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# Novel Methods for Corrosivity Assessment of Automotive and Industrial Lubricants

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Prova Interlaboratorio Prodotti Lubrificanti  
Riunione Plenaria: Electrochemistry 14 June 2022

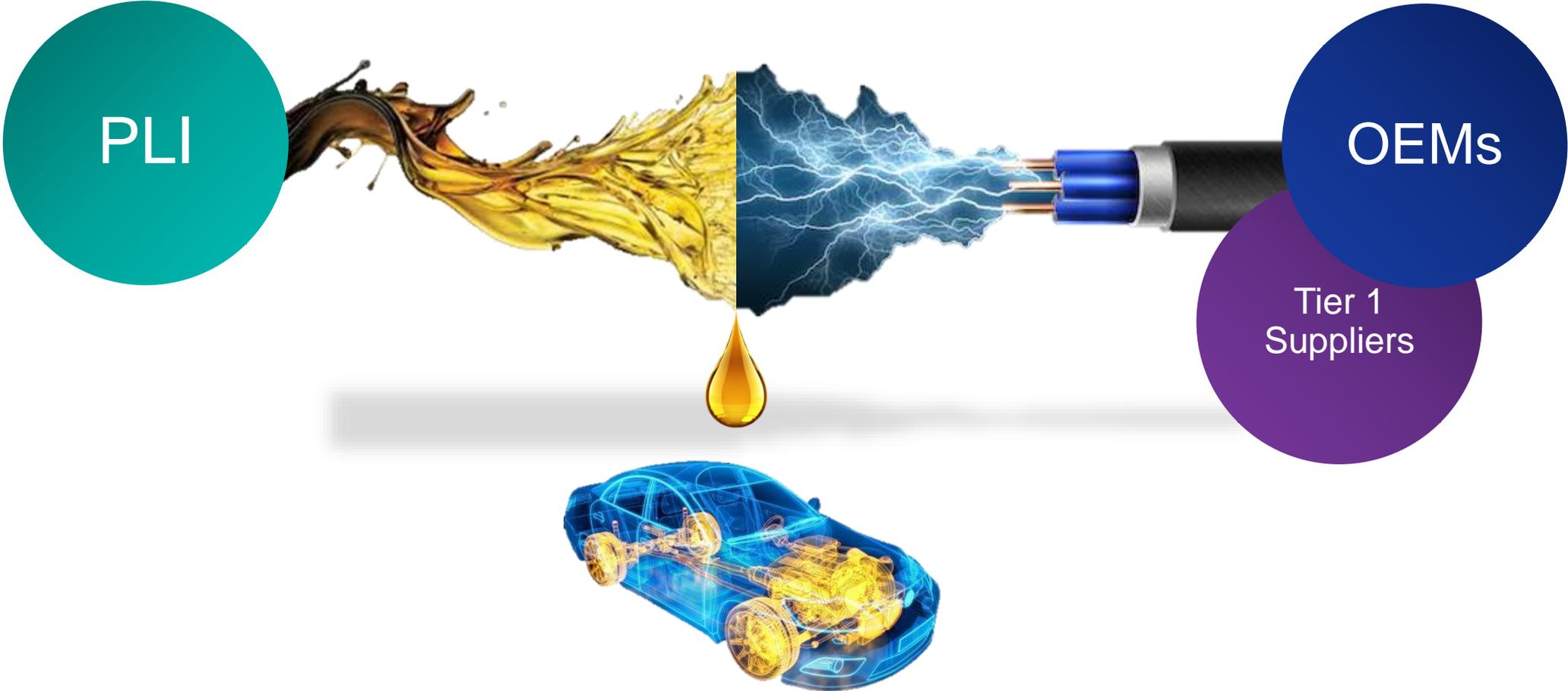
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New testing methodologies are available at PLI to design, together with OEMs, tailor made E-fluids for E-application.

