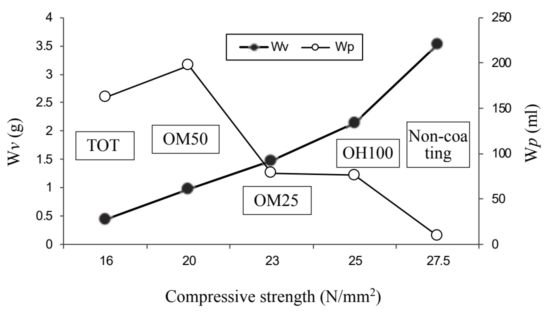


Fig. 20 The relationship among compressive strength, Wv, and Wp

Based on Sections 4.4.1 to 4.4.3 above, Fig. 20 shows the relationship among compressive strength, Wv, and Wp. The figure shows that OM25 or OH100 are suitable repair materials because they offer a good balance of suppressing water permeability, ensuring moisture permeability, and having minimal adverse effects on strength.

5. Conclusions

(1) No significant discoloration or peeling was observed at the application site of Agent A (a strengthening preservative for stones and rocks), and it was confirmed that the predominant frequency after coating was higher than that before coating.

(2) At the site where non-shrink cement slurry was applied, the predominant frequency after construction increased significantly in the crack-filled part compared to that before construction, and the repair effect was thus recognized. In addition, no drying shrinkage cracks or peeling were observed, even 5 months later.

(3) The hammer test confirmed that the quality of the bedrock of the underground burial chamber of Mastaba Idout on the walls and ceiling was lower than that of the outdoor.

(4) In a quality test conducted with Ryukyu limestone in Japan, OM25 and OH100 were considered to be suitable repair materials because they have a good balance of suppressing water permeability, ensuring moisture permeability, and having minimal adverse effects on strength.

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References

1) Macramallah, M. Le Mastaba d'Idout, Cairo (1935)

2) T. Nishigata and A. Ito, Stability Research and Restoration Work for Deterioration Mother Rock in Burial Chamber of Idout, Semawy Menu – Institute for Conservation and Restoration of Cultural Properties, Kansai University, Japan, 4, 1-7 (2012)

3) Y. Nakamura, H. Tsuruta, A. Ito, H. Suita, R. Tsutsumi, S. Okada, T. Nakamura and S. Fujii, Coagulation Strength of Some Adhesives for Conservation of Cultural Heritage, MEXT Supported Program for the Strategic Research Foundation at Private Universities, Final Report of the Center for the Global Study of Cultural Heritage and Culture, Kansai University: Comprehensive Global Studies for Conservation and Utilization of Cultural Heritage, 101-111 (2018)

4) T. Nakamura, Y. Nakane, H. Tsuruta, A. Ito, H. Suita, K. Suemori, T. Hirai, S. Fujii and Y. Nakamura, Improvement of Reinforcement Effect of Alkoxy Silanes for Conservation of Stone Cultural Assets, Journal of Adhesion Society of Japan, **56-10**, 9-14 (2020)