Effects of the Presence of Water on Ultrasonic Devulcanization of Polydimethylsiloxane

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ABSTRACT: The effects of the presence of water on ultrasonic devulcanization of 30 phr silica-filled polydimethylsiloxane (PMDS) were investigated at increasing feed rates and different die gap sizes. The results showed that the initial die entrance pressure without ultrasound for wet rubber was higher than in the case without water and then it decreased monotonously with applying ultrasound. The die pressure for wet rubber decreased significantly even at low ultrasonic amplitude, while that for dry rubber changed little at low amplitude. The power consumption at an amplitude of 10 μm, where devulcanization was most effectively achieved, was lower for wet rubber even though the pressure was lower. The crosslink density and gel fraction after the devulcanization of wet rubber were lower than those of dry rubber, indicating that the presence of water facilitates the devulcanization process under the same devulcanization conditions. The good mechanical properties of recycled silica-filled PDMS were obtained at higher feed rates and at lower ultrasound amplitudes, which are directly related to the economics of a recycling process. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 88: 2630–2638, 2003

Key words: vulcanization; silicones; silicas; rubber

INTRODUCTION

In considering the higher cost of silicone rubber [polydimethylsiloxane (PDMS)] compared to most other rubbers, recycling of waste silicone rubber has been of interest from both commercial and environmental point of views. However, the recycling of silicone rubber is more difficult due to the bond energy of silicon–oxygen atoms in the main chain, which is greater than that of the carbon–carbon bonds in various organic elastomeric materials. For comparison purposes, Table I lists the bond energies of silicone and SBR rubber networks according to refs. 1–3.

To date, there has been a relatively small amount of work related to the recycling of silicone elastomer. Although silicone rubber has a unique chemical structure and unique properties, the methods applied to recycling of silicone rubber were not much different from those for other waste rubbers. Largely, the recycling methods of waste silicone rubber can be classified into three categories: (1) thermal depolymerization in a thermal cracking bed reactor[4] or in a closed vessel,[5,6] (2) chemical treatment by dissolving the waste silicone rubber in amines[7] or in a mixture of 2.5% sulfuric acid/KOH and 75% butyl carbitol in the presence of a catalyst,[8] and (3) grinding waste silicone rubber scraps into very fine particles and then compounding them with virgin silicone rubber.[9,10]

Another most recent and actively studied approach in the the recycling of various waste rubbers is the use of high-power ultrasound. Isayev and coworkers[11–13] investigated the possibility of the recycling of unfilled and silica-filled silicone rubber using ultrasound, where excellent and satisfactory recovery, respectively, of the physical properties of devulcanized unfilled and silica-filled silicone rubber was obtained.

Interestingly, water vapor under high pressure has been occasionally employed to enhance the thermal depolymerization process of PDMS in a closed system at elevated temperature for long operating times.[5,6] Its acting mechanism has not yet been clearly demonstrated. However, taking into account the fact that a water molecule is released from the condensation of the end groups of PDMS during the production of PDMS as shown in Figure 1, an excess amount of water may accelerate a degradation reaction, that is, a reverse condensation reaction, of PDMS at elevated temperature.[14] In the current study, the effects of water in the ultrasonic devulcanization process of silica-filled silicone rubber was investigated with the aims to achieve a higher output of the ultrasonic reactor and to reduce processing cost.

EXPERIMENTAL

Sample preparation

A polymeric network was prepared by crosslinking PDMS, SE 64 made by General Electric Co., with a

TABLE I
Bond Energies Existing in the Silicone Rubber and SBR Network

Bond type/ energy	Bonds					
	Si—O	C—C	Si—C	C—S	S—S	S_x
Bond type	Main chain in PDMS	Crosslink in PDMS (VMQ) main chain in SBR	Crosslink in PDMS	Crosslink in SBR	Crosslink in SBR	Crosslink in SBR
Bond energy (kJ/mol)	451 [1]	345 [1,2]	318 [1,2]	285 [3]	268 [3]	251 [3]

weight-average molecular weight $M_w = 4.14 \times 10^5$ and a number-average molecular weight $M_n = 2.34 \times 10^5$ [measured by gel permeation chromatography (GPC)]. It contained 0.6 mol % vinyl groups. Hi-Sil® 132 (PPG Industries, Pittsburgh, PA), which is a precipitated amorphous-type silica having a surface area of 200 m²/g, was used as a reinforcing filler at a concentration of 30 phr in PDMS. Dicumyl peroxide (DCP), LUPEROX® 500R (Pennwalt Corp., Philadelphia, PA), was used as the curative. Various concentrations of Hi-Sil® 132 were incorporated into PDMS by a Moriyama mixer having a chamber capacity of 3 L. Half the amount of the filler was added to the PDMS, and after 3 min, the rest was added and mixed for 7 min at room temperature. The silica-filled PDMS compounds were then homogenized by a two-roll mill (Dependable Rubber Machinery Co.). DCP, 0.5 phr, was added to the compounds on the two-roll mill at 25°C. After mixing, the compounds were precured by a compression-molding press (Wabash) in 260 × 260 × 12-mm³ slabs at 170°C and then postcured in a ventilated oven at 200°C for 2 h.

