

Imaging and analyzing nanoparticles in an easy way: An exploration

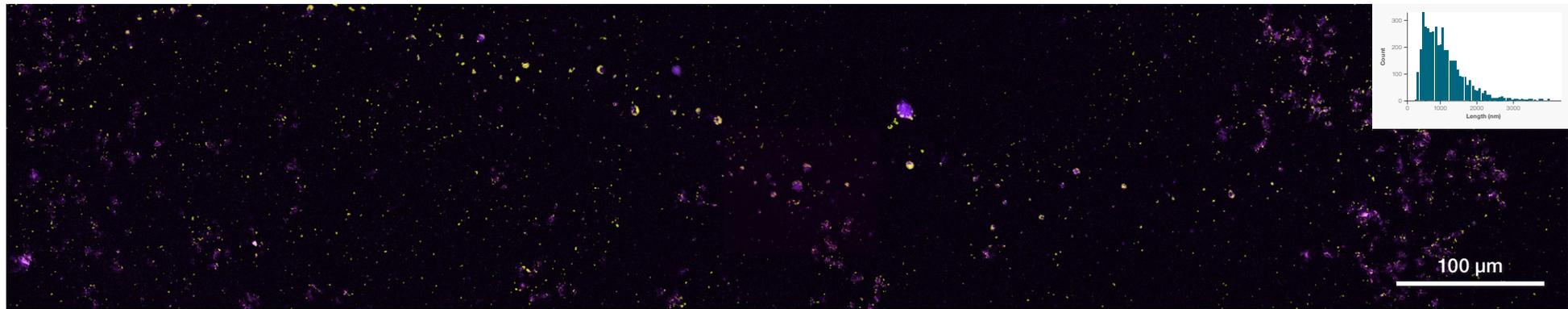
Effectively examine statistically relevant data, no matter
the nanoparticle type.

Nanoparticles. Seen nowhere, found everywhere.

Typically ranging from 1 to 100 nanometers (nm), these particles can be found in materials in almost every modern-day industry imaginable. Their broad applicability in materials is widely due to their unique chemical properties, high surface area, and nanoscale size. Their reactivity and bonding properties make these particles a frequent additive in various industries, including food products, cosmetics, energy-based research, aerospace engineering, and environmental applications.

Therefore, the quantification and characterization of chemical properties of nanoparticles within a material are crucial first steps to designing materials that are more intentionally designed, structurally stable, and safer for the public.

How TEM and EDS are uniquely suited for nanoparticle imaging



Thanks to improvements made by nanotechnology in the 20th century, we have enjoyed various revolutionary developments in a wide range of everyday materials. These improvements are partly due to parallel improvements made to resolution in transmission electron microscopy (TEM) used in conjunction with energy dispersive X-ray spectroscopy (EDS).

Today, we employ TEMs that can acquire images at the nanometer and even sub-nanometer scale for truly high-quality chemical data. The Thermo Scientific™ Talos™ TEM (Transmission Electron Microscope) used with EDS offer resolutions well below a nanometer, fit for a wide variety

of sample analyses. Additionally, Thermo Scientific Maps™ Software, enabled by Thermo Scientific Velox™ UI (User Interface) Software, automatically acquires an array of images across a sample and stitches them together to create one large final image. The acquisition of the images is done unattended. For streamlined image analysis, the Thermo Scientific Avizo2D Software allows researchers to perform image analysis by automating workflows for on-the-fly processing. Avizo2D Software generates information about nanoparticle samples, such as the size of the particles, surface area, perimeter, distribution, and chemical composition. It also contains deep learning / AI to accelerate the quality of the outcome. Therefore, the fully automated nanoparticle workflow (APW) that takes care of everything from acquisition to processing provides meaningful statistics on a wide variety of nanoparticles and precipitates.

