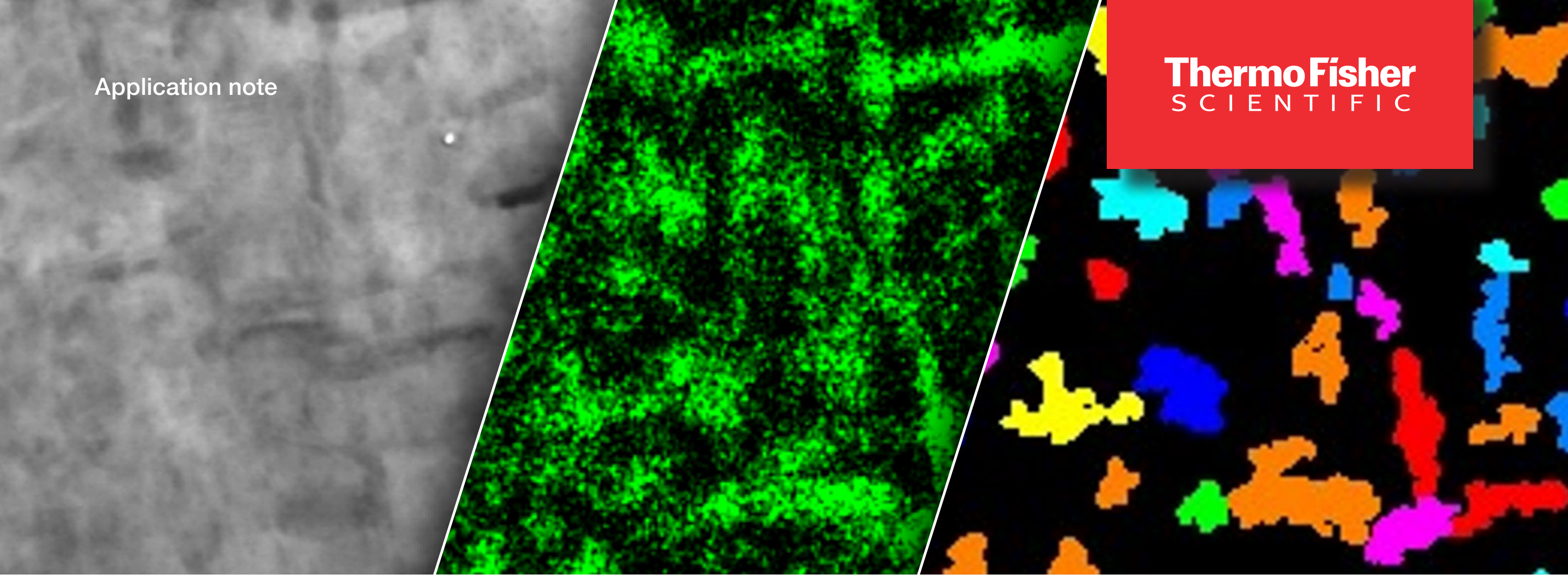

Application note

Micro- and nano-scale analysis of passivated stainless-steel landing gear with XPS, SEM, and TEM

Introduction

The aerospace industry has developed many high-strength, high-toughness alloys for demanding landing gear components such as hydraulic cylinders, pins, and axles. Several of these alloys are in the AISI 4340 carbon steel family, which have less than 3 wt% (Ni + Cr) and therefore require coatings to prevent oxidation. Historically, these parts have been coated with cadmium or chromium, which provide superior oxidation and abrasion resistance but can negatively impact the environment. Today, there is a push for alternative solutions that avoid these hazardous metals, such as high-velocity oxygen-fuel (HVOF) thermal spray coatings on carbon steel or stainless steel. This application note describes the surface chemistry and microstructure of a GKN Aerospace stainless-steel landing gear cylinder as a complete replacement for coated carbon steel.

Figure 1. Example of an Airbus A340-600 landing gear.

This alloy is produced by vacuum induction melting and vacuum arc re-melting. The final composition was determined using a Thermo Scientific™ ARL™ iSpark 8860 Optical Emission Spectrometer (Table 1). Having light elements such as carbon, nitrogen, and sulfur below 25 ppm suggests that the refining conditions are highly controlled for a specific purpose. This steel is also deoxidized by aluminum, yielding a dissolved oxygen content around 2 ppm.

The most unique part of this alloy is the >1.5 wt% titanium content. This elevated titanium wt% is likely used for precipitation hardening, producing the intermetallic η-Ni_3Ti phase. Titanium will, however, react with carbon, nitrogen, sulfur, or oxygen in steel if given the opportunity, so every measure has been taken to reduce their presence and minimize the formation of TiC, TiN, TiS, or TiO_2.

