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EXPERIMENTAL ANALYSIS OF THE OBTURATION OF PIPES BY ICE PLUGS

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This paper assembles together the knowledge acquired during seven tests of freezing with liquid nitrogen on pipes representing circuits in thermal or nuclear power plants, using different geometrical configurations, steels and initial conditions. These tests made it possible to link the physical phenomena to the appearance of the thermal stresses caused by the freezing. The stresses measured were below the elasticity limit. The speed of temperature fall and the thermal gradients (in the thickness, axial and azimuthal) were measured; this made it possible to define a typical transient regime that may be considered representative of a freezing operation. The tests of the resistance of the plug to pressure showed that it could withstand high pressures. The tests also led to improvements in the technology of the process and the methods used.

1. Why freezing a pipe?

When a defect (or leak) has been revealed on a circuit, pipe or valve and it therefore becomes necessary to carry out a repair operation without first emptying the circuit, and when there is no section division by which the pipe in question can be isolated, it is frequently possible to block it off with an ice plug, and so to repair it or add a valve, or even leave a part of the circuit in service.

This is the purpose of the method of section division by freezing. The ice plug acts as an artificial valve. It enables the circuit to be serviced and repair or modification operations to be carried out without interrupting the functioning of the installation; emptying the pipe system or rendering it inert is avoided.

2. What is the principle of the freezing method?

The principle is as follows: the fluid (water, for example) under pressure in the pipe in question is locally frozen with a refrigerant (liquid nitrogen – LN_2 – for example), thus isolating two sections which are rendered physically independent.

In this paper we are essentially concerned with the case that occurs most frequently in Electricité de France: the freezing of a water pipe with liquid nitrogen. However, what follows remains applicable whatever the fluid to be frozen or the refrigerant.

A box of parallelepiped shape (also called a sleeve or caisson), made to fit the external diameter of the pipe and heat-insulated, is placed around the pipe at the desired position for freezing. It is filled with fine chips of copper to encourage thermal exchange and to render the heat flow homogeneous. The box is made fluid-tight by a "clay" type joint, which behaves very well at low temperature and compensates for metal shrinkage. The liquid nitrogen (at 77 K) is injected into the box until the pipe is completely surrounded with it. The nitrogen level is maintained thereafter. The cold is transmitted to the fluid to be frozen by conduction through the pipe wall. The plug is then formed by annular progression of excentric concentric cylinders and develops towards the axis of the pipe.

3. Application of the process in Electricité de France

This method is used in the gas and oil industry [1,2] on gas and oil pipelines, essentially when a modification is necessary (deviation or connection of circuits, adding valvegear) and for repairing and isolating leaks. Electricité de France proposes to use it for repairing circuits in thermal and nuclear power stations.

To this end, studies on both theoretical and experimental level have been undertaken in order to understand better the physical phenomena that occur during freezing and to evaluate the effects of the latter on the piping.

4. The thermal phenomena

The thermal phenomena that occur during the process are extremely complex:

- conduction in the different materials: steel, ice, water;
- forced convection with nucleate boiling between the liquid nitrogen and the wall of the pipe;
- natural convection or leak flow bringing heat that discourages the formation of ice;
- phase change at the ice-water interface; the problem is thus not linear.

Furthermore, it is three-dimensional: there are temperature gradients in the thickness, around the circumference (the level of refrigerant varies, at least at the beginning of the process) and along the axis.

A complete model of the thermal field covering all these phenomena would therefore be very complex and so far it has not been possible to establish one.

A methodology based on a number of simplifying hypotheses (in particular for the thermal shock, etc.) and some initial experiments was thus developed. This analytic methodology is briefly described in refs. [3] and [4]. It was then completed and a computer program prepared with the essential object of answering the questions that the operator may ask before any freezing operation: is freezing physically possible? Can it be carried out without harming the piping? How big is the stress field?