As nanotechnology-based products begin to proliferate, their safety and environmental impacts are being studied. One area of research involves the exploration of the health impacts of nanoparticles, including titanium dioxide, silver, and gold, used to enhance food and cosmetics. The goal is to formulate a standard approach to the identification and characterization of the concentration of nanoparticles in food additives and to determine the volume of nanoparticles that are safe for human contact and consumption. Higher resolutions from Talos TEM instruments will enable researchers, even those still unfamiliar with TEM techniques, to generate quality data to help build the future of the safety and regulatory questions related to nanoparticles, in a very intuitive way.

Analyzing nanoparticles for safer food products

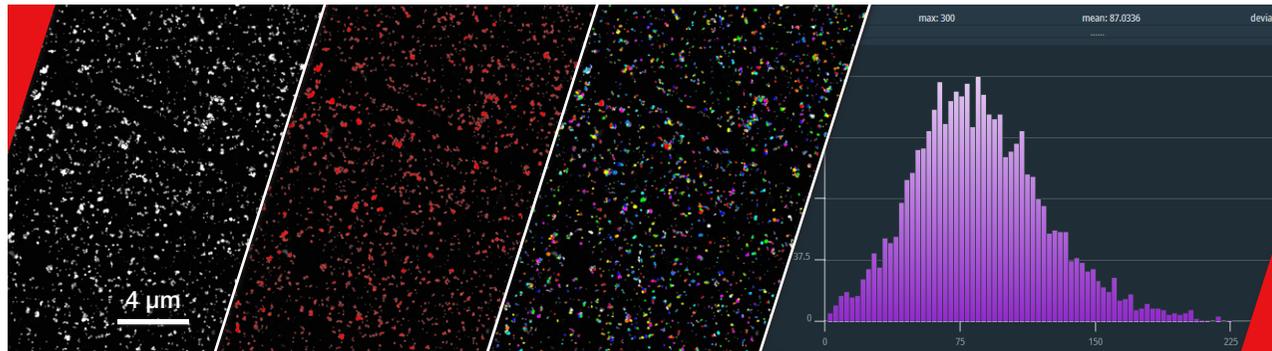


Figure 1. Particle size distribution of Ti-oxide nanoparticles (additive E171) in commercially available candy.

Facilitating an optimal nanoparticle characterization is imperative to engineering food and cosmetic products that align with governmental regulations and promote better health outcomes. Therefore, quantifying a nanoparticle's composition, morphology, and scale in consumables early on in food or cosmetic products research strengthens a material's stability and furthers our understanding of nanoparticle chemistry.

Studying nanoparticles such as silver, gold, and titanium dioxide (TiO_2) continues to be an area of interest to ensure that these nanoparticles provide their intended effects within consumables. In food products, TiO_2 primarily functions as a whitener or adds opacity to a food product. Titanium dioxide or ultrafine titanium dioxide particles (TiO_2) in particular make a frequent appearance in many of the chemically complex food and household items that we eat and use in our day-to-day lives—from sunscreens to toothpaste and over 3,000 food items.

Therefore, a multifaceted approach is required to observe these hard-to-study materials. To start in data acquisition, the Talos TEM is capable of high enough resolutions to image even the most chemically complex food products, such as commercially available candy. In addition to the sugars, proteins, and normal foodstuffs found within candies, acquiring a clear image becomes even more complicated when there are also other nanoparticles in most candies, such as iron oxides and silvers. However, Figure 1 clearly illustrates how the Talos TEM can clearly capture the morphology and chemistry of these TiO_2 .

After the Talos TEM completes initial data acquisition, manually going through information-dense data can distract from making accurate analyses. Large area imaging has been a valuable technique to protect the context of observations while also providing statistically relevant data.

The Maps Software enabled with the Velox Software can enhance large area imaging by automatically collecting an array of images across a food product sample and stitch these images together to create one large final image, seen in Figure 1. The Avizo2D Software can be trained to perform automated segmentation and generate statistics such as size, surface area, perimeter, distribution, and chemical composition of nanoparticles in food products.

