Sense the power of light

Need for Speed?

A comparative study of H_2S-corrosion tests

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Motivation and Goal

Motivation

Find a corrosion test for Automotive, that is closer to real life conditions.

	Test	Details	Used Today/2025	Proposal AECQ102 2025
Interior Automotive	*FMG*	*H2S 10ppb, SO2 200ppb, NO2 200ppb, Cl2 10ppb IEC 60068-2-60 method 4*	*y*	
	H2S 10ppm	*H2S 10ppm 75%r.h. T=25°C 500h, IEC 60068-2-43*	*y*	
	NO2&H2S	H2S 2ppm NO2 4ppm 75% r.h. 40°C, IEC 60747-5-13		y
Exterior Automotive	*FMG*	*H2S 10ppb, SO2 200ppb, NO2 200ppb, Cl2 10ppb IEC 60068-2-60 method 4*	*y*	
	H2S 15ppm	H2S 15ppm 90%r.h. T=40°C 336h, IEC 60068-2-43	y	y

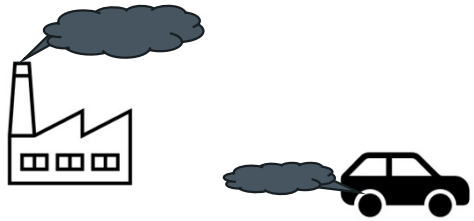
Goal

Find out whether the proposed change of conditions brings an advantage for testing.

Criteria: Suitability of Test (closer to real application, handling, repeatability), Speed, others.

Why we need tests with H2S?

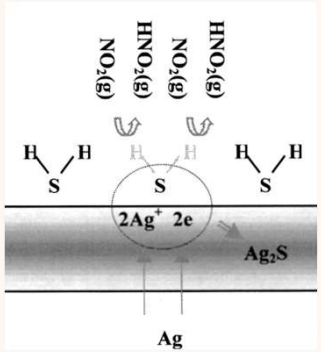
H2S is seen in our environment



What does H2S do?

Simulation of failure mechanisms:
Sulfidation of Ag→ reflector tarnishing (colorshift) → brightness degradation
In real world: Environments with fuel vapour, S-containing rubbers, industrial atmospheres

Reaction NO2 & H2S [1]:

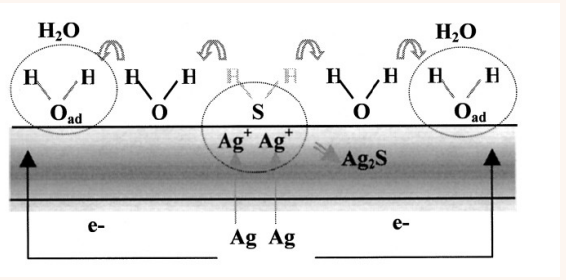


$$H_2S + 2NO_2 \rightarrow S + 2HNO_2$$
$$2Ag + S \rightarrow \mathbf{Ag2S}$$

Reaction product: Ag2S

- NO2 is an oxidation agent→ HNO2/3 acid → highly accelerated test
- Lower dependency of corrosion rate by humidity
- Field representative/realistic corrosion crystal morphologhy.

Reaction H2S [1]:



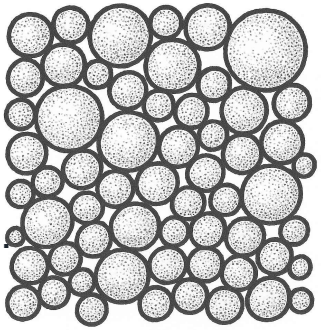
$$4Ag(s) + 2H_2S(aq) + O_2(g) \rightarrow 4Ag^+ + 2S^{2-} + 4H^+ + O_2$$
$$\rightarrow \mathbf{2Ag2S}\,(s) + 2H_2O(l)$$

[1] Source: H. Kim, "Corrosion process of silver in environments containing 0.1 ppm H_2S and 1.2 ppm NO_2," *Materials and Corrosion*, vol. 54, 2003

H2S & NO2 React with Silver as in Real-World Conditions

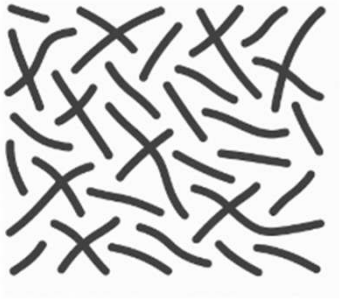
From AECQ102 expert discussion:

For H2S & NO2:



- Granular crystals
- Comparable to natural environmental conditions
- Stable sulfidation

H2S only: - Filamentous crystals.



