

How Sulfur Corrosion of LEDs Occurs and How to Avoid it

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1. Overview

This document provides further explanation pertaining to the Nichia's specifications (see Cautions, (1) Storage), for how corrosive gases (particularly, the sulfur content of these gases) can affect the characteristics and reliability of the LEDs and detailed cautions and suggestions on how to store/use the LEDs.



Before being exposed to a corrosive environment containing sulfur

After being exposed to a corrosive environment containing sulfur

1.1 How the Lead Frames Discolor/Corrode

Most Nichia LEDs have internal lead frames that are made of silver-plated copper alloy. Silver is excellent at both reflecting light and conducting electricity; however, if silver is exposed to a corrosive gas (e.g. hydrogen sulfide, etc.), then it can react with the sulfur content and may discolor. This is due to a chemical reaction between silver and sulfur (i.e. $2Ag + S \rightarrow Ag_2S$) and the resulting layer of silver sulfide on the surface. The level of discoloration can vary from yellow, then brown to black depending on the thickness of this layer.

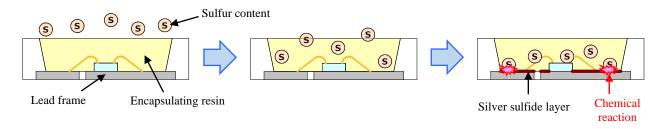


Figure 1. Schematic diagram of how sulfur causes a silver-plated lead frame to corrode (discolor)

1.2 Potential Issues Caused by Corroded Lead Frames

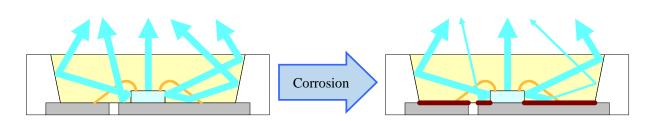
1) Reduction in the Luminous Intensity/Flux

If the lead frame discolors, the light that is reflected from the lead frame and extracted from the package is reduced; this causes the luminous intensity/flux to decrease (See Figure 2).

2) Color Shift

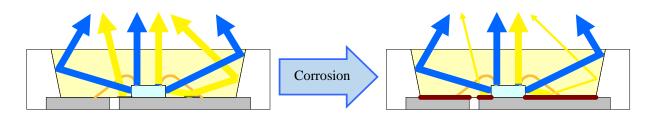
In order to produce white light, Nichia LEDs use a combination of phosphor with die that emits blue light: when the blue light from the die excites the phosphor it fluoresces yellow light; as both blue light and yellow light are combined together within the LED package, the light from the LED appears white, depending on the spectrum distribution. While most of the blue light exits the LED after turning yellow through the phosphor, some of the blue light instead strikes the lead frame surface first; if the lead frame is discolored, more of the yellow light may be absorbed by the lead frame; this can cause the spectrum distribution to change and the color of the light to shift to the blue region (See Figure 3).

Application Note



*When the lead frame becomes discolored, the amount of reflected light at the lead frame surface decreases.

Figure 2. How the Luminous Intensity/Flux Decreases



*When the lead frame becomes discolored, the amount of reflected light at the lead frame surface decreases and it changes the spectrum distribution of the LED.

Figure 3. How the Color Shift Occurs

3) Wire Breakage/Open Circuit

If corrosion extends under the surface of the lead frame, it can cause the bonds between the wires and the lead frame to become easily damaged; this may cause the LED not to illuminate.

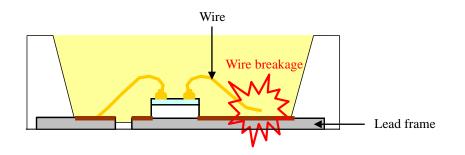


Figure 4. How Wire Breakage occurs due to Corrosion

1.3 LED Failure Modes As a Result of Sulfide Corrosion

Based on both Nichia's customer return history and test results, if the LEDs are used in indoor environments, Nichia considers sulfide corrosion of LEDs to be due to the following reasons:

1) In cases where corrosion occurs in general indoor environments

(i.e. the concentration of corrosive gas is low)

It can depend on the concentration level of sulfur-containing gases; however, both luminous flux reduction and color shift progress over time; after a long period of time has elapsed, LEDs can fail as an open circuit.

