EXPERIMENTAL STUDY OF CHLORIDE-INDUCED STRESS CORROSION CRACKING (CISCC) FOR AUSTENITIC SUS 304L UNDER THERMAL INSULATION

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ABSTRACT

Chloride-Induced Stress Corrosion Cracking (CISCC) has been frequently reported in the petrochemical industries where it usually causes failure to the austenitic stainless steel structures encased with thermal insulation in a chloride-containing environment. This study aims to analyze and evaluate the risk of chloride concentration and different types of insulation materials on CISCC of Austenitic SUS 304L. The experimental study was carried out for 14 days using U-bend specimens through the Drip Test apparatus, as per ASTM G30 and ASTM C692. U-Bend specimens were installed onto the test rig and shielded with different insulation materials. Sodium chloride (NaCl) salt solutions were periodically dripped onto the specimens, to simulate the wet and dry cycle. Insulation materials used were rockwool, calcium silicate and perlite, whereas the concentrations of NaCl solutions were set at 0.1, 1.0 and 3.5 wt.%. The specimens were inspected using dye penetration tests (DPT), stereo microscope and optical microscope throughout the study to determine the CISCC susceptibility. Results showed that cracking was observed on the specimen with rockwool insulation at 3.5 wt% NaCl and 90°C. Rockwool has a high water absorption capacity at which saltwater will evaporate when in contact with a hot metal surface, resulting in the build-up of salt deposits of high concentration over time. SCC was not observed on other specimens with different conditions, but salt deposits, general corrosion and pitting corrosion were found. From the study, the thermal insulation of rockwool was found to have the highest tendency to cause SCC, followed by calcium silicate and perlite.

Keywords: *SUS304L, chloride-induced stress corrosion cracking, insulation materials, U-Bend specimen, drip test*

INTRODUCTION

Petrochemical and oil and gas industries mostly use stainless steel SUS 304L as the main structural materials for the pipelines and pressure vessels due to its ability to withstand corrosive conditions [1]. Thermal insulation (usually rockwool, calcium silicate and perlite) is used to minimize the heat loss to the surroundings, but its performance will be reduced when in contact with water [2]. As such, jackets (usually aluminium roll) is used to prevent water from entering the insulation layer. However, improper installation or breakage in jacketing and not quickly repaired can lead to the ingress of water moisture to the insulation materials [3]. The presence of moisture resulted from the rainwater could cause the chloride ions to be leached out from the insulation and trapped on the metal surface [4]. Salt compounds could accumulate on the material surface as a result of the wet and dry cycle, or more specifically, the repeated rainy and sunny days [5]. The mechanism behind this is during evaporation, only water evaporates but not the salt contents.

SCC is the cracking failure resulted from three factors –susceptible material, corrosive environment and tensile stress [6]. It can lead to unexpected sudden failures of normally ductile materials, especially at elevated temperatures. Since SCC is highly chemically specific, certain materials are likely to undergo SCC even they are just exposed to mild chemical environments. Chloride-Induced Stress Corrosion Cracking (CISCC) is prevalent in the chloride-containing environment, such as the petrochemical plants at the coastal sites, and this usually happens under thermal insulation [7]. CISCC is one of the most common reasons why austenitic stainless steel pipelines deteriorate in the petrochemical industries [8]. Austenitic stainless steel pipeworks, which are usually covered with insulation layers, are prone to CISCC in the presence of moisture and chloride ions [9].

A recent study done by Mintz et al. [10] on the coastal salt effects on SCC of SUS 304 showed that SCC took place between 35°C and 80°C in the presence of high calcium chloride humidity. Another similar study done by He et al. [11] on the effects of salts on SCC of SUS 304 showed that SCC did not occur on any specimen, but there was general corrosion that occurred on the as-received and sensitized specimens deposited with NH_4HSO_4. Besides, the grain boundary attack has occurred on the welded specimens exposed to NH_4HSO_4. However, the recent studies are done only on the SCC or under thermal insulation itself but not the combination of both the SCC with thermal insulation [10]-[15]. Specifically, there are limited experimental work on CISCC under thermal insulation, hence the need to perform this work.

