

Rupture of Rubber. VII. Effect of Rate of Extension in Tensile Tests

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INTRODUCTION

There are few published measurements on the effect of rate of extension in tensile rupture tests on rubber vulcanizates. The most extensive data on a single vulcanizate are those of Dogadkin and Sandomirskii¹ and of Smith.² These measurements were made on GR-S gum vulcanizates and covered a similar range of rates of extension, from about 0.02 to 20%/sec. Ring specimens were used in both cases, and tensile strength and breaking extension were measured over a wide temperature range. Villars³ has made tensile strength and breaking extension measurements on double dumbbell specimens of several gum and filled vulcanizates extended at various rates in the range 10,000 to 100,000%/sec. Kainradl and Handler⁴ have reported tensile strength measurements for several filled vulcanizates, obtained with dumbbell specimens extended at four different rates of extension covering a range from about 1 to 100,000%/sec. All these results indicate that tensile strength and breaking extension can vary appreciably with the rate of extension of the specimen. Complete load-extension curves are not given in any of these papers.

In the present paper an autographic method is described for obtaining the load-extension curves of ring specimens extended at various rates from about 0.1 to 2000%/sec. Results showing the effect of the rate of extension on the tensile strength and breaking extension and on the load-extension curve are given for GR-S vulcanizates. The data were obtained primarily for the comparisons of tear and tensile rupture measurements given in a subsequent paper (Part VIII).⁵

EXPERIMENTAL

Method of Measurement

The main features of the tensometer which was used are shown schematically in Figure 1. The ring specimen was looped, as shown, over two small

rollers, one roller being mounted on an auxiliary crosshead attached to the main crosshead of the tensometer, the other roller and its mounting being coupled to a cantilever spring which measured the force on the ring. The tensometer drive consisted of an endless chain mounted on a pair of sprocket wheels immediately below the crossheads and driven via a multispeed gear box by a variable-speed motor. The chain was driven continuously at the chosen speed, and the main crosshead was set in motion by depressing a pin in the crosshead so as to engage with the links of the chain. Provision was made for the pin to move out of engagement with the chain at the end of the crosshead traverse. A slotted coupling between the main and auxiliary crossheads ensured that the auxiliary crosshead was not set in motion until the main crosshead was moving at uniform speed. The deflection of the cantilever spring was magnified by an optical lever (not shown), and the deflection of the light spot was recorded on a drum camera geared to the chain drive, the curve traced out on the drum camera film giving, therefore, the force on the ring as a function of the distance travelled by the crosshead.

The cantilever spring system was sufficiently stiff for the displacement of the roller coupled to the spring to be negligible in comparison with the extended length of the specimen. The response time of the spring system was about 2 msec. The rollers were 6 mm. in diameter, machined from reinforced resin material, and rotated on steel pins. The reproducibility of load-extension measurements indicated that the rollers adequately equalized tensions in the ring specimens. The ring specimens were immersed during extension in a water or alcohol bath maintained at the test temperature to $\pm 1^\circ\text{C}$. Crosshead speeds from about 6×10^{-3} to about 60 cm./sec. could be obtained. The speeds were calculated from the motor speed as measured by a tachometer.

Ring specimens were cut out from a sheet of

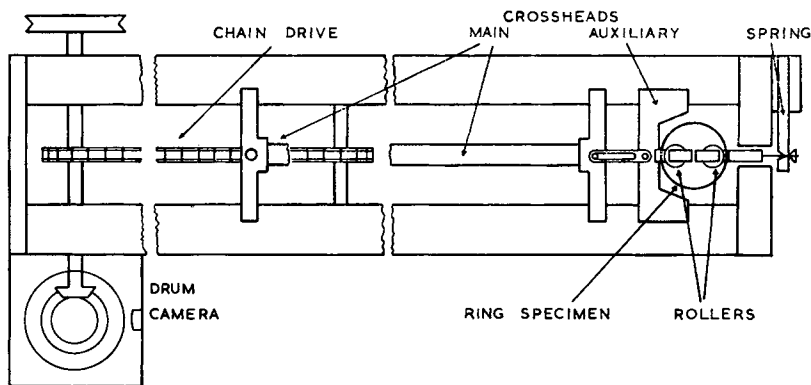


Fig. 1. Schematic diagram of tensometer.

vulcanizate by means of double-bladed cutters. Rings of 2 and 3 cm. internal diameter were used, the larger rings being employed for vulcanizates of relatively low breaking extension. The thickness of the rings, i.e., the difference between the external and internal radii, was about 0.7 mm. and the width was 1.5–3 mm., depending on the thickness of sheet from which the rings were cut. The mean cross-sectional area of the ring was derived from the weight and internal diameter and the density of the vulcanizate.

