

## User Guide to Operating the Zeiss Sigma 500VP SEM

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### Sample Preparation

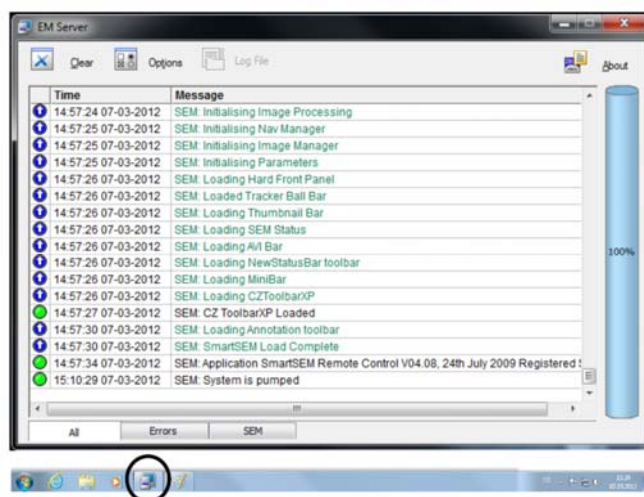
- Put on a pair of gloves. Samples should be clean, as small as possible, stable in vacuum. If needed, clean samples, stubs and the specimen holder with isopropanol and kimwipe papers.
- Mount fragment or powder samples firmly on Al-stubs using C-tape. Fix stubs onto the ZEISS specimen holder. 9 stubs may be loaded. Mount thin sections directly on the specimen holder.
- Make maps if necessary to be used to locate each sample in the SEM.

*Warning* Salt and oils from fingerprints will contaminate the FESEM vacuum and your samples if you do not wear gloves. Fingerprints can be removed with isopropanol and kimwipe.

### Starting ZEISS SmartSEM Program

First, the tool needs to be activated by logging onto the Zeiss SEM in FOM system.

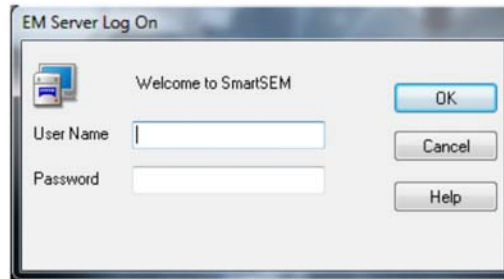
The EM Server, implementing the internal communication between software and hardware, is always running too. It is sometimes minimized to a small element (icon) on the right side of the Windows task bar.