C (ppm)	N (ppm)	S (ppm)	Cr	Ni	Ti	Mo	Al	Fe
24	18	6	11.6	11.2	1.6	1.0	0.05	~74 (Bal.)

Table 1. Composition of stainless steel landing gear by weight percentage, unless otherwise noted.

Methods

This work was initially motivated by a surface staining issue that needed to be remedied. A secondary electron image taken with the Thermo Scientific™ Axia™ ChemiSEM shows machining and grinding lines, along with a hint of discoloration (Figure 2). Thermo Scientific™ ChemiSEM™ Technology is integrated, always-on energy dispersive spectroscopy (EDS) in a scanning electron microscope (SEM) that can clearly visualize minute fractures, inclusions, and particles. Here, the fractured titanium nitride particles on the surface were readily identified (Figure 3), but the identity of the surface stain could not be confirmed.

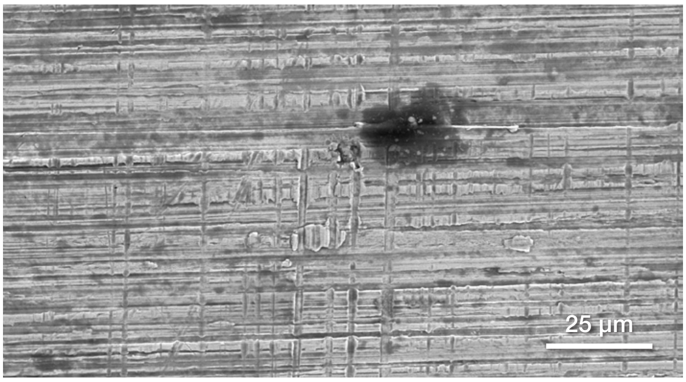

Figure 2. SEM image of the stained landing gear surface.

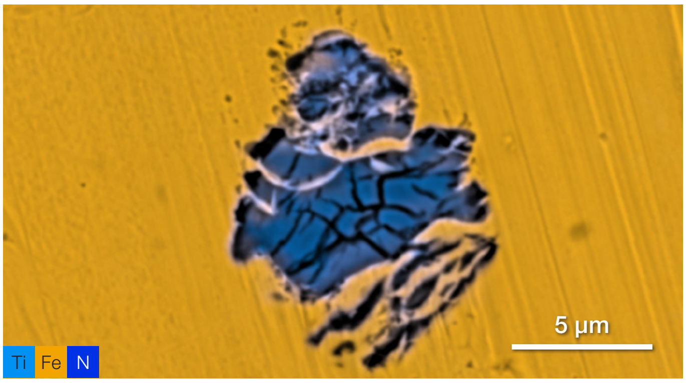

Figure 3. ChemiSEM image of a fractured titanium nitride particle in the steel surface.

Since EDS interacts with the entire top micrometer of a sample, a more sensitive method was needed to identify the stain. X-ray photoelectron spectroscopy (XPS) quantitatively analyzes the top 10 nanometers of a material, with the added capability of depth profiling. The Thermo Scientific™ Nexsa™ G2 Surface Analysis System was used to profile the top 80 nm of both a clean and stained landing gear cylinder (Figure 4). The clean sample surface showed chromium and oxygen concentrated in the top 20 nm, with metallic iron, chromium (and nickel) underneath. This indicates that the sample was properly passivated (i.e., chemically protected by forming Cr_2O_3 at the surface) whereas the stained sample was not. The latter showed oxidized iron and chromium through the full 80-nm depth. As a result of this analysis, surface cleaning and passivation processes were revised to prevent any staining on future landing gear cylinders.

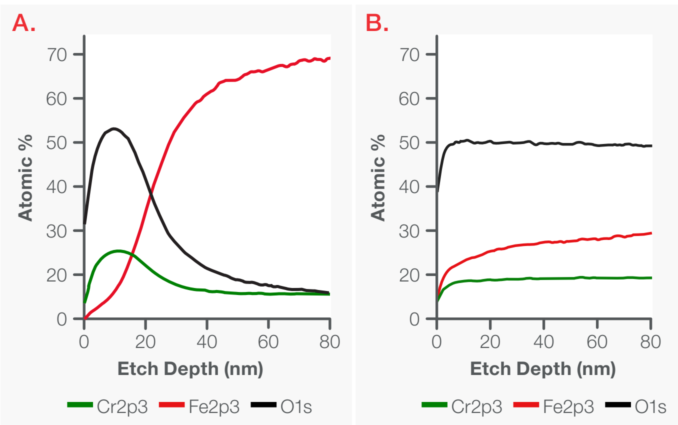

Figure 4. Surface analysis of the clean (A) and stained (B) landing gear cylinders.

