Fundamental Study on Fretting Corrosion in Solid Breeder Blanket*)

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Lithium titanate (Li_2TiO_3) is candidate tritium breeder of the fusion blanket systems. The small pebbles of Li_2TiO_3 are packed in the blanket box. The coolant tubes made of RAFM steel F82H (Fe-8Cr-2W-0.1C) are installed in the blanket box and their oscillation is possibly induced by the coolant flow. The fretting corrosion is then caused by the reciprocating slip of the pebbles on the tube wall. The purpose of the present study is to investigate the fundamental behaviors of fretting corrosion between Li_2TiO_3 pebble and the F82H plate. The small pebble of Li_2TiO_3 or Al_2O_3 was placed on the plate specimen of F82H in point contact, and the plate specimen was oscillated horizontally and lineally at the frequency of 50 Hz and the amplitude of 120 μm. The constant load of 4.9 N was applied between the small pebble and the plate specimen. The fretting tests were conducted for 10 and 300 minutes in an air atmosphere at a room temperature. The Li_2TiO_3 pebble was significantly abraded in the tests. The surface of the F82H specimen was damaged due to the scratch with the broken particles of the pebble. The fretting corrosion of F82H was promoted in the test with Al_2O_3 pebble, since the formation and destruction of the oxide layer were repeated on the steel surface in the fretting cycle.

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1. Introduction

Lithium titanate (Li_2TiO_3) is candidate tritium breeder in the solid breeder blanket of fusion reactors [1, 2]. The small pebbles of Li_2TiO_3 are packed in the blanket box. The coolant tubes are installed in the blanket box [2] to remove heat generated in the pebbles under neutron irradiation. The blanket coolant is pressurized water and the coolant tubes are made of reduced activation ferritic martensitic steel F82H [3, 4].

The oscillation of coolant tubes induced by the flowing coolant has been recognized in nuclear power plants [5–7]. The coolant tube then slips against the peripheral structures such as anti-violation bars [8]. The occurrence of fretting corrosion and tube failures has been recognized in the steam generators of pressurized water reactors (PWRs) [9]. The fretting corrosion has been identified as a serious problem for the damage of steam generator tubes of nuclear reactors [10]. The fretting corrosion is also leading cause of fuel rod failures of PWRs [11].

Small pebbles of ceramic breeders and neutron multiplier are randomly filled in the blanket box of the Japanese fusion DEMO reactor [4]. The filling factor is expected to be 64%. The small pebbles are stuck and do not move freely even when the coolant tubes oscillate by the coolant flow. The fretting corrosion may occur between the Li_2TiO_3 pebbles and the tube wall in the blanket. The occurrence of the fretting corrosion is serious concern, since it can influence on the operating life of the blanket. However, the information of fretting corrosion between the Li_2TiO_3 pebble and F82H was limited so far.

The purposes of the present study are to develop the test apparatus and to investigate fundamental fretting behaviors of the Li_2TiO_3 pebble and the F82H plate. The preliminary fretting corrosion tests were performed in an air atmosphere at room temperature.

2. Experimental Conditions

2.1 Test materials

Fretting tests were performed with the pebble specimen of Li_2TiO_3 and the plate specimen of F82 H. Table 1 presents the chemical compositions of F82 H. The surface of the F82H specimen was mirror-polished. Table 2 presents major features of small pebble specimens. The Li_2TiO_3 pebble has a porous structure, in which the release of tritium from the pebble is promoted [12]. The fretting behaviors between the pebble and the plate were

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Table 1 Chemical compositions of reduced activation ferritic martensitic steel F82H [wt%].

	Cr	W	Si	Others	Fe
F82H	7.7	1.94	0.1	0.01Ti-0.01Cu	Balance

Table 2 Major specifications of Li_2TiO_3 and Al_2O_3 pebbles.