 In cases where either sulfur-containing materials/parts are used in the same product as the LEDs or many materials/parts containing corrosive substances are placed near the LEDs (i.e. the concentration of corrosive gases is high)

These LEDs are exposed to high concentrations of corrosive gases and it can increase the possibility that these LEDs will fail as an open circuit in a short period of time. For the details about the differences in both the chemical composition and concentration of corrosive gas, see Section 2.2.1.

1.4 Sources and Items that Release/Contain Corrosive Gas

Normally, a small amount of sulfur can be found in the general environment (i.e. the air). The following are examples of where or when corrosive gases can be generated and items containing sulfur.

- Sources that generate corrosive gases:

Diesel-powered cars, human waste treatment plants, livestock barns, kraft process, starch factories, manufacturing/combustion facilities at plants (e.g. thermal power stations, petroleum refineries, steel mills, workplace boilers, etc.), sulfur production, metalworking/smelting, hot springs, etc.

- Items containing sulfur:

Cardboards, rubber products (e.g. rubber bands), rubber-based adhesives, moisturizers, ointments, cosmetics, surface-active agents (e.g. detergents), etc.

2. Verification of the Corrosion Test

2.1 Corrosivity Categories of Indoor Atmospheres Defined in ISO 11844 and General Indoor Environments

In order to determine the test conditions for Nichia's corrosion test, Nichia performed an investigation on the relationship between the corrosivity categories of indoor atmospheres defined in the international standard specification ISO 11844 and field tests of general indoor environments near Nichia facilities.

2.1.1 Corrosivity Categories of Indoor Atmospheres Detailed in ISO 11844-1

ISO 11844-1 classifies the corrosion that develops in general indoor atmospheres (see 1.3 1) for examples) as shown in Table 1. The corrosivity categories for the ISO standard are based on measurements of the corrosive effect on standard specimens of four reference metals (i.e. carbon steel, zinc, copper and silver).

Since it is generally considered that silver corrosion can proceed lineally with time in actual indoor atmospheres, in order to determine the corrosivity category of an indoor atmosphere, Nichia has used silver plates as the standard samples for this evaluation.

Table 1. Description of typical environments related to the estimation of indoor corrosivity categories with the rate of mass increase (i.e. silver) according to ISO 11844-1¹⁾

Corrosivity category		Increase of the mass for the silver plates (mg/m ² per year)	Typical environments	Examples	
IC1	Very low	Heated spaces	1<25	Heated spaces with controlled stable relative humidity (<40%) without risk of condensation, low levels of pollutants, no specific pollutants.	Computer rooms, Museums with controlled environment
IC1	indoor		1\25	Unheated spaces with dehumidification, low levels of indoor pollution, no specific pollutants.	Military stores for equipment
IC2	Low	Heated spaces	fluctuation of relative humidity without risk of condensation, spaces		Museums, Control rooms
IC2	indoor Unheated spaces		25 <r<100< td=""><td>Unheated spaces with only temperature and humidity changes, with no risk of condensation, low levels of pollution without specific pollutants.</td><td>Storage rooms with low frequency of temperature changes</td></r<100<>	Unheated spaces with only temperature and humidity changes, with no risk of condensation, low levels of pollution without specific pollutants.	Storage rooms with low frequency of temperature changes
	Medium	humidity, medium levels of pollution, certain risks to pollutants.		Heated spaces with risk of fluctuation of temperature and humidity, medium levels of pollution, certain risks for specific pollutants.	Switchboards in the power industry
IC3	indoor	Unheated spaces	100 <r<450< td=""><td>Unheated spaces with elevated relative humidity (>50% - 70%) with periodic fluctuation of relative humidity, without risk of condensation, elevated levels of pollution, low risk of specific pollutants.</td><td>Churches in non-polluted areas, Outdoor telecommunication boxes in rural areas</td></r<450<>	Unheated spaces with elevated relative humidity (>50% - 70%) with periodic fluctuation of relative humidity, without risk of condensation, elevated levels of pollution, low risk of specific pollutants.	Churches in non-polluted areas, Outdoor telecommunication boxes in rural areas
		Heated spaces		Heated spaces with fluctuation of humidity and temperature, elevated levels of pollution including specific pollutants.	Electrical service rooms in industrial plants
IC4	High indoor	Unheated spaces	450 <r<1000< td=""><td>Unheated spaces with high relative humidity (>70%) with some risk of condensation, medium levels of pollution, possible effect of specific pollutants.</td><td>Churches in polluted areas, Outdoor boxes for telecommunication in polluted areas</td></r<1000<>	Unheated spaces with high relative humidity (>70%) with some risk of condensation, medium levels of pollution, possible effect of specific pollutants.	Churches in polluted areas, Outdoor boxes for telecommunication in polluted areas
IC5	Very high	Heated spaces	1000 <r<2500< td=""><td>Heated spaces with limited influence of relative humidity, higher levels of pollution including specific pollutants like H₂S.</td><td>Electrical service rooms, Cross-connection rooms in industries without efficient pollution control</td></r<2500<>	Heated spaces with limited influence of relative humidity, higher levels of pollution including specific pollutants like H ₂ S.	Electrical service rooms, Cross-connection rooms in industries without efficient pollution control
	indoor	Unheated spaces		Unheated spaces with high relative humidity and risk for condensation, medium and higher levels of pollution.	Storage rooms in basements in polluted areas

