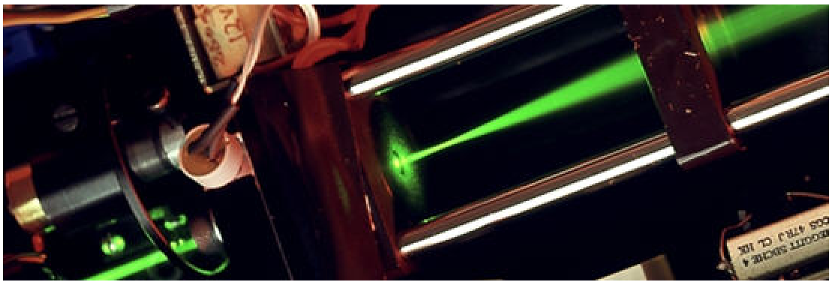
Guide to “The Risø Single Grain Laser OSL system”



DTU Nutech, Roskilde, Denmark
October 2016

Contents

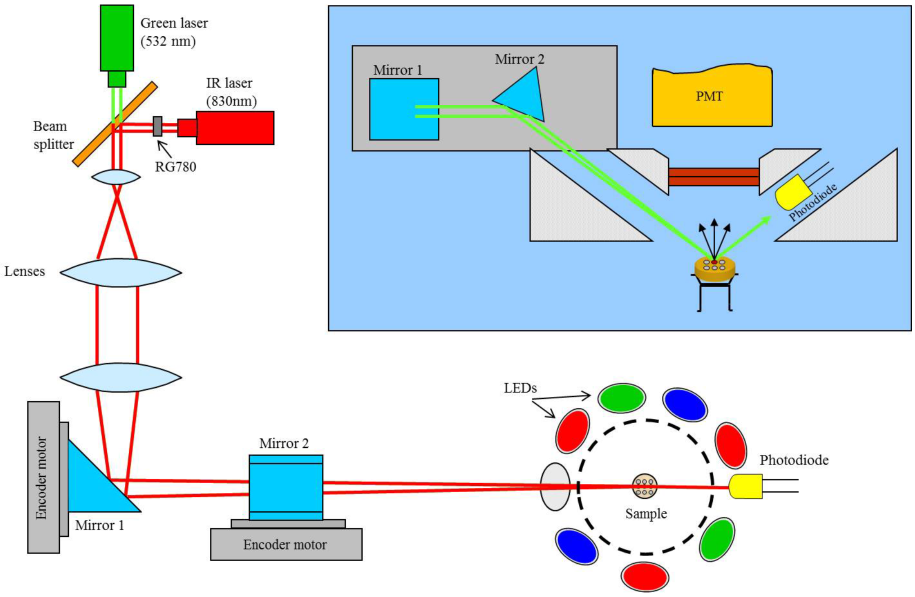


Figure 1: Schematic diagram of the single grain OSL attachment. Optical stimulation is achieved using a laser beam focused by three lenses. The position of the laser spot on the sample is controlled by moving two mirrors. **a)** Single grain OSL attachment seen from above. **b)** Cross-section of the single grain OSL attachment

1 The Single Grain OSL System

The Risø single grain laser OSL system is an attachment to the conventional Risø TL/OSL luminescence reader. This attachment enables routine measurements of sand-sized single grains. The sample is loaded into special aluminium discs each containing 100 grains in holes of known position on the disc surface. Irradiation and heating can thus be performed simultaneously on all 100 grains, whereas the OSL signal can be measured separately from individual grains by using a focused laser (beam diameter on the sample disc is $< 20\,\mu$m). This laser spot is steered to each of the grain holes in turn and switched on. The focused laser enables a high stimulation power and reduces the risk of optical cross-talk by ensuring that the entire spot enters the $300\,\mu$m diameter hole (discs with other hole diameters are available upon request). Only a small part of the grain will be stimulated directly by the laser beam, but internal reflection within the grain hole is assumed to provide a uniform illumination of the grain. The emitted OSL is usually detected by a photomultiplier tube that is shielded from the stimulation light by different glass filters. A schematic diagram of the single grain OSL attachment is shown in Figure 1.

A reader with a single grain attachment can be used in the same manner as a conventional Risø TL/OSL reader; e.g. using blue, green or IR LED stimulation. In a conventionally reader, a 3 mm thick quartz window is positioned between the measurement chamber and the OSL stimulation head. This quartz window serves two purposes: 1) it makes the measurement chamber air tight, and 2) it prevents evaporated silicon oil from reaching the stimulation optics. However, this quartz window must be removed when the *Single Grain Attachment* is to be used to allow the laser beam to enter the measurement chamber unhindered. This means that if silicon oil is used to prepare samples then it can reach the OSL stimulation head. Thus, if silicon is to be used it is important to re-insert the quartz window.

1.1 Single grain sample discs

The aluminium sample discs designed for mounting single grains are 1 mm thick and have a diameter of 9.7 mm (i.e. same surface area as the conventional sample discs). The individual grains are placed in 100 holes drilled into the surface of the sample discs. These holes are 300 μm deep by 300 μm in diameter on a 10 by 10 grid with 600 μm spacing between hole centres (see Figure 2, other hole sizes are available upon request). The preferred grain size for these discs is the $180 - 250\mu$m fraction[1]. The 100 sample holes are drilled using a CAD/CAM controlled computerised numerical control system type DMU 50V from DMG, Germany, which has an accuracy of $1-2\,\mu$m. The holes are drilled automatically one at a time using hardened drills. At the same time three further holes are drilled at the periphery of the disc. These holes (called location holes) have a diameter of $500\,\mu$m and are drilled all the way through the disc. The relative positions between the location holes and the grain holes are well-known, but the relative distance between the holes and the circumference of the disc may vary slightly from disc to disc. The purpose of the location holes is to form a basis for the calculation of the positions of the individual grain holes. Before every OSL measurement the system determines how the single grain sample disc is positioned using the disc location routine (see section 1.2)

