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「収差補正による電子顕微鏡の新次元」

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With Aberration Correction to a New Dimension in Electron Microscopy

Commemorative lecture at the 29th Honda Prize
awarding ceremony on the 17th November 2008

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このレポートは、2008年11月17日 東京、帝国ホテルにおいて行われた第29回本田賞授与式記念講演の要旨をまとめたものです。
This report is the gist of the commemorative lecture at the 29th Honda Prize Awarding Ceremony on the 17th November 2008 Imperial Hotel,Tokyo.

With Aberration Correction
to a New Dimension in Electron Microscopy

Maximilian Haider, Harald Rose and Knut Urban

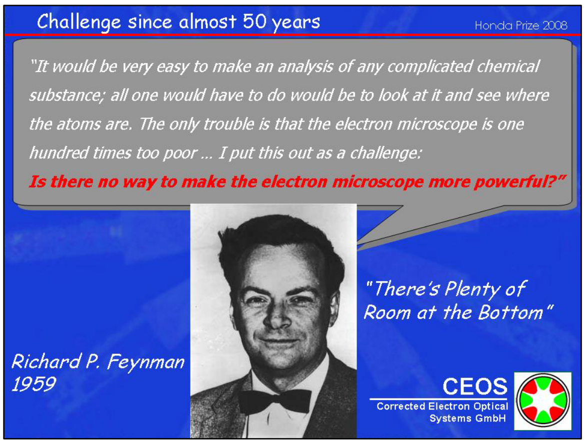

Fig 1　Richard P. Feynman,
„There's Plenty of Room at the Bottom", 1959

〈Fig 1〉　*It would be very easy to make an analysis of any complicated chemical substance; all one would have to do would be to look at it and see where the atoms are. The only trouble is that the electron microscope is one hundred times too poor … I put this out as a challenge:* ***Is there no way to make the electron microscope more powerful?"***

1. Introduction.

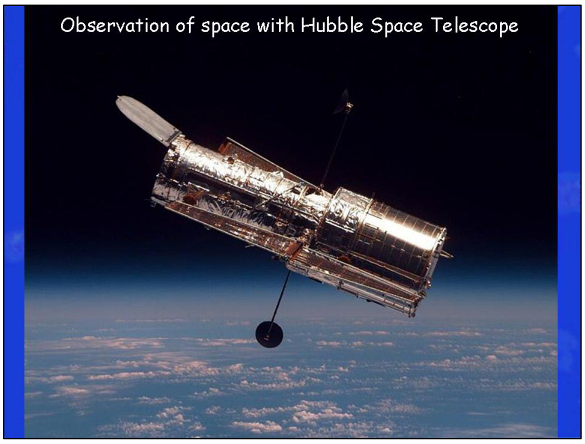

Fig 2

Fig 3

〈Fig 2〉〈Fig 3〉　To see the atoms is an old dream in science. To be able to derive properties and functions directly from an observation of the basic building blocks of matter appeared to be like a view into the workshop of nature. Is there no way to make the electron microscope more powerful, i.e. to increase its resolution? Today we know the answer: Yes, there is a way, and we were able to realize it in recent years. It is based on aberration-corrected electron optics.

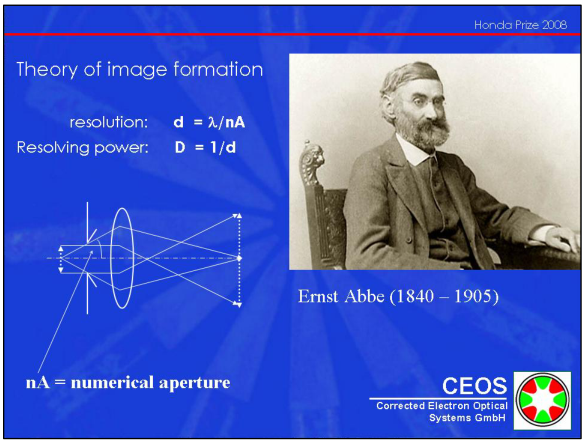

Fig 4

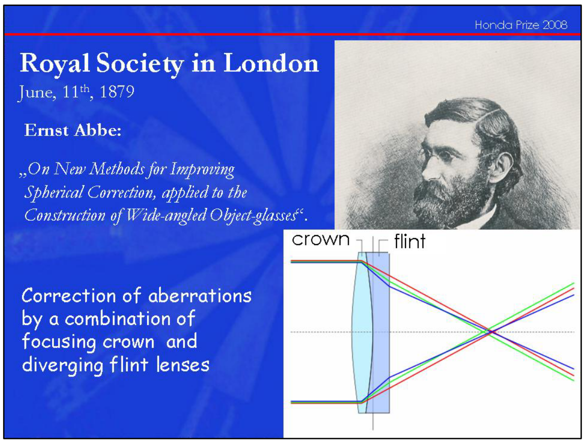

Fig 5

〈Fig 4〉〈Fig 5〉 The resolution limit of a microscope is defined as the smallest distance between two object points which can just be distinguished as two separate points in the image. More than 130 years ago, Ernst Abbe showed that this resolution improves when the wavelength of the employed radiation decreases and when the angular range of rays leaving the object and entering the objective lens of the microscope increases.

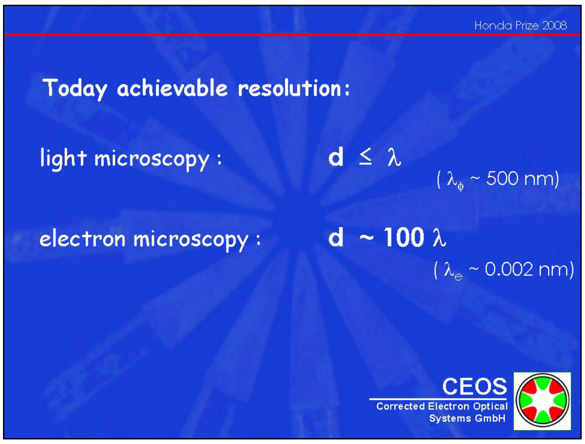

Fig 6

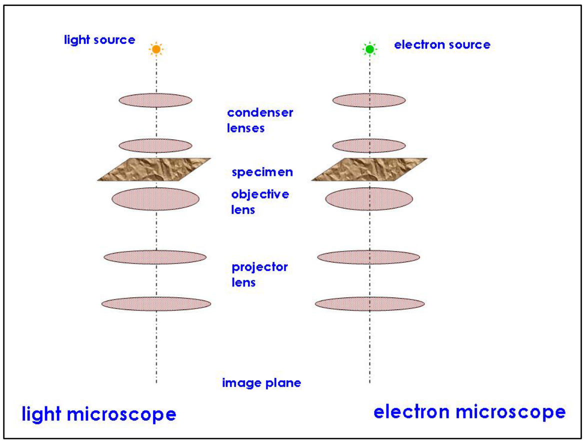

Fig 7

〈Fig 6〉〈Fig 7〉 Electron microscopy makes use of the remarkable property of fast electrons to behave not only as particles but also as waves. Fast electrons can be produced in accelerators where they traverse an electric tension of a few hundred thousand Volts. The faster the electrons the smaller is their wavelength. The accelerator of a modern electron microscope operates typically at 200,000 or 300,000 Volts, and the corresponding wavelength amounts to 2.5 or 2.0 picometers, respectively (1 pm = 10^{-12} m).

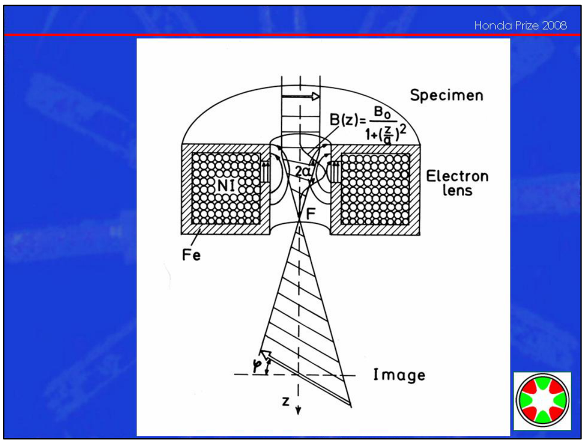

Fig 8

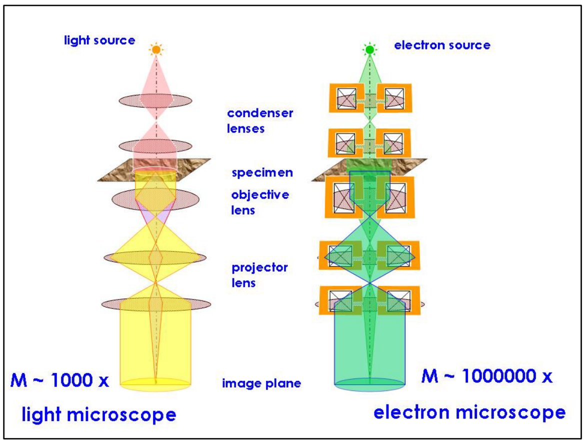

Fig 9

〈Fig 8〉〈Fig 9〉 Modern light microscopes have reached their maximum physically possible resolution in the order of magnitude of the wavelength of light (about 0.5 micrometers). This achievement results from the fact that high-quality lens systems can be constructed essentially without aberrations. In contrast, until recently the quality of electron lenses was so bad that, as a result of aberrations, the resolution was limited to about 200 picometers (about one hundred times the wavelength).

