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APPLICATION NOTE

Advances in Compositional Analysis Using Analytical S/TEM

Characterization of superalloy material

The ultimate performance of superalloy materials is influenced by the finest distribution of the elements comprising the material. The ability to perform nanoscale compositional characterization, both as 2D chemical maps and as 3D volume rendering of structure, is critical to achieving full understanding for predicting the performance of the material.

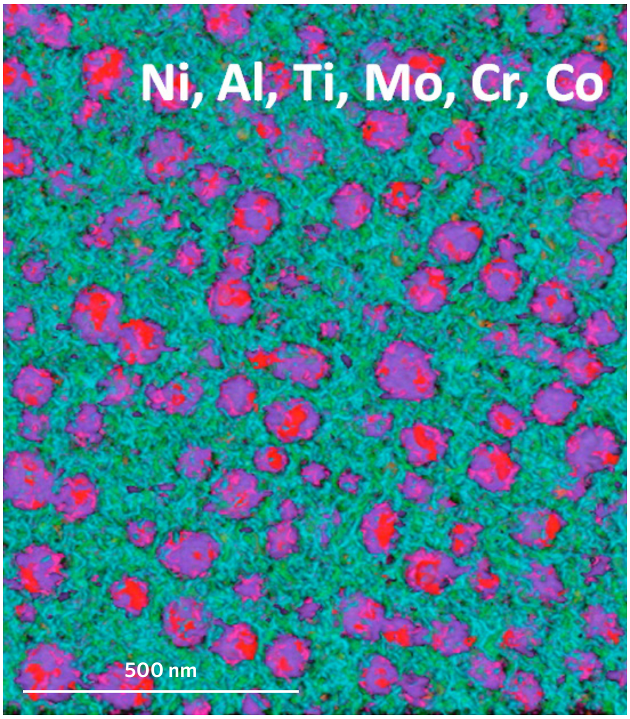

Figure 1a. Compositional map of the Ni based superalloy sample: 2D EDS multi-element compositional map of 512 × 512 pixels. Acquired in <5min.

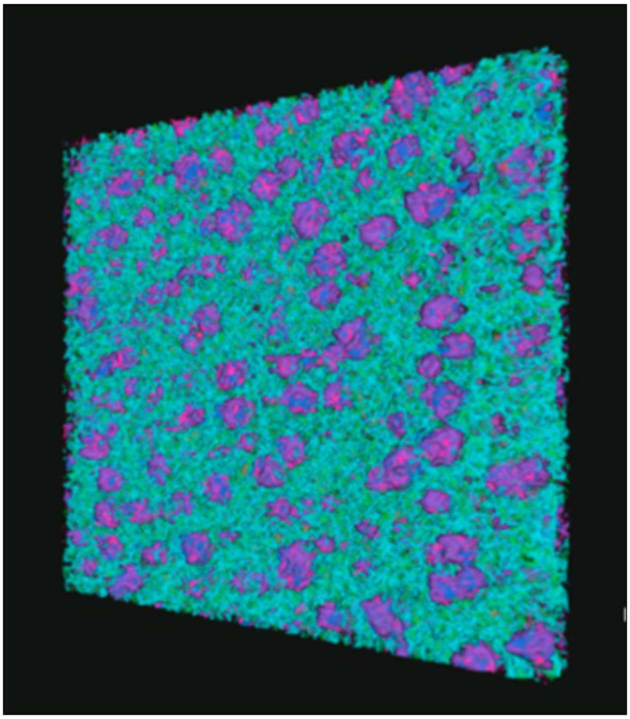

Figure. 1 b. 3D EDS multi-element compositional map, tilt range +/-70 degrees; 512 × 512 pixels at each tilt – total acquisition time <3h.

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High performance materials in extreme conditions

Modern engineering applications strive for new materials with high performance in extreme conditions such as elevated temperatures, chemically-aggressive environments and high mechanical loads. The metal superalloys discovered in the last century are a focus of continuous innovation and improvement for such applications. In today's engineering, superalloys most typically have application in aerospace, jet and high performance engines, and in turbine designs, among other applications for the material.