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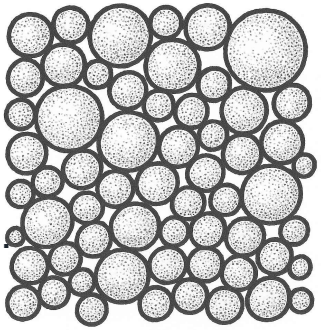

- Granular crystals
- Comparable to natural environmental conditions
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Literature check [2] confirms statement

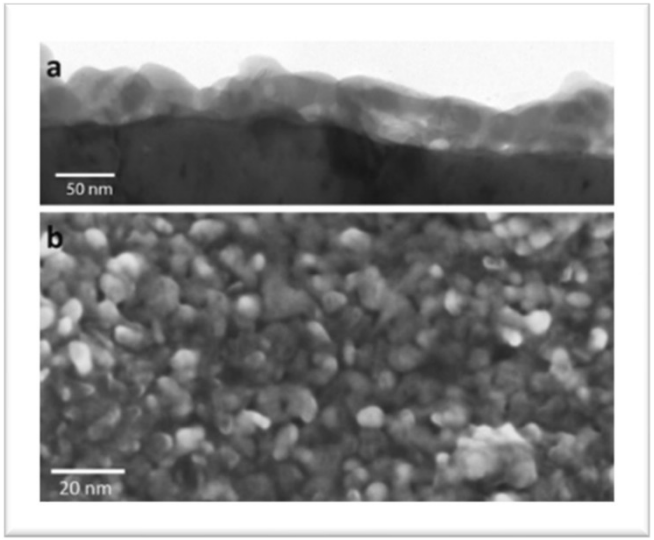
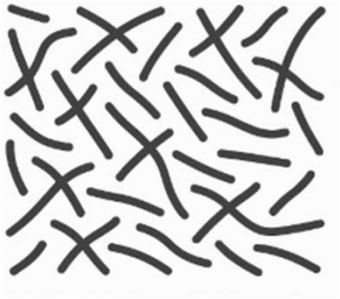


Figure 3. Corrosion layer formed on bulk silver imaged with (**a**) transmission electron microscopy (TEM) in cross-section and (**b**) scanning electron microscopy of the surface. This corrosion layer was one that had formed after many years of exposure to ambient air and can be seen to be particulate in nature.

H2S only: - Filamentous crystals.



Ag2S crystal structure in real world environment is granular, as can be seen from the reaction of H2S&NO2

Source [2]: Keast, V. J. (2022). *Atmospheric corrosion of silver and silver nanoparticles. Corrosion and Materials Degradation, https://www.mdpi.com/2624-5558/3/2/13*

Gas test comparison from literature

Acceleration for the new H2S&NO2 test

Gas Mixture	Environment Type	Synergistic Factor
Hydrogen sulfide (H2S)	Industrial, simulated, real-world	Baseline for silver corrosion
Hydrogen sulfide (H2S) + chlorine gas (Cl2)	Industrial, simulated	Synergistic, corrosion rate up to 4.6 times higher than additive effects
Hydrogen sulfide (H2S) + sulfur dioxide (SO2)	Industrial, coastal, simulated	Synergistic, SO2 accelerates H2S-induced corrosion
Hydrogen sulfide (H2S) + nitrogen dioxide (NO2)	Industrial, simulated	Synergistic, but less than Cl2 or SO2

Acceleration less than 4,6

Acceleration Factor new test

(very rough estimations!!!):

1. **Temperature**

$AF1=e^{Ea/R(1/T1-1/T2)}$ → with T1=25°C and T2=40°C

AccelerationFactor=3,7 (0,7eV) AECQ102 [6]

Literature values lead to a large range [4].