Extensions were calculated from the increase in length of the ring as given by the force-distance record, taking as the initial length of the ring the mean circumferential length. It was observed that a portion of the ring in the region of contact with the rollers was not fully extended, and it was estimated that this would cause the extensions calculated as described above to be about 5% low. This was the main source of error in the measurement of the extension of the ring. Loads/unit undeformed area were calculated on the basis of the mean undeformed cross-sectional area.

The number of rings extended at each rate of extension varied from three to six, depending on the scatter in the values for breaking load and extension. Mean values for the tensile strength (the breaking load/unit undeformed area) and breaking extension at each rate of extension were obtained from the measurements on the individual rings. The tensile strengths obtained with the ring specimens were found to be equal to or, more usually, somewhat higher than tensile strengths obtained with die-cut dumbbell specimens for approximately equal rates of extension.

Vulcanizates

The vulcanizates were prepared from GR-S (Polysar S) polymerized at 50°C. and containing

23% of styrene. The compounding recipes and cures are given in Table I. Vulcanizate A was a gum compound, and vulcanizates B and C were filled compounds containing, respectively, High Abrasion Furnace (HAF) and Semireinforcing Furnace (SRF) carbon black.

TABLE I

Vulcanizate	Compounding recipe, (parts by weight)			Cure
	A	B	C	
Polysar S	100	100	100	50 min., 145°C.
HAF black	—	50	—	
SRF black	—	—	50	
Sulfur	1.75	1.75	1.75	
Zinc oxide	5	5	5	
Stearic acid	2	2	2	
Santocure	1	1	1	
Antioxidant	1	1	1	
Dutrex R	—	6	6	

RESULTS

Tensile Strengths and Breaking Extensions

Tensile strength measurements for the gum vulcanizate A at 25 and 90°C. are shown plotted against the rate of extension in Figure 2; breaking extension measurements are shown in Figure 3. The results at 25°C. represent mean values for three specimens and those at 90°C. represent mean values for four to six specimens. With this vulcanizate the tensile strength and breaking extension increase uniformly with the rate of extension.

Results for the filled vulcanizate B at 25°C. are given in Figure 4, these being mean values for three specimens. The tensile strength appears to pass through a maximum as the rate of extension is

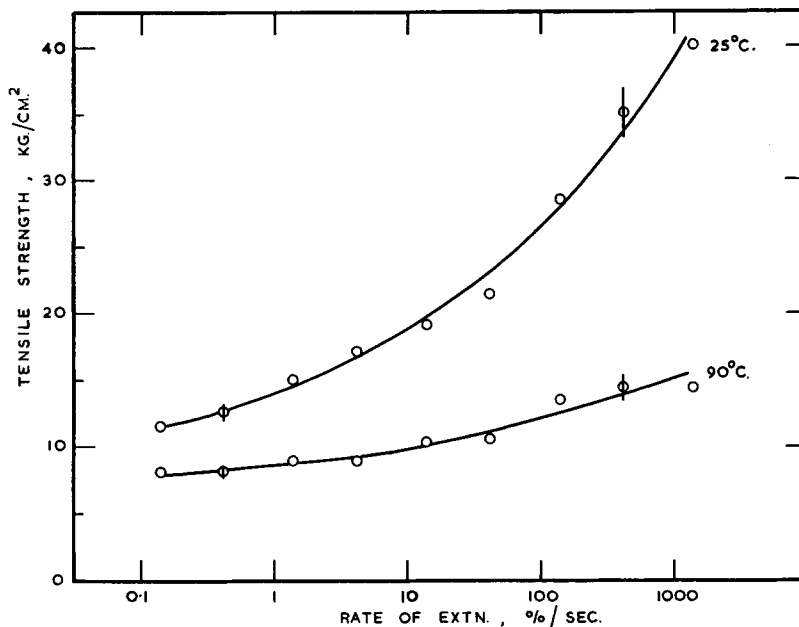


Fig. 2. Mean values of tensile strength vs. rate of extension for the gum vulcanizate A at 25 and 90°C. Vertical bars indicate standard deviation.