1.2 Disc location

Manual positioning of the sample discs on the sample carousel can not be precise on a $10 - 100\,\mu$m scale. Furthermore, every time the sample is lifted into the measurement position some uncertainty in the position of the sample disc relative to the laser system will be introduced. Thus, it is necessary to determine the exact location of the sample disc relative to the laser system on each occasion before any measurements are carried out. The three location holes at the periphery of the disc are used for this purpose. The position of the location holes are determined by scanning the laser beam at reduced power ($\sim 4\%$) across the periphery of the disc. The power is reduced during the location hole scans to minimise the amount of scattered light that might reach grain holes adjacent to the location holes. A photo-diode is positioned in such a way that it will be struck by laser light reflected from the surface of the sample disc. When

[1] Sample discs with different grain hole dimensions can be delivered upon request

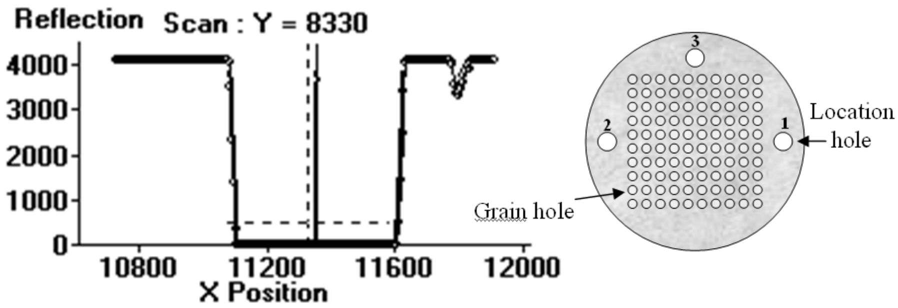


Figure 2: Example of a location hole scan. The change in reflectivity as the laser passes over the location hole is clearly seen. The diameter of the location hole is $500\,\mu$m as expected. Also shown is a schematic drawing of a single grain disc.

the laser beam passes over a location hole the intensity of the reflected light decreases significantly.

Figure 2 shows an example of the measured reflection during a location scan. Scanning is performed in both X and Y directions and these measurements form the basis for the calculation of the centre of the location hole. This process is repeated for the three locating holes.

2 Single Grain Light sources

There is a choice of two different laser sources:

1. Green laser (532 nm)
2. Infrared (IR) laser (optional, 830 nm)

One of the two lasers available for single grain measurements is a 10 mW Nd:YVO_4 solid state diode-pumped laser emitting at 532 nm focused to a spot about 20 μm in diameter. The maximum power density at the sample is estimated to be $50\,W/cm^2$ (Duller et al., 1999).

The other laser available for single grain measurements is a 140 mW 830 nm IR laser positioned perpendicular to the green laser beam. A beam splitter enables the use of the same optics to focus the IR laser beam onto the sample disc as used for the green laser. The maximum energy fluence rate of the IR laser is estimated to be $500\,W/cm^2$ (Bøtter-Jensen et al., 2003). A Schott RG 780 filter is placed directly in front of the IR laser (see Figure 1) to cut a small resonance emission at 415 nm
The power of the laser beams can be electronically controlled to vary between zero and full power, thus enabling linearly modulated OSL measurements on

single grains.

Three lenses (see Figure 1) are used to focus the laser beam, which is approximately Gaussian, with 90% of the power contained within a spot of $< 20\,\mu$m on the sample disc. The laser spot is steered by two orthogonal mirrors and can be positioned arbitrarily on the sample disc. The mirrors are moved by two motor driven stages equipped with position encoders. The mirror in the x-direction is placed at an angle of $45\,^\circ$ to the direction of the laser and the y-mirror at an angle of $22.5\,^\circ$ to obtain an angle of incidence on the sample disc of $45\,^\circ$. Ideally the angle of incidence on the sample should be $90\,^\circ$ to reduce the possibility of scattered light affecting the adjacent grain, but that would greatly increase the distance between the sample and the photomultiplier tube and so decrease the light collection efficiency. Thus, the angle of incidence of $45\,^\circ$ is a compromise between minimising optical cross-talk and optimising detection efficiency.

3 Setting up the software

The single grain attachment is run using the standard *Sequence Editor* and the user is assumed to be familiar with this and the basic operations of the TL/OSL reader.

3.1 Setting up the Controller/Minisys

The default settings of the Controller/Minisys must be changed in order to run the *Single Grain Attachment*. To enable single grain operations:

1. Start the Control program and select "Connect to MiniSys"
2. Select the tab "Reader Settings"
3. Check the option "Single grain reader YES" and press "send" to send the information to the Controller/Minisys. You will be prompted for a password. This can be obtained by contacting DTU Nutech.

IMPORTANT!!! If the *Single Grain Attachment* is to be disconnected it is imperative that the option "Single grain reader NO" is sent to the Controller/Minisys before disconnection.

Controller/Minisys parameters for single grain operation

In order for the *Single Grain Attachment* to run it is important that the Controller/Minisys has the appropriate parameters.

X- and Y-rails The number of encoder steps for the X- and Y-rails are set using parameter "39" and "40", respectively. For new SG units (after 2007) the value is "2158". For older units the value is "4960".

Gearbox ratio The gear box ratio is set using parameter "56". For new SG units the value is "35.55". For older units it is "16".

To check the value of parameters:

1. Start the Control program and select "Connect to MiniSys"
2. Select the tab "Dialogue"
3. Use the command "ra 39" to read the status of parameter "39". Do the same for the other parameters ("40" and "56")

In order to change the value of parameters "39", "40" and "56" you will need the system password. This can be obtained by contacting DTU Nutech.

1. On the "Dialogue" tab type "ep *password*"
2. Type "sa 39 2158" ENTER and "sa 40 2158" ENTER to change parameters for the Y- and X-rails. Type "sa 56 35.55" to change the parameter for the gearbox.
3. Save the change by the command "wp" ENTER.