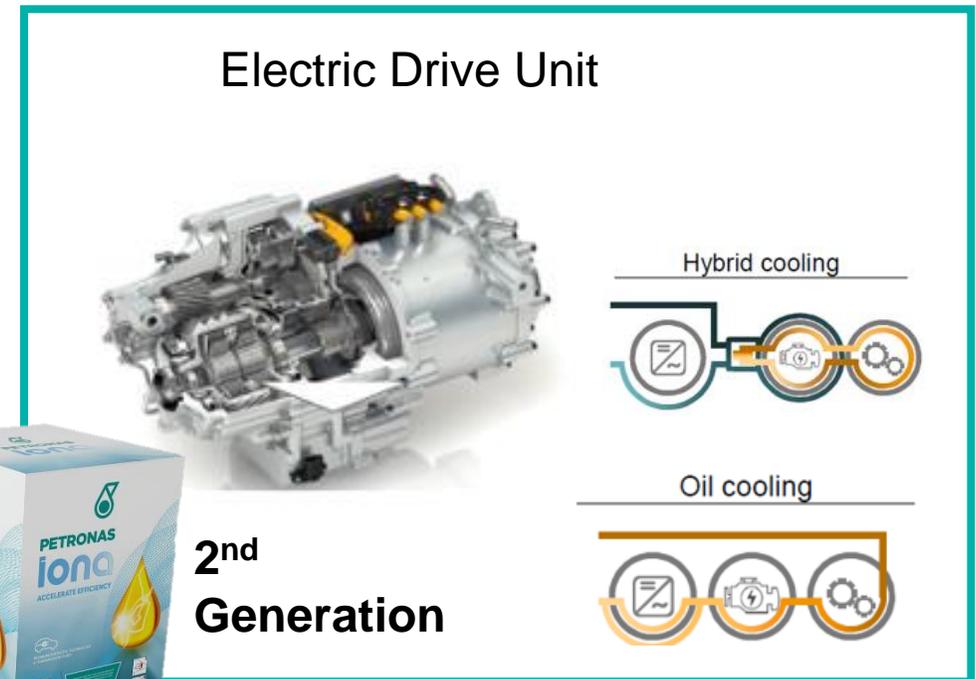
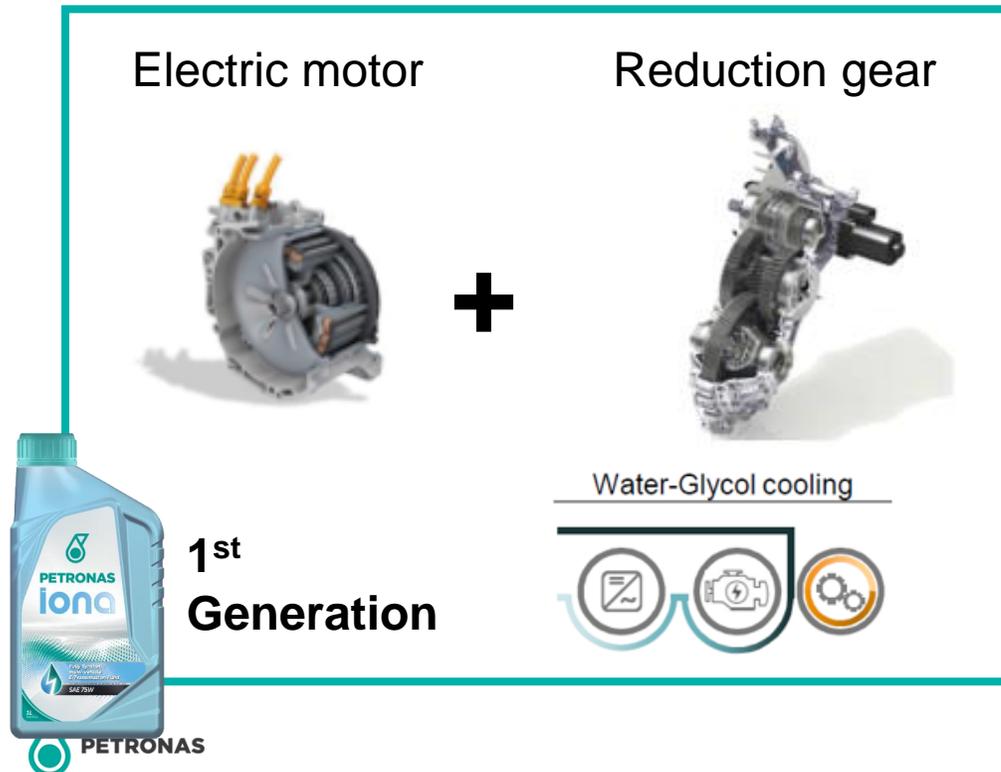


# New Generation of Electric Drive Units (EDUs)

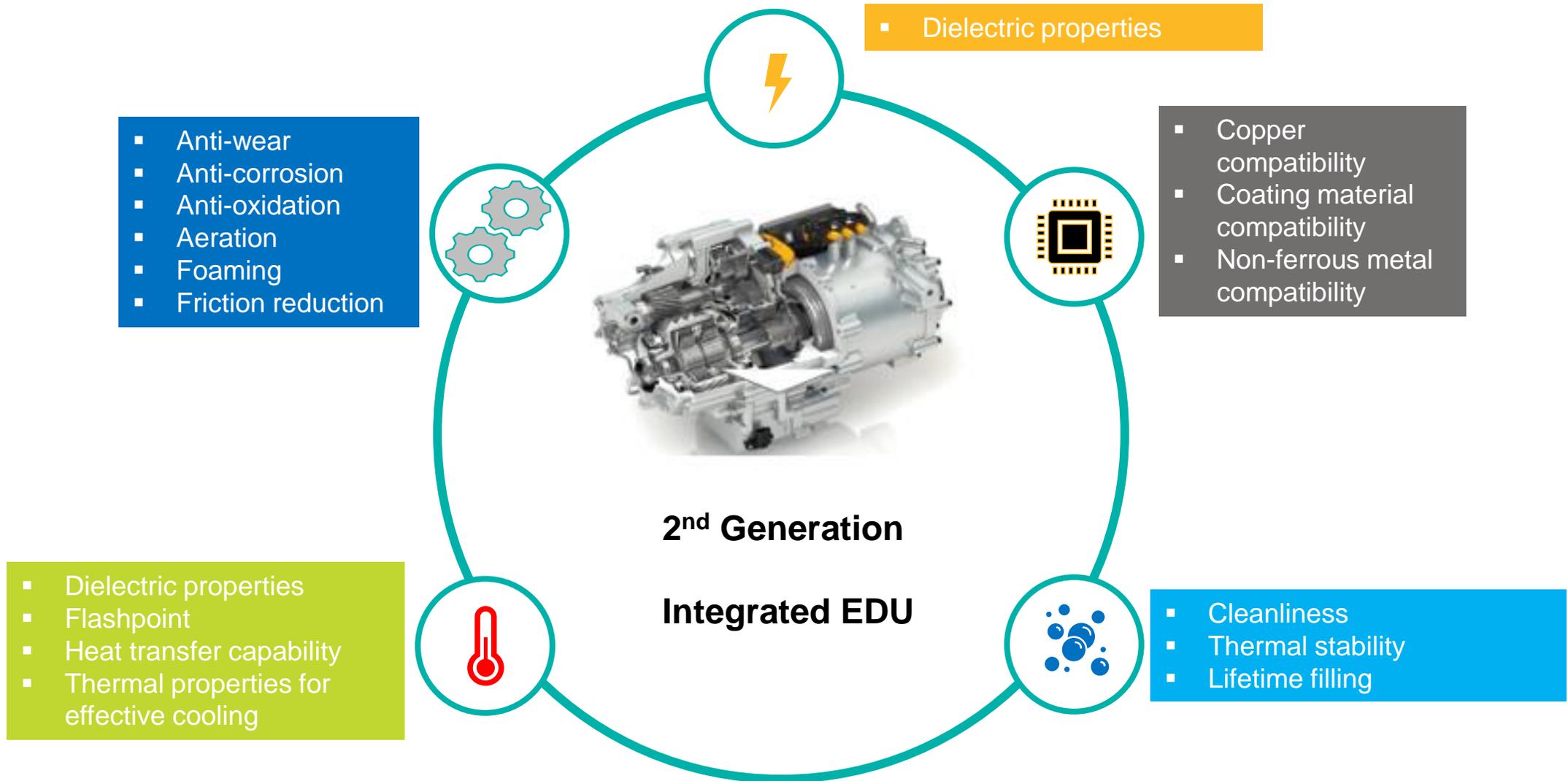
The Electric Drive Unit (EDU) is made by 3 key elements:

- Power electronics/Inverter
- Reduction Gear
- E-Motor

Direct cooling can increase the output up to 50%  
Possible to downsize the E-Motor



# 2° Generation requirements

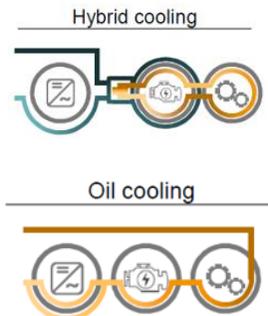


# Iona – Fluids for 2nd Generation of EDUs

## Electric Drive Unit



2<sup>nd</sup> Generation



## E-Axle



- Usually, higher scuffing protection needed
- Usually no clutches involved (multi-stage e-Axle only on high power applications)
- E-motor can be integrated

## DHT (e-DCT / e-ATF)



- Usually, lower scuffing protection needed
- Clutches involved
- E-motor is integrated

# Critical components

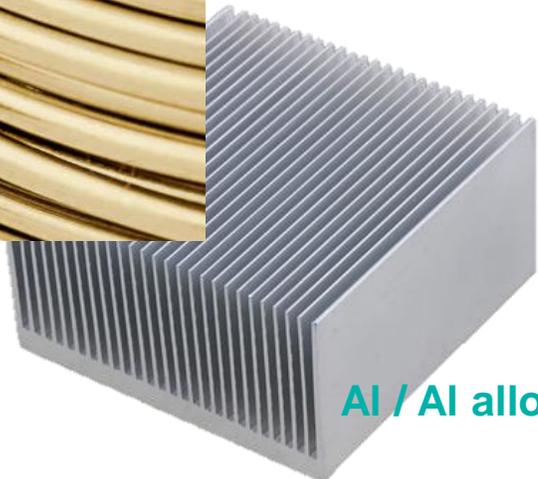