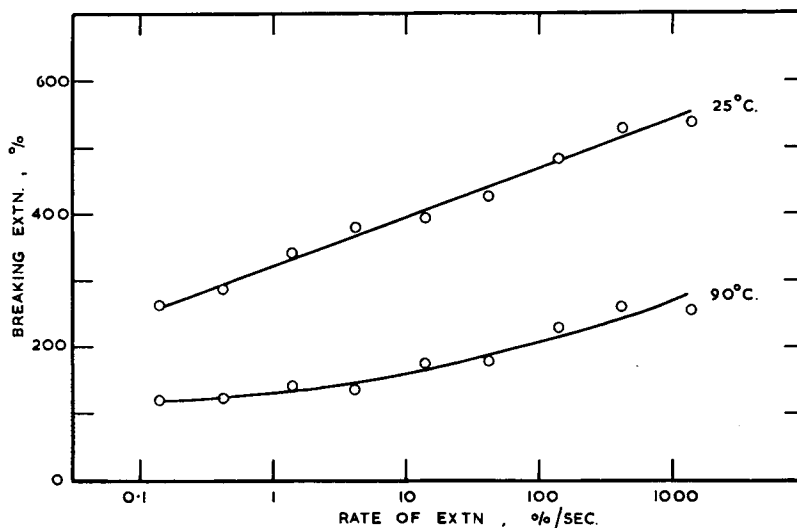


Fig. 3. Mean values of breaking extension vs. rate of extension for the gum vulcanizate A at 25 and 90°C.

increased, but the breaking extension shows very little variation with the rate of extension.

Results for the filled vulcanizate C at 0 and 25°C. are shown in Figures 5 and 6, these being mean values for four to six specimens. A pronounced maximum in the tensile strength is observed at 0°C., and there are indications of a maximum also at 25°C.; the maximum at 25°C. occurs at a higher rate of extension than that at 0°C. The

breaking extensions show rather less variation with the rate of extension.

Load-Extension Curves

Load-extension curves for various rates of extension are shown in Figures 7-11. Apart from the variation in the breaking point, the effect of the rate of extension on the load-extension curves for the gum vulcanizate A is small. The

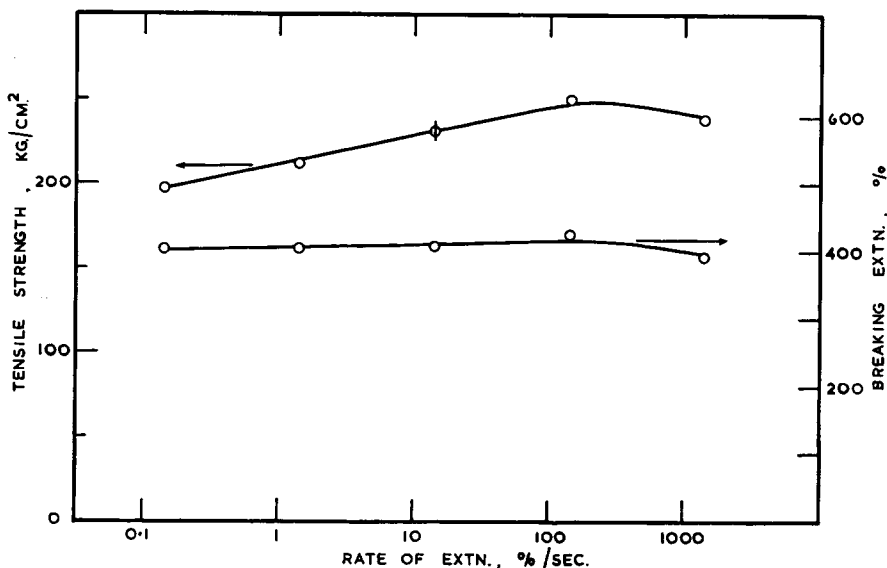


Fig. 4. Mean values of (top) tensile strength and (bottom) breaking extension vs. rate of extension for the filled vulcanizate B at 25°C. Vertical bar indicates standard deviation.