Employing the Talos TEM with data correlation and segmentation enabled by Maps and Avizo Software can streamline the typical bottlenecks of workflows, so researchers can focus on how to formulate better food and cosmetic products that promote healthier and more environmentally friendly outcomes.



Video 1: Talos APW analysis of TiO_2 nanoparticles for food. Duration 1.18

Ensuring intended chemical reactions with TEM

Nanoparticles are a common class of catalysts due to their high surface area, which provides many sites for chemical reactions. Therefore, refining a nanoparticle's shape and size is key to facilitating the intended chemical reaction in a catalyst. This surface can also be functionalized with additional catalytic compounds or stabilizing molecules that prohibit unwanted side reactions. The Talos TEM can image a catalyst's surface area at high resolutions, so researchers can better understand, then reverse engineer, a catalyst's properties to increase their performance and catalytic efficiency.

Nickel catalysts are used in the petrochemical industry, where the challenge is to create a specific size distribution that will promote a desired reaction. If a particle is too large, a reaction may not happen, but, if the nanoparticle is too small, the reaction may not be the intended one.

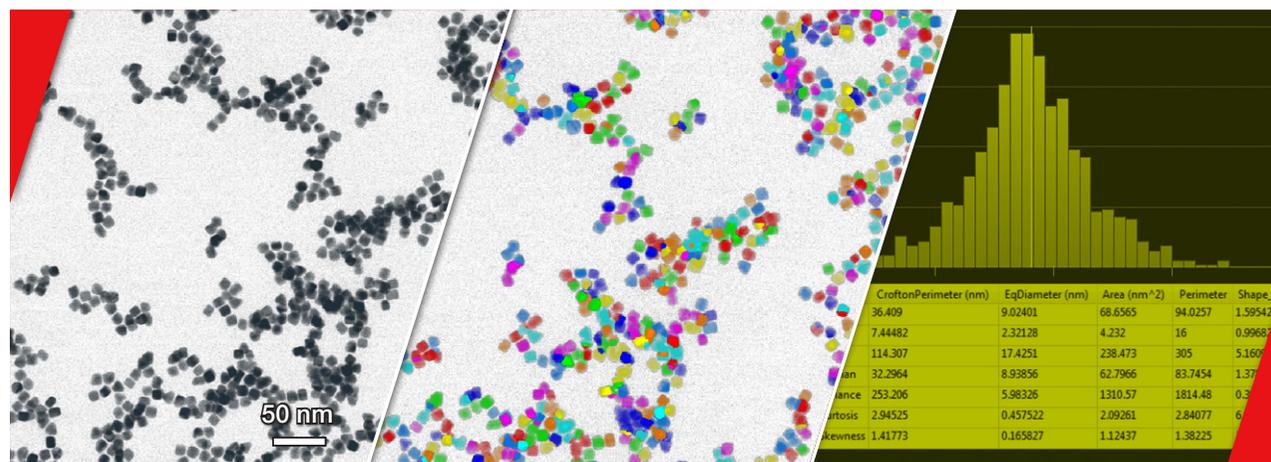


Figure 2. Example of individual Pt-Rh nanoparticles analysis done on a Thermo Scientific Talos F200X TEM: size, area, perimeter, shape factor, contacts, etc. *Sample courtesy of Prof. B Gorman and Prof. R. Richards, Colorado School of Mines.*