5. Experimental analysis

Tests on the freezing of pipes typical of circuits in thermal and nuclear power plants were then carried out with different geometrical configurations (thickness,

Table 1
The freezing tests carried out

Date	Test	Steel type	Int. diameter (mm)	Thickness (mm)	Int. flow (kg/s) (1)	Init. temp. °C (3)	Final temp. (°C)	Remarks
10 03 76	1	316 L Z3 CND 17.12	289	33	? (2)	120 (max) 50 (min)	−196	Inclined pipe (45°)
07 03 79	2	A 37	340	8,5	0	50		Horizontal pipe Ferritic steel brittle at low temp.
12 03 80	3				0,06	22		
05 11 80	4	type 316 Z5 CND 17.12	173	23	0,06	27		Horizontal pipe
11 03 81	5				0,06	37		
29 09 82	6	304 L-Z2 CN 18.10	222	25,4	0,04	35		Horizontal pipe Circumference weld in the frozen part (for metallurgical tests)
27 10 82	7				0	37		

Notes:
(1) The circulation flow corresponds to that of a realistic leak in a power plant.
(2) In this case the circulation flow was caused by movements of natural convection inside the pipe.
(3) The initial pressure of the fluid to be frozen was around 2 to 3 bar.

diameter inclination), initial conditions (pressure, temperature, internal flow), and steel nature (ferritic or austenitic). The objectives were as follows:

- to check the hypotheses and the analytical process [5] adopted;
- to ascertain the speeds of temperature fall and the thermal gradients, as well as the stresses occurring during the process;
- to test improvements to the technology and freezing procedure;
- to provide samples that have undergone actual freezing one or several times, for metallurgical checking and expert opinion (low-temperature characteristics, tenacity, speed of appearance of martensite); this metallurgical aspect will not be covered here.

The characteristics of these seven tests are given in table 1.

After a brief description of the test methods, this paper gives a general summary of the tests, explains the physical phenomena that were observed and links these to the stresses of thermal origin that appeared. It then gives measurements of some characteristic parameters of the freezing process.

6. The test method and equipment

A test facility was constructed to enable freezing tests to be performed on any form of pipe (not flanged) and to simulate any initial condition. The initial conditions for each test are given in table 1; they correspond to very low initial stresses, not taken into account in the recordings.

Numerous measurements (pressure, water and metal temperature, strain, refrigerant level, flow of circulating water) were carried out (about 70 per test); they required measuring methods at very low temperature to be developed. Measurement error was less than 4°C for the temperatures and than 30 MPa for the stresses. Fig. 1 gives an example of the siting of the measuring points. Note that the zone with the most instrumentation was the freezing zone (which was three times the diameter of the pipe long), and that the metal temperatures were measured at different depths on the circumference of the pipe, and that the strains were measured in the two principal directions.

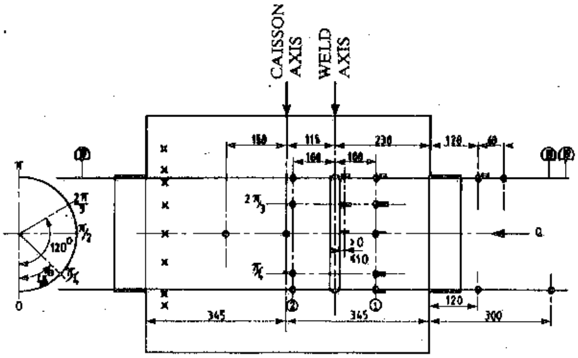

FIG. 1 : AN EXAMPLE OF THE INSTRUMENTATION (TEST N° 6)

KEY :
- • THERMOCOUPLE (METAL)
- ○ THERMOCOUPLE (WATER)
- ⇔ STRAIN GAUGES
- x MEASUREMENT OF LN2 LEVEL
- Ⓟ MEASUREMENT OF PRESSURE

Tests of detection of the ice plug (by ultrasonics and acoustics) were carried out but were not conclusive (difficulties in performing and interpreting the tests). They are not mentioned here.

Photo 1 is a general view of the test device with a freezing test in course. Photo 2 shows the freezing caisson and reveals the presence of an ice plug by the frosting on the pipe on each side of the caisson.

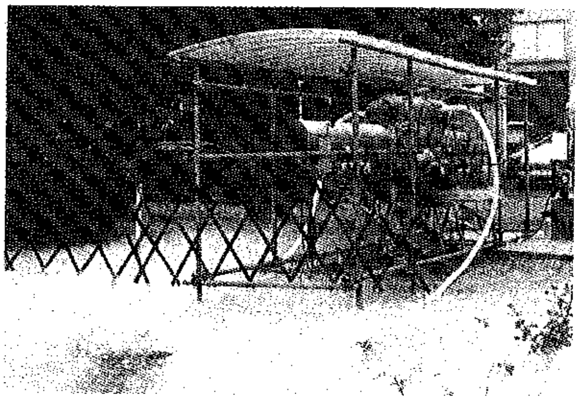

Photo 1. The freezing apparatus during a test.

