# 2-7. Internship Researchers



\* The 1st term started in December 2022 of FY 2022 and the 2nd term was supposed to end in March, 2023 (FY2022). However, it was postponed to FY2023 due to unavoidable reasons. Therefore, their final reports are posted in "Annual Report for FY2023".



The topic of my internship project was " Investigation of shear behavior of high-strength stirrup concrete beams after high temperatures " and I would like to describe what I gained from this internship in the following ways.

### **Research background**

For reinforced concrete structures exposed to high-temperature environmental conditions, the degree of damage to the reinforcing and concrete materials directly affects the working and safety performance of the structure. To avoid serious failure damage of the structure under high temperature fire environment, high tensile steel bar instead of ordinary steel bar is a very effective solution. High-strength reinforcing bars have received widespread attention for their high tensile strength, small deformation, and corrosion resistance. Therefore, research on the mechanical properties of bars above 600 MPa used in concrete structures, especially in special environments such as high temperature and corrosion, can help promote the widespread use of high strength bars in practical projects.

## **Tasks and results**

#### *1. Material test results and loading programme*

The tensile test of the rebar material was carried out using a 600KN microcomputer screendisplay hydraulic universal machine (WEW-600D) as shown in Fig. 1(a). It was found that the fracture of the rebar was more zigzag at room temperature, while the fracture after high temperature was smaller and the crack was straight, as shown in Fig. 1(b). The stress-strain curves of the HRB600 and HRB400 reinforcement materials of the beams are shown in Fig. 2.







**Fig. 2.** Stress-strain curve of steel reinforcement.

RC beam was placed on two rigid supports and line displacement transducers (LVDTs: L1~L5) were arranged on the upper surface of the beam above the supports to measure the vertical displacement at the supports, and in addition, three line displacement transducers were arranged in the midspan and loading point of the RC beam to measure the vertical displacement in the pure bending section. The schematic diagram of the four-point bending test loading of the RC beam is shown in Fig. 3.



Fig. 3. Loading device and testing instruments (unit: mm).

#### *2. Experimental results and analysis of beams*

Fig. 4 shows the load-displacement curves of the numerically simulated beams after high temperature compared with the measured results. It can be seen from the figure that the numerically simulated ultimate loads are very close to the test values with a maximum error of no more than 10%. With the increase of exposure temperature, the shear capacity of the beams without web reinforcement decreases rapidly, while the shear performance of the beams with web reinforcement is better, especially the residual capacity of the beams with high-strength stirrups can still reach more than 80% of the room temperature. The test shows that the high strength stirrups have a significant improvement on the shear performance of the beams, especially after experiencing high temperatures, the shear performance of the high strength stirrups is more

superior.



Fig.4 Comparison of simulation and experiment.

#### *3. Simple calculation of shear capacity*

The simplified shear calculation model derived was used to compare with the collected literature data, including a total of 140 beams at room temperature and 48 beams after high temperature. Data parameters:1≤a/d≤5,200 mm≤d≤700 mm, 0.14%≤ρl≤4.53%, 300 MPa≤fy≤820 MPa, 14.94 MPa≤fc≤119.04 MPa, and 20℃≤T≤800℃. The comparison between the experimental values (Vexp) and the values calculated by the computational model (Vcal) for the beams after different temperatures is given in Fig. 5. From the figure, it can be seen that the calculated results are in good agreement with the experimental values and are concentrated within the 20% error range. The mean value of Vcal /Vexp for the beams at room temperature is 1.11 with a standard deviation of 0.23 and a coefficient of variation of 0.21.The mean value of the beams after high temperature is 0.96 with a standard deviation of 0.09 and a coefficient of variation of 0.10.The experimental results show that the computational model can be adapted to the prediction of shear capacity of reinforced concrete beams under different temperature conditions.



Fig. 5 Comparison of calculated and experimental values.

### **Achievement**

The content reported here will be collated into a scientific article with an in-depth discussion and summary.

