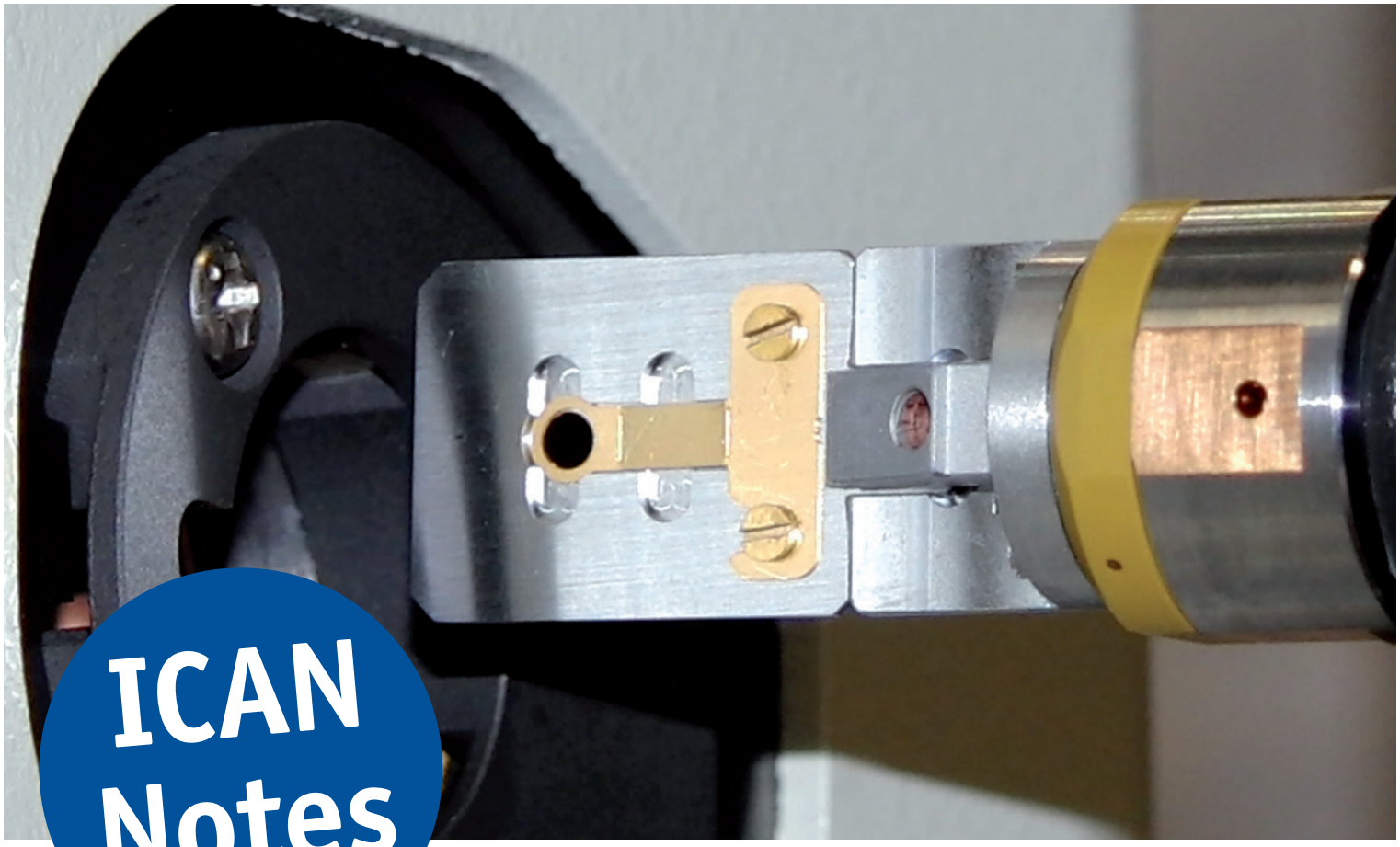

ICAN Notes

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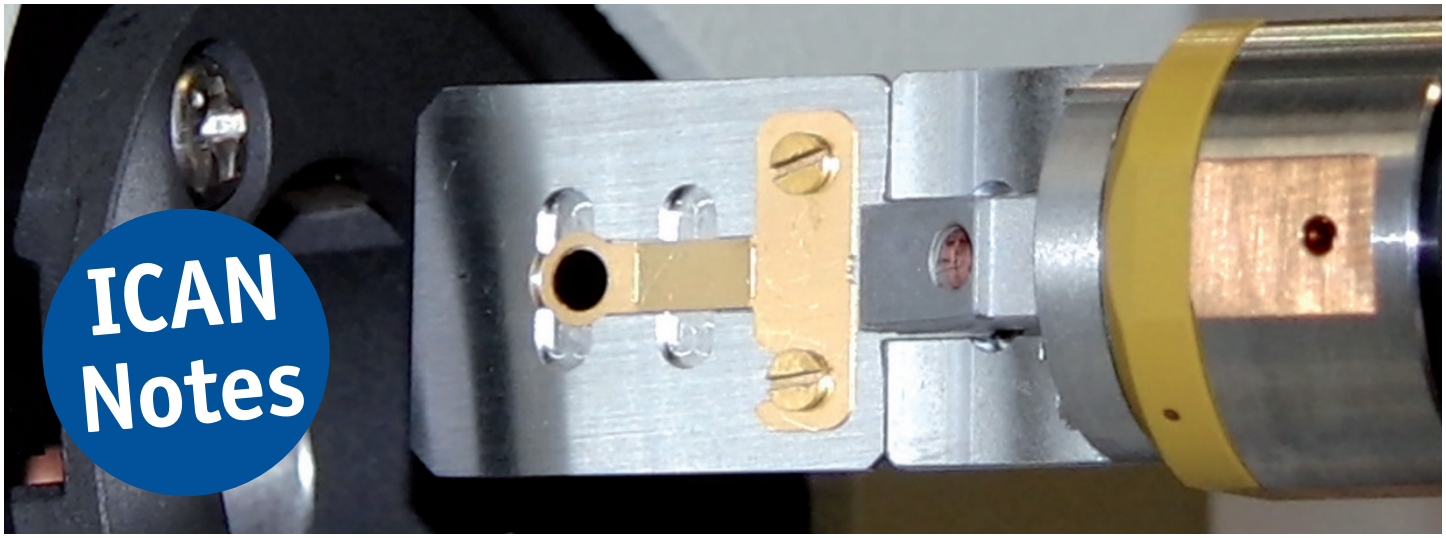

High-Angle Annular Dark-Field (HAADF) Contrast

by Markus Heidelmann* - Interdisciplinary Center for Analytics on the Nanoscale (ICAN)

Transmission electron microscopy is a versatile tool for the characterization of nano-material with high spatial resolution. High-angle annular dark-field (HAADF) [1, 2] contrast in the scanning mode has several advantages especially compared to conventional parallel bright-field illumination. First, there is no ambiguity about the observed structure when changing the microscope's focus. In conventional bright field TEM the observed pattern is always a rather complicated quantum mechanical interference pattern and the conclusion about the actual structure can be time-consuming. Whether the atoms appear a black or white is just a matter of focus and keeps changing back and forth with changing focus. In HAADF atoms always appear white on black background. Non-optimal focus conditions usually result in blurred micrographs with no structure at all, so finding the optimal focus conditions is much easier. Moreover, HAADF contrast is dependent on the atomic number Z, in other words, heavier atoms appear brighter in the micrograph. This is known as "elemental contrast". An example of a HAADF micrograph can be seen in Figure 1a showing a strontium-titanate ($SrTiO_3$) crystal along the [100] direction. The image intensity for atomic columns containing strontium is higher compared to the titanium columns, as can also be seen in the intensity profile in Figure 1b. In this respect HAADF micrographs are often attributed to be directly interpretable since at first glance no simulations are needed to compare the model structure and the micrograph.

