
Morphology and Physico-Mechanical Properties of Closed Cell Microcellular EPDM Rubber Vulcanizates: Effect of Conductive Carbon Black and Blowing Agent

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ABSTRACT

The morphology of the composite based on a new controlled long chain branching grade, oil extended EPDM (Keltan 7341A) and highly conductive carbon black (vulcanXC72) filled compound has been studied from SEM photomicrographs with variation of blowing agent and conductive carbon black filler loading. The average cell size, maximum cell size and cell density varies with variation of blowing agent and filler loading. Physical properties like relative density, hardness, tensile strength, modulus, tear strength decreases with blowing agent concentration. Enclosed gas pressure in a closed cell increases relative modulus at higher strain. Elongation at break decreases with blowing agent loading at higher filler loading. At lower filler loading it increases both with filler and blowing agent loading. The elastic nature of closed cells reduces the hysteresis loss compared to solid compounds. The stress relaxation behavior is independent of blowing agent loading.

INTRODUCTION

Electrically conductive cellular and microcellular ethylene propylene diene terpolymer (EPDM) finds increasing special application in electronic sectors as a packaging and semi conductive polymeric material due to its high filler loading, low cost and good aging properties. The systematic study of the properties of microcellular and cellular EPDM rubber particularly filled with highly conductive carbon black with blowing agent variation has not received much attention till today.

In case of microcellular EPDM foams, very few articles have appeared in literature⁽¹⁻²⁾. The morphology and physical properties of closed cell microcellular ethylene-octene copolymer have also been reported⁽³⁾. Previous

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investigation on elastomeric foams⁽⁴⁻⁸⁾ were primarily concerned with development of compounding techniques for various elastomers and studies of their physical properties like tensile strength, compression deflection, water absorption, and ozone resistance etc. The morphology of elastomeric foam⁽⁹⁾ and characterization of microcellular foam⁽¹⁰⁾ have also been reported. Mechanical properties and modeling of cellular materials have been published by several authors⁽¹¹⁻¹³⁾. Theoretical modeling of the elastomeric latex foam has been developed by some authors for both open and closed cell foam to predict the failure properties⁽¹⁴⁻¹⁷⁾.

The mechanism of nucleation and bubble growth in elastomers⁽¹⁸⁾ thermoplastics⁽¹⁹⁾ thermoplastic elastomers⁽²⁰⁾ and microcellular thermoplastics⁽²¹⁻²³⁾ using a blowing agent and a super saturation method is also subjected to earlier investigation. Physical properties of elastomeric foam such as hysteresis, damping, cell size and thermal insulation properties have also been reported in literature⁽²⁴⁻²⁸⁾.

The preparation of electrically conductive polymer composites⁽²⁹⁻³¹⁾ and rubbers⁽³²⁾ by filling conductive fillers have already been reported by several researchers. The relation between electrical and mechanical properties of conductive polymer composites⁽³³⁾ have also been subjected of recent research.

In the present study we have investigated several processing and compounding techniques of a new controlled long chain branching (CLCB) grade oil extended EPDM rubber to achieve closed cell conductive microcellular EPDM filled with excess conductive black i.e. VulcanXC72 with uniform cell distribution. KELTAN 7341A, has been selected as this is a new CLCB foam grade rubber for high performance and consistency. In addition to the improved product consistency CLCB polymers shows a reduced sensitivity to carbon black scorch⁽³⁴⁾. In comparison to EPDM based conventional technology K7341A shows a better overall performance at higher compound loadings. We selected Vulcan XC 72 as filler since it is the excess conductive carbon black. We also selected a peroxide curing system instead of a sulphur curing system since sulphurous vapors emitted from EPDM rubber can damage electronic instruments⁽³⁵⁾. The chemistry of peroxide vulcanization⁽³⁶⁾ explaining that peroxide contain an oxygen-oxygen bond that breaks homolytically when heat is applied to yield free radicals. It also explains the effect of oils on the peroxide vulcanization of a basic EPDM compound which shows that paraffinic oil provides the best results since it does not consume free radicals preferentially over the polymer. So in the present work we used paraffinic oil rather than naphthanic or aromatic oil. Here we have studied morphology and physical properties of micro cellular KELTAN 7341A (EPDM) rubber with special

reference to variation of blowing agent and vulcan XC72 (Excess conductive carbon black) filler loading.

EXPERIMENTAL

Materials

The EPDM rubber [Keltan 7341A, (a new CLCB grade rubber) ENB wt%-7.5, oil (phr)-20, Mooney viscosity 53 (at 150 °C) manufactured by DSM Elastomers, Singapore] was used. Vulcan XC72 (excess conductive carbon black) was used as Filler, supplied by Cabot Corporation Ltd, USA. The curative used as the Dicumyl Peroxide (DCP) with a purity of 98%, manufactured by Aldrich Chemical Company, USA. Azo di carbonamide (ADC), the blowing agent used was of ADC-21, manufactured by High Polymer Lab, India and Paraffinic oil used was Sunpar oil, supplied by Sun Oil Company Pvt. Ltd, Kolkata.