2.1.2 Field Investigation Results - Categorization of the Surrounding Environments and Nichia Facilities

To determine the corrosivity categories of both surrounding environments of Nichia facilities and general outdoor atmospheres, Nichia conducted an investigation by using silver plates to measure how much the mass increased as a result of corrosion in those atmospheres. For the results of this measurement refer to Figure 5 below.

The results of the investigation are as follows:

Nichia facilities

- Production rooms/reliability laboratory fall into upper IC1 to lower IC2
- Workshop/service entrance fall into middle IC2 to lower IC3
- Bicycle parking area/the inside of instrument shelter fall into lower IC3 to middle IC4

General indoor atmospheres

- Private residences (e.g. living room and kitchen) fall into lower IC2 to lower IC3
- Commercial buildings (e.g. automobile repair shop/garage and clothing shop) fall into lower IC2 to lower IC3

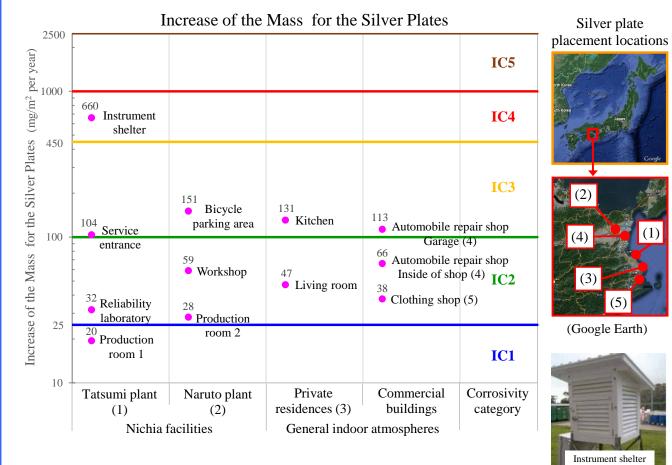


Figure 5. Field Investigation Results

2.2 Development and Evaluation of Accelerated Corrosion Test Conditions

LED corrosion tests that are performed under general environments can take significant time to complete; in order to shorten the test duration, accelerated testing is useful.

In order to determine the accelerated test conditions, Nichia has performed corrosion tests by using different test conditions (e.g. gas composition, temperature, etc.). The test results and the evaluation of the test conditions used are shown in Pages 6 to 11.

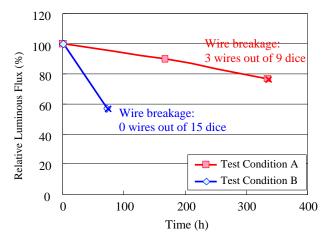
2.2.1 Difference in the effect of corrosion due to the gas composition/concentration

Silver can corrode differently depending on the gas composition/concentration. In the case of LEDs, if they are exposed to corrosive gas containing sulfur, it can cause the LED characteristics to change and/or the wires to break. Since the effect on the LED can differ depending on the gas composition/concentration and/or the conditions/environment where the corrosion occurs, the degree of the change in the characteristics and the occurrence rate of the wire breakage are not directly proportional. In other words, if the luminous flux decreases to a certain level as a result of exposure to corrosive gas, it will not necessarily cause the wires to break.

Application Note

Nichia's corrosive test results (see Figure 6 below) shows that the group of test LEDs with a greater reduction in relative luminous flux (shown in blue) had no LEDs with broken wires (0/15 dice), while the other group with a smaller reduction in relative luminous flux (shown in red) had LEDs with broken wires (3/9 dice).