3.2 Setting up the *Sequence Editor*

The default settings of the Controller/Minisys must be changed to enable single grain operations. This is normally done by DT Nutech before shipment or in connection with installation, but for completeness the procedure is described below:

- Open the *Sequence Editor* program and type in your User Access name (see Figure 3).
- In *Options* select *user options* (see Figure 4). In this dialogue box you can set up the sequence and data paths.
- In *Options* select *system options* In this dialogue box you can choose the appropriate system type. In order to be able to run the *Single Grain Attachment* you need to choose *single grain* in *System options* (see Figure 5. You will now be able to access the single grain software in *Options* you need to click the last option *Single Grain system.* Click *OK*. You will now be able to access the single grain software in *Options*.

In *Options* you will now be able to choose: ***Single grain system***.

4 Single Grain options

When the *Sequence Editor* has been set up properly, the *Single Grain Options* will appear. Here you have four additional options (see Figure 6)

Figure 3: User Access. Typing in your username ensures that the *Sequence Editor* points to the directories specified in sequence and data paths (see Figure 4)

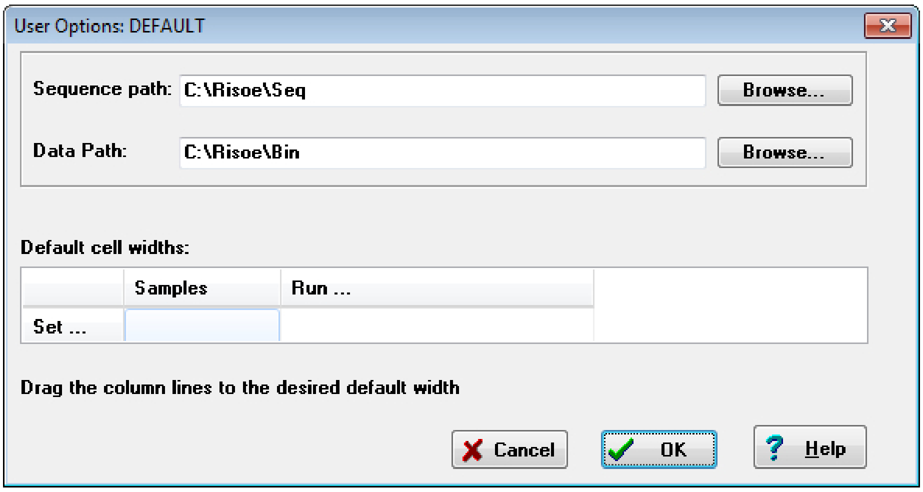


Figure 4: In this dialogue box the Sequence and Data path can be specified. These paths must exist prior to typing them here.

1. System set up (see section 4.1)
2. Check Disc Locations (see section 4.2)
3. Check centre position (see section 4.3)
4. Check laser power (see section 4.4)

4.1 System Set Up

System Set Up is the first option in *Options/Single Grain Setup* (see section 3). In this dialogue window there are two tabs:

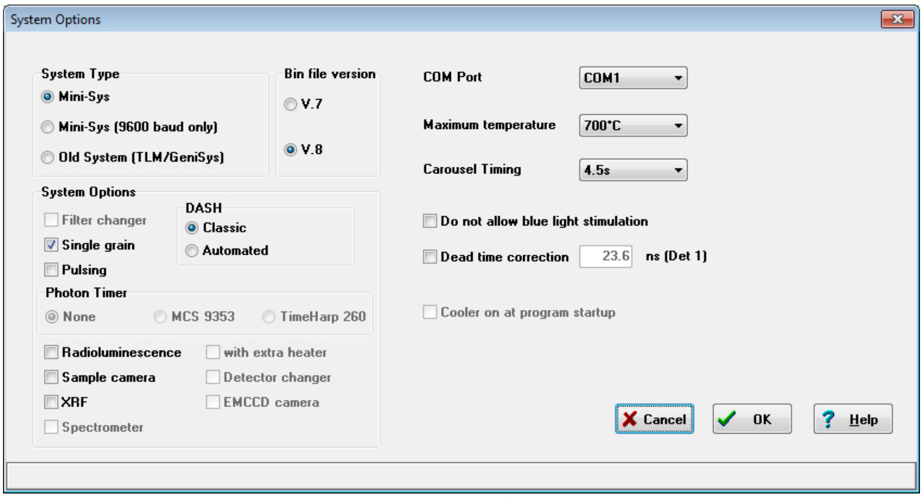


Figure 5: To be able to use the *Single Grain Attachment* the *Single Grain system* must be selected under *System Options/ System Options*.

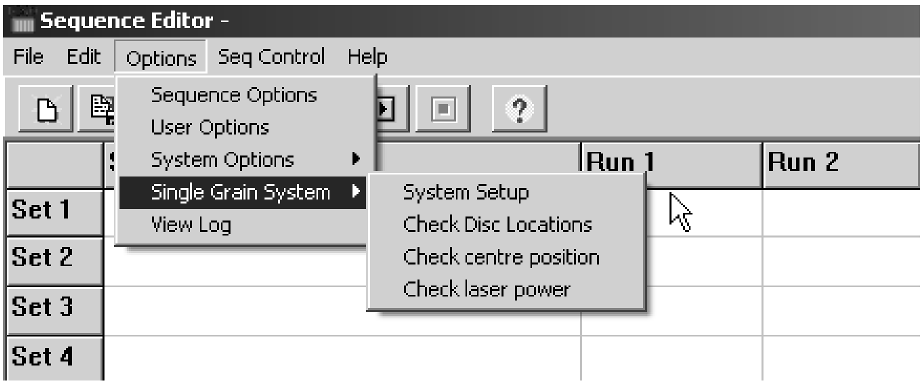


Figure 6: *Single Grain System* menu

1. *Initial Disc Positions and Disc Description*
2. *Encoder System and Search Parameters*

For most applications you will only need to work with the *Initial Disc Positions and Disc Description* tab.
The system set up is unique for each attachment. A specification sheet for the individual *Single Grain Attachment* is provided with the system. All values presented in square brackets [ ] in the following sections are typical values.