# *Appendix*

*The following photos show what the internship looked like at that time.*





During this first week of my internship in Japan, I conducted extensive research to better understand the various techniques used as well as the different reactions involved.

On Tuesday, January 23<sup>rd</sup> and Wednesday, January 24<sup>th</sup>, I had two phone interviews with Dr. Emilie Pouget to ask her questions about the subject and obtain very useful documentation for the future.

As I familiarized myself in the laboratory, I continued bibliographic research on the subject to begin the literature review section of my internship report.

Additionally, I conducted experiments with silica samples from Bordeaux, preparing them for TEM analysis.



During a zoom meeting with Dr. Emilie Pouget, I discussed the obtained images, which included nanohelices functionalized by APTES on the left and nanohelices made by Mr. Naoya Ryu on the right:



With the assistance of Mayuka Sakai, a Japanese student, I learned about the operation of a hydrophobic column and filtration equipment:



Every two weeks, I met with Dr. Emilie Pouget, Dr. Reiko Oda, and Prof Makoto Takafuji to discuss results and plan future experiments. After this first meeting, we have decided the different powder which I will work on supercritical carbon dioxide and the main objective that I must reach.

With the selected material, the objective was to find favorable conditions for subsequent tests with the nanohelices. There is different condition that I can varied for example, the temperature, the pressure, the time, the ration between polymer particle et nanosphere of silica.

Because it will take a lot of time to try all these conditions, we decided to fix these following parameters:

- Time: 2h
- Temperature: 80°C
- Pressure: 20,7MPa

We decided to work on this condition because the latest results from a study subject **[1]** of M. Takafuji, N. Hano and H. Ihara were similar to mine and were very conclusive.

**[1]** Takafuji, M., Hano, N., Yamamoto, H., Ryu, N., Horikawa, M., Nagaoka, S., & Ihara, H. (2017). One-pot green process for surface layering with nanodiamonds on polymer microspheres. *The Journal Of Supercritical Fluids*, *127*, 217-222. https://doi.org/10.1016/j.supflu.2017.02.025

Additionally, initial calculations of the ratio between powders have been made for the first synthesis, to be ready for the following week.

During the first weeks of my internship, I also completed e-learning courses on Research Ethics. Below are the two certificates of my courses.



The Japanese school system is different from the French system. The scholar year of Japanese

student finished at the end of march, so they must make some report of their year of research. In France is different, the scholar year finished in June. So, during the visit I was able to attend the dissertation of three master's student from our laboratory. One of the presentations was in English and focused on the surface modification of silica spheres to be able to bind polyurethane macromolecules. This made it possible to increase the mechanical characteristics of the polyurethane.



With the guidance of Yudai Oishi, I conducted the 1<sup>st</sup> test experiment with scCO2. We ran into some difficulties, such as the first reactor we used was leaking (the large reactor). So, we decided to switch to the smaller reactor with a Teflon seal.



I analyzed FE-SEM images derived from our first sc-CO2 experiment. This analysis was crucial in guiding our future research directions and determining the efficacy of our current approach. These are the different picture that I get from the first coating:



Additionally, I dedicated time to exploring various methods for removing aggregates. Through experimentation with water and ethanol, followed by analysis of FE-SEM images, we aimed to optimize our approach. Furthermore, we considered alternative synthesis conditions to enhance our overall methodology.



After this experimentation, I prepared FE-SEM samples and with the help of Daiki Aome, and I get this differents images of my powders:



On the left and the right, we can see polymer sphere graphed by nanosphere of silica after washing. These results are good for the future of my experiment.

After to be able to conducte manipulation with scCO2 alone, Yudai Oishi makes sure that I've got all the information and details of the experiment. This experience was important for building my confidence and independence in handling such procedures.



I also got the chance to see the presentation of the student from the bachelor's degree of their first year in laboratory. It was interesting to see all the different subject led in our laboratory.