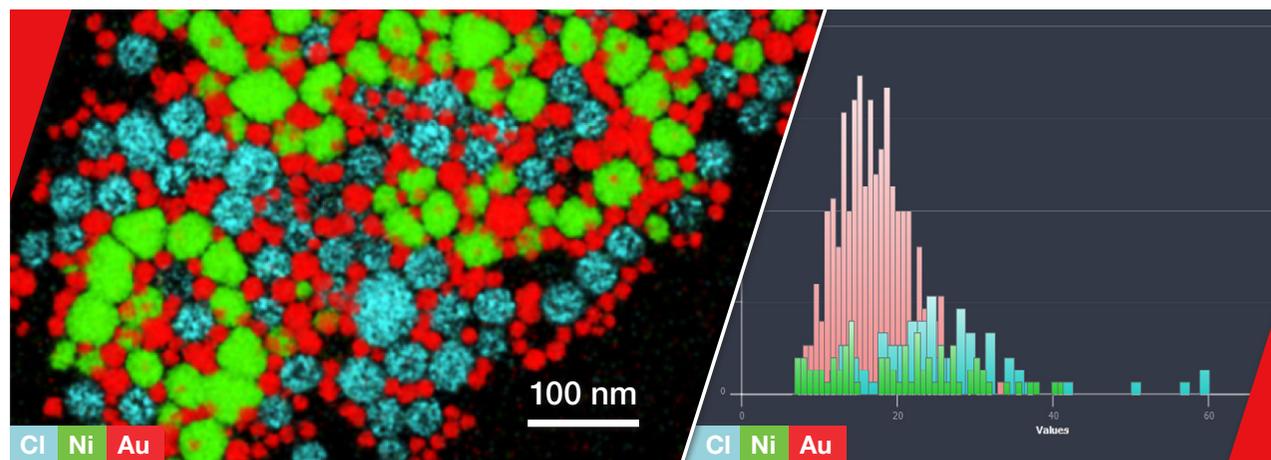


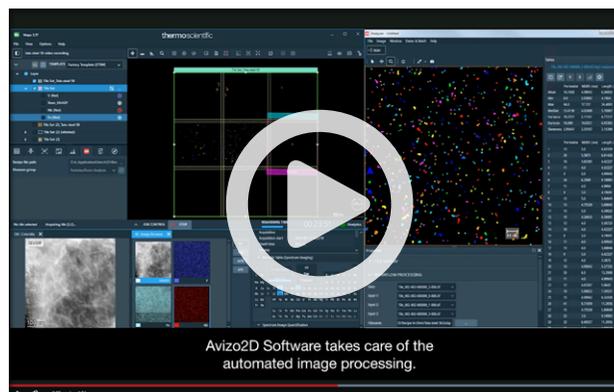
Figure 3. Example of large-area, high-resolution HAADF STEM and EDS mapping with Dual Bruker X-flash 100 Racetrack detectors (On a Thermo Scientific Talos F200i TEM) on gold-nickel nanoparticles, acquired in less than one minute. *Sample courtesy J. Bursik, Institute of Physics of Materials, Brno.*

Making stronger materials with metal precipitates

Metals researchers seek to improve the various mechanical properties of materials, such as microalloys, or high-strength low-alloy (HSLA) steels, in order to meet modern industrial and manufacturing demands of industries such as oil and gas extraction, construction, and transportation. Precipitates formed during steel manufacturing are known to have a significant impact on the mechanical properties of the resulting material.

Small additions of vanadium, niobium, and titanium have been shown to improve strength and toughness compared to mild carbon steel. These microalloys (<0.10% alloying elements) react with carbon and nitrogen to form nanoscale carbonitride precipitates. The exact nature of these metal precipitates continues to be an active area of research, and the Talos TEM is preferred for its high-resolution imaging, providing nanoscale information about these nanoparticles.

Video 2 examines the ease of data acquisition of HSLA steel data with a Thermo Scientific workflow.