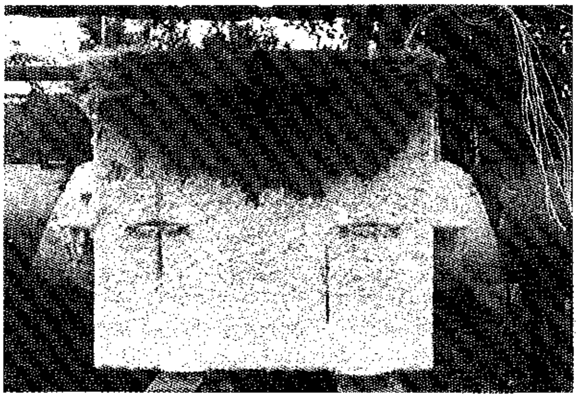

Photo 2. Frosting on the pipe indicates the formation and position of the ice plug.

7. The technology of the process

Compared with the studies [1,2] a significant improvement in the freezing procedure was achieved. In particular, the caisson was provided with a cover, thus creating a closed parallelepiped box with two lateral orifices which, when connected to two flexible pipes, enabled the nitrogen vapour to be evacuated away from the experimental zone (a possibility that is feasible in situ).

The cover, with its filling tube was made fluid-tight with thermoplastic paste. Nitrogen was supplied automatically via a cryogenic electric valve actuated by position sensors (on or off sensors affected by the physical state of the fluid – liquid or vapour).

After filling, this detection device maintained the nitrogen level to ensure that the pipe remained covered throughout the period of freezing.

8. The different phases of a test

All the freezing test followed the same pattern:

- an initial condition corresponding to an actual situation in a power plant was established;
- a freezing phase was begun, divided into two sequences:
  - filling the caisson (transient regime with thermal shock), beginning with the injection of liquid nitrogen and ending when the pipe is completely immersed,
  - the freezing proper during which cold is transferred, starting with complete immersion of the pipe and ending with formation of the plug;
- the tests of the resistance to pressure of the plug thus formed, up to the test pressure of the test circuit;
- the thawing phase, with the plug still under pressure.

Each of these phases is mentioned in turn in the following sections.

9. The filling phase

9.1. Thermal analysis

This phase begins with injection of liquid nitrogen up to complete immersion of the pipe. It is during this phase that the greatest temperature differences, the greatest thermal shocks and consequently the most severe thermal stresses occur. From the stress point of view, this is the critical part of the process.

All points on the pipe experience successively:

- *Nitrogen vapour:* the convectional exchanges are small, the temperature decreases slowly, the tensile stresses due to thermal gradients in the thickness $(\partial\theta/\partial r)$ and axial $(\partial\theta/\partial z)$ are small and the compressive stresses due to thermal bending caused by the azimuthal gradient $(\partial\theta/\partial\omega)$ are not negligible.
- *The liquid-nitrogen level:* this level fluctuates widely. It is the level of a liquid that is boiling at atmospheric pressure; there are greater thermal exchanges, the temperature falls, and stresses increase.
- *Liquid nitrogen:* convectional exchanges are very substantial, and the phenomenon of nucleate boiling occurs. The steep gradients observed cause very heavy tensile stresses. The moment when the liquid nitrogen just touches the point under observation is the most critical one. All the stresses are of tension and they are cumulative.
- *The liquid nitrogen after homogenization of the metal temperatures:* the gradients die away and the stresses decrease with the return to thermal equilibrium.

Fig. 2, which shows the temperature movements during test No. 7 at a given point (the middle generating line) and at different depths, illustrates these different stages well. Fig. 3, which is obtained from test No. 5, also gives the changing temperatures in the metal at the middle generating line at three depths, and the water temperatures opposite this point.

9.2. The thermal gradients observed

During the tests, some temperature dissymetry caused by the distribution of the liquid-nitrogen flow around the pipe, was observed.