On the left, we can see students from our laboratory, and on the right, we have Eda Nur Donmezier (left) from Turkey, who is studying in Kumamoto as an exchange student for six months, Moustapha Mohamed Mahamoud (right), a PhD student from Djibouti.

During the last week in Japan, our plans included cleaning the nanohelices samples from Bordeaux and utilizing the freeze-drying system with assistance from Kotomi Higuchi. Additionally, I attempted to graph this nanohelices on polymer particle and twisted ribbons. The following pictures show the setup required for using the freeze-drying system.



During the last weeks, we welcomed new students to our lab, offering a brief presentation about my home country, France, my academic journey, and student life in Bordeaux. We capped off the event with a barbecue, fostering collective discussions.

In my final experiments, I tried to graph nanohelices on polymer, our primary objective, and achieved very promising results:



The laboratory team organized a meeting for my departure. They gave me a frame with pictures of our lab team, which a was very kind gesture. I took a moment to express my gratitude to Takafuji sensei and the students for their assistance throughout these two months.



During my last day I went to Sendai for a business trip to meet Oda sensei, but also to discuss about the future research plans. I also met Oda sensei's team at Tohoku University.



I am grateful to Takafuji sensei for the opportunity to work with his team during this two month. It was the most beautiful experience that I have lived in my life. But I wanted to also thanks IROAST for this scholarship.

Thank you for the invaluable cultural, lifestyle and language lessons. I hope to return to Kumamoto and contribute further to our research endeavors.

Mina Ruff



## **Details of activities**

## **1. Tasks you have engaged in and what you have learnt**

Geopolymer materials have been recently used for barrier in waste containment instead of cement because of low cost and  $CO<sub>2</sub>$  emission reduction. To evaluate the barrier performance of geopolymer, some hydraulic barrier parameters including hydraulic conductivity and adsorption coefficient should be carried out. During the IROAST internship in Prof. Mukunoki 's laboratory, I performed hydraulic conductivity and adsorption tests using geopolymer materials.

The hydraulic conductivity of geopolymer specimens were determined by constant rigid flow rate hydraulic conductivity test (Figure 1). The samples are geopolymer synthesized from coal fly ash, local soils mixed with different ratio of coal fly ash based geopolymer. The solution for this test are deionized water and ammonium solution with different concentration.



Figure 1. Hydraulic conductivity test and geopolymer specimens

In addition, batch adsorption test of geopolymer, mixures of geopolymer and local soils were also conducted in this internship in Kumamoto University (Figure 2). The solution was used for this test are ammonium solution with different concentration (20, 50, 70, 100, 150, 200, 250 mg/L NH<sub>4</sub><sup>+</sup>-N). The ammonium concentration was analysed using UV-Vis spectrophotometer (Shimadzu, UV mini-1240, Japan) from Prof. Kawagoshi's Laboratory. Also, porous geopolymer from coal fly ash was synthesized using solution  $H_2O_2$ . These materials were tesed ammonium adsorption capacity in the form of granules.



Figure 2. Adsorption test and ammonium analysis

Some characterizations of these geopolymer materials including XRD, XRF, pore structures of porous geopolymer via nano X-ray computed tomographt (CT) scanner (Figure 3) were examined to evaluate the relation with hydraulic conductivity and adsorption coefficients results.



Figure 3. Nano CT scanning for geopolymer specimen

## **2. Future research plans**

After going back my home country after this intership, I will analyse the results from experiments and numerical works (X-ray CT images) in Kumamoto University. Also, I will continue to discuss with Prof. Mukunoki about the results and write a publication together. The research field of Prof. Mukunoki and his laboratory are extremely close to my further research plan. I believe we could develop some research collaboration in the near future; especially the research proposal relates to utilization of nano and micro CT scanner apparatus in X-earth Center of Kumamoto University.



