

Validating Glass Annealing Thresholds in an ISO World

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Introduction

Annealing is a useful technique that can be applied to classify glass as tempered or non-tempered in situations where questioned glass fragments lack discriminative morphology. Toughened glass is commonly encountered in forensic cases due to its use as vehicle side window and large commercial windows. The toughening or tempering of glass is due to stress imparted during the manufacturing process. This stress induces a change in the refractive index of the glass. Annealing, the process of heating fast and cooling slow, relieves that stress and results in a change in the refractive index of the glass to a higher value. The magnitude of the change in refractive index can then be used to classify the origin of a glass as tempered, semi-tempered, or non-tempered.

In order to establish a data range that can distinguish these types of glasses, a database of pre- and post-annealed refractive indices is needed. At the United States Army Criminal Investigation Laboratory, a database of glass samples was created between 1993 and 1998 using the *Emmons Double Variation* method for refractive index determinations. The transition to ISO accreditation in 2010 spurred the need for a new validation. The ISO validation centered on determining the appropriate cut-off value for classifying tempered glass with newer RI technology.

Furthermore, since the National Institute of Standards and Technology (NIST) does not provide a tempered glass standard, one sample from an automotive side window was selected as a standard due to its known origin and characteristics typical of tempered glass. This sample was analyzed multiple times to determine reproducibility of the oven program and its potential as a control standard for annealing runs.

Experimental Design

A *Foster and Freeman GRIM 3* was used to determine the pre and post annealed refractive index (RI) values for the 43 glasses collected for this study. The samples were annealed by placing them in a 16 slot annealing block made of Type 316 stainless steel. A *Fisher Programmable Furnace, Model 495A*, with linear heating and cooling rates was utilized. The change in magnitude of the pre- and post-annealed refractive indices was recorded. These refractive indices were assessed to determine the threshold criteria for tempered and non-tempered glass classification specific to the equipment/method used in our laboratory.

Oven Program

- 20°C per minute to 200°C
- No hold
- 20°C per minute to 400°C
- No Hold
- 20°C per minute to 550°C
- Hold for 60 minutes
- Cool Down
- 0.07°C per minute to 500°C
- Hold for 1 minute
- 2.53°C per minute to 300°C
- No Hold
- 2.53°C per minute to 35°C

