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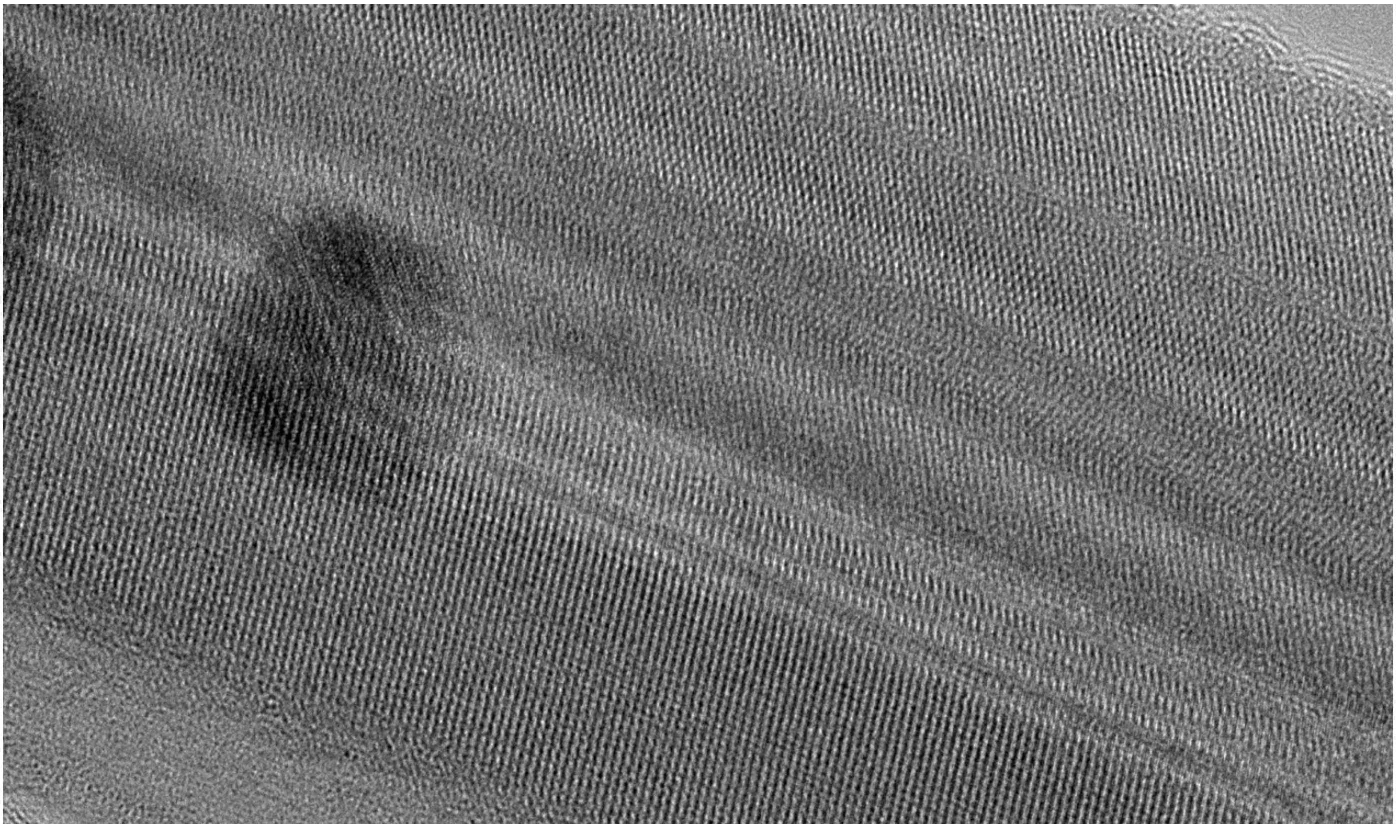
APPLICATION NOTE

Advances in Compositional Analysis using Analytical S/TEM

Characterization of Boron-Nickel composite nanowires

The ability to perform elemental characterization of nanomaterials, both at room temperature and during heating, provides important insight to structural and compositional changes. In this application brief, we demonstrate the use of energy dispersive X-ray spectroscopy (EDS) on a catalyst specimen during heating.



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Fast compositional characterization

Today's compositional analysis for *in situ* characterization of catalysts requires instrumentation with fast and quantitative data acquisition as well as dynamic high resolution imaging. This becomes particularly pronounced in the *in situ* experiments where the outcome of the measurements is dependent on the rapidly changeable parameters such as temperature, mechanical stresses or electric fields. For such dynamic experiments, the combination of flexibility in the sample imaging optimization, the ability to use different settings and modes, such as TEM and S/TEM, and the fast collection of the compositional data are indispensable for capturing the properties of nanomaterials.

