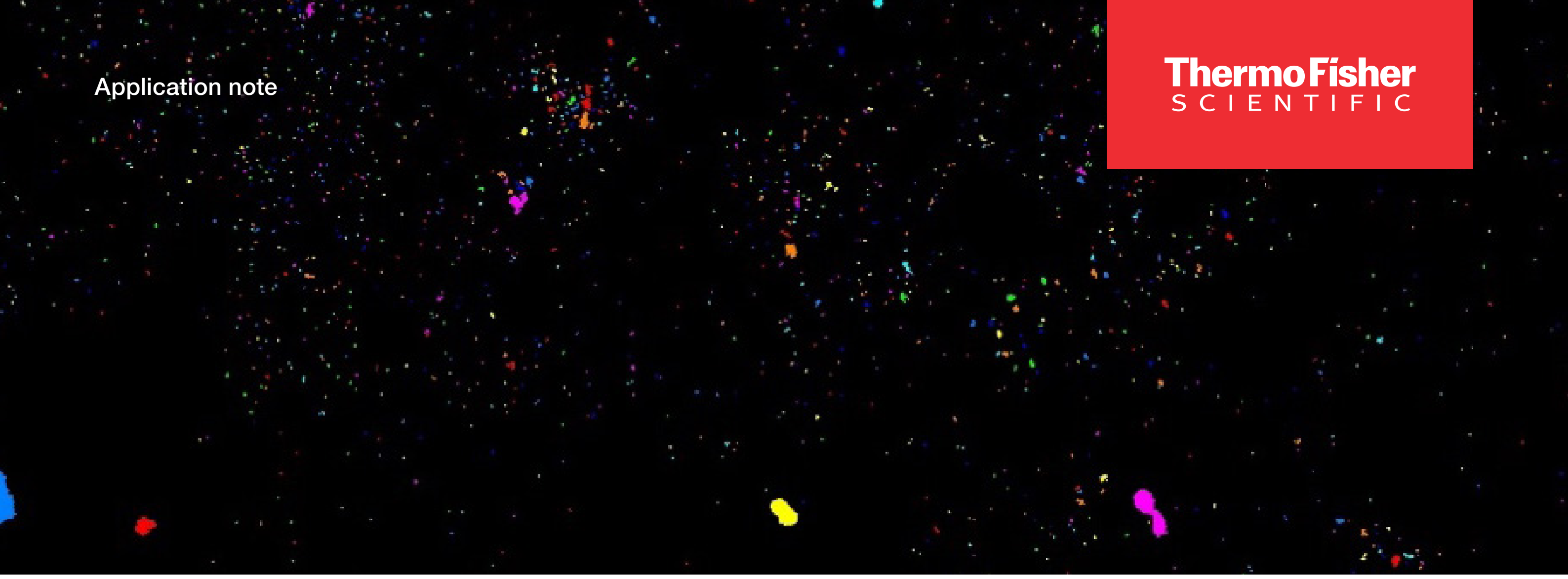
Application note



Microalloyed steel precipitate characterization by TEM

Evaluation of a hot-rolled, heat-treated steel structure

High-strength low-alloy (HSLA) steels are used in automotive, structural, and other applications since they can obtain more favorable strength and ductility properties over conventional grades of carbon steel.

Small amounts of elements such as niobium, titanium, and vanadium are employed to promote a finer austenite grain size during hot rolling (Table 1). A finer grain size is a key component to achieving steel with higher strength. The microalloy additions will react with carbon and nitrogen to form carbides, nitrides, or carbonitride precipitates. If these nanosized precipitates are permitted to coarsen, their ability to minimize austenite grain growth will decrease, along with their positive effect on hardening.

HSLA steels are produced by the Tata Steel Port Talbot Works, UK, using the blast furnace, oxygen steelmaking, and slab casting process route. The hot rolling process reheats slabs to approximately 1,200°C, then reduces their thickness to about 3 mm. Water cooling is applied between the hot mill and the coiler to control the grain size as the steel transforms from an austenitic to ferritic structure (Figure 1). The two samples in this study had a difference in ultimate tensile strength (688 MPa and 608 MPa, respectively), which needs to be understood.

Methods and results

An imaging study was initiated to correlate precipitate distribution with ultimate tensile strength on samples of hot-rolled, heat-treated coils. One sample was taken from the head of a coil; the second sample was taken from the middle of the same coil. The Thermo Scientific™ Helios™ 5 UX DualBeam (focused ion beam scanning electron microscope [FIB-SEM]) was used to prepare transmission electron microscopy (TEM) lamellae and to conduct preliminary analysis with S/TEM-in-SEM imaging.