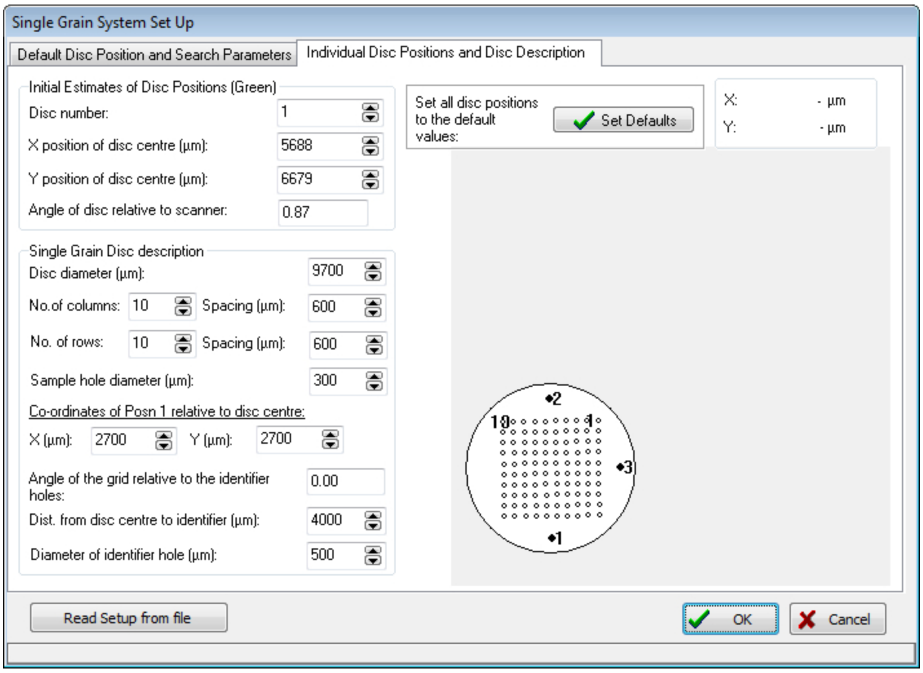


Figure 7: Screen shot of the *Initial Disc Positions and Disc Description* tab found in *Options/System Set Up*

4.1.1 Initial Disc Positions and Disc Description

A screen shot of this dialogue box is shown in Figure 7.

Initial Estimates of Disc Positions (Green)

1. *Disc number*: number of the sample disc on the carousel [1]
2. *X position of disc centre*: the X coordinate of the centre of the disc in μm. [6000]
3. *Y position of disc centre*: the Y coordinate of the centre of the disc in μm. [6000]
4. *Angle of the disc relative to scanner*: the angle of rotation of the disc relative to the laser beam. Zero degrees corresponds to locating hole 1 closest to the laser source, locating hole 2 furthest away, and locating hole 3 positioned towards the outside of the carousel. Initially, the user enters the “best guess” of the appropriate angle [0].

The values of the X- and Y coordinates of the centre are initially determined using the Options/Single grain system/Check centre position (see section 4.3).

These values are then updated automatically for each disc. In normal operation (after setting up the machine for the first time) one should never alter these values.
The system remembers the last determined value of the angle of rotation for each disc, and it is advisable ro reset this value before each new sequence is started. D10 was positioned at $\sim 0^\circ$ when "Sequence 1" was started. Before the final OSL measurement the angle of rotation was 5°. A new disc is now placed on position 5 at $\sim 0^\circ$. Before starting "Sequence 2" the angle of rotation ought to be changed manually back to 0° (see **Set Defaults**).

Single Grain Disc description

Used to define the sample disc characteristics. These values should not normally be changed.

1. Disc diameter (μm) : [9700]
2. No. of columns : [10]
3. Spacing (μm) : [600]
4. No. of rows : [10]
5. Spacing (μm) : [600]
6. Sample hole diameter (μm) : [300]
7. Co-ordinates of position 1 relative to disc centre - X (μm) : [2700]
8. Co-ordinates of position 1 relative to disc centre - Y (μm) : [2700]
9. Angle of the grid relative to the identifier holes : [0]
10. Distance from disc centre to identifier (μm) : [4000]
11. Diameter of identifier hole (μm) : [500]

Set Defaults

If this button is pressed then the values entered for any given disc will be made the default value for all discs; e.g. if:

1. Disc 1; X pos = 6000; Y pos = 6000; Angle = 5
2. Disc 2; X pos = 6100; Y pos = 5900; Angle = 0

and the *Set Defaults* button is presses while the data for Disc 2 is displayed then all discs will be assigned the same values as Disc 2, that is: Disc x; X pos = 6100; Y pos = 5900; Angle = 0. Usually, the centre coordinates are very similar for different discs, so they only change marginally from disc to disc, but the angle of rotation may vary considerable (several degrees) from disc to disc, so it advisable to reset the angle of rotation to zero before beginning a new sequence (on different discs).