Cu / Cu alloys

Sn

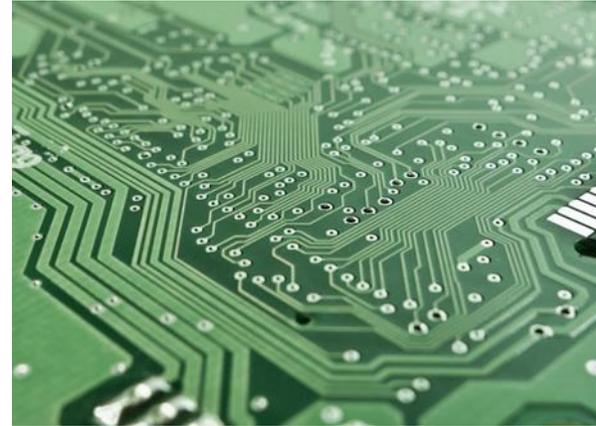


Brass

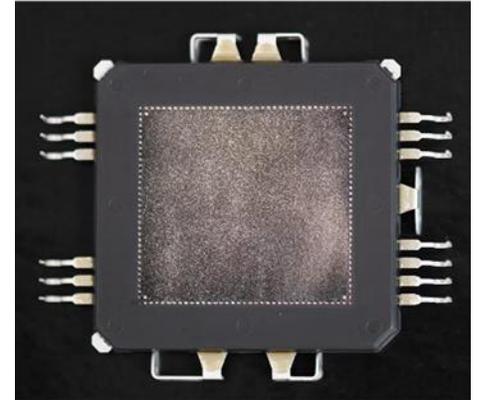


Al / Al alloys

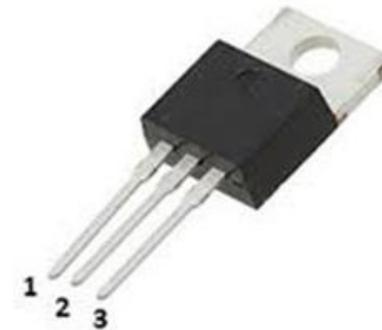
## Printed Circuit Boards



## Microelectronics packaging



## Transistors and electronic components



## Hairpins Insulating Lacquers



# Critical components



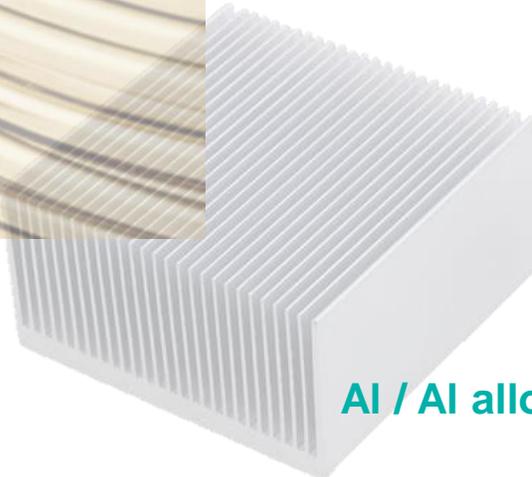
Cu / Cu alloys



Sn



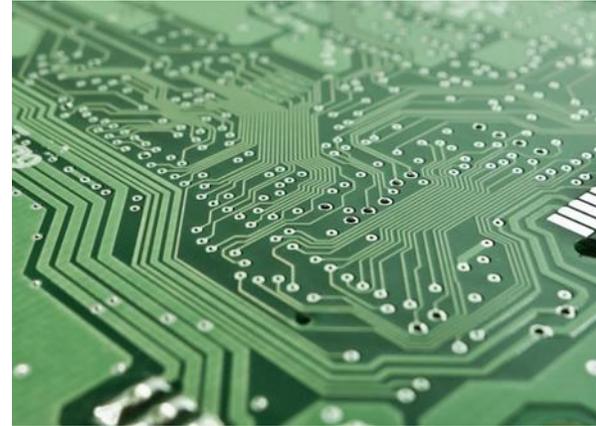
Brass



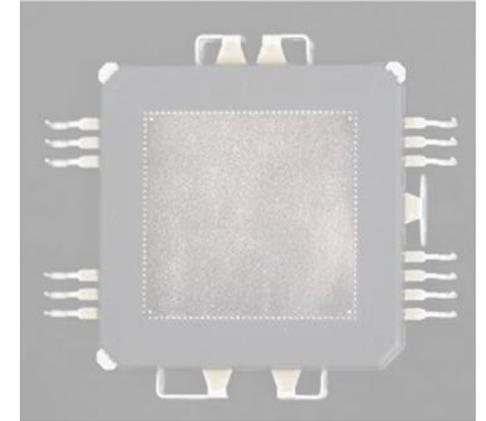
Al / Al alloys



## Printed Circuit Boards



## Microelectronics packaging



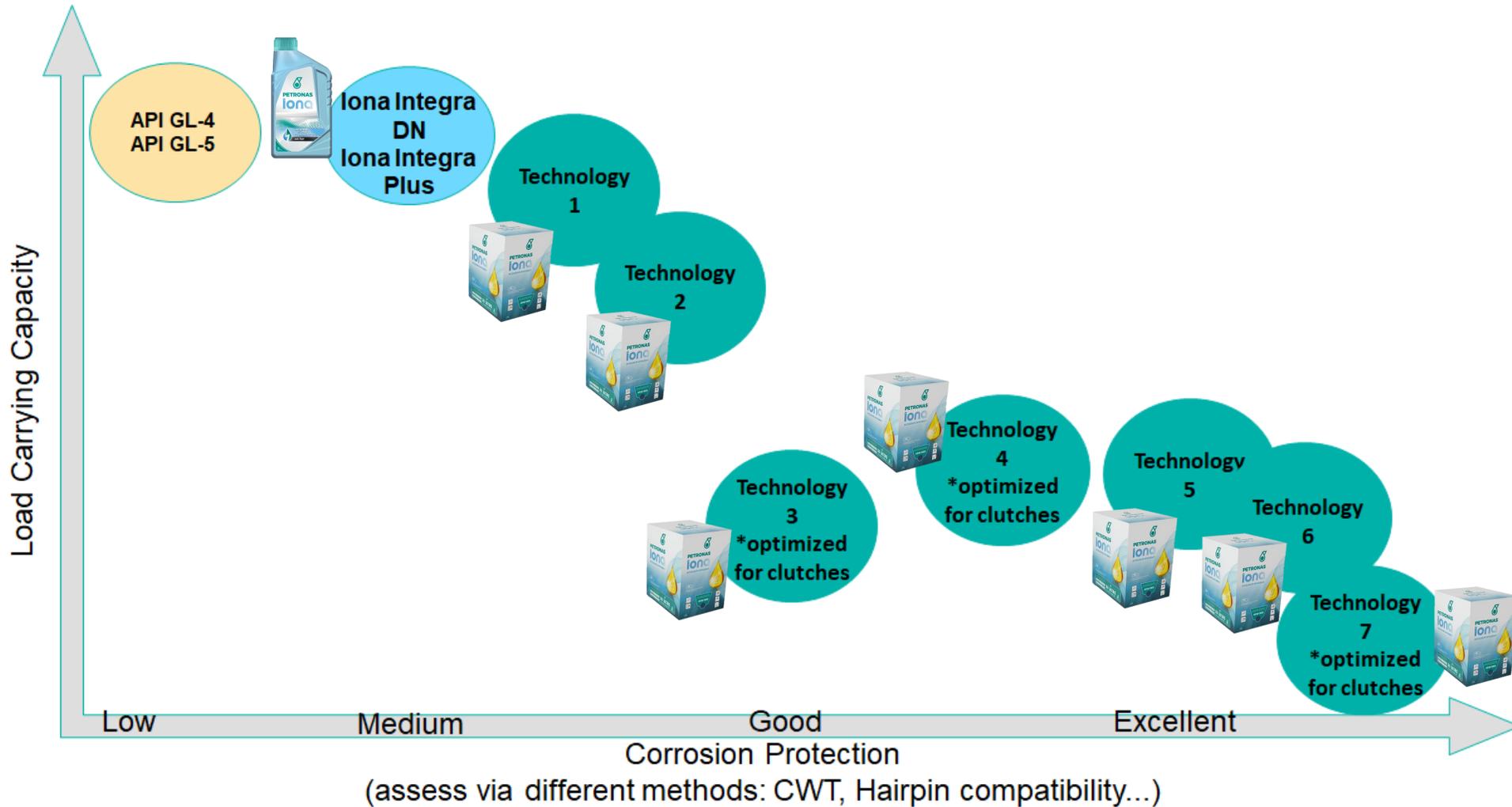
## Transistors and electronic components