Ultrasonic devulcanization

The vulcanized sheets were ground into particles using a Nelmor grinding machine with a 5-mm screen. To investigate the effects of water on the devulcanization process, the ground particles were soaked in water for 5 min and excess water was removed. No swelling of the PDMS particles in water was observed. The approximate water content was measured to be 10%.

These wet silicone particles were then fed into a rubber extruder with an ultrasound coaxial die attachment[15] to achieve devulcanization. The temperature of the extruder barrel was set at 180°C. The screw speed was 20 rpm and both the die and horn cooling water flow rate were set to be 0.09 m³/h. The gap δ between

the flat face of the horn and the die exit surface was either 0.35 or 0.63 mm. The flow rates, Q, were 0.32, 0.63, and 1.26 g/s. A 3000-W ultrasonic power supply, a converter, and a booster were used to provide longitudinal vibrations to the horn at a frequency of 20 kHz. The amplitudes, A, of the ultrasonic wave were 5, 7.5, and 10 µm. The devulcanized silicone rubber exiting from the die was collected for further investigation. The devulcanized rubber was revulcanized with 0.5 phr DCP in slabs of dimensions of 180 × 130 × 3 mm³ at 170°C. The revulcanizates were also postcured under the same conditions as were the virgin compounds.

Characterizations

The cure behaviors of the silicone rubber compounds were investigated using a Monsanto oscillating disc rheometer at 170°C following ASTM D 2084. The cure time was determined based on the time required to achieve 90% of the maximum torque from the cure curve.

Gel fractions of the vulcanized and devulcanized samples were measured by Soxhlet extraction, using benzene as the solvent.[13] The extraction time was set at 24 h. Crosslink densities of the gel were determined by the swelling method. The weights of the swollen samples were measured after removing the surface solvent. Then, the samples were dried in a vacuum oven at 50°C for 24 h and were weighed again. The crosslink density was calculated using the Flory–Rehner equation[16] with the Kraus correction[17] to prevent misleading effects of the filler on the swelling of the reinforced particulate-filled rubber.

The weight- and number-average molecular weights of the sol of PDMS devulcanized under dry and wet conditions were measured by GPC. The apparatus consisted of a Waters 510 GPC, a differential Viscotek 100 viscometer, and a Waters 410 differential
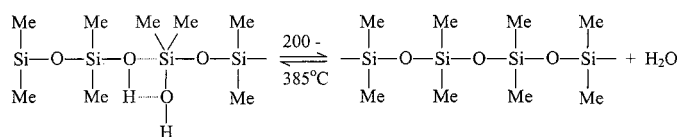

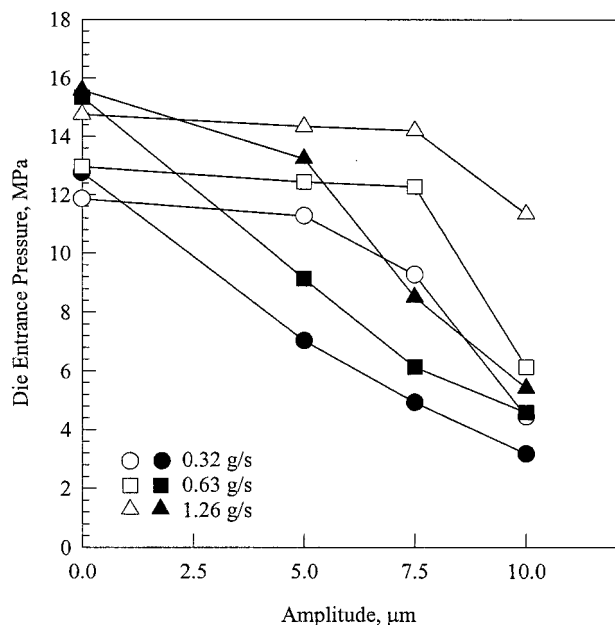**Figure 1** Condensation reaction of the end groups of PDMS.

Figure 2 Die entrance pressure versus ultrasound amplitude for dry (open symbols) and wet (solid symbols) 30 phr precipitated silica-filled PDMS devulcanized at a die gap of 0.63 mm and a barrel temperature of 180°C.

refractometer. Three Waters Styragel high-resolution columns were used. The calculation of the molecular weight was based on a universal calibration performed by using the Aldrich Polystyrene Standard Kit (including 13 standards). The solvent used was THF. The sample was filtered and then injected into the column. The mechanical properties of the virgin vulcanizates and revulcanized samples were measured at a crosshead speed of 50 mm/min by an Instron 5567 tensile test machine with a 500-N load cell following ASTM D 412 (type C) at room temperature.