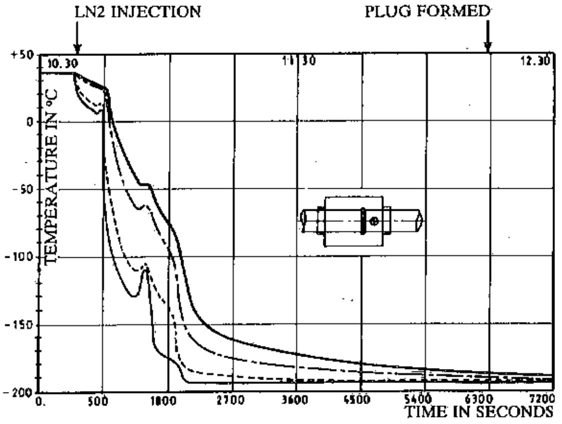

FIG. 2 : TEST N° 7 ON 27.10.82
TEMPERATURE CHANGE METAL $\omega = \pi/2$

KEY : ___ EXT. METAL TEMPERATURE
_._ METAL TEMP. AT 14 MM DEEP
.... METAL TEMP. AT 7 MM DEEP
▬ METAL TEMP. AT 21 MM DEEP

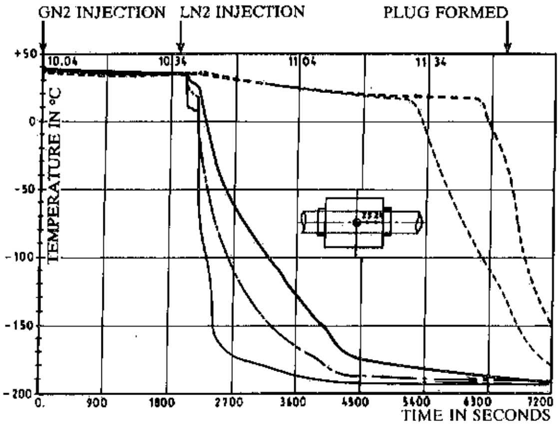

FIG. 3 : TEST N° 5 ON 11.03.81
MIDDLE GENERATING LINE $\omega = \pi/2$

KEY : —— EXT. METAL TEMPERATURE
▬▬ METAL TEMP. AT 17 MM DEEP
– – – METAL TEMP. AT 7 MM DEEP
–·–·· WATER TEMP. NUMBER 23
– – – WATER TEMP. NUMBER 24

Table 2 gives the maximum experimental values for the speed of temperature drop $\partial\theta/\partial t$, the temperature gradients, the temperature difference between the upper and lower generating lines, for the pipes under experiment. These parameters are large, and are responsible for the thermal-stress field

Some degree of homogeneity was found for the gradients measured in tests Nos. 1, 4, 6 and 7, made on austenitic steels and thick pipes. In test No. 5, also on a pipe in austenitic steel, the freezing process was much faster and caused a greater thermal shock, with a steep gradient in the thickness (and so heavier thermal shocks in the thickness), but on the other hand a smaller difference between the upper and lower generating lines (and thus small stresses on a given level). With regard to tests Nos. 2 and 3 on the ferritic-steel pipe, it is difficult to form a conclusion. For, we did not have control of test No. 3 and we met several problems in filling (leaks, level stabilization, etc.) and the filling phases were very sudden.

The convection coefficients between the liquid nitrogen and the steel were deduced from the test data; they are of the order of 900 W m^{-2} K^{-1} and 2200 W m^{-2} K^{-1} for the metastable and nucleate boiling phase, for the austenitic and ferritic steel respectively.

9.3. Mechanical analysis

The gradients observed cause stresses of thermal origin attributable to the freezing alone.

