Understanding corrosion to enable next-generation metals

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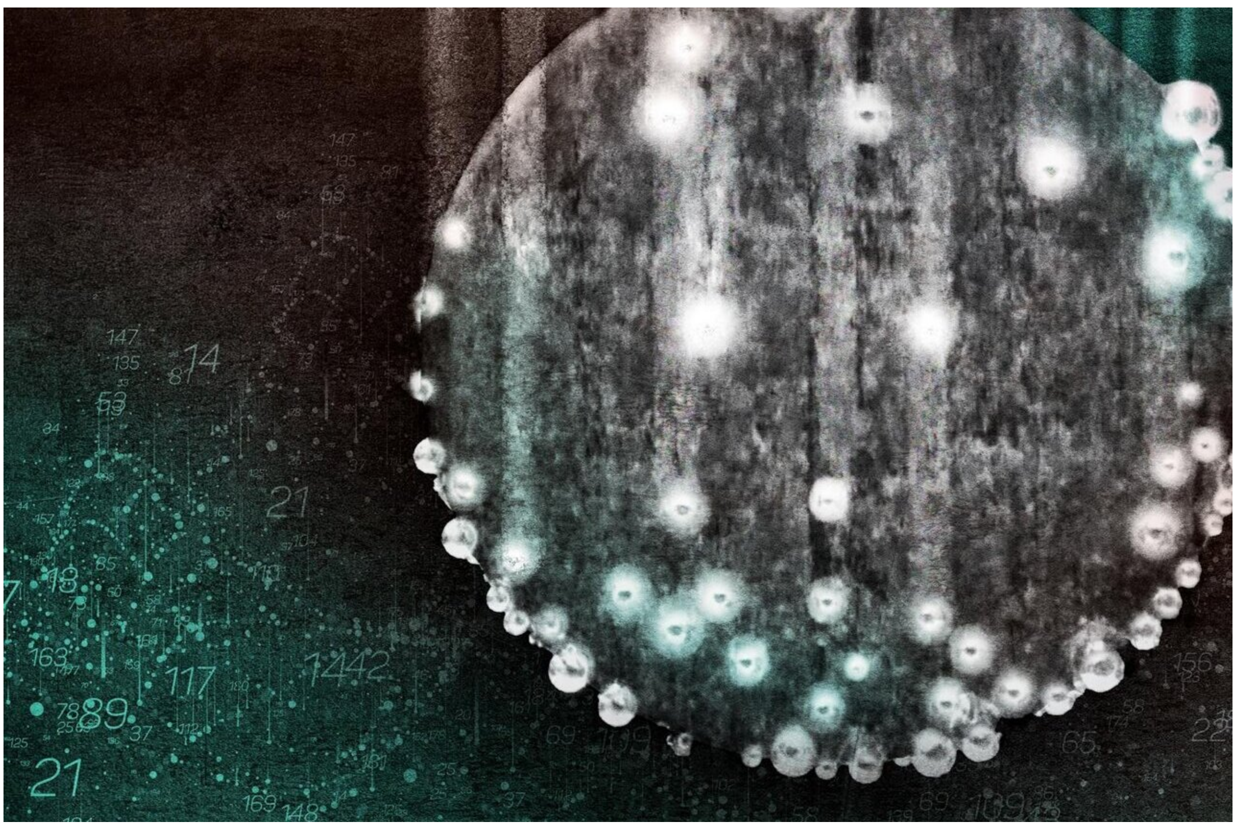

PNNL's new method of monitoring corrosion offers higher resolution and better reliability. Credit: Melanie Hess-Robinson | Pacific Northwest National Laboratory

Researchers are using new, experimental techniques like Shear Assisted Processing and Extrusion (ShAPE) and friction stir welding to produce metal components that are lighter, stronger, and more precise than ever before. But as we enter those new frontiers of metalworking, it's crucial to understand the performance and properties of the resulting metals and the bonds between them.

Corrosion—a process by which metals degrade—can pose serious problems over time, but until now, it's been tricky to visualize and explain exactly how corrosion progresses through a metal or a bond between two metals.

Now, researchers at Pacific Northwest National Laboratory (PNNL) have developed a new technique to get a high-resolution look at how—and why—corrosion happens. Their research was highlighted in the August 2023 and October 2023 issues of *Scientific Reports* and in the July 2022 issue of *The Journal of Physical Chemistry*.

The problem with 'cook-and-look' and other methods

"One of the main challenges when it comes to measuring corrosion is that it's mostly 'cook-and-look,'" explained Vineet Joshi, a materials scientist at PNNL. "Typically, researchers take a sample, immerse it in their chosen medium and, after a certain period of time, observe the corrosion—but only after it has occurred. Then, they generate numerous hypotheses to explain the corrosion."