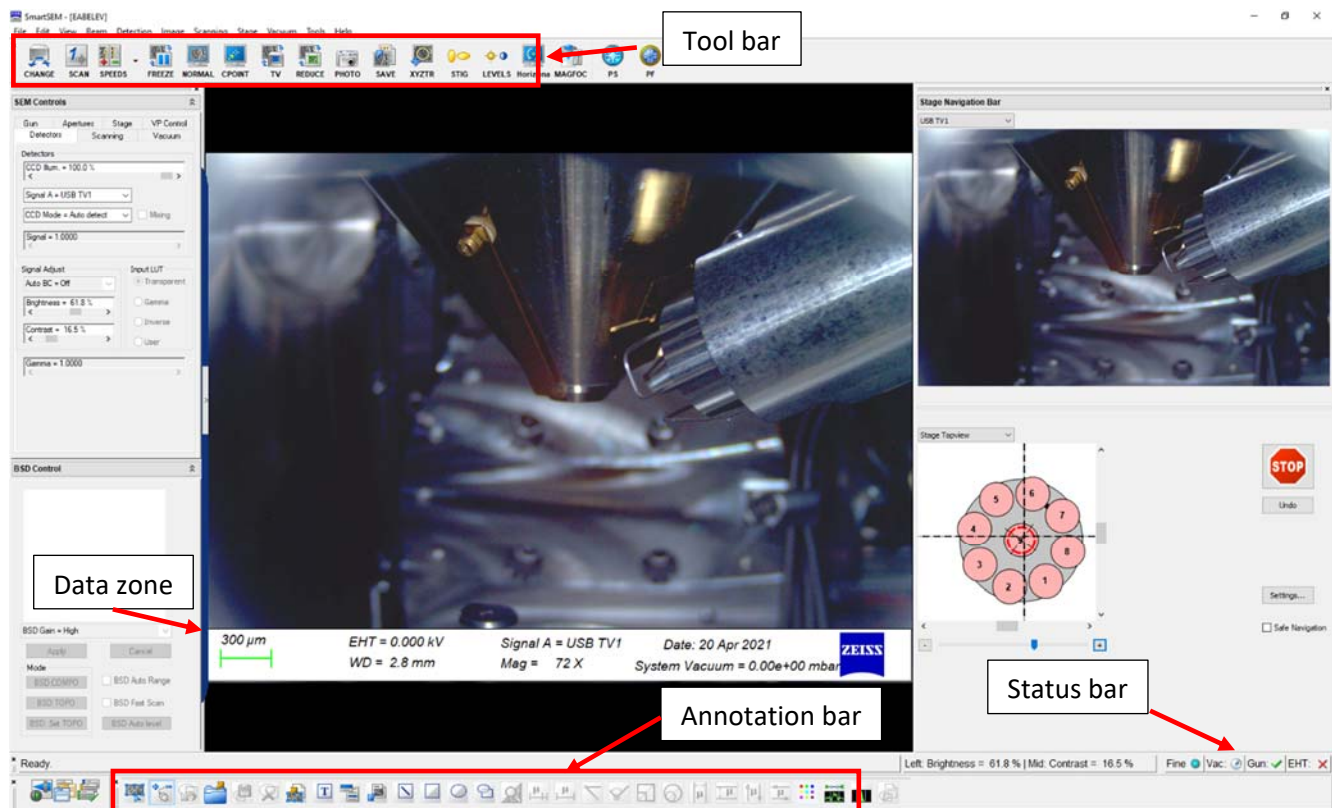
Note: If the EM Server was closed by the last User, Starting the **ZeissSmartSEM** software will first reload the EM server and recover software/hardware communication.

Double click on **ZeissSmartSEM** icon.

Alternatively, select **Start/Programs/SmartSEM/SmartSEM User Interface**. The EM Server Log On dialogue appears.



By logging, the SmartSEM user interface opens and is ready to operate the tool. By default, a TV view inside the specimen chamber is shown.



The data zone is a special and useful group of annotation objects which are used to display useful parameters. If it is not open, select **View/Data Zone/Show Data Zone** from the menu. Alternatively, type **<Ctrl+D>** to toggle the data zone.

### Loading the Specimen Holder

- VENTING: Make sure EHT is off. Click VAC → VENT button on the toolbar, then wait approximately 1 minute.



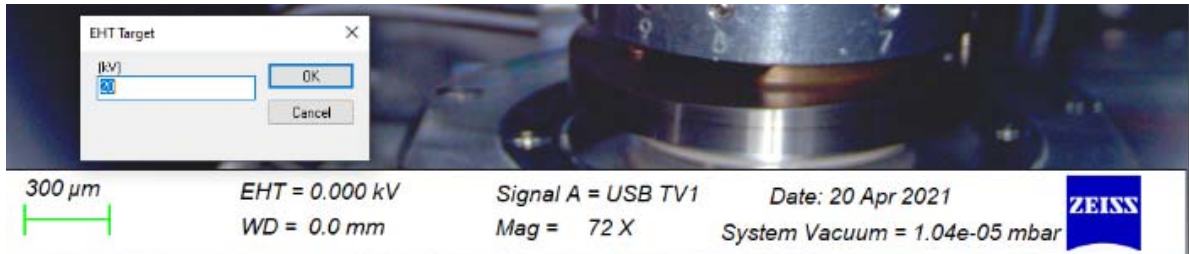
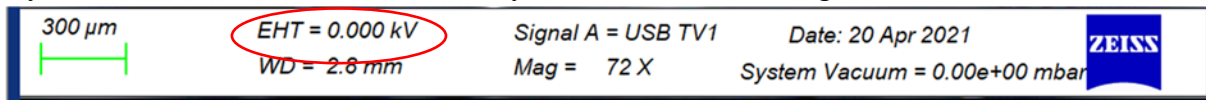
- OPENING CHAMBER: Use the handle to open the door. Take the carousel (sample holder) out and close the door completely. Place sample stubs into designated holes on the carousel and screw to tighten. Open chamber, put holder back into SEM, and close the door.