In this application example, we show the importance of the fast compositional characterization provided by EDS together with the dynamic S/TEM and HR-TEM imaging to study the structure of boron-nickel (B-Ni) composite nanowires. These nanostructures combine refractory and conductive properties which are critical for future applications in the field of nano-electronics. To study these properties, we employ our newest generation 200kV analytical FEG S/TEM, the Thermo Scientific™ Talos™ System, which is equipped with our fast EDS system, dynamic HR-S/TEM and the MEMS-based heating device.

Structural characterization with high resolution transmission electron microscopy (HR-TEM) (Figure 1 a, b, c) and dark field scanning transmission electron microscopy (DF-S/TEM) (Figure 2) reveals the geometry and presence of the fragmented and continuous nickel crystalline rods constituting a core of the crystalline boron nanowires. This in particular is obvious in the high contrast DF-S/TEM images showing the Ni catalyst. High speed EDS elemental mapping confirms this in series of multi-element EDS maps. Ni core structures are verified by EDS and the presence of oxidized layer on the surface of the B nanowires is shown as well (Figure 3).

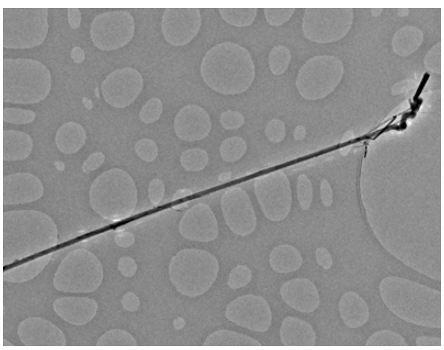


Figure 1a. Low magnification TEM image showing a geometry of B-Ni composite nanowire

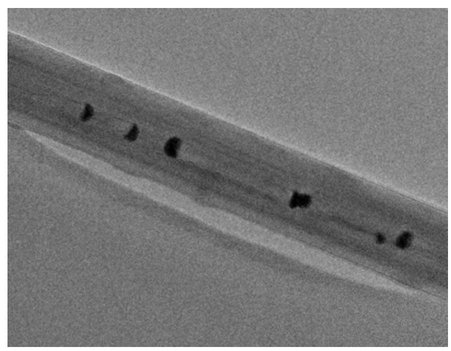


Figure 1b. TEM image showing the presence of Ni catalyst particles/fragments in Boron matrix

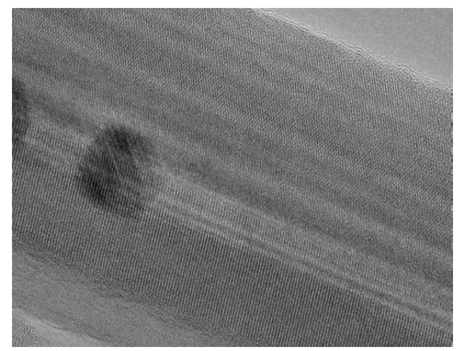


Figure 1c. HR-TEM image showing crystalline lattice of B shell and Ni nanocrystal