2. **Gas Mix:** Adding NO2 up to factor 5 [5]

3. **Gas concentration:**

Reduction from 10ppm H2S to 2ppm H2S

Literature supports: adding NO2 & increased Temperature lead to test acceleration

[3] Source: Elicit summary based on Vargas et al., 2009; Chen et al., 2011; Xiao et al., 2017; Huo & Lee, 2015; Reid et al., 2007; Fu et al., 2014; Liao et al., 2021; Lee et al., 2014; Zhang et al., 2020; Furusawa et al., 1986; Lee & Lee, 2021; Hendriks, 1986; Mazurkiewicz, 2006; Valdez et al., 2003; Kolbinger et al., 2018; Wang et al., 2023; Fu & Lee, 2017; Zhang et al., 2010; Zhuang et al., 2023)

4].Source: Wang, Z., Gu, T., Kadohira, T., Tada, T., & Watanabe, S. (2008). Migration of Ag in low-temperature Ag_2S from first principles. *The Journal of Chemical Physics*

[5] Source: Muller C., (1990). Multiple Contaminent Gas Effects on Electronic Equipment Corrosion; Corrosion 90

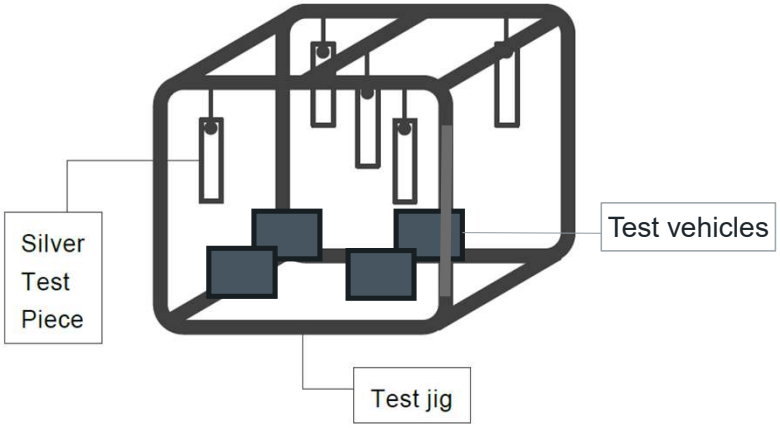
[6] Source: Automotive Electronics Council. (2020). Failure mechanism based stress test qualification for optoelectronic semiconductors in automotive applications (AEC-Q102 Rev A). http://www.aecouncil.com/Documents/AEC-Q102_Rev_A.pdf

Method

Test setup

4 test vehicles with different Ag-leadframe platings and sputtered silver plates where tested with:

1. existing 10ppm H2S test, 500h, T=25°C
2. new 2ppm H2S and 4ppm NO2 , 168h, 336h, 500h, T=40°C



Test setup

IEC 60747_5_13: Hydrogen sulphide corrosion test for LED packages

What do we learn from the NO2&H2S specification

8 Details to be specified

The relevant specification shall specify the following details:

a) measurements and checks to be made prior to the test;

b) electrical loading or operational conditions of the specimens, if applicable;

c) duration of the test;

