

High-resolution imaging on C_s -corrected Titan™ 80-300

A new era for new results

In NanoResearch a detailed knowledge of the structure of the material down to the atomic level is crucial for understanding the physical properties of nano-devices. As has been proven in a multitude of materials systems, the physical properties depend directly on the observed micro- or nanostructure; complete with interfaces and all defects. Particularly for computer simulation and modeling, it is not sufficient to just use the ideal crystalline structure; the complete microstructure needs to be accurately represented down to the atomic level, including defects and local alloying variations, in order to get an accurate representation. For this to be effective, the highest resolution imaging methods are essential. In conventional (Scanning) Transmission Electron Microscopy (S/TEM) the image resolution is limited predominantly by the spherical aberration (C_s) of the objective lens of the microscope. By correcting the spherical aberration, the resolution is enhanced and images become easier to interpret. When equipped with a C_s -corrector, Titan™ 80-300 breaks the important 1 Ångström (100pm) barrier in lateral resolution and gives images with unprecedented resolution. In STEM, aberration correction will mean tighter, brighter beams, yielding a stronger signal, higher imaging contrast, greater analytical sensitivity and unprecedented spatial resolution. The introduction of this technology is therefore an important breakthrough for atomic resolution (S)TEM in NanoResearch.

This application note describes the basic technology of C_s -correctors and the very necessary electron column requirements. Since on C_s -corrected microscopes the spherical aberration is no longer the resolution-limiting factor, the stability of the microscope column becomes critical. Titan 80-300 is the ideal platform to acquire HR-TEM and HR-STEM images, as it has been designed and dedicated to incorporate corrector technology and has unique inventions for ultimate stability. To illustrate the improvements in atomic resolution imaging, application examples are presented showing results on various materials like twinned diamond, silicon, TiB_2 and $Nb_7W_{10}O_{47.5}$. We demonstrate the strength of this new technology using comparisons of corrected and uncorrected images in STEM and TEM on the same sample area and also discuss the important role of focus series reconstruction in C_s -corrected TEM.

Titan: Spherical aberration corrector and column technology

Until recently, the application of S/TEM to obtain ultra high-resolution information involved the limitation of objective-lens aberration [1]. Correctors have the capability to minimize this aberration and consequently effectively remove the barrier to better resolution, direct interpretation and highest sensitivity. In any round magnetic lens, the parts of the object wave that carry the most interesting information pass through the outer zone of the objective lens and suffer from large additional phase shifts. Additional wave phase shifts are mainly introduced by the third-order

spherical aberration of the objective lens. As early as 1936, Scherzer proved that the spherical aberration couldn't be avoided in rotationally-symmetrical electro-magnetic fields [2]. A proven method to correct this type of aberration is by means of multipole fields, which effectively produce a negative third-order spherical aberration (See figure 1). In 1990, Rose described a hexapole corrector based on this principle allowing compensation of the C_s -aberration of the objective lens [3,4]. By changing the multipole excitation the value of the total spherical aberration of the TEM objective imaging system can be set to any value. This gives an additional parameter to tune the microscope to optimum imaging conditions.

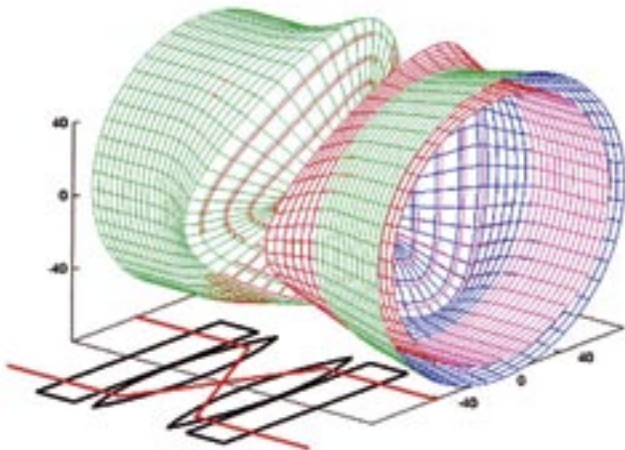


Figure 1: Principle of C_s -corrector. The graph illustrates the creation of a negative spherical aberration with the hexapole lens system. Note that the electrons further away from the optical axis (green) leave the optical system under a higher negative opening angle than the electrons closer to the optical axis (red). Figure courtesy of Dr. S. Uhlemann, CEOS GmbH.

