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Application of Ice Plug Technology to Nitric Acid Pipe Systems

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ABSTRACT

At the maintenance of hydraulic pipe system during continuous operation in many industrial plants, an ice plug technology for temporary partial isolation is generally applied. We have been developing ice plug technology in order to apply to nitric acid pipe systems. As chemical reagents such as nitric acid possess volume shrinkage characteristic in contrast to water, we implemented mock-up test in order to study specific freezing process of nitric acid and application ability of the ice plug technology.

Consequently, we confirmed nitric acid transferred completely to white solid phase around -60 °C to -70 °C. Furtehrmore, in mock-up test, approximate 40 °C of freezing point depression originated from freezing concentration process of nitric acid was observed. It did not affect ice plug formation, it was possible to form ice plug possessed sufficient pressure resistance performance with freezing completion time of estimated freezing process.

From the above results, we obtained the prospect that our technology is applied to nitric acid pipe systems.

KEYWORDS

Ice plug technology, Nitric acid pipe system, Volume shrinkage characteristic, Freeze concentration process, Freezing time

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1. Introduction

At the maintenance of hydraulic pipe system during continuous operation in many industrial plants, an ice plug technology for the temporary partial isolation with freezing a part of internal water is generally applied. On the other hand, many number of nitric acid pipe system with their various concentrations are installed in the irradiated nuclear fuel reprocessing plant, so that we have been developing the ice plug technology to apply to these nitric acid pipe systems.

Water possesses expanding characteristic when it is frozen, but chemical reagents such as nitric acid possess shrinkage characteristic in reverse. In addition, they show freezing point depression originated from freezing concentration process. Because of these unique characteristics, it is important to confirm specific freezing process of nitric acid solution to realize safe and reliable ice plug application to nitric acid pipe systems.

Here in, we report an ice plug application technology which we have studied based on the above backgrounds, and ice plug formation process.

2. Overview of ice plug technology

Ice plug technology, as shown in Fig. 1, is to make a blockage plug by freezing liquid in pipe systems under operation. In order to implement maintenance work of valves which are difficult to be isolated mechanically, this technology is applied to a part of pipe systems which are under continuous operation. The blockage plug is formed on upstream side from maintenance objects by forcibly cooling liquid in upstream pipe from its outer surface with refrigerant such as liquid nitrogen.

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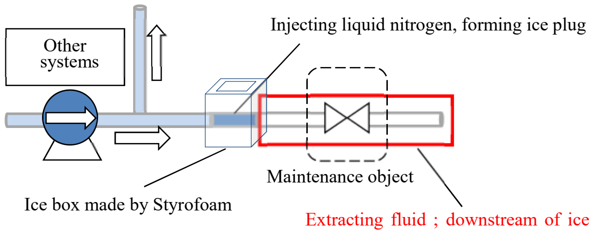


Fig. 1. Schematic description of ice plug technology

3. Nitric acid for test

Nitric acid passes through specific freezing process as compared with water. Moreover, it is known that nitric acid possesses volume shrinkage characteristic. Sections 3.1 to 3.3 describe these characteristics of nitric acid.

3.1. Freezing point

Fig. 2. shows various crystal structures of nitric acid [1]. In order to simulate actual plant situation, 3 kinds of concentrations (3.0, 6.0, and 13.6 mol/L) are selected in this paper. This concentration range covers the maximum concentration using in plant (13.6 mol/L) and the concentration (6.0 mol/L) which shows the lowest freezing point.

3.2. Freeze concentration process

Once nitric acid starts to freezing, it passes through freeze concentration process, which leads to freezing point depression and hindrance of crystal growing rate [2]. This is caused by crystallization of the water including nitric acid solution. The viscosity of remained solution increases greatly and diffusion characteristic decreases. In addition, this phenomenon is significant in higher concentration solution.

3.3. Volume change rate

Table 1 shows volume change rate of both water and nitric acid when they are frozen. We can understand that the volume shrinkage of nitric acid increases with the increase in its concentration.

4. Beaker test

As preparation of mock-up test simulated actual scale application, we implemented beaker test in order to study the effect that characteristics described in Section 3 affect ice plug feasibility.
The purpose of this test is to study 3 items according to freeze concentration process on each nitric acid concentration, that is, (1) Whether freezing point depression is or not, (2) Crystal formation process, and (3) Freezing time until ice plug forming completely.