However, looking at Figure 2a showing a HAADF micrograph of cobalt-ferrite ($CoFe_2O_4$) nanoparticles using a state of the art aberration-corrected Jeol 2200fs TEM two things stand out. First, the observed structure can be easily compared to the model structure in Figure 3a. White spheres represent the atomic columns containing cobalt and iron whilst oxygen atoms are not visible due

* *Corresponding author; eMail: markus.heidelmann@uni-due.de*

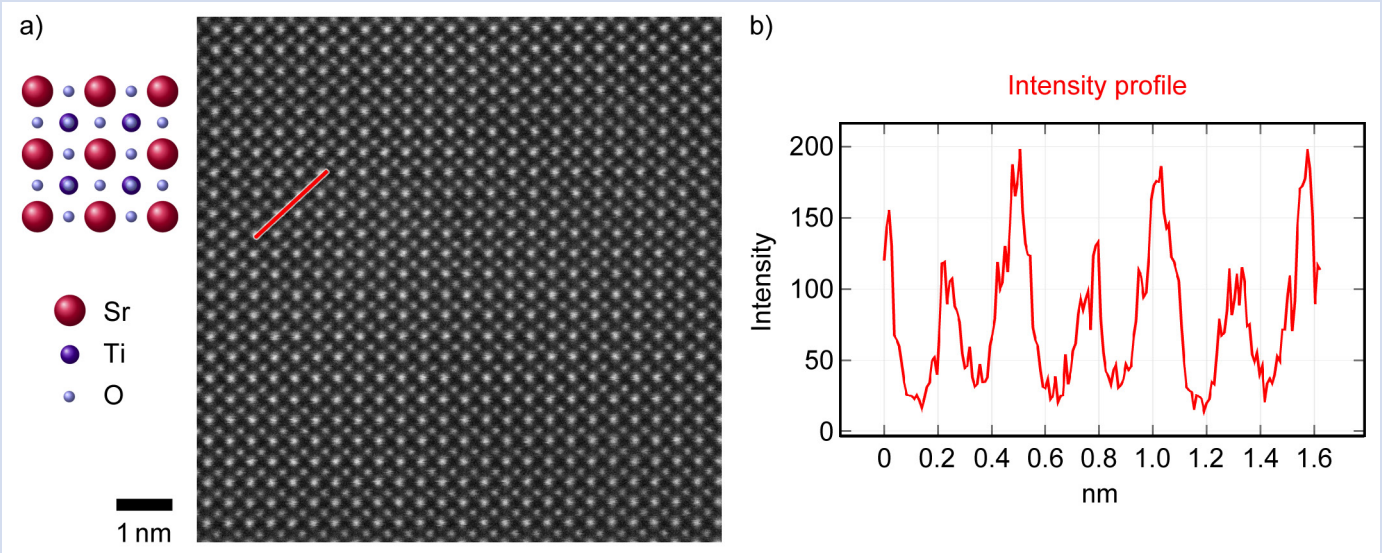

Figure 1: a) Crystal model and experimental HAADF image of $SrTiO_3$ [100]. b) Intensity profile along the red line in Figure 1a.

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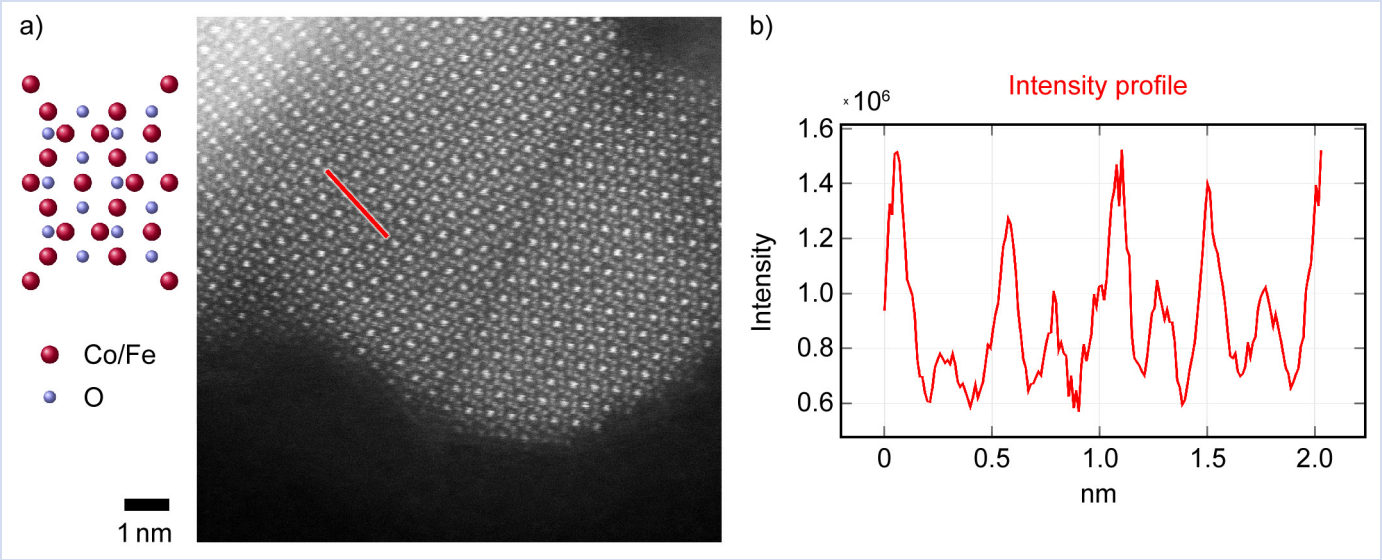

