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Blowout of Rubber Vulcanizates: Influences of Cure Systems, Content of Carbon Black, and Organic Additives

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Blowout of NR and SBR vulcanizates was studied using a microwave oven. Rubber vulcanizates with different contents of carbon black (0, 30, 50, 70 phr) and various cure systems (conventional, semi-EV, and EV) were prepared. Unfilled rubber vulcanizates did not explode by irradiation of microwave, while carbon black-filled ones exploded within 10 min. A blowout time of the carbon black-filled rubber vulcanizate decreases with an increase of the content of carbon black in the vulcanizate. A blowout temperature of the organic additive-extracted vulcanizate is higher than that of the not-extracted one, but the extracted vulcanizate blows out faster than the not-extracted one. A blowout temperature of the overcured vulcanizate is higher than that of the undercured one with the same cure system. Temperatures of unfilled SBR vulcanizates heated by the microwave irradiation are lower than those of unfilled NR ones. The carbon black-filled SBR vulcanizates blow out at higher temperatures than the carbon black-filled NR ones. Blowout times of the carbon black-filled SBR vulcanizates are longer than those of the carbon black-filled NR ones.

Introduction

When rubber is deformed, some part of the energy of deformation is transformed into heat as a result of various dissipative processes. When thick rubber blocks are subjected to repeated deformations, they can become so hot in the interior that they explode. This phenomenon is known as *blowout*. It is an important mode of failure in thick rubber articles, such as tire treads and tank track pads.¹ It is attributed to thermal decomposition of the rubber compound when the heat generated internally is not conducted away rapidly enough and the internal temperature becomes high enough to cause decomposition. Volatile products then develop an internal pressure sufficiently large to tear open a path to the exterior.

It should perhaps be pointed out that there is not at present a single, well-accepted, mechanism for blowout. It was first thought to be a mechanical fatigue cracking process, aggravated by the high temperatures set up by cycling stressing.² Other studies have attributed the failure to a biaxial tensile fracture in the center of the block, where the material is also weakened by high temperature.³ Failure is attributed to the development of an internal pressure large enough to expand any pre-existing internal cavity to the

point of rupture. A similar hypothesis has been shown to account for internal fractures produced in rubber by superheating dissolved liquids.⁴

Engelhardt reported variation of blowout times of NR vulcanizates due to types of carbon black using a flexometer.⁵ Gent and Hindi studied blowout phenomena of NR (natural rubber), SBR (styrene butadiene rubber), BR (butadiene rubber), and IIR (butyl rubber) vulcanizates using a flexometer and a microwave oven.⁶ They found that the blowout temperatures measured using a flexometer were consistent with those measured using a microwave oven, although the times taken to reach blowout were quite different. The times taken to reach blowout in the microwave oven are much faster than those in the flexometer experiments by over 10 times. In this study, blowout experiments were carried out using a microwave oven to study the influences of organic additives in rubber vulcanizates, content of carbon black, and cure system and time on blowout.

Experimental

Thick rubber specimens, 12 mm thick, were made in a compression mold. The mix formulations and vulcanizing

Table 1. Formulations, cure characteristics, and total crosslink densities of NR vulcanizates

Compound	NR1	NR2	NR3	NR4	NR5	NR6
SMR20	100.0	100.0	100.0	100.0	100.0	100.0
N351	0.0	30.0	50.0	70.0	50.0	50.0
ZnO	5.0	5.0	5.0	5.0	5.0	5.0
Stearic acid	3.0	3.0	3.0	3.0	3.0	3.0
Oil	5.0	5.0	5.0	5.0	5.0	5.0
TBBS	1.2	1.2	1.2	1.2	0.6	2.5
Sulfur	1.2	1.2	1.2	1.2	2.5	0.6
Rheometer (MDR 2000) at 160 °C						
ML (dN·m)	0.17	0.26	0.37	0.62	0.39	0.37
MH (dN·m)	1.14	1.92	2.55	3.56	3.00	2.52
t_{02} (min)	2.28	1.48	1.25	1.08	0.95	1.27
t_{90} (min)	9.98	6.12	5.33	4.33	6.10	7.12
Undercure time (min) at 160 °C (overcure time: 20 min)						
	9	5	4	4	4	5
Total crosslink density ($\times 10^3$ g·mol/mL)						
Undercured	3.17	1.55	2.51	2.73	3.32	2.25
Overcured	3.23	2.66	2.61	2.84	3.72	2.43