	Supplier	Diameter [mm]	Density [g/cm^3]	Theoretical density [%]	Hardness [HV_{10}]	Purity
Li_2TiO_3	QST	1.2	2.74-2.92 [13]	80-85 [13]	363 [13]	-
Al_2O_3 (SSA-999W)	Nikkato corp.	1	3.9	98	1800	99.9

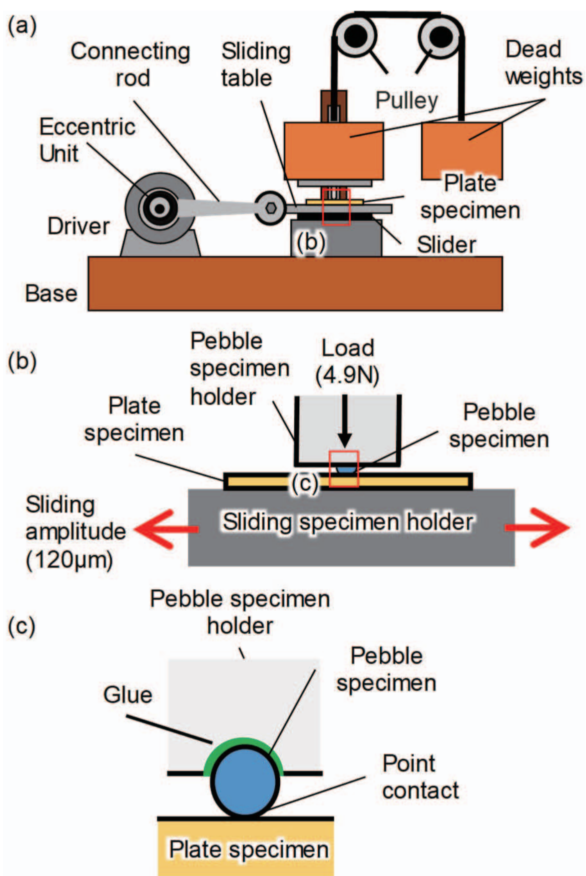

Fig. 1 Fretting test apparatus, (a) Schematic diagram of fretting test apparatus, (b) pebble specimen holder and sliding specimen holder, and (c) Specimen arrangement.

assumed to be influenced by the porosity and the hardness of the pebble. Therefore, the fretting tests were also performed using the Al_2O_3 pebble which had lower porosity and higher hardness than Li_2TiO_3, though the Al_2O_3 pebble is not used in the solid breeder blanket of fusion reactors. The fretting behaviors of the Li_2TiO_3 pebble and the F82H plate were then featured by comparison with the results of the fretting tests with Al_2O_3 pebble.

Table 3 Experimental conditions of fretting tests.

Temperature	Room temperature
Time [min]	10 and 300
Load [N]	4.9
Sliding Frequency [Hz]	50
Sliding amplitude [μm]	120
Sliding cycles	30,000 and 900,000

2.2 Experimental apparatus

Figure 1 (a) shows the experimental apparatus newly constructed for the fretting tests in the present study. The eccentric unit connected to the motor driver converted the rotary motion into a linear reciprocating motion of the sliding specimen holder. The sliding specimen holder then oscillated horizontally and linearly. The pebble specimen of Li_2TiO_3 or Al_2O_3 was fixed on the pebble holder by glue as shown in Figs. 1 (b) and (c). The pebble specimen was then pushed onto the plate specimen in point contact. Fretting corrosion tests were performed by means of the slip oscillation of the plate specimen against the pebble specimen. The load applied by the pebble onto the plate was adjusted with two weights, and measured using a weighing scale before the fretting tests.

2.3 Experimental conditions

Table 3 presents the experimental conditions of fretting tests. The fretting tests were performed in air atmosphere at room temperature. The load applied between the pebble specimen and the plate specimen was 4.9 N. The sliding frequency was 50 Hz, and the sliding amplitude was 120 μm. The test durations were 10 minutes and 300 minutes. The vibration conditions of the ceramic breeders and the coolant tubes in the solid breeder blanket were not made clear so far. Therefore, the experimental conditions in the present study were based on those in the previous studies [10].