This work aims to simulate the SCC of service pipelines in the petroleum refinery plants through the use of drip test apparatus and U-Bend Specimen [16],[17]. The drip test apparatus has the closest similarity to the real-life scenario on simulating the pipelines and environment in petrochemical industries. The previous test methods, dana test (transfer of NaCl solution through wicking process), simple immersion test in saltwater and other test methods have different study purposes and limitations. Hence, they are not a suitable method to be used for this study. As such, the specific objective of this study was to evaluate the risk of sodium chloride concentration and different types of insulation materials on CISCC of Austenitic SUS 304L. At the end of the study, a CISCC susceptibility graph combining all the variables was constructed.

METHODOLOGY

Sample Preparation

Laboratory experiments using drip test apparatus conducted to imitate the service pipelines in practice to understand the mechanism of SCC on Austenitic SUS 304L under thermal insulation. This experiment leads to the use of a U-Bend specimen as per ASTM G30 for the experimental simulation [17]. The sample preparations are discussed below shows the preparation of the U-Bend specimen was done by bending a flat bar into a U shape via two-stage stressing method as per ASTM G30 [17], and Figure 1 shows the final dimensions of the U-Bend specimen.

i) U-Bend Specimen (ASTM G30)

U-Bend specimen is able to simulate the two criteria of imitating the stress corrosion cracking due to its characteristics of having tensile stress and being a susceptible material (Austenitic SUS 304L). The corrosive environment was simulated through the drip test apparatus at which a NaCl solution with different concentrations was dripped onto the insulated test specimen.

ii) Insulation Materials

Insulation materials were used to cover the U-Bend specimen, with a cavity that is hollowed out in half part of the insulation material, as shown in Figure 2. The insulation materials to be tested in this study include rockwool, calcium silicate and perlite.

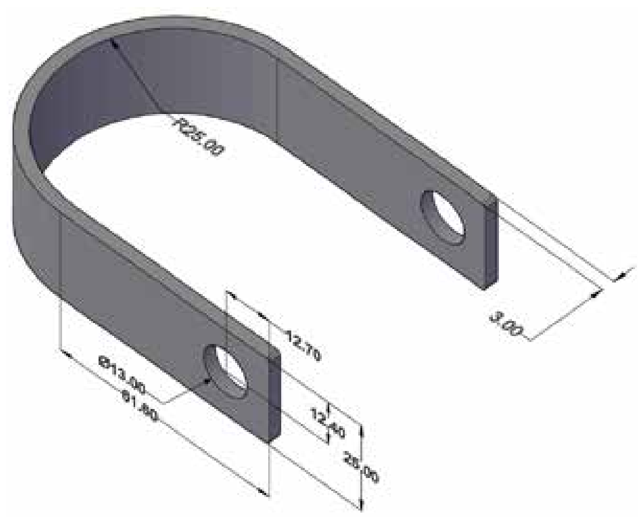

Figure 1 Final U-Bend specimen with dimensions

Figure 2 Preparation of insulation materials

iii) NaCl Solution

The preparation of NaCl solution was prepared by simply adding 0.1, 1 and 3.5 wt.% NaCl into distilled water and stirred well. The concentrations of NaCl solution were set at 0.1, 1.0 and 3.5 wt.% to simulate inland to the coastal environment.

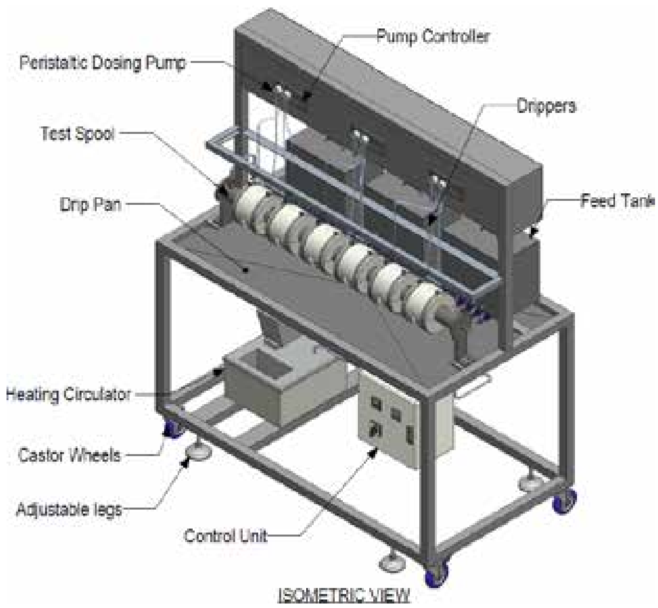

Figure 3 Drip test apparatus set-up

Drip Test Apparatus Set-up (ASTM C692)