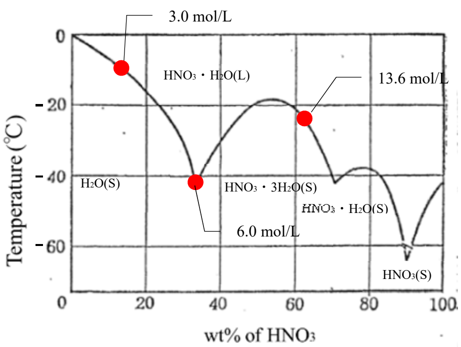


Fig. 2. Solid-liquid phase equilibrium diagram for a binary system composed of Nitric acid and Water

Table 1. Volume change rate of both water and nitric acid

	Water	Nitric acid (HNO_3)		
Concentration (mol/L)	−	3.0	6.0	13.6
Volume change rate (%)	+9.0	−1.0	−6.0	−15.0

4.1. Test method

(1) 1200 ml nitric acid is poured into 2L beaker and resistance temperature detector is putted in center of beaker. This detector is set by clamp so as its tip is at half of liquid level height.

(2) This beaker is inserted into Dewar vessel which is filled with liquid nitrogen. Moreover, this beaker is set so as liquid level height of liquid nitrogen is higher than one of nitric acid solution. Liquid nitrogen is added whenever necessary in order to keep up its liquid level height.

4.2. Results and Discussion

(1) As shown in Fig. 3, temperature reduction of each nitric acid is suppressed at freezing point described in Reference [1] (3.0 mol/L : -10°C, 6.0 mol/L : -42°C, and 13.6 mol/L : -23°C) and temperature shows a constant value, which means each nitric acid reaches solid-liquid phase equilibrium. That is to say, we could not find freezing temperature depression originated from freeze concentration process.

(2) While observing white crystal formation process of 13.6 mol/L nitric acid, as shown in Fig. 4 (a) – (d), we confirmed that ice crystal formation started from surface of beaker after 5 minutes from liquid nitrogen pouring into (Fig. 4(a)). As time passes, ice crystal grew into white crystal toward center of beaker.

Ice crystal just after formation is translucent sherbet state, but when temperature of beaker central reaches -60°C to -70°C, and after 55 minutes from cooling start when phase transition can be completed (Fig. 4 (d)), translucent crystal changed to white crystal. We confirmed white crystal possessed functionable hardness as ice plug.

(3) According to other report [2], hindrance of nucleus growing rate by freeze concentration process is significant on higher concentration solution. In this test, as nitric acid concentration was higher, it was necessary long time to reach -60°C to -70°C.

From the results of beaker test, we confirmed ice plug formation of 13.6 mol/L nitric acid could be completed by securing 55 minutes of freezing time after pouring liquid nitrogen.

5. Mock-up test

Fig. 5 shows mock-up test equipment simulated actual scale application. We implemented mock-up test by using this equipment. The purpose of this test is to confirm: (1) freezing process of 13.6 mol/L nitric acid in plant process pipe systems and (2) pressure resistance performance of formed ice plug.

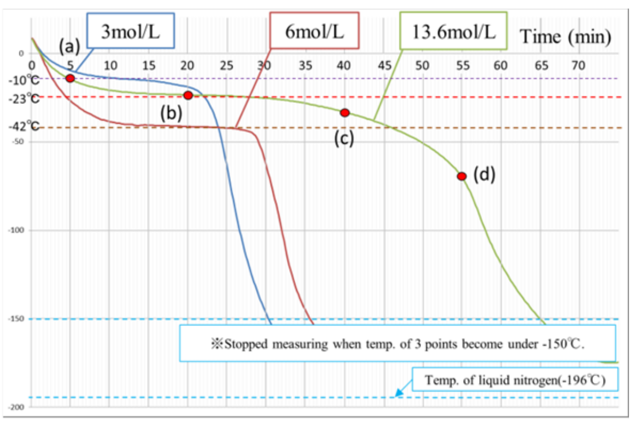


Fig. 3. Results of Beaker test

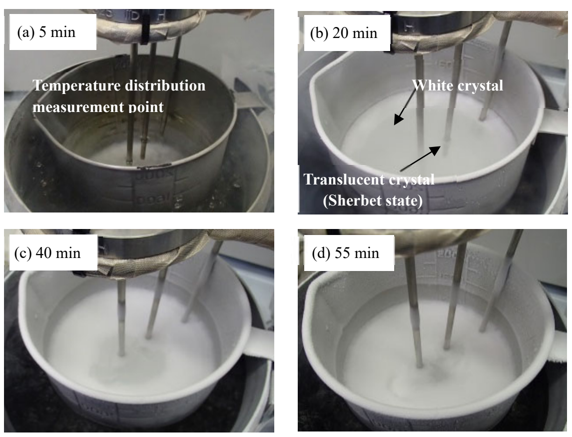


Fig. 4. White crystal formation process (13.6 mol/L)