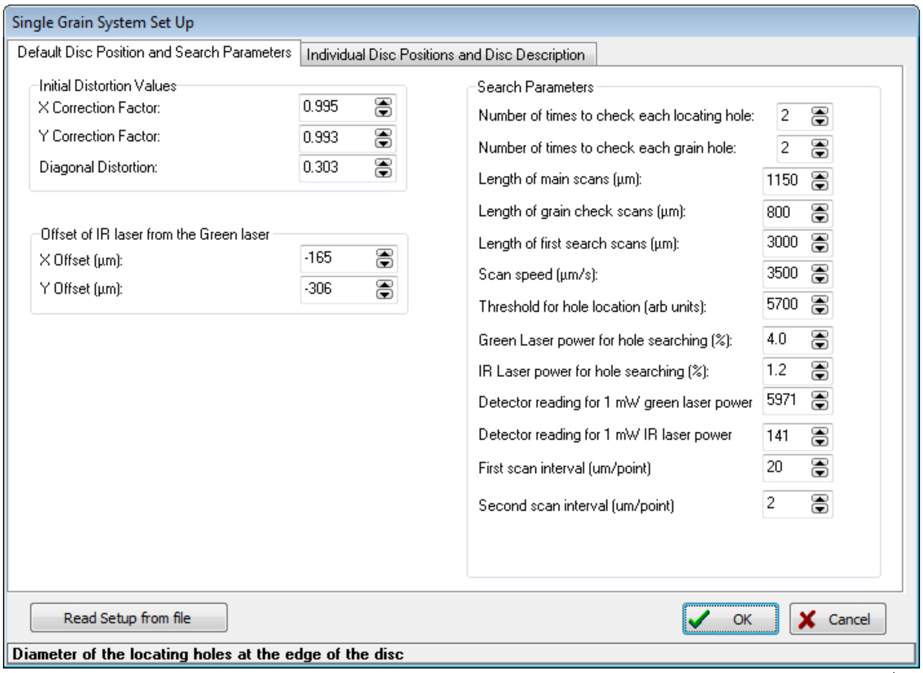


Figure 8: Screen shot of the *Encoder System and Search Parameters* found in *Options/Single Grain System/ Set Up.*

4.1.2 Encoder System and Search Parameters

A screen shot of this dialogue box is shown in Figure 8

Encoder Parameters

1. Maximum X travel: The maximum travel in (μm) of the X movement. DO NOT ALTER THIS VALUE. [14000]
2. Maximum Y travel: The maximum travel in μm of the Y movement. DO NOT ALTER THIS VALUE. [14000]
3. X (μm) per 1000 encoder steps: Calibration of X movement [2158]. DO NOT ALTER THIS VALUE.
4. Y μm per 1000 encoder steps: Calibration of Y movement [2158]. DO NOT ALTER THIS VALUE.
5. First scan interval: The number of data points transferred from the MINISYS/CONTROLLER to the host PC after an initial hole search scan. This value significantly affects the speed of the disc location routine, and

should be kept as large as possible consistent with reliable initial identification of a locating hole or a grain hole. [20]

6. Second scan interval: The number of data points transferred from the MINISYS/CONTROLLER to the host PC after the second or subsequent scans of a hole. This value significantly affects the speed of the disc location routine, and should be kept as large as possible consistent with precise measurement of the centre of the hole. [2]

Initial Distortion Values

1. *X Correction Factor*: a linear multiplication factor used to allow for a small distortion in the X axis. This can arise, for example, in disc manufacture or if the heater plate is not exactly in the same plane as the X movement. [1.0000]

2. *Y Correction Factor*: a linear multiplication factor used to allow for a small distortion in the Y axis. This can arise, for example, in disc manufacture or if the heater plate is not exactly in the same plane as the Y movement. [1.0000]

3. *Diagonal distortion*: Allows for small non-normal relative movement in the X and Y directions, or for small diamond distortion in the disc manufacture. [0.0000]

The user should enter average values for each of these distortion factors derived from successful disc location runs using derived from Options/Single grain system/Check Disc Locations. The system will use these values as first approximations when it begins further disc locations, whether manual or automatic.

Offset of IR laser from the Green laser

1. *X Offset in* μm: [0]

2. *Y Offset in* μm: [0]

Search Parameters

1. *Number of times to check each locating hole*: the number of X and Y scans used to determine the centre of a disc locating hole. [2]

2. *Number of times to check each grain hole*: the number of X and Y scans used to determine the centre of a grain hole. [2]

3. *Length of main scans*: the distance in μm the laser spot will move in the X or Y direction when checking the centre position of a locating hole. [1200]

4. *Length of grain check scans* the distance in μm the laser spot will move in the X or Y direction when checking the centre position of a grain hole. This scan length must be chosen with the grain hole spacing (usually 600 μm) and grain hole diameter (usually 300 μm) in mind. [800]

5. *Length of first search scans*: the distance in μm the laser spot will move in the X or Y direction when searching for a locating hole. This longer scan is used when the system was unable to locate a hole in the position it expected. [3500]

6. *Scan speed*: the speed of the X and Y movement in μm/s. High scan speeds can cause errors. [2000]

7. *Threshold for hole location*: the value of the reflected laser signal used by the hole locating routine to determine whether a hole exists. When the signal crosses this threshold from above or below a significant change in reflection has occurred; this may indicate the presence of a locating hole or a grain hole. [900]

8. *Green Laser power for hole searching*: sets the laser power (%) to be used during any hole search. This should be set, in conjunction with the A-D converter gain for hole scans, to as low as possible a value consistent with a just saturated (65,000) signal from the flat surface of a clean disc. Note that on some systems very low values may result in the laser not switching on during grain scans. This offset results from the laser manufacturer's set up, and is outwith DTU Nutech's control. [5]

9. *IR Laser power for hole searching*: sets the laser power (%) to be used during any hole search.

10. *Detector reading for 100% green laser power*: [6400]

11. *Detector reading for 100% IR laser power*: [180]

4.2 Check Discs Locations

This dialogue window can be used to locate discs without performing any OSL measurements, and to determine the accuracy with which the laser spot can be directed to grain holes (see Figure 9). The same location routine is also used automatically in routine OSL measurement, but this window allows the user to be sure that the disc can be found before embarking on a long measurement sequence.