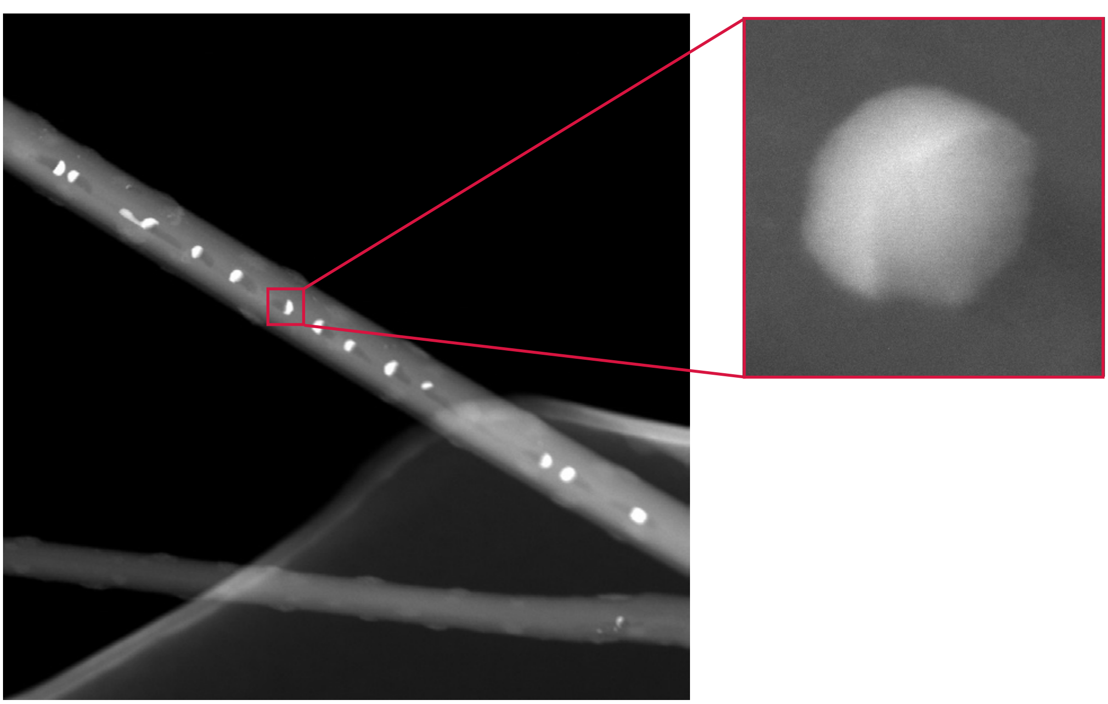


Figure 2. DF-STEM image revealing high contrast crystalline Ni fragments in Boron nanowire; Enlargement shows individual Ni crystal at higher magnification