conditions are given in Tables 1 and 2 for NR and SBR compounds, respectively. NR and SBR compounds are most popular as a tread one of tire. Rubber compounds with different contents of carbon black (0, 30, 50, and 70 phr) and with different cure systems (conventional, semi-EV, and EV cure systems) were prepared. Rubber vulcanization with a high sulfur content is called as conventional cure system (NR5 and SBR5) and those with a high ratio of accelerator to sulfur are called as EV cure system (NR6 and SBR6). A conventional vulcanizate consists mainly of polysulfidic crosslinks, while an EV vulcanizate consists mostly of monosulfidic crosslinks.⁷ Cure characteristics of NR and SBR compounds were also listed in Tables 1 and 2, respectively. Cure characteristics were obtained using a Flexsys rheometer (MDR 2000) at a frequency of 100 cycles/min and ± 1.5 arc for the vulcanizing temperature of 160 °C.

The overcured NR and SBR vulcanizates were prepared by curing at 160 °C for 20 and 40 min, respectively. The undercured NR and SBR vulcanizates were prepared by curing at 160 °C for shorter than t_{90} . The undercure times are also listed in Tables 1 and 2. The mono- and disulfidic crosslinks in the overcured vulcanizates are more than those in undercured ones.⁸ The total crosslink densities of the vulcanizates were measured by the swelling method.⁹ Organic additives in the vulcanizates were removed by extraction with THF and *n*-hexane each for 1 day. After extraction of organic additives, the samples were dried for 3 days at room temperature. *n*-Decane was employed as a swelling solvent. The total crosslink densities of the vulcanizates were also summarized in Tables 1 and 2. The total crosslink density of the overcured vulcanizates are higher than those of the undercured ones.

In order to investigate the influence of organic additives in the vulcanizates on blowout, organic additive-extracted vulcanizates were prepared by extraction with solution of acetonitrile/acetone/methanol (40/40/20 volume %). Organic additives in the vulcanizates were extracted in the solution for 3 days and this process was repeated twice. The ex-

Table 2. Formulations, cure characteristics, and total crosslink densities of SBR vulcanizates

Compound	SBR1	SBR2	SBR3	SBR4	SBR5	SBR6
SMR20	100.0	100.0	100.0	100.0	100.0	100.0
N351	0.0	30.0	50.0	70.0	50.0	50.0
ZnO	5.0	5.0	5.0	5.0	5.0	5.0
Stearic acid	3.0	3.0	3.0	3.0	3.0	3.0
Oil	5.0	5.0	5.0	5.0	5.0	5.0
TBBS	1.2	1.2	1.2	1.2	0.6	2.5
Sulfur	1.2	1.2	1.2	1.2	2.5	0.6
Rheometer (MDR 2000) at 160 °C						
ML (dN·m)	0.13	0.26	0.41	0.76	0.42	0.41
MH (dN·m)	1.04	1.82	2.43	3.43	3.14	2.30
t_{02} (min)	4.18	2.77	2.27	1.73	2.43	2.35
t_{90} (min)	24.02	20.13	17.38	15.17	23.17	15.03
Undercure time (min) at 160 °C (overcure time: 40 min)						
	17	15	12	11	16	11
Total crosslink density ($\times 10^3$ g·mol/mL)						
Undercured	1.43	1.29	1.33	1.53	0.93	0.25
Overcured	1.51	1.45	1.63	1.69	1.61	1.54

tracted samples were dried for 7 days in a vacuum chamber at the room temperature.

Blowout experiments were carried out using a Samsung RE-777BR microwave oven with a microwave frequency of 2450 MHz. The irradiation power was set with 450 W. Blowout temperatures were measured with a thermocouple thermometer kept in a boiling oil bath. The temperature of the oil bath was about 210 °C. Experiments were performed three times and averaged.

Results and Discussion

Temperatures in the center of the rubber blocks were measured immediately after blowout. The blowout temperatures (T_b) and times (t_b) are given in Tables 3 and 4 for NR and SBR vulcanizates, respectively. Figure 1 gives blowout sample and its cross section of the overcured NR4 vulcanizate. The blowout sample has a large region decomposed. There are sticky substances and soots in the decomposed region.

