Post-Irradiation Heat Treatment of B3 Dosimeter Products

SCOPE

The use of post-irradiation heat treatment (annealing) of B3 film dosimeters, and the process verification and validation testing of the heat treatment methodology that the user employs.

PURPOSE

To define and elaborate on the post-irradiation behavior of B3 film, and how to use heat treatment processes to manage film stability.

HISTORY / BACKGROUND

GEX recommends the heat treatment of B3 film dosimeters after they have been irradiated in order to complete B3 dosimeter color development, and eliminate post-irradiation measurement variability. Heat treatment or annealing is defined at GEX as: the process used to stabilize the chemical properties of the B3 material. The following information is useful in understanding the requirements needed to establish an effective post-irradiation heat treatment process.

B3 film post-irradiation color development is dependent on the relative humidity and temperature, both during and after irradiation, as well as the dose magnitude that the B3 dosimeter was exposed to during the irradiation process. The temperature and dose during irradiation are the two most influential quantities that affect the performance of the film after irradiation. The effects of these influence quantities is interdependent and complex during that color development period and cannot be independently evaluated or corrected. The post-irradiation color change will continue for hours unless an effective heat treatment process is applied to complete the color development, with the length of time being dependent on the quantities noted prior. Therefore, we have found the best practice for B3 films is to heat treat them after irradiation. This heat treatment process must be implemented, validated, and maintained by the user.

Once the films are heat-treated properly they become very stable, particularly if the storage conditions are controlled. This is a by-product of the processes that is one of the highlights of B3 performance characteristics.

Significance of Post-Irradiation Response Variance:
One of the most significant factors in determining the accuracy of a dosimetry system is the post-irradiation stability of the dosimeter response or absorbance value. The impact of post-irradiation dosimeter response variance can account for a significant portion of the overall uncertainty of the dosimetry system. The quantity of post-irradiation change is typically non-linear, in that a higher percent change generally occurs in the lower doses, and with little or none associated with higher doses.

The response signal of some dosimeters may be highly stable over a long time period, but suffer a high degree of initial short-term response signal instability. These dosimeters can be managed by simply controlling the initial holding time period needed before allowing dosimetry measurements to be taken, in order to provide sufficient time for the response signal to stabilize.

Other and more difficult to manage dosimeters may exhibit an unstable response immediately after irradiation and never achieve an acceptable level of stability; they may only demonstrate both signal growth and fading. These dosimeters will add significantly to the overall dosimetry system uncertainty, and at best may only be time control measured. These dosimeters cannot be managed with a simple time holding period before readout. They require use of multiple post-irradiation time period calibration curves to best manage their post-irradiation instability.

Post-Irradiation Stability of B3 Film Dosimeters:
B3 users have been able to implement and validate heat-treatment processes that render the films stable. Measurement data indicates that the post-irradiation stability of properly heat treated B3 dosimeters is as good as or better than other dosimeters. In fact, irradiated B3 film is available from Risø DTU National Laboratory as certified references for use with the

RisøScan Dosimetry Software. In addition, because the B3 film dosimeters are stable after a heat treatment process, they can also be used as reference standards for the dosimetry system (see *GEX Doc# 100-210, Genesys 20 – General Practices and Information*, for detail on using B3 dosimeters as references).

Historical Specification of B3 Film Dosimeter Heat Treatment:
It was determined early in the development of the B3 film dosimeter that a heat treatment process provides completion of the film's color development. The founder of B3, Arne Miller, established the basis that post-irradiation heat treatment in a box-style incubator for a five minute time cycle at 60.0°C provides a 100% color development, and B3 dosimeters heat treated to these specifications would remain stable for more than one year. This specification has been used successfully at Risø since the early 1980's.

A number of questions arise when the heat treatment specification mentioned expands beyond a single user. For example:

- How much variation is acceptable in terms of the temperature?
- Are there maximum and minimum temperature range limits?
- Is there a minimum or a maximum time limit at these temperatures?
- Does it matter how long after irradiation the heat-treatment process is started?

