High-Tension Determination of Transmission Electron Microscopes by means of Convergent-Beam Electron Diffraction (CBED)

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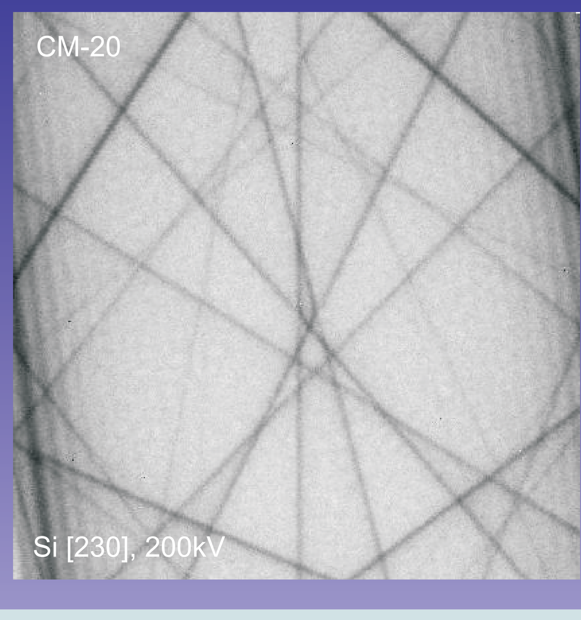
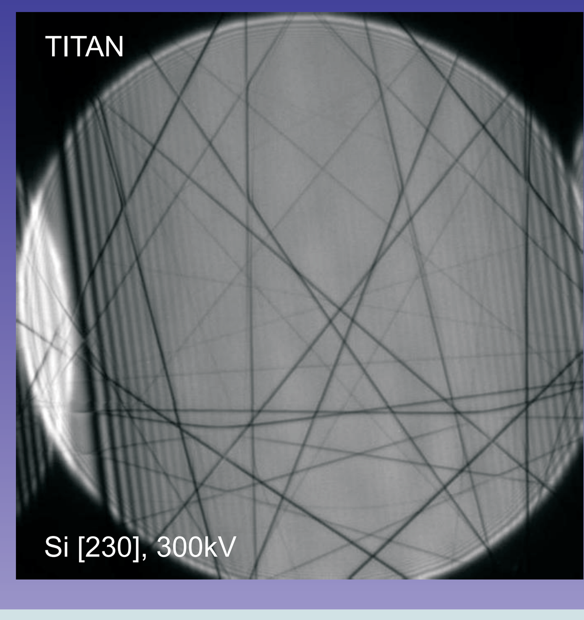
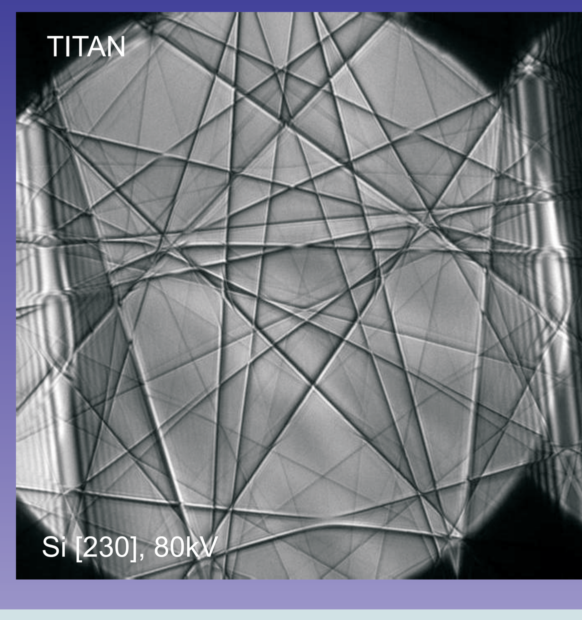
MOTIVATION

CBED is a well established method for local strain and lattice parameter measurements [1-4]. Application of line detection algorithms based on Hough transformation allows HOLZ-Line detection with accuracy far below one pixel [3, 5] and thus line detection does not contribute significantly to the resulting accuracy of lattice parameter measurement. The main sources of errors, physically limiting the accuracy, are the image noise [5] and the systematic errors. The latter include HT drift during experimental time-slice and thickness determined shift of HOLZ lines [6].

In this work we evaluate and compare the long term HT stability for CM-20 and TITAN 80-300 microscopes for the range of operating conditions. Absolute value of HT is determined by CBED method exploiting Si [230] zone as a standard. As far as absolute values are measured, the a priori knowledge of the measurement errors became a key issue. The roadmap for thorough evaluation of measurement accuracy including SNR related errors and thickness correction is suggested and approved.







METHODS

Experimental Setup

- **Investigated sample:** Si plane-view sample in (110)-orientation, lattice constant 5,431Å to get large areas without deflections.