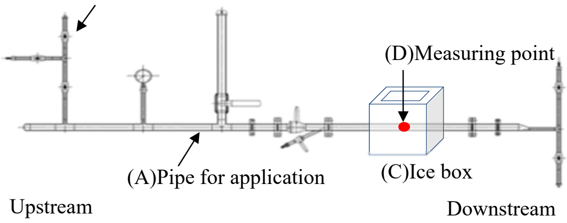


Fig. 5. Mock-up test equipment

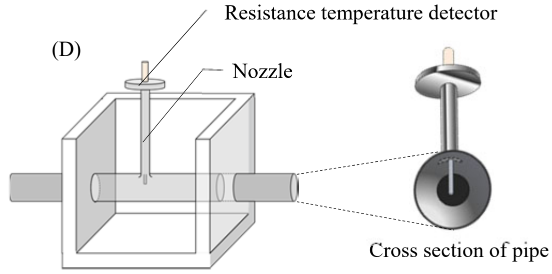


Fig. 6. Schematic description of measurement area of temperature distribution in pipe

5.1. Setting test condition

(A) Pipe for application

As the 40 A-sized pipe which is one of SUS304L pipe is mainly used as nitric acid pipe systems in actual plant, this pipe is selected for the mock-up test.

(B) Pressurization line

To confirm pressure resistance performance of the formed ice plug, a nitrogen gas cylinder is connected with the application pipe, and this system is pressurized from its upper stream side. The test pressure is set on 0.9 MPa by assuming 1.5 times of the pump discharge pressure of chemical reagent pipe system in the actual plant.

(C) Ice box

As we are aiming to simple application of ice plug technology, an ice box (W:294 × D:240 × H:420 × t:34 mm) made by expanded polystyrene is used because of its easy molding. As the aim of this test is ice plug formation which possesses 5 times length of the internal diameter of the pipe (40 mm) [3], the ice box with liquid nitrogen immersion area of over 200 mm height is used. Moreover, the dimensions of ice plug is changeable by taking account of usability on site.

(D) Measuring point of temperature distribution inside pipe

As the nozzle is attached with center of ice box as shown in Fig. 6, resistance temperature detector enables us to measure temperature distribution in pipe.

5.2. Confirmation test for temperature distribution

In order to study the freezing process of nitric acid in pipe, ice plug technology is applied to test equipment shown in Fig. 5, and also temperature distribution in pipe is measured at measuring point shown in Fig. 6.

13.6 mol/L nitric acid was used for confirmation of temperature distribution, because it showed significant hindrance of crystal growing rate originated from freeze concentration process and the highest volume change rate in freezing process.

5.2.1. Test method

(1) The mock-up test equipment is filled with nitric acid and subsequently an ice box is set on application area,
(2) Ice plug technology is performed after this ice box is filled with liquid nitrogen,
(3) Temperature of nitric acid in the pipe is measured from liquid nitrogen filling to ice plug formation completion with resistance temperature detector.

5.2.2. Results and discussion

(1) Compared with the freezing point (-23°C) of 13.6 mol/L nitric acid described in Reference [1], when temperature of nitric acid in pipe reached -60°C, it reached solid-liquid phase equilibrium state at A in Fig. 7. As a result, the freezing point depression of about 40 °C was observed.
(2) Slight temperature rise was observed at B in Fig. 7. Phase transition from liquid phase to solid phase caused this latent heat release.

(3) Subsequently, at C in Fig. 7, the temperature of the nitric acid in pipe was decreased sharply from around -70°C and it reached the liquid nitrogen temperature (-196°C) and became the constant temperature. This phenomenon shows that nitric acid transfers to white crystal completely around -70°C. And after transition, the temperature decrease steeply due to heat release.

Based on results of beaker and mock-up test, we estimate freezing process of nitric acid in the pipe used in this paper. Table 2 shows estimated freezing process of 13.6 mol/L nitric acid.

This estimated freezing process takes account of the result that translucent crystal of nitric acid changes to white crystal around -70°C in beaker test. In addition, it takes account of the freezing point depression originated from freeze concentration process which was observed in mock-up test. We considers that in like manner of beaker test, ice crystal formation starts from pipe wall and it grows toward center of pipe as time passes.

Moreover, 60 minutes was set as the freezing completion time in estimated freezing process. It takes account of the freezing time in mock-up test when nitric acid reaches temperature of liquid nitrogen and becomes completely stable crystal.

5.3. Pressure test for formed ice plug

The function of the ice plug depends on mainly contact state (adhesion / friction force) between internal surface of pipe and ice plug [4]. In general, nitric acid solution shows shrinkage characteristics during freezing and shrinkage change rate is greatly in relation to concentration change of solution. So, we concerned about the decrease of adhesive force. Therefore, in order to confirm pressure resistance performance of each ice plug formed from 3 concentrations, pressure test was implemented under the condition shown in Table 3.