The experimental set-up of the drip test was designed and developed as per the ASTM C692 [16]. It was developed such that the insulated U-bend specimen could be mounted on the test spool and heated through a heat circulator. This was intended to simulate the wet and dry cycle of the repeated rainy and sunny days in real-life cases. Besides, three feed tanks containing sodium chloride solution with different concentrations were controlled by the pump controller to drip the solution on to the insulated U-bend specimens. The whole system was powered up by a control unit connected to the main socket. Figure 3 shows the entire system of the drip test apparatus set-up developed.

Test Matrix

The test matrix was designed by varying NaCl concentration and insulation materials. The duration of the SCC test was 14 days. The test matrix of the SCC tests for the study is shown in Table 1. All the 18 tests were inspected through visual examinations, dye penetration tests and stereo microscopic views. Before proceeding to the optical microscopic views, the specimens were cut, ground and polished as well as using an electrolytic etching process to expose the grain boundary.

Table 1 Test matrix for CI-SCC Tests

Parameter	Description
Duration (Days)	14
Initial pH	6.0
Type of Specimen	U-Bend Specimen (As-received SUS 304L)
Total Tests	18
Thermal Insulation	Rockwool, Calcium Silicate & Perlite
Temperature (˚C)	90
NaCl Concentration (wt%)	0.1, 1.0 & 3.5
Drip Details	21 ml/ drip & 12 drips/ day
Evaluation	Visual Inspection, Dye Penetrant Inspection, Stereo Microscopic View & Optical Microscopic View

RESULTS & DISCUSSION

Drip Test (Overall Results)

Based on the inspections done through visual inspection, dye penetration tests, stereo microscopic views as well as the optical microscopic views, the overall results, in terms of salt deposits, general corrosion, pitting corrosion and SCC occurrence were recorded and are tabulated in Table 2 to Table 5.

SCC only happened on the condition where the thermal insulation used was rockwool with the 3.5 wt% NaCl concentration. U-Bend specimens with other conditions found to have salt deposits, general corrosion and pitting corrosion. The thermal insulation layer created an environment where it would trap the moisture of the NaCl solution once it dripped onto the specimen. Rockwool had the highest absorption rate, being one of the reasons that caused SCC to occur on the specimen. Besides, the NaCl solution dripped on the specimen was dried up instantaneously when the solution touched the specimen. Thus, the high-temperature test spool would cause the solution to dry up faster, and the repeated wet/ dry cycle would accumulate the salt contents on the specimen. The mechanism behind this was that during evaporation, only water evaporated, leaving salt contents to remain on the trapped surface. As a result, the highly concentrated salt solution would result in higher and higher concentration overtime on the surface. Generally, rockwool had the highest tendency to cause corrosion due to its highest water absorption capacity, and this followed by calcium silicate and perlite.

Besides, corrosions and cracks happened underneath the salt deposits because that was the spot with the highest concentration. It also created a close environment as the salt deposits had already hardened. The reaction underneath the salt deposits region was not disturbed by external factors but an enclosed environment with a localized attack. The pH values on this spot might be different, and it would be referred as crevice corrosion. Pitting corrosion could initiate underneath the salt deposits, and it might also propagate and caused SCC. The location of the salt deposits consisting high concentration of chloride ions would be harmful to stainless steel as it could damage the protective layer or the chromium oxide. It would cause the protective layer to disappear locally, provoked all the other aggressive ions, including the hydrogen ions, to start attacking the point. Hence, chloride ions would be dangerous to SUS as it would dissolve the protective layer, damaging the spots locally. In turn, it would result in general or pitting corrosion, being the initiation to cause SCC.

In this work, the results showed that all the variables play their role in contributing to SCC. With the high-temperature specimen and through the wet and dry cycle, the NaCl solution would immediately vaporize once it touched the specimen, leaving the salt contents trapped on the surface.