RESULTS AND DISCUSSION

Effects of feed rate on devulcanization of PDMS

Figure 2 shows the die entrance pressure versus the ultrasound amplitude upon the devulcanization process of 30 phr silica-filled PDMS at various material feed rates of 0.32 (2.5), 0.63 (5), and 1.26 g/s (10 lb/h). The devulcanization was achieved at a gap of 0.63 mm and barrel temperature of 180°C. It is seen that the die entrance pressure at a higher feed rate was higher for both wet and dry rubbers. As the ultrasound amplitude increases, the die entrance pressure for the wet sample continuously decreases from 5 μm for all feed rates, while that for dry rubber changes little at a low ultrasound amplitude and at a high feed rate. The pressure for the wet rubber is always lower compared to that for the dry upon ultrasound exposure. Considering the relationship of the die entrance pressure to the degree of devulcanization, lower pressure at the

same devulcanization condition is caused by reduced viscosity of the material due to more devulcanization. It is noted that the initial pressure without ultrasound for wet rubber was higher than in the case of dry rubber for all the feed rates. This could be due to the vaporization of water contained in the sample at high barrel temperatures. In addition, ultrasound generates a significant amount of thermal energy by bubble cavitation.

The power consumption of the devulcanization of both wet and dry rubber is represented in Figure 3. All the devulcanization conditions were the same as in Figure 2. As expected, the power consumption at a higher feed rate is greater for both cases. The power consumption at 5 and 7.5 μm for wet rubber is higher than in the case of dry rubber. However, the power consumption for wet rubber at 10 μm, where a severe devulcanization reaction takes place, is lower, which implies that less energy is required. In addition, in the case of dry rubber, the power consumption at 10 μm sharply increases while the increase of the power consumption for wet rubber at 10 μm was less.

The crosslink density and gel fraction of the devulcanized rubber obtained at several feed rates are given in Figure 4. More devulcanization is obtained at a lower feed rate for both dry and wet rubber as shown in Figure 4(a,b) by lower values of the crosslink density and gel fraction. The crosslink density and gel fraction for wet rubber are lower than those of dry rubber, indicating that the soaked water facilitates the devulcanization process under the same devulcanization conditions. The decrease of the crosslink density
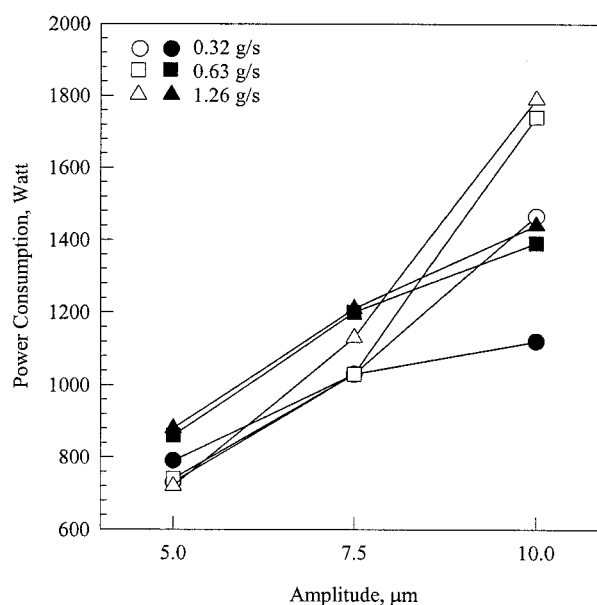

Figure 3 Power consumption versus ultrasound amplitude for dry (open symbols) and wet (solid symbols) 30 phr precipitated silica-filled PDMS devulcanized at a die gap of 0.63 mm and a barrel temperature of 180°C.
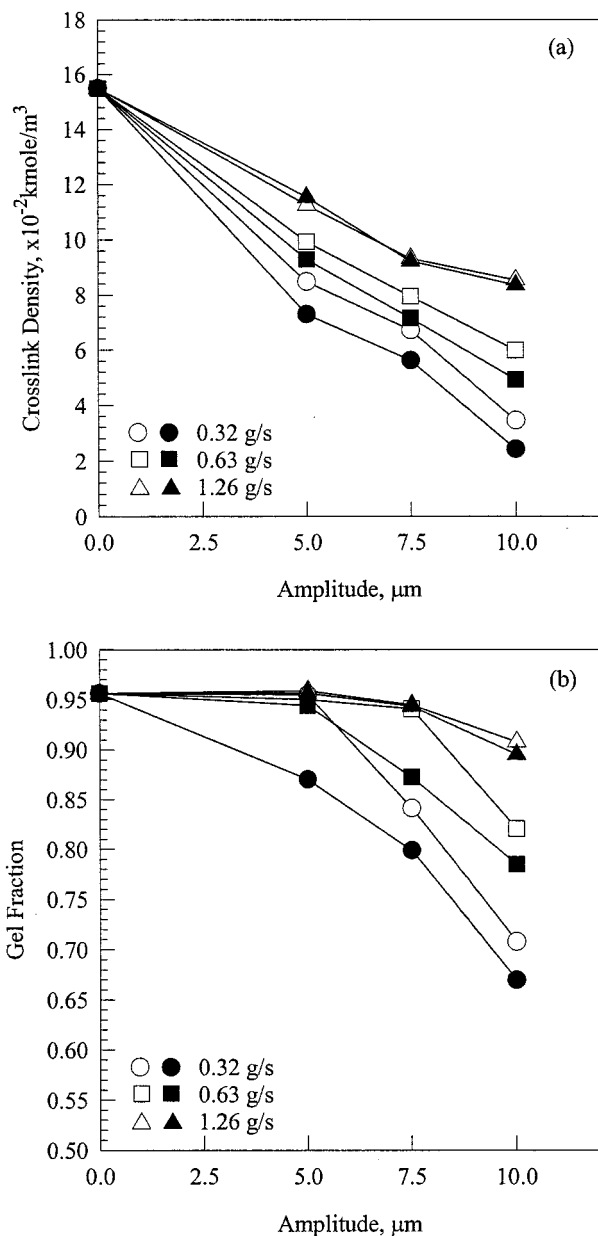
Figure 4 (a) Crosslink density and (b) gel fraction of dry (open symbols) and wet (solid symbols) 30 phr precipitated silica-filled PDMS devulcanized at a die gap of 0.63 mm and a barrel temperature of 180°C.