Physical Characteristics of Vulcan XC72 carbon black

Nitrogen Surface Area (m ² /g)	180
DBP Absorption number (ml/100g)	178
Particle Diameter (nm)	29
Electron Microscopy Surface Area (m ² /g)	86
CTAB Surface Area (m ² /g)	86
Pore Area (m ² /g)	94
Electrical Resistivity (Wcm)	0.015

Compounding and Sample Preparation

The rubber was compounded with the ingredients according to the formulations of the mixes (Table 1). Compounding was done in a laboratory size two roll mixing mill at room temperature according to ASTM D3182. Cure and blowing characteristics of the compounds were determined in a Monsanto Rheometer, R-100. The vulcanizates were press-molded at 150°C to obtain a closed cell microcellular sheet. As the press is closed, the compounds completely fill the mold, expelling the air and sealing the cavity. The typical compound flows readily in the molds, coalesces, and eliminates trapped air blisters. As the stock temperature increases the cure starts and the decomposition of the blowing agent begins. Carbon dioxide is released and cell starts to form. As the

Table 1 Formulations of unfilled and Vulcan XC72 filled vulcanizates

	G ₀	G ₂	G ₄	G ₆	EB ₁	EB ₂	EB ₃	EB ₄	EB ₅	EB ₆	EB ₇	EB ₈	EB ₉	EB ₁₀	EB ₁₁	EB ₁₂
Keltan 7341A	120	120	120	120	120	120	120	120	120	120	120	120	120	120	120	120
Vulcan XC72	0	0	0	0	20	20	20	20	40	40	40	40	60	60	60	60
Paraffinic oil	0	0	0	0	2	2	2	2	4	4	4	4	6	6	6	6
ADC 21	0	2	4	6	0	2	4	6	0	2	4	6	0	2	4	6

Each mix contains ZnO-5 phr; Stearic Acid-1.5 phr and Dicumyl peroxide (98% pure) - 1 phr

decomposition progresses, an exotherm develops and pressure builds up. These factors accelerate the curing rate. The press is opened before the cure has been completed. A very small closed cell is obtained after expansion. The precured sheet is then post cured at 100°C (for 1 hr) to complete the curing.

Test Procedures

The specific gravity of the samples was measured according to ASTM D 3574-77. The hardness of the micro cellular sheets was measured using a Shore-A Durometer as per ASTM D 676-59T. The stress-strain properties like tensile strength, modulus, elongation at break and tear strength were measured on a computerized Zwick Universal Testing Machine according to ASTM D 3574-77. Measurement of hysteresis is also carried out in the same machine according to ASTMD 3574-77. All these tests were performed at room temperature (25±2°C). At least five specimens per sample were tested for each property and mean values are reported.

SEM Studies

SEM studies were carried out for understanding the cell structure using JEOL JSM 5800 SCANING ELECTRON MICROSCOPE. Razor cut surfaces from micro cellular sheets surfaces of the tensile specimen were used as samples for SEM studies. The surfaces were gold coated before being studied.

RESULTS AND DISCUSSIONS

Rheometric Characteristics

The cure characteristics with effect of blowing agent and excess conductive carbon black filler, obtained from Monsanto rheographs are shown in Figures 1 and 2 and are summarized in Table 2. The maximum rheometric torque decreases with increase in blowing agent loadings. The minimum rheometric torque also sometime decreases very slightly which is negligible. This decrease

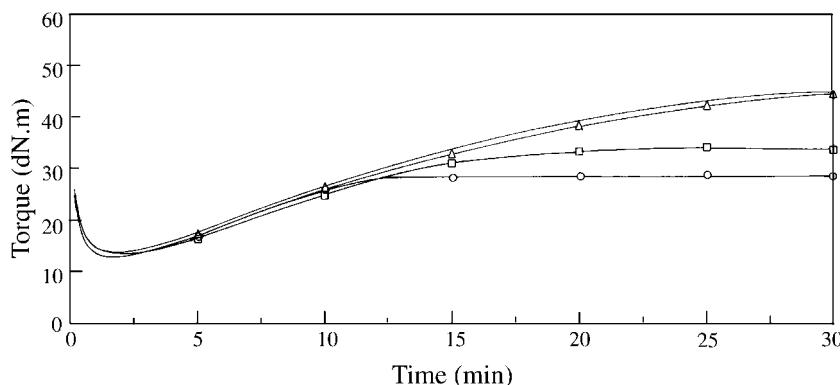


Figure 1 Representative rheographs of unfilled compounds: Effect of blowing agent loading (—) G₀, (—▲—) G₂, (—□—) G₄, (—○—) G₆