Glass Annealing Database Results

ID	Make/Model	Year	VIN/Class	Glass Type	Annealing Date	RI	Temp	Annealed RI	Temp	Change in Temp	Change in RI
1	Ford F150	2004	1FTPX14544N036338	semi-tempered	9/20/2010	1.52111	62.78	1.52274	58.43	4.35	0.00163
2	Ford Ranger	1999	1FTYR10C39PA94403	tempered	9/20/2010	1.51951	67.15	1.52182	60.90	6.25	0.00231
3	Ford Ranger	1997	1FTCR140V0926285	tempered	9/20/2010	1.51999	65.82	1.52225	59.72	6.10	0.00226
4	Mazda/B3000	1998	4F4YH16U2WTM15943	semi-tempered	9/20/2010	1.52102	63.01	1.52234	59.48	3.53	0.00132
5	Chevrolet Silverado C1500	2001	1GCEC19VX12170803	tempered	9/20/2010	1.52088	63.41	1.52348	56.38	7.03	0.00260
5	Chevrolet Silverado C1500	2001	1GCEC19VX12170803	tempered	1/28/2011	1.52099	63.13	1.52325	56.97	6.16	0.00226
6	Dodge Dakota	2000	1B7GL2N1Y5S87352	tempered	9/20/2010	1.52222	59.76	1.52454	53.47	6.29	0.00232
7 QC	Dodge Ram 2500	1998	3B7KC2622WM226368	tempered	9/20/2010	1.51982	66.37	1.52184	60.85	5.52	0.00202
7 QC	Dodge Ram 2500	1998	3B7KC2622WM226368	tempered	9/27/2010	1.51979	66.38	1.52182	60.85	5.53	0.00203
7 QC	Dodge Ram 2500	1998	3B7KC2622WM226368	tempered	9/29/2010	1.51979	66.38	1.52181	60.88	5.50	0.00202
7 QC	Dodge Ram 2500	1998	3B7KC2622WM226368	tempered	12/8/2010	1.51978	66.45	1.52182	60.89	5.56	0.00204
7 QC	Dodge Ram 2500	1998	3B7KC2622WM226368	tempered	1/28/2011	1.51977	66.46	1.52189	60.68	5.78	0.00212
8	Ford Explorer XLT	2006	1FMEU63E46JA00867	tempered	9/20/2010	1.52072	63.93	1.52289	57.97	5.96	0.00217
9	Ford Ranger	2006	1FTYR10U66FA86335	semi-tempered	9/27/2010	1.52129	62.30	1.52229	59.57	2.73	0.00100
11	Pontiac Grand Prix	1999	1GZWP12K70317229	tempered	9/27/2010	1.52138	62.12	1.52364	55.95	6.17	0.00226
12 a	Pontiac G6 GT	2006	1G2ZH55806A215640	tempered	9/27/2010	1.52127	62.42	1.52344	56.50	5.92	0.00217
12 b	Pontiac G6 GT	2006	1G2ZH55806A215640	semi-tempered	9/27/2010	1.52247	59.15	1.52354	56.22	2.93	0.00107
13	Pontiac Grand Prix SE	2003	1G2WKS5243F153641	tempered	9/27/2010	1.51914	68.24	1.52116	62.73	5.51	0.00202
15	Honda Accord	2004	1H4CM58834A118203	tempered	9/29/2010	1.52029	65.00	1.52262	58.67	6.33	0.00233
16	Chrysler PT Cruiser	2007	3A4FY48607T510684	tempered	9/29/2010	1.52081	63.61	1.52302	57.00	6.61	0.00223
19	Mazda Protege DX/LX	2002	1M1B022410470995	tempered	9/29/2010	1.52039	70.21	1.52073	63.84	6.37	0.00234
20	Acura Integra	1995	1H4DC46858030772	tempered	9/29/2010	1.52051	64.51	1.52296	58.66	5.85	0.00215
21	Nissan Altima	2005	1N4AL11D13W445543	semi-tempered	9/29/2010	1.52214	60.07	1.52347	56.47	3.60	0.00133
22	Volkswagen Jetta	1999	3VWRA81H0X0240834	tempered	9/29/2010	1.51822	70.77	1.52024	65.27	5.50	0.00202
23	Lexus LS-400	1991	1JTBFP11EXM0089192	tempered	9/29/2010	1.51535	78.60	1.51738	73.01	5.59	0.00203
24	Honda Civic	2005	1H4GS16365003584	tempered	9/29/2010	1.51890	68.91	1.52107	62.99	5.92	0.00217
25	Suzuki Verona	2005	KLSV156458156108	tempered	9/29/2010	1.51821	70.78	1.52037	64.92	5.86	0.00216
26	Dodge Caravan	2000	2B4GP2536YR811762	tempered	9/29/2010	1.52062	64.22	1.52275	58.41	5.81	0.00213
GBI Glass Collection Samples											
27 (09.20)	PPG Architectural Glass colored "Aurora"	na	sheet glass	non-tempered	12/8/2010	1.52231	59.61	1.52309	57.48	2.13	0.00078
28 (10.02)	PPG Architectural Glass colored "Caribbe"	na	sheet glass	non-tempered	12/8/2010	1.52303	57.40	1.52389	55.26	2.34	0.00086
29 (10.12)	Glass USA Corp	na	sheet glass	non-tempered	12/8/2010	1.51972	66.62	1.52057	64.30	2.32	0.00085
30 (10.18)	Tequila Beer Agave Line Anheuser Busch	na	container	non-tempered	12/8/2010	1.51876	69.27	1.5194	67.52	1.75	0.00064
31 (10.19)	New Castle Brown Ale	na	container	non-tempered	12/8/2010	1.52092	63.37	1.5216	61.53	1.84	0.00068
32 (10.21)	Mike's Hard Lemonade	na	container	non-tempered	12/8/2010	1.51812	71.02	1.51881	69.12	1.90	0.00069
33 (09.07)	Beering White Zinfandel 2000	na	container	non-tempered	1/28/2011	1.51915	68.14	1.51982	66.31	1.83	0.00067
34 (09.09)	Architectural Door GBI	na	sheet glass	tempered	1/28/2011	1.51495	79.62	1.51711	73.75	5.87	0.00216
35 (09.10)	Architectural Door Holiday Inn	na	sheet glass	tempered	1/28/2011	1.51800	71.30	1.51991	66.1	5.20	0.00193
37 (09.14)	Cutliss Ciera Oldsmobile	1984	GBI Auto	semi-tempered	1/28/2011	1.51999	65.89	1.52115	62.71	3.18	0.00116
38 (09.17)	PPG Architectural "Atlantica"	na	sheet glass	non-tempered	1/28/2011	1.52178	61.02	1.52268	58.54	2.48	0.00090
39 (10.05)	Architectural Glass Centers USA	na	sheet glass	tempered	1/28/2011	1.52039	64.81	1.52263	58.71	6.10	0.00224
40 (10.09)	Sliding Glass Door from GBI	na	sheet glass	non-tempered	1/28/2011	1.51611	76.50	1.51727	73.33	3.17	0.00116
41 (10.16)	Guadalupe Salva	na	container	non-tempered	1/28/2011	1.51618	76.28	1.51684	74.48	1.80	0.00066
42 (10.20)	Captain Morgan Spiced Rum	na	container	non-tempered	1/28/2011	1.51610	76.49	1.51690	74.29	2.20	0.00090
43 (10.32)	Mud Lubo Front Door	na	sheet glass	tempered	1/28/2011	1.51775	71.98	1.51972	66.61	5.37	0.00197
44 (10.38)	Northstar Grocery Store	na	sheet glass	tempered	1/28/2011	1.51807	71.11	1.52008	65.85	5.46	0.00201
45 (10.39)	Pharmacy Door	na	sheet glass	tempered	1/28/2011	1.51777	71.94	1.51971	66.64	5.30	0.00194
46 (10.76)	Sheet Glass Door	na	sheet glass	tempered	1/28/2011	1.51747	72.75	1.51967	66.76	5.99	0.00220

Summary

Tempered
RI Range
0.0026-0.00191

Semi-Tempered
RI Range:
0.00163-0.00100

Non-Tempered
RI Range
0.00090-0.00064

QC Tempered
Glass Average
RI: 0.00205

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Acknowledgments
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