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Optical Isolation of Scintillating Tiles Using TiO_2 Doped Epoxy for the D0 ICD in Run II

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A Run II DØ Inter Cryostat Detector tile array will be composed of 16 identical modules. Each module contains 12 optically isolated scintillating tile elements, each with dimension of 0.1×0.1 in η and ϕ in the pseudorapidity region from 1.1 to 1.4. The 12 tiles in a module are formed by routing grooves in a single piece of scintillator - optical isolation is achieved by filling the grooves with a white reflective epoxy. The procedure for filling these isolation grooves is described here.

Contents

1 Overview

1.1 ICD tile array

The Inter Cryostat Detector (ICD) [1] is a single layer of scintillator placed on the inner face of each of the calorimeter end cryostats. The scintillator is cut to yield a 0.1 by 0.1 segmentation in η and ϕ. To minimize dead space between channels while retaining modularity (to ease construction, installation and cosmic ray testing), the tile array is composed of sixteen identical *tile modules*, each covering three units in η and four units in ϕ. Shown in Figure 1 is a schematic of the 16 identical modules which compose an ICD tile module array.

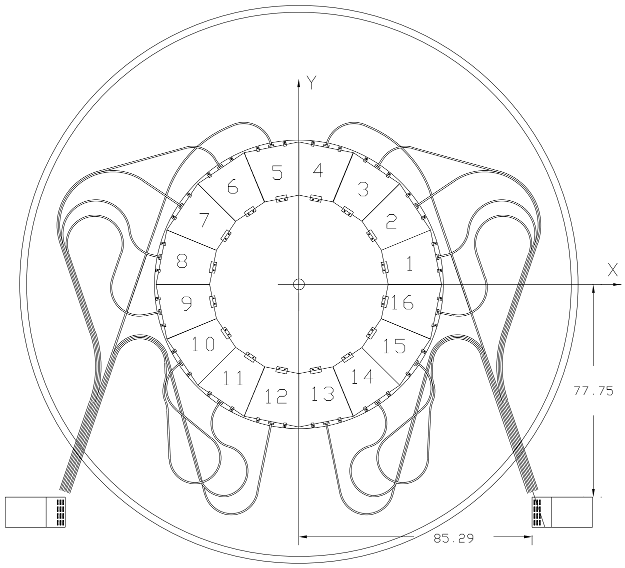

Figure 1: Schematic of the ICD tile array to be hung on the inner surface of each of the end cryostats for Run II. The array is composed of 16 identical tile modules.

1.2 ICD tile module

Figure 2 shows a schematic of the scintillator within an ICD tile module. Each tile unit is cut from a single 1/2″ thick sheet of scintillator 22″ square. Grooves for isolation (straight lines) and fiber (closed curved loops) as well as the final cut out are performed by the Thermwood routing machine in Lab 8 at Fermilab. Initially, isolation grooves are cut. There are 3 ϕ grooves and 2 η isolation grooves. Fiber grooves are cut after the epoxy is injected and has cured in the isolation grooves (the subject of this note). Finally, the outside edges of the tile unit are cut out.

Figure 3 shows a cross section of the isolation and fiber grooves in a tile. The cross hatched area indicates the region to by filled with epoxy.

The purpose of the epoxy in the isolation grooves is to optically isolate the 12 tiles in the module while holding them together in mechanically stable unit. The reflective (white) quality of clear 24 hour epoxy is obtained via doping with TiO_2 (Titanium DiOxide).