Fig 10

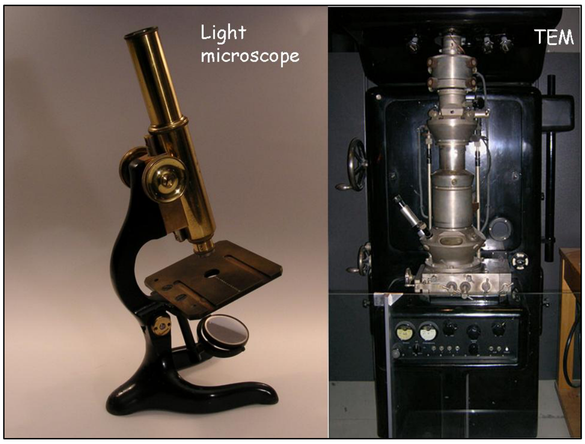

Fig 11

〈Fig 10〉〈Fig 11〉 This behavior largely excludes the possibility to image and study the atomic structure of matter, to the great regret of scientists and engineers. Actually, modern science, from materials via technology to biology and medicine, experiences a burning need for atomic studies.

In principle one always has to deal with two fundamentally different types of lens aberrations, so-called geometrical aberrations and chromatic aberrations. The latter originate from the fact that the refractive power of a lens varies with the wavelength of the radiation (color) and therefore, when light with a spectrum of colors is employed or an electron beam with a certain energy width, the lens is unable to focus the constituent rays of different color (energy) into a common point. The most important geometrical aberration is spherical aberration. It causes the focal length of a lens to depend on the angle of incidence of the radiation entering the lens. As a result, the focal length is shorter for rays passing the lens at its periphery compared to paraxial rays passing the lens in its central region. In the past, as a result of the very low quality of electron lenses, electron-optics engineers made the angle of the bundle of rays coming from the object and entering the lens very small by placing an aperture stop at the back focal plane of the lens. However, the information on high-resolution details (e.g. the atomic structure) is lost by this measure because the high-angle beams carrying the high-resolution information are excluded from image formation.

Again it was Ernst Abbe who showed (for light optics) that systems consisting of converging and diverging lenses can be designed in such a way that the individual lenses are mutually compensating their aberrations. Electron lenses consist of suitably formed magnetic fields produced by coils through which an electric current is flowing. Unfortunately, a major difference between light-optical (glass) lenses and electron lenses is that, according to the laws of physics, diverging round lenses cannot be produced for electrons.

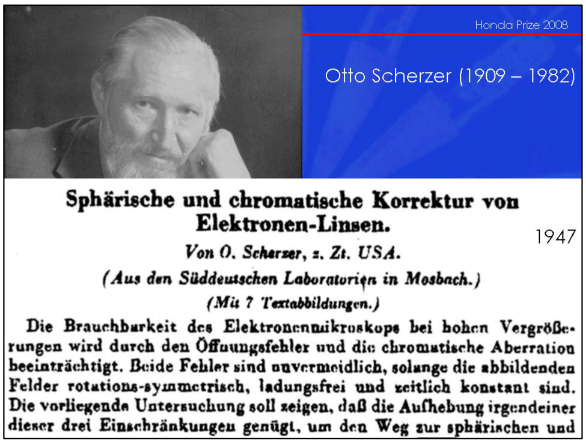

Fig 12

〈Fig 12〉 This fundamental difficulty was first recognized in 1936 by Otto Scherzer of Darmstadt University. He also suggested, on the basis of extended theoretical work, a way out of this dilemma by adding multipole lenses to the round lenses of the microscope.

Fig 13

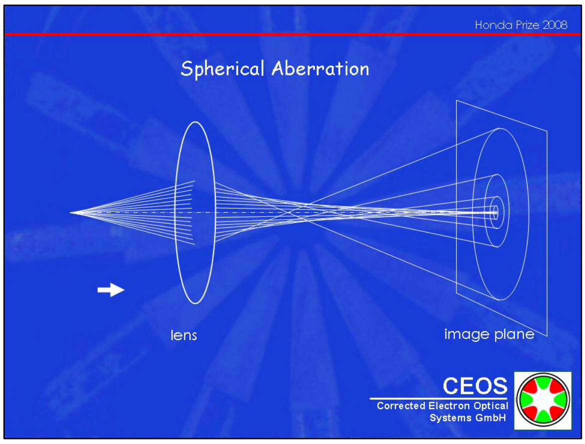

Fig 14

〈Fig 13〉〈Fig 14〉 Moreover, he proved mathematically that special systems consisting of both types of elements are-in principle-largely free of aberrations.

Since then several fruitless experimental attempts were made to realize Scherzer's correction principles and to construct correctors compensating for the spherical and chromatic aberrations. Although these projects demonstrated that correction works in principle ("proof of principle"), none of them achieved an improvement of resolution.

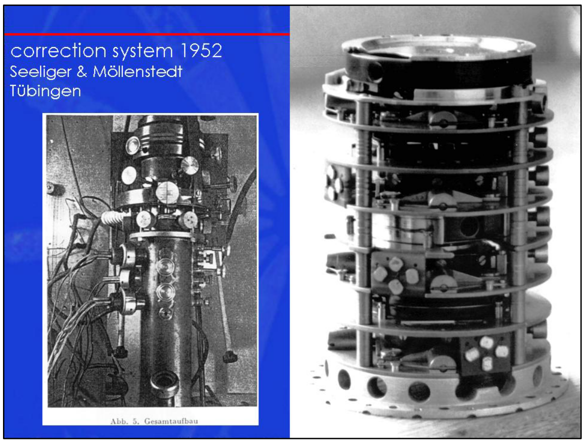

Fig 15

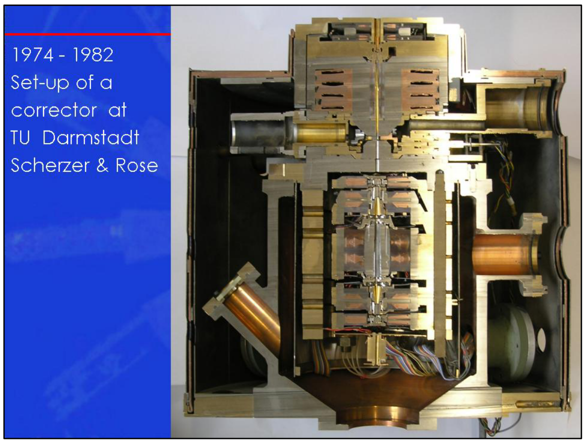

Fig 16

〈Fig 15〉〈Fig 16〉 The main reasons for this failure of the correction experiments were: (a) the basic microscope was not stable enough, (b) the interference with the environment had been underestimated, (c) the entire system was too complex and could not be aligned within a sufficiently short time, and (d) it was not possible to measure all resolution-limiting aberrations at a sufficient rate and precision and to correct them with the electronic equipment available at that time. The disappointment was so severe that at the end of the nineteen eighties a panel of experts advised the American National Science Foundation to no longer fund such developments since it appeared unlikely that any further progress could be expected within a reasonable period of time.

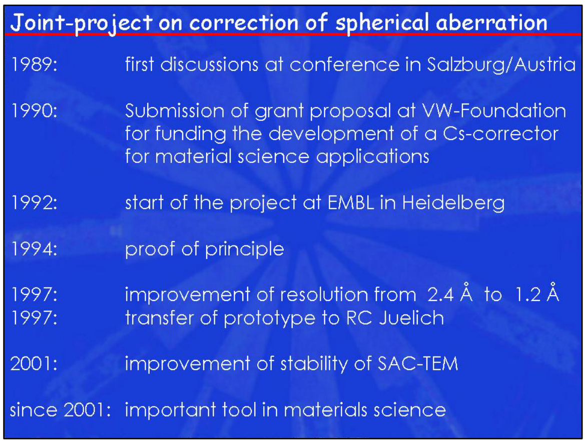

Fig 17

〈Fig 17〉 At about the same time, in 1989, Max. Haider, Harald Rose and Knut Urban had intensive discussions on the prospects of aberration correction during the “Three Countries Meeting” on electron microscopy (organized by the Austrian, the German and the Swiss Electron Microscopical Societies) in Salzburg, Austria. At the end, the three were convinced that with the help of a new type of corrector involving two hexapole lenses the successful correction of spherical aberration in a modern transmission electron microscope (TEM) should be feasible. They hoped that this would provide for the first time a basis for atomic resolution. An advanced high-resolution TEM was selected as the basic instrument. In particular this microscope was equipped with a highly stable field-emission electron source providing electron beams with an extremely narrow range of energies. Therefore, the expected result could be achieved by spherical-aberration correction alone, additional chromatic correction would not be necessary thus substantially simplifying the task.

