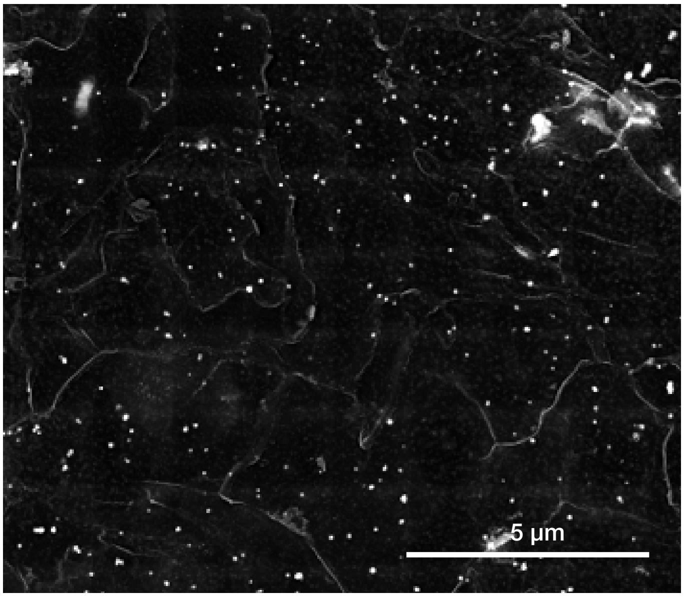
Metal Precipitate Analysis with the Automated Particle Workflow

Transmission electron microscope workflow for unattended, high-throughput imaging and data analysis of nanoscale precipitates in steel alloys.

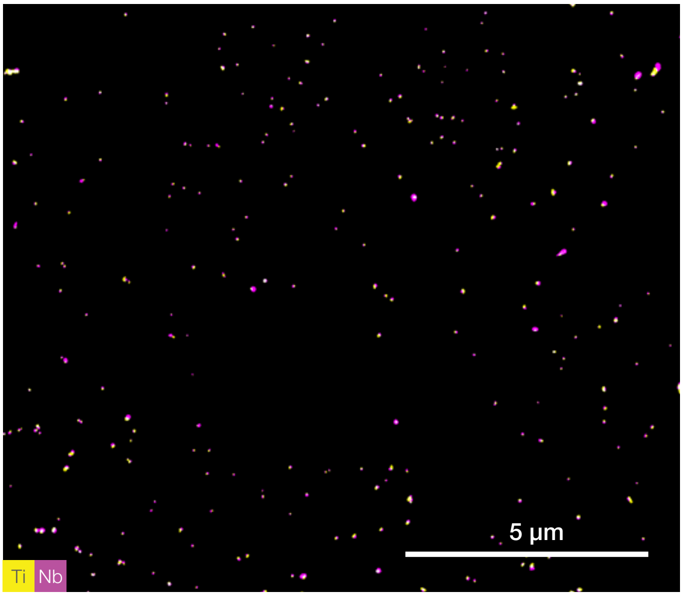
Introduction

Ongoing metals research seeks to improve the various mechanical properties of materials such as steels in order to meet modern industrial and manufacturing demands. In particular, precipitates formed during steel manufacturing are known to have a significant impact on the mechanical properties of the resulting material. The exact nature of these precipitates continues to be an active area of research, and transmission electron microscopy (TEM) is a preferred tool for this analysis as it is capable of providing high-resolution nanoscale information on the sample.

This routine analysis, however, requires large datasets in order to obtain reliable, statistically significant results. The Thermo Scientific Automated Particle Workflow (APW) combines our unique hardware and software into a robust, automated, and unattended nanoparticle characterization workflow ideally suited for precipitate analysis in steels.



This STEM image shows the heterogeneous distribution of precipitates on this microalloyed steel carbon replica sample. Complete area scanned (not shown) is 32 µm x 25 µm.



EDS maps of the corresponding area shows precipitates including titanium (yellow) and niobium (purple).

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Background

Microalloy or high-strength low-alloy (HSLA) steels are common in many industries such as oil and gas extraction, construction and transportation. These steels have shown improved strength and toughness compared to mild carbon steel due to small additions of vanadium, niobium and titanium. These microalloys (i.e. <0.10%) react with carbon and nitrogen to form nanoscale carbonitride precipitates. After casting, Ti-Nb-V carbonitrides are partially dissolved by reheating, and then re-precipitated during rolling and subsequent thermomechanical processing. The improved mechanical properties of HSLA steels result from grain refinement during hot rolling, which is governed by complex precipitates, and precipitation hardening.

