Single grain OSL analysis: A discussion of how to clean and check single-grain discs

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Abstract
We compare possible methods to remove grains from single grain discs including heating, tapping, vibration in a sieve shaker and use of an ultrasonic bath. These methods are quick, simple and effective at removing smaller grains or those that have become stuck as a consequence of settling during pre-heating. Holders for single grain discs to facilitate thermal and vibrational cleaning approaches have been designed and manufactured. Heating and tapping seem to be useful additional techniques for cleaning single grain discs, resulting in insignificant residual sensitivity in the majority of cases.

Introduction

Optically stimulated luminescence (OSL) analysis of single grains has proven to be a valuable means of enhancing luminescence dating. It has been shown that there is significant variability in luminescence properties and sensitivity between grains (Duller et al., 2000; McCoy et al., 2000). With single-grain analysis it is possible to investigate such behaviour, and importantly, attempt to separate components within complex mixtures (e.g. Thomsen, 2004) or identify grains that have been heated in a fire (Moska et al., in press). However, the sensitivity of single grains of quartz frequently leaves many observations at, or close to, statistical detection limits for photon counting. Thus it is extremely important to check the blank levels of cleaned discs between runs, and to carry forward realistic estimates of detection limits into the analysis. In this paper we look briefly at how to clean single grain discs from the Risø DA-20 system, using blank scans in the reader and scanning electron microscopy to evaluate performance. The Risø guide (2007) suggests that the safest cleaning method is an ultrasonic treatment with water or alcohol. Here we have also evaluated thermal and vibrational approaches with promising results.

After each cleaning process was applied, discs were analysed in three ways to check for residuals. The disc was first observed under an optical microscope, which was rapid but did not provide enough detail to fully confirm that there were no grain fragments. This detail was provided by looking at the disc under a scanning electron microscope (SEM). Finally, to check for remaining OSL signals a “disc cleaning check” was carried out on the Risø single grain reader. This comprised application of a fixed beta dose of roughly 10 Gy (supplementing the signal induced by electron beam irradiation under SEM examination) followed by preheating to 160°C (at 5°C/s and held for 10 s) and an OSL measurement for 2 s. Data from the OSL check were classified on the basis of the number of times that observed net OSL signals exceeded estimated poisson uncertainties. The signal from the first 0.5 seconds of optical stimulation was integrated and signal during the last 0.5 seconds was used for background subtraction to calculate the “net” signal. The detection limits, applicable to subsequent runs, for each disc and hole were then defined as the mean value plus three standard deviations. Even unused discs and cleaned discs produced a small, but detectable, signal (Fig. 1). To ensure that each single grain disc could be subsequently tracked, a number was engraved on its back.

Discussion of methods

Twenty-six discs were examined using the methods outlined below. These discs had previously been used to measure quartz from a range of sample sites (Orkney, Italy and Sri Lanka) and all had been sieved to 150–250μm diameter.

Cold tapping

The majority of grains were found to be easily removed by “cold tapping” – tapping the discs upside down followed by brushing and spraying with compressed air. This only removed all the grains on three discs out of 17 (there were 9 discs for which this method was not used) but for the discs that were not completely cleaned, only between one and ten grains remained. The removal of the loose smaller grains prevented any grains from returning to holes as well as isolating these stuck grains. To improve the cleaning process, a single grain disc holder was designed (Fig. 2) which holds the discs upside down above a pit for collecting fallen grains and, when the