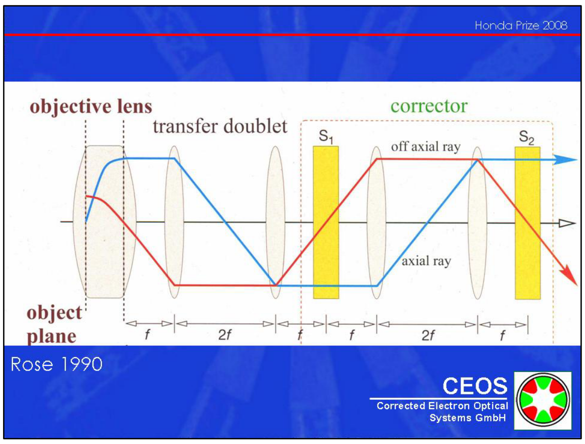

Fig 18

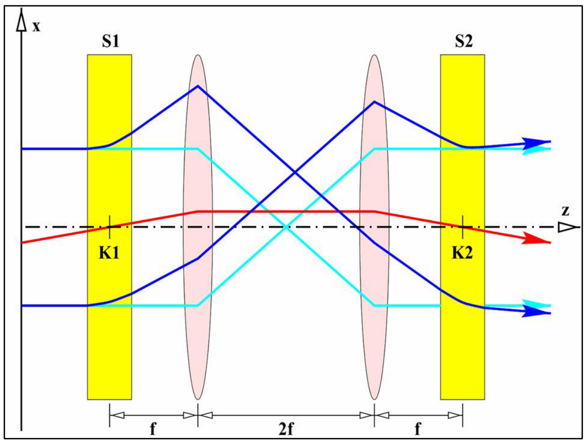

Fig 19

〈Fig 18〉〈Fig 19〉 At the end of 1990 the three submitted a grant application to the German Volkswagen Foundation. Their proposal was approved one year later and the development of the corrector started in January 1992. The joint project to improve the resolution of a mediumvoltage instrument for atomic characterization of materials was covered by the electron-optical theory of Rose at the Technical University of Darmstadt, the experimental development of Haider at the EMBL (European Molecular Biology Laboratory) at Heidelberg, and the subsequent application in materials science by the group of Urban at the Research Centre Jülich. Already in 1994, the correction power of the novel hexapole-corrector could be demonstrated successfully on an optical test bench. This corrector generates a negative spherical aberration that suffices to compensate for the spherical aberration of the objective lens. Based on this proof of principle, the project obtained from the Volkswagen Foundation the final funding for realizing the corrected microscope at the Heidelberg laboratory.

2. Design Principles of the Hexapole Corrector

A magnetic hexapole field produces a force on an electron, whose strength is proportional to the square of the lateral distance from the optical axis. The direction of this force depends on the azimuth angle about the axis and changes its sign every 60 angular degrees. Therefore, a bundle of electrons propagating on the mantle of a cylinder parallel to the axis experiences a three-fold deformation (ray deviation of second order) after passing the hexapole field. However, due to the particular dependence of the force on magnetic-field strength also a third-order rotationally symmetric deformation occurs. As a result, a three-fold axial aberration of second order and a rotationally symmetric spherical aberration of third order arise at the image plane. The sign of the latter aberration is opposite to that of the spherical aberration of a round lens. Therefore, hexapoles can be employed to compensate for the spherical aberration of the objective lens of an electron microscope.

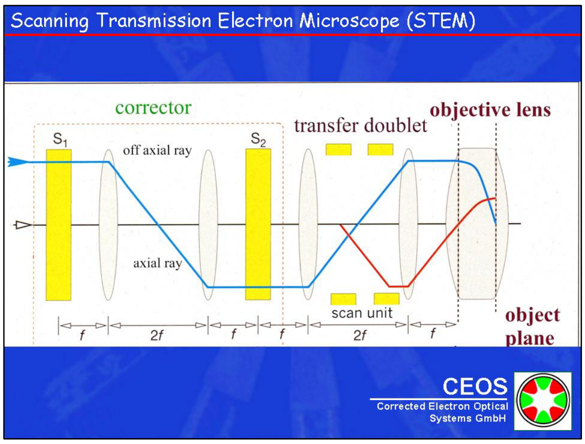

Fig 20

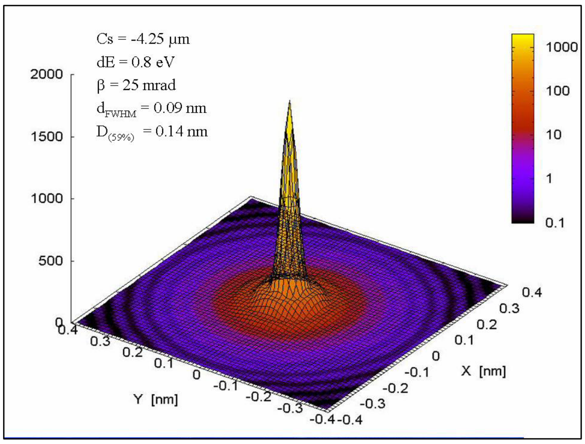

Fig 21

〈Fig 20〉〈Fig 21〉 In order to utilize the negative spherical aberration of the hexapoles, one must first get rid of their awkward large second-order aberrations. However, it is not possible to eliminate the second-order path deviation in a system consisting solely of hexapoles. To compensate for this deviation, one needs two identical hexapoles and a telescopic round-lens doublet. One hexapole is placed at the front focal plane of the first round lens, the other hexapole at the back focal plane of the second lens. The transfer-doublet images the first hexapole inversely onto the second hexapole. In this way, the second-order path deviations cancel out whereas the third-order deviations add up. The resulting negative spherical aberration at the image plane is proportional to the square of the hexapole strength. Hence, by appropriately adjusting this strength, the negative spherical aberration of the corrector compensates for the positive spherical aberration of the objective lens. Since this corrector only introduces a negative spherical aberration, one can conceive it as "spectacles" for the imperfect objective lens of the electron microscope.

3. Adjustment of the spherical-aberration corrected TEM

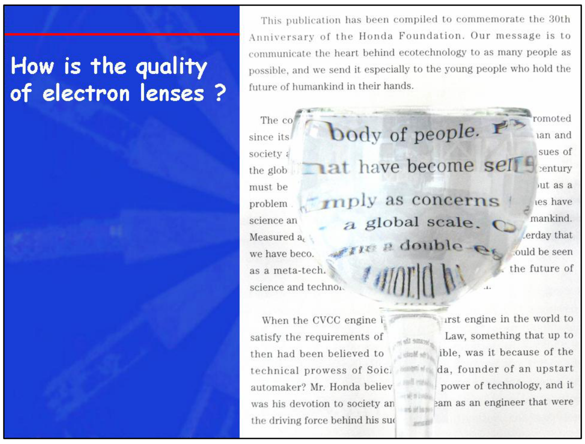

Fig 22

〈Fig 22〉 The lenses of light-optical microscopes are manufactured with micrometer (10^{-6} m) accuracy and subsequently glued together in an essentially for ever stable arrangement. In order to achieve atomic resolution in an electron microscope, the electron lenses would have to be produced and aligned with a precision in the nanometer (10^{-9} m) range. However, the electrodes and the pole-pieces of electron-optical elements can neither be produced nor mechanically aligned with this accuracy. Even if an ideal geometry of the pole-pieces could be attained, the unavoidable magnetic inhomogeneities prevent the necessary accuracy of the imaging field. To maintain the required precision of the ray path within the lens system, additional electromagnetic adjustment elements are needed. For this purpose, additional so-called stigmators and deflectors are incorporated into the system at appropriate positions. With the help of these elements the unavoidable residual aberrations can be subsequently eliminated.