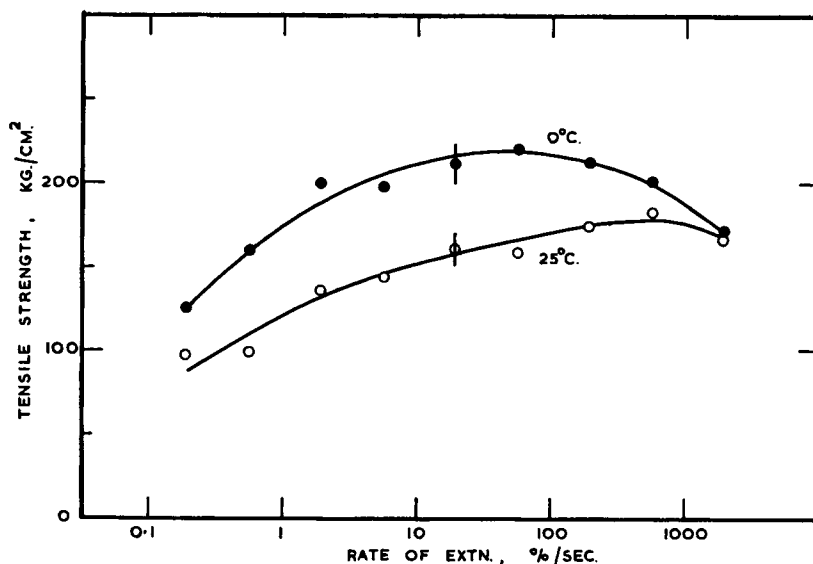


Fig. 5. Mean values of tensile strength vs. rate of extension for the filled vulcanizate C at 0 and 25°C. Vertical bars indicate standard deviation.

effect of the rate of extension is more pronounced in the filled vulcanizates, particularly in the vulcanizate C. The initial slope of the load-extension curves for the filled vulcanizates increases progressively with the rate of extension, but the slope at high extensions near the breaking point passes through a maximum as the rate of extension is increased.

DISCUSSION

The experimental results for the GR-S gum vulcanizate A, showing that the tensile strength

and breaking extension increase with the rate of extension, are in accord with earlier findings.^{1,2} Smith² found that tensile strengths and breaking extensions measured at different temperatures and rates of extension could be superposed according to the transformation scheme employed by Williams, Landel, and Ferry⁶ for small-strain viscoelasticity data, and this is true also for the present results.

The high tensile strengths attained by the filled vulcanizates in comparison with the gum vulcanizate are evidence of the reinforcing action of the

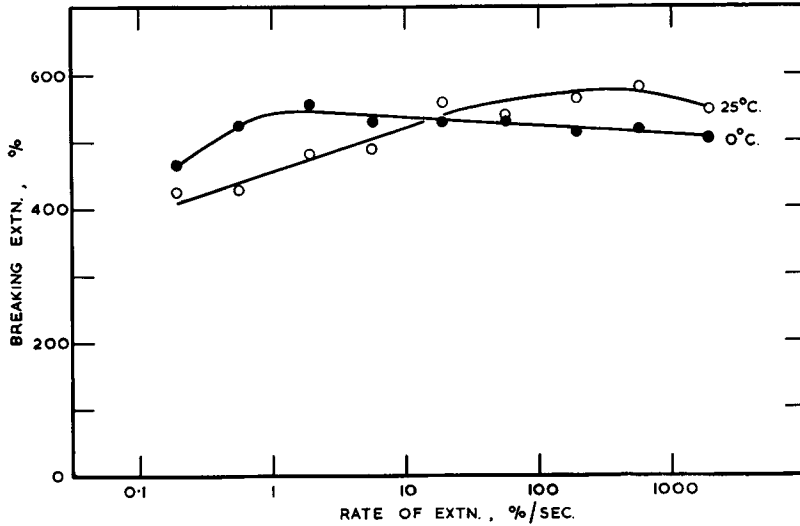


Fig. 6. Mean values of breaking extension vs. rate of extension for the filled vulcanizate C at 0 and 25°C.