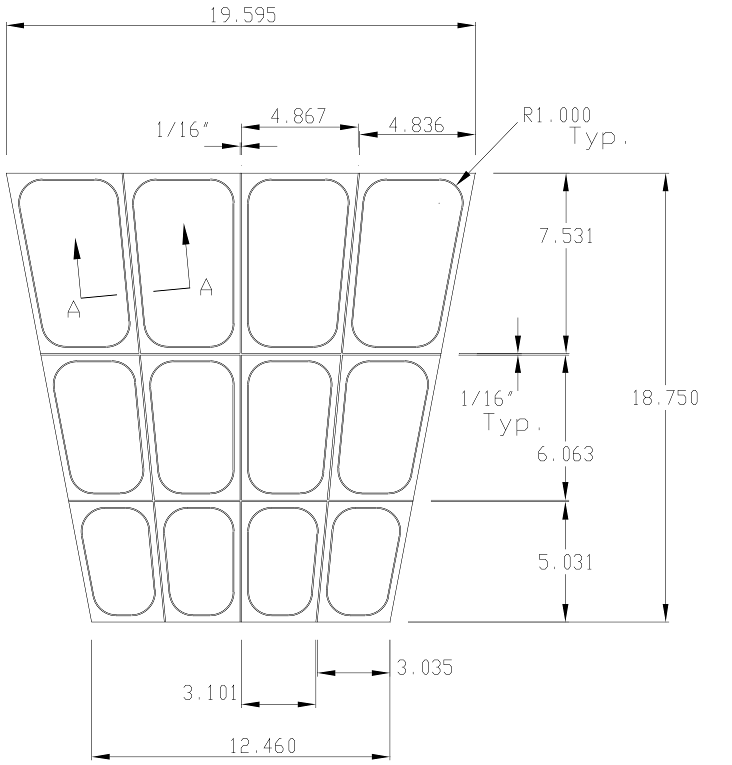

Figure 2: Schematic of the scintillator in an ICD tile module. Straight lines indicate the isolation grooves. WLS fibers are placed in the curved grooves.

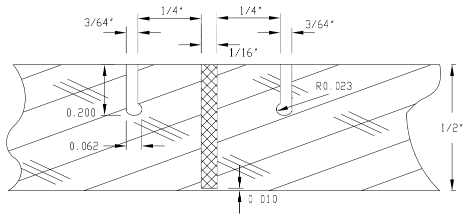

Figure 3: Schematic of a cross section of an ICD tile module showing the fiber and isolation grooves. The rounded key in the fiber grooves holds the fiber to the outer edge of the groove, eliminating the need for epoxy.

1.3 Quantity of materials required

Isolation grooves are nearly $1/2'' = 1.27$ cm deep. The groove width is about $1/4'' = 0.15875$ cm over a total length of about $92'' = 284$ cm (over five sections: 3 in ϕ and 2 in η). The quantity of epoxy needed for a single tile unit is therefore $1.27 \times 0.15875 \times 284\text{cm}^3 = 57\text{cm}^3$. In terms of weight (components are mixed by weight, not volume) about 40 grams of epoxy is combined with $(40 \times 32/150 =)8.53$ grams of hardener to fill one syringe. At least two 30 cc syringes will be required to inject epoxy into each tile unit.

The table below lists the quantity of materials needed for production of 40 tiles (to make 32 tile modules forming the array, plus 25% spares).

Material	Measure	Minimum quantity required	Production quantity (add 15%)
epoxy	by volume	2400 cm^3	
DER-332 epoxy resin (Texaco)	weight	3200 grams	3700 grams
TiO_2 powder (Dupont)	weight	1600 grams	1850 grams
epoxy hardener (Dow Chemical)	weight	683 grams	800 grams
30 cc syringes	unit	80	100

Table 1: Chemicals/materials required to fill ICD tile module isolation grooves with TiO_2 doped epoxy.

1.4 Precautions

Human skin oils are know to cause the surface of scintillator to craze, reducing the surface's reflective properties. Therefore, cotton or nylon gloves must be worn at all times when handling the tiles or anything that comes in contact with the tiles. Wash hands before putting gloves on because skin oils eventually will penetrate the gloves.

2 Producing TiO_2 Doped Resin

2.1 Materials required

Note: Precautions are indicated in parentheses. Please refer to the Material Safety Data Sheet for full details on precautions.

- DER-332 Epoxy Resin - (avoid contact with skin - clean spills or skin with ethyl alcohol). This material is a pure resin and may crystalize at room temperature - heat mildly near a space heater or put in an oven at 62 degrees centigrade for a few hours until enough resin is liquified for use.
- TiO_2 titanium dioxide [white dopant - a powder] (handle wearing gloves and face mask).
- Roalox [3] grinding mill jar [a jar made of wear-resistant chemical porcelain fortified with alumina, porcelain mixing balls].
- Jar mill which rolls the jar at a steady slow rate - about once every 8 seconds.
- Oven set at 62 degrees centigrade [large enough to hold the jar]
- Digital scale.
- Nylon gloves.

2.2 Doping procedure

- Note all batches in the log book including your name, time/date/ weight of measurements, time/temperature required to decrystalize resin (if any).
- Tare cup on scale for TiO_2 powder.
- Add 150 grams of powder to cup. Pour into mill jar.
- Using same cup, add 300 grams of DER-332 epoxy.
- Add epoxy to mill jar. Clean any spills around jar opening.
- Securely fasten the lid to the jar without overtightening.
- Place jar on the roller and engage power to rotate jar at slow speed (rotating the jar once every 8 seconds or so).
- Check the jar after 15 minutes for leakage around lid.
- Leave on roller for 24 hours. If leaving for the day, place jar in the oven at 62 degrees centigrade. Place on roller for one hour before adding curing agent (just before application).