Networks of dislocations and coarse precipitates were observed using this technique (Figure 2), but varying contrast made quantification challenging.

TEM analysis was performed on the lamellae using the Thermo Scientific Talos™ F200X G2 TEM. STEM and energy dispersive X-ray spectroscopy (EDS) imaging were used to search for small precipitates (Figure 3). EDS mapping more successfully identified precipitates in the ~90 nm thick lamellae than S/TEM-in-SEM imaging.

Elements	C	Mn	Si	N	Nb	Ti	P	Excess N
Weight (%)	0.067	1.32	0.208	0.0050	0.42	0.016	0.02	0.003

Table 1. Composition of Tata Steel Port Talbot Work's S450 grade.

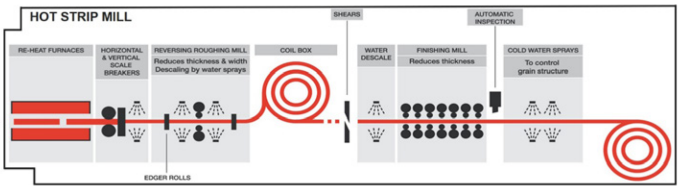


Figure 1. Schematic diagram of Tata Steel Port *Talbot Work's hot rolling process.* Steel is passed through a reversing roughing mill and a finishing mill to reduce thickness. Cold-water sprays are used to control grain structure. *Image courtesy of Tata Steel Europe Ltd., London, UK.*

Following manual TEM imaging, wide areas were characterized with the Thermo Scientific Automated Particle Workflow (APW) in overnight scans (Figure 4). This system utilizes Thermo Scientific Maps™ and Velox™ Software to collect STEM images and EDS maps in a tile-by-tile manner across the sample. As each tile is captured, the EDS map is instantly characterized by user-defined criteria in the Thermo Scientific Avizo2D Software.

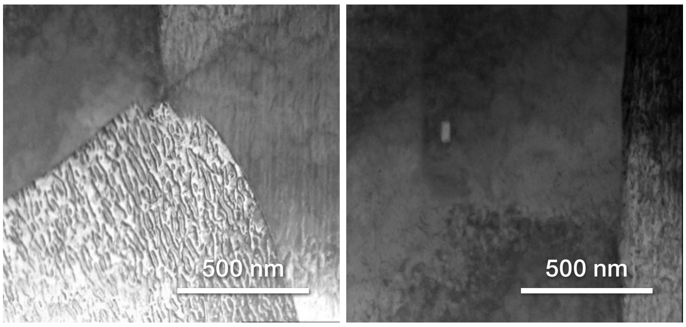


Figure 2. S/TEM-in-SEM imaging of (left) dislocation networks and (right) 100 nm precipitate.

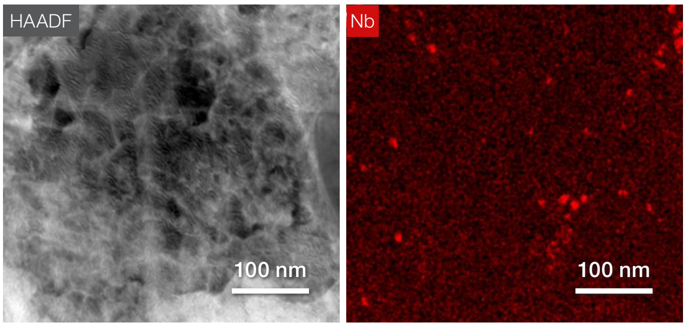


Figure 3. Imaging and mapping of the same regions in a steel lamella: (left) STEM mode imaging and (right) EDS mapping.

A comparison of two different samples from the same coil (Figures 5-6) shows a marked difference in precipitate size and number density (count/µm^2). The head of the coil has a higher precipitate density (118/µm^2) than the coil middle (85/µm^2); the average precipitate size is smaller in the coil head (9.0 nm) than in the coil middle (12.4 nm).

Conclusions

The APW characterized the niobium carbide precipitate size and number density for two samples from the same coil. The higher number and smaller precipitate size in the coil head explained the much higher ultimate tensile strength of the product. This process evaluation guided Tata Steel Port Talbot to choose their hot rolling cooling pattern to achieve the desired properties determined by these precipitates.