Fig 23

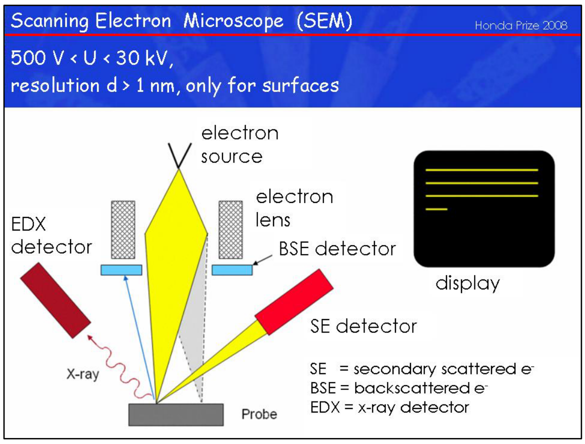

Fig 24

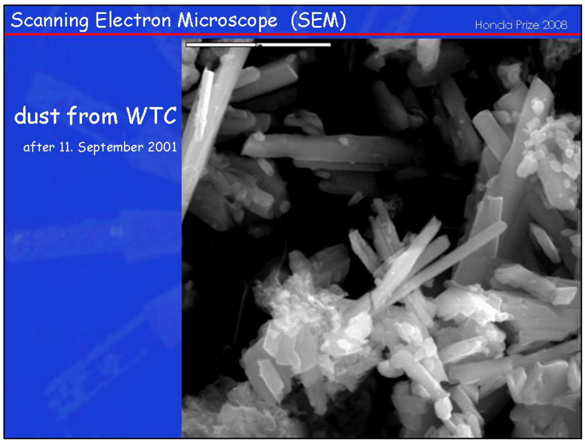

Fig 25

〈Fig 23〉〈Fig 24〉〈Fig 25〉 In order to be able to utilize the hexapole corrector and to successfully set the stigmators and deflectors, the current state of the aberrations and of the alignment has to be known. Actually the design and implementation of high-precision diagnostics and alignment procedures turned out to be a major task in the successful realization of the project. The residual aberrations can be measured by employing the so called "diffractogram-tableau" method. A diffractogram is obtained by taking an image of an amorphous specimen which is then subjected to a Fourier transform in a computer and squared. The diffractogram tableau is composed of several such diffractograms each taken with a different direction of illumination and arranged according to the direction of incidence. By evaluating the tableau, one can measure the resolution limiting coherent aberrations and thus determine the state of correction of the whole microscope.

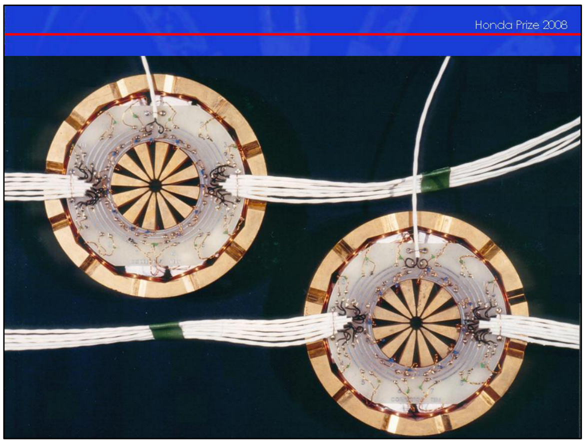

Fig 26

Fig 27

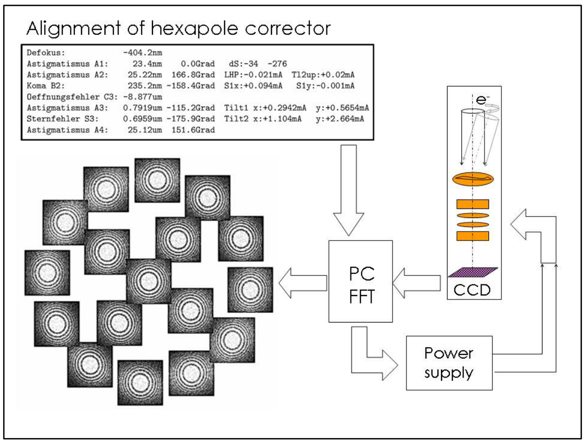

Fig 28

〈Fig 26〉〈Fig 27〉〈Fig 28〉 To achieve the required precise adjustment in the shortest possible time, the images are acquired with a CCD camera and the associated diffractograms of the images are calculated on-line with a computer. The aberrations are deduced from the diffractograms by measuring the changes of focus and two-fold astigmatism induced by altering the direction of incidence. This analysis provides also the values of the remaining higher-order aberrations. An additional calculation gives the needed output of the voltages and current drivers of the stigmators and deflectors for the compensation of all relevant aberrations. By means of microprocessors the voltages and currents are applied to the appropriate electrodes and coils.

4. Experimental results and applications

The demonstration of the improvement of the resolution limit from 0.245 nm for the uncorrected microscope down to about 0.125 nm for the corrected instrument was achieved in the first half of 1997. At this time, Bernd Kabius, a member of Urban's group, started working with the instrument in order to learn how to operate it for real materials science applications. On June 24th, 1997, he succeeded to obtain the first atomic-resolution images of gallium-arsenide, a semiconductor employed in high-speed electronics. After this success, the first ever aberration-corrected transmission electron microscope was transferred to Jülich and installed there.

Fig 29

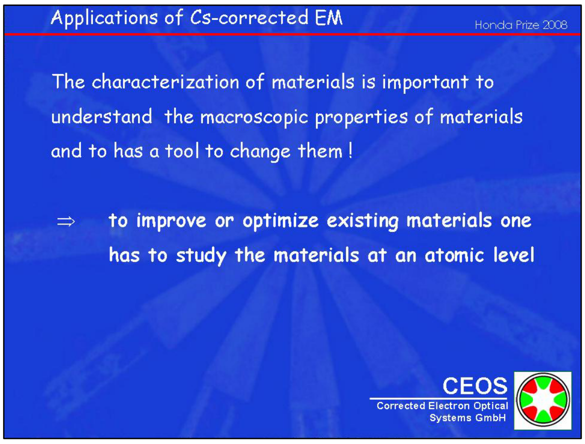

Fig 30

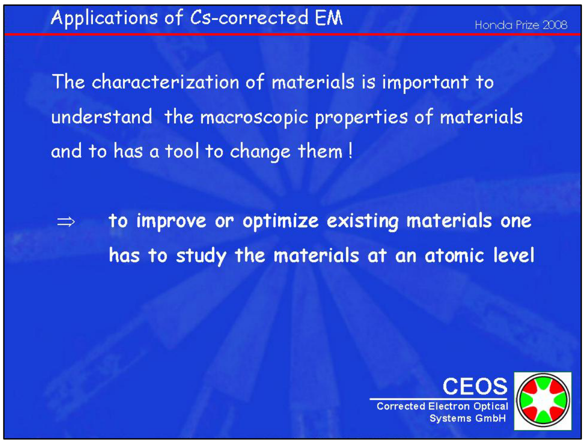

Fig 31

〈Fig 29〉〈Fig 30〉〈Fig 31〉 The first years of operation of the instrument at Jülich required some patience. Although the innovative optics performed perfectly, it took painstaking two additional years to trace down and cure a number of conventional technical problems. During this time Haider also implemented a few modifications, which largely improved the electrical stability of the instrument. By the end of 2000 the instrument was in a perfect shape, and demonstrates till today extraordinary performance. The materials science community was enthusiastic about the progress achieved, i.e. genuine atomic resolution, when the group gave their first extended report on a meeting of the American Materials Research Society in San Francisco on April 17th, 2001. The auditorium could not accommodate all the scientists who wanted to hear about the new developments, the doors had to remain open to allow participants standing in the corridor to listen.

The work at Jülich in the following years revealed that with an aberration-corrected instrument a number of entirely new operation modes are possible. These were worked out in detail and set on a theoretically sound foundation. In particular, it is the NCSI (negative spherical aberration-imaging) technique which has opened up entirely new possibilities for atomic-resolution materials investigations. Since for the first time oxygen and other light nuclear-charge elements can be imaged directly (oxygen could in fact never be studied before by any microscopic technique) scientists working on oxides, ceramics or other technically important compounds were enthusiastic about the potential of aberration-corrected electron microscopy for their fields.