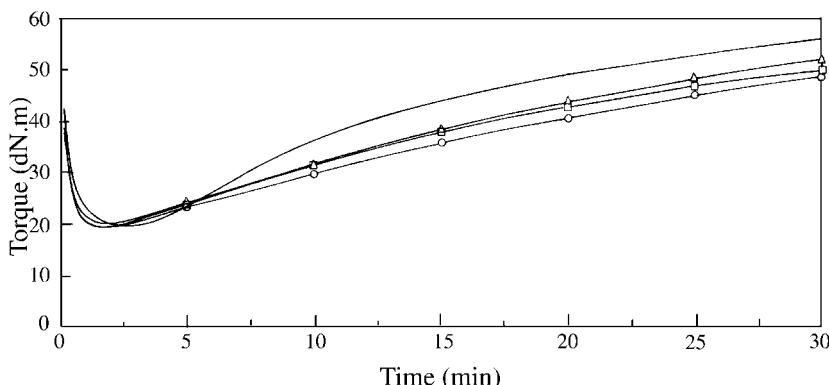


Figure 2 Representative rheographs of 60 phr Vulcan XC 72 filled compounds: Effect of blowing agent loading (—) EB₉, (—▲—) EB₁₀, (—□—) EB₁₁, (—○—) EB₁₂

Table 2 Rheometric characteristics of unfilled and Vulcan XC 72 filled vulcanizates

Mix No.	Mooney Viscosity (ML ₁₊₄ 100°C)	Minimum Rheometric Torque, (dN-m)	Maximum Rheometric Torque, (dN-m)	Rheometric scorch time at 150°C, min.	Optimum cure time at 150°C, min.
G ₀	65	14.0	45	1.25	6.0
G ₂	63	13.0	44	1.30	5.0
G ₄	61	13.5	33	1.35	4.5
G ₆	58	13.0	28	1.35	4.0
EB ₁	69	16.0	50	1.3	6.0
EB ₂	65	15.0	46	1.35	5.5
EB ₃	56	14.5	43	1.4	5.0
EB ₄	50	14.0	41	1.45	5.0
EB ₅	83	17.0	48	1.35	6.5
EB ₆	82	16.0	46	1.35	6.0
EB ₇	80	15.5	44	1.35	6.0
EB ₈	77	15.0	43	1.35	6.0
EB ₉	108	20.0	56	1.35	6.5
EB ₁₀	107	20.0	52	1.4	6.5
EB ₁₁	106	19.5	50	1.45	6.5
EB ₁₂	105	19.0	48	1.55	6.5

in maximum torque is due to decomposition of blowing agent, which form microbubbles. These microbubbles reduce melt viscosity and hence maximum torque decreases. With incorporation of conductive carbon black filler both minimum and maximum rheometric torque increases. However there is a significant change in the Mooney viscosity values. Rheometric scorch time (t_2) increases with increasing blowing agent concentration and with filler loading it increases very slightly due to low scorching nature of rubber with carbon black. The optimum cure time decreases with increase in blowing agent in case of unfilled compound and at lower filler loading due to oil extended nature of rubber, which gives low viscosity. But at higher filler loading (above

40 phr) it does not vary with filler loading. However in general with increasing filler loading optimum cure time increases. From rheographs, actual cure characteristics may not be obtained but resultant effect of curing and blowing can be obtained.

Morphology of Razor Cut Surfaces

SEM photomicrographs of razor cut surfaces of various unfilled and filled microcellular rubber vulcanizates are shown in Figure 3. These photomicrographs are analyzed in terms of average cell size or diameter, maximum cell size and cell density (number of cells per unit volume of microcellular rubber). The number of cell increases as the blowing agent loadings increase. Average cell size decreases with increase in blowing agent loading from 2 to 6 phr. Photomicrographs shows that with filler loading the average cell size increases. The microbubbles formed by decomposition of the blowing agent defused and collapsed with each other due to decrease of the cure rate with increase in filler loading. Thus with increase in filler loading average cell size as well as maximum cell size increase (Table 3). The number of cells of microcellular rubber vulcanizates at maximum expansion is calculated using the following relation⁽⁹⁾:

$$N = \left\{ \left\{ 6/(\Pi d^3) \right\} \left\{ (\rho_s/\rho_f) - 1 \right\} \right\}$$

Where N is the number of cells per unit volume of microcellular rubber, d is the average cell diameter and ρ_s and ρ_f are the density of the solid and microcellular rubber vulcanizates respectively. The number of cells per unit volume (cm^{-3}) are calculated and shown in Figure 4. The number of cells per unit volume increases with increase in blowing agent loading. The cell density decreases with increase in filler loading. The carbon black (Vulcan XC 72) filler, being basic in nature, doesn't act as a nucleating agent. This also reduces the acidity of field compounds compared to the unfilled compounds, which may result in a decrease in cell density.