The NR vulcanizates blow out at lower temperatures than

Table 3. Blowout temperatures T_b and times t_b of filled NR vulcanizates

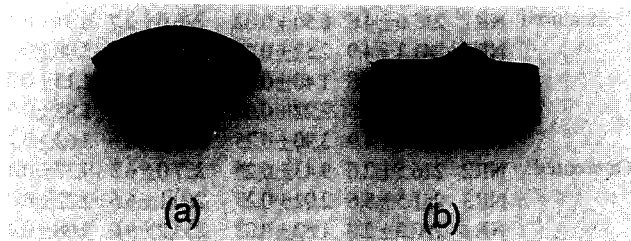
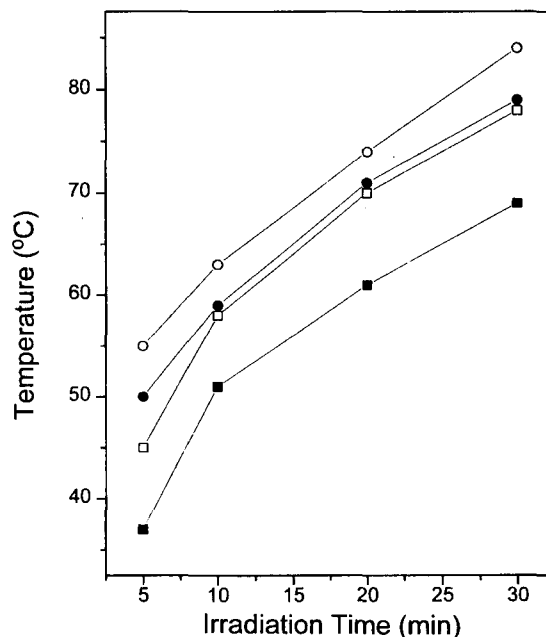
Vulcanizate	not-extracted		extracted		
	T_b (°C)	t_b (min)	T_b (°C)	t_b (min)	
Undercured	NR2	213.6 \pm 4.8	6.50 \pm 0.02	226.0 \pm 2.7	6.31 \pm 0.22
	NR3	241.1 \pm 4.9	2.52 \pm 0.27	247.1 \pm 9.6	2.08 \pm 0.23
	NR4	244.3 \pm 1.8	1.40 \pm 0.18	250.1 \pm 6.4	1.15 \pm 0.05
	NR5	246.9 \pm 9.5	2.22 \pm 0.08	257.3 \pm 8.0	1.83 \pm 0.22
	NR6	230.3 \pm 4.9	3.90 \pm 0.25	231.6 \pm 0.6	3.62 \pm 0.27
	Overcured	NR2	216.5 \pm 2.6	9.43 \pm 0.25	227.0 \pm 6.3
NR3		251.5 \pm 5.6	2.92 \pm 0.27	260.1 \pm 6.6	2.22 \pm 0.07
NR4		250.3 \pm 2.3	1.82 \pm 0.03	253.2 \pm 9.0	1.08 \pm 0.45
NR5		259.1 \pm 2.0	2.52 \pm 0.20	264.1 \pm 8.4	2.27 \pm 0.30
NR6		262.9 \pm 4.5	2.73 \pm 0.03	264.3 \pm 7.9	2.42 \pm 0.77