This would result in an increased NaCl concentration over time due to the accumulated salts. It should be noted that the fibrous structure of the rockwool imaterial has a higher water absorption capacity, acting as a collecting media of the salts as it tends to retain the moisture within its insulative space. Perlite, on the other hand, is hydrophobic and does not hold the moisture well becasue of its expanded granular structure. There was no any SCC occurred on the specimen because salt compounds were not trapped on the specimen due to the perlite's characteristic of having low water absorption capacity.

Table 2 Overall drip test results

Thermal Insulation		Rockwool			Calcium Silicate			Perlite		
Temperature (˚C)		90			90			90		
NaCl Concentration (wt%)		0.1	1.0	3.5	0.1	1.0	3.5	0.1	1.0	3.5
Results:	**Salt Deposits**	✓	✓	✓	✓	✓	✓	X	X	X
	General Corrosion	✓	✓	✓	X	✓	✓	X	X	✓
	Pitting Corrosion	✓	✓	✓	X	X	✓	X	X	X
	Cracks	X	X	✓	X	X	X	X	X	X

Table 3 Results (Day 14) for specimens under rockwool insulation

Insulation Material: Rockwool		Temperature: 90°C	
Concentration of NaCl (wt.%)	0.1	1.0	3.5
Visual Inspection			
Dye Penetration Tests			
Stereomicroscopic Views			
Comments	a. General corrosion and pitting on the surface. b. Minor rockwool precipitate.	a. General and pitting corrosion occurs on specimen. b. Minor rockwool and NaCl precipitate.	a. Major pitting and general corrosion occur on specimen. b. Major rockwool and NaCl precipitate.

Table 4 Results (Day 14) for specimens under calcium silicate insulation

Insulation Material: Calcium Silicate		**Temperature: 90°C**	
Concentration of NaCl (wt.%)	**0.1**	**1.0**	**3.5**
Visual Inspection			
Dye Penetration Tests			
Stereomicroscopic Views			
Comments	a. Minor discolouration on the surface of specimen.	a. General corrosion occurs on the part where the NaCl solution dripped.	a. Major pits occur on the centre of the U-bend specimen. b. Indication of cracking.

Table 5 Results (Day 14) for specimens under perlite insulation

Insulation Material: Perlite		**Temperature: 90°C**	
Concentration of NaCl (wt.%)	**0.1**	**1.0**	**3.5**
Visual Inspection			
Dye Penetration Tests			
Stereomicroscopic Views			
Comments	a. No discolouration observed on the specimen. b. Coagulates of NaCl form red spots.	a. No discolouration observed on the specimen. b. Coagulates of NaCl form red spots.	a. Minor general corrosion on the surface of the specimen.