## Hairpins Insulating Lacquers



# Iona 2<sup>nd</sup> Generation: Copper compatibility vs. Load Carrying Capacity



**Standard ASTM methods  
for corrosivity assessment  
&  
their limitations**

# Compatibility with Copper: Standard ASTM methods and their limitations



Designation: D130 – 18

Federation of Societies for  
Paint Technology Standard No. Dt-28-65  
British Standard 4351

## Standard Test Method for Corrosiveness to Copper from Petroleum Products by Copper Strip Test<sup>1</sup>

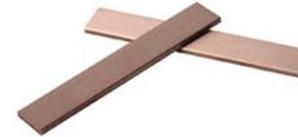
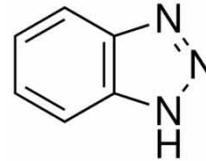
This standard is issued under the fixed designation D130; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the U.S. Department of Defense.*

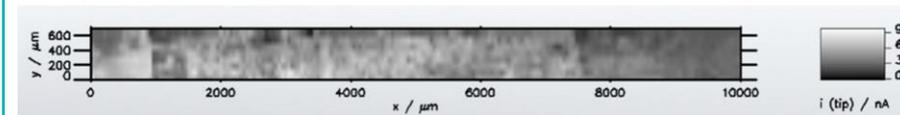


<sup>1</sup>Scanning electrochemical microscopy (SCEM) Principle and Application. *Dr. Michaela Nabel. Metrohm Users Meeting 2019, Zofinghen.*

Benzotriazole,  
Cu Corrosion inhibitor



Scanning Electrochemical  
Microscopy



↑ Tarnishing  
degree

↓ Corrosion  
Current

**In presence of surface-active molecules  
ASTM D130 results can be misleading**

# Compatibility with Copper: Standard ASTM methods and their limitations



Designation: D 6594 – 04

An American National Standard

## Standard Test Method for Evaluation of Corrosiveness of Diesel Engine Oil at 135°C<sup>1</sup>

This standard is issued under the fixed designation D 6594; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

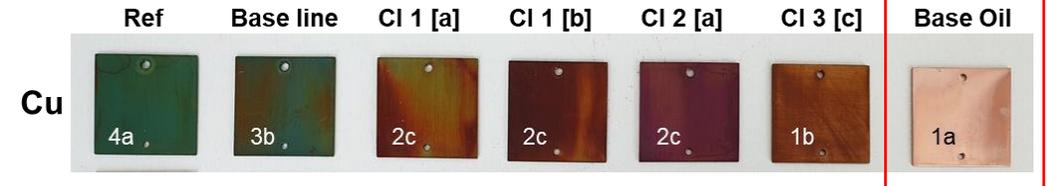


ASTM D5185 ICP  
+  
ASTM D130 Rating

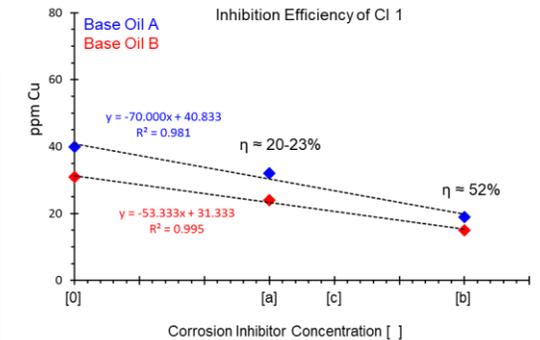
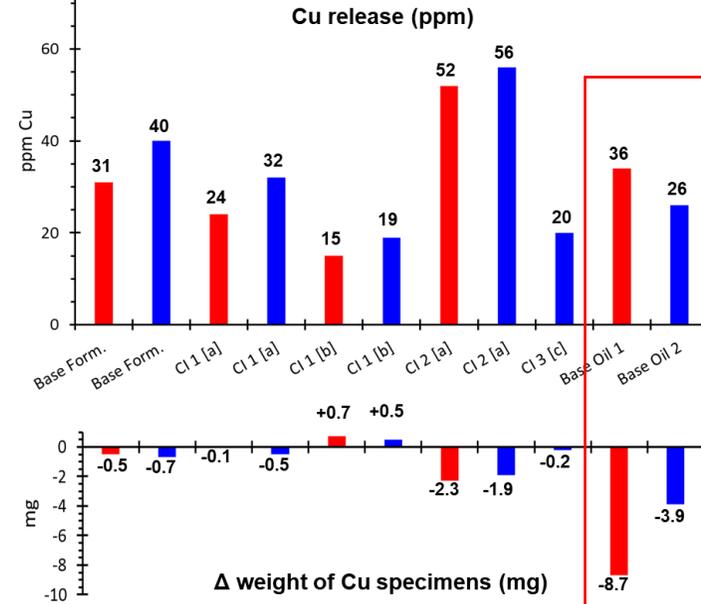