Table 4. Blowout temperatures T_b and times t_b of filled SBR vulcanizates

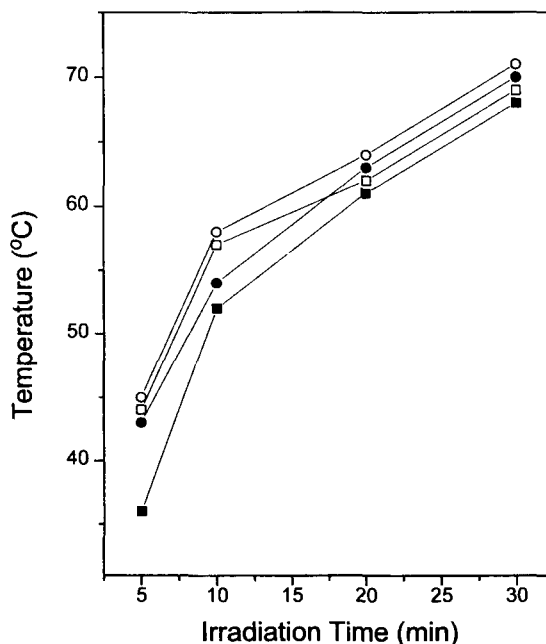
Vulcanizate	not-extracted		extracted		
	T_b (°C)	t_b (min)	T_b (°C)	t_b (min)	
Undercured	SBR2	242.5±2.5	7.73±0.03	257.6±6.0	7.32±0.37
	SBR3	305.7±4.2	4.10±0.27	308.2±8.6	4.02±0.33
	SBR4	296.1±4.5	2.20±0.23	298.2±8.4	2.15±0.05
	SBR5	296.8±1.0	3.83±0.07	297.6±8.7	3.72±0.33
	SBR6	293.5±3.6	4.45±0.33	296.0±1.0	3.87±0.23
	Overcured	SBR2	264.7±9.6	6.83±0.25	266.7±4.6
SBR3		305.9±5.1	2.73±0.37	331.3±7.6	2.70±0.28
SBR4		313.9±8.3	2.18±0.52	322.0±9.7	2.13±0.02
SBR5		301.9±8.2	3.85±0.08	302.2±8.4	3.83±0.17
SBR6		298.8±9.2	3.67±0.32	301.6±7.9	3.12±0.18

the SBR ones with the same contents of carbon black and cure system by over 30 °C as demonstrated in Tables 3 and 4. The NR vulcanizates blow out at 213-264 °C, while the SBR ones blow out at 242-331 °C. Blowout times of the NR vulcanizates are shorter than those of the SBR ones except overcured NR2 and NR3 vulcanizates. Gent and Hindi⁶ reported that different elastomers had strikingly different blowout temperatures. They also reported that IIR compounds blew out at relatively low temperatures, about 180 °C, whereas NR and SBR compounds blew out at temperatures of about 200 °C or higher. These values are lower than those obtained from this study by over 50 °C. This is due to the initial temperature of the thermocouple thermometer. In our work, the thermocouple thermometer was kept in a boiling oil bath with about 210 °C to minimize the temperature difference between a blowout sample and the measuring probe. Thus, our results may be closer to a real blowout temperature since the temperature of the thermometer probe is close to the blowout temperature.

Unfilled rubber vulcanizates (NR1 and SBR1) did not blow out, while carbon black-filled rubber vulcanizates blew out. This means that one of principal factors to cause blowout is carbon black. Temperatures of the unfilled vulcanizates increase by increasing irradiation time. Figures 2 and 3 show the temperature increases of the unfilled vulcanizates with an increase of irradiation time. The temperatures of the undercured vulcanizates are lower than those of the overcured ones. This may be due to the crosslink density and the length of crosslinks. When the crosslink density of the vulcanizate is high or the length of crosslinks is short, motion of a rubber chain is restricted so that much part of the energy of microwave can be

**Figure 1.** Blowout sample (a) and its cross section (b) of the overcured NR4 vulcanizate.**Figure 2.** Variation of temperatures of unfilled NR vulcanizates depending on irradiation time. Solid and open symbols indicate not-extracted and extracted vulcanizates, respectively. Squares and circles indicate under- and overcured vulcanizates, respectively.

transformed into heat. Thus, the unfilled vulcanizates with a high crosslink density or a high ratio of monosulfidic crosslinks are heated faster than those with a low crosslink density or a low ratio of monosulfidic crosslinks. The temperatures of the organic additive-extracted vulcanizates are higher than those of the not-extracted ones. This may be be-

**Figure 3.** Variation of temperature of unfilled SBR vulcanizates depending on irradiation time. Solid and open symbols indicate not-extracted and extracted vulcanizates, respectively. Squares and circles indicate under- and overcured vulcanizates, respectively.

cause some parts of irradiated microwave are absorbed by the organic additives or the heat generated in the rubber chains is dissipated by collision of the organic additives.

