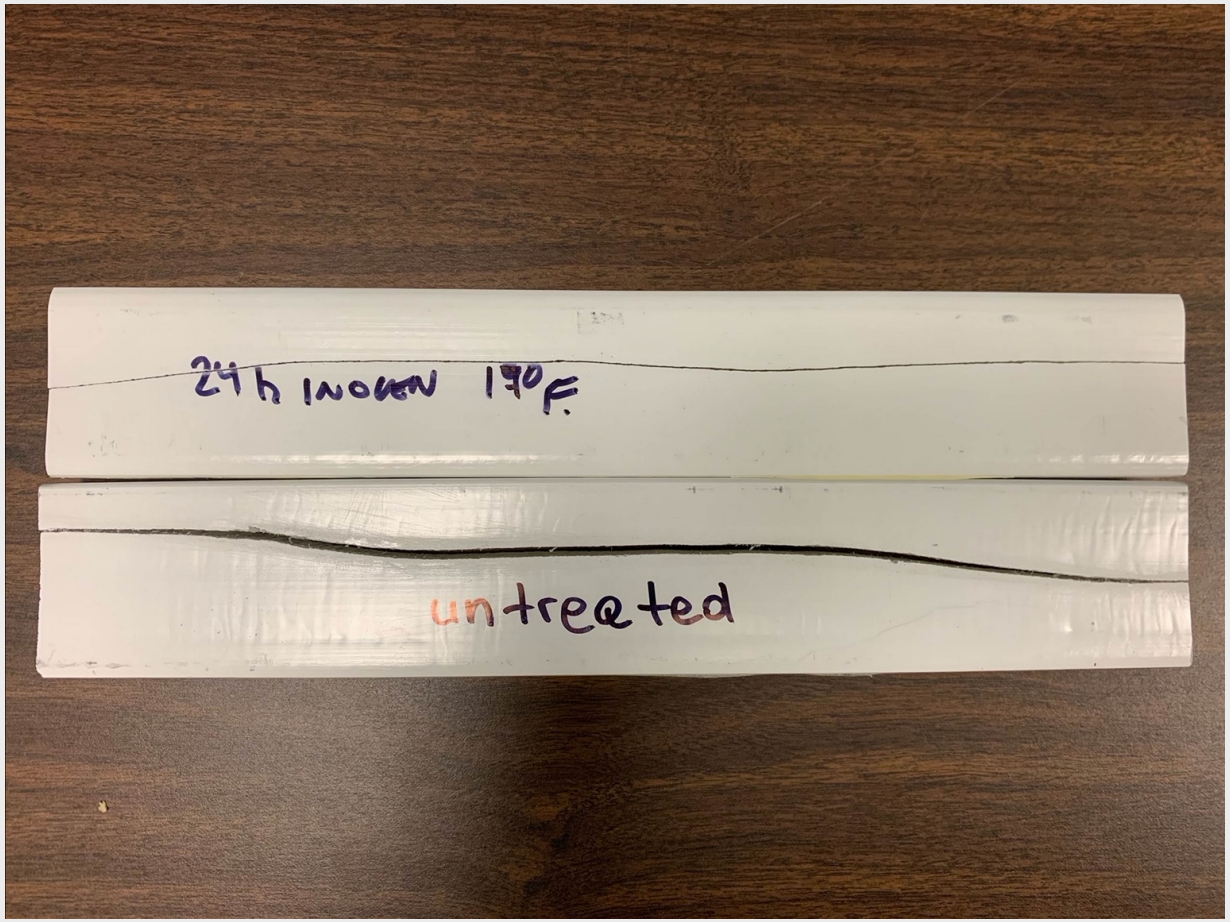
Annealing studies

Yuri Oksuzian and the UVa team

Annealing procedure

- CRV counters are exposed to a large temperature gradient during the production
  - This creates a large internal stresses that can be visualized



Because water evaporates very rapidly when maintained at a temperature of 184 degrees, Fahrenheit, ethylene glycol is often added to the bath. If it is used, the annealed styrene parts must be carefully washed after their treatment to remove any trace of glycol since the latter is not a very good electric insulator and does not dry readily from the plastic. Swishing the baskets in several changes of water held at a temperature of about 140 degrees, Fahrenheit, will usually do the rinsing satisfactorily.