- PUMPING: Click the VAC → PUMP button on the toolbar. Wait for (Vac status = ✓, System Vacuum ~ 5.0e-5) in the data zone and Vac is green.

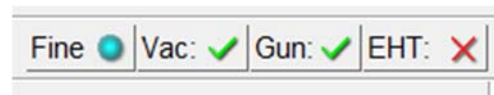


### Switch ON the EHT

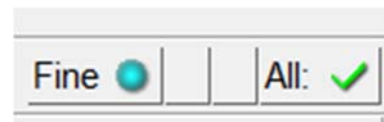
- ‘EHT’ means the extra-high tension acceleration voltage. This voltage must be applied to the gun to make the gun emit electrons.
- By double click on Data Zone EHT=0, you will be able to change EHT to the desire value.



- After adjusting EHT, go to EHT tab and Turn EHT On.

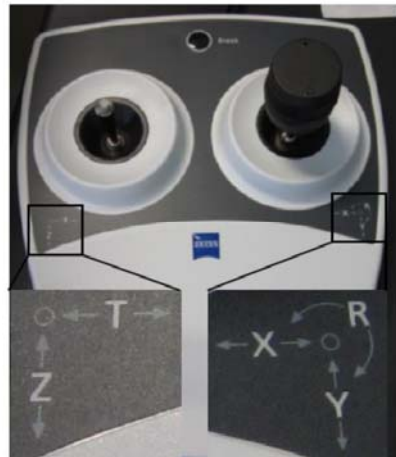
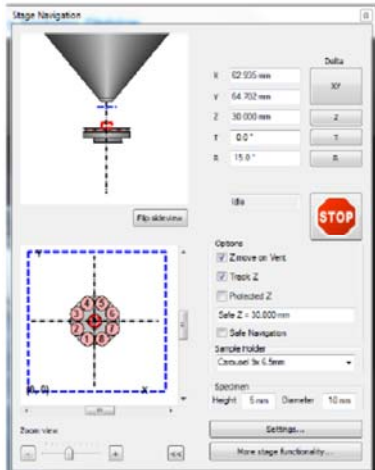


- Wait until EHT turns ✓ and EHT=value on the Data Zone.



### Stage settings and motions

- Adjust specimen height with joystick. Move to desired sample slot by using X/Y/R joystick (hardware) or double-clicking carousel graphic (software). Adjust height of stage using Z joystick, noting that the Z-movement automatically stops at the blue line drawn in the Stage Navigation Bar.



Left joystick (Z and T) has to be used with great care: a risk of damaging the tool and/or the specimen is present if hitting the objective lens while driving the stage. Watch the moving stage in TV mode.

- For correct carousel adjustment see “Setting...” → Sample Holder Gallery.

#### Stage Navigation Settings

**Behaviour**

Stage XY+Z

Track Z

On Goto Point

On Centre Point

**Safety**

Z move on Vent

Protected Z

Safe Z = 0.000 mm

Safe Navigation

**Camera**

CCD Illum. = 100.0 %

CCD Brightness = 61.8 %

CCD Contrast = 16.5 %

CCD Resolution = High

CCD Mode = Auto detect

**Stage Enable / Disable**

Joystick Disable

Stage Disable

**Navigation**

Go to \$NavCam on Vent

**Sample**

Carousel 9x9mm

Sample Holder Gallery..