Rubber vulcanizates with the same cure system but different cure times have different crosslink types and densities. Rubber vulcanizates with different crosslink types and densities were found to have different blowout temperatures and times. The overcured vulcanizates blow out at higher temperatures than the undercured ones by 3-32 °C as listed in Tables 3 and 4. This is due to not only crosslink density but also crosslink type. When the vulcanizates having a high crosslink density, they will hold a highly internal pressure. Since mono- or disulfide linkages are stronger than polysulfide linkages by more than 30 kJ/mol,¹⁰ the vulcanizates with a high ratio of mono- and disulfide linkages will blow out at higher temperatures than those with a low ratio of mono- and disulfide ones. The crosslink densities of the overcured vulcanizates are higher than those of the undercured ones as listed in Tables 1 and 2. Since a ratio of mono- and disulfidic crosslinks in a rubber vulcanizate increases with an increase of cure time,¹¹ the ratio of mono- and disulfidic crosslinks in the overcured vulcanizates will be higher than that in the undercured ones.

The vulcanizates with a high crosslink density blow out at higher temperatures than those with a low one. Let us compare blowout temperatures of the vulcanizates having the same content of carbon black, 50 phr. The NR vulcanizates containing 50 phr of carbon black are NR3, NR5, and NR6. The SBR vulcanizates containing 50 phr of carbon black are SBR3, SBR5, and SBR6. For the undercured NR vulcanizates, the higher its crosslink density is the higher its blowout temperature is. Crosslink densities of the undercured NR3, NR5, and NR6 vulcanizates are 2.51, 3.32, and 2.25×10^{-3} g·mol/mL, respectively, and their blowout temperatures are 241, 246, and 230 °C, respectively. However, the overcured NR vulcanizates do not follow this trend. The crosslink density of the overcured NR6 vulcanizate is lower than those of the overcured NR3 and NR5 ones, but its blowout temperature is higher than those of the overcured NR3 and NR5 ones. This may be due to the crosslink type. The NR6 compound has a EV cure system which gives a high ratio of monosulfide linkages. As discussed previously, the vulcanizates with a high ratio of monosulfidic crosslinks blow out at high temperatures than those with a low ratio of monosulfidic crosslinks. Thus, this can lead to a conclusion that blowout is mainly initiated by dissociation of sulfur crosslinks. For the SBR vulcanizates, not only the undercured vulcanizates but also the overcured ones, the vulcanizates with a high crosslink density blow out at higher temperature than those with a low crosslink density. This is due to non-reversion characteristics and a relatively low polysulfidic crosslinks of a SBR compound.¹² Since a ratio of crosslink types of SBR compounds is nearly constant irrespective of cure times, the ratio of monosulfidic crosslinks of the undercured SBR vulcanizates will be close to that of the overcured ones.

The content of carbon black has a great influence on the blowout temperatures and times of the filled rubber vulcanizates. The blowout temperatures of the filled vulcanizates remarkably increase by about 28-61 °C as the content of carbon black in the vulcanizate increases from 30

phr (NR2 and SBR2) to 50 phr (NR3 and SBR3). By increasing the content of carbon black from 50 to 70 phr, the blowout temperatures of the filled vulcanizates increase (undercured NR4 and overcured SBR4) or decrease (overcured NR4 and undercured SBR4). The blowout times of the filled vulcanizates strikingly decrease by increasing the content of carbon black. By increasing the content of carbon black from 30 to 50 phr and from 50 to 70 phr, the blowout time becomes faster by about twice. This is due to a high thermal conductivity of carbon black.^{13,14} Thermal conductivities of NR compounds containing HAF black of 0, 20, 40, 60 phr are 0.175, 0.247, 0.301, and 0.331 W/m·K, respectively.¹³ Since the thermal conductivity of the vulcanizate increases with an increase of the content of carbon black, the more the content of carbon black in the vulcanizate is the faster the heat transfer becomes. Thus, the vulcanizate with a high content of carbon black blows out more rapidly than that with a low content of carbon black.