Physical Properties

The percent of volume expansion of the Keltan 7341A vulcanizates are shown in Figure 5. It is observed that the percent of volume expansion increases with increase in loading of blowing agent and conductive carbon black filler. Increase in percent of volume expansion is due to more decomposition of blowing agent and less diffusion of decomposed gas during curing. Physical properties like relative density, hardness is also given in Table 3. The relative density (ρ_f/ρ_s) decreases with increase in blowing agent loading. The decrease

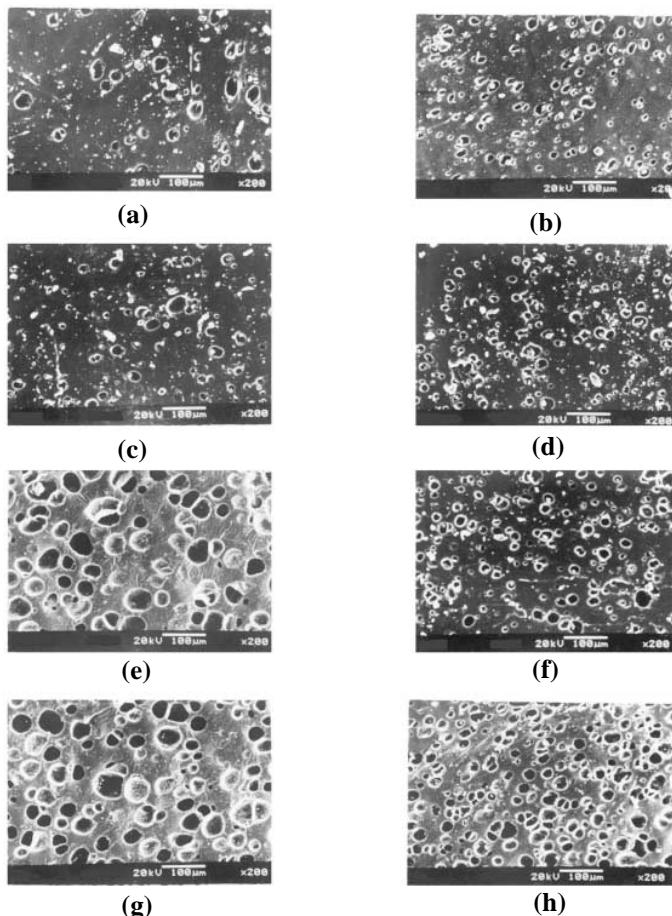


Figure 3 SEM photomicrographs of razor cut surfaces of microcellular Keltan 7341A (oil extended EPDM) vulcanizates. (a) G₄; (b) G₆; (c) EB₃; (d) EB₄; (e) EB₇; (f) EB₈; (g) EB₁₁; (h) EB₁₂

in relative density is much more pronounced in filled compounds. With increase in filler loading, gas permeability decreases and hence there is less possibility of the loss of gas by diffusion. As a result more gas remains in the rubber matrix. Thus in filled compounds, the decrease in relative density is more for the same blowing agent loading. The hardness of the closed cell microcellular rubber decreases with increase in loading. As the enclosed gas in the closed cell has little elastic property, hardness decreases with decrease in relative density.

Table 3 Physical properties of unfilled and Vulcan XC72 filled vulcanizates

Mix No.	Relative density	Hardness (shore A)	Average cell size (mm)	Maximum cell size (mm)	Cell density (cells/cm ³) x10 ¹³
G ₀	1.0	30	—	—	—
G ₂	0.92	25	30	55	.61
G ₄	0.88	20	27	49	1.32
G ₆	0.85	17	23	40	2.76
EB ₁	1.0	38	—	—	—
EB ₂	0.89	35	35	65	.55
EB ₃	0.84	33	32	60	1.11
EB ₄	0.78	30	28	52	2.45
EB ₅	1.0	43	—	—	—
EB ₆	0.85	40	41	75	.49
EB ₇	0.78	38	39	70	.91
EB ₈	0.67	36	36	55	2.01
EB ₉	1.0	45	—	—	—
EB ₁₀	0.8	44	48	90	.43
EB ₁₁	0.71	42	46	80	.78
EB ₁₂	0.60	40	42	60	1.7

Tensile strength, elongations at break modulus and tear strength are given in Table 4. Tensile strength, tear strength and modulus values are decreased with increase in blowing agent loading. For filled samples tensile strength, tear strength and modulus increases up to 40 phr filler loading and with higher filler loading tensile strength and tear strength decreases but modulus increases. At 40 phr filler loading Keltan 7341A giving highest value of tensile strength which means that at this loading polymer-filler interaction is maximum where as at higher filler loading filler-filler interaction exceeds. Tensile strength and tear strength increases with strain in distinct steps. At small strain closed cell microcellular rubber shows the linear elastic modulus. This is caused by the cell edge bending, face stretching and enclosed gas pressure⁽¹⁴⁾. Non-linear elastic behavior of closed cell microcellular rubber is observed at higher strain.