- **Analysed projection** : [230] incident direction.

- **Exposure techniques:**

CM-20 (200kV): LACBED was used to filter out thermal diffuse scattered electrons to increase SNR.

TITAN (80kV/300kV): Normal CBED was used. To filter out inelastic scattered electrons and increase SNR, images were ZL filtered by using GIF.

Prerequisites

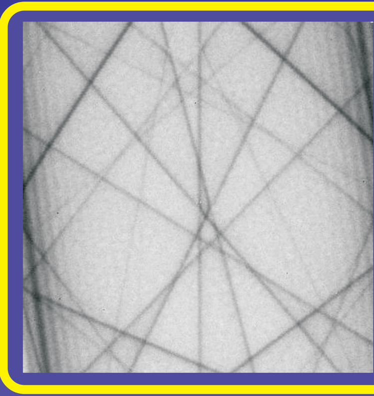
- Absolute HT value (energy at sample location) of interest to lattice parameter determination by comparing HOLZ-Line positions of experimental and calculated CBED patterns.

- Reduction of systematical errors: Usage of dynamical calculations and consideration of the thickness-depending HOLZ-Line position by thickness correction.

- Errors of HT values important to evaluate influence of HT accuracy on lattice parameter determination. Therefore the total error of the experimental image was calculated, considering SNR.

Workflow

- HT determination by calibration
- Error calculation
- Thickness correction



Experimental CBED/LACBED patterns taken.

Signal to Noise Ratio (SNR) of experimental pattern for each line [5]:

$$SNR = \frac{\mu_{line} - \mu_{bgr}}{\sigma} = \frac{S}{\sigma} \quad (1)$$

Statistical error ΔL_{stat} of each line depending on SNR, line width W and line length L [5]:

$$\Delta L_{stat} = 0.66 \cdot \sqrt[3]{\frac{W^4}{SNR^2 L}} \quad (2)$$

Line detecion and calculation of line-ratios using Hough-transformation.

Geometrical error ΔR of line-ratio $R = d_1/d_2$ depending on Hough-transformation accuracy ΔL [5]:

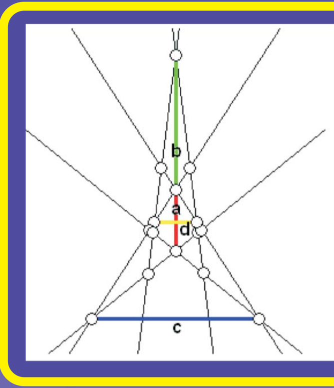
$$\Delta R = \frac{1}{d_2^2}\sqrt{d_1^2\left(\frac{\Delta L_1^2 + \Delta L_2^2}{\sin^2\gamma_1} + \frac{\Delta L_3^2 + \Delta L_4^2}{\sin^2\gamma_2}\right) + d_2^2\left(\frac{\Delta L_5^2 + \Delta L_6^2}{\sin^2\gamma_3} + \frac{\Delta L_7^2 + \Delta L_8^2}{\sin^2\gamma_4}\right)} \quad (3)$$

total error of experimental pattern

Error of single HT value (gaussian error):

$$\Delta U = \left|\frac{dU(r)}{dr}\right|_R \cdot \left(\Delta R_{geo} + \Delta R_{SNR}\right) + \Delta U_{Curve} \quad (4)$$

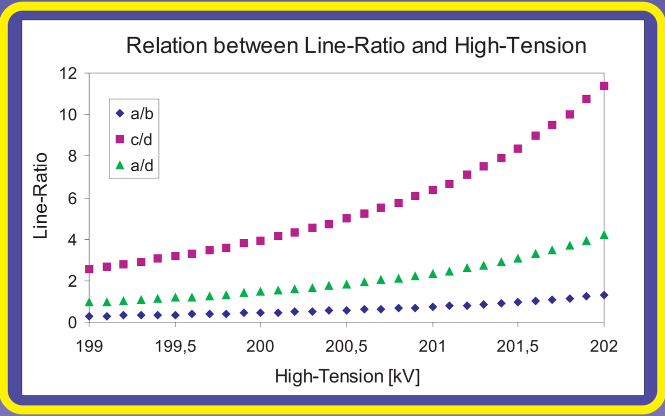
Dynamical calculation of CBED patterns at constant sample thickness and variation of HT using MBFIT program [7]:



Line detection and calculation of line-ratios using Hough-transformation.

Geometrical error of line-ratios depending on Hough-transformation accuracy.

