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A Revised Glass Annealing Method to Distinguish Glass Types

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ABSTRACT: A revised glass annealing method is presented that results in the separation of tempered glass from the other common glass types. The method uses a temperature programmable muffle furnace that allows a very slow annealing process. The possibility of separating bottle glass from laminated windshield glass emerged. The ability to classify small pieces of glass removed from clothing as to type increases the value of existing glass databases compiled by forensic scientists and, thus, the value of glass evidence in the courts.

KEYWORDS: forensic science, glass, refractive index, glass classification, annealing, tempered, laminated

Much work has been done by the forensic science community to establish the value of glass evidence in a court of law. The ultimate goal is to turn glass from a "comparison" type of evidence to an "identification" type, something not yet possible. At present, a statistical approach using frequency of occurrence data is used to produce probability values. Databases of glass samples are built which rely on the glass standards submitted in criminal cases [*1,2*]. Physical properties such as color, density, refractive index, and thickness are measured and entered into these databases. A frequency of occurrence is assigned in casework, sometimes based only on microscopic observation and refractive index measurements. Since very different types of glass possess overlapping refractive index values [*1,2*], type cannot be determined from refractive index values alone.

Various approaches have been taken to determine glass origin and type [*3–9*]. Locke et al. [*3*] published an annealing method to separate toughened windshield from non-toughened window glass. Adopting Locke's work, Ryland [*4*] was able to separate tempered from non-tempered glass. Laminated glass (partially tempered) defied Ryland's classification attempts. My study used a programmable furnace which annealed the glass at a considerably slower rate than Ryland's, which permits all laminated glass to be separated from tempered. Using this equipment with an appropriately revised method, the change in refractive index (ΔN_D) was observed for multiple samples of the more common types of glass found in the United States.

Materials and Methods

Glass Samples

Laminated glass can be divided into two areas. The laminated glass used in automobile windshields, for example, in the United States, is a partially heat-strengthened glass

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which is produced by reheating nontempered glass to bend it. Flat laminated glass, such as would be found in patio doors, is not partially tempered.[2] This study concentrates on the partially tempered laminated glass. The other types of glass used are self-explanatory and are discussed below.

Equipment

A Fisher Scientific temperature programmable ashing furnace, Isotemp Model 495-A, with a fractional linear heating and cooling rates program, was used in the annealing routines. The furnace is programmable to 0.01°F min (17.77°C/min), with three sets of heating and three sets of cooling rate capabilities. The furnace's internal chamber measures 16.5 cm in width by 12 cm in height by 27.3 cm in depth. A stainless steel annealing block identical to the one described by Locke et al. [*3*] was fabricated for use as the sample holder.

Samples and Optical Measurements

The refractive index at the sodium *D* line, N_D (589 nm) for 60 window (float and nonfloat), 43 laminated, 29 tempered, 22 bottle and container, and 4 reinforced glass samples were determined, using the double variation method. All optical measurements were performed using the Mettler FP 52 hot stage for temperature control and the Bausch and Lomb grating monochromator for wavelength control. The size of the glass particles used in the study ranged between 0.8 and 1.5 mm at the longest dimension.

Temperature Stability Determination

The furnace and sample holder were tested to ensure their noninvolvement in the glass annealing process. The tests were run using a sample of tempered, laminated, window, and bottle glass. The N_D was determined for each of 16 samples from each of the 4 types of glass. The temperature distribution, across the 16 sample compartments of the sample holder, was indirectly observed, by determining the ΔN_D after annealing for each sample in the sample compartment. The ΔN_D for the 16 samples of each type varied no more than 1×10^{-4}. Next, the temperature distribution within the furnace's chamber was observed. The same 4 sample types were used. The difference in the ΔN_D values after annealing with the sample holder in the front of the oven, versus placement in the rear of the oven, ranged from 0 to 1×10^{-4}. Good temperature uniformity across the chamber and sample holder could be inferred.

Particle Shape Effects

The effects of glass particle shape were also observed. Both the flat and chunky glass samples of each type that fell within the size parameters listed above were annealed. The entire flat sample was crushed and the N_D determined. The same was done with the chunky samples. There were no significant differences found in ΔN_D that could be attributed to the shape of the glass particles.

Annealing Procedure

A temperature program was identified to produce the largest ΔN_D after annealing over a reasonable time period. The fractional heating and cooling program provided superior

[2]T. Draper, PPG Industries, personal communication, 1989.

flexibility in this part of the study. Locke et al.'s [3] temperature/rate/time values were used as a starting point. Twenty-two complete temperature/rate/time methods were evaluated. Much like Locke's approach, each succeeding method changed a value in order to attempt to optimize the variables. The method selected must allow the equipment to reproduce temperatures accurately over time. Table 1 contains a listing of the values tested. The method utilized in the study is outlined in Fig. 1.