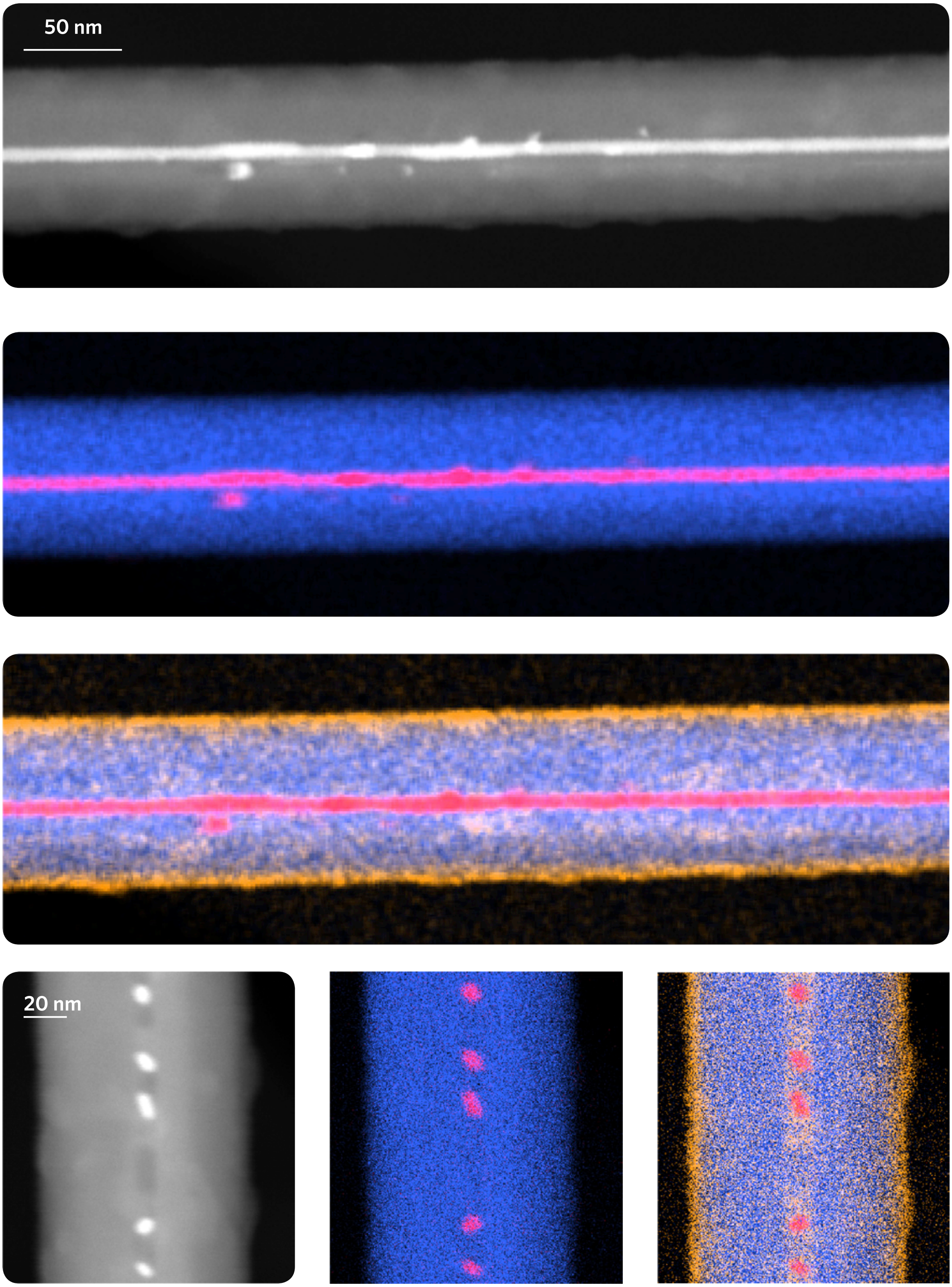
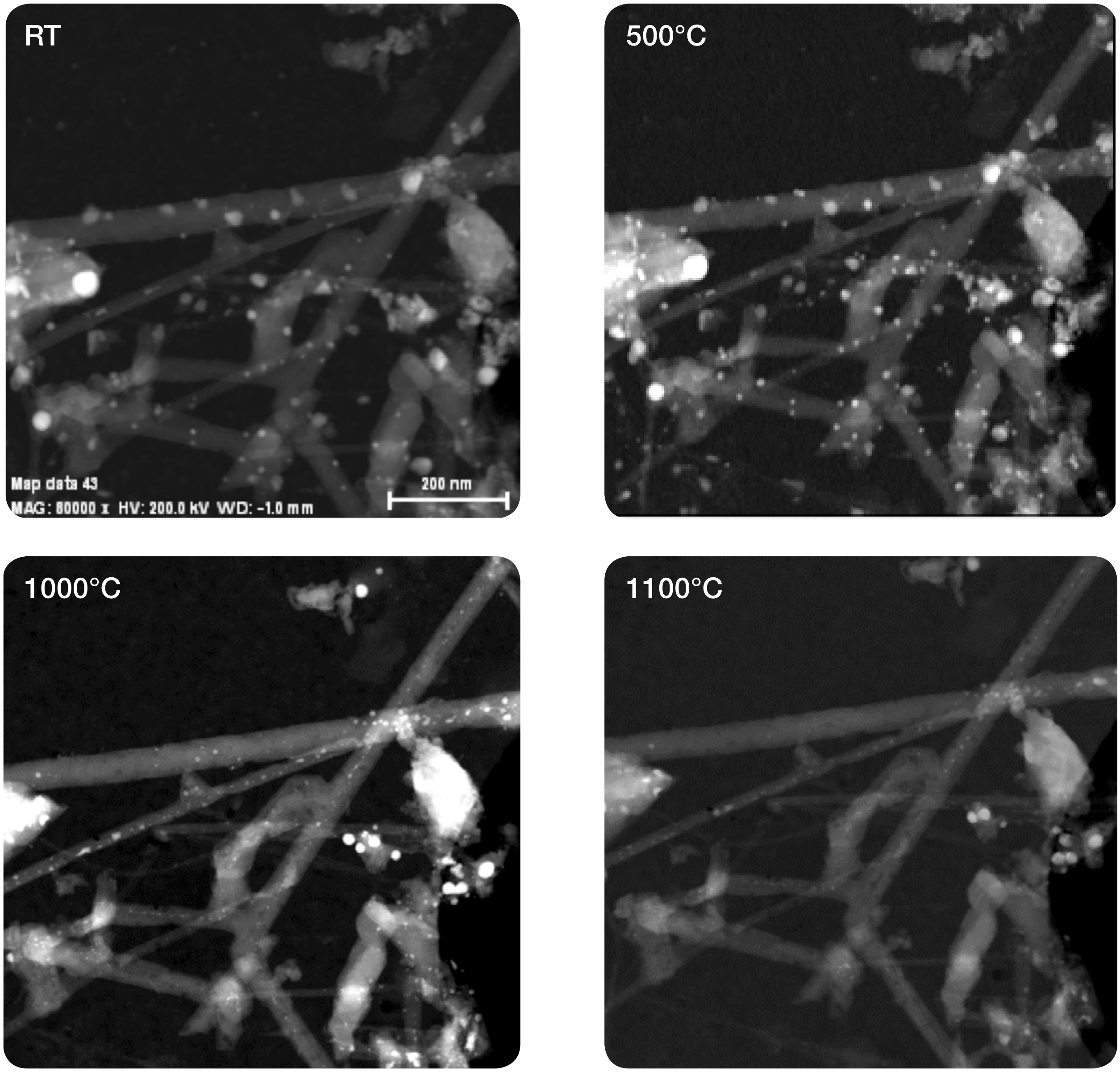


Figure 3. EDS compositional maps of 500 × 120px (top series) and 300 × 250px (bottom series) collected and quantified in 10 min. Light elements such as Boron, Carbon and Oxygen are mapped in the composite nanostructure