Based on these results, when determining the conditions for the accelerated tests, it is important to consider the conditions/environment where the LEDs are actually used and ensure that appropriate conditions/environments are used for the tests.



Test Condition A Test LEDs are operated within a sealed flask (360ml) with rubber bands (4g).
Operating conditions: T_A=65°C, I_F=100mA

-Test Condition B

Test LEDs are stored (unpowered) within a sealed flask (360ml) with a rubber gasket/seal (0.5g). Storage conditions: $T_A=100^{\circ}C$

Figure 6. Reduction in Relative Luminous Flux and Occurrence of Wire Breakage in Corrosive Atmospheres

2.2.2 Verification of the Corrosion Test Conditions

When performing the corrosion tests, Nichia used Method 3 as defined in IEC 60068-2-60 Environmental testing – Part 2: Tests – Test Ke: Flowing mixed gas corrosion test. Nichia considers it appropriate to use Method 3 as the accelerated test method/conditions for the following reasons:

- Method 3 uses a mixed gas and will be able to provide a better test environment to obtain fine silver sulfide corrosion crystals than using a single component gas; these crystals are as fine as crystals that could occur in natural atmospheres.
- Compared to the other three methods, Method 3 uses a higher concentration of hydrogen sulfide and that can cause the silver corrosion to occur faster.

For the details of the different IEC 60068-2-60 Method test conditions, refer to Table 2.

Method	Gas Composition	Temperature and Humidity Level
1	$SO_2 0.5ppm + H_2S 0.1ppm$	25°C 75%RH
2	$H_2S \ 0.01ppm + NO_2 \ 0.2ppm + Cl_2 \ 0.01ppm$	30°C 70%RH
3	$H_2S 0.1ppm + NO_2 0.2ppm + Cl_2 0.02ppm$	30°C 75%RH
4	$H_2S \ 0.01ppm + NO_2 \ 0.2ppm + Cl_2 \ 0.01ppm + SO_2 \ 0.2ppm$	25°C 75%RH

Table 2. IEC 60068-2-60's Test Methods and Conditions²⁾

2.2.3 Optimizing the Accelerated Test Conditions

In order to further optimize Method 3 and to develop more severe (accelerated) test conditions, Nichia reviewed the composition and concentration of the gas used in Method 3 and performed a corrosion test by: using the same mixed gas as used in Method 3 at the same ratio of the gas ingredients (i.e. $H_2S:NO_2=1:2$) as well as an increased H_2S gas concentration of up to 20 times that of Method 3. The chlorine (Cl₂) component in the original Method 3 composition was removed from this improved Method. For tests that use Method 3, the resulting products of corrosion will mostly consist of silver sulfide; very little silver chloride will be produced. With the improved Method, the form of corrosion should remain the same but the speed of corrosion should be further accelerated.³⁾

Based on this consideration, Nichia conducted an LED corrosion test by using the tentative accelerated test conditions (see Table 3) to investigate the extent of the corrosion and the additional weight created by the corrosion formed over the test silver plates.

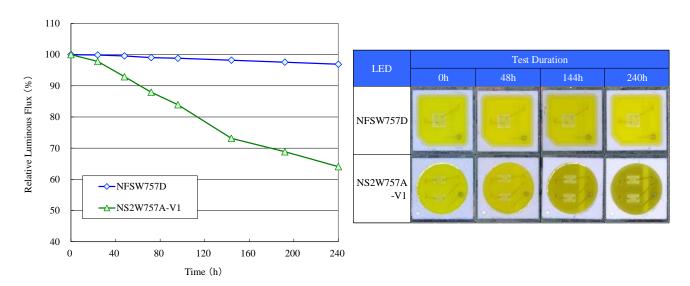
Table3. Improved Corrosion Test Method

Gas Composition	Temperature and Humidity Level	Test Duration	
$H_2S 2ppm + NO_2 4ppm$	40°C 75%RH	240h	

2.2.4 Accelerated Corrosion Test Results using the Improved Test Method

Nichia used two LED models (i.e. NFSW757D and NS2W757A-V1) for the improved accelerated corrosion test (see 2.2.3). During this test the LEDs were not operated.

As the test results show (see Figure 7), the luminous flux of the NS2W757A-V1 LEDs decreased more than that of the NFSW757D LEDs. Note that the NFSW757D is an improved model of the NFSW757A-V1 with better gas barrier properties.