GEX began its effort to develop a general specification for post-irradiation heat treatment of B3 film dosimeters as a part of the initial release of its WINdose Dosimetry System in 1999. GEX utilized the Risø practice as a baseline for testing and to determine answers to these basic questions. The 60.0°C temperature was validated by testing at temperatures ranging from 50.0°C to 70.0°C in 5.0°C increments for times from five to sixty minutes, in five minute increments. Beyond 60 minutes, the intervals were increased to one hour up to six hours, with a total tested time of twenty hours.

It was observed that temperatures below 55.0°C do not complete the B3 color development, and temperatures above 65.0°C could cause the B3 film surfaces to stick and even fuse together if they came into contact with one another, which destroys the film when the user tries to separate them from each other.

The conclusions based on these tests led to the formation of GEX recommendations with typical box-style incubators, also known at GEX as the P4800 Incubator. User recommendations indicated that targeted temperatures between 55.0°C and 65.0°C could be used to satisfactorily heat treat both bare B3 film as well as packaged B3 WINdose dosimeters, using a minimum five minute time period for bare B3 film, and a minimum fifteen minute time period for pre-packaged B3 dosimeters. The 15 minute time for the packaged dosimeters was to accommodate for the heat-sink response which is associated with the additional packaging material.

Initial testing in 1999 also confirmed that heat treatment at 60.0°C for times above six hours (results were for a single time period of approximately 20 hours) resulted in significant fading in the B3 film dosimeter. It was determined that a conservative maximum three hour time recommendation be established to avoid any possible fading.

After performance studies with P4800 incubators, it was evident that heat loss occurred inside the incubator when the door was opened in order to load and retrieve the B3 dosimeters. It was determined that it could take up to ten minutes for the incubator temperature to fully recover to specification. See Figure 1 below.

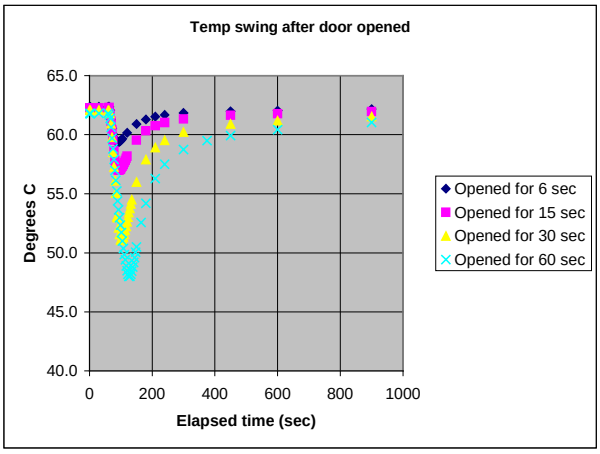

FIGURE 1

Above are results obtained with a P4800 incubator set to 60.0°C, with actual temperatures measured inside the B3 WINdose dosimeter pouch of approximately 62°C. The plot shows temperature recovery times associated with opening and closing the incubator to simulate dosimeter loading and unloading from the incubator.

B3 dosimeter users are encouraged to perform temperature mapping of their particular incubator systems at their facility to verify temperature uniformity inside the incubator.

NOTE: Heat treatment testing up to 70.0°C has not shown any indication of permanent damage B3 film dosimeters in terms of physical properties, unless two B3 film surfaces come into direct contact with one another. Post-irradiation heat treatment on B3 response function does not show a detectable change. It should be noted however, that the surface of B3 film begins to soften at temperatures near 60.0°C and above, and this softening is just enough that the two B3 film surfaces may stick to one another. GEX also recommends avoiding radiation process temperatures above 60.0°C. We realize that some applications may exceed 60.0°C and the recommendation is to use the films with caution. In fact, we recommend to use all chemical dosimeters with caution at temperature above 60.0°C.

METHOD / SOLUTIONS

Recommended Heat Treatment Temperature Specification:
The post-irradiation heat treatment specification temperature range (55.0°C to 65.0°C) established in 1999 has not changed, and can be used for the effective heat treatment of B3 film dosimeters today.