For sustainable energy development, it is of importance to develop and utilize deep underground. However, it is well known that the development and utilization of deep underground cause induced seismicity, which could raise public concern. In the past, generally the occurrence of induced seismicity was investigated predominantly based on the macroscopic stress state of rock discontinuities, such as joints and faults. However, in most of cases, the occurrence of seismic events cannot be explained from the macroscopic stress because seismic events frequently take place in geological structures where anthropogenic activity-induced stress change is infinitesimal. One of the cause of such seismic activities is assumed to be attributed to the stress concentration of asperities on the surface of rock discontinuities, which leads to time-dependent failure of the asperities and thereby causes a seismic event. This study aims at investigating strain localization within asperities on a rock discontinuity.

Figure 1 shows the sandstone specimen used for this study. The sandstone is Kimachi sandstone obtained from a quarry located in Shimane prefecture. From a rock block, the specimen with a diameter of 10 mm was cored, from which the specimen was made. As can be seen from the figure, around the center of the specimen, there is an undulating discontinuity. Then, uniaxial compressive test was performed with a small-scale uniaxial compressive test machine in the micro-focused Xray CT, as shown in Figure 2. The UCS test was performed whilst increasing the load in a stepwise manner. More specifically, the load was increased by 314 N at each stage.

Figure 3 shows an example of CT images taken during the UCS test. As can be seen, the discontinuity is clearly displayed. Note that the CT images were taken at the final stage of the UCS test, where the specimen underwent failure. Hence, as can be seen from the figure, fractures are visible near the pre-existing discontinuity.

Figure 4 shows a displacement field derived from image analysis with the digital image correlation method. In order to obtain a reliable result from the DIC analysis, the DIC parameters were calibrated using a specimen without a discontinuity, which was loaded with the UCS device in the X-ray CT. From the result, Young's modulus of the specimen was evaluated with DIC whilst varying the parameters. As the Young's modulus of the sandstone was already evaluated from ordinary UCS test, the DIC parameters were optimized so that a comparable Young's modulus can be obtained.

Now that the displacement field was obtained, I'm going to compute a strain field in the specimen and focus on micro-scale strain distribution in the asperities located on the discontinuity. The result will help us estimate stress concentration taking place in the asperity when compared to numerical simulation result. To achieve this, the discontinuity surface was measured with a laser scanner owned by Prof. Sainoki. Now, based on the result of the surface scanning, a 3D numerical model is being constructed, whereby the experiment can be reproduced. Then, the strain field obtained from the DIC analysis can be compared to that obtained from the numerical simulation. The duration of this internship is a little bit short to conduct all the experiment and numerical simulation described above. After going back to China, I will continue the study.



Figure 1: Sandstone specimens with a discontinuity



Figure 2: UCS test in a micro-focused X-ray CT



Figure 3: Example of CT images during the UCS test



Figure 4: Displacement fields derived from DIC analysis



3D printing technology with sand powders is a novel and promising method in the field of rock mechanics because of its consistent mechanical properties, not affected inherent heterogeneity and anisotropic characteristics that natural rocks have. However, no fundamental studies have been conducted on the 3D printed specimen, although its mechanical properties are affected by the size distribution of the sand particle. In order to gain a deep understanding on the effect, we performed a series of uniaxial compressive tests in an X-ray CT scanner at Kumamoto University, followed by image analysis to examine strain distribution during the loading stage and its implication for the final rupture.

The first couple of weeks have been spent on developing a method to grind the edges of the 3D printed specimen. During the previous internship in 2022, it was pointed that the accuracy of the edge is not high enough due to the problem of the 3D printing machine. Although the problem was solved in China, it was found that the accuracy is still low. After a number of discussions with a technician at Kumamoto University (Toru Yoshinaga), we decided to use a lathe to grind the edge. Then, all the specimens were grinded, which took a couple of weeks and caused a delay for this project.

Then, we started to conduct uniaxial tests in the X-ray CT. However, the CT is allowed to used only when Prof. Sainoki is here, so that we only conducted 9 experiments in total for specimens made of sand particles with different distributions of particle size. Figure 1 shows the UCS test in the CT.