ANNEALING TESTED — Early in the research work, it was found that in order to ascertain whether or not the annealing work was being done satisfactorily it was necessary to develop a quick, easy, and reliable test of the finished pieces. Consequently the "kerosine" test was worked out. In this test a representative piece of the annealed styrene is immersed in kerosine for one minute at room temperature —20 degrees, Centigrade, plus or minus five degrees—then removed without wiping and observed for results. If the piece is free from cracks at the end of 30 minutes after its removal from the kerosine the annealing is considered satisfactory. This test is applied only to such pieces as will not receive any subsequent machining.

In the case of long rods which will be machined later, however, the kerosine test is applied to a crosswise sample cut from the rod at a point distant about three diameters from one end and at least ¼ inch thick measured in the direction of the original length of the rod. Four small holes are drilled through the sample in the axial direction—one at the center, and the other three spaced at radial distances of ¼ of the radius, ½ the radius, and ¾ the radius from the center. The exact location of the holes is not too important, and the holes themselves may vary from 1/16 to 1/6 inch in diameter.

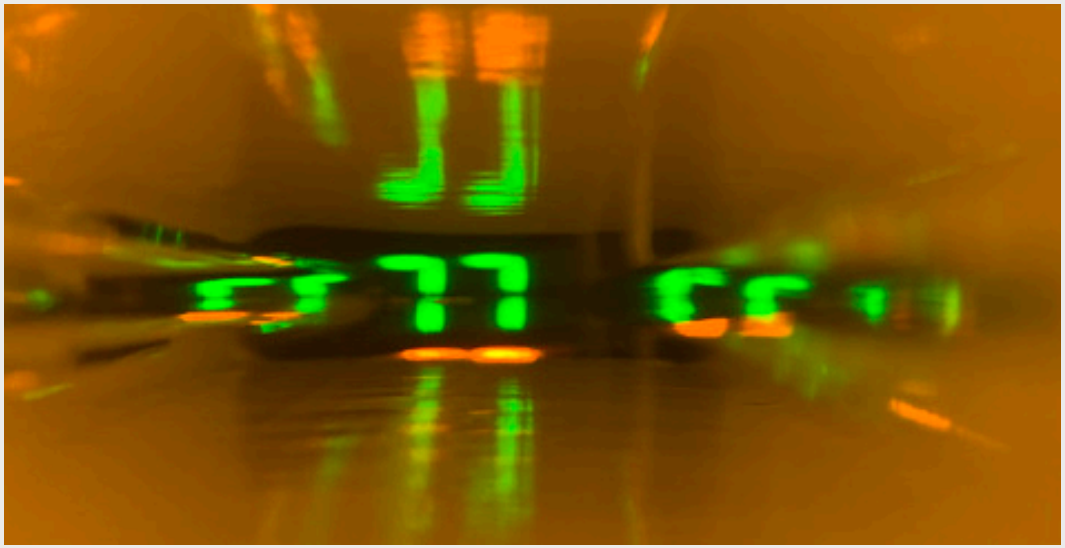
A wet saw and drills are are used in cutting out the specimen and in drilling the holes to avoid the introduction of stresses by these operations. Small cracks around the ends of the drilled holes which show no tendency to spread into the main body of the material when the piece is given the kerosine test may be disregarded and attributed solely to the drilling. The same is true of surface crazing along saw cuts. If a sample indicates deep body cracks, even though small, when given the kerosine test, a second test is made using more care during the preparation of the sample. If this piece also cracks, the annealing is not satisfactory. An accompanying photograph shows a properly annealed sample, a borderline case, and an unsatisfactory annealing job.