Application Note

While performing the improved accelerated corrosion test on the test LEDs by using silver plates, Nichia attempted to determine how much the mass of these silver plates increased during this test. The silver plates were exchanged and measured every 48 hours and the cumulative sum of the increase in the mass of the silver plates was calculated to obtain the gradient of the line. From this investigation, Nichia confirmed that the mass of the silver plates increased linearly and a corrosive atmosphere was kept constant within the test chamber. Figure 8 below shows how the mass of the silver plates increased during this investigation.

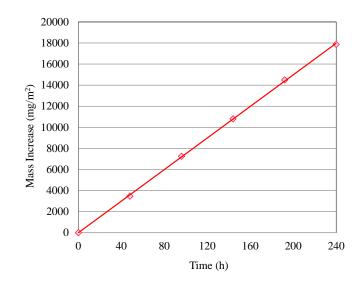


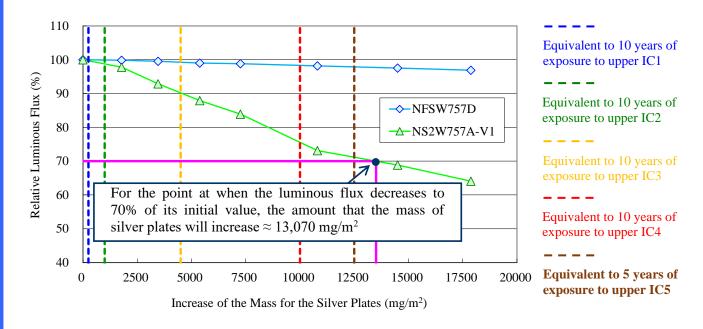
Figure 8. Increase in the Mass of the Silver Plates during the Corrosion Test

2.2.5 Estimation of the time to 70% lumen maintenance by using the corrosion test results

Nichia used the mixed gas corrosion test results (see 2.2.4), the corrosivity categories of indoor atmospheres (ISO 11844 Part 1) and the amount that the mass of the silver plates increased in the field tests (see 2.1.2) to estimate the time to 70% lumen maintenance.

- NFSW757D: The LEDs are less likely to corrode in atmospheres of each of the five ISO 11844 categories (i.e. IC1 to IC5); the resulting luminous flux reduction due to corrosion will not occur.
- NS2W757A-V1: The LEDs are less likely to corrode in atmospheres up to upper IC2 (potentially, lower IC3); the resulting luminous flux reduction due to corrosion will rarely occur in these atmospheres.

Figure 9 and Tables 4 to 5 show the estimated times to 70% lumen maintenance of both LED models. These estimates do not include deterioration from use or during storage. Ensure that this data is only used for reference purposes.



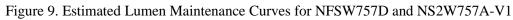


Table 4. Estimated Time to 70% Lumen Maintenance for Ead	ach ISO 11844 Corrosivity Category
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	ISO 11844	Calculated Lumen Maintenance by Using Accelerated Corrosion Test Results (% in 10 years)		Estimated Time to 70% Lumen		
Corrosivity	Increase of the Mass for the Silver Plates			Maintenance (Years)		
Categories	(mg/m ² per 10 years)	NFSW757D	NS2W757A-V1	NFSW757D	NS2W757A-V1	
Upper IC1	250	100	99.7	>100	>100	
Upper IC2	1000	99.9	98.8	>100	>100	
Upper IC3	4500	99.3	90.2	>100	29	
Upper IC4	10000	98.4	76.4	>100	13	
Upper IC5	12500 (5 years)	97.9 (5 years)	71.1 (5 years)	>100	5	

* These estimates do not include deterioration from use or during storage. Ensure that this data is only used for reference purposes.