Applying line-ratios to HT calibration curves



HT calibration curves U(r)

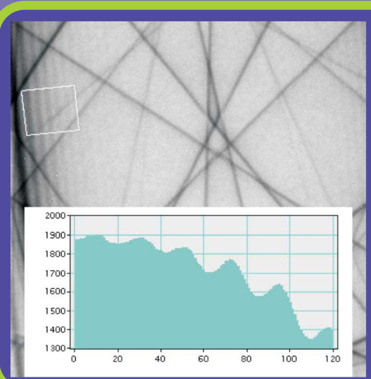
calibration error ΔU_{Curve} (std. dev. of regression U(r))

Single HT value U for each line-ratio.

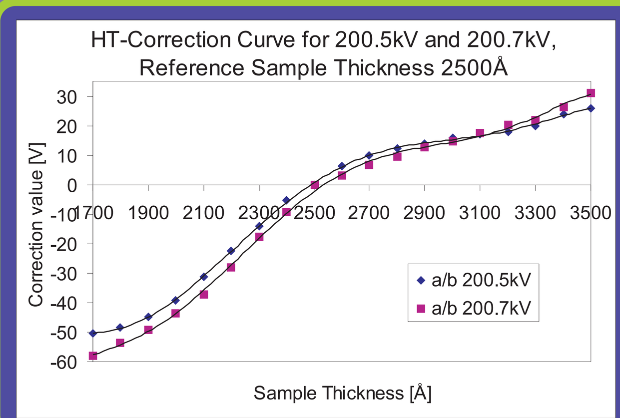
Corrected HT values U^{cor}.

Correction value

Dynamical changing of HOLZ-Line position with sample thickness.



Determination of sample thickness by analysing intensity oscillation of rocking curves.



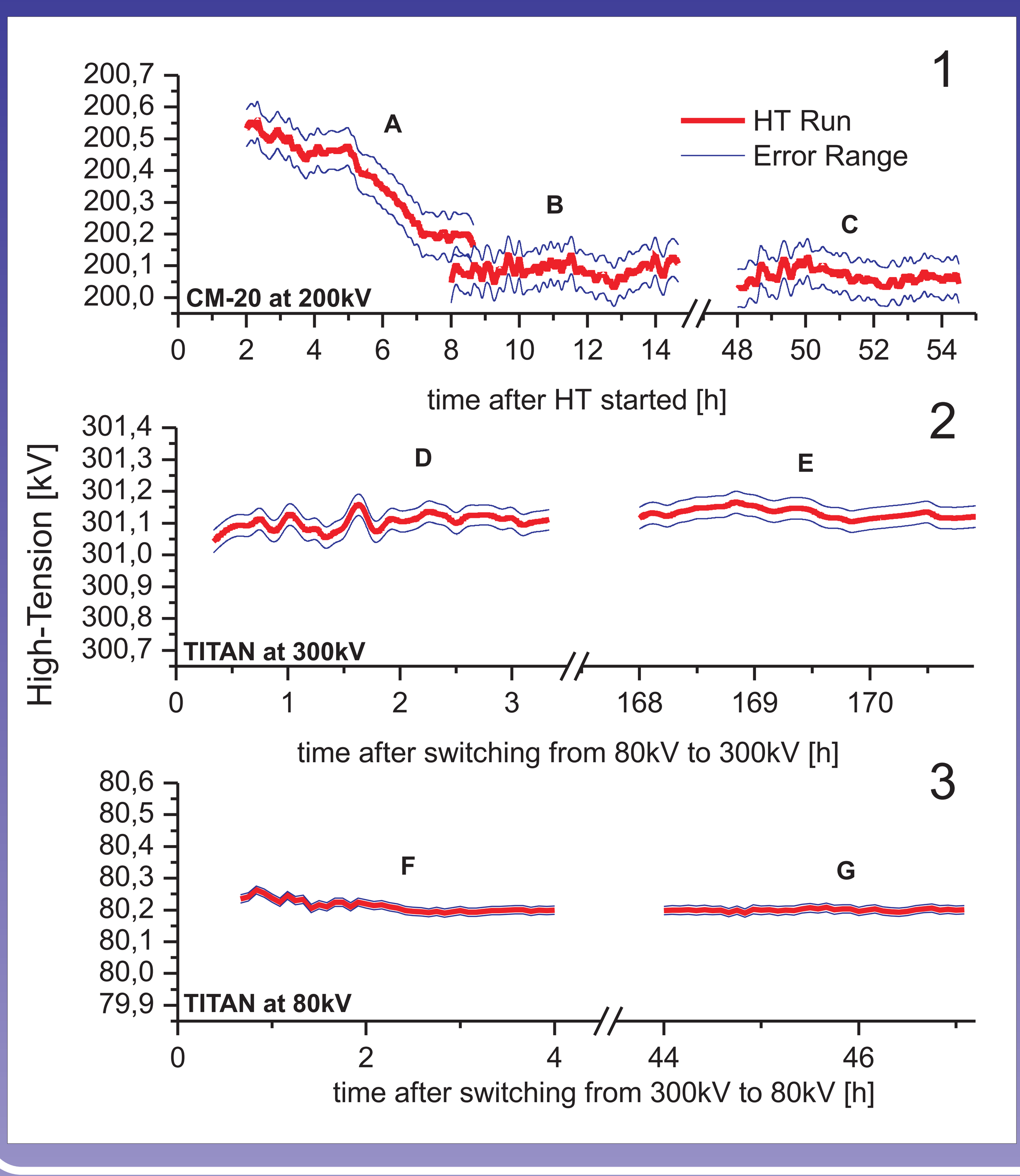
1. Calculation of CBED-patterns by variation of thickness at constant HT (here 200.5kV).
2. Applying line-ratios to HT-calibration curves.
3. Differences of HT-values to 200.5kV give HT-correction values depending on sample-thickness.