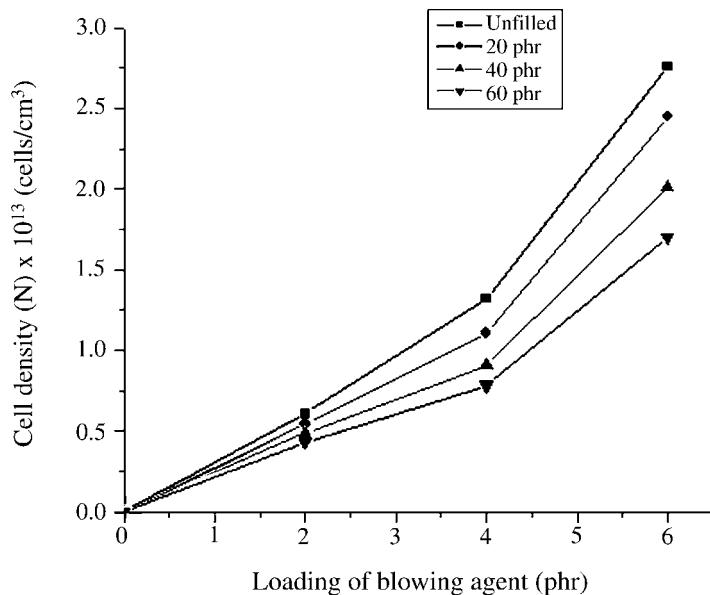


Figure 4 Cell density (N) of microcellular Keltan 7341A (Oil extended EPDM) vulcanizates: Effect of blowing agent and filler loading

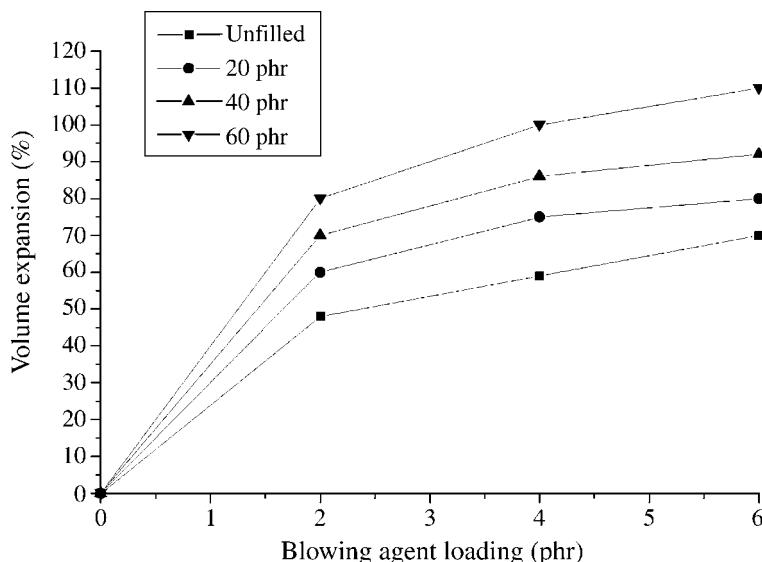


Figure 5 Variation of volume expansion (%) with blowing agent loading

Table 4 Physical properties of unfilled and Vulcan XC72 filled Microcellular Vulcanizates

Mix no.	Tensile strength (Mpa)	Elongation at break (%)	Modulus (100%) MPa	Modulus (200%) MPa	Modulus (300%) MPa	Tear strength (N/mm)
G ₀	2.22	1480	0.64	0.75	0.82	12.48
G ₂	1.21	1787	0.35	0.51	0.60	10.37
G ₄	1.01	1878	0.30	0.48	0.54	8.32
G ₆	0.96	1923	0.22	0.32	0.39	7.26
EB ₁	6.78	2043	0.66	0.74	0.84	20.51
EB ₂	6.0	2589	0.64	0.73	0.83	19.26
EB ₃	5.41	3006	0.60	0.70	0.81	16.98
EB ₄	3.77	3161	0.55	0.67	0.70	14.33
EB ₅	10.61	1748	0.94	1.12	1.44	34.93
EB ₆	8.0	2483	0.84	1.03	1.36	33.03
EB ₇	6.0	2693	0.73	0.89	1.22	28.33
EB ₈	4.64	2735	0.62	0.79	1.08	24.27
EB ₉	6.39	1488	1.08	1.26	1.62	25.37
EB ₁₀	4.9	2351	0.92	1.11	1.49	24.93
EB ₁₁	3.97	2209	0.76	0.98	1.35	22.7
EB ₁₂	3.3	1964	0.7	0.88	1.25	21.91