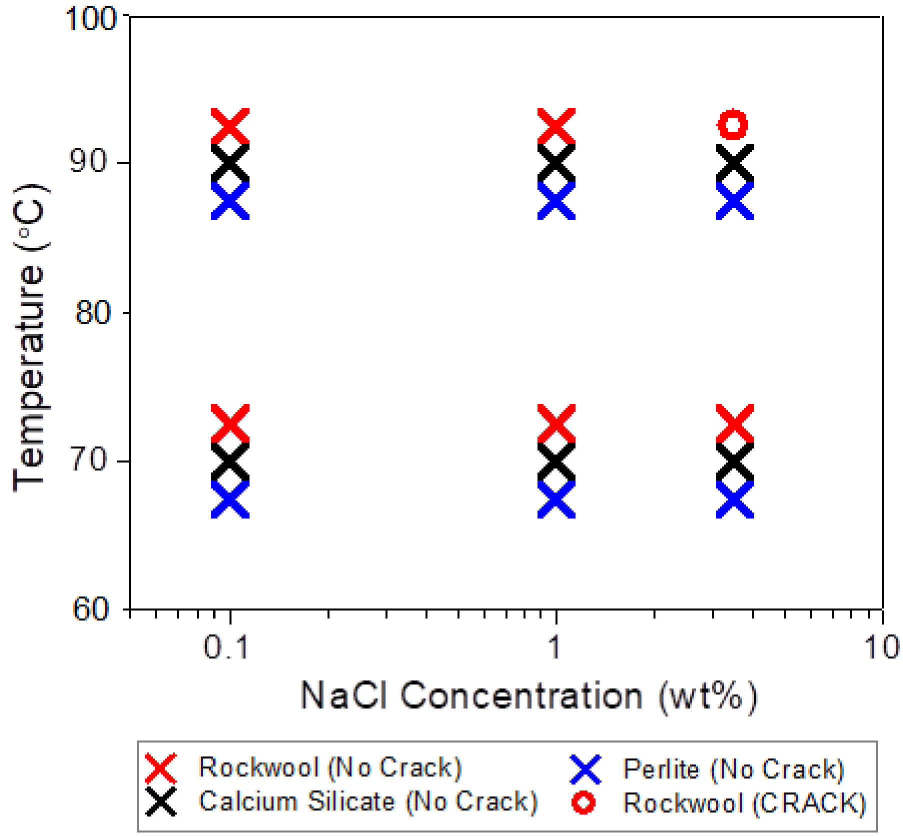

Figure 4 Effect of NaCl concentration on the Cl-SCC susceptibility of SUS 304L at 90°C

Changes in pH Measurement

Initial pH values of the NaCl solution was adjusted to 6.0. During the SCC test, in-situ pH was measured using pH strip paper at the dripped spot on the insulated test specimens. All pH values were found to have increased because of the insulation materials' characteristic of being alkali in nature. Specifically, the pH value of the specimen increased to 7.14, 7.94 and 8.98 for perlite, calicum silicate and rockwool insulations, respectively. The rockwoll insulation showed the highest rise in pH due to certain soluble constituents in the rockwool material that were mostly oxides of alkaline metals.

Visual Inspections/ Dye Penetration Tests/ Stereomicroscopic Views

Tables 1 to 4 show the visual inspection, dye penetration tests and stereomicroscopic views of the specimens under rockwool, calcium silicate and perlite insulations, respectively. Visual inspection results showed that the corrosion severity was the highest for specimens under rockwool insulation. Detailed observations for all the specimens exposed to different conditions can be referred to in the tables. From dye penetrant inspections, cracks were found before proceeding with further inspections.

Optical Microscopy

Once the crack locations were identified, cracked surface was prepared into metallographic sample and optical microscopy was performed at 200X and 500X magnification to determine the exact crack profile and its length.

Of all the specimens, cracks were only detected in U-bend specimens under rockwool insulation at 3.5 wt.% NaCl, as shown clearly in Figures 6 and 7. The microstructure of the uncracked specimen at a condition of 70°C, 1.0 wt% NaCl under perlite as thermal insulation is shown in Figure 5 for comparison. It can be seen that the cracks are branched out into two at some points, and the total length of the cracks on the specimen is expected to be 800 μm with transgranular type. Transgranular crack is a crack where the crack splits (cleaves) through the grains that results from the repeated breaking of atomic bonds along specific planes.

No cracks were found on other U-bend specimens under calcium silicate and perlite thermal insulations, hence their microphotographs were not presented here. A sample image of uncracked specimen is shown in Figure 5. There were general corrosions that occurred on some of the specimens, some with pitting corrosion but only one condition that subjected to SCC.

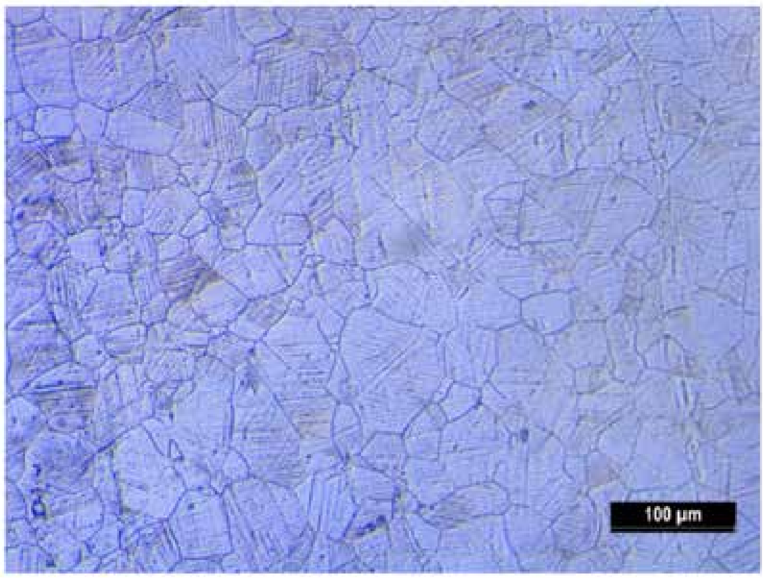

Figure 5 Microstructure of uncracked specimen (70°C, 1.0 wt% NaCl & Perlite) (200X)