1. *Function - Find disc*: Used to search for the three location holes on the disc defined under Disc Location Options in this window, and *Search Parameters* defined in section 4.1.2. The search can be repeated an arbitrary number of times ("0" means infinite number of measurements), as required, with or without lowering the heater between each search. The results of the search are stored in the data box, and these results may be copied to

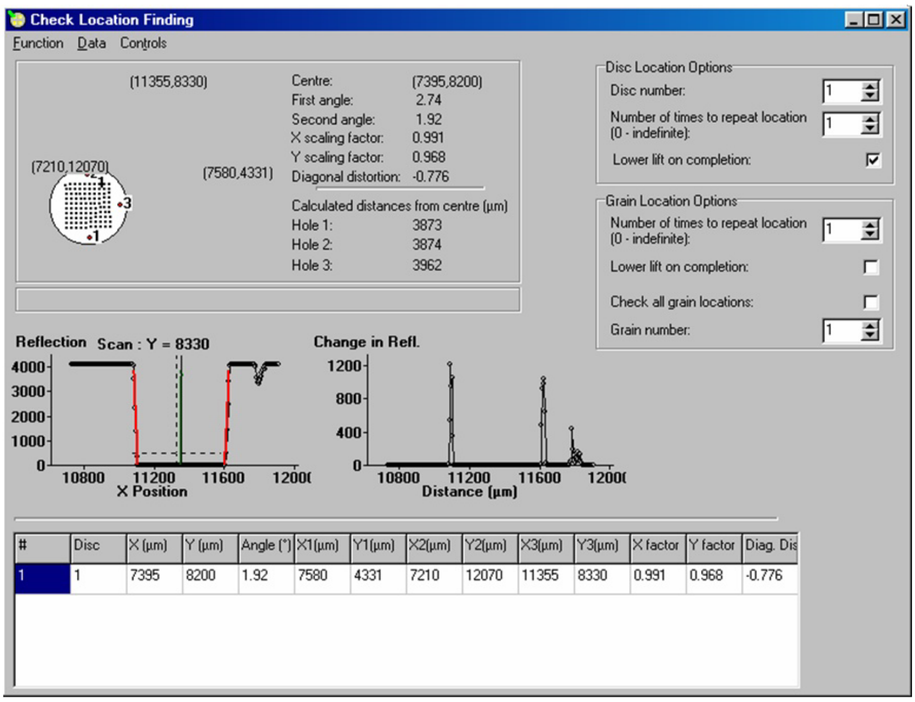


Figure 9: Dialogue box used to manually locate discs and/or grain holes.

the clipboard using Data - Copy data to clip board. The results are also used to automatically update the location and distortion values stored under *Encoder System and Search Parameters* (see section 4.1.2). Note that lowering the heater will probably result in a small change in position of the disc the next time the heater is raised. If the results of a *Find disc* operation are to be used in a subsequent *Find grain operation*, then the *Lower lift on completion* box should not be checked.

2. *Function - Find grain*: Used to search for one or more grain holes, using the same routine used for location hole searching, and search parameters defined in section 4.1.2. The search can be repeated an arbitrary number of times, as required, with or without lowering the heater at the end of the operation. The results of the search are stored in the data box, and these results may be copied to the clipboard using Data - Copy data to clip board. Note that lowering the heater will probably result in a small change in position of the disc the next time the heater is raised. If the *grain hole search* is to be repeated as a check on reproducibility, either the heater should not be lowered (the *Lower lift on completion* box should not be checked), or a new *Find disc* search should be conducted each time.

The two options under Data allow the results of the disc and grain searches to be exported for statistical analysis. The options under Control allow a forced lowering of the heater, or a rest of the X and Y movements.

4.2.1 Precision of the location routine

Before an OSL measurement is undertaken the system must find the three location holes. If all three holes are located successfully, the software then calculates the positions of each of the 100 grain holes based on the known geometry between the location holes and the grain positions. To estimate the precision of the location routine a sample disc was located 88 times and the standard deviations of the calculated x- and y-values were $0.7\,\mu$m ($\sim 0.012\%$) and $0.9\,\mu$m ($\sim 0.012\%$). These figures translate into a maximum error at the locating holes of $2.2\,\mu$m (3σ, x-direction) and $2.6\,\mu$m (3σ, y-direction).

4.2.2 Accuracy of the location routine

The system can be set up to determine the position of the individual grain holes by scanning the laser beam across the surface of the disc, while measuring the intensity of the reflected laser light (analogous to the procedure used in the location routine). The difference between the calculated and the measured position of a given grain hole can thus be compared. However, the individual grain holes are drilled one at a time and therefore might be slightly off-set with respect to each other. In order to determine the accuracy of the software calculations, the grain holes were defined to coincide with the location holes themselves. After successful location of a disc, the system predicted the position of the three "grain holes" (in this case identical to the location holes). If the software is able to calculate these positions accurately, they should be identical to the measured positions of the three location holes. This was tested using 20 different discs placed at different angles of rotation. The differences between the measured and calculated positions ranged between -1 and $2\,\mu$m and the average difference was $0.2\,\mu$m with a standard deviation of $0.7\,\mu$m ($n = 120$). Rounding errors in the computer calculations are assumed to be responsible for the small difference between the measured and calculated values. No systematic dependence on angle of rotation was observed.
For one disc the grain search routine was performed 51 times. The relative standard deviations of the determined positions for all three location holes were 0.01% (comparable to the precision determined in the location routine, section 4.2.1) and the average difference between the calculated and the measured position was $-0.16\,\mu$m with a standard deviation of $0.06\,\mu$m ($n = 306$).