After the tests, the pebble and plate specimens were taken out from the specimen holders. The fretting debris produced in the tests was collected and analyzed by scanning electron microscope/energy dispersive X-ray spec-

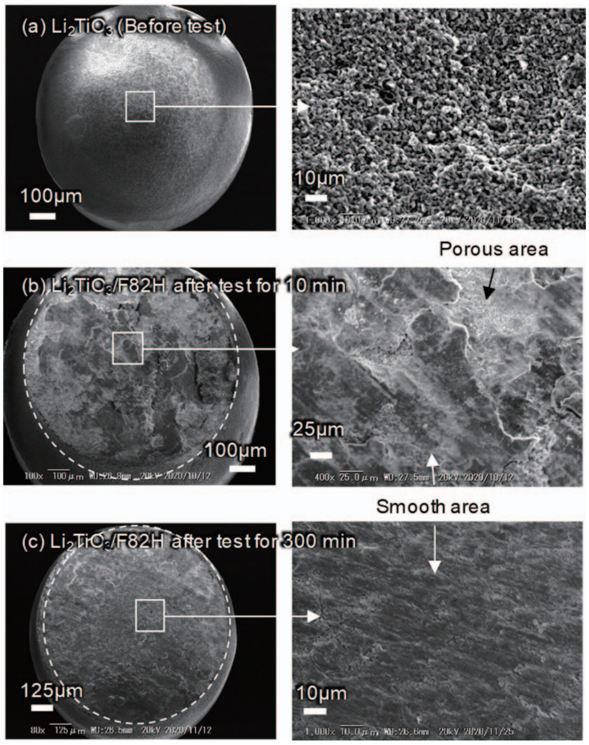

Fig. 2 SEM images of Li_2TiO_3 pebble (a) before test, (b) after test with F82H plate for 10 minutes, (c) after test with F82H plate for 300 minutes.

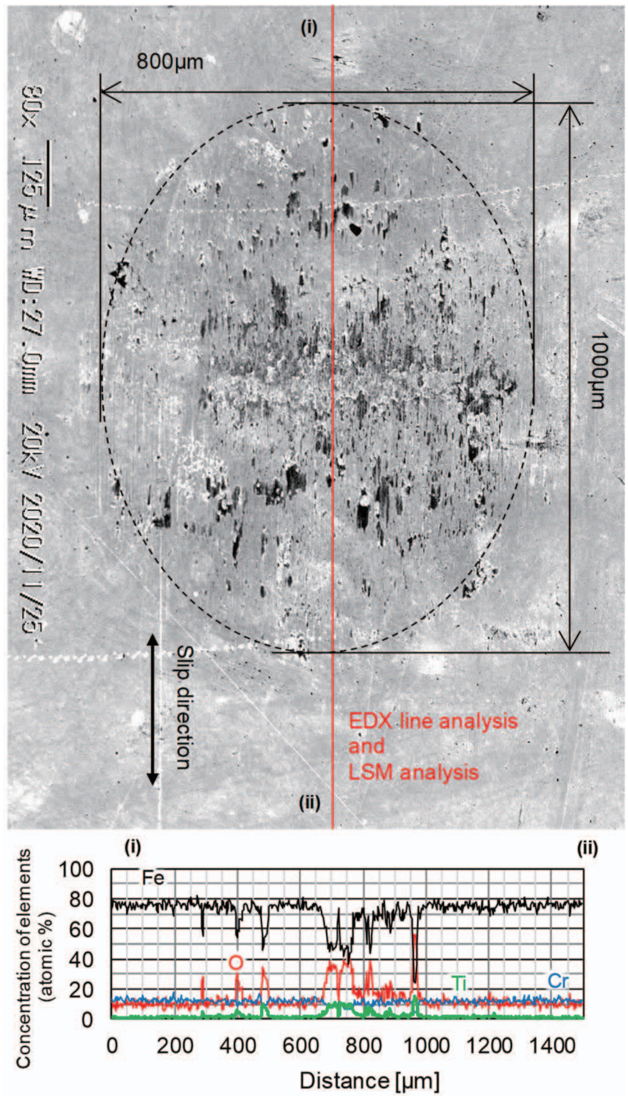

Fig. 3 SEM/EDX analysis of fretting scar of F82H steel after test with Li_2TiO_3 for 10 minutes.

troscopy (SEM/EDX). The ultrasonic cleaning of the pebble and plate specimens were performed with acetone. The surface of the specimens was analyzed by 3D laser scanning microscope (LSM, Nano search microscope of OLYMPUS corp.) and SEM/EDX.