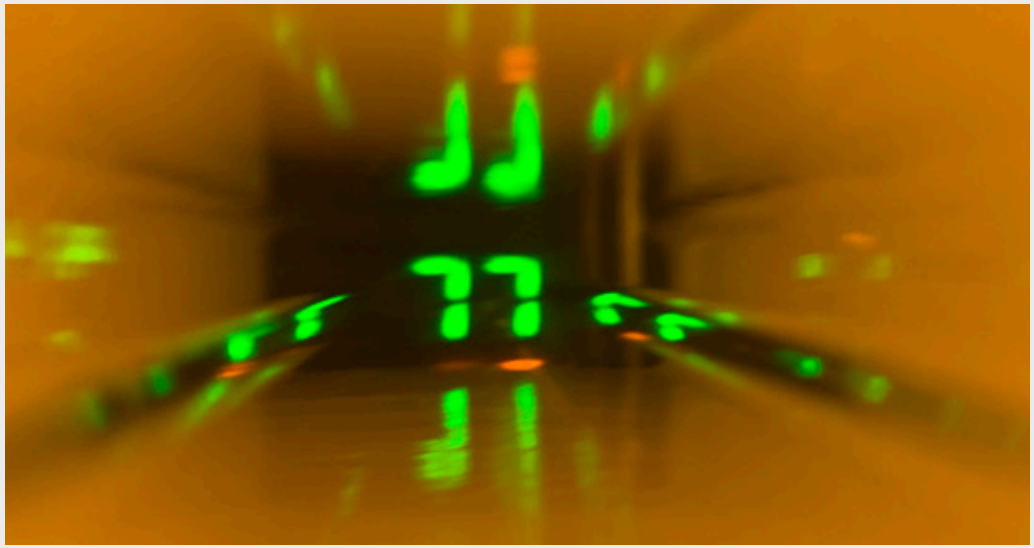
OTHER TECHNIQUES — The Bell Telephone Laboratories, among others, have worked upon the styrene annealing problem and specified a procedure for certain items. In this company's specification the procedure calls for an oven treatment at a single temperature of 170 degrees, Fahrenheit, followed by cooling in still air at room temperature. The maximum diameter of the part allowed for in this system is 1¾ inches, and the piece must be heated in an air oven at the specified temperature for 16 hours.

The advantage of this method is that the air oven employs a simple

Images distortion

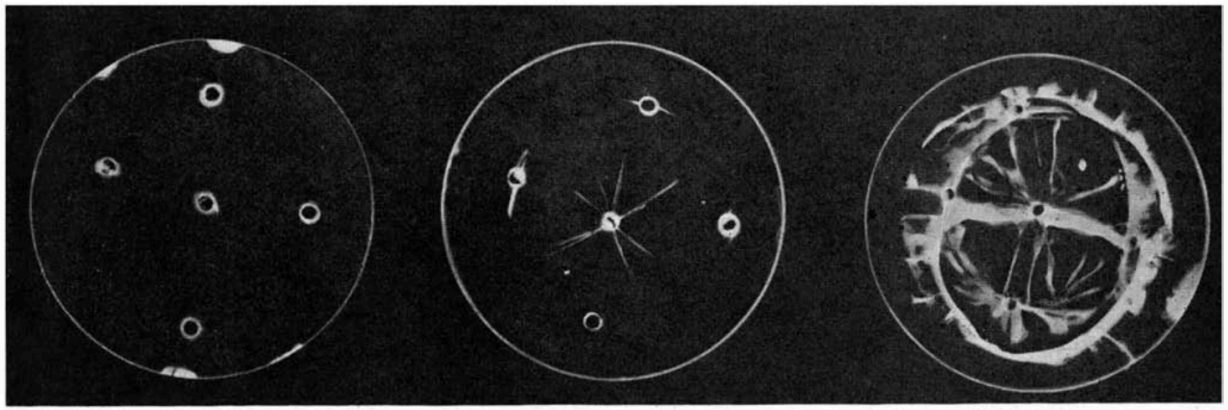
- Another technique to visualize internal stress is visual distortion test
- If looked through a 2’ long counter on distant objects:
  - Left: Uncured counter distorts images
  - Right: Annealed counter acts as a regular piece of glass
- It’s hard to capture this effect on the phone camera





“Kerosene” test

- Another technique to visualize internal stress is through a “kerosene” test
- “Kerosene” test 1:
  - Submerge a counter end into kerosene for 1 min
  - “If the piece is free from cracks at the end of 30 minutes after its removal from the kerosine the annealing is considered satisfactory.”
- Kerosene test 2:
  - Drill holes or cut a piece and repeat kerosene test. Use water as a coolant while drilling/cutting
  - This method might be more sensitive to deeper internal stresses



All illustrations courtesy Plax Corporation

Polystyrene disks, after kerosine test, show (left to right): proper annealing, unsatisfactory annealing, and an unannealed piece