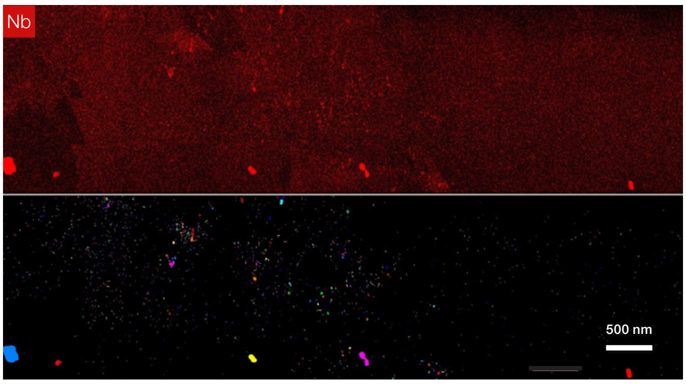


Figure 4. Scan of 13.5 µm^2 area. (top) EDS map of niobium and corresponding (bottom) APW characterization of nanoparticles. APW segments different particles by changing the color.

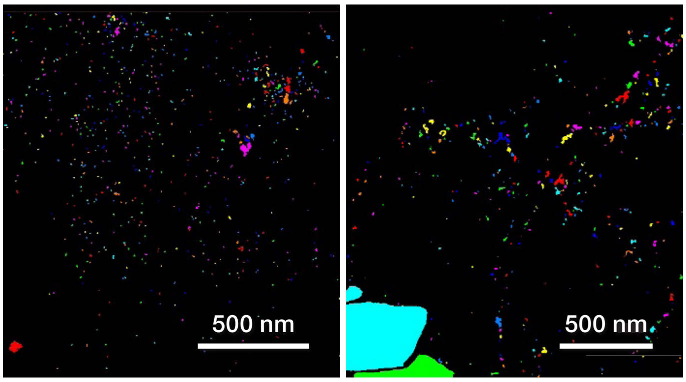


Figure 5. APW was used to produce these close-ups of Nb particle maps for the (left) coil head and (right) coil middle steel lamellae.

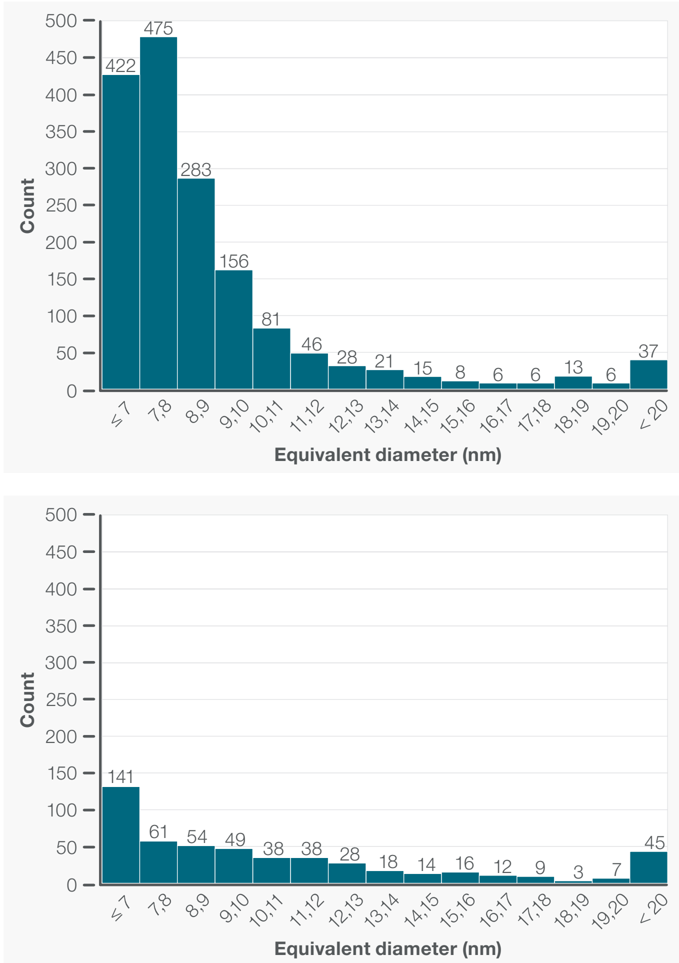
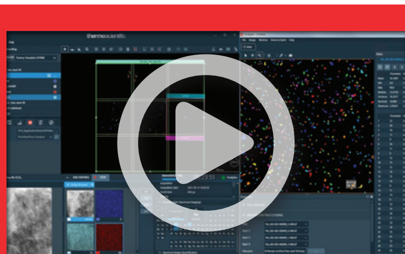


Figure 6. Histogram of particle sizes for the (top) coil head and (bottom) coil middle.



Automated Particle Workflow on HSLA steel lamellae

Duration 1:05

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