Application Note

Locations of the Silver Plates			Increase of the Mass for the Silver Plates (mg/m ² per year)	ISO 11844 Corrosivity Categories of Indoor Atmospheres	Corrosivityby Using Accelerated Corrosionategories of IndoorTest Results (% in 10 years)		Estimated Time to 70% Lumen Maintenance (Years) NFSW757D NS2W757A-V1	
	Tatsumi Plant	Production Room 1	20	Around Upper IC1	100	99.7	Over 100	Over 100
		Reliability Laboratory	32	Around Lower IC2	100	99.6	Over 100	Over 100
Atmospheres		Service Entrance	104	Around Lower IC3	99.9	98.8	Over 100	Over 100
within Nichia Facilities		Instrument Shelter	660	Middle of IC4	98.9	84.8	Over 100	20
T definitions	Naruto Plant	Production Room 2	28	Around Lower IC2	100	99.6	Over 100	Over 100
		Workshop	59	Middle of IC2	99.9	99.3	Over 100	Over 100
		Bicycle Parking Area	151	Around Lower IC3	99.8	98.0	Over 100	Over 100
	Private Residences	Living Room	47	Around Lower IC2	99.9	99.4	Over 100	Over 100
		Kitchen	131	Around Lower IC3	99.8	98.4	Over 100	Over 100
General indoor Atmospheres	Commercial Buildings	Automobile Repair Shop (Garage)	113	Around Lower IC3	99.9	98.6	Over 100	Over 100
I to the		Automobile Repair Shop (Inside of Shop)	66	Middle of IC2	99.8	99.2	Over 100	Over 100
		Clothing Shop	38	Around Lower IC2	100	99.6	Over 100	Over 100

* These estimates do not include deterioration from use or during storage. Ensure that this data is only used for reference purposes.

Based on the results of the field tests and estimation, the following conclusions can be summarized: -NS2W757A-V1:

Even if the LEDs are used for 10 years in a semi-outdoor location (i.e. the instrument shelter example in the field tests), the expected lumen maintenance is approximately 85%; there should be no major concern that corrosion will occur as a result of being exposed to corrosive gas in the other locations of the field tests.

-NFSW757D:

The reduction in the luminous flux of the LEDs is expected to be small in any field test location; there should be no major concern that corrosion will occur as a result of being exposed to corrosive gas in the other locations of the field tests.

There will be little possibility of corrosion regarding the Nichia 757 series LEDs as far as when LEDs are used in general indoor atmospheres similar to the locations of the field tests. However, if the LEDs are used in corrosive atmospheres (e.g. used with parts/materials containing sulfur, etc.) and the concentration of corrosive gas is high - as shown in Nichia's data that as the mass of silver plate increases, the luminous flux decrease - it may cause the luminous flux to decrease.

Application Note

2.2.6 Corrosion Test Results under High Temperature Conditions

Nichia also has used a different ambient temperature (i.e. $T_A=60^{\circ}C$) to perform further evaluation of the test conditions for the corrosion test. When using silver plates for this high temperature corrosion test, the corrosion rates were not much different compared to when tested at $T_A=40^{\circ}C$; on the other hand, when using LEDs, the corrosion rates of the test LEDs were different between these ambient temperatures and these LEDs corroded faster when they were tested at $T_A=60^{\circ}C$. This can be explained by the deterioration of the encapsulating resin used in these LEDs; the gas barrier property of this encapsulating resin can deteriorate faster when the ambient temperature is high. During the tests, at a certain point of time the luminous flux of the test LEDs decreased followed by a slower decline. When the LEDs were tested at $T_A=60^{\circ}C$, the luminous flux reached this point faster than when tested at $T_A=40^{\circ}C$. This explains that the test conditions that were used for the high temperature corrosion test was too severe and is not suitable to evaluate the LEDs. For the details of the test conditions and results, see Table 6 and Figures 10, 11 and Table 7

respectively.

Table 6. High Temperature Corrosion Test Conditions for NS2W757A-V1

	Temperature and Humidity Level		Test Duration	
Test Conditions in Table 3	40°C 75%RH	U.S. Januari, NO. Array	240h	
High Temperature Test Conditions	60°C 75%RH	H_2S 2ppm + NO ₂ 4ppm		

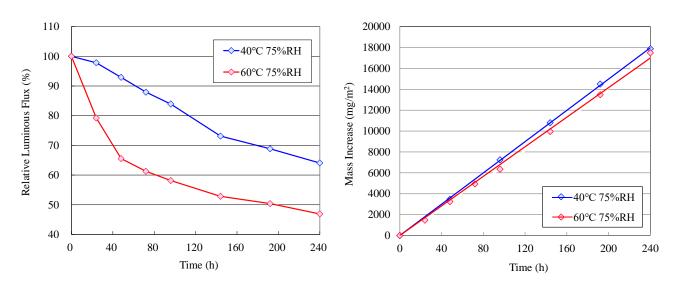


Figure 10. Lumen Maintenance for NS2W757A-V1

Figure 11. Increase in the Mass of the Silver Plates

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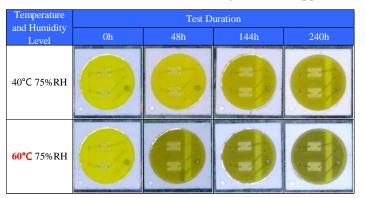