Superalloys have complex compositional structure which typically constitutes a single crystal Ni- or Co-based γ type matrix having fcc symmetry blended with γ' type precipitates composed typically of Ni, Ti, Al and Mo which have cuboidal structure. There are many other metallic elements that are added to a superalloy to tailor its properties to a specific application, for example adding Cr for corrosion resistance or Nb for increased creep resistance at elevated temperatures. Such variety of elements present together in a single material drives the need to have precise, detailed compositional and structural analysis to verify that composition is correct for a desired application. As a result, high speed elemental analysis and precise quantification are predominant requirements in characterization of these materials. The complexity of superalloy materials also drives the requirement for knowledge of 3D spatial distribution of every individual elemental constituent, in addition to the 2D elemental maps used for compositional analysis with EDS/EELS. Such 3D reconstructed elemental mapping is shown based on the tilt series where superalloy lamella was mapped at various tilt angles to construct a 3D volume with compositional map of all elements in the sample. (Figure 1 a, b)

In this application example, we show the importance of the fast compositional characterization based on the EDS /EELS methods together with the flexible S/TEM and HR-TEM imaging used to study the structure of these composite materials. To study the superalloy material we employed the new 200 kV analytical FEG S/TEM, Thermo Scientific™ Talos™ System, which is equipped with fast EDS/EELS, and multi-mode and dynamic HR-S/TEM.

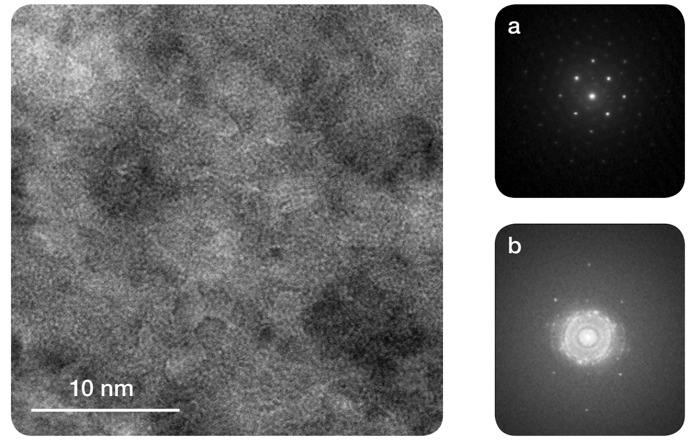

Figure 2. HR-TEM image of superalloy, 4k × 4k pixels; **a)** SAED diffractogram; **b)** FFT diffractogram from the HRTEM image.

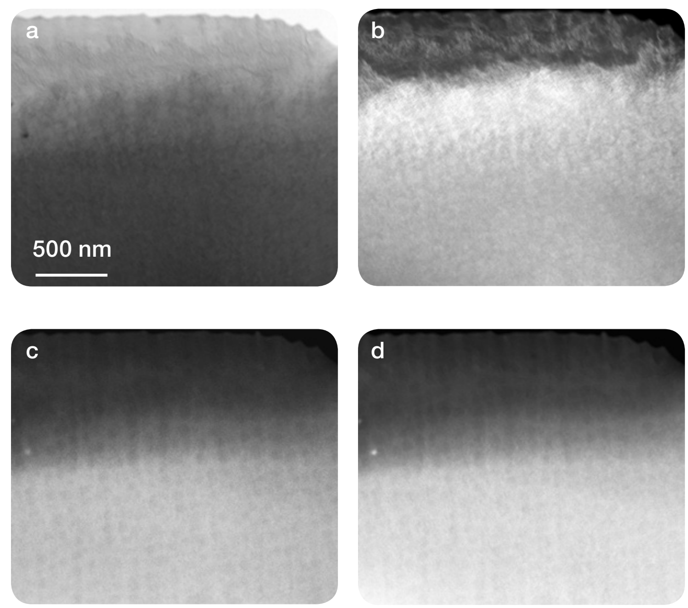

Figure 3. S/TEM images acquired simultaneously from 4 different STEM detectors: a) Bright field STEM; b) dark field STEM; c, d) high angle dark field.