After each experiment, image analysis based on digital image correlation method was performed in order to obtain strain distribution in the specimen before failure. The UCS test was performed in a stepwise manner while increasing the uniaxial load by 314 N at each stage, so that strain increment can be captured. Figure 2 shows an example of the image analysis result showing strain localization with the increase in the loading. However, the strain increment from 0 to 1884 N still contains errors, so that the strain concentration is not reasonable. Further optimization of parameters used for the image analysis is needed. The strain increment from 0 to 2198 shows a reasonable strain localization along a potential shear failure plane. Figure 3 shows strains less than 0.01. Although it is not so clear, it seems that strain localization is taking place prior to the occurrence of final rupture.

After going back to China, further study will be conducted in order to characterize shear strain

localization for specimens made of different sizes of sand particles. Based on the result, it is aimed to optimize physical properties of sand particles used for 3D rock-like specimen for future experiments.



Figure 1: Uniaxial compressive test in the micro-focused X-ray CT.



0-1570  $0 - 1884$  $0 - 2198$ 0-final Figure 2: Example of image analysis result showing strains above 0.015 and below 0.024.



Figure 3: Example of strain distribution showing only strains less than 0.01.



The study conducted during this internship pertains to the microscale mechanical characteristic of 3D printed rock-like specimen made of silica sand powders. The factor that contributes to the mechanical and physical properties of the 3D printed specimen is binder and sand particle. This study particularly focuses on the effect of silica sand particle size on the mechanical and physical properties of the 3D printed specimen. Nine types of 3D printed rock-like specimens were generated with a 3D printer in China, using different sizes of silica sand particles, which were then shipped to Japan to conduct uniaxial compressive tests in a micro-focused X-ray CT.

During the period from August  $1<sup>st</sup>$  to Sept.  $30<sup>th</sup>$ , nine groups of specimens were tested in the microfocused X-ray CT with the device shown in Figure 1, which is the specially designed small-scale UCS test machine for the micro-focused X-ray CT. In the device, it is possible to install a specimen with a diameter of up to 10 mm and height of approximately 20 mm. It is very difficult to place the specimen at the center of the device. The UCS test was performed whilst increasing the uniaxial load. At each stage, CT scanning is performed. Generally, the increment of load is 314 N, and the specimen will fail when the load is increased to 2000 N approximately, which corresponding to the uniaxial compressive strength of 20 MPa.

Then, digital image correlation method (DIC) was applied to CT images at each loading stage to calculate strain distribution inside the specimen. Figure 2 shows an image obtained by subtracting images at different stages. Figure 3 shows strain distribution obtained from the DIC analysis. The DIC analysis requires optimization of parameters. The DIC parameters affect the result significantly, which necessitates the optimization. In order to optimize the parameters, Young's modulus was calculated based on the stress and strain relationship obtained from the test. The strain field can be obtained from displacements derived from the DIC, while the stress can be calculated from the load measured during the UCS test. The Young's modulus derived from the DIC analysis was then compared to that derived from a UCS test using an ordinal specimen with a diameter of 50 mm and height of 100 mm. Figure 4 shows Young's moduli estimated from the DIC for the purpose of optimization. The most important parameter is *hws* in the figure, which needs to be optimized based on the particle size whilst considering computational cost. I performed a number of analysis whilst varying the parameters to obtain a reliable result from the DIC.

Eventually the parameters were optimized, whereby strain distribution was calculated to examine strain localization in the specimen before the occurrence of final rupture and investigate the influence of particle size.