3. Experimental Results

3.1 Li_2TiO_3 pebble vs. F82H plate

Figure 2 shows the SEM images of Li_2TiO_3 pebble before and after the fretting tests. The Li_2TiO_3 pebble initially had the porous structure as shown in Fig. 2 (a). Figure 2 (b) shows the surface morphology of the pebble after the test for 10 minutes. The flat area was indicated by a dotted circle, and its diameter was approximately 920 μm. The flat area could be classified into porous and smooth regions. Figure 2 (c) shows the surface morphology of the pebble tested for 300 minutes. The diameter of the flat area was approximately 1080 μm, and was just slightly larger than that tested for 10 minutes.

Figure 3 shows the results of SEM/EDX analysis on the specimen surface after the test for 10 minutes. The fretting scar was indicated by a dotted circle. The length of the fretting scar was approximately 1000 μm. This length corresponds to the sum of the sliding amplitude (120 μm) and the diameter of the flat area (920 μm) of the pebble. The element concentrations in the results of EDX analysis were expressed as their total of the Fe, Cr, O, and Ti concentrations was equal to 100%. The results of EDX analysis on the mirror finished specimen before the test indicated that the concentrations of Fe, Cr, O were approximately 78 (85 wt%), 12 (12 wt%), and 10 at% (3 wt%), respectively. The oxygen concentration of the initial specimen surface was high possibly because of the surface oxidation after the mechanical polishing procedures. The oxidation was detected around the center region of the fretting scar. Ti was detected in the oxidized area. These results indicated that the formation of Fe-Ti-O or the adhesion of Li_2TiO_3 on the surface.

Figure 4 shows the LSM fretting scar profile along slip direction as indicated by solid line in Fig. 3. Some small cavities were detected around the center region of the fretting scar. The depth of the cavity was less than 30 μm. Some protrusions were also detected. They were due to the adhesion of the fretting debris on the surface as explained in the next chapter.

Figure 5 shows the SEM image of the surface of F82H specimen after the test for 300 minutes. The length and

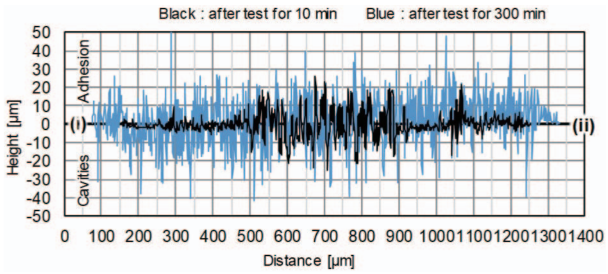

Fig. 4 Fretting scar profile of F82H surface by 3D laser scanning microscope after test with Li_2TiO_3 for 10 minutes and 300 minutes.