5.3.1. Test method

After ice plug application in same manner described in Section 5.2.1, the mock-up test equipment is pressurized by nitrogen gas from its pressurization line for 15 minutes in order to check presence or absence of liquid leakage resulting from collapse or dissolution of formed plug.

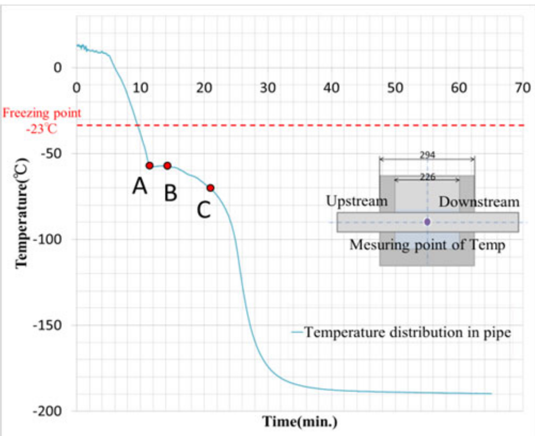
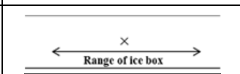
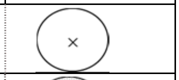
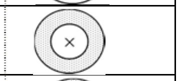
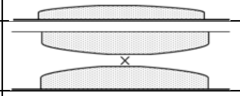
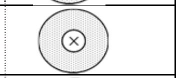
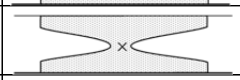
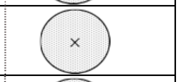
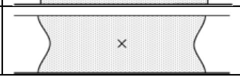
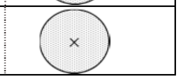


Fig. 7. Result of mock-up test (13.6 mol/L nitric acid)

Table 2. Considered freezing process of nitric acid (13.6 mol/L)

Time (min)	Temperature (°C)	Pipe	Cross-section
Start 1.0	20		
10	−10		
15	−60		
25	−70		
60	−190		

× : Measuring point　　: White crystal

Table 3. Pressure test conditions for formed ice plugs

Concentration of HNO_3	3.0, 6.0, and 13.6 mol/L
Atmosphere temperature	30 °C ± 2 °C
Freezing time	60 min.
Pressure	0.9 MPa, 15min. *

* According to“Chosou no taiatsu siken oyobi kimitsu siken” issued by METI.

Table 4. Application and operation condition of ice plug application to nitric acid pipe systems

Operation Condition	**Atmosphere temperature**	**30 °C ± 2 °C**
	Pipe system	Horizontal pipe (～40A Sch20s SUS304L)
	Refrigerant	Liquid nitrogen(LN_2)
Application Condition	**Freezing time**	60 min.
	Ice box *	W:294×D:240×H:420×t:34 mm
	Pressure	≦ 0.9 MPa

*: W: Length of liquid nitrogen immersion area, D : Pipe inner diameter

5.3.2. Results and discussions

As results of the mock-up test, 13.6 mol /L nitric acid shows the highest volume change rate, and the ice plug formed under freezing time (60 minutes) possessed pressure resistant against test pressure. This ice plug possessed sufficient pressure resistance, so it is considered that volume change rate of nitric acid solution does not affect contact state with internal pipe wall during ice plug formation process. Therefore, similar pressure resistance performance will be obtained in ice plugs formed form 3.0 and 6.0 mol/L nitric acid. Table 4 shows application and operation condition of the ice plug technology which is obtained from the mentioned test results and enables us to apply it to nitric acid pipe systems.

6. Conclusions and future efforts

We obtained a prospect that the ice plug technology is possible to apply 40 A-sized nitric acid pipe system installed in the irradiated nuclear fuel reprocessing plant by using liquid nitrogen (-196°C) as refrigerant with 60 minutes of freezing time. The results in this paper are as follows:

(1) In the beaker test, although nitric acid solution reaches solid-liquid phase equilibrium state at freezing point described in Reference, it is necessary to cool it up to -60°C to -70°C until nitric acid solution transfers white crystal completely.
(2) In the mock-up test, although freezing point depression of nitric acid originated from freeze concentration process was observed, but any effect on plug formation did not be observed.
(3) Ice plug formation can be completed with 60 minutes of the freezing time from estimated freezing process. It was confirmed that ice plug possessed the hardness standing up to test pressure and continuous liquid blockage function.
(4) As it is confirmed that any effect of volume shrinkage characteristic of nitric acid do not affect the ice plug function, it is possible to obtain the ice plug which possesses sufficient pressure resistance.

As future efforts, we will expand application scope of ice plug technology. To achieve this purpose, we are going to apply this technology to pipe systems with other diameters, and also study freezing process and pressure resistance performance of ice plug formed from chemical reagents other than nitric acid used in the plant (sodium hydroxide etc.).

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