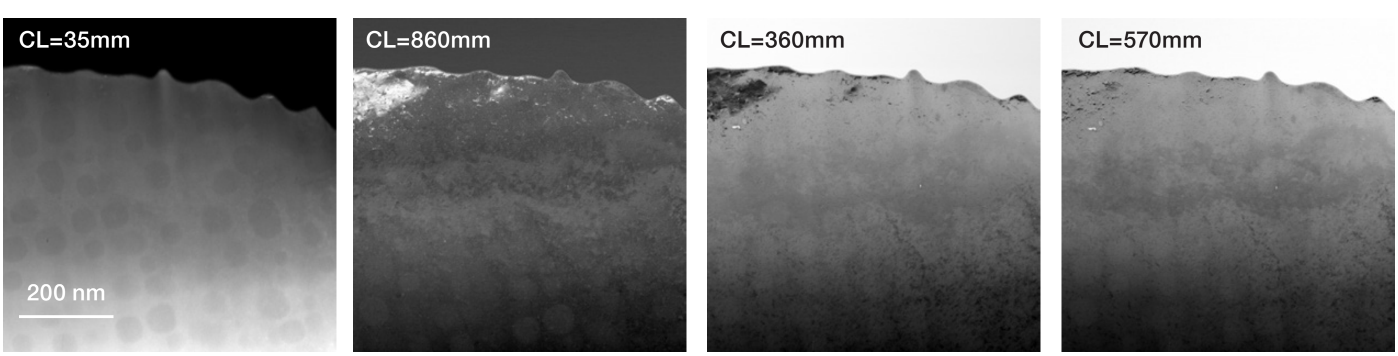

Figure 4. Series of S/TEM images with HAADF STEM detector at selected camera length (CL) values.

Structural characterization with HR-TEM (Figure 2) and S/TEM (Figure 3) reveals the morphology of the superalloy sample and presence of the γ' precipitates, which are particularly pronounced in DF-S/TEM images (Figure 3- 4). The advantage of imaging the sample in STEM with 4 parallel channels and using multiple STEM detectors is well illustrated in Figure 3, where the sample is imaged with electrons scattered by the sample at various angles. The flexibility of such multi-detector STEM imaging engine is enhanced with available choice of 25 settings in camera length which can be changed in the 14mm-5.7m range and corresponding 1-200mrad scattering angle range. (Figure 4)

The compositional analysis was performed with EDS and EELS with both techniques used in STEM-SI acquisition mode. The elemental maps together with the multi-element color compositional maps demonstrate good accordance with respect to the element distribution. The γ' precipitate's elemental structure is confirmed to have Ni-Ti-Al-Mo composition, while the γ matrix is built on the combination of Co and Ni with the addition of Cr. The uniform presence of oxygen revealed by both EDS and EELS is conjectured to be caused by the aging and oxidation of the sample.

The capability of extracting the 3D distribution in space for every constituent element is demonstrated with attached 3D EDS maps shown for Ni and Ti (Figure 7). It is important to state that the high acquisition speed, fast STEM engine and symmetric design of the EDS system with a large solid angle on the Talos System were major contributors in successful 3D elemental mapping of this super alloy sample.