and gel fraction with an increasing ultrasound amplitude at a lower feed rate is more pronounced for the wet rubber.

Effects of gap size on devulcanization of PDMS

Figure 5 represents the die entrance pressure versus the ultrasonic amplitude of 30 phr silica-filled PDMS by changing gap sizes. The material feed rate was fixed at 0.32 g/s (2.5 lb/h) and was chosen as the best condition in the previous section. The barrel temperature of the extruder was set at 180°C. The die pres-

sure for wet rubber is always lower upon ultrasound treatment than that for dry rubber. As expected, a smaller die gap results in higher pressure for all cases. It is shown that the die entrance pressure for wet rubber monotonously decreases as the ultrasonic amplitude increases. However, there is little decrease in the pressure at low amplitude dry rubber. The difference of the die entrance pressure for wet rubber becomes smaller with an increasing ultrasound level, indicating that water enables devulcanization to be possible at a higher flow rate with a greater gap size.

The power consumption at the same devulcanization condition as in Figure 5 is depicted in Figure 6. The power consumption at a narrower gap size is higher due to the higher die entrance pressure. Similarly as in Figure 3, the increase in power consumption for wet rubber at 10 μm is found to be less than that of dry rubber. At a lower ultrasound amplitude, the die entrance pressure was higher for wet rubber due to the vaporized water, but it becomes lower at 10 μm where an active devulcanization reaction occurs. The difference in die pressures between the wet and dry rubbers at a 0.35-mm die gap becomes greater. In addition, the die pressure with a 0.35-mm die gap at 7.5 μm for wet rubber was lower than that for dry rubber. This may be explained by that the water molecules are already involved in the devulcanization reaction at this smaller die gap.

The effects of gap size upon the change in crosslink density and gel fraction is demonstrated in Figure 7. The devulcanization conditions were the same as in
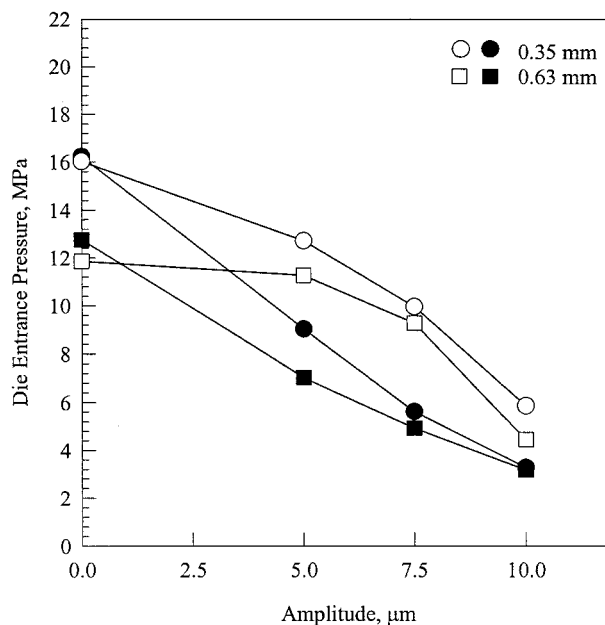

Figure 5 Effect of gap size on die entrance pressure versus ultrasound amplitude for dry (open symbols) and wet (solid symbols) 30 phr precipitated silica-filled PDMS devulcanized at a flow rate of 0.32 g/s and a barrel temperature of 180°C.

Figure 6 Effect of gap size on power consumption versus ultrasound amplitude for dry (open symbols) and wet (solid symbols) 30 phr precipitated silica-filled PDMS devulcanized at a flow rate of 0.32 g/s and a barrel temperature of 180°C.
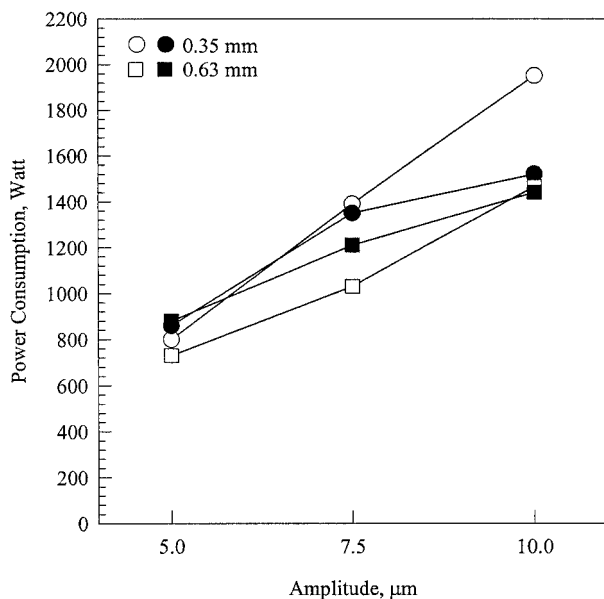
Figures 5 and 6. In comparison with dry rubber, the crosslink density and gel fraction for wet rubber are also lower at all conditions. The values in the crosslink density and gel fraction for dry rubber at 5 μm change slightly while they decrease significantly for wet rubber. The difference in the crosslink density values of wet rubber is smaller than that of dry rubber.