Video 2: Talos APW analysis of precipitates in steel. Duration 1.04

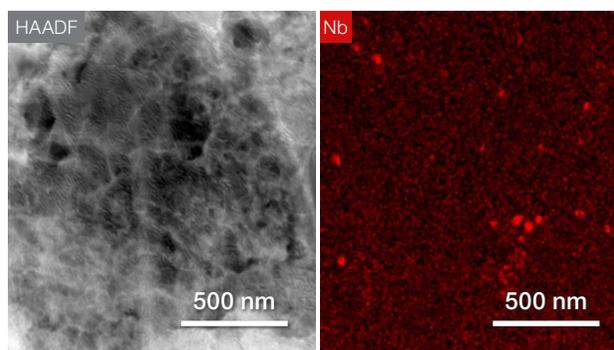


Figure 4: STEM mode imaging (left) and EDS mapping (right) of the same region in a steel lamella done with the Thermo Scientific Talos™ F200X TEM.

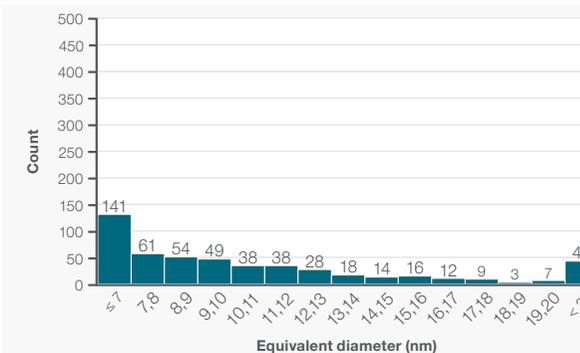
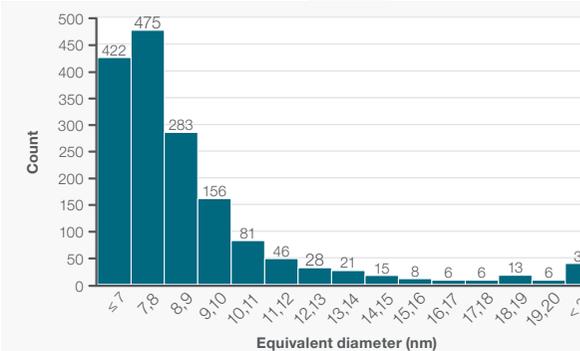
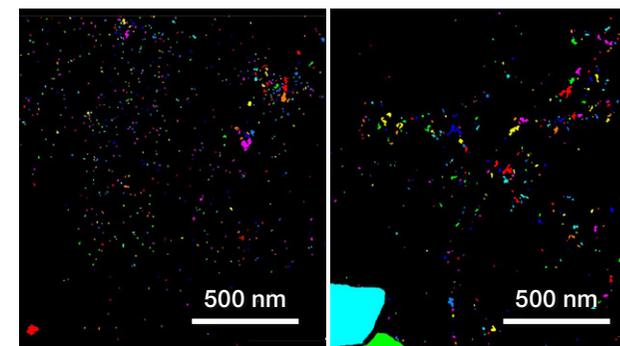


Figure 5: Closeup of APW particle maps for two steel lamella samples (top, right and left) with their associated particle size histograms. The map on the top-left correlates to the upper histogram, and the map on the top-right correlates to the lower histogram.