However, GEX has determined that utilizing a temperature setting of 58.5°C ± 1.0°C provides optimal B3 heat treatment results, and is an appropriate recommendation based on reported user success with this temperature setting. The 58.5°C temperature specification has been used internally by GEX since 2004, and has demonstrated to be a sufficiently high enough temperature to complete the color development and stabilize B3 film dosimeters, but low enough to minimize the potential of damage to the film surfaces in factory packaged products. For users that utilize the films outside of GEX packages, a higher minimum set point is acceptable up to 70.0°C.

The refined optimal temperature setting of 58.5°C was established after working closely with several users who reported B3 film surfaces sticking together following the heat treatment process. Investigations revealed significant temperature differences (>10.0°C) can exist between the upper and lower shelves in a box-style incubator. One simple resolution to improve temperature uniformity is to restrict dosimeter placement to the middle and/or upper shelves of the incubator.

Performance Requirements for an Optimum Heat-Treatment System when using packaged B3 Film Dosimeters:
Based on investigations into B3 user complaints about dosimeter films sticking together when there are more than one dosimeter in a sealed package, a series of tests were conducted that helped GEX to determine a more “optimum” heat treatment temperature of 58.5°C.

As part of product specification verification, the 55.0°C minimum temperature required for 100% color development and B3 dosimeter stabilization was re-validated in 2004. More importantly, however was the discovery that B3 film could actually reach full color development in less than ten seconds at the 55.0°C temperature. It was observed during these investigations that a trigger-like color development in B3 film dosimeters occurs once the minimum temperature is achieved throughout the dosimeter film. Figure 2 demonstrates confirmation of the 55.0°C minimum temperature requirement for post-irradiation heat treatment of B3 film dosimeters.

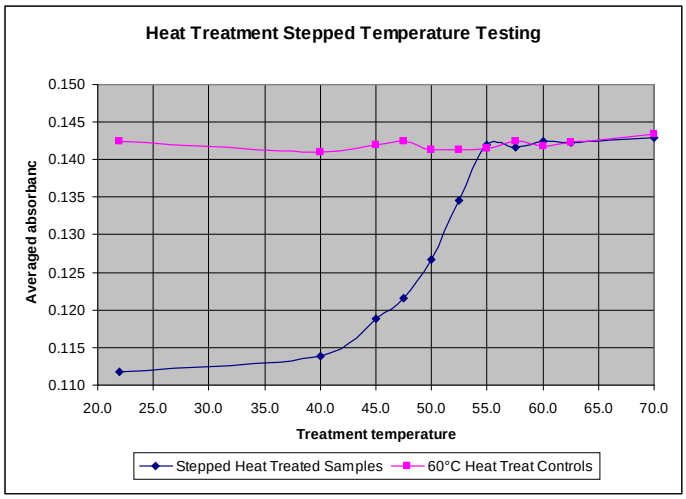

FIGURE 2

GEX also confirmed the early Risø findings that B3 dosimeters which have undergone effective post-irradiation heat treatment will remain stable for long periods of time. It was also confirmed that B3 dosimeter heat treatment intervention does not vary with the time after irradiation, and that there is no measurable difference in the results observed with sets of dosimeters that were heat-treated and measured immediately after irradiation compared with dosimeters that were held for up to fourteen days before being heat treated and measured.

Since then, process validation testing by customers has shown that the time of intervention can have an impact on the resulting measurement and should be considered before determining the SOP's for the heat treatment process. The impact of this on business operations needs to be evaluated based on the user's situation.

For those users whom are not in a rush, the best recommendation is to wait a couple hours before heat treatment. Users whom are in a hurry for measurement results may want to implement a process that treats the films much sooner. In such cases, the user should evaluate the process with performance of verification testing, which will be used to derive the measurement uncertainty contribution from post-irradiation handling. Keep in mind that the time begins when the dosimeter leaves the irradiation zone. This may be a while in gamma or certain e-beam designs and helps the dosimeter. It is important that each user spend the proper time to validate a process that meets their individual needs.

The Optimum B3 Heat Treatment System:
The P4900 Micro Incubator is the final output of GEX performance requirements and subsequent product specifications. The P4900 was specifically designed for post-irradiation heat-treatment of B3 dosimeters in either the WINdose or DoseStix factory pouches, and it provides uniform heat treatment (± 0.5°C) at the preset temperature (± 1.0°C) displayed on the front of the unit.