filler. The occurrence of a maximum in the tensile strength as the rate of extension is increased indicates that part, at least, of this reinforcing action develops during extension and requires a finite time for development so that it does not occur at high rates of extension. A similar explanation has been given to account for the tear behavior of filled vulcanizates.⁷ The results for the vulcanizate C, showing that the maximum in the tensile strength shifts to higher rates of extension as the temperature is increased, suggest that the time required for the development of the reinforcing action decreases as the temperature is increased. Tensile strength data for filled vulcan-

izates do not appear to transform according to the scheme of Williams, Landel, and Ferry.

A possible alternative explanation of the observed decrease in the tensile strength of the filled vulcanizates at high rates of extension is that it is caused by the heating-up of the specimen during extension at high rates. Previous observations^{3,4} suggest, however, that this explanation is unlikely. Kainradl and Handler⁴ found that the

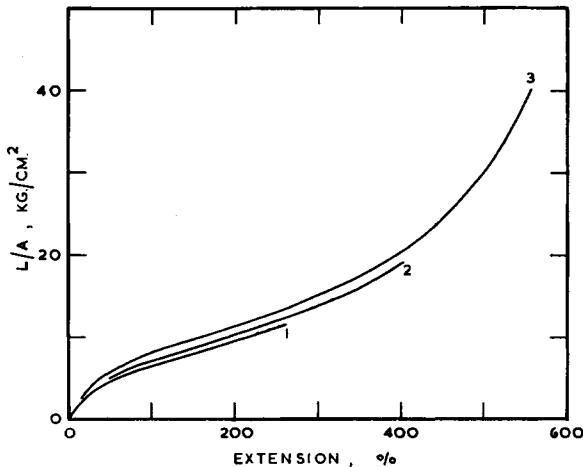


Fig. 7. Curves of load/unit undeformed area (L/A) vs. extension for the gum vulcanizate A at 25°C. at rates of extension of (1) 0.14, (2), 14, and (3) 1400%/sec.

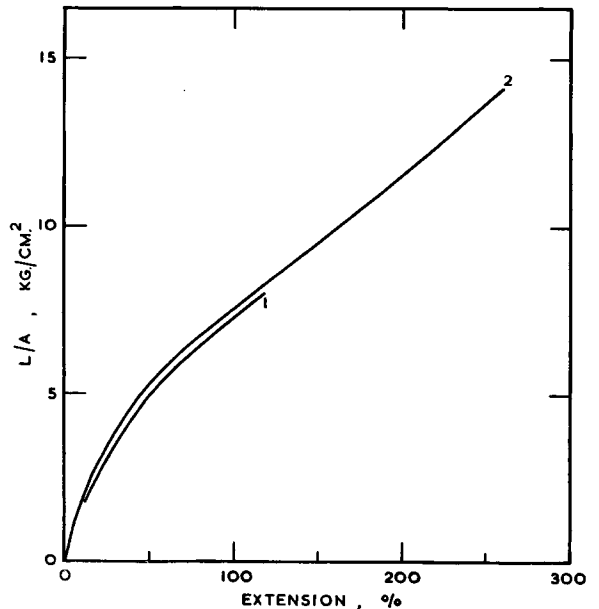


Fig. 8. Curves of load/unit undeformed area (L/A) vs. extension for the gum-vulcanizate A at 90°C. at rates of extension of (1) 0.14 and (2) 1400%/sec.