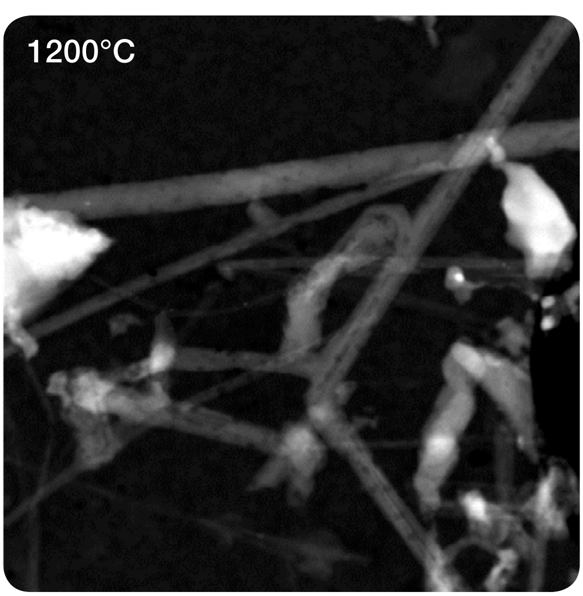


Figure 4. Series of DF-STEM images at room temperature (RT), 500˚C, 900˚C, 1100˚C and 1200˚C.