3 Epoxy Application

3.1 Materials required

- TiO_2 doped epoxy resin as described in the previous section
- Jeffamine D-230 Curing Agent [hardener] - (avoid contact with skin - clean skin with ethyl alcohol)
- The following items are obtained from EFD, Inc [2]. Check the UTA cabinet at lab 8 and/or supplies in the DØ UTA office before ordering any additional items.
  - EFD manually controlled, air powered dispenser with hoses and adapters for compressed air and 30 cc syringes
  - 30 cc syringes [Between 2 and 3 syringes contain enough epoxy volume to complete a single ICD tile unit. EFD item number 5112CP-B]
  - needle tips , white piston caps, orange caps [EFD item numbers 5120-1.5-B-Pink, 5112PE-B, 5113-B]
  - barrel storage rack for holding syringes upright for filling [EFD item 910BR]
  - adjustable barrel production stand [EFD item 7300A]
- compressed air
- thin kapton tape - silicon based adhesive tape 1″ wide, 1.5 mils thick [4].
- thick kapton tape [1] - silicon based adhesive tape 1″ wide, 3 mils thick - available from the Fermilab stockroom as stock number 1130-2600 (acrylic based adhesive tapes are known to cause crazing of the scintillator).
- razor blades
- K-dry low lint, two-ply, absorbant, non-abrasive tissues
- ethyl alcohol for cleaning epoxy resin or hardener spills (isopropyl alcohol is normally used to clean the scintillator - CDF allowed k-dry tissues and ethyl alcohol be used to clean up epoxy spills on the scintillator surface).
- disposable mixing cups
- tongue depressors for stirring epoxy resin and hardener and for pressing air pockets out of the kapton tape on the tile
- calculator

[1] An alternative to this thicker kapton tape is a 3M product - blue colored tape with a silicon based adhesive. It was identified and used by Todd and Dan Ruggiero of the CMS group. It is available in 2″ wide 2.5 mil thick and 1″ wide and 3.5 mil thick varieties. In principle, it is the same as kapton, but does not have the electrical insulation properties (which are not needed here). The advantage to switching is that the blue tape is much cheaper than kapton ($7/roll versus $26/roll for kapton). Another advantage may be that the blue is more transparent than the kapton, making it easier to see the grooves.

3.2 Filling the isolation grooves

The procedure for getting the epoxy into the isolation grooves is described here. Insure that all needed materials are readily available before any white epoxy is poured from the mill jar. The procedure should be aborted immediately if any spillage occurs or is about to occur on the tile elements.

The tiles are cut with bridges halfway up the 3 ϕ isolation grooves. These bridges separate the isolation grooves on a tile into 2 distinct sections, the larger (lower η) section from the smaller (higher η) section. The purpose of these bridges is to eliminate multiply connected grooves during the epoxying process. It may be wise to move the bridges toward the larger section so that each section requires only one syringe of epoxy. Prepare all needed supplies before injection begins.

3.2.1 Epoxy and tile preparation

1. Clean nylon or cotton gloves must be worn while handling all materials.

2. Note all batches in the log book including your name, time/date/ weight of measurements, crock time in oven, crock time on roller.

3. Note the time in the logbook as the jar is taken out of the oven. Place it on the mill, operating at slow speed.

4. Place the tile on a clean flat surface. Ideally the tile is moved about and epoxied on a supportive white plastic sheet or 'stretcher' at least a few millimeters thick. Never place anything on the bare scintillator except the tape. Note any defects on the scintillator surface in the logbook. Some labeling scheme should be devised to distinguish scintillator tiles. Apply kapton tape on the back of the tile along any punch through segments along the isolation groove.

5. Blow any chips, dust or debris off the tile surface and from the isolation grooves with a compressed air gun.

6. Cut 1″ long pieces of the thinner kapton tape and place them centered at the intersection of all η and ϕ isolation grooves. Cut this tape with a razor blade to ensure a smooth cut edge. Make sure the tape does not wrinkle or bow into the groove at any point.

7. Use a single piece of thicker kapton tape to cover the entire length of each of the ϕ and η isolation grooves, extending at least an inch past the end of each groove. Press the tape down along either side of the groove. Do not press the tape into the groove.