Beside this coherent aberration, incoherent disturbances are limiting the information transfer of the TEM. The chromatic aberration of the objective lens in conjunction with the energy spread of the illuminating electron beam dampens the contrast transfer of the TEM at high spatial frequencies. This damping can be overcome by reducing the energy-spread of the illuminating electrons [5] using a monochromator in the illumination system [6]. The monochromator can also be used to improve the analytical capabilities of the S/TEM. However, as an additional bonus, both advanced optical components (monochromator and

C_s -corrector) improve the information transfer of TEM if the remaining residual incoherent disturbances can be suppressed below the desired lateral resolution of the TEM. It is the dependency on column stability that has encouraged FEI Company to develop the new Titan platform, which is dedicated for aberration correction, operable in the high-tension range from 80kV to 300kV, offering ultra-high resolution.

The Titan provides an information transfer deep into the sub-Ångström level. Disturbances due to mechanical vibrations have been reduced by introducing a design concept that separates the magnetic flux circuit from the mechanical forces in the column. Additionally the stiffness of the column is improved by making use of the 4th power dependency of the stiffness on the column diameter, by increasing the column diameter to 300mm from the previous standard which was 220mm. To further reduce the damping of the information transfer of the TEM due to misalignments, the Titan uses a new-patented invention, which ensures the mechanical alignment of the optical components of the column during assembly with extreme precision. The modular design of this technology enables C_s -corrector on-site upgrades to the Titan.

Like the chromatic aberration the current noise of the magnetic lens drivers and deflectors will reduce the temporal coherence of the imaging electrons, leading to limitations in the information limit. The Titan therefore has redesigned electronics, which attain noise levels below 0.1 ppm RMS. Such low noise levels are mandatory for a system that allows an information transfer down to deep sub-Ångström levels. For an optimum use of the monochromator the high tension must be very stable, with a noise/ripple ratio below 0.1 ppm RMS. Only with such stability it is possible to reach an energy resolution of 0.15 eV.

For a robust and reliable working S/TEM it is not only the short-term stability that is important but also the long-term stability like mechanical drift and especially optical alignment drift that will play a much more important role. Temperature-induced drift of the S/TEM needs to be reduced to negligible values to avoid changes in optics in