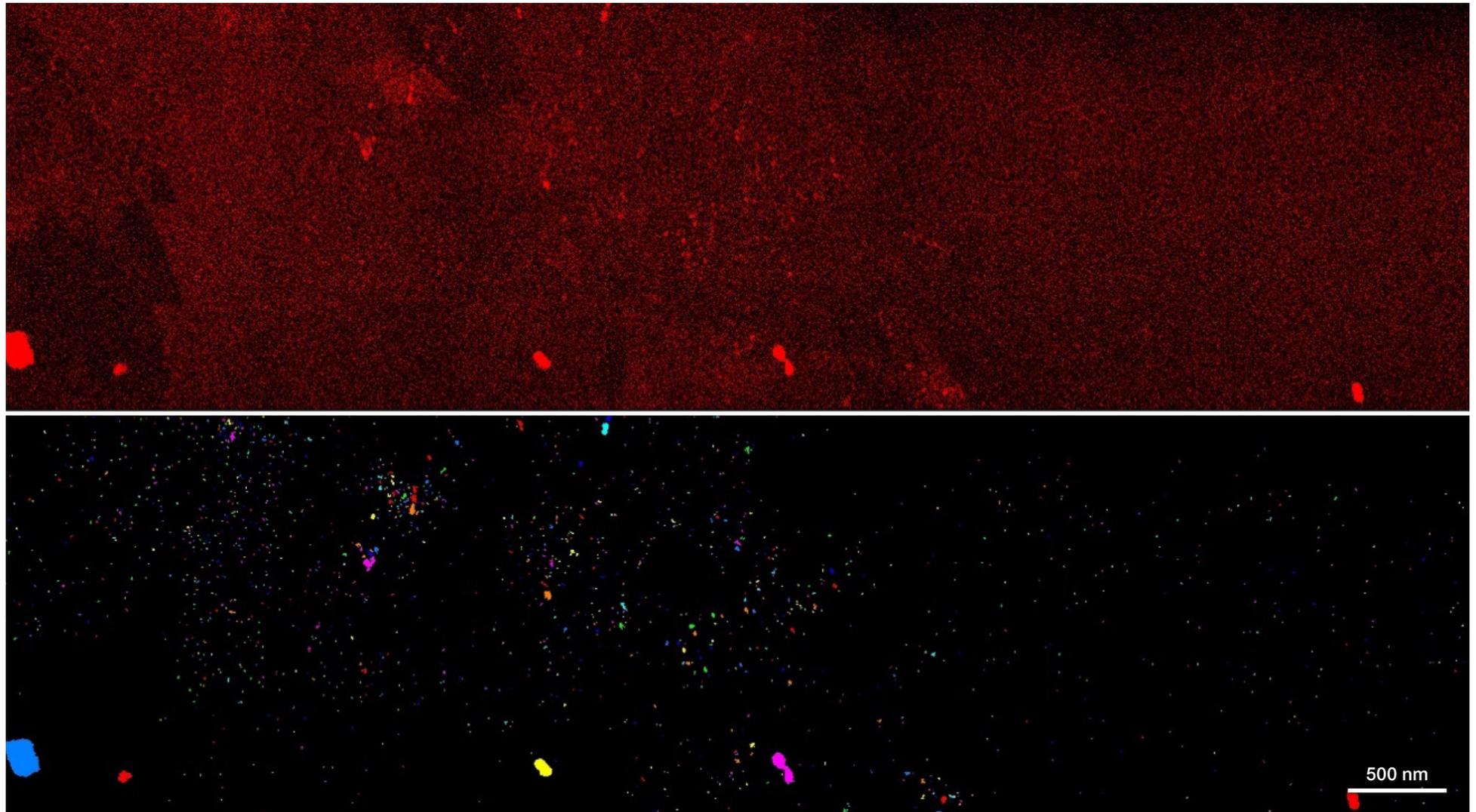


Figure 6: More images were collected using automated particle workflow (APW): EDS map of Niobium (top) and corresponding APW characterization of nanoparticles (bottom) of this $13.5 \mu\text{m}^2$ wide area scan. APW, for example, can segment different particles by changing the color.