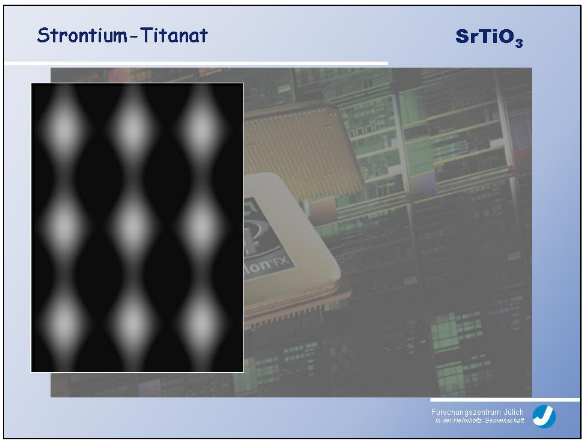

Fig 32

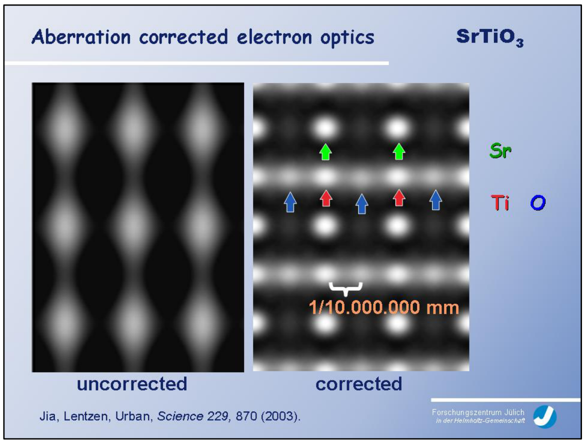

Fig 33

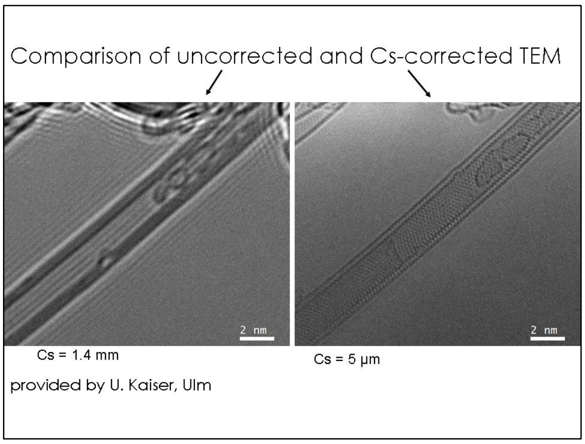

Fig 34

〈Fig 32〉〈Fig 33〉〈Fig 34〉 An example of the results obtained in Jülich is the measurement of how oxygen occupies particular atomic sites in Bariumtitanat ($BaTiO_3$) and Strontiumtitanat ($SrTiO_3$). Both ceramic materials belong to the group of perovskites according to their atomic structure. They are used in the form of thin films in the electronics field and can, for example, be found in memory chips used in cash cards. Their attractive functional properties are however only guaranteed if the material sticks to the chemical formula down to the atomic level. Previously, one could only measure the mean occupation of the atomic sites with the individual elements by chemical methods. This turned out to be not sufficient for a reliable materials characterization. Taking advantage of the novel imaging modes applicable in the corrected TEM a new measuring technique was developed by which the oxygen distribution over particular atomic sites can be measured very precisely. Thus it was found that, employing current thin-film production techniques, a high density of defects is formed in which the chemical formula is violated and a third of the oxygen positions is left vacant. This changes dramatically the electronic properties there. The corresponding publications in prestigious scientific journals found a lot of interest, and these results will help to improve thin-film deposition techniques to achieve better materials and superior device performance.

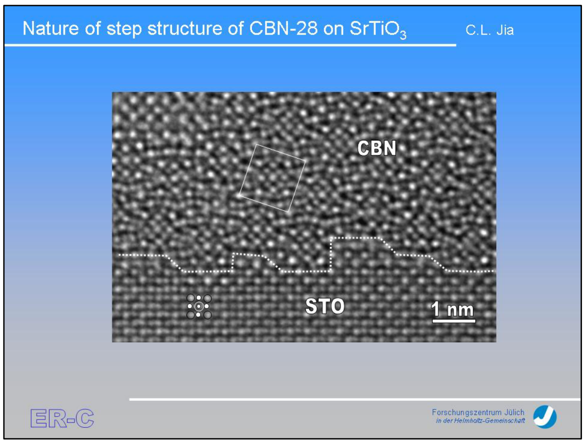

Fig 35

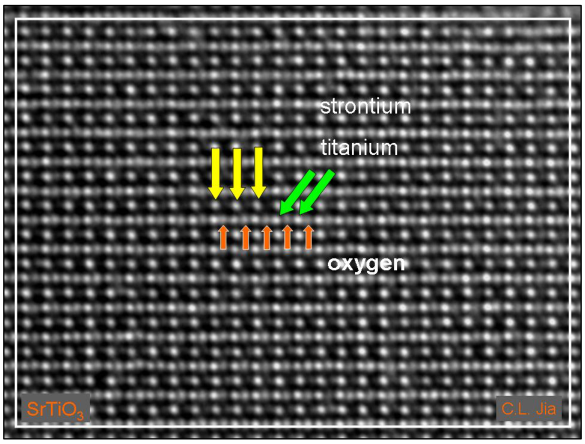

Fig 36

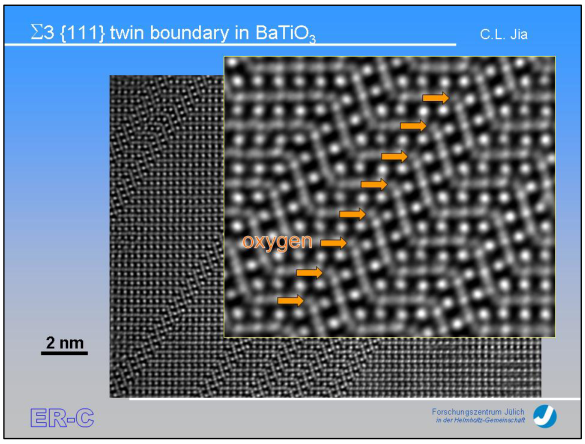

Fig 37

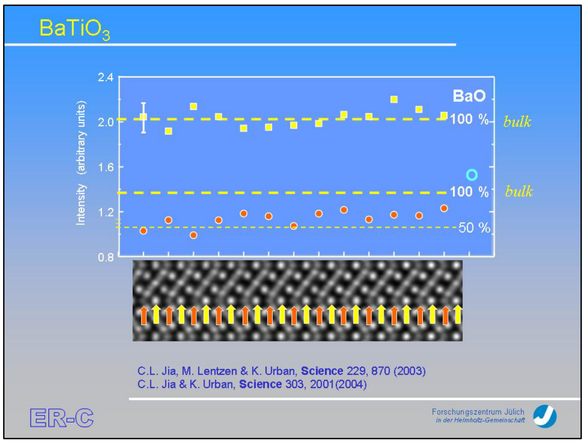

Fig 38

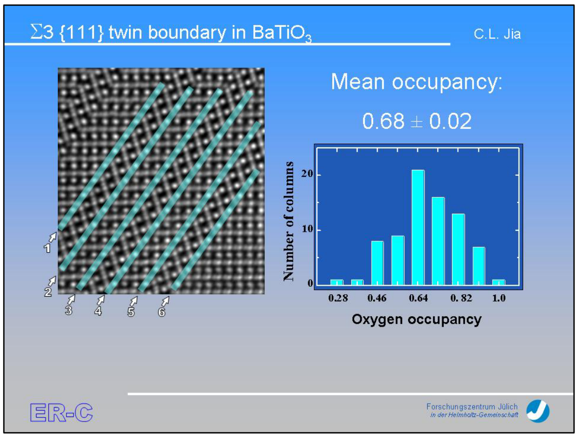

Fig 39

〈Fig 35〉〈Fig 36〉〈Fig 37〉〈Fig 38〉〈Fig 39〉 A second example of the innumerable important applications, which can only be realized with the aberration-corrected electron microscope, is the measurement of atomic positions with an unprecedented precision in the picometer range. Just as a reminder: a picometer is typically a hundredth of the diameter of an atom.

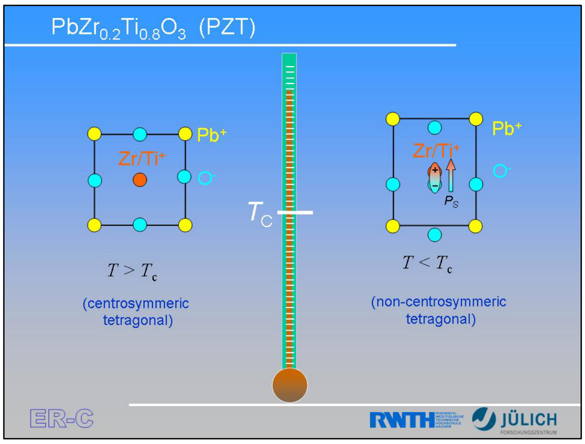

Fig 40

〈Fig 40〉 The Jülich group investigated the well-known compound $Pb(Zr_{0.2}Ti_{0.8})O_3$, a technically important ferroelectric material, used for instance in the form of thin films in ferroelectric memories.