The enclosed gas within the cells is compressed as the cell is stretched. Thus membrane stresses appear in the cell faces. The axial stretching of the cell walls is also responsible for increase in stress. At sufficiently large extension the cell walls become stretched along the tensile axis and further extension will cause the elongation of cell walls themselves. This will result in a step wise of stress-strain. In case of elongation at break it increases with filler loading and blowing agent loading at lower filler loading (20 phr). It may be due to oil extended nature of rubber, which causes low viscosity at lower filler loading so it extended up to maximum extent. At lower filler loading crosslinking density decreases which may also cause increase in elongation at break. Whereas at higher filler loading crosslinking density and viscosity both

increases which causes decrease in elongation at break. As mentioned earlier at higher filler loading filler-filler interaction also plays a great role. At same filler loading microcellular rubber vulcanizates are having more elongation at break than solid rubber.

Tear strength of the closed cell conductive carbon black filled microcellular rubbers are given in Table 4. It is seen that tear strength is found to decrease with increase in blowing agent loading. With increase in blowing agent loading cell sizes decreases and cell density increases which causes decrease in tear strength. Above all tear strength increases up to 40 phr, but at 60 phr like tensile strength, tear strength also decreases. It may be due to increase in crosslinking density at higher filler loading. And it may be concluded that around 40 phr of Vulcan XC72 is optimum filler loading for Keltan 7341A.

Figure 6 shows the relative modulus (σ_f/σ_s) and the relative tensile strength (E_f/E_s) at 40 phr Vulcan XC72 loaded closed cell microcellular vulcanizates plotted against relative density (ρ_f/ρ_s). The relative modulus, 100, 200 and 300% and relative tensile strength decreases linearly with decrease in relative density. Similar trend is also obtained with other filler loading. The decrease in relative

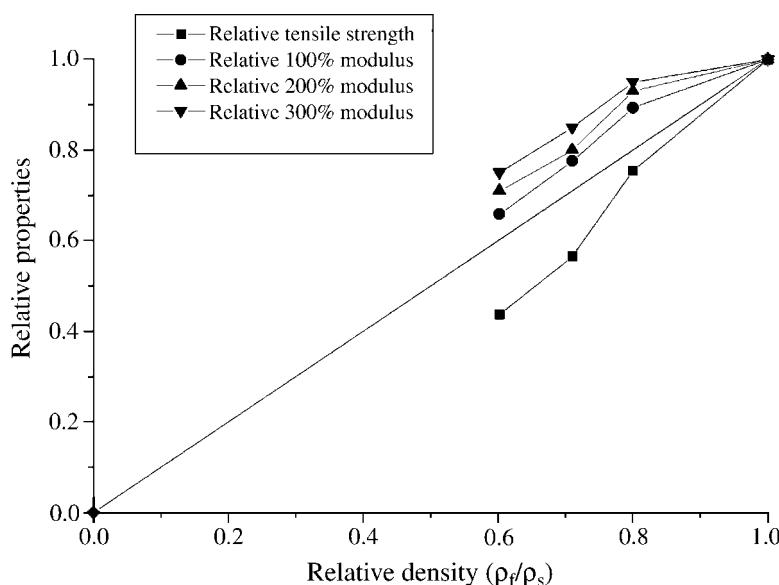


Figure 6 Effect of relative density (ρ_f/ρ_s) on relative properties of 40 phr vulcan XC 72 filled microcellular vulcanizates

tensile strength is sharper than the decrease in the relative modulus. The maximum flaw size i.e., flaws, affect the tensile strength but not the modulus. Therefore relative tensile strength behaves differently than the relative modulus. Here relative tensile strength gives negative deviation where as relative modulus gives positive deviation. It is also observed that the 300% relative modulus shows higher value than 100 and 200% relative modulus. According to additive rule, if the modulus depends only on relative density then it will follow the line joining (0,0) and (1,1) points. So this increase in relative modulus may be due to the increase in enclosed gas pressure inside the cells⁽¹⁾.