Table 7. Corrosion Test Results - Change in LED Appearance

2.2.7 Accelerated Corrosion Tests Overview and Planned Further Improvements

Nichia's accelerated corrosion test results show that in the case of the corrosion of LEDs: the luminous flux does not decrease linearly with time; however in the case of the corrosion of the silver plates, the mass of the silver plates does increase linearly with time. A possible cause of this difference is the gas barrier function of the encapsulating resin used in LEDs.

The test conditions provided in this document are tentative. Nichia considers that its accelerated corrosion test should be effective and fit for the intended purpose (i.e. using Nichia LEDs in general indoor atmospheres); after reviewing all the data/results detailed in this document, Nichia believes that the accelerated test conditions used have achieved a satisfactory level of effectiveness/fitness for purpose. However, Nichia plans to make improvements to these test conditions and perform further accelerated corrosion tests to provide more accurate and useful data/results to Nichia customers.

3. LED Precautions for Corrosive Gas

3.1 Storage Considerations

Nichia LEDs have alloy-plated parts containing silver both inside and outside of the package body. Exposure to a corrosive environment may tarnish the plating, which may result in a reduction in the solderability and may also cause an adverse effect on the optical characteristics. When storing the LEDs, a hermetically-sealed container should be used.

It is possible to protect the lead frames from corrosion by preventing them from being exposed to corrosive gas. In order to store the LEDs properly after opening the aluminum bag, the following examples are recommended:

- Storing the LEDs in a desiccator filled with nitrogen.
- Placing the LEDs into a hermetically-sealed container (e.g. the original aluminum bag and resealing).
- * Do not store the LEDs in the same desiccator/container with parts/materials that can produce corrosive gas.

3.2 Design Considerations

When using Nichia LEDs for the chosen application, ensure that the following points are considered.

- Materials/parts (e.g. gaskets/seals, glue, adhesive, etc.) containing sulfur may affect the plated surfaces; do not use these materials/parts for the chosen application. If gaskets/seals are used, silicone rubber gaskets/seals should be used with great care to ensure that contact failure does not occur due to low-molecular weight siloxane.
- In addition to outgassing from some items (e.g. cardboard boxes, rubber items, etc.), a trace amount of corrosive gas may be found in the ambient air. Halogen-based compounds can also have an adverse effect on the plated surface and they are found in some resin or resin-based materials/parts.
- Even if the LEDs have been soldered to a PCB and/or the PCB has been installed in an end product, the plated surfaces may be adversely affected by gas that is released from materials/parts near the LEDs and/or gas that enters from the outside environment.

4. Summary and References

LED corrosion occurs due to multiple factors that are intricately linked, rather than a single independent factor. Examples of these factors are the concentration/composition of gas, temperature/humidity, etc. When designing a product that will use Nichia LEDs, consider the ambient conditions of the environment where the LEDs will be used (e.g. temperature, humidity, etc.) and the possibility that corrosive gas exists in this environment; if any, the composition/concentration of the gas should be identified. Materials/parts that will be used with or near the LEDs should also be checked to see if there is any possibility of these materials/parts generating corrosive gas. Additionally, if corrosive tests are performed on the LEDs, ensure that these factors are considered for the intended environment and that appropriate test conditions are determined.

The evaluation results/data in this document are provided for illustrative purposes; ensure that necessary evaluations/tests are carried out for the chosen application.

References

- 1) ISO 11844 Corrosion of metals and alloys Classification of low corrosivity of indoor atmospheres
 - Part 1: Determination and estimation of indoor corrosivity
 - Part 2: Determination of corrosion attack in indoor atmospheres
 - Part 3: Measurement of environmental parameters affecting indoor corrosivity
- 2) IEC60068-2-60: 2015 Environmental testing Part 2-60: Tests Test Ke: Flowing mixed gas corrosion test
- 3) Japan Society of Corrosion Engineering. Questions and Answer regarding the Corrosion and Corrosion Protection of Electrical Equipment Parts. Tokyo: Maruzen, 2006.

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SP-QR-C2-211213-1 Jun. 26, 2023