4.2.3 Locating individual grain holes

In the preceding sections it has been shown that the single grain OSL system is able to determine the position of a disc very reproducibly. In this section the ability of the system to steer the laser spot to the centre of individual

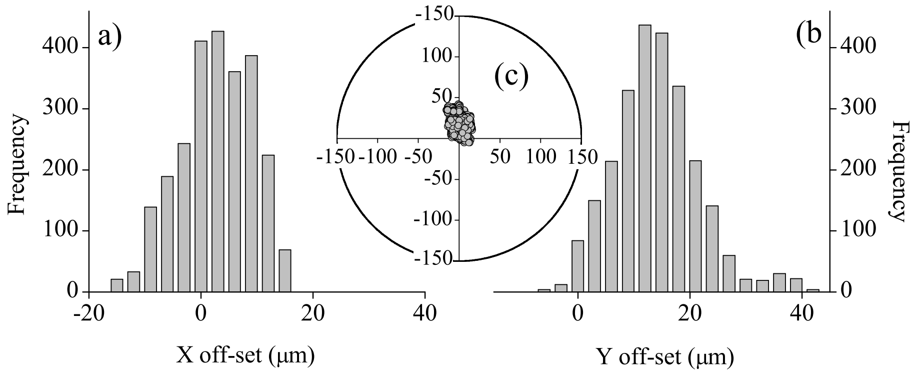


Figure 10: Accuracy and precision of the location routine in locating individual grain holes. The grain hole search routine was repeated 2,500 times. **a)** Frequency histogram of the off-set on the x-axis. **b)** Frequency histogram of the off-set on the y-axis. **c)** The off-sets on the y-axis as a function of the corresponding off-sets on the x-axis. The circle represents the dimensions of a grain hole. Data from Thomsen (2004)

grain holes is examined. The location routine was performed on an empty disc (i.e. not loaded with grains) and the grain hole search was then initiated (without lowering the lift) and repeated 2,500 times (i.e. 25 times on each of the 100 grain holes). The individual position of each of the 100 grain holes was defined to lie as specified in section 1.2. In this test the average off-set (between predicted and measured hole centres) on the x-axis was 2.7 μm (standard deviation was 6.5 μm) and on the y-axis 14.1 μm (standard deviation was 7.6 μm). The results are shown in Figure 10. The important conclusion to draw from this test is that the 20 μm laser spot would have entirely entered each 300 μm grain hole even in the worst case. Given the accuracy and precision found when relocating the three location holes (previous section) it is presumed that most of the variability seen above is in the manufacturing of the disc.

4.3 Check centre positions

Before using the *Single Grain Attachment* the approximate centre of the sample discs must be determined. This is done by manually adjusting the laser spot to the centre of a sample disc. This should only be required when the system is initially set up, or if components are dismantled or replaced. It is not a routine operation.

To manually set the laser spot to the centre of a sample disc load a sample disc onto position 1 and click *Options/Single grain system/Check centre positions*. This opens up a dialogue box. Note that it may take some time, because the reader will reset the turntable and raise the lift before the box appears. The PM tube and optical filter should be removed (remember to remove the HV cable before the signal cable!) so that the location of the laser spot can be seen by eye.

(ALWAYS USE APPROPRIATE SAFETY GLASSES WHEN THE BEAM IS EXPOSED!) The laser spot is manually steered to the centre of the sample disc by varying the X co-ordinate and Y co-ordinate. Simply place the cross-hair on the picture of the single grain disc where the laser spot appears on the real disc. The positioning should be as accurate as can be readily determined by eye. When OK is selected, the new centre position is automatically stored for position 1. This can be confirmed in *Options/Single Grain System/System Set Up* (see section 4.1). If this value is to be used as the default for all positions (this is normally the case), it should be entered into disc position 0, and then all discs will be set to this value by selecting Set Defaults. Remember to replace the optical filter before the PM tube. The signal cable MUST be connected before the HV cable.

4.4 Check laser power

Reads the laser power monitor. This window is also used to calibrate the laser power in %. The Laser control voltage is set to 100%, and the Display format to raw data. The photodetector reading is then displayed above the graph. The time average of this value should be entered in the Set up - User Information - Photodetector reading for 100% laser power box (the averaging need not be precise).
NB. This option is only working correctly for the green laser

5 "The Single Grain OSL" command

This is the option that begins the editing of a measurement sequence. Only the single grain option will be discussed here.

- **Single Grain Light Source** choose either the *Green Laser* or the *IR laser* [Green Laser]
- **Measure grain** the user can choose to only measure a selected number of grains or even a single grain [1 to 100]
- **Power (%)** the power of the laser light. It is unadvisable to run above 90 % power for prolonged periods of time [90]
- **Total Time** the length of the measurement in seconds [1]
- **Time per datapoint (s)** *Total time* divided by *Total Datapoints*
- **Total Datapoints** the number of consecutive time intervals the measurement time in divided into [60]
- **Before stimulation** the number of data points that will be collected before the stimulation light is switched on. Used to check dark count, and to ensure that the entire OSL signal is recorded