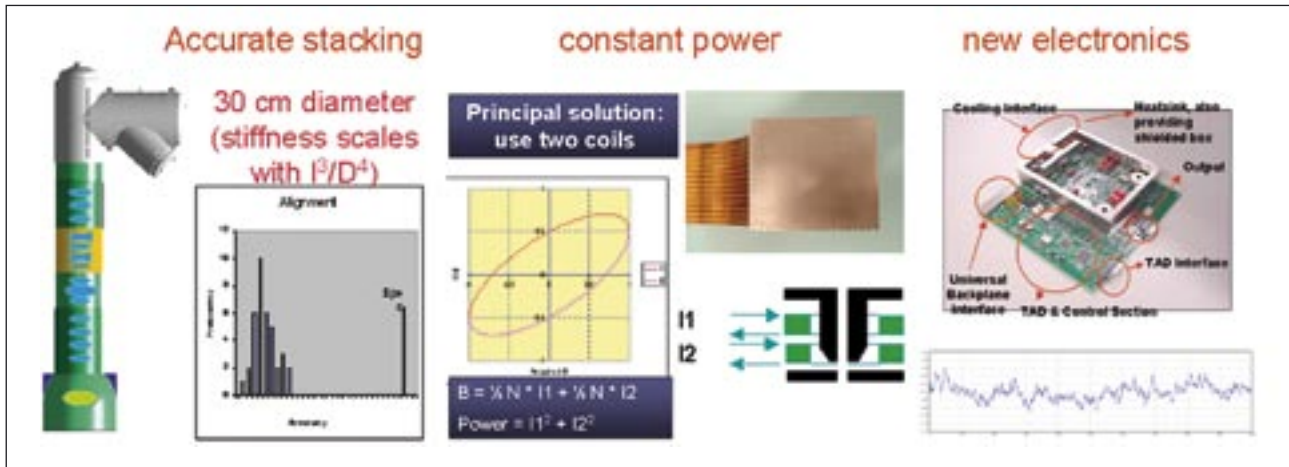


Figure 2: An overview of the new key design technologies that have proven to deliver ultimate stability, ultimate flexibility and ultimate performance: in basic operation, corrected TEM or STEM and or in combination with monochromator. Titan stability stability; ConstantPower Lenses for excellent thermal stability and new electronics with stability level down to 0.1ppm.

the hour time frame. The Titan has at least two coils per lens, making it possible to change magnetic-lens strength without changing the total lens current (ConstantPower), thereby holding the temperature in the column constant for all optical working modes.

All the aforementioned design elements of the Titan make it a platform capable to serve future needs such as the “chromatic-aberration correction project” planned in the TEAM project of the Department of Energy in the USA [7].

Application examples for improved sensitivity and resolution

Improvement in resolution

The combination of corrector optics and a highly stable column drastically improves the image resolution in HR-TEM and HR STEM. Before the C_s correctors era, resolution of around 140pm could be achieved, but images were noisy in STEM – and suffered from delocalization effects in TEM-imaging. With the introduction of the corrected Titan platform the lateral resolution has been improved to the sub-Ångström level. An example of this improved resolution is given in Figures 3 and 4. Figure 3 shows a HR-TEM image of diamond in $\langle 110 \rangle$ direction. The carbon atom dumbbell can be resolved in the image to as small as 89pm.

The same distance can be resolved in HR-STEM, which is remarkable because carbon is an ultra-light element with a very low scattering power for z-contrast imaging (Figure 4). The atomic structure of the defect line D3 as determined from the micrograph is overlaid in figure 4 and shown separately on the right. The defect line D3 does not show the ideal mirror symmetry of a S3 CSL twin boundary but has a displacement along the boundary. This is information that is only visible because of the corrector technology.

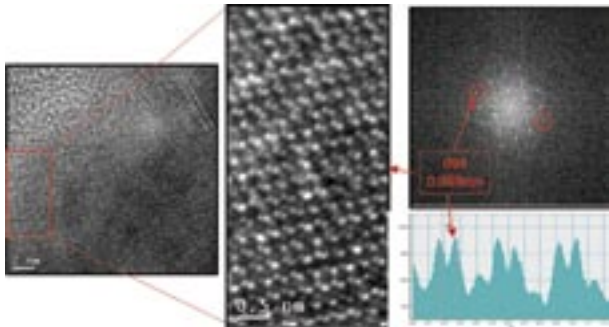


Figure 3: C_5 corrected HR-TEM image of diamond in the $\langle 110 \rangle$ zone axis. The dumbbell structure with a distance of 89pm can be resolved in the image. This is not an artifact as this resolution cannot be obtained by double diffraction effects.

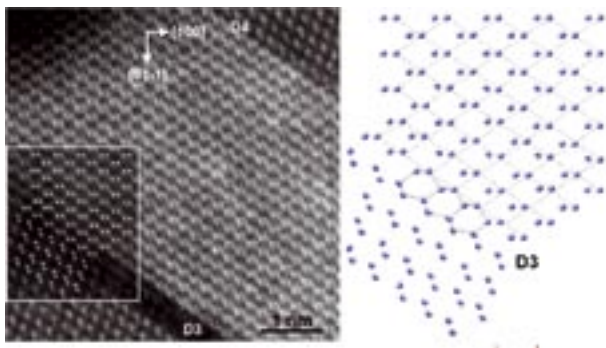


Figure 4: C_5 corrected HR-STEM image of diamond in the $\langle 110 \rangle$ zone axis. The dumbbell structure with a distance of 89pm can be resolved in the image. This resolution cannot be obtained by double diffraction effects. The atomic structure of the defect line D3 as determined from the micrograph is overlaid and separately shown on the right.