## Use of ASTM D6594 for Automatic Transmission Fluid (ATF) Development

### Visual Rating



### Quantitative Rating



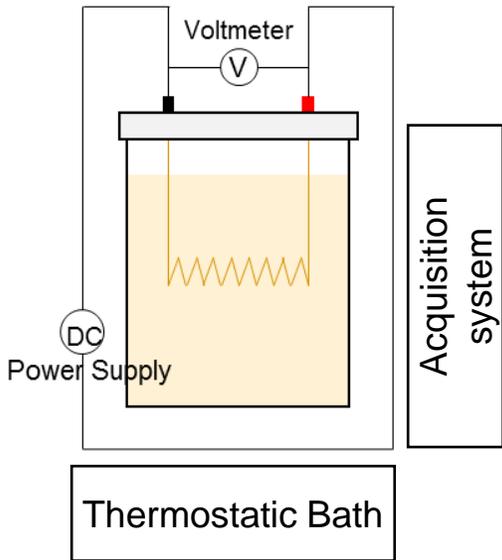
ASTM D6594 results can be misleading

# **Novel Method for Corrosivity Assessment: Copper wire resistance method**

# Copper wire resistance method<sup>1,2,3</sup>: Principles

Despite the method is not an ASTM standard, PLI recognized its potentialities and decided to invest resources to implement it in house as key method for e-fluids development and technology selection.

## Experimental Set-up



## Outputs information

$$Resistance = \frac{\rho Cu_T l}{\pi r^2}$$

$\rho Cu_T$  Resistivity of Copper at Testing Temperature  $T$  ( $\Omega m$ )  
 $T$  Testing temperature ( $^{\circ}C$ )  $\rightarrow$  Constant (Imposed/Measured)  
 $\rho Cu_{20^{\circ}C}$  Resistivity of Copper at  $20^{\circ}C$  ( $\Omega m$ )  $\rightarrow$  Constant  
 $\alpha$  Coefficient of thermal expansion of Cu  $\rightarrow$  Constant  
 $\rho Cu_T = \rho Cu_{20^{\circ}C} + \alpha(T - 20)\rho Cu_{20^{\circ}C}$   
 $l$  Copper wire length (m)  $\rightarrow$  Constant  
 $r$  Copper wire radius (m)

## Practical meaning of the outputs

### Realtime Resistance evolution:

$\Omega$  Constant

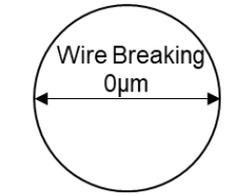
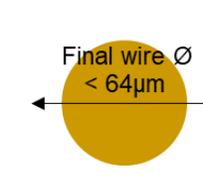
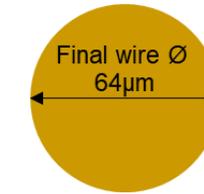
$\Omega$  Constant  
Increase

$\Omega$  Exponential  
Increase

$\emptyset$  Constant

$\emptyset$  Reduction

Wire braking



Corrosivity:

No/Low

Cu compatibility:

High



Corrosivity:

Low/Medium

Cu compatibility:

Medium/TBD



Corrosivity:

High

Cu compatibility:

No

<sup>1</sup> New Insight on the Impact of Automatic Transmission Fluid (ATF) Additives on Corrosion of Copper. Michel P. Gahagan et al. International Journal of Automotive Engineering 7 (2016) 115-120

<sup>2</sup> Wire resistance method for measuring the carrion of copper by lubricating fluids. Gregory J. Hunt et al. Lubrication Science 29 (2017) 279-280

<sup>3</sup> Automatic transmission fluid corrosion inhibitor interaction with copper. Michel P. Gahagan et al. Lubrication Science 30 (2018) 301-315

# Copper wire resistance method<sup>1,2,3</sup> : PLI Measurement set-up



**Instrument:** Metrohm Autolab Multi 204.  
**Temperature Measure:** pX1000 Module  
**Software:** NOVA 2.1.5  
**Acquisition mode:** Galvanostatic  
**Applied Current:** 1 mA  
**Sampling interval:** 10 s  
**Temperature:** TBD typical 50 – 160°C  
**Time:** TBD, typical 168 – 336 hours  
**Raw Data Analysis:** Excel