**Specimen**

Height 1 mm

Diameter 10 mm

Focus on the highest part of the specimen and press <Shift + F2> first.

Calibrate specimen height

**Compucentric**

Compuc. Mode = Off

Calibrate compucentric rotate

Calibrate compucentric tilt

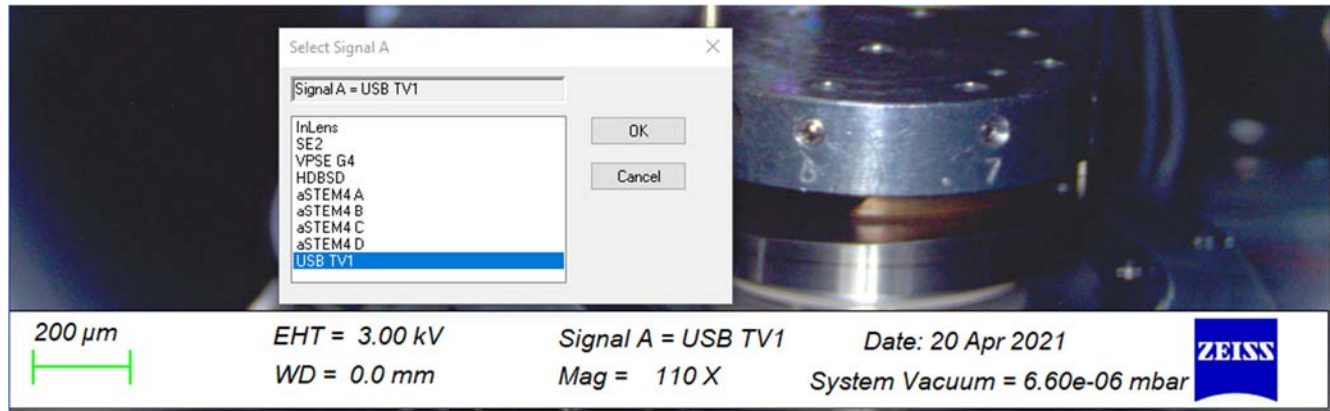
Stage Topview

B TV1      Date: 20 Apr 2021      **ZEISS**

System Vacuum = 9.65e-05 mbar

## Initial Image Adjustments

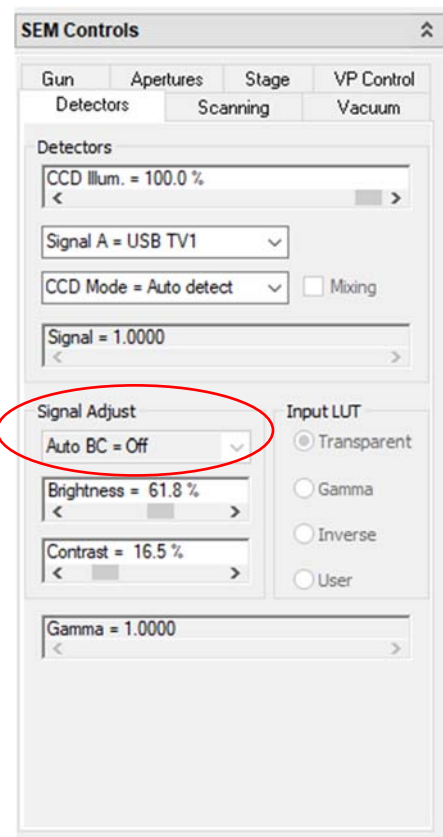
- Choose Detector: always start imaging with SE2 detector. Double click on “Signal A = ..” choose SE2



- Optimal imaging distance for SE2 detector 5-8mm (WD), for InLens detector (3-4mm).
- Use magnification knob on keyboard panel to set magnification **to a minimum**. Adjust brightness and contrast using knobs on keyboard panel in order to see relevant features on your sample(s). Or put go to **SEM Controls** → **Detector** → **Auto BC=BC**. Aim for a starting working distance close to 6-8 mm. Make sure that the detector written in the data zone (“Signal A = ”) label matches the detector you are using.