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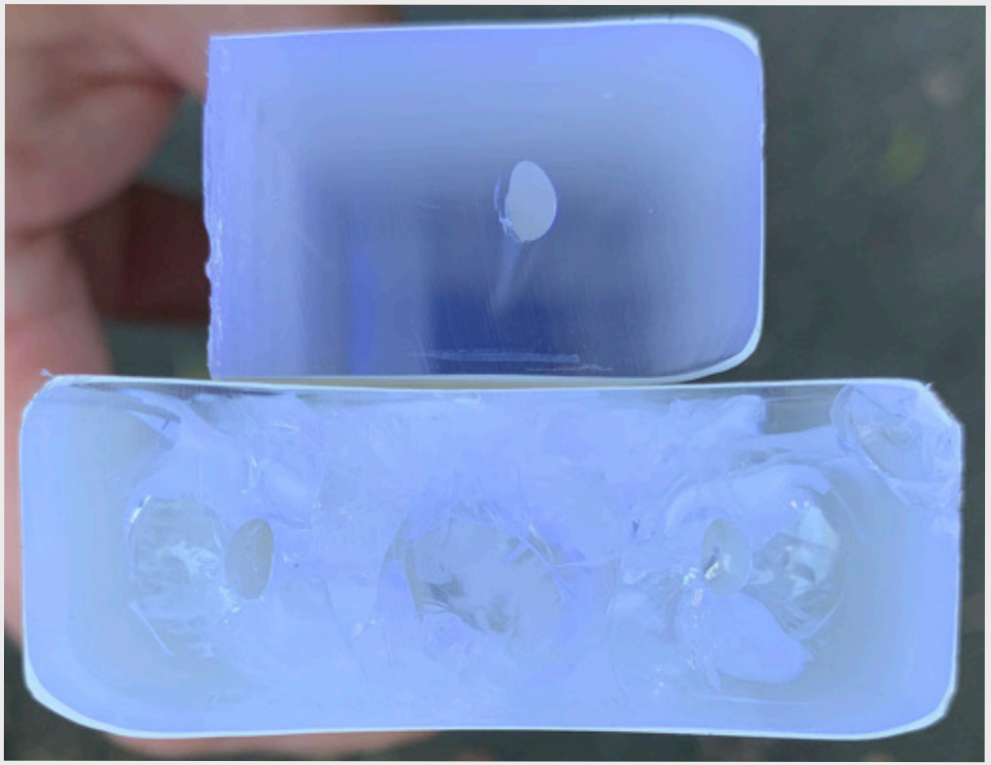
The advantage of this method is that the air oven employs a simple

“Kerosene” test in pictures

- Top: counter annealed for 24 hours at 170F in the oven
- Bottom: a production CRV counter
- Both counters were exposed to the “kerosene” test
- Left: Few minutes after the exposure
- Right: An hour after the exposure

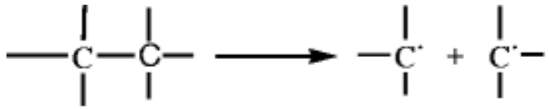


Yellowing due to aging

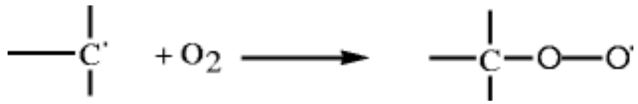
- We have recently received ‘fresh’ counters from Anna
  - The counters were produced in May 2021
- Left: annealed production CRV counter
- Middle: uncured production CRV counter
- Right: ‘fresh’ CRV counter
- Production CRV counters were produced in the first half of 2018

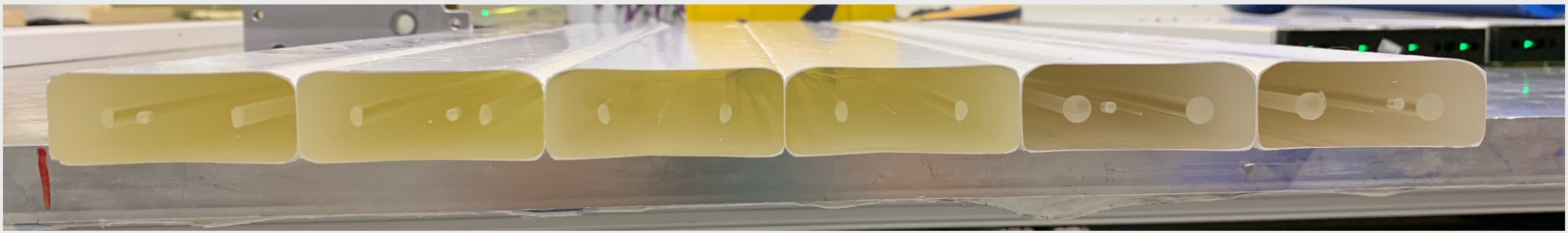
Mechanical degradation

This generally, refers to macroscopic effects brought about under the influence of shear forces. These forces result in the formation of macro radicals as follows:



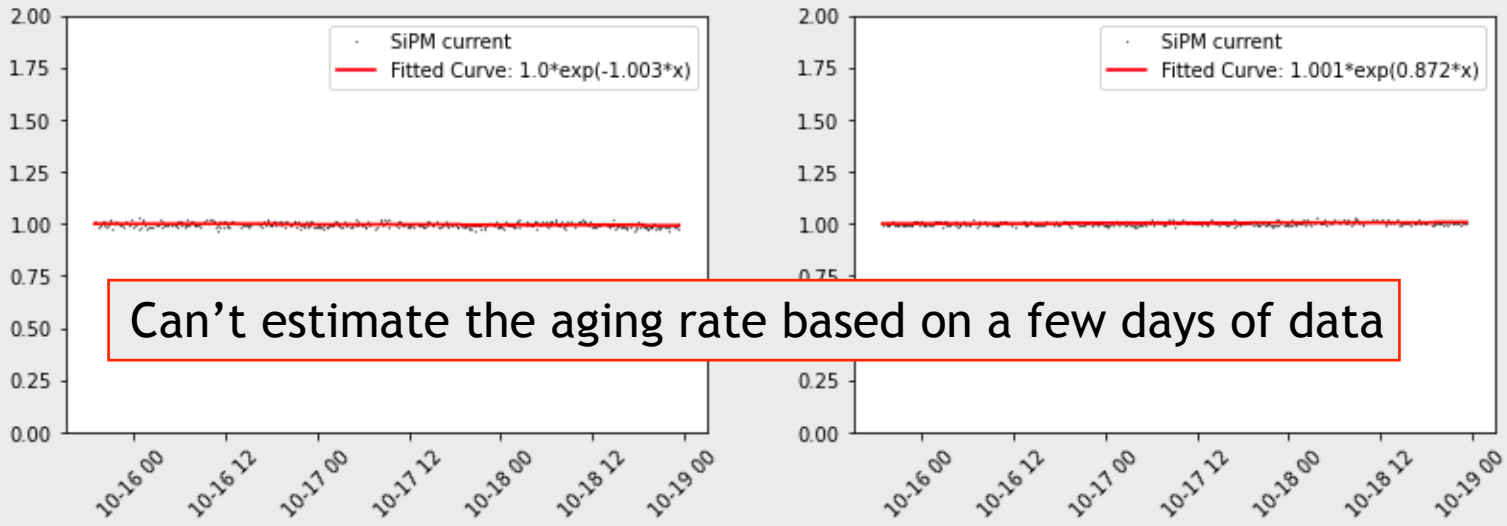
Such radicals can recombine in the absence of oxygen. In the presence of oxygen peroxy radicals may be formed, which leads to the degradation of polymeric chains Potts (1991).