A five minute dwell time has been shown to be effective, and some users have validated shorter times. Testing of the P4900 Micro Incubator confirmed that B3 dosimeters can be left at 58.5°C for up to three hours without damage or measurable change in B3 dosimeter response values when compared with the response values of B3 films that were held at 58.5°C for only a few minutes.

Post-Irradiation Heat Treatment Options and Accessories:
GEX continues to offer its P4800 Incubator System as a cost effective solution for post-irradiation heat treatment of B3 dosimeters, although the P4900 Micro Incubator System is offered as the optimum solution. A new incubator (P4850 Forced Air Incubator) will be offered upon verification, and is a premium box-style incubator solution for this application.

The P4901 Digital Thermometer and P4902 T-Type Thermocouple B3 probe are accessories of the P4900 Incubator, and provides a simple, calibrated means of verifying the temperature settings of the P4900 Incubator as well as a way to validate that temperatures within each of the cavities of the P4900 Incubator are uniform. The user is encouraged to map and validate each incubator purchased under their chosen conditions of use. Not only is validation the proper approach, but the user will gain invaluable information in doing so.

The Appendix below provides a test method to verify the effectiveness of the P4900 Micro Incubator System. The protocol can be easily modified for use in validating other incubators. Users that wish to re-verify the GEX testing and/or establish their own specifications are encouraged to contact GEX customer service for assistance in planning their test protocol.

LIMITATIONS/PRECAUTIONS

The implementation and maintenance of heat treatment systems is the responsibility of the user. GEX cannot warrant our products when used without proper validation of the processes used to handle dosimeters under the actual conditions of use.

REFERENCES

GEX Technical Reports:

- *GEX TIR# 100-205, B3 Film Dosimetry*
- *GEX TIR# 100-210, Genesys 20 – General Practices and Information*

APPENDIX

Validation of a P4900 Heat Treatment System:
B3 Post-Irradiation Heat Treatment Validation Testing in the P4900 Incubator
Post-irradiation heat treatment of B3 dosimeters is intended to effectively complete the color development of B3 dosimeters resulting in stable B3 dosimeters. The effectiveness of the post-irradiation heat treatment process can be verified by monitoring the stability of the B3 dosimeter absorbance over time. The time period should extend to or beyond the expected potential measurement and re-measurement time period in which B3 dosimeters may be used. GEX currently recommends use of 58.5°C ± 1.0°C as the optimum post-irradiation heat treatment temperature setting for the P4900 Incubator.

Heat-Treatment Effectiveness Verification Criteria
B3 dosimeter stability should be demonstrated by reproducible absorbance measurements of ± 1.0% (excluding expected measurement instrument variance of 0.4%) compared with the initial B3 absorbance measurement throughout the complete time period tested in order to evidence B3 dosimeter stability.

Test Method
Sample the B3 dosimeter stock (DoseStix or WINdose format) for enough representative samples. The dosimeters should be irradiated to a low, medium and high dose levels (spanning the user's expected dose range). Use a minimum of three dosimeter packages per treatment time and temperature.

1. Test the 5 sub-groups of specified dwell times listed below:
   - 1 minute
   - 2 minutes
   - 4 minutes
   - 8 minutes
   - 16 minutes

2. Heat treat each sub-group at the 58.5°C GEX recommended temperature setting using any of the micro incubator wells for the time specified at a given time after irradiation.

3. Test sub-groups at the following post-irradiation intervals

4. Open the dosimeter pouches, and measure and record the absorbance values of all dosimeters into each of the sub-groups after applying heat treatment.

5. Re-measure each of the dosimeters once per day for a minimum of seven days. (Note: stability time test can be extended to accommodate longer time periods).

6. Compare the average of subsequent absorbance values against the average of the first measurement value of the absorbance for each sub-group of dosimeters. Results should be within ± 1.0% of the baseline absorbance values to satisfactorily verify stability.

REVISION CONTROL HISTORY

DATE	CHANGE DESCRIPTION	REVISION
05/03/16	The sections quantifying stability were changed to a softer one. The document was made more concise and direct. See ECO# 70244.	A

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