Figure 8 is a normalized gel fraction versus normalized crosslink density plot of 30 phr silica-filled silicone rubber devulcanized at die gaps of 0.35 and 0.63 mm, feed rates of 0.32, 0.63, and 1.26 g/s, and amplitudes of 5, 7.5, and 10 μm. Normalized values are the ratios of the current values of the treated sample to those of the initial values. Lines 1 and 2 represent the cases where only crosslinks and only main chains, respectively, are subjected to rupture.[18] Clearly, the experimental data lie closer to line 2, indicating a substantial breakage of the main chains. This breakage of main chains, as explained in ref. 13, is caused by their reduced mobility due to attachments of the chains to the filler surface. In this figure, rectangular and circular symbols denote the devulcanization of wet and dry rubber, respectively. The experimental data points between the wet and dry rubbers have no noticeable differences in Figure 8. This suggests that the water included in the rubber facilitates the devulcanization reaction of silicone rubber while it has no influence on the structural differences. As mentioned earlier, taking into account that water molecules are released from a condensation of the end groups of PDMS during the production of PDMS (Fig. 1), the

presence of water molecules can cause the reverse reaction, that is, a degradation reaction, of PDMS in the temperature range of 200–385°C.[14] Another possible reason would be the sonolysis reaction of water molecules by ultrasound. It is known from sonoluminescence studies that ultrasonic irradiation of water leads to electronically excited water molecules, hydroxyl and hydrogen radicals in the cavitation of a bubble, as follows[19–23]:

$$H_2O \rightarrow H_2O\cdot \rightarrow HO\cdot + H\cdot \qquad (1)$$
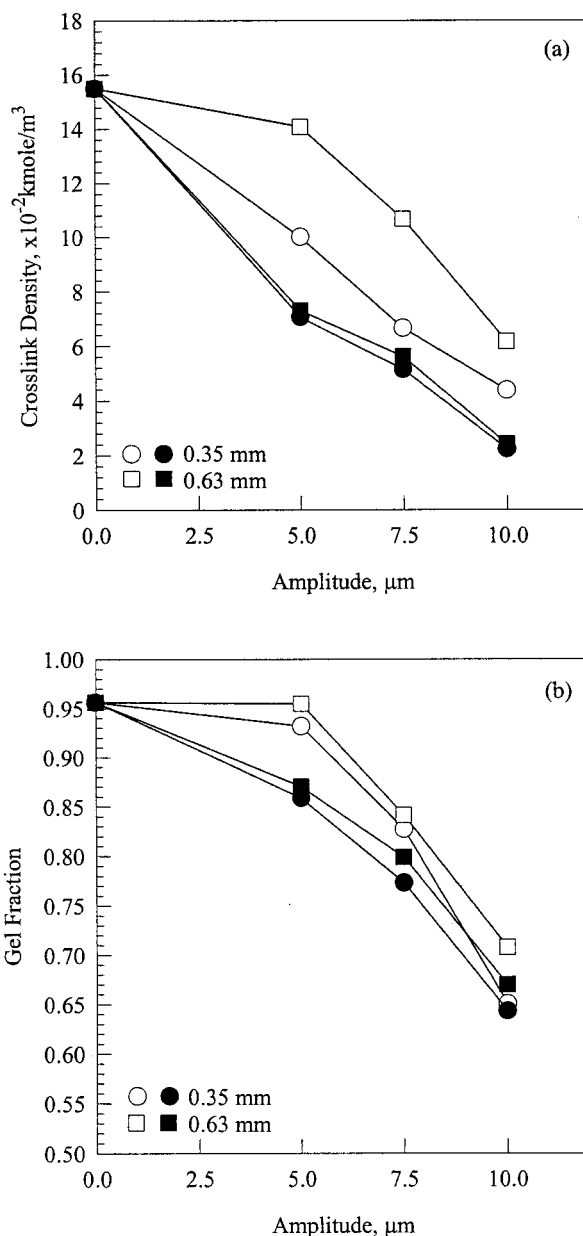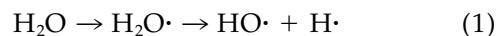