While HSLA steels have been utilized for over 50 years, the science behind how these precipitates form is still an area of ongoing research. Transmission electron microscopy can be used to answer vital questions such as:

- Which types of precipitates are better for pinning austenite grain boundaries?
- What effect do compound precipitates have?

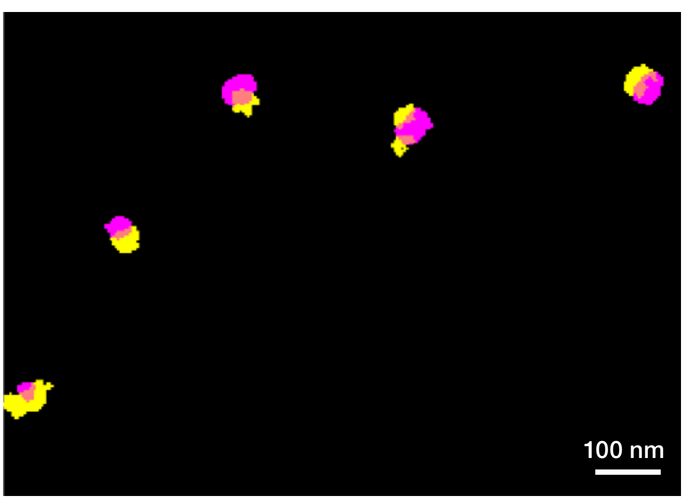
Additionally, recent second- and third-generation advanced high-strength steels (AHSS) have been replacing some HSLA steels due to their substantially higher strength and potential for light-weighting. AHSS are a wholly new grade of steel with a much higher amount of alloying (e.g. >1% aluminum and silicon in third-generation AHSS), which in turn means that they include new types and sizes of nanoscale precipitates.

Whether the grade of steel is old or new, the analysis of precipitates is key to understanding the effectiveness of the alloying and heat-treating processes. Our Automated Particle Workflow is specifically designed to facilitate this kind of crucial characterization.

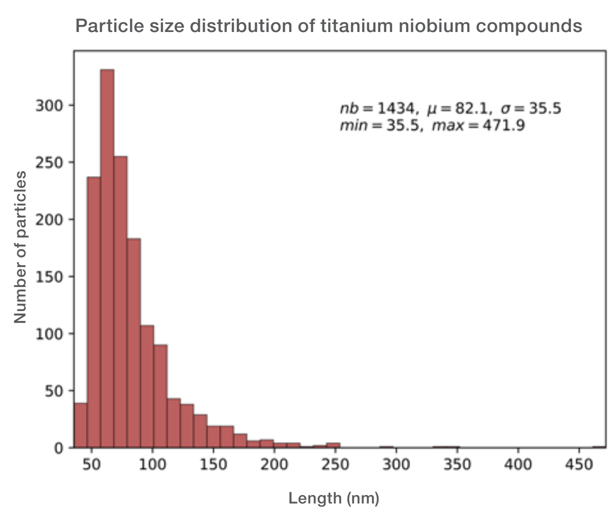
Automated Particle Workflow (APW)

Traditional TEM methods consist of manual spot analysis of the precipitates' chemical composition, or separate particle imaging without chemical information. Additionally, energy dispersive spectroscopy (EDS) can be performed on the TEM in order to determine the composition of individual precipitates, as it can be heterogeneous across a sample. The problem is that this method of analysis is time consuming and tedious, and the TEM operator can only collect compositional information for a few dozen particles per day. It would, therefore, take a researcher a week or more to collect EDS data for just a few hundred particles.

With APW, however, this entire process becomes automated, and can be left unattended, freeing the researcher's time for more critical tasks. Thousands of data points can be easily collected and characterized in one day, and statistically relevant data sets can, in some cases, be produced in just one hour. This accelerated analysis facilitates faster alloy and heat treating process development by significantly reducing the time spent on characterization.



Magnified area of the EDS map shows the compound of titanium (yellow) and niobium (purple) and zones where they overlap (orange).



Over 1,400 compound particles were characterized; the distribution of the overall particle length is shown here.

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