Improvements in sensitivity

In addition to the improvement in lateral resolution, the C_5 -corrected Titan enhances the sensitivity in atomic imaging and spectroscopy. Figure 5 shows this effect on a TiB_2 sample along the hexagonal $\langle 0001 \rangle$ zone axis. The lattice of the extremely light element boron can be imaged in HR-TEM imaging, revealing the hexagonal boron rings around the heavier titanium atom in the center of the ring.

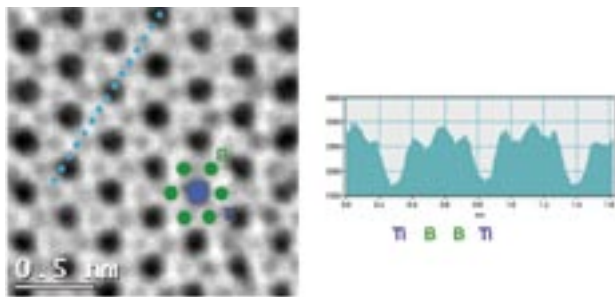


Figure 5: C_5 -corrected HR-TEM image of TiB_2 along the $\langle 0001 \rangle$ zone axis. The hexagon of boron atoms around the heavier Ti atom can be imaged directly. The intensity profile shows the contrast of the different atomic species.

Figure 6 shows a comparison of a corrected and an uncorrected STEM Ronchigram of amorphous material. The Ronchigram of the corrected Titan is flat up to 30mrad, which proves that small (sub Ångström) probes with higher beam currents can be formed. The probe current is determined by the brightness of the electron source and the maximum opening angle transmitted by the illumination system (including the C_5 -corrector). Since the brightness is a fixed value on every system only the correction of the spherical aberration of the illumination system allows in increase of probe current by increasing the opening angle without increasing the probe diameter. This increase in currents of C_5 -corrected STEM lowers the noise level in HR-STEM imaging and improves its sensitivity. The advance in forming smaller electrons probes with higher probe currents is demonstrated in the z-contrast image of diamond (fig.4), as carbon is a light material with low scattering power and the distance between the diamond dumbbell is only 89pm. This resolution cannot be obtained by double diffraction effects.

The higher probe currents in probe diameters of a few Ångströms improve the sensitivity in chemical analysis with high lateral resolution for analytical work. Although an extremely important topic, it is not the focus of this application note. For more information refer to Energy dispersive X-ray analysis (EDS) or electron energy loss spectroscopy, EELS.

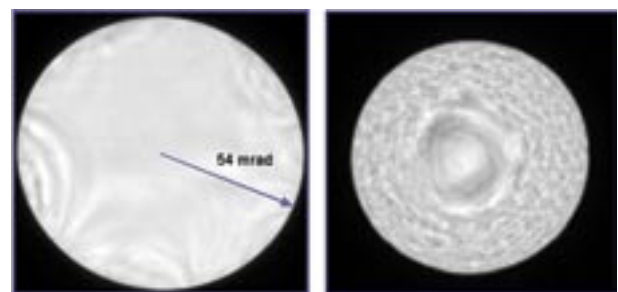


Figure 6: Ronchigram of a probe C_5 -corrected (left) and uncorrected Titan (right). The Ronchigram on amorphous material of the corrected Titan is flat up to 30mrad, allowing sub-Ångström probes with higher opening angle than in the uncorrected Titan, where the smallest probe has a diameter of 136pm.

Comparison of corrected and uncorrected images

The benefit of corrector technology can be demonstrated by comparing images taken on the same area with and without the spherical aberration correction. For this purpose the corrector on the Titan has been switched off to obtain the uncorrected images.

Figure 7 shows a pair of HR-TEM images of Nb₇W₁₀O_{47.5}. In the thin region of the corrected image each dark dot represents the position of Nb or W (Figure 7, inset left side). This is the case because the Cs-corrected image no additional wave phase shifts are introduced by the third-order spherical aberration (Cs) of the objective lens. In an uncorrected image the atoms can have bright or dark contrast due to the phase scrambling, so the atomic positions cannot be extracted directly (Figure 7, inset right side).