Figure 7 Effect of gap size on (a) crosslink density and (b) gel fraction of dry (open symbols) and wet (solid symbols) 30 phr precipitated silica-filled PDMS devulcanized at a flow rate of 0.32 g/s and a barrel temperature of 180°C.
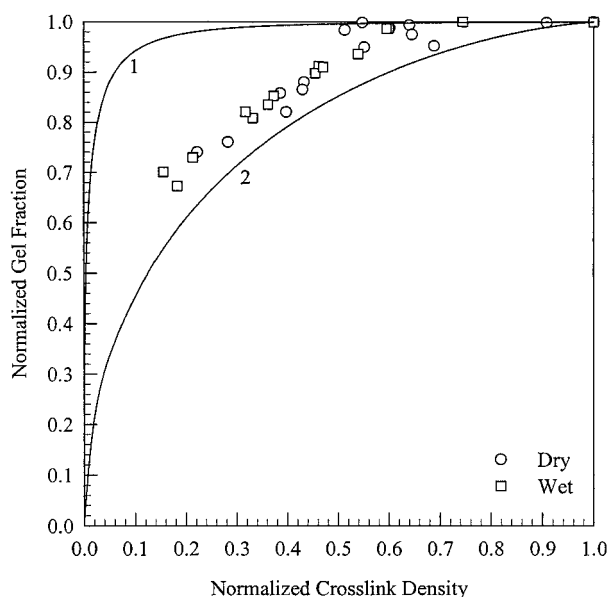
Figure 8 Normalized gel fraction versus normalized crosslink density of dry (circles) and wet (rectangles) 30 phr silica-filled silicone PDMS devulcanized at die gaps of 0.35 and 0.63 mm, feed rates of 0.32, 0.63, and 1.26 g/s, and amplitudes of 5, 7.5, and 10 μm. Lines 1 and 2 represent, respectively, only crosslinks and main chains subjected to rupture.
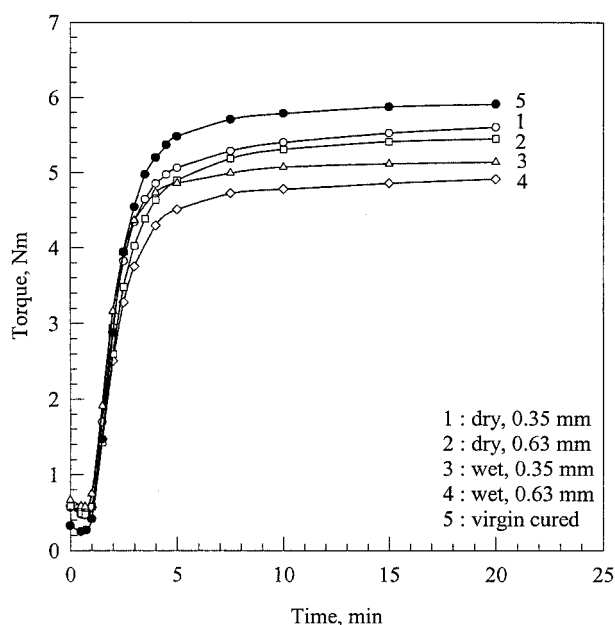

Figure 9 Cure behaviors of 30 phr precipitated silica-filled virgin and 25/75 devulcanized/virgin PDMS blends. PDMS was devulcanized at a feed rate of 0.32 g/s and an ultrasound amplitude of 10 μm.

The thermal degradation of the PDMS main chain is known to occur via an ionic mechanism.[24] Also, the degradation of PDMS takes place via a radical mechanism through homolytic silicon–carbon bond scission of the side methyl group attached to the silicon atom of the main chain.[24] Evidently, the advent of free radicals from sonolysis may participate in a chain-scission reaction during the ultrasonic devulcanization process. In addition to chain scission, the breakage of crosslinks and polymer–filler bonds would lead to the production of sol and the reduction of crosslink density of the remaining gel. In this regard, it is interesting to determine the molecular weights and polydispersity index (PDI) of the sol extracted from the devulcanized dry and wet PDMS. These measured data are given in Table II. The devulcanization for both samples was achieved under the same conditions of a die gap of 0.63 mm, feed rate of 0.63 g/s, and amplitude of 10 μm. It is seen that there are not significant differences in the M_n, M_w, and PDI values between the devulcanized dry and wet samples. The values of M_n and M_w of the extracted sol are about one

order of the magnitude lower than those of the virgin PDMS with a slight change of the PDI value.

The present findings in silica-filled PDMS are contrary to our earlier measurements on devulcanized unfilled PDMS, indicating, respectively, a higher and lower value of M_w and M_n with a significant increase of the PDI value of the extracted sol in comparison with corresponding values of the virgin PDMS.[11] This observation is a clear indication of a severe degradation of polymer chains during ultrasonic devulcanization of silica-filled PDMS. Therefore, in contrast to unfilled PDMS, the presence of a large amount of bound rubber generated by polymer–filler bonds and filler–filler interaction, restricting the mobility of polymer chains in silica-filled PDMS, is the main reason for such a severe molecular degradation. The degradation of the main chains and deactivation of the filler, resulting from the breakage of silica–rubber bonds, cause a deterioration of the ultimate mechanical properties of silica-filled PDMS, as reported below.