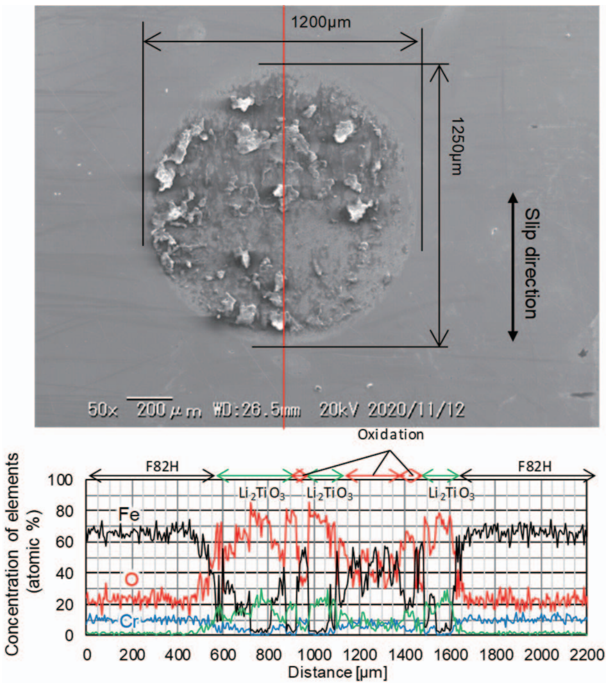

Fig. 5 SEM/EDX analysis of fretting scar of F82H steel after test with Li_2TiO_3 for 300 minutes.

the width of the fretting scar were approximately 1250 μm and 1200 μm, respectively. The results of EDX analysis indicated that the fretting scar was partially covered by Li_2TiO_3. The maximum depth of the fretting wear was 42 μm as shown in Fig. 4. A lot of protrusions on the specimen surface was due to the adhesion of the Li_2TiO_3.

Figure 6 shows the fretting debris ejected and accumulated uniformly around the fretting scar in the test for 300 minutes. The color of the debris was white. The size of the debris particles was approximately 5 μm, and was almost the same with the grains recognized in the microstructure of the Li_2TiO_3 pebble as shown in Fig. 2 (a). Table 4 presents the results of EDX analysis on the debris produced in the fretting tests for 300 minutes. The debris mainly consists of Ti and O, and does not contain Fe and Cr. The chemical compositions were almost the same with that on the flat area of the pebble. These data indicated that the debris was mainly the broken particles of the Li_2TiO_3

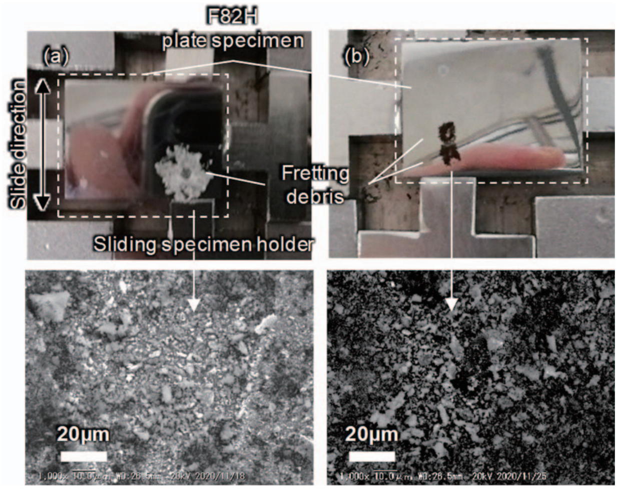

Fig. 6 Fretting debris produced in the tests (a) with Li_2TiO_3 pebble and (b) with Al_2O_3 pebble.

Table 4 EDX analysis of fretting debris produced in tests for 300 minutes (unit: atomic %).

Test	Fe	Cr	Ti	Al	O	Total
Li_2TiO_3	0.99	0.13	13.87	-	85	100
Al_2O_3	34.27	3.98	-	1.62	60.13	100

pebble, which were produced in the abrasion procedure of the pebble in the fretting cycles.

3.2 Al_2O_3 pebbles vs. F82H plate specimens

The number and size of pores in the Al_2O_3 pebble are much less than those in the Li_2TiO_3 pebble as shown in Fig. 7 (a). Figure 7 (b) shows the Al_2O_3 pebble after the test for 10 minutes. The abrasion of the pebble was not caused unlike the result of the test with the Li_2TiO_3 pebble. The abrasion was not observed even in the test for 300 minutes as shown in Fig. 7 (c). This feature was much different from that of the Li_2TiO_3 pebble. The Al_2O_3 pebble had larger density and hardness than the Li_2TiO_3 pebble as presented in Table 3. Therefore, the Al_2O_3 pebble revealed abrasion tolerance more than the Li_2TiO_3 pebble.