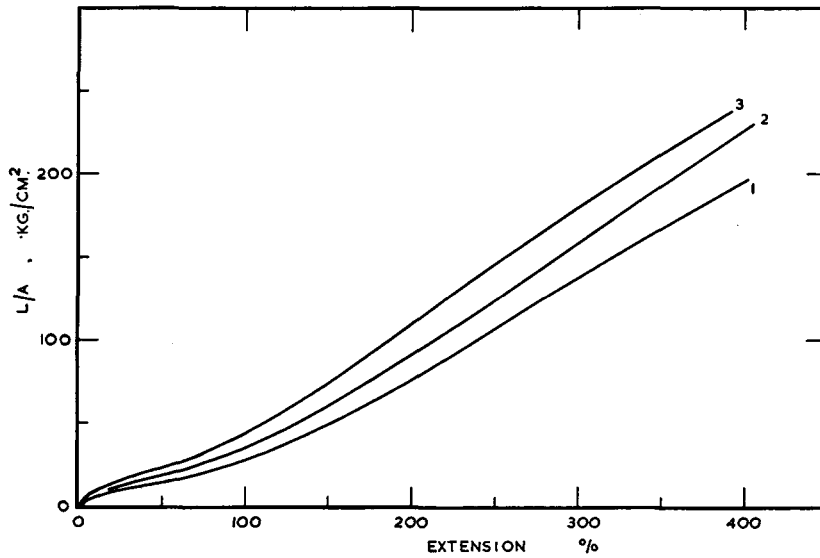


Fig. 9. Curves of load/unit undeformed area (L/A) vs. extension for the filled vulcanizate B at 25°C. at rates of extension of (1) 0.14, (2) 14, and (3) 1400%/sec.

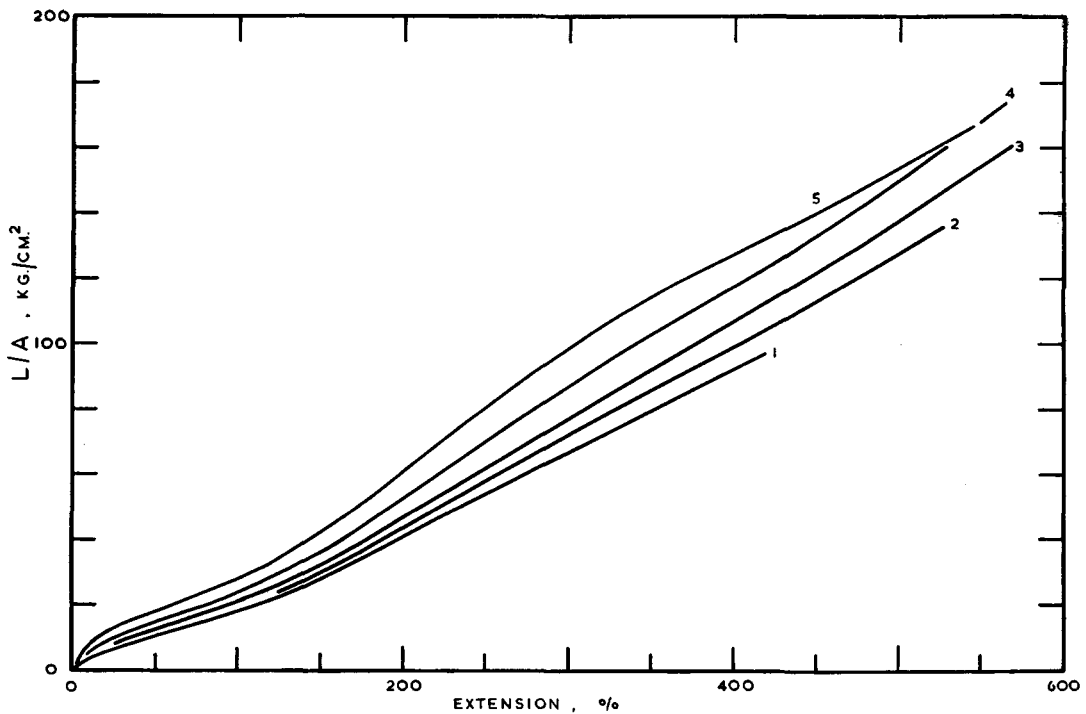


Fig. 10. Curves of load/unit undeformed area (L/A) vs. extension for the filled vulcanizate C at 25°C. at rates of extension of (1) 0.19, (2) 1.9, (3) 19, (4) 190, and (5) 1900%/sec.

tensile strength of a butadiene/styrene vulcanizate containing ISAF black increased again with the rate of extension for rates of extension above 10,000%/sec. Villars³ also found that, for rates of extension above 10,000%/sec., the tensile strength

of a GR-S carbon black-filled vulcanizate increased with the rate of extension.