Conclusion

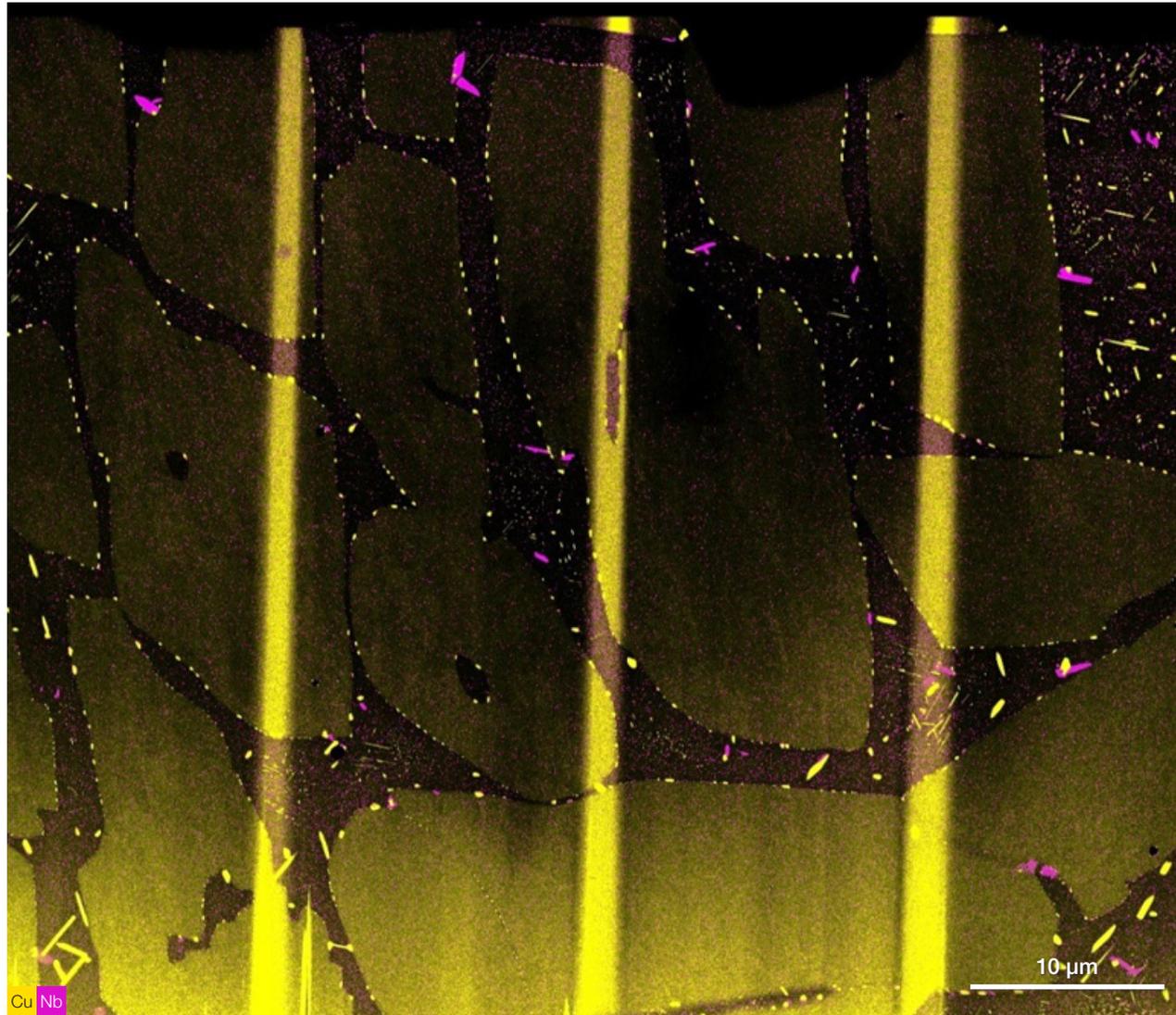


Figure 7. Visualization of multi-grain boundary segregation of nanometer-scale Cu/Nb precipitates in surgical-grade stainless steel.

The Talos TEM, used with the APW Pack, consisting of Maps Software, Avizo2D Software, and Velox Software, provides materials researchers with the necessary, high-resolution data to make significant chemical analyses for a wide variety of nanoparticle and precipitate data. The Talos F200X G2, F200S G2, and the F200i TEMs offer resolutions well below 1 nm, suitable for the acquisition of almost any type of nanoparticle data. This high-resolution enables research on catalysts, metals, batteries, polymers, and novel materials to accelerate the development of next-generation technologies.