Final HT value for each image and error (weighted average and error of weighted average):

$$\overline{U}_{gew} = \frac{\sum_{i=1}^{3} g_i U_i^{cor}}{\sum_{i=1}^{3} g_i} \qquad g_i = \frac{1}{\Delta U_i^2} \qquad \Delta\overline{U}_{gew} = \frac{1}{\sqrt{\sum_{i=1}^{3} g_i}} \quad (5)$$

RESULTS

HT Runs for CM-20 and TITAN Microscope



- Errors of the HT values in diagram 1: ±55V.

- A: Large HT down-drift directly after HT started. After 7h HT stable.

- B, C: As expected in respect of A, HT stable and constant within the errors 8h/48h after HT started. HT-jump (~100V) between A and B probably because of temperature changes between different days.

- Errors of the HT values in diagram 2: ±34V.

- D: Small up-drift (~50V) reaching HT-Level of E. HT oscillations observed until 2h after switch-over.

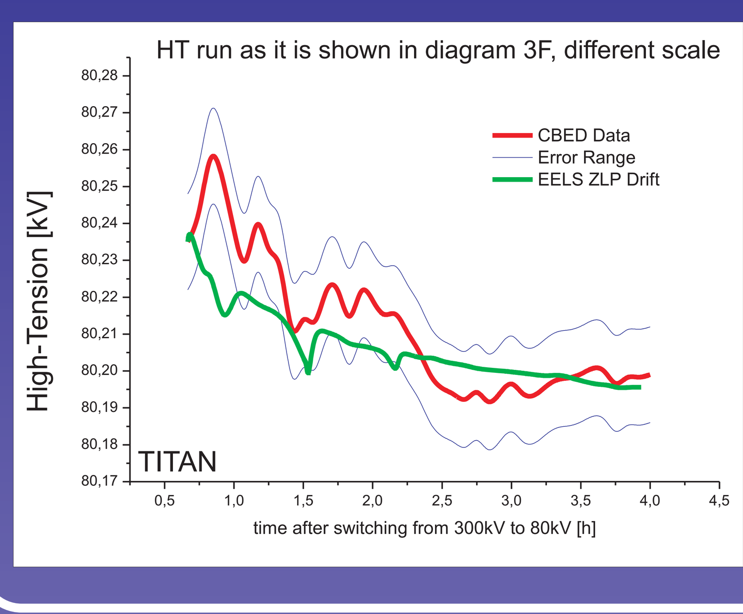
- E: Very stable HT run without drifts and constant within the errors.

- Errors of the HT values in diagram 3: ±12V.

- F: Small down-drift (~40V) with HT oscillations until 3h after switch-over. Afterwards HT-Level of G reached. HT very stable without drifts.

- G: As expected in respect of F, HT very stable without drifts and constant within the errors.

Comparison of CBED and Zero-Loss-Peak Drift



- Simultaneously recorded drift of ZLP plotted in addition to CBED data.

- Only changes of the ZLP known, not the absolute HT values.

- First ZLP position calibrated to 0V and fitted to the first value of the CBED run.

- ZLP drift confirmes HT down-drift determined by CBED. The peaks in the ZLP run indicate calibration problems of the GIF during HT oscillations.

Conclusions

It was shown that CBED provides a possibility to determine absolute high-tension values (electron energy at the sample location) of the microscopes with an accuracy in volt-range.

For the microscopes examined a relaxation period was found directly after HT switch-on (CM-20) and after 80kV/300kV HT switch-over, during which HT oscillates and drifts in the range of hundred volts. After this relaxation period the value of HT remains stable within the measurement error (see formula 5).

In particular such stability of the instruments allows to neglect the systematic error due to HT drift during lattice parameter measurements by means of CBED because HT drifts introduce an error which is much smaller than the one determined by the image noise.

References

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Acknowledgement

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