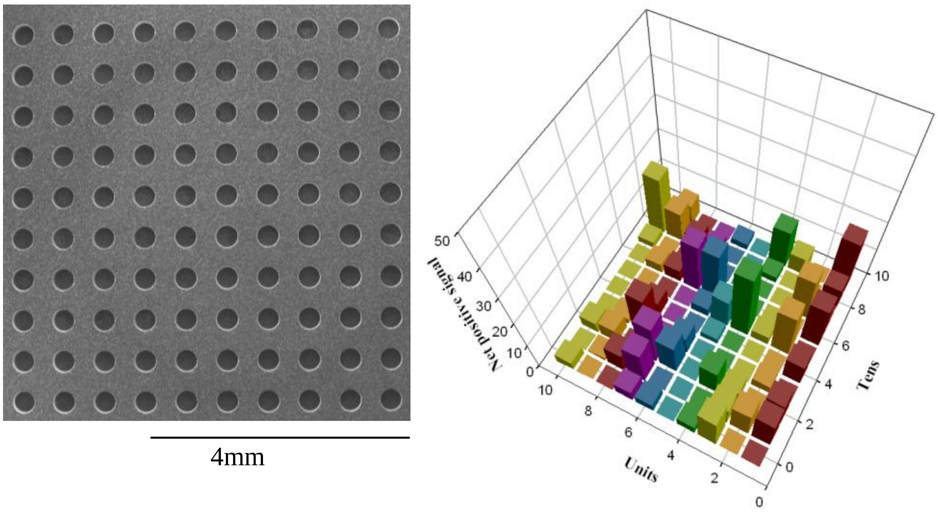


Figure 1: *An SEM picture of a cleaned disc and the net OSL signal (photon counts) produced by that disc for each hole.*

Figure 2: *Single grain disc holders to help with the cleaning process and to store the discs.*

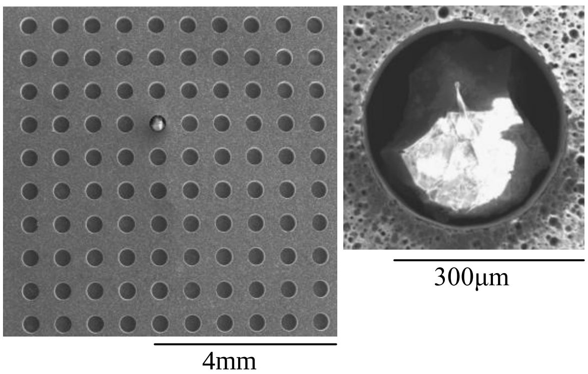


Figure 3: *A grain wedged in a hole because of two points of contact on opposite sides of the hole (note the charging on the grain which has occurred because the discs were not coated).*

discs are clean, prevents them from being contaminated. Furthermore, the holder can be used for all the different cleaning methods and discs can safely be stored with grains in.

Hot tapping

Some grains are the correct size and shape to become wedged in the holes on the single grain discs (Fig. 3). It was thought that these grains may have become wedged in the hole as a result of the hole expanding during preheating in the Risø Reader. If the disc is preheated to 220°C (a temperature rise of roughly 200°C), this corresponds to an increase of 1.39µm in the diameter of the hole since the coefficient of thermal expansion for aluminium is 23.1 x 10^{-6} K^{-1}. This may cause grains to settle into positions which trap them when the holes contract on cooling. Therefore, theoretically, heating the disc upside down to a temperature above the preheat temperature, such as 400°C, and giving it a sharp tap should remove grains that had become stuck through that process. This method was quite successful at dislodging large grains such as the one shown in Fig. 3. All but eight discs were fully cleaned from only using cold tapping and one to three cycles of hot tapping. Statistics of the photon count from a disc as it passed through the methods outlined above are given in Table 1.

Shaking

It was noticed under the SEM that the sides of the holes were not completely smooth (Fig. 4) and may also cause grains to have a difficult path out of the hole even when it has been expanded using heat in the "hot tapping" method.

	Number of holes with signals significant at 3σ	Mean net signal of the holes with insignificant signals (photon counts)	Largest net signal (photon counts)
Before cleaning	24	1.6	4913
After "cold tapping"	13	-1.0	2336
After "hot tapping"	2	-1.8	30
Mean of all 25 cleaned discs	3.3 ± 0.4	-0.7 ± 0.3	45.3 ± 6.2

Table 1: *An example of what happened to one of the discs as it was cleaned using the methods outlined above. The final row gives statistics of the 25 cleaned discs (not including the one disc that had two grains remaining in it) compiled from the "disc cleaning check" runs.*

Shaking the discs while they were still hot was attempted as an enhancement to the "hot tapping" method so an adapter to fit onto a Fritsch ®pulverisette ®analysette ®laborette laboratory sieve shaker was manufactured (Fig. 5). This holds three of the single grain disc holders (Fig. 2) which can be preheated in an oven. This method appeared reasonably successful, and can be readily incorporated into standard procedures. At present it is unclear whether the performance exceeds that from "hot tapping" alone.