Table 2
The thermal gradients observed (maximum values)

Test		1	2	3	4	5	6	7
Steel type		316 L	ferritic		316		304 L	
Caisson filling time (mins.)		40 ?	31	29 ?	38	2	34	11
Thickness of pipe	mm	33	8.5		23		25.4	
Speed of temperature drop ($\partial\theta/\partial t$) (1)	°C/mn	– 66	– 50	– 144	– 66	– 151	– 59	– 59
Gradient in the thickness ($\partial\theta/\partial r$) (2)	°C/mm	4.2	7.7	3.5	6.1	7.2	4.2	4.5
Axial gradient ($\partial\theta/\partial z$) (3)	°C/mm	0.3	0.5	1.2	1.2	1.2	0.9	0.8
Max. temperature difference between upper and lower generating lines of pipe (4)	°C	not mea-sured	207	194	185	75	185	132

Notes:
(1) calculated speed, obviously on the external skin.
(2) Average calculated gradient between the thermocouple on the external skin and the deepest internal one.
(3) Value between two distant points on the same generating line – one in the frozen zone, and the other outside the caisson.
(4) Maximum difference noted, equal to the difference between the highest and lowest temperatures in the same section.

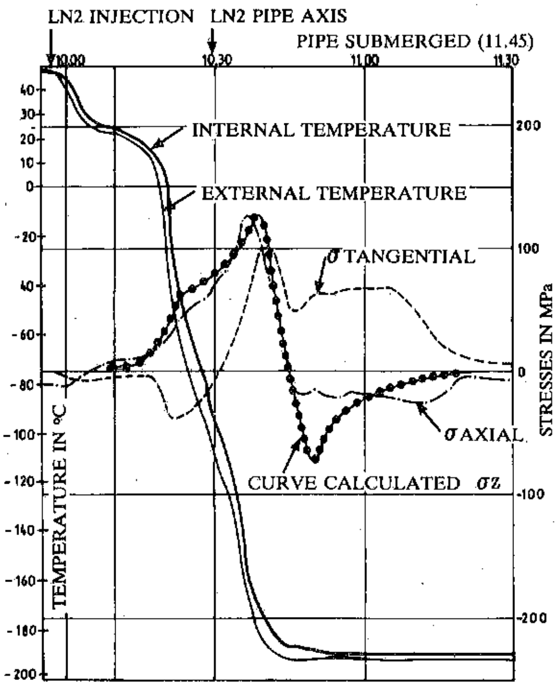

FIG. 4 : TEST N° 2 [3]
TEMPERATURES AND STRESSES AT $\omega = 0$

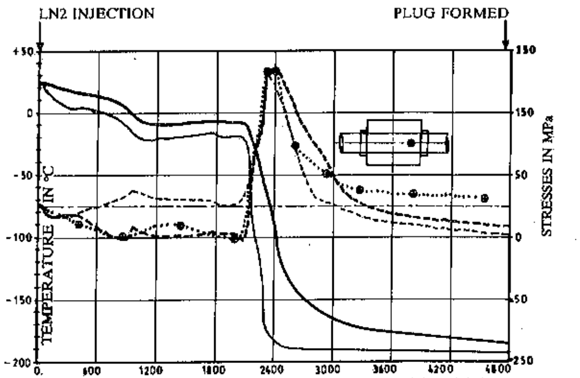

FIG. 6 : TEST N° 4
TEMPERATURES AND STRESSES AT $\omega = \pi/2$

KEY :
— EXTERNAL METAL TEMPERATURE
— INTERNAL TEMPERATURE (AT DEPTH OF 14 MM)
--- TANG. STRESS
--- AXIAL STRESS
-.-- ORIGIN GAUGE AXIS
..●.. σz CALCULATED

Figs. 4 to 8 illustrate our results for different geometrical positions and different tests. In these figures are shown the external and internal temperatures for the point in question, the main stresses measured in the tangential and axial directions, and the resultant axial thermal stress, calculated with the help of ref. [5], these stresses being due to the freezing alone.

We deduced the following information:

(a) During the phase of convectional exchange between the nitrogen vapour and steel, the tensile stress due to the gradient in the thickness is deduced from the compressive stress due to the azimuthal gradient.

(b) During the phase of convectional exchange between the liquid nitrogen and the steel, the two previous stresses are tensile and cumulative. It is during this phase that the steepest thermal gradients and the highest stresses occur.