d) measurements, correction, checks and visual inspection to be made at the end

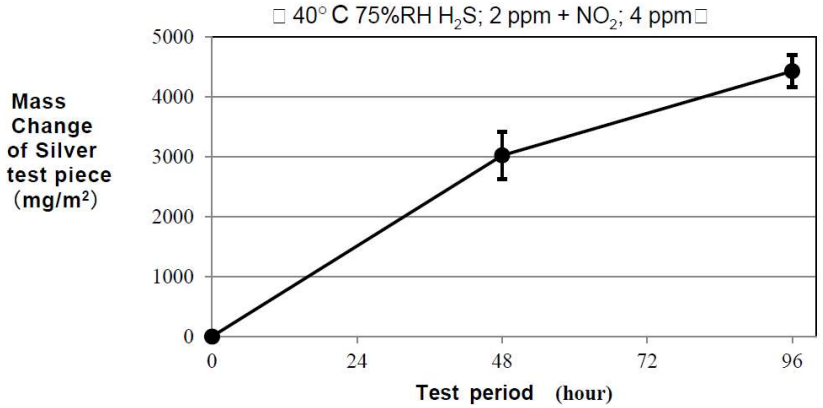
A.2 Correspondence relation between the result of this corrosion test and the corrosion in the field environment (Case example)

Hydrogen sulphide corrosion test for 96 hours gets silver test pieces mass increase by about 4 500 mg/m^2 (See The test results by test apparatus in Figure A.1). Method of confirmation of the mass increase of silver test pieces is detailed in Annex B.

Table A.2—The upper limit of Mass increase of silver test pieces in the indoor environment of Corrosivity category in ISO 11844-1:2006 for ten years' usage

Class	The upper limit of mass increase of silver test pieces mg/m^2·year	The upper limit of mass increase of silver test pieces mg/m^2·10year
IC 1	25	250
IC 2	100	1 000
IC 3	450	4 500
IC 4	1 000	10 000
IC 6	2 500	25 000

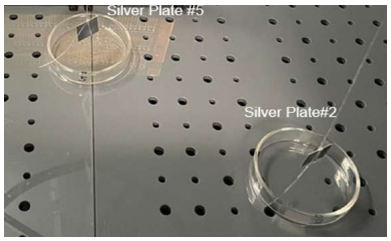
Figure A. 1—Mass increase of silver test piece Hydrogen sulphide corrosion test

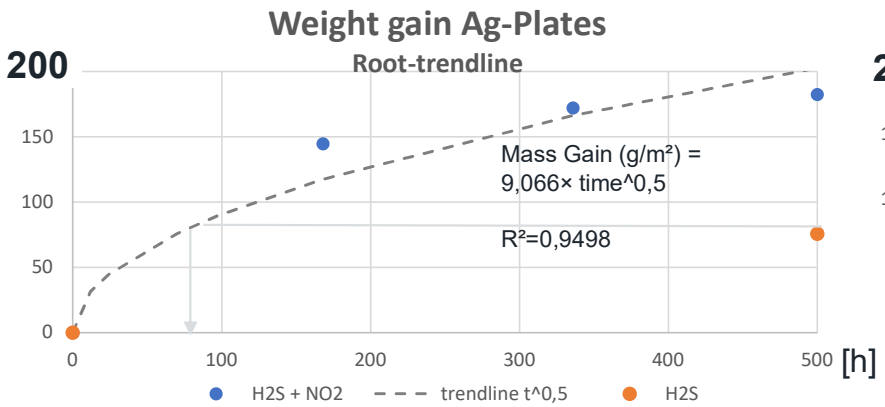


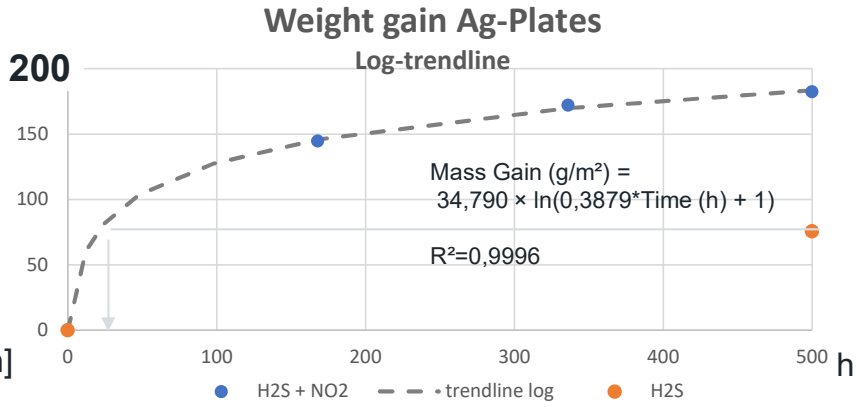
[6] Source: IEC, *Semiconductor devices – Part 5-13: Optoelectronic devices – Hydrogen sulphide corrosion test for LED packages*, IEC 60747-5-13, Ed. 1.0, 2021. https://webstore.iec.ch/en/publication/62836