The 158 glass samples, their N_D previously determined, were annealed using Method No. 22. Each of their N_D was measured after annealing and their ΔN_D was calculated. The original N_D versus ΔN_D was graphed (Fig. 2). The total time for a single run of Method 22 was approximately 28.5 h.

Results and Discussion

The separation of tempered glass from the other glass types is easily seen when the points are plotted on a graph (Fig. 2). Similar findings concerning tempered glass have been reported by Locke et al. [3] and Ryland [4]. The exact point of separation, that is, the ΔN_D value, is different in each study because it is directly dependent on the temperature/rate/time values used.

Both Locke et al. [3] and Ryland [4] felt it was advantageous to keep their programs less than 24 h in length to expedite casework. This study found it was not only advantageous, but also necessary, to make the program over 24 h in length to ensure that there were no electrical or mechanical interruptions with the equipment during the annealing. As has been stressed, temperature reproducibility is vital. With a program more than 24 h long, it could be started at 9:00 a.m. on one day. The next day it could be checked at 9:00 a.m. to ensure that the program was at the correct temperature and time values. With programs less than 24 h long, samples could be subjected to the wrong temperatures and go undetected if such precautions were not taken. If a shorter program were found to be as effective, the end of the program could be monitored by starting the program later in the first day. A computerized furnace that printed out temperature and time values at programed intervals would provide the most flexibility.

The lengthened cool-down process produced some added benefits. Tempered glass was separated not only from nontempered, but also from laminated glass. These results appeared to contradict previous findings by Ryland [4]. He reported laminated glass falling into both tempered and nontempered regions of Fig. 2.[3]

An interesting observation was noted with the 22 bottle-glass samples that were annealed: all exhibited a ΔN_D of 1×10^{-3} or less. All of the partially tempered laminated glass produced changes of 0.9×10^{-3} or greater (see Fig. 2). The laminated glass sample that produced a ΔN_D of 0.8×10^{-3} is not partially tempered. If glass annealing permits the separation of even some bottle glass from laminated, it becomes possible to eliminate or include the possibility that a very small piece of glass might have originated from an automobile in a hit-and-run, for example, or from a container in an assault or death investigation. The bottle-glass samples were taken from four areas of a standard bottle (see Fig. 3). No further work was done in this study with bottle glass.

Finally, all of the window samples with an original N_D of less than 1.5170 produced a ΔN_D of 1.1×10^{-3} to 1.7×10^{-3}. The N_D is large relative to window glass having an N_D greater than 1.5170. More window glass samples, along with bottle and laminated samples, having an N_D value of less than 1.5170 need to be run to assess the possibility of separating window, laminated, and bottle glass types in this N_D range.

[3]In personal communication (1988) with S. G. Ryland, the author requested his laminated samples that fell into the tempered region. He said he would rerun them instead, and subsequently found that the laminated samples did indeed fall into the nontempered region, thus confirming my findings.

TABLE 1—*Values tested for 22 complete temperature/rate/time methods.*

Method	R_1	T_1	H_1	R_4	T_4	H_4	R_5	T_5	H_5	R_6	T_6	H_6	Approximate Total Run Time, h	Annealing Rate, °C/h
1	20	600	720	1	240	1	10	40	1	...	...	...	14.5	60
2	20	600	1	1	240	1	10	40	1	...	...	...	8.5	60
3	20	650	1	1	240	1	10	40	1	...	...	...	9.5	60
4	20	700	1	1	240	1	10	40	1	...	...	...	10.5	60
5	20	550	1	1	240	1	10	40	1	...	...	...	7.5	60
6	20	500	1	1	240	1	10	40	1	...	...	...	7	60
7	20	550	1	0.13	480	1	10	40	1	...	...	...	7.5	8
8	20	550	1	0.05	480	1	10	40	1	...	...	...	28	3
9	20	600	1	0.05	240	1	10	40	1	...	...	...	123	3
10	20	600	1	0.05	480	1	10	40	1	...	...	...	45	3
11	20	600	1	0.05	400	1	10	40	1	...	...	...	70	3
12	20	600	1	0.05	450	1	10	40	1	...	...	...	54	3
13	20	550	1	0.05	400	1	10	40	1	...	...	...	53.5	3
14	20	550	1	0.05	450	1	10	40	1	...	...	...	37	3
15	20	550	1	0.10	450	1	10	40	1	...	...	...	20.5	6
16	20	550	1	0.03	450	1	10	40	1	...	...	...	59.5	2
17	20	550	1	0.15	450	1	10	40	1	...	...	...	15	9
18	20	550	1	0.08	450	1	5	40	1	...	...	...	24.5	5
19	20	550	1	0.07	450	1	8	40	1	...	...	...	27	4
20	20	450	1	10	40	1	8	39	1	...	...	...	4	...
21	20	550	1	0.07	450	1	10	120	1	5	40	1	27	4
22	20	550	30	0.07	440	1	5	80	1	3	40	1	28.5	4