Further detail can be revealed by looking at representative binding energies of different chemical states. The sum of all titanium (Ti_2p) spectra from the region surrounding the passivation layer is shown in Figure 5. The area under each curve accounts for the intensity of each specific state. This confirms that titanium was oxidized along with the chromium in the passivation process, and that TiN is present, as was indicated by the SEM results.

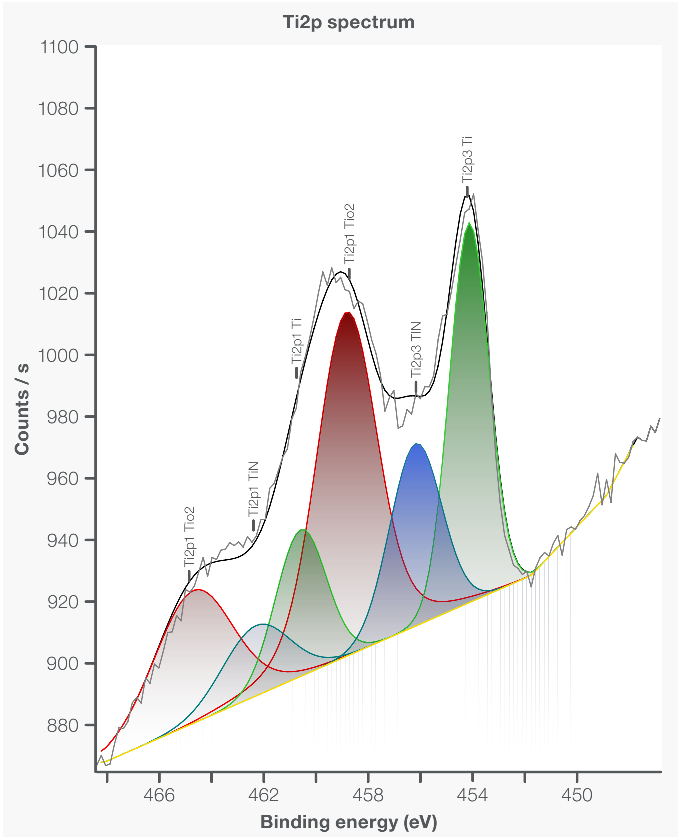

Figure 5. Surface analysis spectra of the clean landing gear cylinder, showing TiO_2 (red), TiN (blue), and metallic titanium (green).

The presence of fractured TiN particles on the surface led to further evaluation of the microscale inclusions in the cross section (with SEM), as well as the nanoscale precipitates that produce desirable properties (with TEM). The Thermo Scientific™ Phenom™ ParticleX™ Steel Desktop SEM can be used for manual imaging or for automated feature analysis of particles down to one micron or smaller. Backscattered electron (BSE) imaging revealed contrast based on atomic weight. (I.e., the large TiN inclusion shown in Figure 6 is darker than the stainless steel background.) Neither this inclusion nor any other in the cross section showed any signs of cracking or deformation. This implies that surface particle cracking is the result of the machining or grinding process.

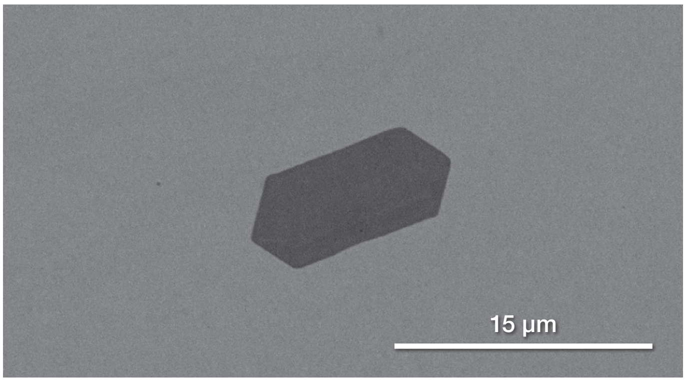

Figure 6. SEM image of a coarse TiN inclusion.

In addition to TiN, a population of TiS was also identified throughout the cross section. Automated scans in the ParticleX Steel Desktop SEM characterized all inclusions larger than 1 µm over a 50 mm^2 area for both the clean and stained samples. The inclusion count, size, shape, and chemistry were nearly identical for both samples, where the combined TiN + TiS inclusion area fractions were 2.278 x 10-4 and 2.275 x 10-4 for the clean and stained samples, respectively. Examples of the automatically captured images are shown in Figure 7, and a Ti-N-S ternary diagram reveals the inclusion size and composition distribution for the clean sample (Figure 8). By contrast, the surface did not show any TiS inclusions, as sulfides are readily dissolved in acid during passivation.