Figure 5a

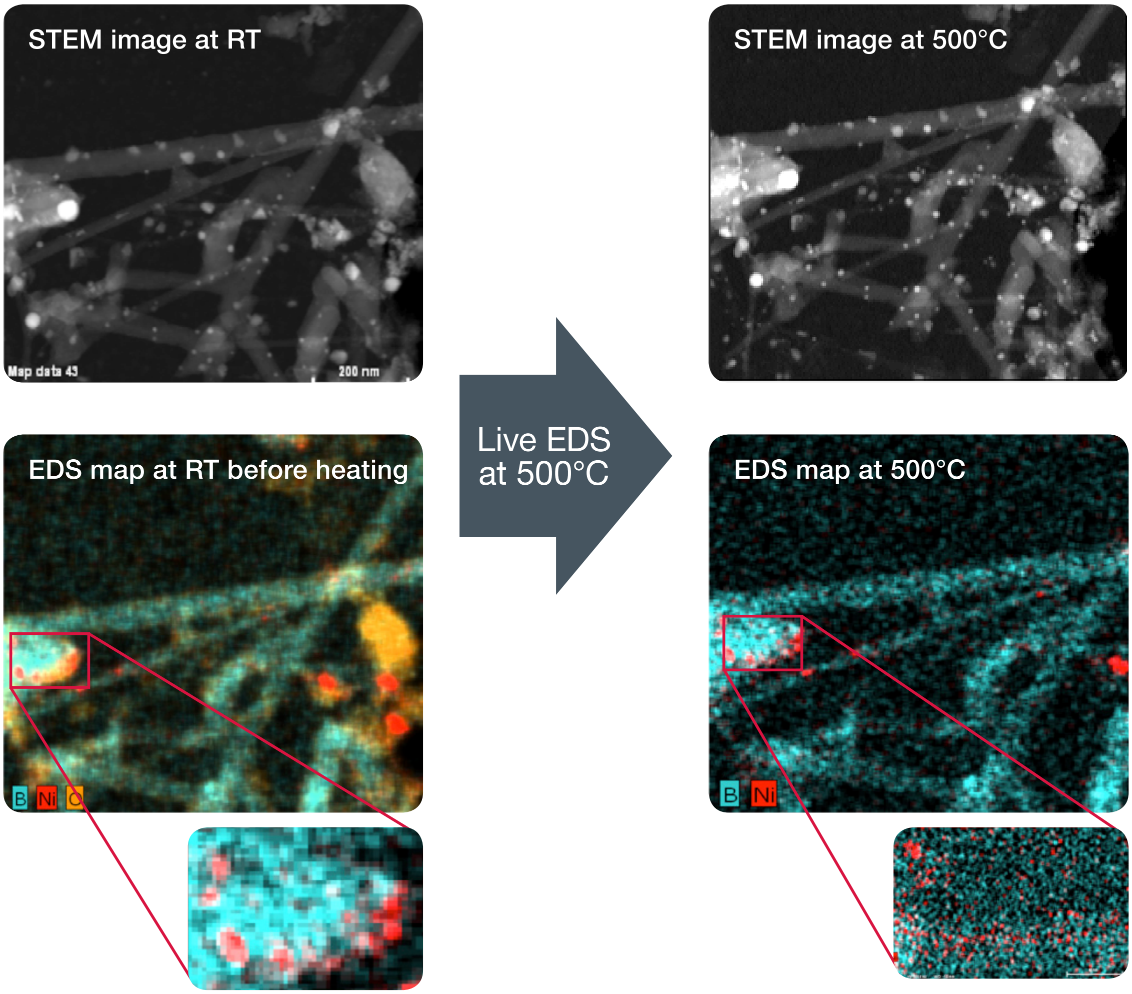
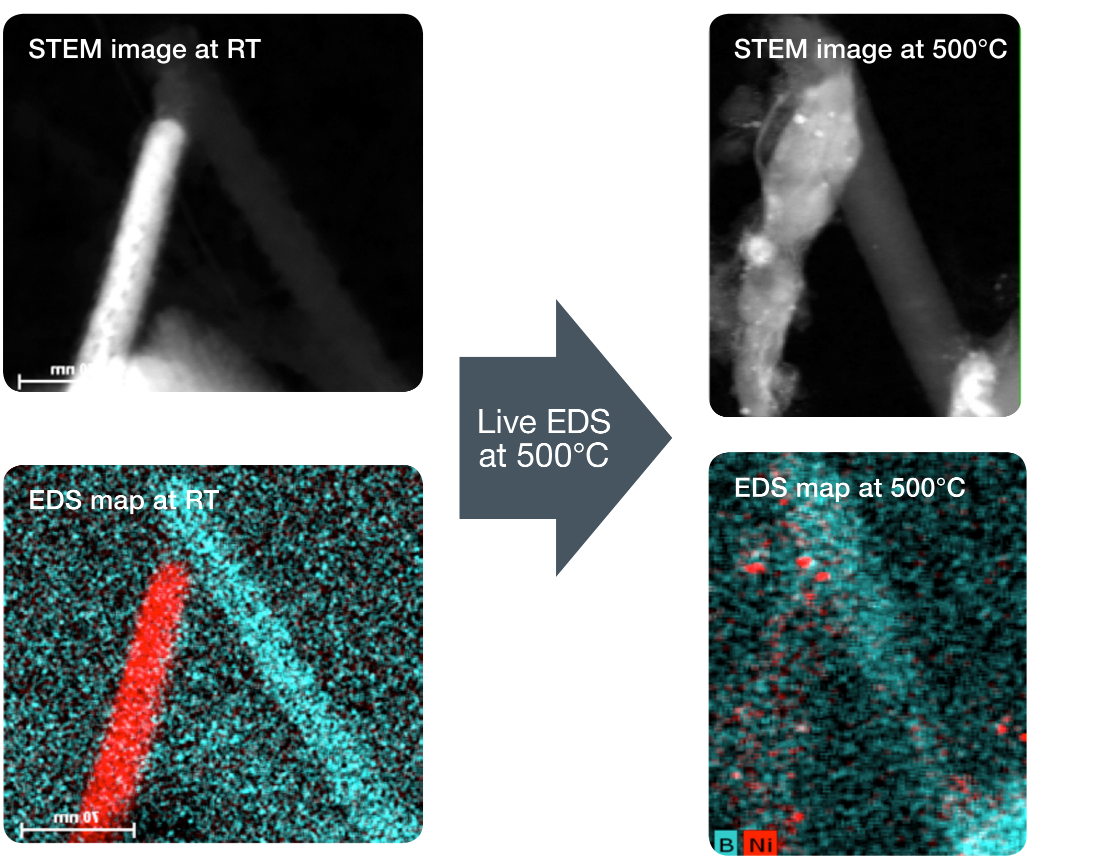


Figure 5b



The B-Ni composite nanowires were rapidly heated and the dynamic compositional analysis was conducted in parallel with S/TEM, with *in situ* observations at higher temperatures (Figure 4). We show that heating to 1100° C results in "extrusion" of Ni nanocrystals from boron solid and further heating to 1200° C confirms that Ni catalyst becomes extinct. EDS compositional mapping, HR-TEM and dynamic DF-S/TEM confirm these conclusions. The examples of the EDS maps acquired at 500° C show gradual changes of the composition with temperature (Figure 5 a, b) and the EDS maps collected after sample cooling to room temperature (RT) verify complete extinction of Ni from the nanowires. (Figure 6).

Figure 7 shows the composite nanowire structure (Figure 7a) and EDS map with a specific area selected (Figure 7b). The elemental composition was instantaneously calculated using new Velox™ analytical software, with the resulting EDS spectrum (Figure 7c) from the selected area of interest further quantifying the elemental composition.