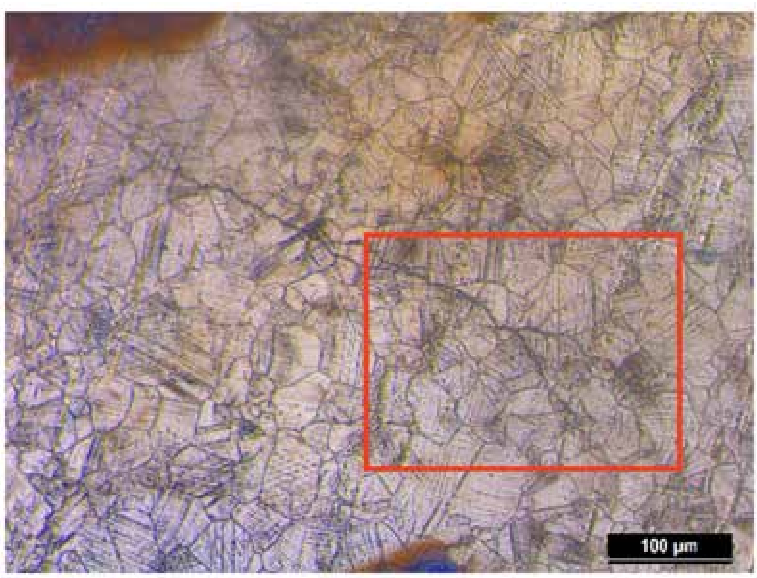

Figure 6 Microstructure showing transgranular crack in SUS 304L, 3.5 wt% NaCl, 90°C under Rockwool Insulation (200X)

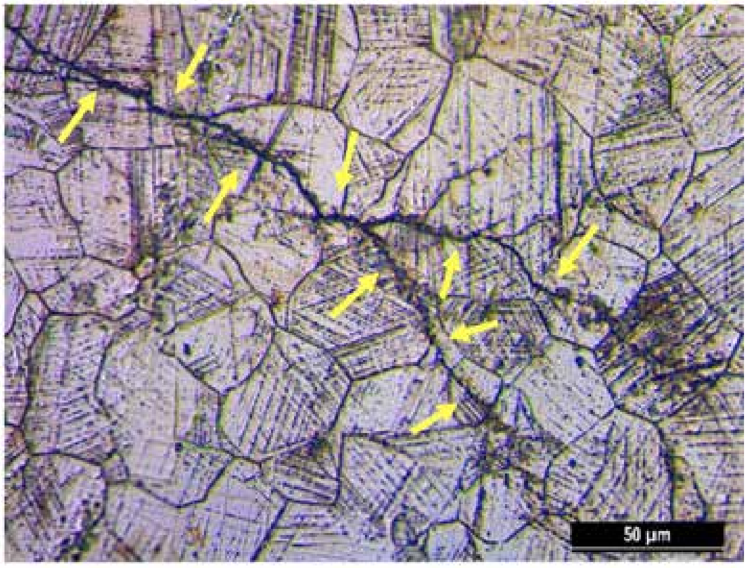

Figure 7 Microstructure showing transgranular crack in SUS 304L, 3.5 wt% NaCl, 90°C under Rockwool Insulation (500X)

CONCLUSION

The following specific conclusions can be drawn from this work:

a. Cracking was observed on the U-Bend specimen with rockwool as thermal insulation at 90°C and 3.5 wt% NaCl concentration only, while no crack was found on other insulated specimens.

b. Good water absorption capacity of the rockwool material has helped longer water retention and resulted in more severe salt deposit built-up on the hot metal surface when subjected to wet/dry cycle.

c. Specimens under perlite insulation showed the least salt deposit and corrosion features, which can be attributed to its relatively poor water absorption capability.

d. With 14 days of SCC tests done through the drip-test method, the results showed that only one out of 18 tests had occurred with SCC. If the duration of the tests prolonged following ASTM C692, which was 28 days, there might be more cases of SCC occurred especially the ones with rockwool as thermal insulation.

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