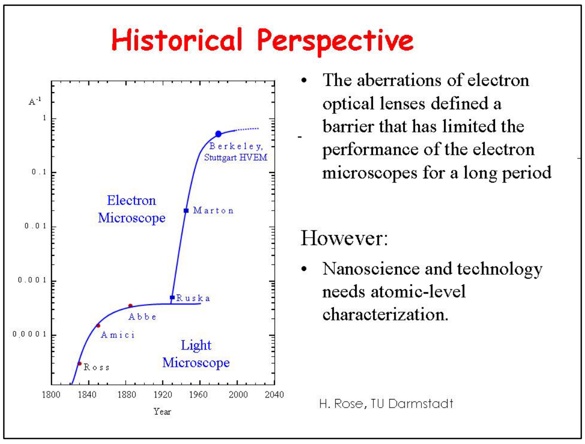

Fig 41

〈Fig 41〉 Employing aberration-corrected electron microscopy, they were able to study the atomic details of switching the direction of the ferroelectric polarization, i.e. the elementary process which allows one to write and store information into the material. Over and above the new insight that can be obtained this way with respect to the atomic background of technologically relevant processes, this study demonstrated that it is now possible to derive and measure physically relevant materials properties on the basis of observations atom for atom.

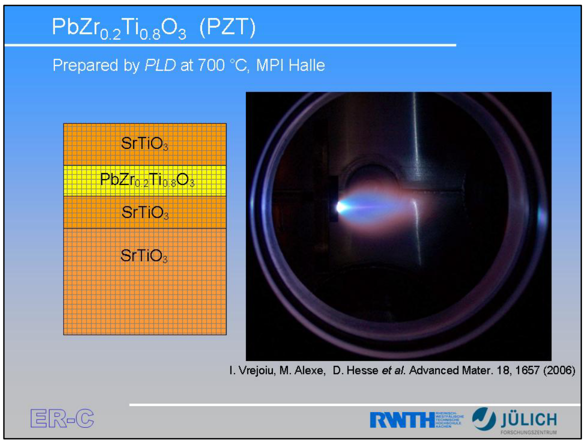

Fig 42

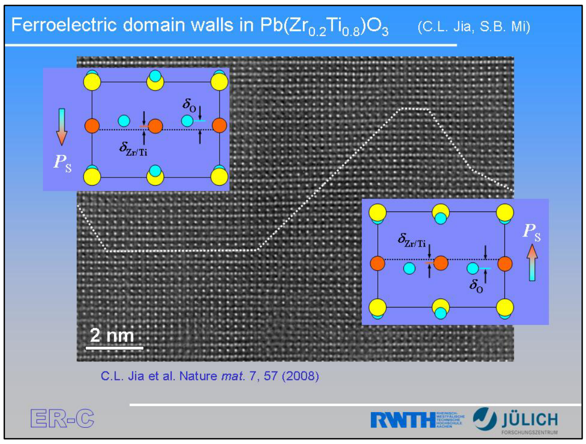

Fig 43

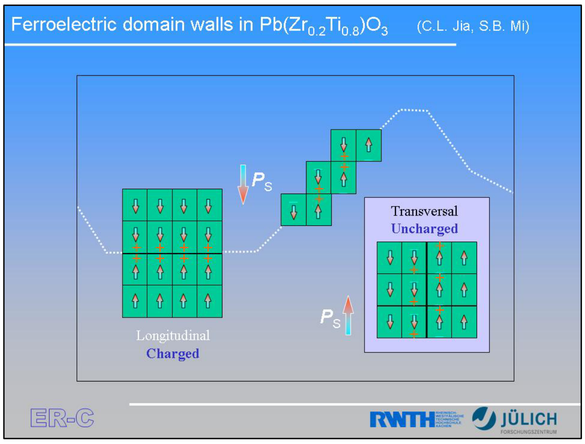

Fig 44

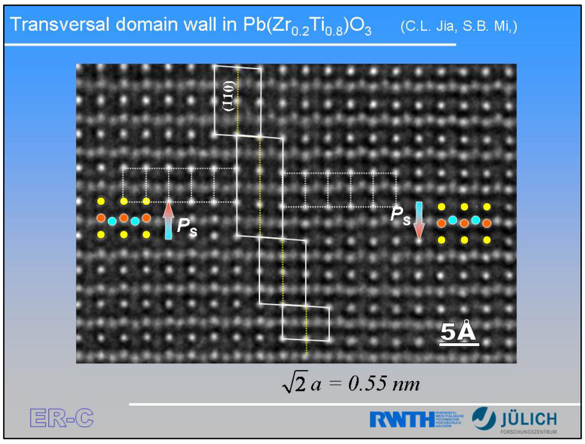

Fig 45

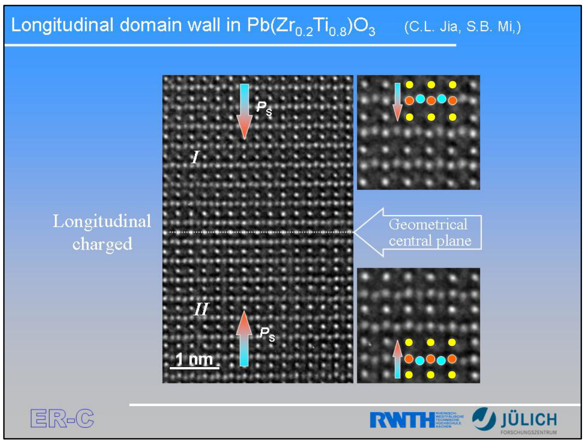

Fig 46

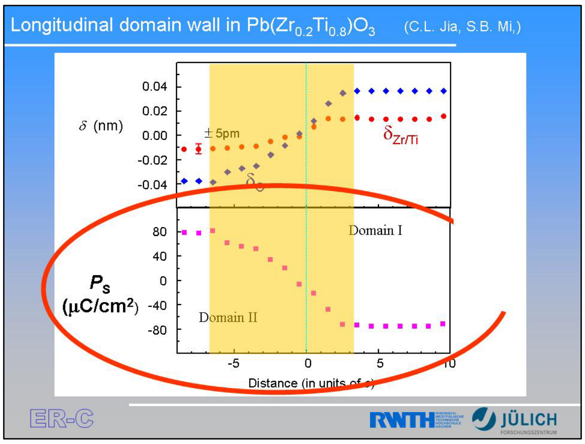

Fig 47

〈Fig 42〉〈Fig 43〉〈Fig 44〉〈Fig 45〉〈Fig 46〉〈Fig 47〉 This means that about fifty years after Richard Feynman's famous lecture the materials scientists dream has become true, thanks to aberration-corrected electron microscopy.

5. Impact on jobs

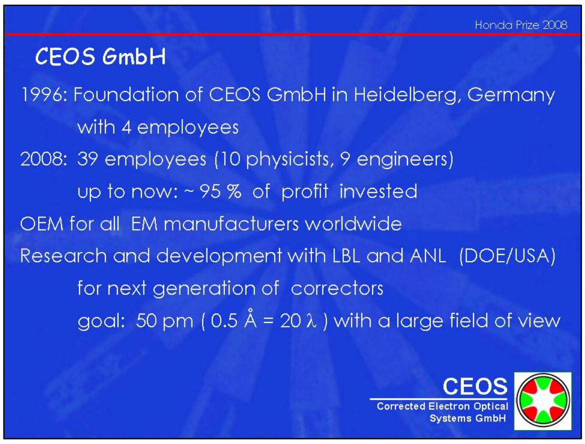

Fig 48

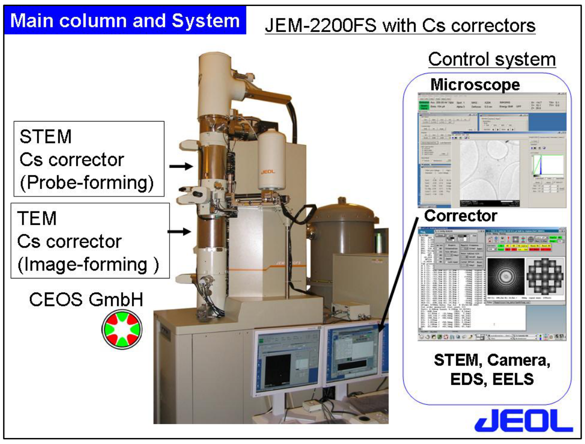

Fig 49

〈Fig 48〉〈Fig 49〉 In 1996, Max. Haider and Joachim Zach founded the company CEOS GmbH (Corrected Electron Optical Systems) at Heidelberg.