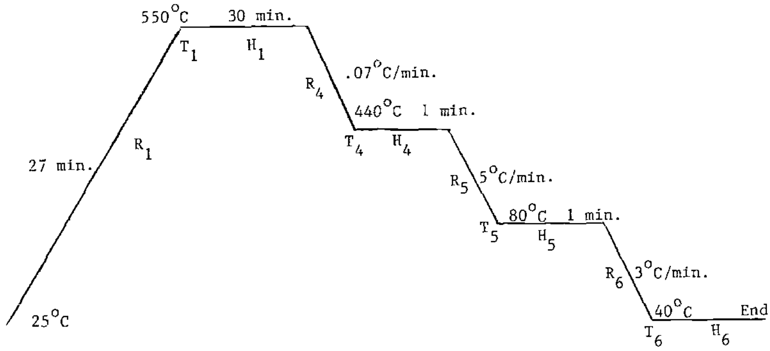

FIG. 1—*Values for Method 22.*

Conclusions

In glass annealing work, temperature reproducibility is paramount. The equipment, method, and technique must take this into account. A program run of over 24 h allows the analyst to check manually to ensure that the correct and entire temperature, rate, and time program were run by the equipment. The method evolved and used in this study demonstrates the ability to separate tempered from laminated and the other common types of glass encountered in casework. Other possibilities, including separating bottle glass and even some window glass, from both tempered and some laminated glass, emerged. More work needs to be done with glass annealing. The ability to classify glass as to type increases the value of the existing glass databases compiled by forensic scientists and thus the value of glass evidence in court.

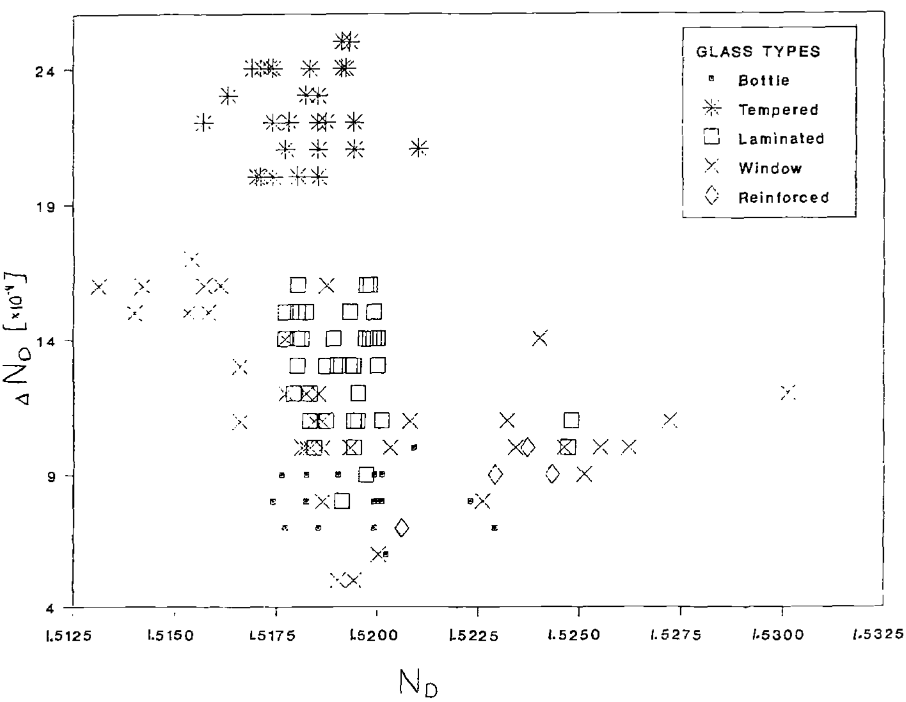

FIG. 2—N_D *versus* ΔN_D *by glass type.*

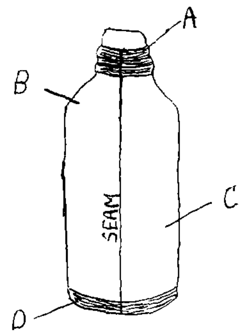

FIG. 3—*Four sample areas of bottle.*

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