8. Press all air pockets and seams out from underneath the kapton, especially at the overlap seams between the thicker and thinner tape using a tongue depressor.

9. The injection point in the larger and smaller η sections is the center-most crossing point since this minimizes the pressure required overall during injection. Place at

least 3 pieces of thicker kapton tape parallel to each other but overlapping slightly centered on each of the 2 injection points. Press out excess air.

10. Using the scribe, puncture a small hole at the end of each groove. These holes allow air to escape as the grooves are filled from the injection point to the end of the groove. There are 16 air escape holes per tile.

11. Cut 18 $3'' \times 3''$ pieces of K-dry wipes (at least one for every air hole plus two for the injection points).

12. Cut at least 18 $2''$ long pieces of thicker kapton tape, which are used to cover each scribed air hole once that section of groove is filled with epoxy. Keep these items within easy reach.

13. Scribe a small hole at the center of the two injection points using extra care not to make a single large cut. When the injection needle is inserted, the tape must seal well around the needle or leakage will occur.

14. Check the level and orientation of the barrel production stand. Adjust it so that it holds a 30 cc syringe equipped with a $1.5''$ needle pointing directly downward with the needle tip about 2 mm below the surface of the table the stand is resting on. Place the stand in position to inject at the injection point of the smaller side of the tile.

15. Note the time as the jar is taken off the mill.

16. Prepare 2 or 3 syringes with epoxy :

    - Place 2 or 3 30 cc syringes with orange barrel tip caps in the barrel holding rack.
    - Tare a cup on the scale.
    - Pour about 40 grams of doped epoxy resin into the cup (about 40 grams will fill one syringe). Note the exact amount in the log book.
    - Always keep the digital scale, the opening of the mill jar and the lid clean. Wipe up spills immediately with K-dry wipes (and ethyl alcohol).
    - Using the calculator, multiply the amount of resin by 32 and divide by 150. This is the amount of hardener that must be added. Tare the cup of doped resin. Add the calculated amount of hardener to the cup and note the amount in the logbook.
    - Stir the hardener into the doped resin for at least 3 minutes with a tongue depressor.
    - Pour epoxy into syringe, filling it so as to leave at least enough space for the white barrel piston and the barrel adapter assembly (about 2 times the height of the white piston).
    - Insert the white barrel piston. Turn the syringe over and unscrew the orange cap. Press the white piston into the syringe until all air has been released.

- Replace the orange cap on the syringe and place the syringe back into the rack.

17. Note the time that the epoxy is loaded and ready in the syringes.

3.2.2 Injection procedure

1. Ensure that all materials are ready and close at hand as described in the previous sub-section.

2. Power up and plug in the compressed air to the dispenser. Turn pressure to 20-30 psi (or 2 bar) on the EFD meter.

3. Attach an epoxy filled syringe to the adapter leading to the dispenser.

4. Replace the orange cap with a pink needle dispensing tip. If doing this for the first time on this pressure setting, press the foot pedal of the dispenser to briefly start the flow of epoxy through the needle into the trash or into a k-dry tissue. Adjust pressure to obtain a slow to moderate flow rate (not fast).

5. Clean off any epoxy from the needle tip and load the syringe into the production stand, gently popping the needle through the tape at the scribed injection point.

6. Press the foot pedal to start the epoxy flow. If epoxy leaks significantly above the tape, a new injection point must be chosen.

7. Continue pressing the foot pedal, stopping only if significant amounts of spillage on the surface appears (clean up larger spillage immediately!). Watch carefully to anticipate which grooves will be filled first.

8. STOP THE INJECTION ! if the syringe is in danger of running out of epoxy (air will be injected into the groove) or if spillage on the tape appears that might run onto the scintillator. Continue injection as soon as possible.

9. Increase the dispenser pressure if the flow rate is too slow (the leading edge of the flowing epoxy should move about 1-2 cm per second).

10. Release the pedal just as epoxy approaches each air hole. Pump the pedal until epoxy reaches each air hole - cover it with a 2″ piece of kapton before epoxy flows out of the hole, releasing the pedal. If epoxy flows out of the hole, quickly wipe it up with a k-dry tissue and then apply the piece of kapton.

11. Continue this process until all grooves are filled (all air holes are taped).

12. Remove syringe/stand from the injection hole, placing it to the side, taking care not to drip epoxy from the needle onto the scintillator. Remove the used syringe from the stand, replacing the pink tip with an orange cap. Note the amount of epoxy left in the syringe.