Figure 8 shows the SEM image of the F82H plate specimen after the fretting tests. The fretting scar was clearly observed after the tests. The length and the width of the fretting scar formed in the test for 10 minutes was smaller than that with Li_2TiO_3 pebble. The results of EDX analysis indicated the fretting scar was locally oxidized. The oxide layer formed on the fretting scar might be Fe oxide containing Cr. The broken particles of the Al_2O_3 pebble was rarely detected on the fretting wear. The oxidation was more obvious in the test for 300 minutes.

Figure 9 shows the LSM fretting scar profile along slip direction as indicated by solid line in Fig. 8. The profile indicated the fretting wear in the test for 10 minutes and the adhesion of the fretting debris in the test for 300 minutes. The maximum depth of the fretting wear in the test

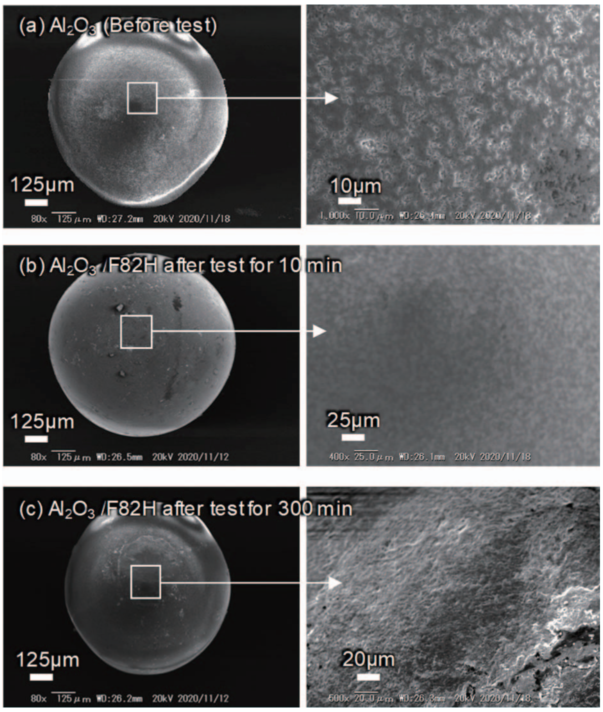

Fig. 7 SEM image of Al_2O_3 pebble (a) before test, (b) after test for 10 minutes, (c) after test for 300 minutes.