## Method improvement

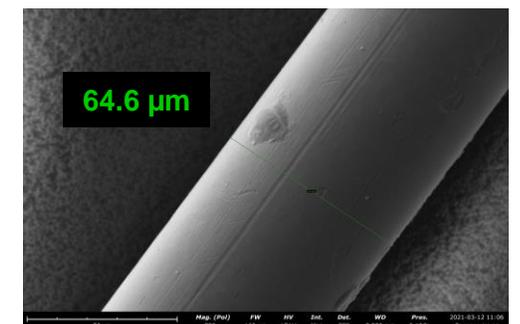
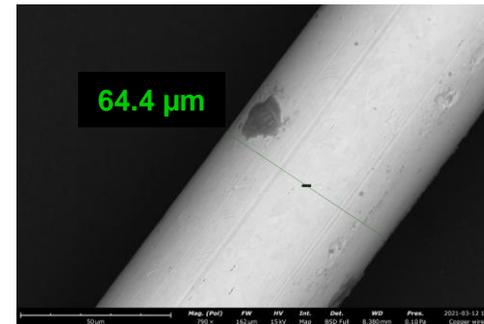
Scanning Electron Microscopy (SEM)+  
Energy Dispersive X-Ray Analysis (EDX)



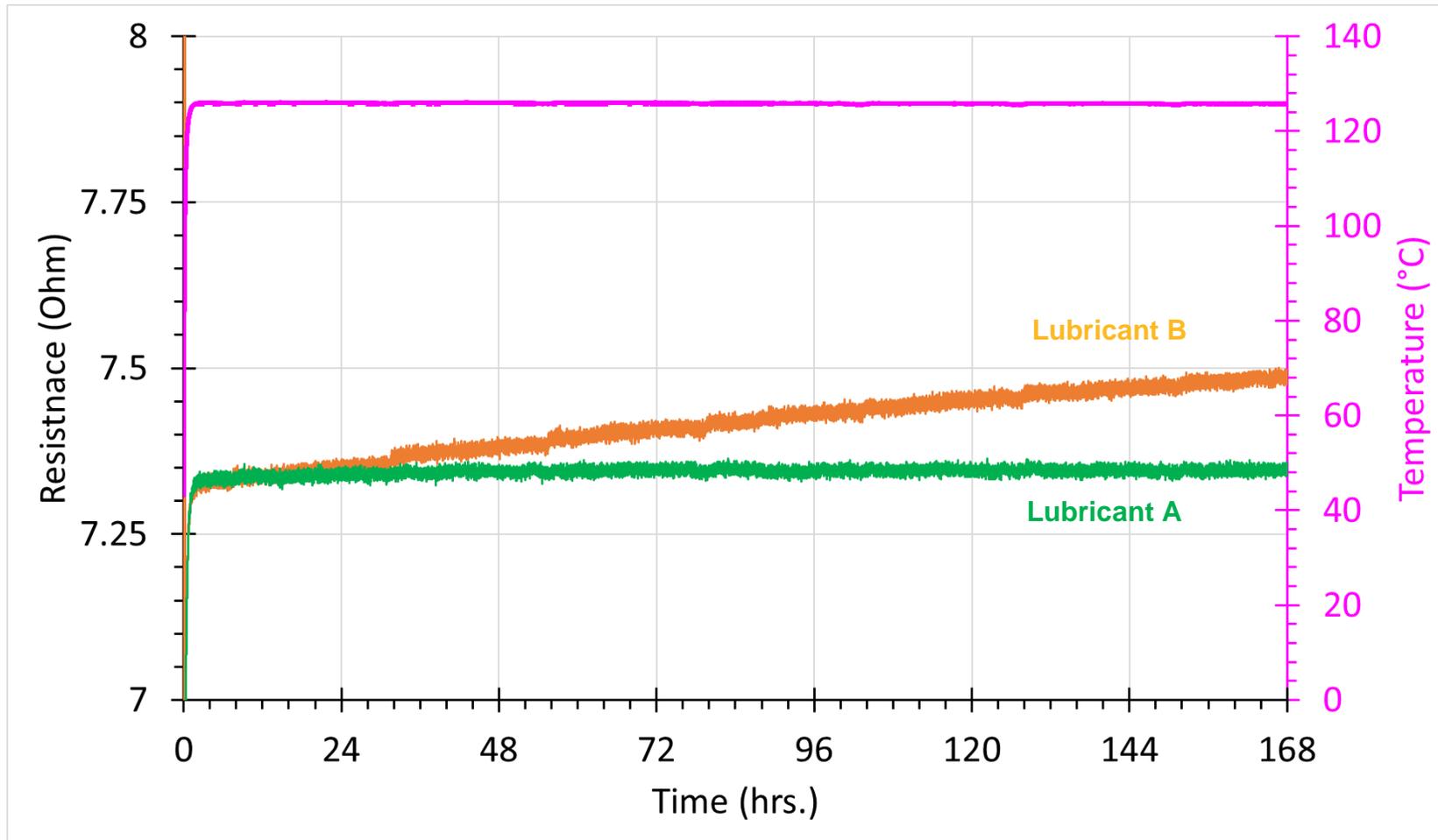
PLI decided to invest in a Benchtop SEM + EDX to maximise the information and to achieve a deep understanding of the corrosion phenomena taking place on the copper wire surface.