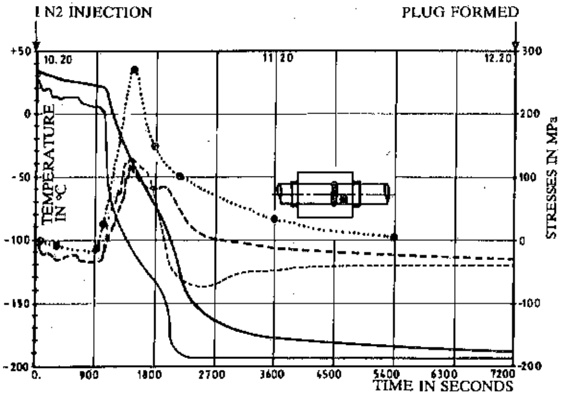

FIG. 5 : TEST N° 6 ON 29.09.82
TEMPERATURES AND STRESSES AT $\omega = \pi/4$

KEY :
— EXTERNAL METAL TEMPERATURE
— INTERNAL METAL TEMPERATURE AT 21 MM DEEP
--- AXIAL STRESS
--- TANG. STRESS
··●·· σz CALCULATED

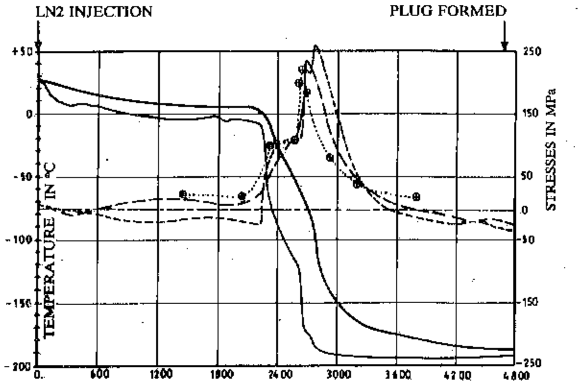

FIG. 7 : TEST N° 4
TEMPERATURES AND STRESSES AT $\omega = 2\pi/3$

KEY :
— EXTERNAL METAL TEMPERATURE
— INTERNAL TEMPERATURE (AT DEPTH OF 17 MM)
--- TANG. STRESS
--- AXIAL STRESS
--- ORIGIN GAUGE AXIS
..●.. σz CALCULATED

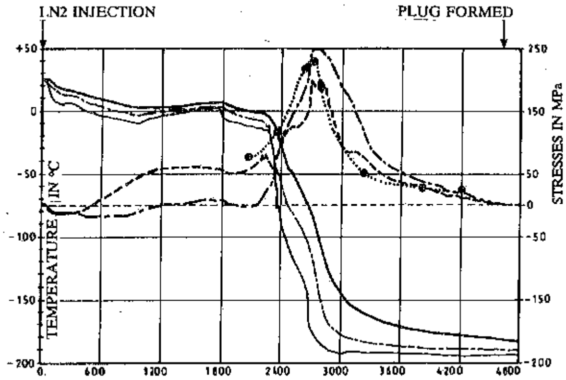

FIG. 8 · TEST N° 4 · TEMPERATURES AND STRESSES AT $\omega = \pi$

(c) When the temperatures are equalling out, the stress due to the azimuthal gradient has completely disappeared and that due to the thermal gradient in the thickness diminishes progressively because of the thermal inertia of the pipe (with the thick pipe) and becomes negligible.

(d) The axial and tangential stresses measured are similar.

(e) The maximum values for the stresses were observed at the generating line $2\pi/3$, when the temperature on the external skin of the metal is of the order of $-110°$C. These stresses (a maximum of 260 MPa, corresponding to 950 μm/m in the case of the austenitic steels) were always below the elasticity limit (0.2% at this temperature), though close to this limit for test No. 3.

(f) The filling mode, which varied widely in all tests, did not significantly influence the degree of stress.

(g) A freezing process constitutes a fatigue cycle with a half-amplitude of a maximum of 370 MPa and 260 MPa respectively for the ferritic and austenitic steels under experiment; this corresponds to the using up of 1/3000th and 1/90000th respectively of the life span of the steel due to the freezing alone, not counting any other stress, which in point of fact can be neglected.

10. The frozen phase

The cold passes towards the inside of the pipe and causes the formation of ice in concentric but excentric cylindrical layers. It combats the various heat contributions: mass enthalpy of the water, latent heat of solidification, contribution by the circulation flow and/or natural convection. This phase of cold transfer thus lasts from immersion of the pipe in the nitrogen up to formation of the plug.

The metal temperatures outside the caisson were also recorded. We did not find any sudden change in these temperatures. The strains and stresses measured at these points proved negligible.