Ultrasonic treatment

For four discs, hot tapping had left one grain stuck in each disc. To try a more vigorous method of shaking the grains out of the hole, an ultrasonic treatment in water followed by drying using methanol was employed as suggested in the Risø Guide (2007). For three of the discs, the ultrasonic treatment removed the final grain. However, for the fourth disc, another cycle of heating and ultrasonic treatment was required to remove that grain implying that a combination of the methods enhances the removal process.

To ensure that ultrasonic treatment was not a sufficient method on its own, three discs were first cleaned using the ultrasonic bath and then photographed under the SEM. An average of 5 grains remained in each discs and these grains were removed by the subsequent heating (Fig. 6).

Figure 5: *An adapter for a laboratory sieve shaker which holds 3 single grain disc holders.*

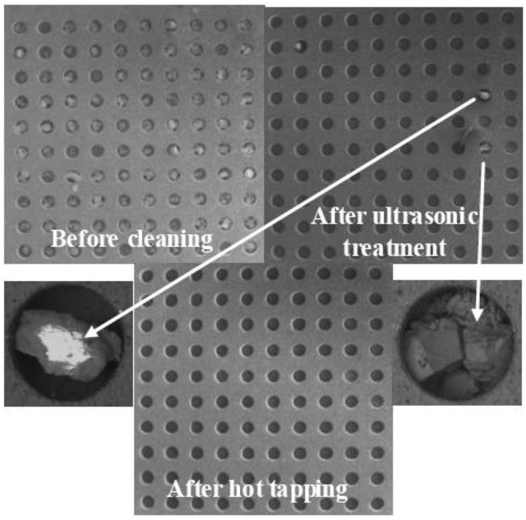
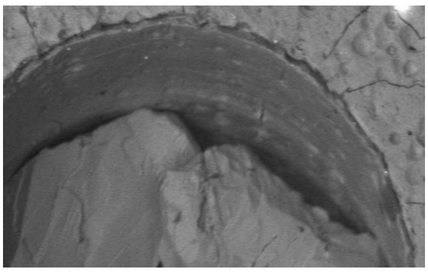


Figure 6: *SEM photographs of a disc that was first cleaned using ultrasonic treatment and then by hot tapping showing some of the grains that were only removed after heating*



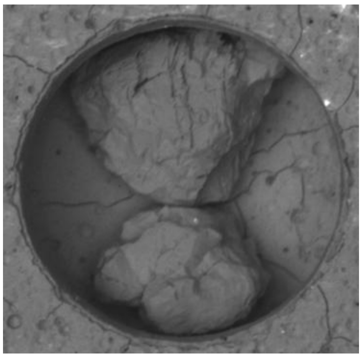
100µm

Figure 4: *An SEM image of the side of a hole where the side shows some roughness.*

The combination of ultrasonic treatment and heating has not been able to clean the final disc which contains two grains stuck in a hole (Fig. 7). It is thought that these two grains are wedging each other into the hole.

Summary and Conclusions

The last line in Table 1 summarises the overall performance achieved in cleaning 25 discs using all methods. This does not include the one disc, from the original 26 examined, that was not completely cleaned (Fig. 7). From this initial study, a combination of heating and ultrasonic treatment was found to be very effective at cleaning all but one hole from a total of 2600 holes examined.



200µm

Figure 7: *The grains that have not been dislodged from the hole of one disc.*

The cleaned discs gave little statistically significant residual signal and no grain fragments could be seen under the SEM. Further work is required to enhance these cleaning methods to ensure that reused discs have been reliably cleaned and contain little residual signal that could contaminate further results.

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Reviewer

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Referee Comments

Ensuring that single grain discs are clean prior to measurement is essential to obtain reliable OSL data. As all users of single grain systems will know, cleaning these discs is a constant challenge. The new methods described here are a valuable addition to those available to practitioners.