Physical properties of devulcanized PDMS

Figure 9 depicts the curing behaviors of 30 phr silica-filled virgin and 25/75 blends of devulcanized/virgin rubber. Devulcanization of both dry and wet rubber was achieved at die gaps of 0.35 and 0.63 mm, at a feed rate of 0.32 g/s and ultrasonic amplitude of 10 μm. DCP, 0.5 phr, was incorporated into the blends of the devulcanized/virgin rubber prior to curing. The amount of DCP was based on the total amount of

TABLE II
Molecular Weights and PDI of the Extracted Sol from Devulcanized Dry and Wet PDMS

State of PDMS	M_n	M_w	PDI
Dry	1.8×10^4	4.8×10^4	2.65
Wet	2.2×10^4	4.9×10^4	2.24

rubber in the blends. The scorch time, the curing time, and the curing kinetics are virtually the same for 100% virgin and 25/75 devulcanized/virgin blends. In the cases of ultrasonically devulcanized sulfur-cured rubbers including SBR[25] and NR,[26–28] the scorch time was shortened and the curing kinetics was accelerated. The recognizable differences in this system are the initial and final torques. In peroxide-cured rubber, no curing agent remains in the rubber after curing since the curing agent is depleted upon curing, which gives the same curing behavior for devulcanized rubber. However, in the sulfur-curing systems, other ingredients such as accelerators and activators stay in the rubber even after curing, leading to a speedup of the vulcanization reaction.[29]

The higher final torque is developed for dry rubber and at a narrower gap size of 0.35 mm. This explains that greater devulcanization is achieved at a smaller gap in both dry and wet rubber, so more sol, which participates in the crosslinking reaction, is produced. In addition, more severe degradation takes places in wet rubber at the same gap size. It is worth mentioning that there is no clear distinction between degradation and devulcanization. However, when rather selective scission of crosslinks is achieved, it is conventionally called devulcanization. Here, the term "devulcanization" is used to define the process in which breakage of crosslinks and partially of main chains occurs, leading to processible rubber with good physical properties. In the case of degradation, the typical assumption is of the breakage of main chains, leading to a deterioration in physical properties.

Figure 10 illustrates the strain–stress curves of virgin-cured and 25/75 devulcanized/virgin blends (same samples as in Fig. 9) after revulcanization. The physical properties of such rubbers are tabulated in Table III. It is seen that the mechanical properties of the vulcanizates made of devulcanized/virgin blends are comparable to those of virgin-cured PDMS. The inferior tensile properties of the blends containing devulcanized wet rubber to those containing the devulcanized dry one may be due to severe breakage of bonds in PDMS upon the ultrasonic process as shown in Figures 4 and 7. Figure 11 shows the results of a comparison of the stress–strain curves for virgin vulcanizate and revulcanizates obtained from devulcanized wet and dry PDMS. Clearly, an inferior performance of revulcanizates made from 100% devulcanized wet rubber compared to that of devulcanized dry rubber is observed. However, one can control the properties of the revulcanizates of PDMS by devulcanizing the wet rubber at a higher feed rate or at a lower ultrasound amplitude, which is not possible in the case of dry rubber. In fact, the highest flow rate achieved in the case of devulcanization of dry PDMS at high amplitude was only 0.32 g/s. This is directly
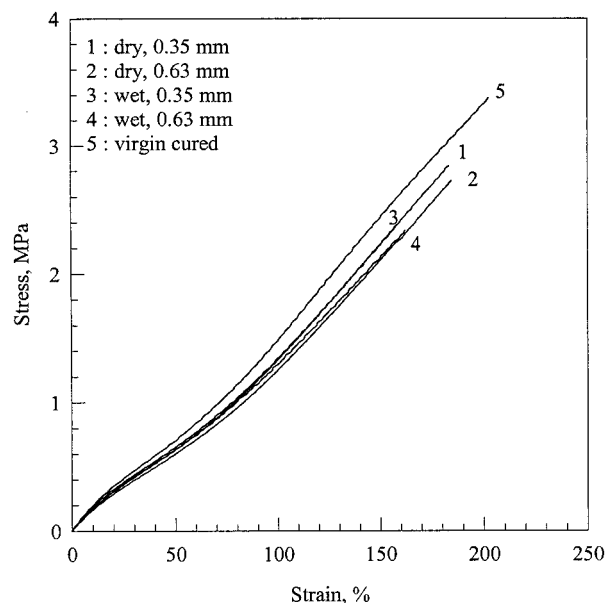

Figure 10 Strain–stress curves of 30 phr precipitated silica-filled virgin and 25/75 devulcanized/virgin PDMS blends. PDMS was devulcanized at a feed rate of 0.32 g/s and an ultrasound amplitude of 10 μm.

related to the economy of the recycling process of PDMS.