Figure 2: a) Crystal model and experimental HAADF image of $CoFe_2TiO_4$ [110]. b) Intensity profile along the red line in Figure 2a.

to their weak scattering power. Cobalt-ferrite crystallizes in the inverse spinell structure of which the Figure 2a shows the [110] projection. The structure consists of a rhomb-like arrangement of 10 atomic columns at the rim with an 11th atom in the middle of the rhomb. This central atomic position gives rise to a higher image intensity, which can also be seen from the intensity profile shown in Figure 2b. Keeping in mind that the HAADF contrast is known for its elemental contrast, it could be deduced that these central atomic columns consist of heaver atoms, i.e., they are cobalt-rich. However, metal atomic columns consist of cobalt and iron in the same fraction, which can be seen in Figure 3a, showing the [110] projection of the cobalt-ferrite unit cell. Red spheres represent positions occupied by cobalt and iron atoms in the respective fractions while blue spheres represent oxygen positions. The reason for the higher image intensity observed for the central atom is actually the increased density of atoms in the projection direction as can be seen in Figure 3b, which shows the cobalt-ferrite unit cell tilted by 90°. Note that the difference in atomic number is too low to be observed for cobalt and iron anyway.

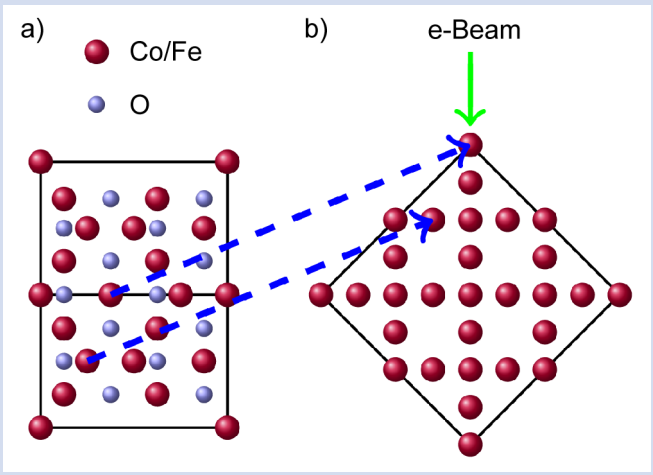

Figure 3: Crystal model of $CoFe_2TiO_4$ along a) [110] direction and b) [100] direction showing the higher density of atoms in the e-beam path for the two marked columns.

In addition, there is a somewhat darker line or border in the HAADF image of Figure 2b going from the lower left to the upper right representing a twin-boundary within a cobalt-ferrite nano-particle. Note the different orientation of the rhombs in the two parts of the nano-crystal. The reduced image intensity within the border region is a result of the distorted crystal structure especially in the projection direction.

To conclude, HAADF contrast is influenced by the atomic number (elemental contrast), local thickness of the object, and the local atomic density.

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Contact

ICAN | CENIDE
Universität Duisburg-Essen
Carl-Benz-Str. 199
47057 Duisburg

Tel.: +49 (0) 201 379 8080
Fax: +49 (0) 201 379 8046
eMail: ican@uni-due.de
www.uni-due.de/ican

ICAN

INTERDISCIPLINARY CENTER FOR ANALYTICS ON THE NANOSCALE

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ICAN | CENIDE
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