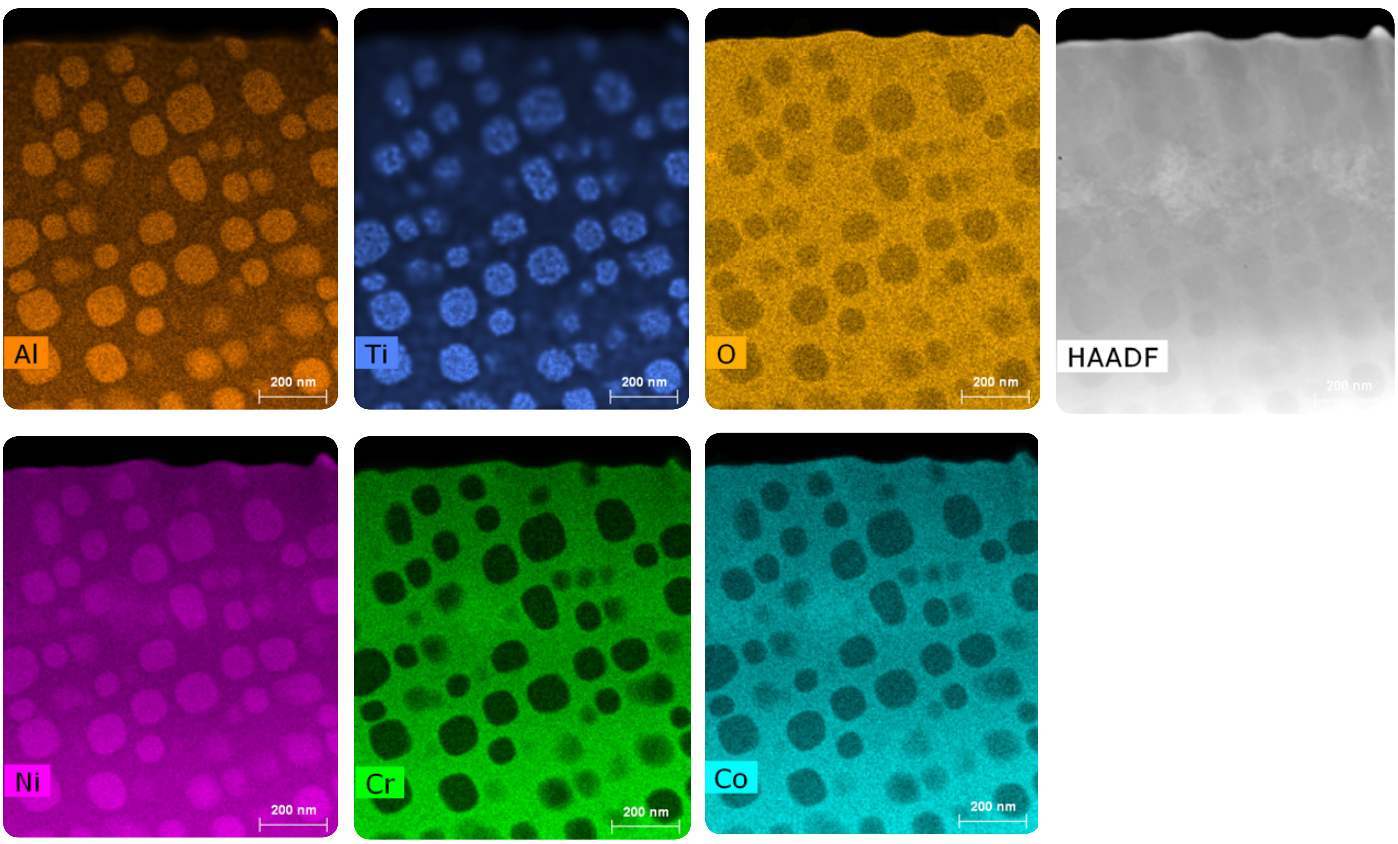

Figure 5. 2D EDS compositional maps of the elements acquired with Super-X system on Talos S/TEM.
Maps of 280 × 235px were acquired and quantified in <10 minutes.

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Talos System

The Thermo Scientific Talos System is a 200kV scanning/transmission electron microscope (S/TEM) that delivers fast, precise, quantitative characterization of nanomaterials in multiple dimensions. With innovative features designed to increase throughput, precision, and ease of use, the Talos System is ideal for advanced research and analysis across academic, government, and industrial research environments.