The Hysteresis Loss values of different vulcanizates at 100% elongation are given in Table 5. Figures 7 and 8 shows the hysteresis loss plots of unfilled and

Table 5 Result of hysteresis studies of unfilled and Vulcan XC72 filled microcellular vulcanizates

Mix no.	1 st cycle	2 nd cycle
G ₀	0.021	0.11
G ₂	0.016	0.008
G ₄	0.013	0.006
G ₆	0.010	0.005
EB ₁	0.029	0.016
EB ₂	0.027	0.013
EB ₃	0.025	0.011
EB ₄	0.021	0.009
EB ₅	0.049	0.029
EB ₆	0.047	0.026
EB ₇	0.045	0.019
EB ₈	0.041	0.016
EB ₉	0.071	0.034
EB ₁₀	0.069	0.033
EB ₁₁	0.067	0.032
EB ₁₂	0.064	0.031
<i>Hysteresis loss in J/m² at 100% elongation</i>		

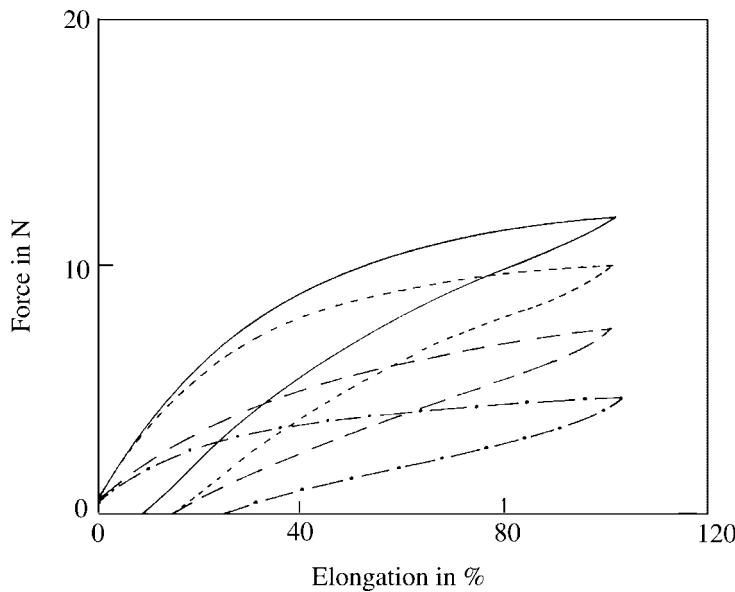


Figure 7 Hysteresis loss plots of closed-cell microcellular unfilled vulcanizates: (—) G₀; (- - -) G₂; (---) G₄; (- · - ·) G₆

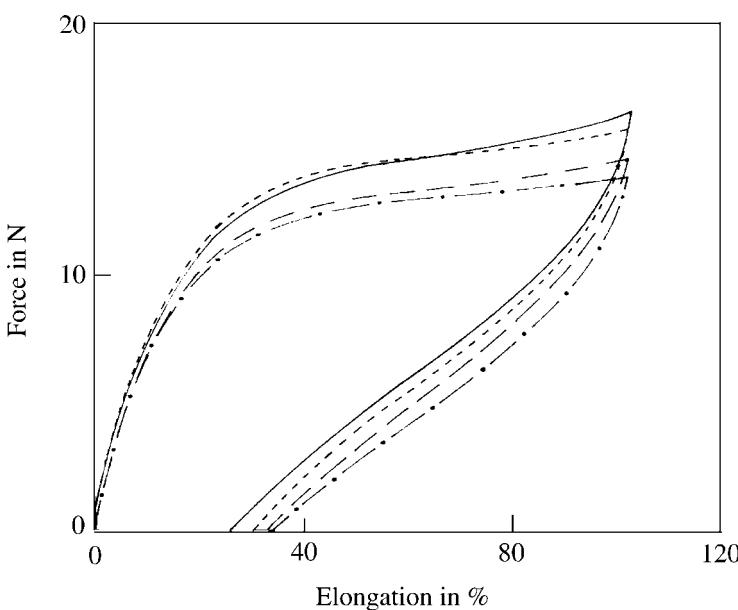


Figure 8 Hysteresis loss plots of 40 phr Vulcan XC 72 filled closed cell microcellular vulcanizates: (—) G₀; (- - -) G₂; (---) G₄; (- · - ·) G₆

40 phr filled vulcanizates. The result exhibits that with increase in blowing agent loading the hysteresis loss decreases for all cycles of measurement. The incorporation of Vulcan XC72 filler causes an increase in hysteresis loss. Solid vulcanizates (both unfilled and filled) exhibit higher hysteresis loss than that of closed cell microcellular vulcanizates. The low hysteresis loss of microcellular vulcanizates can be attributed to the low energy absorption characteristics of the closed cells. With increase in blowing agent loading hysteresis loss decreases since the closed cell in the rubber matrix can act as elastic bodies and is incapable of dissipating energy.

The stress relaxation behavior of closed cell microcellular vulcanizates is determined by stretching the samples at constant strain level of 100%. Figures 9 and 10 shows the decay of stress with time of unfilled and 60 phr Vulcan XC72 filled vulcanizates respectively. The nature of decay is almost similar for closed cell microcellular rubber. The rate of decay is more or less same in case of solid and microcellular Keltan 7341A. Thus the stress relaxation behavior is independent of blowing agent.