This method has major disadvantages. Only measuring at a few time intervals leaves researchers to speculate about how the corrosion began and moved through the metal—and repeatedly removing and reinserting the sample can lead to skewed results.

Other methods, such as the scanning vibratory electrode technique or scanning electrochemical cell microscopy, involve dipping the sample and then using current to measure the electrochemical properties inside the samples—but surface abnormalities and other irregularities can interfere with the results.

Multimodal corrosion analysis

At PNNL, the researchers working to understand results from processes like friction stir welding and ShAPE knew they needed to develop a better approach to monitoring corrosion.

"We specifically wanted to transition from cook-and-look and instead look at specific initiation sites of corrosion to observe the corrosion in real time," Joshi said. "To address this, we created a novel macroscale analysis system called multimodal corrosion analysis."

Through multimodal corrosion analysis, the researchers use sensors, cameras, electrodes, and a hydrogen collection tube to observe the progress of corrosion in simple atmospheres; understand the nature of the surfaces using electrochemical techniques; and image and collect hydrogen gases, which are a byproduct of corrosion.

"By combining data from these simple and diverse modalities in real time, we can address fundamental questions regarding how corrosion initiates and propagates in materials," explained Sridhar Niverty, a materials scientist at PNNL. "The correlative imaging aspect also informs us about where to further investigate our materials to learn about why they corrode. The synergistic combination of these techniques yields significantly more information about a material's performance than was possible until now."

Looking at things from macroscale perspective provided the team with unique insights; however, the process of corrosion happens at a much finer scale.

Scanning electrochemical cell impedance microscopy

So, to analyze corrosion with even more precision, scientists at PNNL developed a new technique called scanning electrochemical cell impedance microscopy that offers much more reliable and high-resolution results.

"In this technique, we have everything needed to initiate the corrosion in a very small tube—or pulled capillary—including the electrolyte, reference, and current-collecting electrode," said Venkateshkumar Prabhakaran, a chemical engineer at PNNL.

"By landing the tiny opening of this capillary on the surface, we measure localized and time-dependent electrochemical properties without getting any interference from nearby regions. That helps us capture weak and strong spots on the surface prone to corrosion, which are otherwise lost when doing the bulk-scale measurement and formulate-suitable mitigation strategies."

This new approach builds on a prior technique called scanning electrochemical cell microscopy that emerged a few years ago. The PNNL team evolved that technique with electrochemical impedance spectroscopy to measure low-frequency impedance, which correlates to the resistance of the metal and allows for a microscopic view of how resistance changes over time.

"Adding impedance spectroscopy to the technique has been invaluable in understanding how a surface changes across metal joint (or alloy) by correlating resistances measured to the physical characteristics of the metal," said Lyndi Strange, a chemist at PNNL. "We have validated our method by comparing bulk impedance responses to responses measured via the new technique, which shows how we can now isolate specific corrosion events on the surface."

Applications for friction stir and more

There are a lot of real-world benefits to this kind of granularity—particularly at PNNL, where researchers are working hard to produce and test lightweight materials and joints for vehicle applications using novel methods like ShAPE and friction stir welding.

"Due to its unique capabilities, the new technique is being employed to acquire electrochemical responses from various microstructural features: grains, grain boundaries, interfaces, second phases, precipitates, and so on," explained Rajib Kalsar, a materials scientist at PNNL. "Obtaining individual electrochemical properties at the microscopic level is beneficial for designing high corrosion-resistant structural materials."

In the friction stir scribe process, for instance, a tiny cutting device is used to join materials with drastically different melting points without the need for fasteners. But researchers needed to understand how this new method of joining affected corrosion at the interface between the two metals—in one case, a friction stir scribe bond between magnesium and steel, which is a crucial bond for producing lightweight vehicles.

"When employing the friction stir scribe technique for joints, we observed a slightly lower corrosion rate," Joshi said. "The decline in corrosion rates can be attributed to the emergence of specific high-resistance pathways at the interface during processing. These pathways led to a reduction in the corrosion rate of the magnesium."

"We're using our new technique left and right now," he added. "If you understand these interfaces for corrosion really well, you can start to design accurately, rather than overdesigning or underdesigning a component."

More information: Sridhar Niverty et al, Probing corrosion using a simple and versatile in situ multimodal corrosion measurement system, *Scientific Reports* (2023). DOI: 10.1038/s41598-023-42249-0

Venkateshkumar Prabhakaran et al, Investigating electrochemical corrosion at Mg alloy-steel joint interface using scanning electrochemical cell impedance microscopy (SECCIM), *Scientific Reports* (2023). DOI: 10.1038/s41598-023-39961-2

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Provided by Pacific Northwest National Laboratory

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