Table 3 gives the experimental values of some characteristic parameters for a freezing operation.

10.1. Resistance of the plug to pressure – Thaw phase

As soon as the plug is formed and the injection of refrigerant consequently stopped, the cold moves from

Table 3
Characteristic parameters of a freezing operation

Test	1	2	3	4	5	6	7
Internal pipe diameter (mm)	289	340		173		222	
Obturation time (minutes)	196	162	117	47	71	87	86
Consumption of liquid nitrogen (dm3)	5000	2001	2000	307	526	580	560
Position of 0°C isotherm (frost) (mm)	not measured			160	150	162	167

Table 4
Tests of resistance of the ice plug to pressure

Test	1	2	3	4	5	6	7
Plug resistance to pressures (bars)	45	16	16	139	80	250	250
Time the plug was maintained at the pressure (hours)	20	46	22	–	–	13	13
Remarks	Maximum pressure for the test circuit			Pressure at which the plug slips		Tests not significant : circumference weld present	

the coldest to the warmest parts. Initially the ice plug progresses, just after its formation. It generally has a symmetrical aspect. The lower part of the plug is longer, and the central part is slightly hollowed out, because of phenomena of natural convection.

Tests of resistance to pressure were carried out, generally just after the formation of the plug, and this pressure was maintained for some time, until the temperature rose to close to 0°C at the ice–steel interface; the pressure was progressively lowered to 1 bar. At this point, the plug lost fluid-tightness.

The results are given in table 4. Note that the seven tests carried out are insufficient for conclusions to be drawn and systematic testing is necessary. The most interesting tests are Nos. 4 and 5. The pressures indicated are those at which the plug first became detached. After this first detachment, we found that the plug could stand even high pressure, and so on on successive movements until the maximum pressure for the test circuit was reached.

Lastly the thaw phase, even when accelerated, did not show significant changes in the thermal gradients and stresses.

10.2. Conclusion

These tests, which are full-scale simulations, have shown that it is possible to freeze on two occasions, at the same place, a thick pipe of large diameter in ferritic or austenitic steel, typical of a circuit in a thermal or nuclear power plant. It was possible to link the physical phenomena to the appearance of the stresses. These stresses, of thermal origin, caused by the freezing process alone, proved to be lower than the limit of elasticity to cold (at about −110°C). The speeds of temperature drop observed were of the order of 80°C/min. The gradients in the thickness (of the order of 5.5°C/mm on average), the axial gradients (of the order of 1.0°C/mm) and the differences between the upper and lower generating lines of the pipe (of the order of 170°C) were responsible for the thermal shocks during the phase of filling the caisson. This was incidentally the critical phase of the process (sudden transient regime of thermal shock). The tests of pressure resistance are insufficient. However, they showed that an ice plug could easily withstand pressures of over 80 bar. The prediction calculation for the characteristics of freezing based on the analytical procedure set out in ref. [5] is pessimistic. The freezing processes used, which varied widely from test to test, did not show any significant difference.

The technology for the freezing operation was improved (automatic nitrogen feed regulation, confined freezing zone, evacuation of vapour, procedure to be used), and was shown to be efficient in practice. However this process must always seek to reduce the thermal stresses as far as possible, by precooling the pipe over its whole circumference followed by immersing it as quickly as possible in a bath of liquid nitrogen. And furthermore, the safety aspect of the use of this process on a power-plant circuit must also be gone into.

References

[1] M. Gatineau and M. Thomas, Recherche de fuite sur canalisation; Méthode de sectionnement par congélation, Conférence ATG-1972.

[2] Pipe-Line Service (PLS), Méthode d'obturation de tuyauteries, PLS report (1976).

[3] A. Lannoy, Problèmes thermiques posés par la congélation d'un fluide dans une tuyauterie, Rev. Gén. Thermique 220 (1980) 311.

[4] A. Lannoy, L'obturation des tuyauteries par bouchon de glace, Technical Seminar on Maintenance, Paris la Défense, 1981.

[5] B. Flaix and A. Lannoy, Détermination des possibilités et des conditions de congélation d'une tuyauterie, EDF, Direction des Etudes et Recherches, Internal report, HP/219/81/12 (March 1981).