The physical properties of 25/75 devulcanized/virgin blends devulcanized at a higher material feed rate and lower ultrasound amplitude for wet rubber are also given in Table III. First, devulcanized rubber was obtained at lower ultrasonic amplitudes (samples 6 and 7 in Table III) at a feed rate of 0.32 g/s and at a die gap of 0.63 mm. Compared to 10 μm (sample 4 in Table III), higher mechanical properties were attained for both 5 and 7.5 μm. Among the three different amplitudes, 5 μm gives the best properties. Second, the material feed rate was increased twofold with an ultrasound amplitude of 7.5 and 10 μm at a die gap of 0.63 mm (samples 8 and 9 in Table III). It is seen that the mechanical properties of the revulcanizates obtained at a higher feed rate are quite similar to the sample devulcanized at lower feed rates (sample 4 in Table III). Therefore, the presence of water expedites the ultrasonic devulcanization of PDMS and the economy of the recycling process of PDMS can be enhanced by water.

CONCLUSIONS

The effects of the presence of water on ultrasonic devulcanization of 30 phr silica-filled PMDS were examined. The vulcanized ground particles were soaked in water prior to feeding them into an extruder and the water content was found to be approximately 10%. It was found that the initial pressure without ultrasound for wet rubber is higher than in the case with-

TABLE III
Mechanical Properties of 30 phr Silica-filled Virgin-cured and 25/75 Devulcanized/Virgin Silicone Rubber

Sample no.	Devulcanization conditions	Modulus at 100%		Elongation at break		Tensile strength	
		Values (MPa)	Recovery (%)	Values (%)	Recovery (%)	Values (MPa)	Recovery (%)
1	Dry, 0.35 mm, 0.32 g/s, 10 μm	1.33	90.5	183.4	90.6	2.84	84.3
2	Dry, 0.63 mm, 0.32 g/s, 10 μm	1.25	85.0	184.7	91.2	2.73	81.0
3	Wet, 0.35 mm, 0.32 g/s, 10 μm	1.29	87.8	162.2	80.1	2.34	69.4
4	Wet, 0.63 mm, 0.32 g/s, 10 μm	1.35	91.8	157.6	77.8	2.36	70.0
5	Virgin cured	1.47	100	202.5	100	3.37	100
6	Wet, 0.63 mm, 0.32 g/s, 5 μm	1.34	91.2	172.1	85.0	2.61	77.5
7	Wet, 0.63 mm, 0.32 g/s, 7.5 μm	1.24	84.4	172.4	85.1	2.45	72.7
8	Wet, 0.63 mm, 0.63 g/s, 7.5 μm	1.33	90.5	165.4	81.7	2.47	73.3
9	Wet, 0.63 mm, 0.63 g/s, 10 μm	1.31	89.1	162.4	80.2	2.39	70.9

out water due to the vaporized water and it decreases significantly with an increasing ultrasonic amplitude, resulting in lower die pressures compared to dry rubber. In addition, the die entrance pressure for wet rubber decreases significantly even at low ultrasonic amplitudes. The phenomena are more pronounced at a lower material feed rate and a narrower die gap clearance. The power consumption at 10 μm, where devulcanization is most effectively achieved, is lower for wet rubber even though the pressure is lower.

The crosslink density and gel fraction after devulcanization of wet rubber are lower than those of dry rubber, indicating that the presence of water facilitates the devulcanization process under the same devulcanization conditions. Based on a plot of the normalized gel fraction versus the normalized crosslink density, no noticeable difference in chain scission between the

dry and wet rubber was observed. This implies that the water included in the rubber only facilitates the devulcanization reaction of silicone rubber but has no influence on the structural differences of devulcanized rubber.

The physical properties of recycled rubber stand considerably good compared with those of virgin-cured PDMS. The inferior tensile properties of the revulcanizates of wet rubber to the dry one may be due to the severe breakage of bonds in PDMS upon ultrasonic treatment at a feed rate of 0.32 g/s and at ultrasound amplitude of 10 μm. The material properties of the revulcanizates of wet rubber are improved by increasing the feed rate or by decreasing the ultrasound amplitude, which is directly related to the economics of a recycling process.
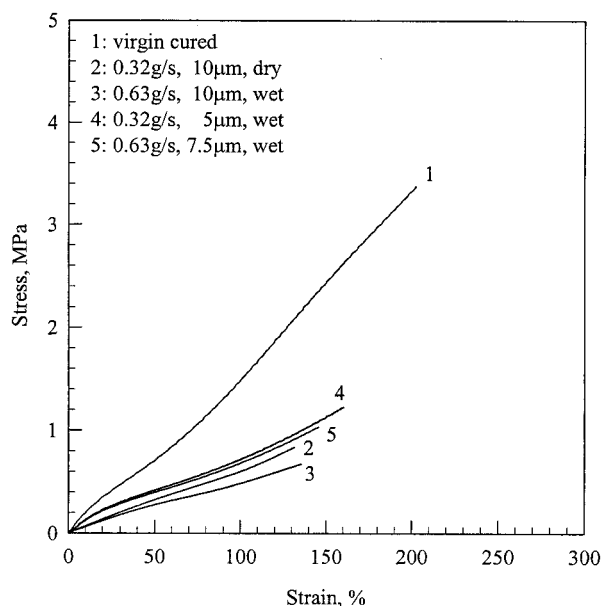
Figure 11 Strain–stress curves of vulcanizates of virgin and 100% dry and wet devulcanized PDMS obtained at a gap of 0.63 mm.

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