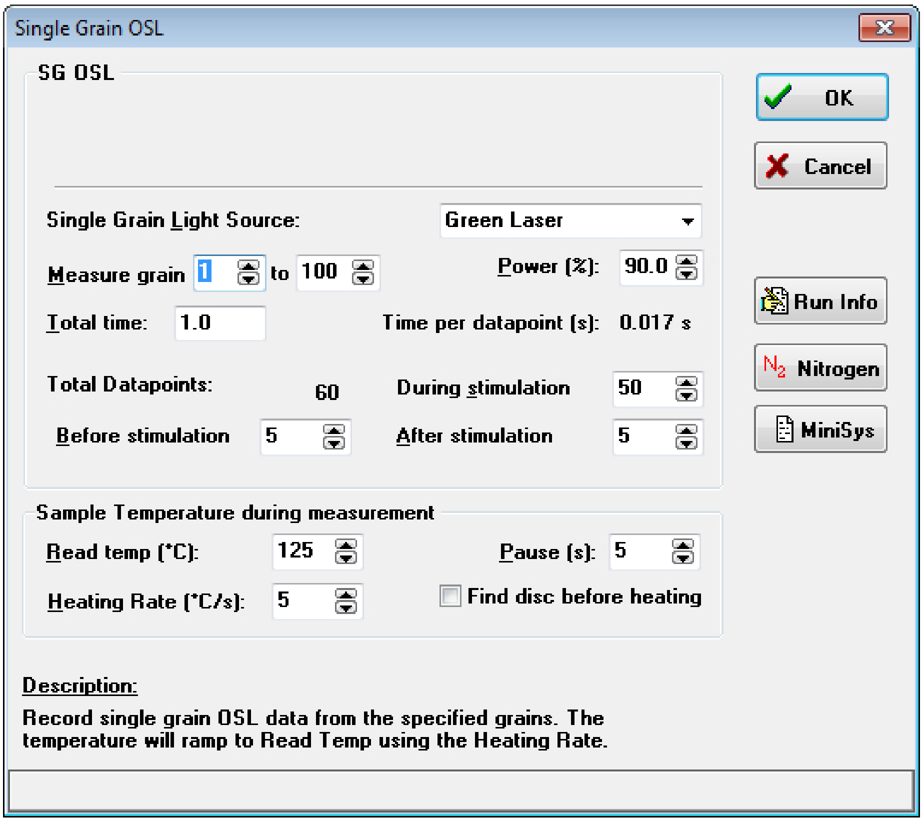


Figure 11: Single grain dialogue box

- **During stimulation** the number of data points that will be collected while the stimulation light is switched on [50]
- **After stimulation** the number of data points that will be collected after the stimulation light is switched off. Used to check dark count, and to estimate the size of any remaining OSL signal at the end of the measurement period. [5]
- **Read temp** (°C) the OSL measurement will be carried out at this temperature, e.g. the heater plate ramps to 125 °C and the measurement will only begin after the 125 °C has been reached [125]
- **Heating Rate** (°C/s) the rate at which the heater plate will ramp to the *Read temp* [5]
- **Pause (s)** the time in seconds for which the sample is held at the *Read temp* before measurement begins

6 What to do if all else fails!

- **No response from the MINISYS/CONTROLLER.** Start by turning off the MINISYS/CONTROLLER and the PC, and rebooting both. When a sequence is started, and *Sequence Editor* gives a message that it cannot contact the MINISYS/CONTROLLER check that the communications port is set correctly for the PC (particularly that it is the right port number). This is done by clicking *Options/System Options/Port.* Only the port number usually needs changing, to suit the user's PC. [COM1, 9600, none, 8,1]

- **Unable to find a disc.** This is most likely to be because the disc is placed incorrectly compared with the disc angle of rotation given in *System Set Up* (see section 4.1). If this is definitely within tolerance (about ±5 ° of the given angle) reset the X and Y movements (Set Up - XY System - Control - reset encoders). If the search routine still cannot find the locating holes, then try turning off both the MINISYS/CONTROLLER and PC. If the program still cannot find the locating holes, the system should then be set up again from the beginning (a disc should be present in carousel position 1).

  IN WHAT FOLLOWS THE LASER BEAM IS EXPOSED. ALWAYS USE APPROPRIATE SAFETY GLASSES IN THESE CIRCUMSTANCES!

  1. Dim the room lights. Remove the PM tube and optical filters (remember to remove the HV cable before the signal cable!) so that the location of the laser spot can be seen by eye
  2. Select *Options/Single Grain System/Check centre position.* This will move the laser spot to where the system thinks the disc centre is. Turn on the laser beam, and check that the laser spot is visible. Check the centre position as described in section 4.3.
  3. Run *Options/Single Grain System/Check Disc Locations* with the PM tube and optical filters still removed. Check the spot is visible, and watch it move across the disc as the system searches for the first locating hole. If the laser spot is not visible the laser power for hole searching can be altered in *Options/System Set Up/Encoder System and Search Parameters* (see section 4.1.2). The power should be as low as possible, consistent with it being clearly visible. Alter the angle of rotation if necessary to ensure that the spot passes across the first location hole. As the spot traverses the hole, the reflected signal should drop from saturation to almost zero. It must pass below the threshold given in *Options/Single Grain System/Encoder System and Search Parameters/Threshold for disc location.* If it does the hole should be found successfully on this scan (indicated by a solid vertical line appearing on the reflection scan, passing through the centre of the reflected image of the locating hole). If the spot definitely crosses

the middle of the hole, but the signal does not drop to a low value, the reflection analogue amplifier is faulty or misadjusted, and DTU Nutech should be contacted for further instructions.

7 Disc Care

The sample discs are made of anodised aluminium. It is very important that the surface is kept clean and in good condition, and that the holes (especially the location holes) are not damaged. The surface is scratch resistant, but nevertheless metal tools (tweezers etc.) should never be used. Plastic tweezers are readily available (e.g. those used by stamp collectors). No attempts should be made to "poke" a grain out of a hole - it will only stick more tightly. The safest cleaning method seems to be to use an ultrasonic cleaner, i.e. place the discs upside down in an appropriate holder (see accessory list for the Risø TL/OSL Reader) and press the tip of the cleaner against the back of the disc for 1 s. If an ultrasonic bath, using water or alcohol, is used then never leave the discs to soak as discolouration may occur. Acetone can be used for drying, but it is dangerous to mix aluminium and acetone for long periods, especially in an ultrasonic bath. Discs have also been damaged by prolonged soaking in Calgon. Remember, one or two old grains stuck in a disc do not significantly reduce its usefulness. Results from particular holes can always be identified and disregarded. But a scratched, discoloured or otherwise damaged disc will have to be discarded. And they cost about US$20 each!

References

Bøtter-Jensen, L., Andersen, C. E., Duller, G. A. T., and Murray, A. S. (2003). Developments in radiation, stimulation and observation facilities in luminescence measurements. *Radiation Measurements*, 37:535–541.

Duller, G. A. T., Bøtter-Jensen, L., Murray, A. S., and Truscott, A. J. (1999). Single grain laser luminescence (SGLL) measurement using a novel automated reader. *Nuclear Instruments and Methods B*, 155:506–514.

Thomsen, K. J. (2004). *Optically Stimulated Luminescence Techniques in Retrospective Dosimetry using Single Grains of Quartz extracted from Unheated Materials*. PhD thesis, University of Copenhagen.