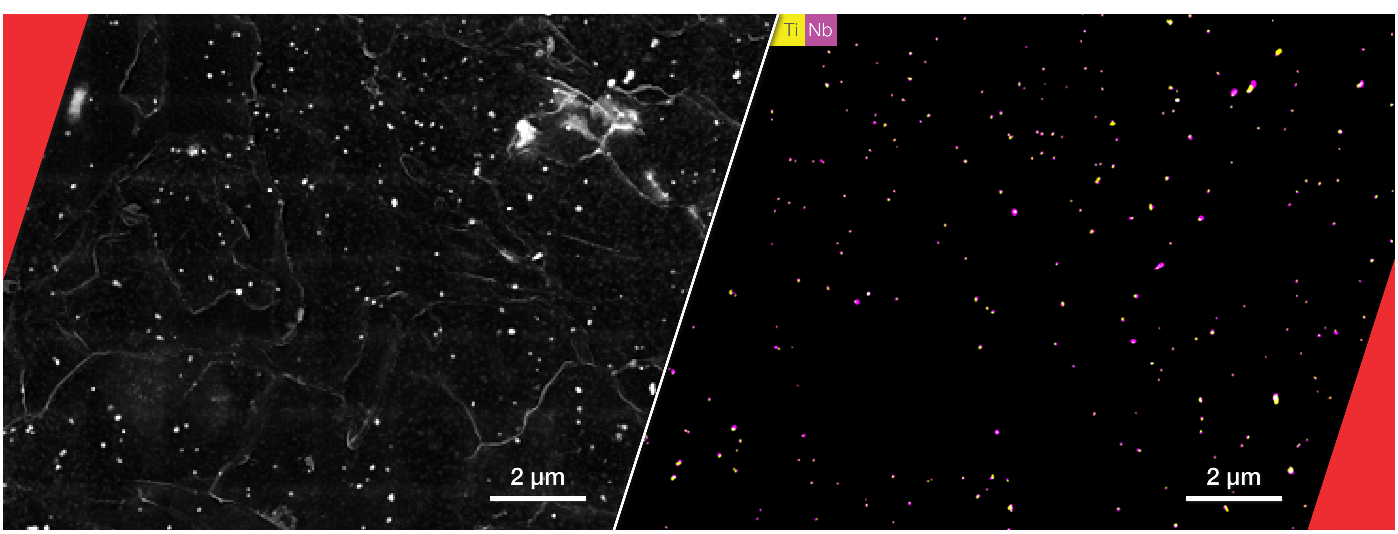
Precipitate Analysis in Metals with the Automated Particle Workflow

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

Introduction

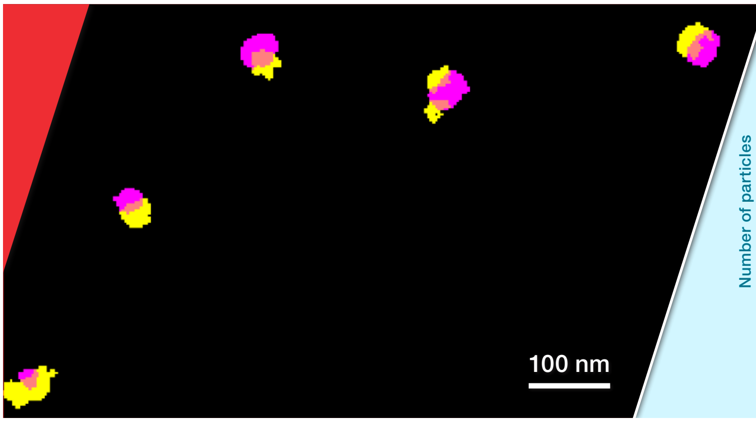
Metals researchers seek to improve the various mechanical properties of materials, such as steels, in order to meet modern industrial and manufacturing demands. In particular, precipitates formed during steel manufacturing are known to have a significant impact on the mechanical properties of the resulting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

This routine analysis, however, requires large data sets 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.

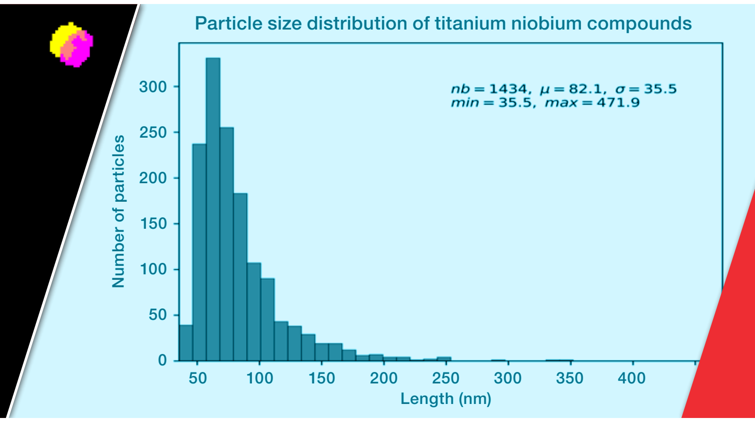


This STEM image shows the heterogeneous distribution of precipitates on this microalloyed steel carbon replica sample.

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



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



Distribution of the overall particle length. Over 1,400 compound particles were characterized. Total analysis time was one hour, including the overview images.

Goal

Microalloy, or high-strength low-alloy (HSLA), steels are common in many industries, such as oil and gas extraction, construction, and transportation. Due to small additions of vanadium, niobium, and titanium, these steels have shown improved strength and toughness compared to mild carbon steel. These microalloys (<0.10% alloying elements) 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 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.

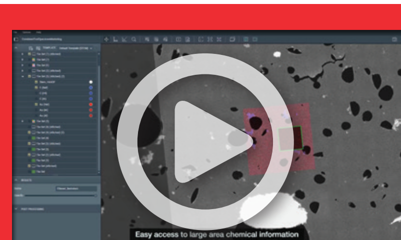
Whether the grade of steel is old or new, the analysis of precipitates is key to understanding the effectiveness of the alloying and heat-treating processes. APW is specifically designed to facilitate this kind of crucial characterization.

Solution

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.

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.



Automated Particle Workflow in action

Duration 2:49

Find out more at **thermofisher.com/APW**