Figure 1: Small-scale UCS test machine for a micro-focused X-ray CT



Figure 2: Showing the first step to conduct DIC Figure 3: Calculation of strain distribution with DIC

$Wdt = 210$ <b>Stage 942N-1256N</b>			Number	-ns		-hws	-glt
			1			10	15000
				◆ The returnStatus appear 1 and 2 when I use the parameter, this situation have not appeared when -ns 20 -			
hws 20.			$\blacklozenge$ The calculation is not including the data that return Status is 1		210-220	3.96	
						220-230	2.78
	elastic position module/G			$20 - 30$	5.57	230-240	1.67
		110-310	6.82	$30 - 40$	6.41	240-250	6.07
		120-300	6.77	$40 - 50$	5.72	250-260	9.99
	pa	130-290	6.86	$50 - 60$	5.59	260-270	6.76
$20 - 400$	4.71			$60 - 70$	5.81	270-280	5.99
$30 - 390$	6.61	140-280	6.97	$70 - 80$ $80 - 90$	7.10 8.87	280-290	6.25
40-380	6.60	150-270	6.94	$90 - 100$	5.67	290-300	5.96
50-370	6.59	160-260	6.98	$100 - 110$	4.98	300-310	6.19
60-360	6.69			$110 - 120$	8.91	310-320	7.55
		170-250	6.83	120-130	6.23	320-330	6.03
70-350	6.78	180-240	6.69	130-140	6.15	330-340	7.99
80-340	6.76	190-230	24.35	140-150	8.76	340-350	7.02
$90 - 330$	6.65			150-160	6.75	350-360	5.61
100-320	6.73	200-220	6.81	160-170	6.21	360-370	5.05
				170-180	9.12	370-380	8.51
				180-190 190-200	7.39 4.35	380-390	6.98
				200-210	24.48	390-400	0.41

Figure 4: Optimization for DIC parameters



3D printing technology with sand powders is a novel and promising method in the field of rock mechanics because of its consistent mechanical properties, not affected inherent heterogeneity and anisotropic characteristics that natural rocks have. However, no fundamental studies have been conducted on the 3D printed specimen, although its mechanical properties are affected by the size distribution of the sand particle. In order to gain a deep understanding on the effect, we are planning to perform a uniaxial compressive test in an X-ray CT scanner at Kumamoto University. As a preliminary analysis, during this internship, we sent some specimens to Kumamoto University and Prof. Sainoki conducted a UCS test without scanning in the CT.

Figure 1 shows the specimen, which is 10 mm in diameter and 20 mm in height printed with silica sand particles sent to Prof. Sainoki at Kumamoto University. The specimen was set in the smallscale uniaxial compression test machine shown in Figure 2. Subsequently, the loading was applied to the specimen whilst controlling the displacement at a rate of 1 μm/s.

Figure 3 shows the result, which was discussed with Prof. Sainoki during an online meeting. As can be seen, the uniaxial strength differs between the specimen, although they were made from the same sand particles. Prof. Sainoki pointed out that the degree of parallelism is not high for the specimen, which is attributed to the generation process with the 3D printing machine. Prof. Sainoki asked us to increase the edge accuracy by the time we conduct UCT tests in the X-ray CT next year (2023).



Figure 1: 3D printed specimen with a diameter of 10 mm and height of 20 mm



Figure 2: Small-scale uniaxial compressive test



Figure 3: Discussion on the result of the uniaxial compressive test



During this internship, preliminary experiments were conducted for 3D printed rock-like specimen made of silica sand particles. Figure 1 shows the specimen with a diameter of 10 mm and height of 20 mm. The specimen is on a small-scale in order to conduct a uniaxial compressive test in a micro-focused X-ray CT in the future. As a preliminary analysis, the accuracy of the specimen was checked, and it was found that the accuracy is not high, requiring further grinding for the edge so that both the edges become parallel with each other. Figure 2 shows a small-scale compressive machine, in which the specimen was placed and underwent uniaxial loading. Figure 3 shows an example of the uniaxial compressive tests. The result shows a discrepancy in uniaxial compressive strength among the specimens, indicating the effect of the edge. Further work is needed to obtain a reliable result, so that reliable UCS tests can be performed in the X-ray CT.



Figure 1: 3D printed specimen with a diameter of 10 mm and height of 20 mm



Figure 2: Small-scale uniaxial compressive test



Figure 3: Result of the uniaxial compressive test