Test results: Test duration of new test

Mass gain of silver plates







Ca. 70h Test duration

Square-root behavior :
Diffusion controlled rate, depends on ion migration through a growing corrosion layer [7].

Ca. 16h Test duration

Log-behaviour [8]:
-due to protective film buildup
-activation-controlled processes, reactions

Test duration has to be reduced for new test!

This evaluation indicates a test duration of 16h to 70h. (16+70)h/2=43h
Exact duration has to determined in second round.

[7] Z. Grzesik, *Methods of studying metal oxidation kinetics* [PowerPoint], AGH Univ. of Science and Technology. https://home.agh.edu.pl/~grzesik/FHTC/2_Methods%20of%20studying%20metal%20oxidation%20kinetics.pdf

[8] Olasunkanmi, L. O. (2021). *Corrosion: Favoured, yet undesirable – Its kinetics and thermodynamics*. In F. Zafar, A. Ghosal, & E. Sharmin (Eds.), *Corrosion - Fundamentals and Protection Mechanisms*. IntechOpen. https://www.intechopen.com/chapters/77297

Test results LEDs overview: with tightened critera

Higher test serverity for H2S&NO2 test

Tighter than AECQ102:
Used robustness criteria

AECQ102 limits	10%
Criteria	**5%**

Test	Test Length [h]	Device/Leadframe	Vf fails
R1 H2S 10ppm	500	A	0%
R1 H2S 2ppm NO2 4ppm	500	A	0%
R1 H2S 10ppm	500	B	0%
R1 H2S 2ppm NO2 4ppm	500	B	0%
R1 H2S 10ppm	500	C	0%
R1 H2S 2ppm NO2 4ppm	500	C	0%
R1 H2S 10ppm	500	D	0%
R1 H2S 2ppm NO2 4ppm	500	D	0%

365 Devices

No Vf fails.

Test results LEDs overview: with tightened critera

Higher test serverity for H2S&NO2 test

Tighter than AECQ102:
Used robustness criteria

AECQ102 limits	10%	0,02	0,02	30%	
Criteria	**5%**	**0,01**	**0,01**	**20%**	

Test	Test Length [h]	Device/Leadframe	Vf fails	cx fails	cy fails	D lv fail	devices fails
R1 H2S 10ppm	500	A	0%	0%	0%	0%	0%
R1 H2S 2ppm NO2 4ppm	500	A	0%	0%	0%	0%	0%
R1 H2S 10ppm	500	B	0%	0%	0%	0%	0%
R1 H2S 2ppm NO2 4ppm	500	B	0%	100%	100%	100%	100%
R1 H2S 10ppm	500	C	0%	0%	16%	0%	16%
R1 H2S 2ppm NO2 4ppm	500	C	0%	100%	100%	78%	100%
R1 H2S 10ppm	500	D	0%	4%	13%	4%	13%
R1 H2S 2ppm NO2 4ppm	500	D	0%	38%	82%	67%	82%

Overall, the new H2S&NO2 test is highly accelerated.
Test serverity depends on LED type/plating.

Summary

Test criterea for the new H2S&NO2 test compared to H2S 10ppm:

- ✓ Closer to real application and test results comparable
- ✓ Handling comparable
- ✓ 2 instead of 4 tests reduces logistics
- ✓ Speed: ca. factor 10 times faster (exact duration will be evaluated in 2nd round)

Well suitable for qualification tests

? Repeatability : has to be confirmed in 2. round

Potential

The new H2S&NO2 Test could also substitute the standard H2S shown in the current version of the AECQ102.

Test duration would have to be adjusted.

Thank you for your attention!