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Step ahead. Step beyond. Duration 1.33

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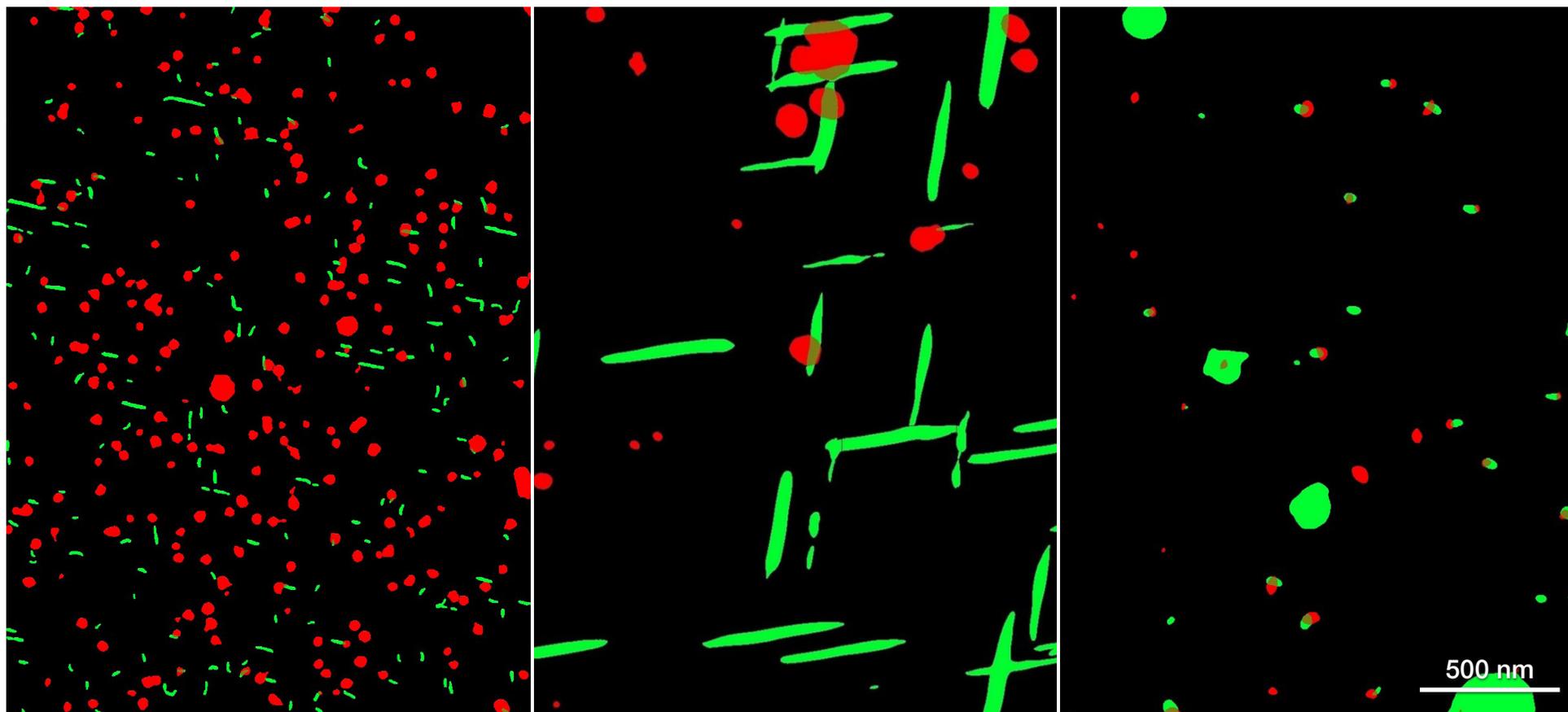


Figure 8. Precipitates containing copper (green) and zirconium (red) in a friction-stir-welded Al-Cu-Li alloy. The three regions represent the base metal (left), the heat-affected zone (middle), and the stirred zone (right).

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