13. Clean up any spills around the injection point and cover the hole with a piece of kapton.

14. Choose another syringe full of epoxy. Replace the orange cap with a pink dispensing tip.

15. Mount this syringe into the stand and place the stand back on the tile with the needle penetrating the second injection point.

16. Repeat the above procedure, watching carefully for spillage. On this larger injection area, stop the injection before all the epoxy in the syringe is exhausted. More than one syringe may be required, and the excess from the syringe used on the first (smaller) injection may have enough to complete the larger injection area. The location of the bridges may be moved to equalize these areas so that only one 30 cc syringe is needed for each injection area.

17. Remove syringe/stand from the injection hole, placing it to the side, taking care not to drip epoxy from the needle on the scintillator.

18. Clean up any spills around the injection point and cover the hole with a piece of kapton.

19. Clean up any spillage and dispose of used wipes, syringes, etc.

20. Leftover epoxy in a syringe may be injected into gaps in a previously injected tile (see Section 4).

21. After about an hour, remove the 2″ long pieces of tape from the air holes at the tile **edges** (not the 6 interior air holes or at the injection points). The epoxy will shrink back from these points as it cures.

22. The curing time of the epoxy is 24 hours at room temperature or 8 hours at 100 degrees F.

If the epoxy resin cools for too long or the the resin/hardener sits too long, the mixture will cure (thicken) more quickly and become more difficult to dispense. If the epoxy resin is just out of the oven and on the mill for only a few minutes, the mixture will be very thin and more difficult to control. While generally these conditions are not fatal to the success of the process, adjusting following factors may make the procedure run more smoothly:

- time between taking the jar out of the oven and the time that the hardener is added
- time on the mill
- room temperature, humidity

The author found about 15 minutes to an hour on the mill gave the epoxy a good consistency for injection. Make a note of the time that injection was completed and the thickness of the epoxy at the start and the end of the injection process, adjusting the time accordingly.

4 Final Inspection

After the epoxy has cured, the tile is placed back on the Thermwood routing table to cut the fiber grooves, cut out the bridges in the 3 ϕ isolation grooves, and cut out the tile from the larger piece of scintillator. The bridge gaps and any gaps in the epoxy on the tile due to shrinkage or air bubbles must be filled with reflective material: fill gaps with either more epoxy (injected using a hand held syringe) or Bicron 620 white reflective paint. If epoxy is available, this option is preferred - it is easier to inject and though it requires 24 hours to cure, the Bicron paint takes longer to dry, especially for holes of any significant size.

Any dripped epoxy or soil on the tile surface will effect the internal reflective properties of the tile. Ideally, the tile must be clean and polished with no scratches for optimal performance. Drips of epoxy may be chipped off carefully with a razor blade, making sure not to scrape the surface. A mixture of 50/50 (50% isopropyl alcohol and 50% water) may be used to clean the scintillator surface using a clean cotton cloth. Paper wipes should be used sparingly and without applying pressure to avoid scratching the surface of the tiles.

Edges of the tile unit must be painted with Bicron 620 white reflective paint.

5 Acknowledgements

There are a number of individuals who have greatly assisted me in bringing this procedure to a useful state. Specifically, I'd most like to thank Howard Budd, Daniel Ruggerio, Ewa Skup, Lupe Rodriguez, Edna Turner, Marc Olsson, Bob Walker, Jay Hoffman and many others working on the CDF Run II upgrade and the CMS EM calorimeter for their careful detector development and documentation, assistance and cooperation.

References

[1] H. Lee Sawyer, *et al.*, "Technical Design Report for the Upgrade of the ICD for DØ Run II", DØ Internal Note 2686 and FERMILAB-TM-2041, June, 1997. `http://www-hep.uta.edu/~gallas/icd_tdr.ps`.

[2] EFD, Inc., Bryan J Gass, Application Specialist, Chicago Sales Office of EFD, Inc., 1 (800) 556-3484, East Providence, RI (401) 434-1680. `http://www.efd-inc.com/index5.htm`.

[3] U.S.Stoneware, 700 East Clark Street, East Palestine, Ohio, Phone: 330-426-4500, Fax: 330-426-1859 `http://www.usstoneware.thomasregister.com/olc/usstoneware/home.htm`.

[4] The thin kapton tape was purchased from J.J.Glenn, 396 Wegner Drive, West Chicago, IL 60185. (630) 293-7800, email: jjglenncor@aol.com `http://www.dwinc.com/cgi-bin/dwin/wccomp/8`, `http://www.jjglenncor@aol.com` or `http://www.jjglenn.com/`