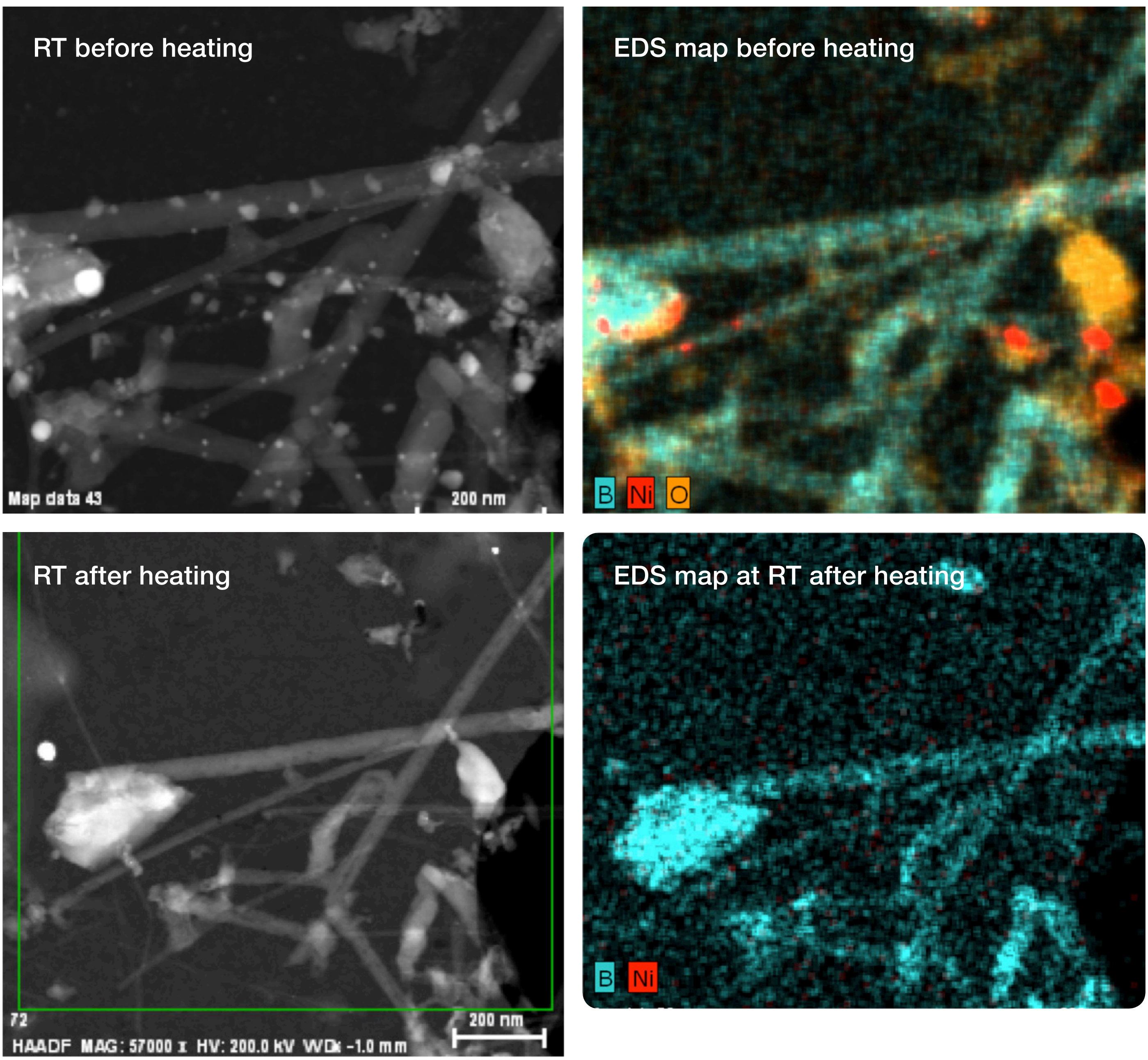


Figure 6. Corresponding EDS maps acquired at RT after cooling sample from 1200˚C

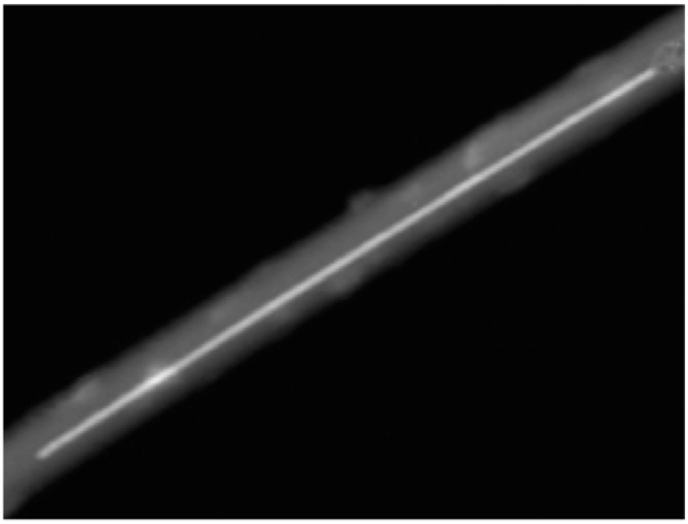


Figure 7a. DF-STEM image of B-Ni composite nanowire

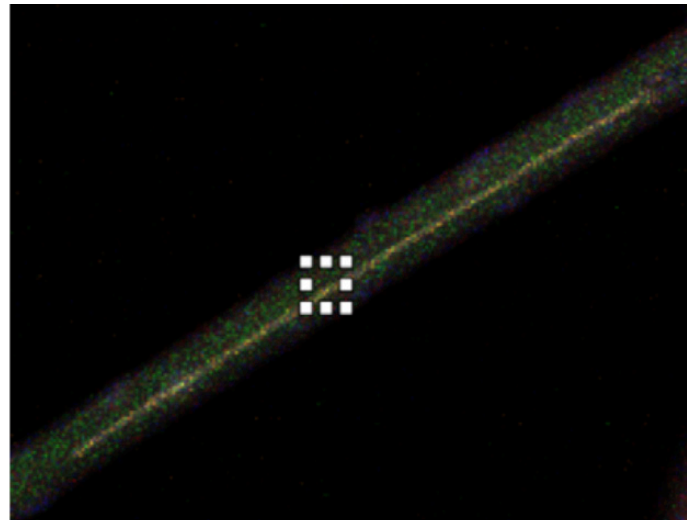