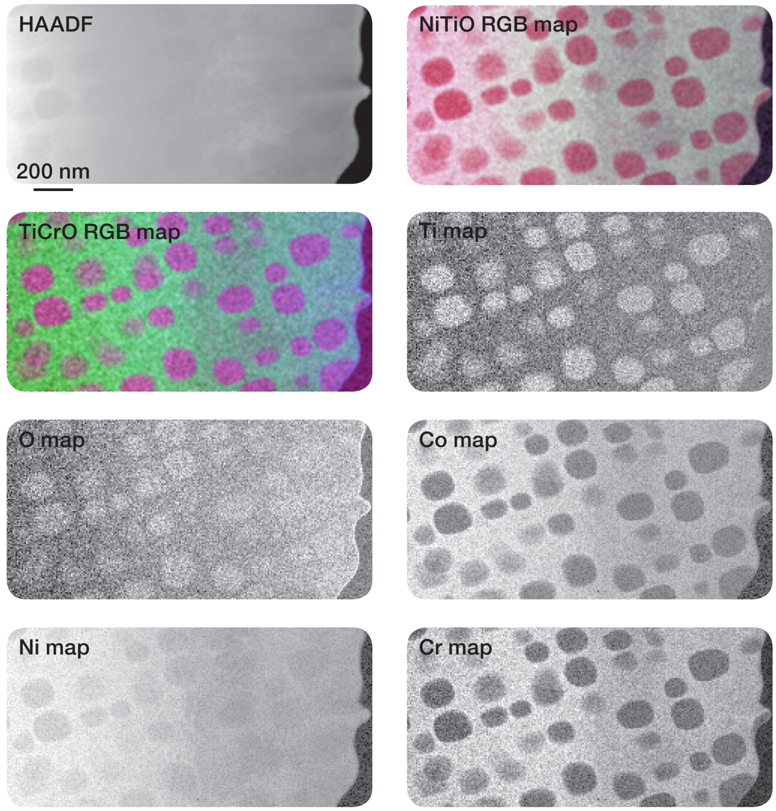

Figure 6. 2D EELS compositional maps acquired with Gatan EELS spectrometer. These maps of 280×235 px were acquired in 30 min. and post-processed separately.

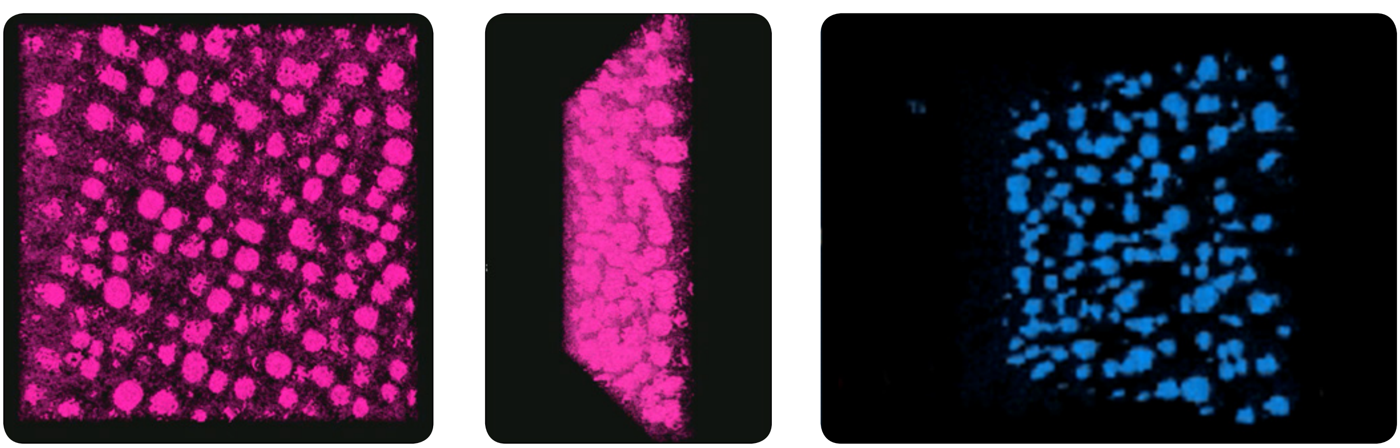

Figure 7. 2D-3D EDS maps showing spatial distribution of individual elements in super alloy lamella (1.2μm × 1.2μm × 0.1μm).

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