Magnification



### ***Focusing Imaging***

- Identify region of sample to image. Zoom in as much as possible using magnification knob. Turn the Focus knob on the keyboard panel to focus as much as possible. Use the Stigmator X & Y knobs on keyboard to minimize image stretch/distortion upon moving in and out of focus.



### ***Aperture Alignments***

- Click the Wobble button on the keyboard. Use the Aperture X & Y knobs on keyboard panel to minimize image oscillations. If you can, it is recommended to use >10,000X magnification for aperture adjustments.

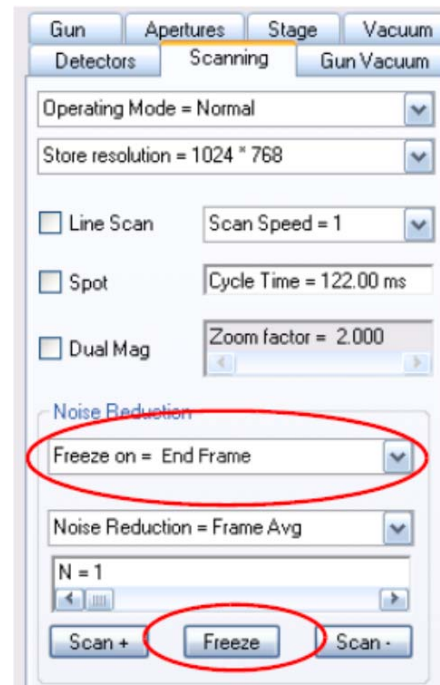


### ***Future Image Adjustments***

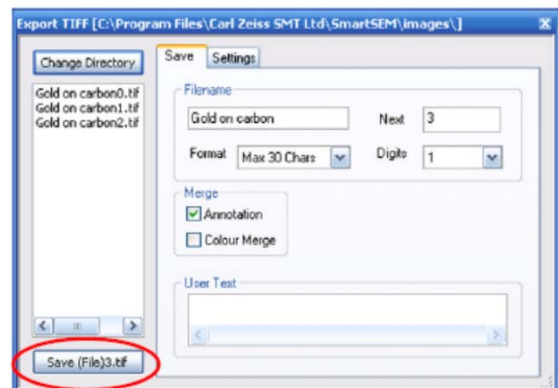
- Iterate steps 9 through 12 until you are satisfied with the image quality you see on the screen.

### *Saving Images*

- Choose Scan Speed 5/10 and Noise reduction=Line Average ~10-20 lines. Freeze (red dot in bottom right corner of data zone signifies ready to save). Save (TIF, JPG, BMP, all with or without Annotation). Make sure to navigate to your user directory before confirming the saving operation.



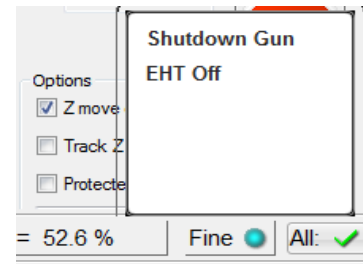
- Select **File > Save Image** from the SmartSEM menu.
- Enter a path and a file name.
- Click **Save...tif** to save the image file.



- To continue imaging, select **Image > Unfreeze** from the SmartSEM menu, or click **Unfreeze** on the Scanning tab.

### ***Taking sample OUT***

- Go to the **ChamberScope** view.
- Click **EHT** and hit **EHT off** to turn off the accelerating voltage.



- Click **VAC** and hit **Vent** to vent the SEM specimen chamber.
- Put on a pair of gloves.
- Once the chamber vented, open the door and remove the specimen holder off the stage.
- Close the chamber door immediately. Then click **VAC** and hit **Pump**. **The chamber must be pumped down at the end of a session.**
- Wait for a green tick for **VAC**.
- Go to **File** and click **Logoff**. Hit **Yes** to the two questions.