Figure 7b. B-Ni EDS map

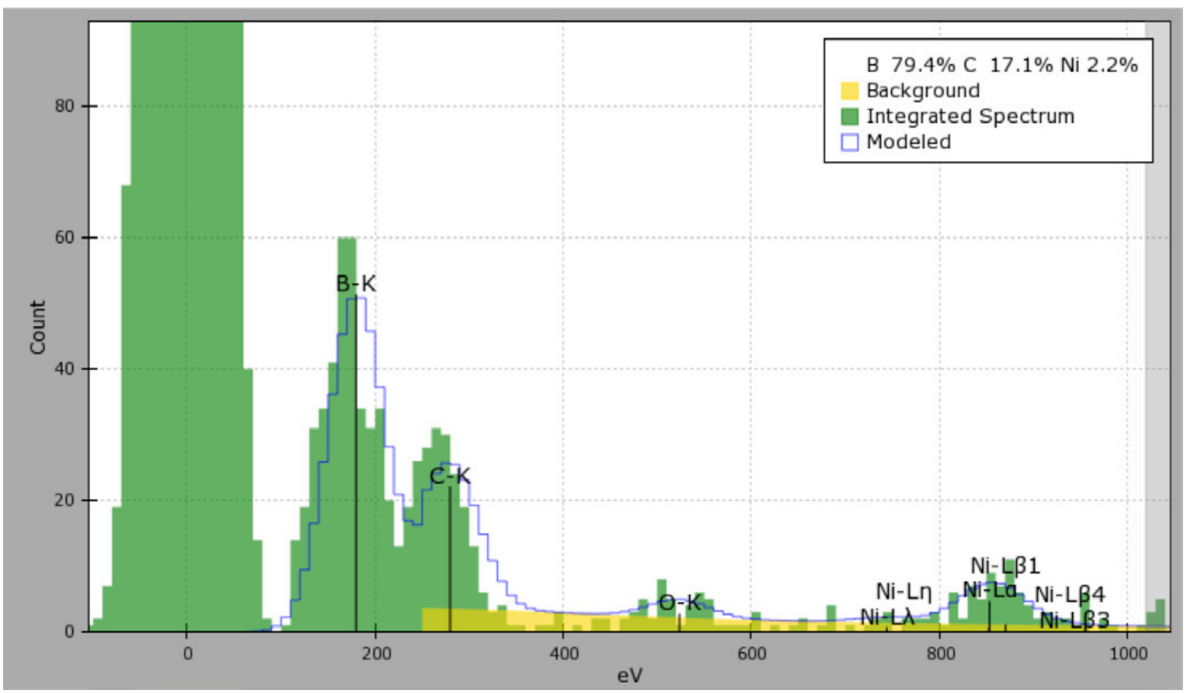


Figure 7c. EDS spectrum from selected area

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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.

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