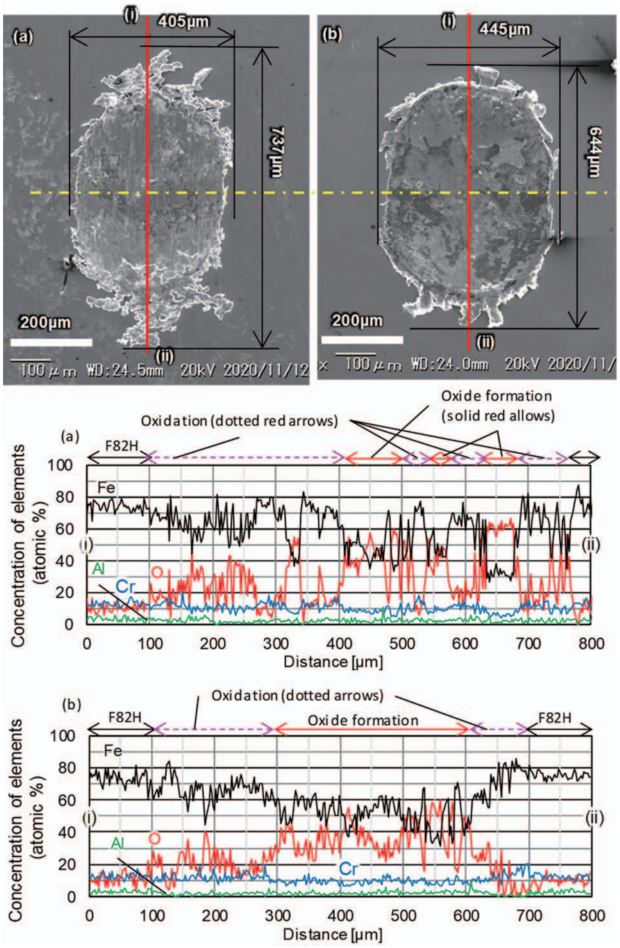

Fig. 8 SEM/EDX analysis on F82H plate specimens fretted by Al_2O_3 pebble after test (a) for 10 minutes and (b) for 300 minutes.

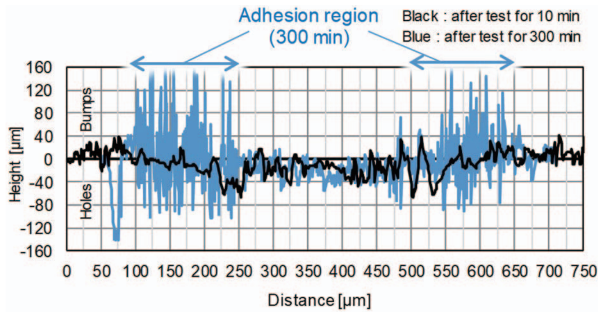

Fig. 9 Fretting scar profile on F82H surface after fretting test with Al_2O_3 pebble by 3D laser scanning microscope.

for 10 minutes was 61 μm, and that for 300 minutes was 141 μm. The damage of the plate specimen in the test with the Al_2O_3 pebble was larger than that with the Li_2TiO_3 pebble.

Figure 6 (b) shows the fretting debris accumulated at the both ends in the linear oscillation of the pebble. The color of the debris was red-brown [14]. The results of EDX analysis indicated that the debris was Fe oxide containing small content of Cr and Al as presented in Table 4.

4. Discussions

The fretting behaviors of the Li_2TiO_3 pebble and the F82H plate were summarized in Fig. 10 (a). The abrasion of the Li_2TiO_3 pebble was caused mainly in the initial stage of the fretting cycles, which corresponds to the test for 10 minutes. The porous structure of the pebble promoted the abrasion. The stress worked between the flattened pebble and the plate became smaller at the constant load after the formation of the flat face by the abrasion of the pebble. The abrasion of the pebble was then mitigated in the steady state conditions. The broken particles of the pebble were produced in the abrasion procedure. The most part of the particles was delivered into the outside of the fretting scar as shown in Fig. 6 (a). Some particles were stuck onto the plate specimen by the oscillating pebble. The particles were removed in the reciprocating slip motion, and fretting scars were then formed with small cavities. The formation of the deeper cavities in the test with longer duration was indicated in Fig. 4. The depth of the cavities can be deeper by the repetition of these procedures. The particles were trapped more easily when the depth of the cavities became deeper. Large adhesion of the Li_2TiO_3 was detected on the surface of the plate specimen in the test for 300 minutes.

Oxygen is one of the key factors which promote the fretting corrosion. The fretting corrosion of the F82H plate was promoted in air atmosphere in the current work. The fretting corrosion of F82H may be mitigated in high-purity He atmosphere, though the behavior in an inert gas atmosphere is affected by non-metal impurities such as oxygen and moisture [14]. Tritium released from the Li_2TiO_3 pebbles in the blanket operation may not affect on the fretting behavior, since hydrogen did not affect on the fretting be-