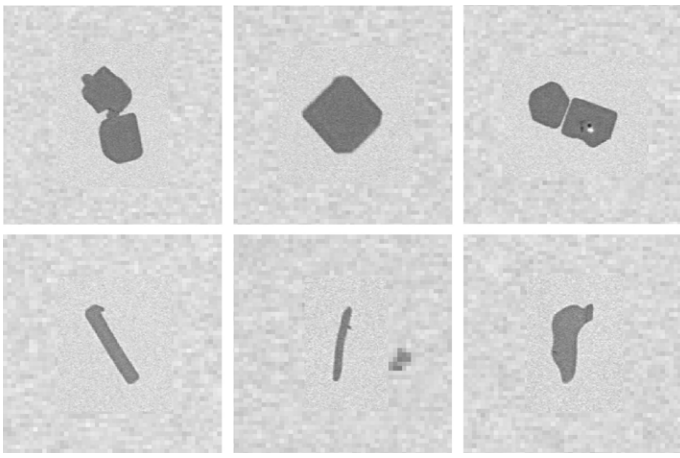

Figure 7. Automated SEM imaging showing the largest TiN (D_{MAX} ≥ 15 µm, top) and TiS (D_{MAX} ≥ 12 µm, bottom) inclusions.

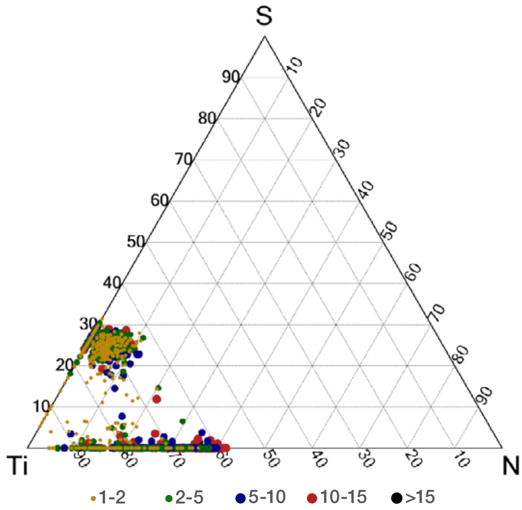

Figure 8. Ti-N-S ternary diagram of inclusions in the clean landing gear cylinder, measured with automated SEM imaging.

Finally, the nanostructure was analyzed with TEM in search of important η-Ni3Ti precipitates. A lamella was prepared with the Thermo Scientific™ Helios™ 5 Laser Plasma Focused Ion Beam (PFIB). The lift-out chunk and the 50-nm thinned section are shown in Figure 9. Bright-field scanning transmission electron microscopy (STEM) imaging and EDS were conducted in the Thermo Scientific™ Talos™ F200X TEM, with representative precipitate images shown in Figure 10.

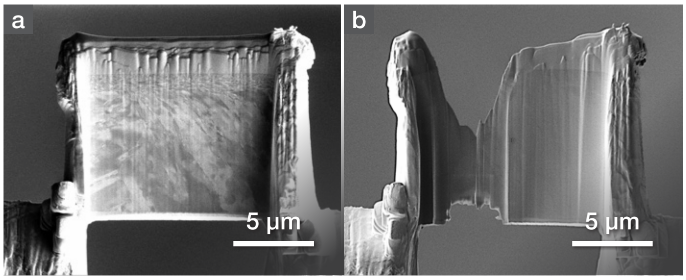

Figure 9. FIB lift-out lamella (A) and thinned section (B).