Fig 50

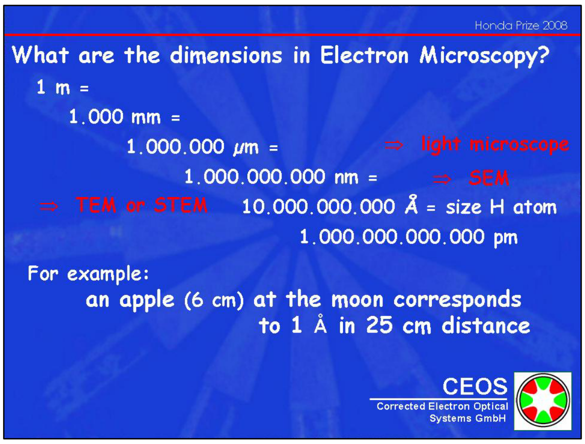

Fig 51

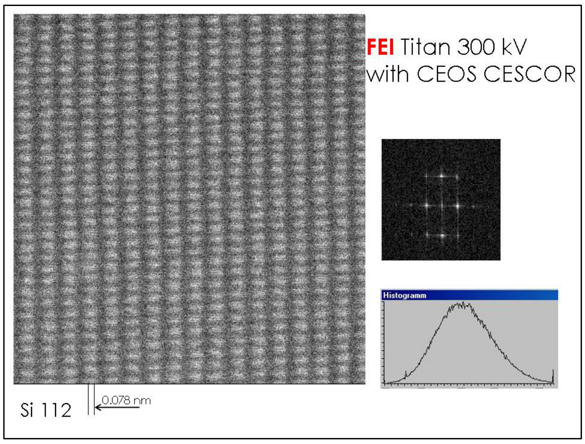

Fig 52

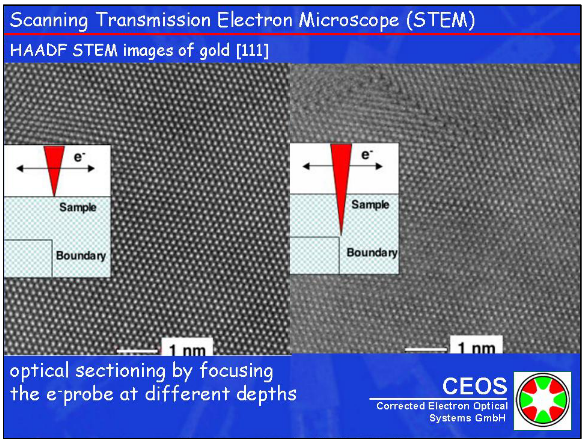

Fig 53

〈Fig 50〉〈Fig 51〉〈Fig 52〉〈Fig 53〉 This company focuses on research, development and production of advanced optical components for modern and future generations of transmission, scanning transmission and scanning electron microscopes. Among these are aberration correction systems and electron monochromators, and the company is continuously developing even more complex components for future generations of instruments. Financially, this start-up was supported by an order for the development of a spherically and chromatically corrected scanning electron microscope by the Japanese company JEOL. Currently besides JEOL other customers of CEOS are the Japanese company HITACHI, the American-Dutch company FEI and the German ZEISS company. CEOS has now generated almost forty solid jobs. The world's first spherical-aberration corrected transmission electron microscope has become the "mother" of an entirely new generation of commercial aberration-corrected TEMs, and currently the production capacity in industry is hardly sufficient to satisfy the extraordinary demand.

Fig 54

〈Fig 54〉 Unfortunately, the flourishing electron optics industry and the research laboratories applying the new technology are facing the problems arising from an especially tense labor market. Since one and a half decades universities worldwide have cut their education and scientific working capacity in the field of electron optics. As a result, there are by far not enough physicists and engineers to satisfy the urgent need for specialists in electron optics technology, and the research laboratories both in industry and in research can hardly find the experts to realize the new opportunities offered by aberration-corrected electron microscopy. It is hoped that the particular attention the new technology is appreciating recently in science policy and in funding institutions will trigger a corresponding initiative in academic education and training.

6. What does the future hold?

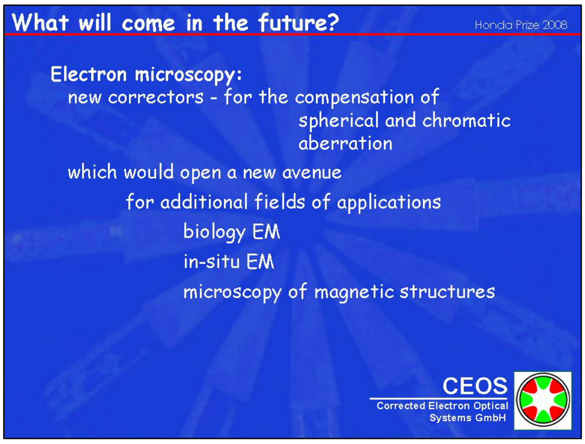

Fig 55

〈Fig 55〉 After spherical-aberration correction has successfully been accomplished and while the new generation of aberration-corrected instruments is in the process of conquering the science laboratories, is there still space for improvement?

Today the best instruments offer a resolution of 0.08 nm and a precision of the measurements of atomic positions of a few picometers. Can one do better? In order to answer this question, we have to explore what is currently limiting the optical performance of these instruments.

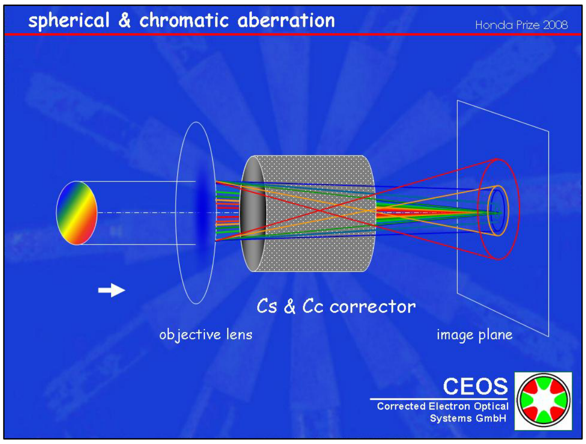

Fig 56

〈Fig 56〉 In any case, there is always an effect on resolution by mechanical vibrations and other instabilities of the microscope column reacting sensitively to environmental effects such as noise, vibration of the microscope foundation or temperature instabilities. In addition, there are effects due to magnetic stray fields which are everywhere in a modern technical environment. Measures have to be taken to reduce these disturbances to the lowest technically feasible level. Aside from these effects there are electronic and optical limitations. These arise from the finite energy spread of the electrons leaving the electron source, instabilities of the electrical supplies of the electron accelerator, the electromagnetic lenses, the stigmators and deflectors and the like.

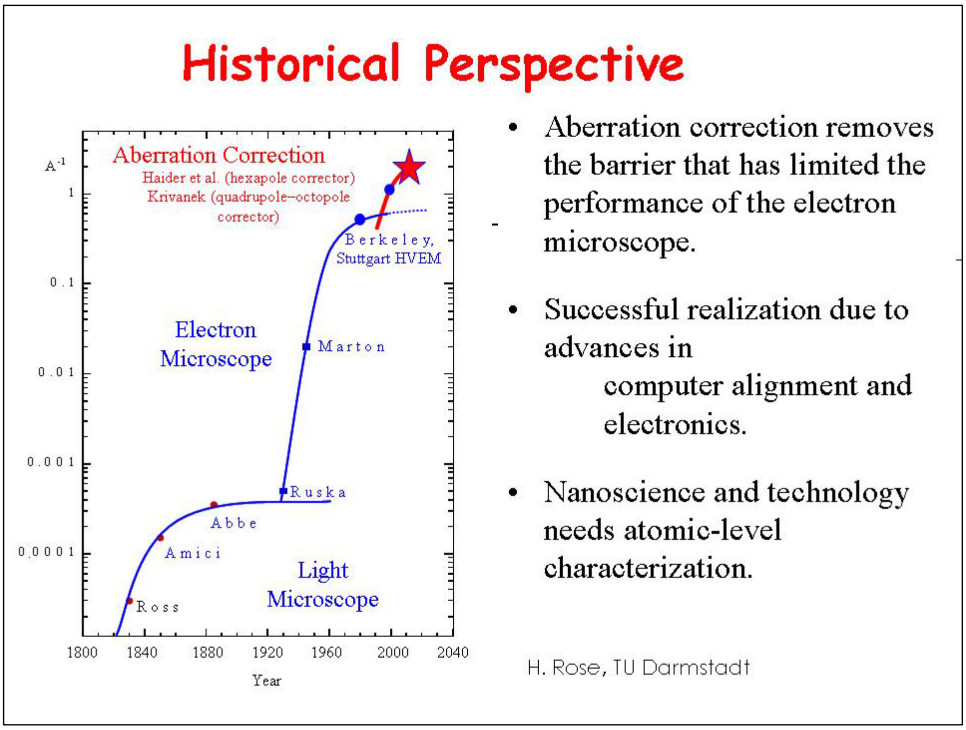

Fig 57

〈Fig 57〉 It has to be pointed out that so far only the spherical aberration has been corrected. However, in order to advance any further, the chromatic aberration of the microscope's objective lens has to be corrected as well. There are two ways to reduce the effect of chromatic aberration. The first is to reduce the energy spread of the electrons by placing an electron energy filter, a monochromator, behind the electron source. The second is to construct a novel multipole correction element which simultaneously corrects both spherical and chromatic aberration. At the same time all the modern technical means have to be exploited to electronically stabilize currents and voltages.