## SEM images of Cu wire in pristine state

Backscattered Electron Detector (**BSD**)    Secondary Electron Detector (**SED**)



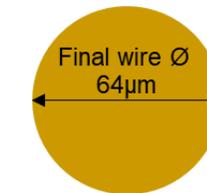
# Copper wire resistance method: Typical output plot



## Lubricant A

$\Omega$  Constant

$\emptyset$  Constant



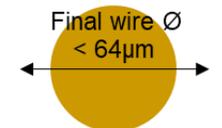
Corrosivity:  
**No**

Cu compatibility:  
**High**

## Lubricant B

$\Omega$  Constant Increase

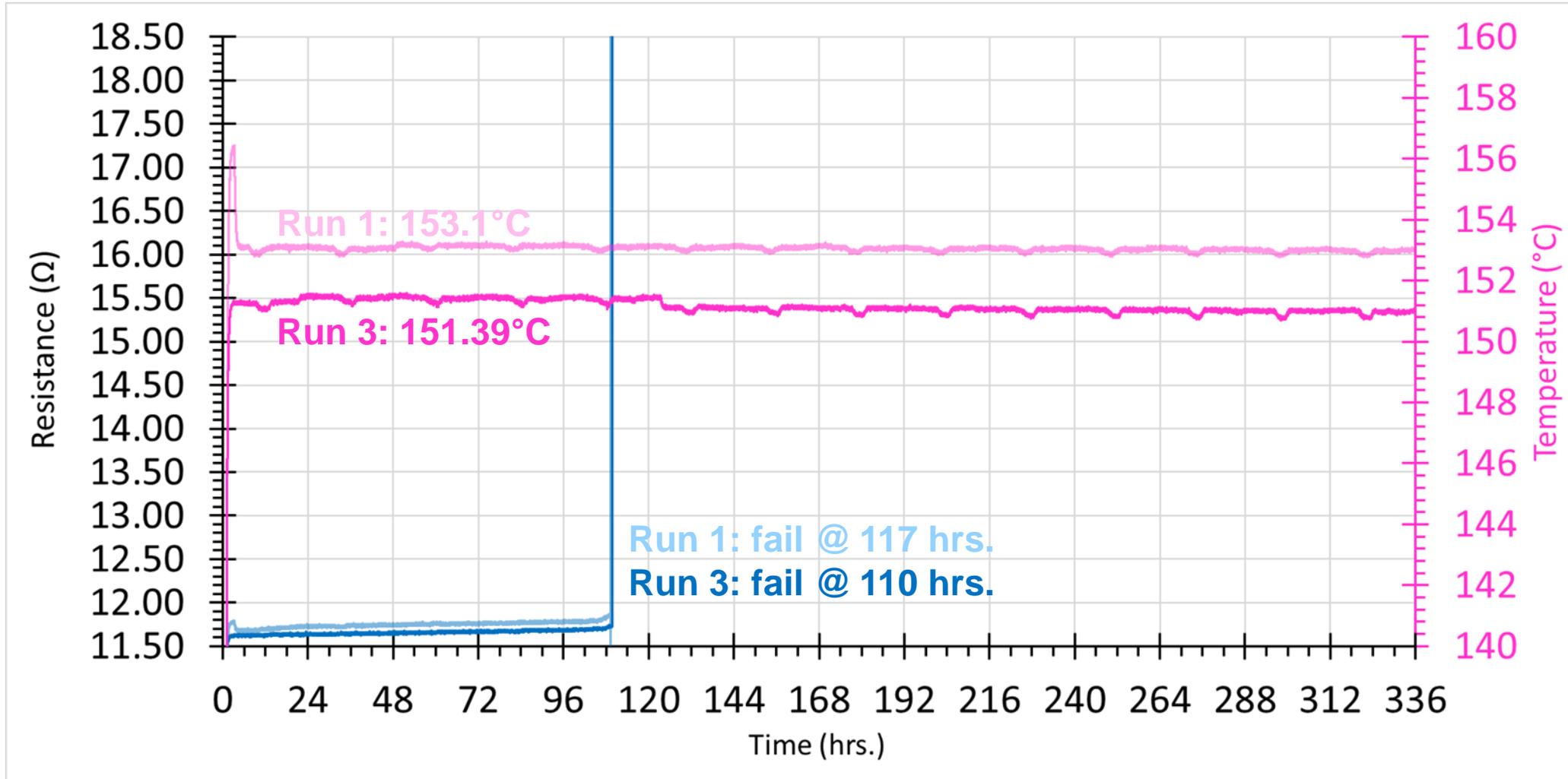
$\emptyset$  Reduction



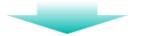
Corrosivity:  
**Low**

Cu compatibility:  
**Good**

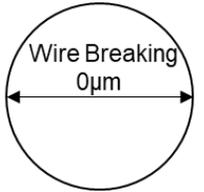
# Copper wire resistance method: Repeatability Assessment



**$\Omega$  Exponential Increase**



**Wire breaking**



 Corrosivity:

**High**

Cu compatibility:

**No**

**Copper wire resistance method.**

**Case Study:**

**Performance additive selection**

**(Technology)**

**Copper wire resistance method.**

**Case Study:**

**Formulation components  
optimization**

**Copper wire resistance method.**

**Case Study:**

**Formulation performances  
optimization**

# Outcomes

## Outcomes

- ASTM D130 and ASTM D6594 results can be misleading in particular if surface-active molecules are present in the formulation.
- The Copper Wire Resistance Method combined with SEM + EDX allows a well understanding of the corrosion mechanism of copper in contact with lubricants.
- The Copper Wire Resistance Method demonstrated to be very important for lubricants development in particular for technology selection and performances – component optimization.
- The application field of the Copper Wire Resistance Method is going to be extended to other metallic materials and other fluid e.g. Coolant and Battery fluids.

**Thank you for your passion!**



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