In order to investigate the influence of organic additives in the vulcanizates on blowout, the organic additive-extracted samples were prepared. The organic additives are vaporized by irradiation of microwave and then develop an internal pressure. Thus, one can expect that a rubber block containing volatile materials blows out at lower temperatures than that not containing ones. The experimental results listed in Tables 3 and 4 show this expectation. The blowout temperatures of the organic additive-extracted vulcanizates are higher than those of the not-extracted ones by about 1-9 and 1-26 °C for NR and SBR vulcanizates, respectively. However, the blowout times of the organic additive-extracted vulcanizates are shorter than those of the not-extracted ones. This may be due to the microwave absorption by the organic additives and the heat transfer from rubber and carbon black to the organic additives by collision as discussed previously. When the organic additives absorb microwave or the heated rubber and carbon black are cooled by collision with the organic additives, the irradiation times taken to occur blowout will become longer.

Conclusion

Unfilled rubber vulcanizates did not exploded by irradiation of microwave and their temperatures increase remarkably with an increase of the irradiation time. Temperatures of unfilled SBR vulcanizates heated by the microwave irradiation are lower than those of unfilled NR ones. A blowout time of the carbon black-filled rubber vulcanizate decreases by increasing the content of carbon black in the vulcanizate. The extracted vulcanizates blow out faster than the not-extracted ones. A blowout temperature of the overcured vulcanizate is higher than that of the undercured one with the same cure system. Blowout times of the carbon black-filled SBR vulcanizates are slower than those of the carbon black-filled NR ones.

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Dynamics of Poly[oxy-1,4-phenyleneoxy-2-{6-(4-(4-butylphenylazo)phenoxy)hexyloxy}terephthaloyl] and Poly[oxy-1,4-phenyleneoxy-2-{10-(4-(4-butylphenylazo)phenoxy)decyloxy}terephthaloyl] Studied by ¹³C CP-MAS NMR

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Carbon-13 CP-MAS NMR techniques were used to investigate dynamics of new combined type liquid crystalline polymers, poly[oxy-1,4-phenyleneoxy-2-{6-(4-(4-butylphenylazo)phenoxy)hexyloxy}terephthaloyl] and poly[oxy-1,4-phenyleneoxy-2-{10-(4-(4-butylphenylazo)phenoxy)decyloxy}terephthaloyl]. Noticeable mobility change of either aromatic groups or methylene groups is not detected between 25 °C and 82 °C from ¹³C spin-lattice relaxation time in the rotating frame ($T_{1\rho}(C)$) and contact time array experiments. However, line shape analysis shows the increase of mobility of methylene carbons in poly[oxy-1,4-phenyleneoxy-2-{6-(4-(4-butylphenylazo)phenoxy)hexyloxy}terephthaloyl] at higher temperature. The dynamics of side chains does not seem to be affected in our experimental temperature range by the length of aliphatic chain which is connecting the side chain group to the main chain.

Introduction

The macroscopic physical properties of solid polymers are mainly determined by their molecular level structure and dynamics. Orientational order plays an important role in solid polymers. The degree of orientation is enhanced by drawing, which leads to the improvement of mechanical properties. Molecular dynamics also influences physical properties, for example, the molecular motion of the rates in the range of the frequency of the alternating mechanical or electric fields is strongly correlated with a change of dynamic moduli or compliances.¹⁻³ The energy-dissipating local molecular motion slowed down too much produces brittle materials, on the other hand, the increase of molecular motion can result in the decrease of the strength of the materials.¹

New combined type liquid crystalline polymers(LCP), poly[oxy-1,4-phenyleneoxy-2-{6-(4-(4-butylphenylazo)phenoxy)hexyloxy}terephthaloyl] and poly[oxy-1,4-phenyleneoxy-2-{10-(4-(4-butylphenylazo)phenoxy)decyloxy}terephthaloyl] (from now on, will be abbreviated as PAZO-6 and PAZO-10, respectively), consist of poly(*p*-terephthalate) main chain and azo group with a aliphatic chain, as a side chain, attached to the main chain. The molecular formulas of the PAZO samples are shown in Scheme 1, where *n* indicates the number of methylene carbon between poly(*p*-terephthalate) and the azo group (*n* is 6 in PAZO-6 and 10 in PAZO-10). Since the alkyl chain length of the side chains may influence the structure and dynamics of the polymers, it is worthwhile to compare the structure and dynamics of PAZO-6 and PAZO-10.

NMR line shapes are sensitive to molecular motions. As the motions are fast and isotropic, the line shapes become narrow in the extreme narrowing limit. In general, the observed full line width at half height $(\pi T_2)^{-1}$ can be described as

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