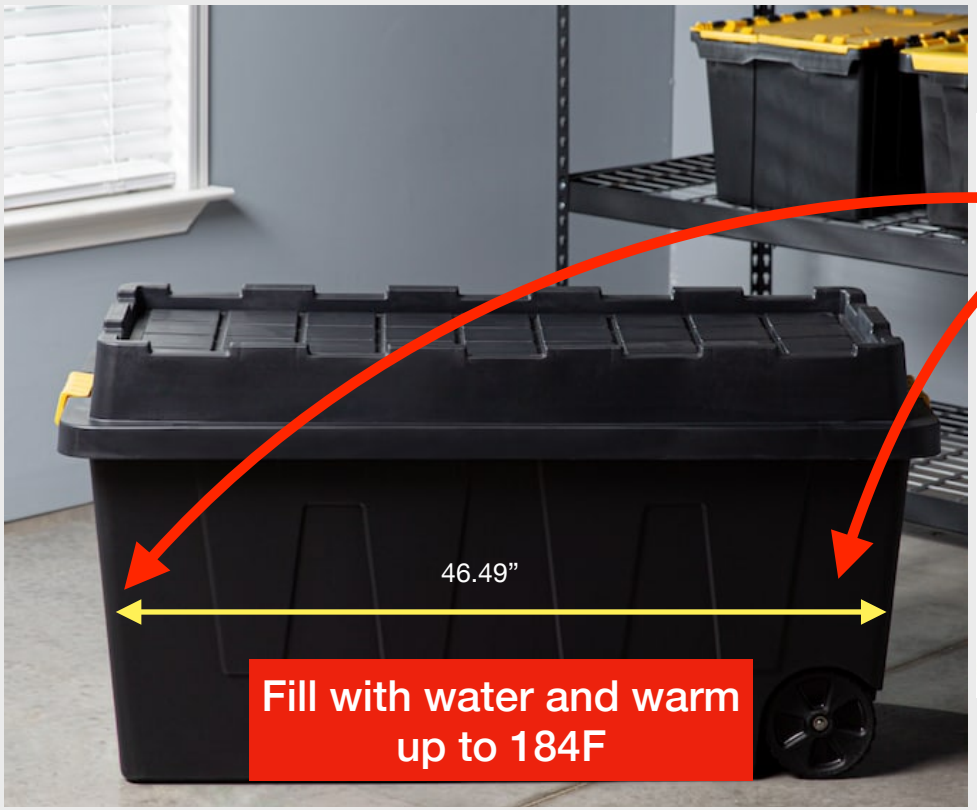
Annealing plan

- We have assembled multiple types of 2’ long di-counters:
  - Fresh (old) CRV di-counters
  - Annealed extrusions of fresh (old) CRV di-counters
  - Annealed fully assembled fresh (old) CRV di-counters
- Preliminary results suggest that annealing does not recover the light yield, but actually degrades (~5%) the performance
  - Annealing at lower temperature and/or shorter time periods may reduce a short term ‘damage’ from annealing
- We installed the counters at the test stand and plan to measure the aging rate



Items for annealing

- Items to purchase:
  - Container (50$), 2 x precision cooker ($70/each), plastic bag



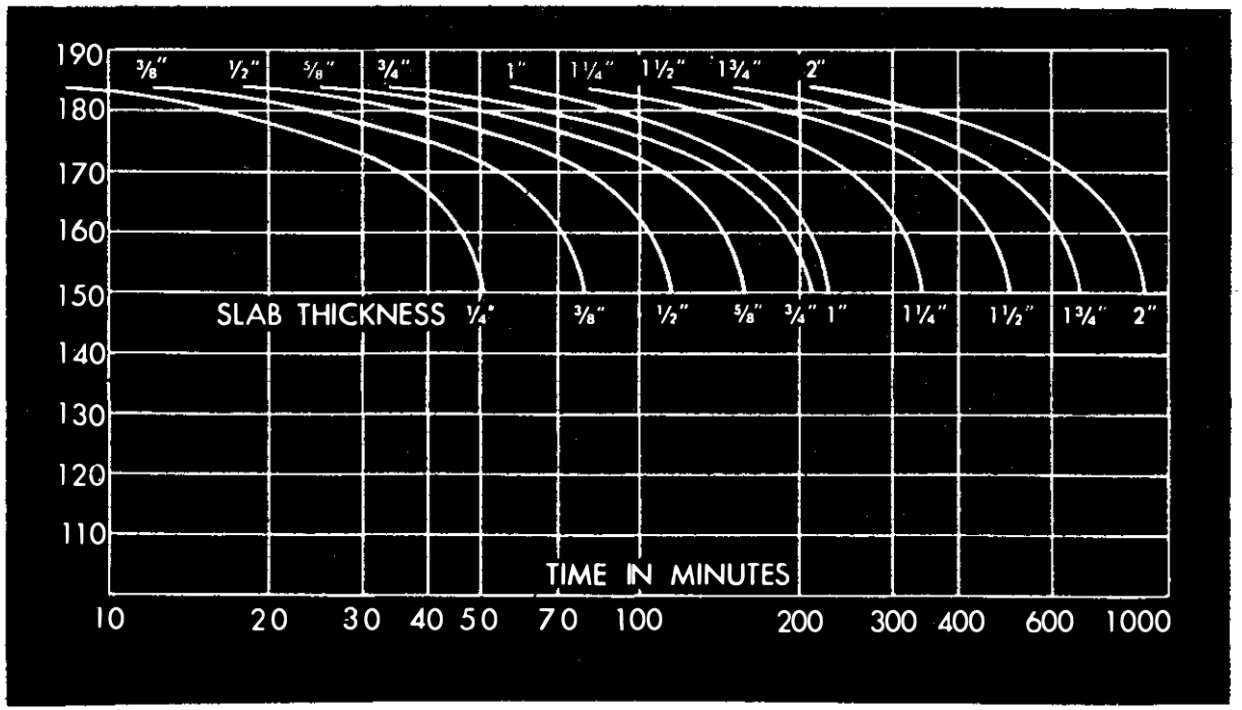
Heat water to a precision temperature value