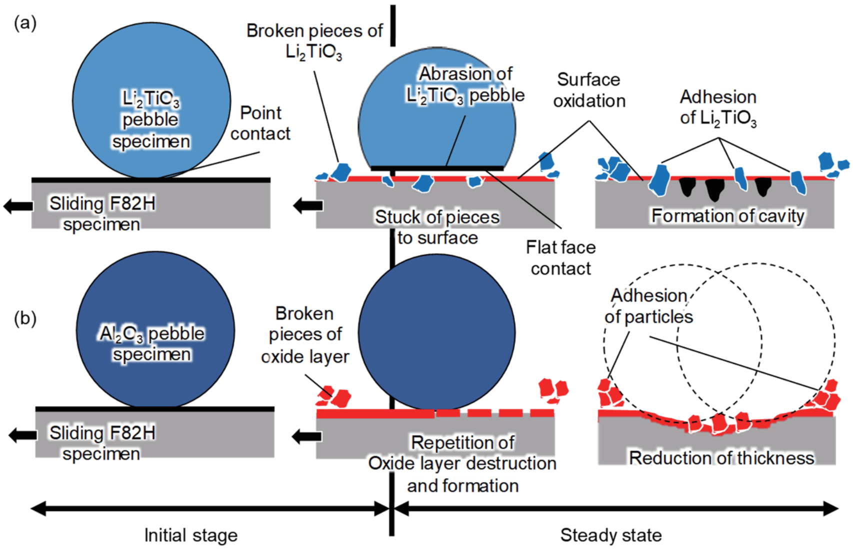

Fig. 10 Schematic diagram of fretting behaviors of pebble and plate specimens.

haviors in the previous study [14].

The temperature effect on the fretting corrosion of high strength alloy steel (Fe-3Cr-1Mo-0.4C) in air atmosphere was investigated in the temperature range between 297 K and 723 K in the previous study [15]. The coefficient of friction between the steels becomes smaller at higher temperature. The volume loss of the steels due to the fretting wear also becomes smaller at low temperature. In these conditions, a glaze layer is formed on the fretting scar due to a tribo-sintering process, and mitigates the fretting corrosion. However, the formation of the glaze layer on the F82H steel under fretting condition in He atmosphere at elevated temperature is not made clear so far. The fretting behaviors between the Li_2TiO_3 pebble and F82H at the practical blanket conditions will be investigated in our further study.

The mechanical damage of the Li_2TiO_3 pebble is caused in the fretting cycles, and this behavior may not be significantly changed in the He atmosphere at high temperature. The effect of the He atmosphere and the temperature on the fretting ablation of the pebble may be small.

The fretting corrosion between the Al_2O_3 pebble and the F82H plate was summarized in Fig. 10 (b). The abrasion of the pebble was not caused since the pebble had compact structure unlike the Li_2TiO_3 pebble. A fresh surface was then formed on the steel after the destruction of the oxidized surface by the slip motion of the pebble. The fresh surface was oxidized and destroyed repeatedly. In this procedure, the fretting debris of Fe oxide was produced.

5. Conclusions

The fretting test apparatus was newly constructed, and the fretting corrosion tests with solid breeder materials were performed. Major conclusions are as follows;

1. The fretting behaviors of the Li_2TiO_3 pebble and F82H steel plate in air atmosphere at room temperature were investigated. The Li_2TiO_3 pebble was severely abraded according to the fretting cycles for 10 minutes. The large abrasion was promoted by the brittle structure due to a lot of pores in the pebble. The broken particles of the pebble adhered on the fretting scar of F82H steel. The particles were ejected into the region outside the fretting wear. The cavities were observed in the fretting scar on the steel, which might be formed by the stuck and the removal of the hard particles of Li_2TiO_3. The steel surface was oxidized. The oxidized surface was not destroyed by the fretting cycle of the pebble. The maximum depth of the cavities was 42 μm in the test for 300 minutes.
2. The damage of the Al_2O_3 pebble was negligibly small in the fretting test with F82H steel. Its large density and high hardness of the pebble might be the reason for the damage tolerant. The oxidized surface was destroyed by the oscillatory slip motion of the pebble, and the fresh surface was oxidized again. The repetition of these procedures caused the fretting corrosion. The fretting debris was ejected into the edge of the fretting scar. Some debris were pressed by pebble in the reciprocating slip motion and accumulated on the fretting scar. The maximum depth of the fretting wear was 141 μm in the test for 300 minutes.

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