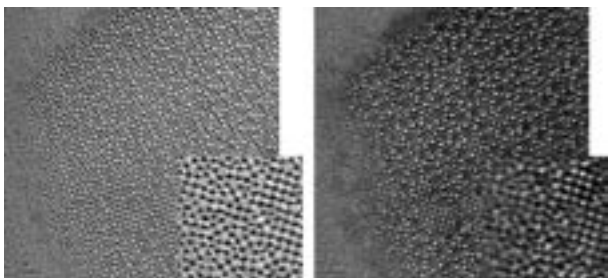


Figure 7: Pair of Cs-corrected (left) and uncorrected (right) HR-TEM images of Nb₇W₁₀O_{47.5}. In the thin region of the corrected image each dark dot represents the position of Nb or W (inset left side). In an uncorrected image the atoms can have bright or dark contrast, so that the atomic positions cannot be extracted directly (inset right side).

In Figure 8 a second comparison of corrected and uncorrected HR-TEM images is shown. Here a grain boundary of silicon is imaged. The improved resolution is demonstrated by resolving the dumbbell structure of 136pm in silicon, which is absent in the uncorrected image. Additionally the atomic structure at the grain boundary can be visualized in the corrected image showing alternating rings of 5 and 7 atoms at the boundary. In the uncorrected image no direct visualization is possible due to the lack of resolution and delocalization effects caused by the spherical aberration.

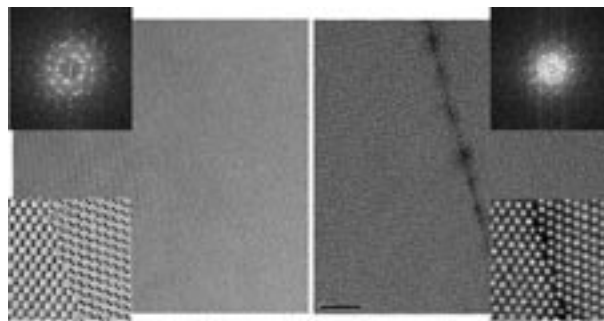


Figure 8: Pair of Cs-corrected (left) and uncorrected (right) HR-TEM images of a grain boundary in silicon. Clearly the dumbbell structure of 136pm and the atomic structure at the grain boundary can be resolved in the corrected image. (Sample courtesy : J.Thibault, Marseille, France)

The leap in performance shown in corrected HR-TEM imaging can also be demonstrated in probe-corrected STEM imaging using the Titan. Figure 9 shows a pair of corrected and uncorrected high-resolution STEM image on the same silicon grain boundary imaged in HR-TEM (Figure 8). In both images the dumbbell distance of silicon <110> of 136pm can be resolved, but the probe size in the corrected image is approximately 80pm, while the uncorrected image was acquired with a probe of about 136pm. This difference in probe diameter can be clearly noticed in the image. The corrected image shows much higher contrast than the uncorrected one due to the fact that the atomic structure is imaged with the smaller probe. The intensity profile in Figure 9 shows the gain in contrast especially in the depth of the minimum between the silicon dumbbell distances.

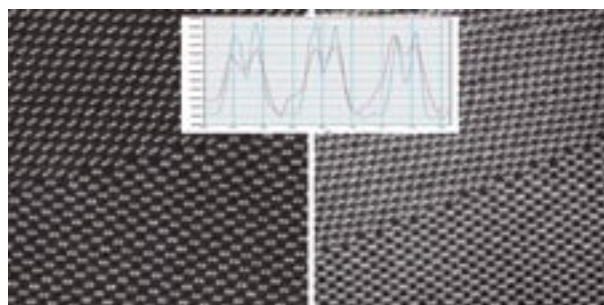


Figure 9: Pair of Cs-corrected (left) and uncorrected (right) HR-STEM images of a silicon grain boundary. The improvement in the resolution is clearly noticeable in the form of higher contrast in the image. The intensity profile shows the improvement is resolution in the increased contrast between the silicon dumbbells.

Focus series reconstruction on C_5 -corrected HR-TEM imaging

FEI offers a full focus series reconstruction solution with Truelmage technology, which not only allows automatic acquisition of such through-focus series on a Titan but also to reconstruct the exit wave from this series. An important extra piece of information this software provides is the exact thickness at any part of the reconstructed image. Since the image C_5 corrected removes the artifacts due to the spherical aberration (like delocalization effects on interfaces and phase scrambling depending on different atomic distances) the question arises of whether focus series acquisition is necessary at all in C_5 -corrected HR-TEM imaging.