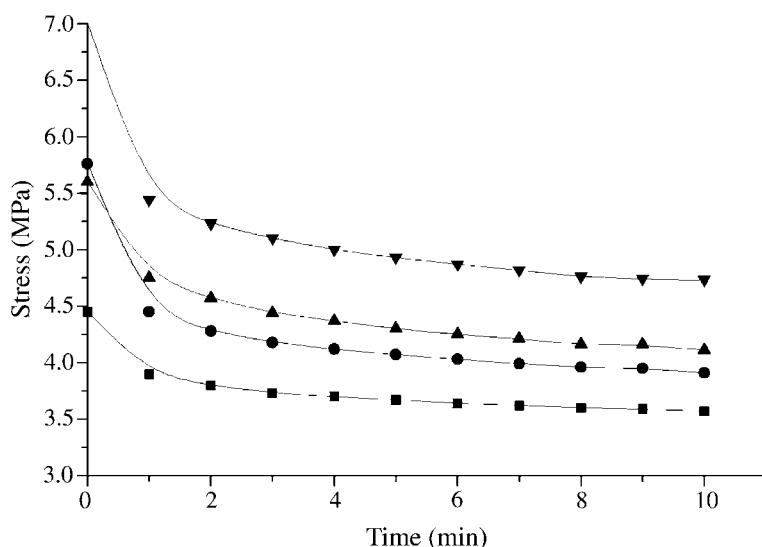


Figure 9 Stress relaxation behavior of closed cell microcellular unfilled vulcanizates (—▼—)G₀; (—▲—) G₂; (—●—) G₄; (—■—) G₆

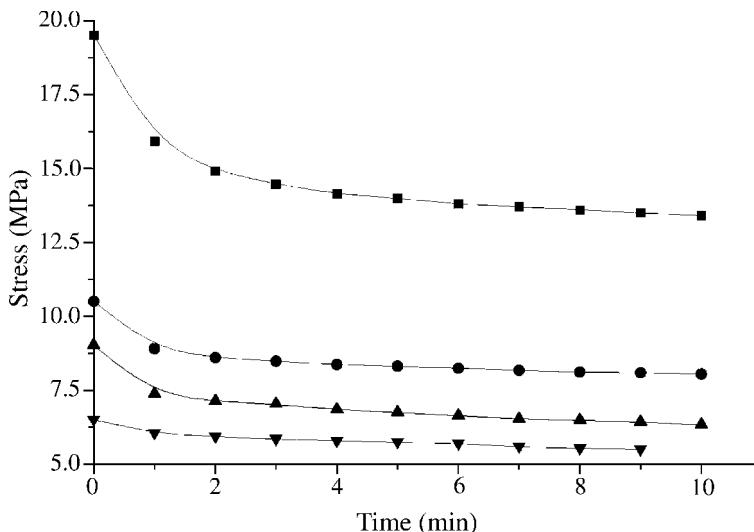


Figure 10 Stress relaxation behavior of closed cell microcellular 60 phr Vulcan XC 72 filled vulcanizates (—■—) EB₉; (—●—) EB₁₀; (—▲—) EB₁₁; (—▼—) EB₁₂

CONCLUSIONS

- i. In case of microcellular Keltan 7341A (oil extended EPDM) rubber vulcanizates the maximum rheometric torque decreases with increase in blowing agent concentration.
- ii. Average and maximum cell size increase with incorporation of Vulcan XC72 filler. This is due to alkaline surface of the carbon black.
- iii. Cell density and percent volume of expansion increases with increasing blowing agent loading as well as Vulcan XC72 filler loading.
- iv. The relative density decreases with increase in blowing agent loading.
- v. Tensile strength and tear strength increases with filler loading up to 40 phr then it decreases but modulus increases at higher filler loading. With blowing agent loading tensile strength, tear strength and modulus are found to decrease for both unfilled and filled compounds.
- vi. Elongation at break for both unfilled and at lower filler loading increases due to low viscosity, oil extended nature of rubber and low crosslinking density.

But at higher filler loading i.e. above 20 phr it decreases with blowing agent loading. Increase in elongation at break may also be due molecular slippage, which does not occur at higher filler loading due to increase in viscosity.

- vii. Enclosed gas pressure in the closed cell increases the relative modulus value in case of higher filler loading, whereas relative tensile strength decreases sharply and does not follow the additive rule.
- viii. The stress relaxation behavior is independent of blowing agent loading, i.e., density of closed cell microcellular rubber.
- ix. The hysteresis loss decreases with increase in blowing agent concentration due to the elastic behavior of the closed cell.
- x. It is concluded that 40 phr Vulcan XC72 can be taken as a optimum filler loading, which gives better physical and mechanical properties.

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