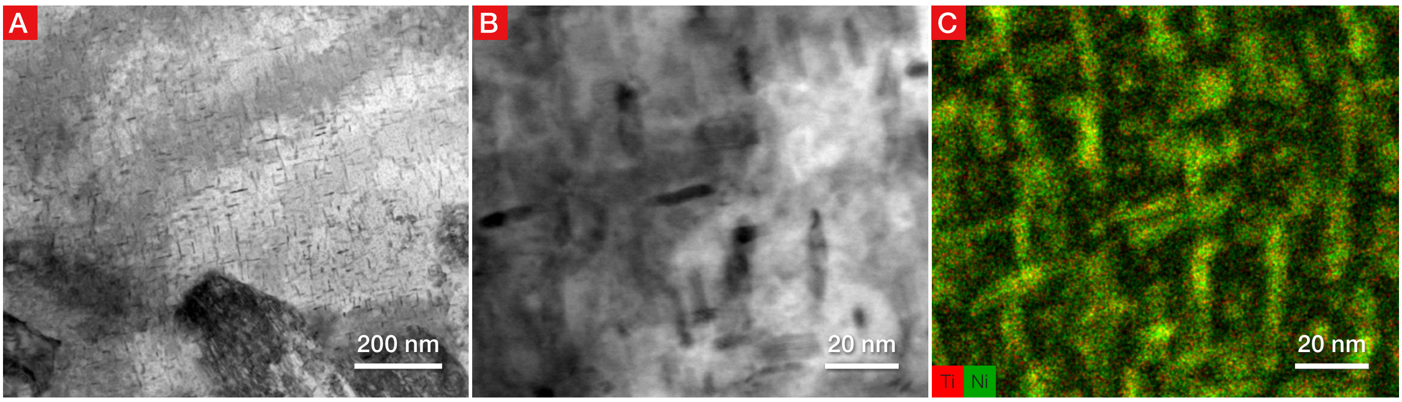

Figure 10. Bright-field STEM images (left, mag = 94 kX; center, mag = 780 kX) with overlapping EDS maps of Ni and Ti (right, mag = 780 kX).

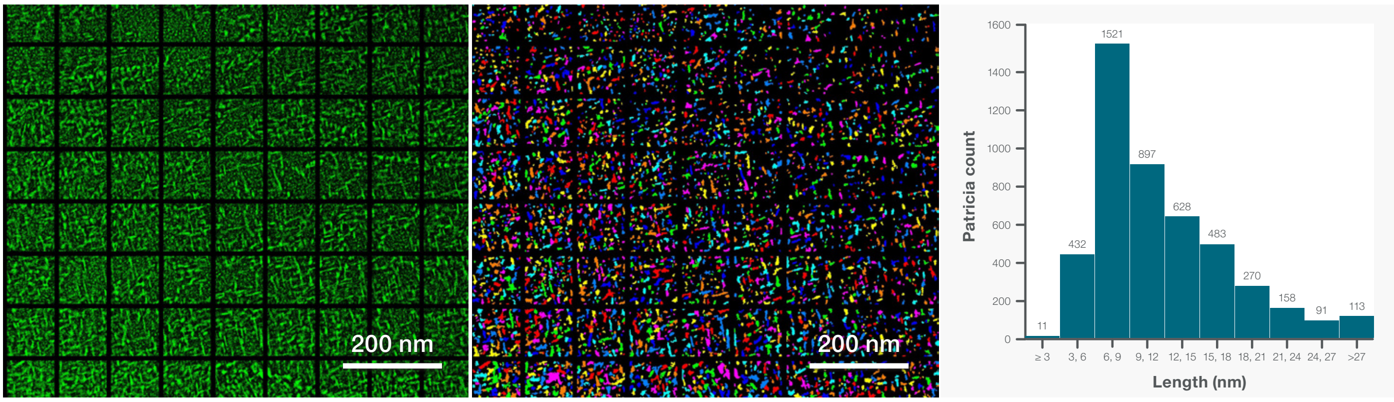

Figure 11. Wide-area automated TEM imaging, EDS mapping, and particle sizing.

Results

Integrated within the Talos F200X TEM is the Thermo Scientific™ Automated Particle Workflow, or APW, which allows for unattended imaging and particle sizing in the TEM. High-resolution images were captured tile-by-tile over a total area of 1 μm^2. For each tile, STEM images and any desired EDS maps were collected. A chosen EDS map, in this case nickel, was then automatically characterized for particle size to represent the η-Ni3Ti precipitates. The nickel EDS map, segmented particle map, and size histogram are shown in Figure 11. The 4,000+ particles measured have an average length of 12 nm. This result confirms the particle size distribution, which is responsible for precipitation hardening. Note that any future changes in composition or heat treatment can be tested in the same way for comparison.

Conclusions

Stainless-steel landing gear cylinders were studied from several different perspectives. First, passivation layers and surface staining were characterized by XPS. Next, surface inclusions (broken TiN) and internal inclusions (unbroken TiN and TiS) were quantified by SEM. Finally, nanoscale precipitates were identified and quantified with TEM EDS and the Automated Particle Workflow. Evaluation by XPS, SEM, and TEM provided a better understanding of the base alloy, which led to adjustments in the parts production process. These revisions increased the viability of stainless steel as an environmentally conscious choice to replace coated carbon steel.

Stainless landing gear

in Axia ChemiSEM

Duration 1:22

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