Wrap extrusions

Annealing cycle

- Anneal at 184F for 4 hours
- Decrease the setting by 2 degrees every hour until 170F
- Decrease the setting by 5 degrees every hour until 160F
- Decrease the setting to 150F and let it run for an hour
- **We've never used this procedure since it's time consuming.**

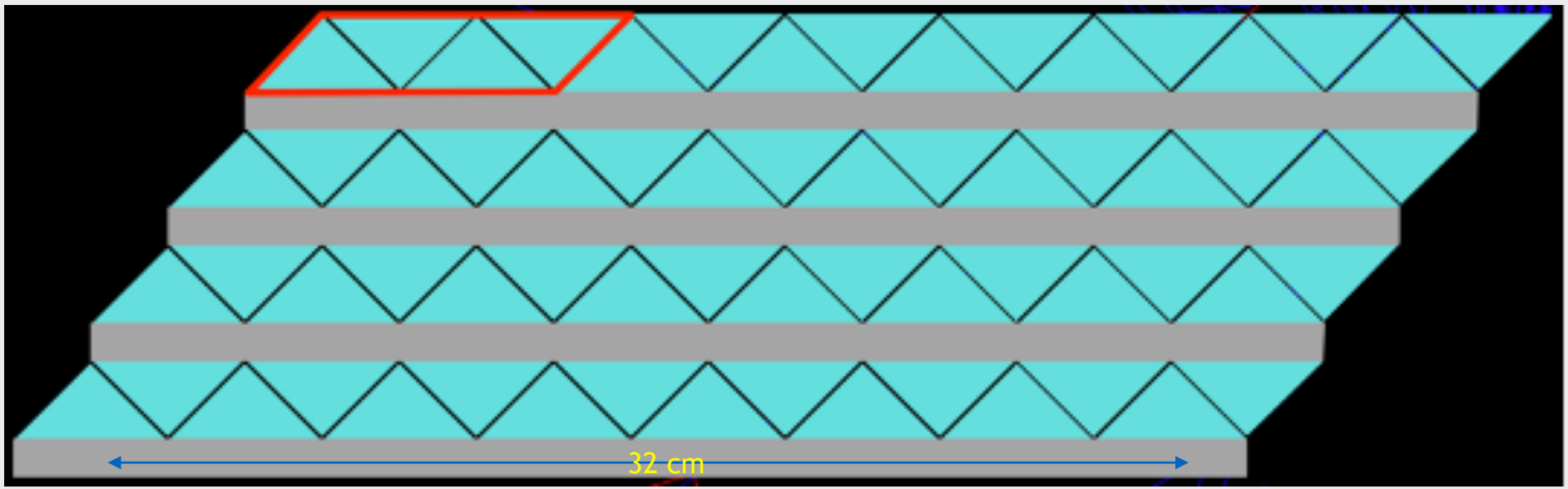
Temp	184	184	184	184	182	180	178	176	174	172	170	165	160	150	Air
Hour	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15



The four parts of each annealing cycle include the holding time at 184 degrees, Fahrenheit; the initial slow cooling rate, 184 to 170 degrees, Fahrenheit; the next slightly faster cooling rate from 170 to 160 degrees, Fahrenheit; and the final slightly faster cooling rate, 160 to 150 degrees, Fahrenheit. At 150 degrees, Fahrenheit, the parts are removed and allowed to air cool.

CRV prototype for Mu2e-II

- Consists of 16 quad-counters
- 1 or 2 meter long
- The prototype below will be able to directly measure 3/4 CRV efficiency
- 1 FEB is required to read-out the entire prototype below
- We can consider building individual layers and later stack them
  - We can start with a single layer and later scale it up
- This prototype can be used at MTest to measure the positional resolution



CRV prototype for Mu2e-II

- Few quad-counters were glued without FGB
  - There is currently no jig to properly glue counters
  - Because of that fiber holes on counters and FGB don't align well
- Only one quad-counter was assembled with fibers and FGB
- It should be easy to scale the production up…