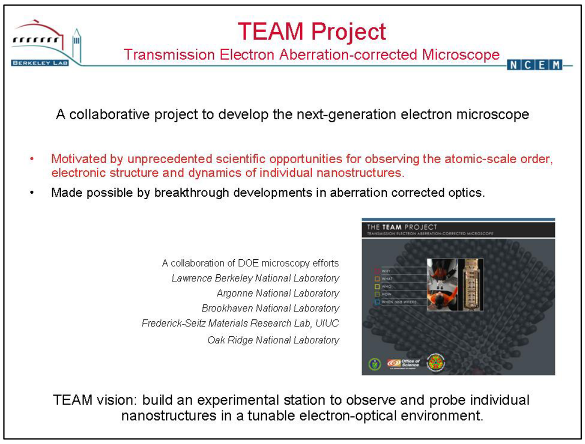

Fig 58

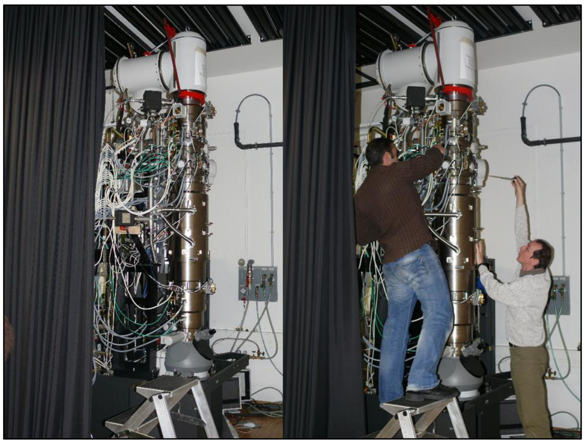

Fig 59

〈Fig 58〉〈Fig 59〉 A project, within which such improvements are in the process of being realized, is the so- called TEAM project of the United States Department of Energy. One of the goals of this endeavor is to construct an instrument offering a resolution of 0.05 nm (= 50 pm). This instrument should in principle allow measurements on atomic positions with a precision of 1 pm.

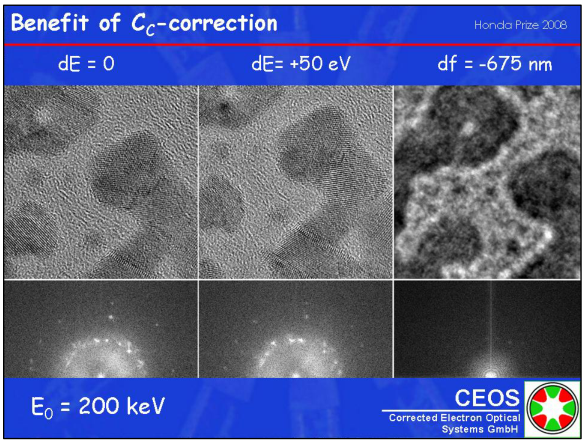

Fig 60

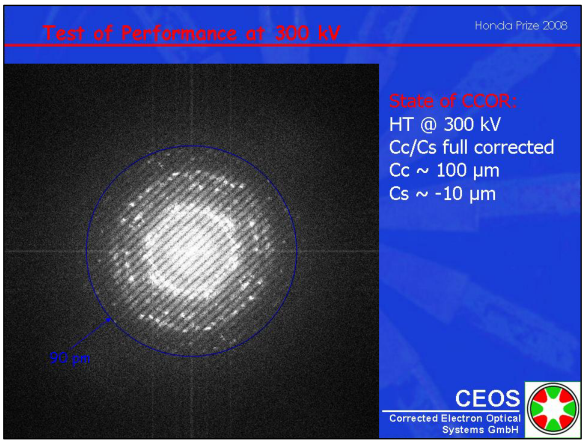

Fig 61

〈Fig 60〉〈Fig 61〉 The project is to a large part carried out by CEOS. To realize its very ambitious goals, requirements for the correction system must be satisfied which are substantially more complex than those for present spherical-aberration correctors. The combined chromatic and spherical aberration corrector is about three times as long as the present double-hexapole corrector. The corrector designed by CEOS consists of 17 optical elements, and for the operation more than 150 current drivers are required instead of the 28 for the hexapole corrector. Chromatic correction requires an extraordinary unprecedented relative stability of about 1: 100,000,000 for the voltage and the current supplies. Such extremely stable supplies were only a few years ago considered as not feasible and their realization by CEOS involves to most recent technical achievements in electronics. The TEAM instrument is currently installed at the laboratories of CEOS in Heidelberg. The simultaneous correction could already be demonstrated successfully, and the project is on its way to realize its objectives on time. With this development science will be offered an instrument which, with respect to the first generation of aberration-corrected instruments, will provide another almost doubling of the primary resolution.

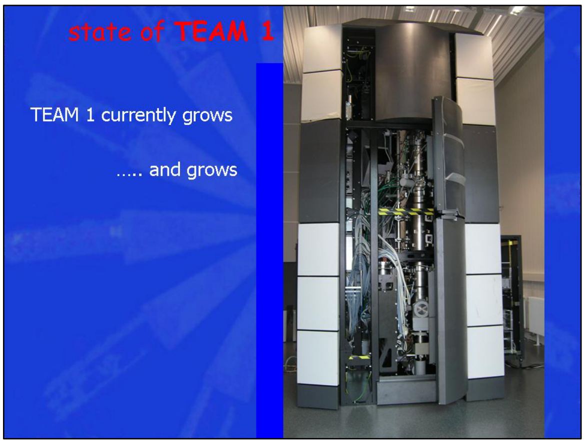

Fig 62

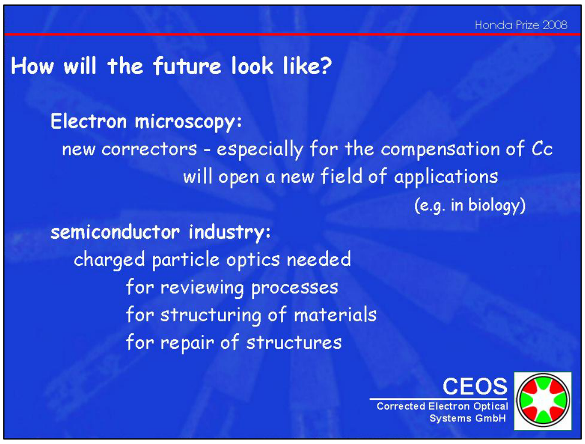

Fig 63

〈Fig 62〉〈Fig 63〉 However, resolution is not an end in itself, this type of instrumentation will be employed in research on topics such as CO_2-free power plants, high-efficiency solar cells, artificial photosynthesis, biological cell functions and molecules and molecular aggregates for life. It will help to tackle the problems for a better and healthier world and it will contribute to open the gate to a better understanding of the world we are living in today with the goal to hand it over intact to our children and future generations.

Acknowledgments

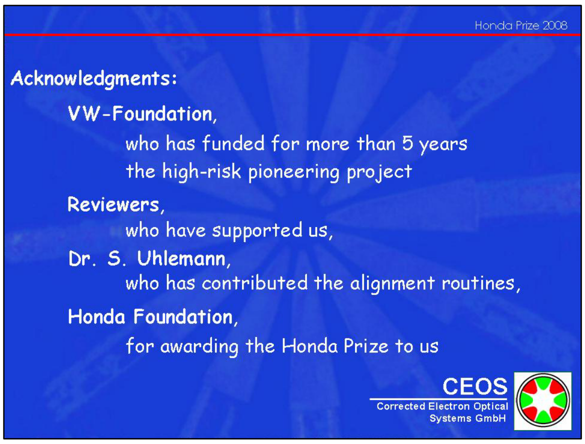

Fig 64

〈Fig 64〉 The success of our project was made possible by the generous financial support of the Volkswagen Foundation and by their reviewers who recommended the project for funding. In order to reach our goal many sub-tasks had to be solved, which were essential for the successful completion of the very demanding project. For example, the development of an efficient alignment procedure would not have been possible without the continuous efforts of Stefan Uhlemann. We are highly honored by the decision of the *Honda Foundatio*n to award us the prestigious Honda-Prize. We would like to express our sincere appreciation for this highly recognized award.

〈MEMO〉

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