To answer this question some fundamental imaging facts have to be considered. When the electron passes through the sample the amplitude and the phase of the electron is altered. The CCD-camera detects only intensity modulations of the image plane, which are caused by both the phase and amplitude change of the electron wave in a particular image point. Hence single image HR-TEM cannot recover the contrast contribution of the phase and amplitude in an image point, but detects always a mixture of both. This leads to a loss of vital information about the atomic structure in the single image.

By the acquisition of a series of images acquired under different focus conditions, Truelmage enables separation of this mixture and allows the amplitude and phase image of the sample to be obtained. This is crucial for quantitative HR-TEM imaging, in which the loss of phase and amplitude information is not acceptable. Two examples of such a reconstruction of a complex image is shown in figure 10 and figure 11. From a focus series of 20 images the amplitude and the phase image of TiB_2 and silicon can be reconstructed. This pair of images gives for every image point a separate value for the amplitude and phase change caused by the sample. With this information for example the exact amount of atoms in one atomic column and their variation in the crystal can be determined.

Therefore - coming back to the fundamental question - an image C_5 -corrected Titan makes the focus series reconstruction method not obsolete, but the combination of both enables a deeper understanding of the structure of the material examined and true quantitative information.

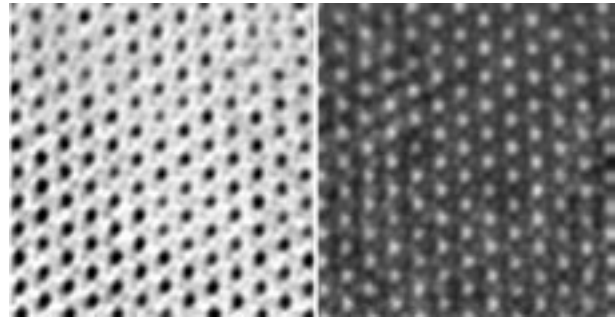


Figure 10: Reconstructed amplitude (left) and phase image (right) of TiB_2 in the $\langle 0001 \rangle$ zone axis. The positions of the light boron atoms can be resolved in the electron amplitude (weak dark dots next to the strong dark dot of the Titanium atom) and phase image (weak bright dots).

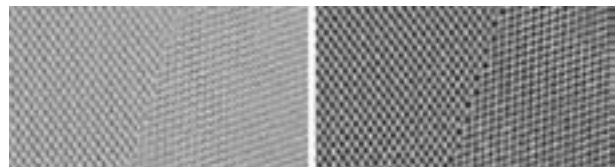


Figure 11: Reconstructed amplitude (left) and phase image (right) of a grain boundary of silicon in the $\langle 110 \rangle$ zone axis. (Sample courtesy: J.Thibault, Marseille, France)

Conclusion

In this note an overview of the Titan platform for corrector technology is given and the break through in atomic resolution S/TEM is shown in high resolution images on various materials using the new platform and both varieties of corrector, those for the imaging (objective) side as well as the probe (condenser) side. For educational purpose the benefits of the correction of the spherical aberration are demonstrated on pairs of corrected and uncorrected images of the same sample area. In addition the role of Truelmage focus series reconstruction technology on the C_s-corrected Titan is discussed, which gives the user an easy access to quantitative and high-precision information on the atomic positions in non-perfect crystal structures. Some of the key issues that should be considered when implementing corrector technologies on any microscope have been discussed. The corrected Titan 80-300 with its S-TWIN objective and its pole pieces gap measuring over 5 mm, delivers sub-Ångström resolution in TEM and STEM while allowing ample space for sample manipulation. Its stability, performance and usability will allow corrected microscopy to be taken to the next level where new discoveries of structure-property relationships of materials are possible at ever decreasing scales.

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- [7.] The Titan platform is the basis of the TEAM project of the USA Department of Energy with the goal to arrive at 0.5 Ångström resolution in TEM and STEM on a single system, in combination with a Cc corrector developed in co-operation with CEOS GmbH.

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