The effect of the rate of extension on the load-extension curves for the gum vulcanizate is that to be expected from the viscoelastic properties of the

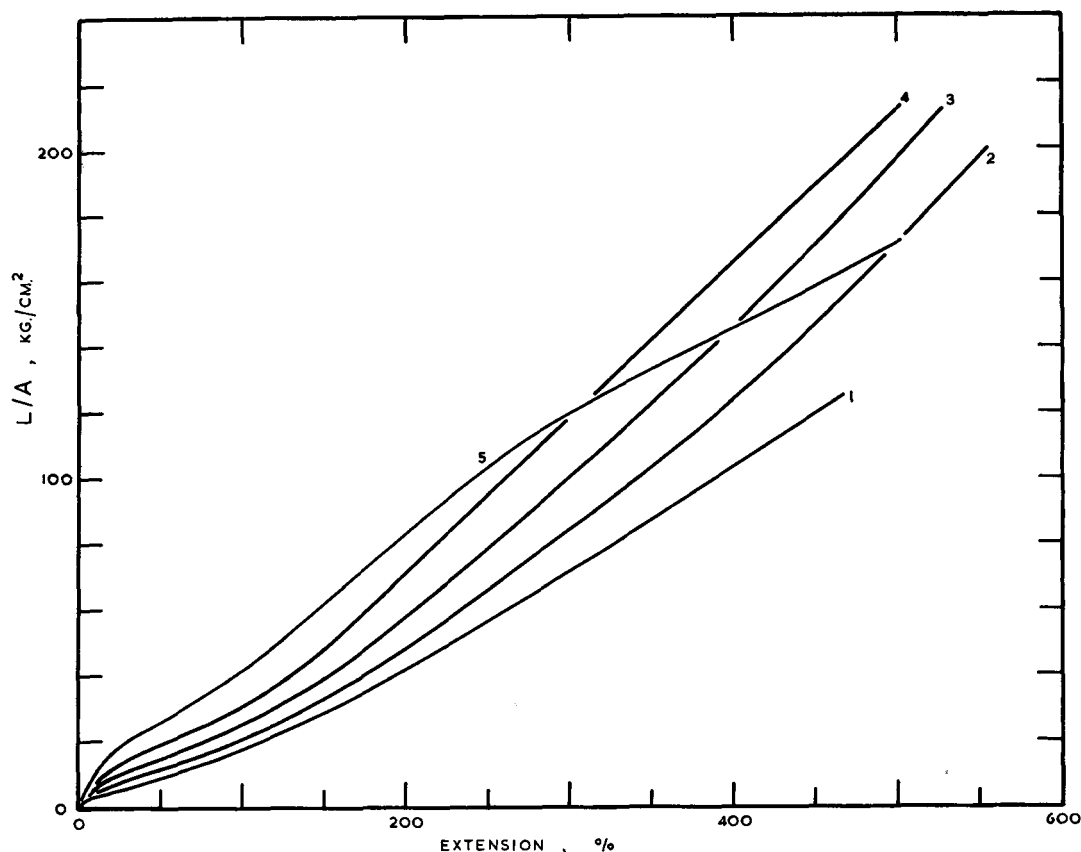


Fig. 11. Curves of load/unit undeformed area (L/A) vs. extension for the filled vulcanizate C at 0°C . at rates of extension of (1) 0.19, (2) 1.9, (3) 19, (4) 190, and (5) 1900%/sec.

material and this applies also, at low extensions, to the load-extension curves for the filled vulcanizates. The changes in the shape of the curves for the filled vulcanizates at higher extensions suggest that the development of the reinforcing action of the filler during extension is accompanied by changes in stress-strain behavior.

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Synopsis

An autographic method is described for obtaining load-extension curves of ring specimens extended at various rates from about 0.1 to 2000% per second. Results showing the effect of the rate of extension on the tensile strength and breaking extension and on the load-extension curve are given for a GR-S gum vulcanizate and for two GR-S vulcanizates containing carbon black. The tensile strength of the gum vulcanizate, at the temperatures at which the tests were carried out, increases uniformly with the rate of extension, but the tensile strength of the filled vulcanizates passes through a maximum. The effect of the rate of extension on the load-extension curve of the gum vulcanizate is that to be expected from the viscoelastic properties of the material; the effect in the case of the filled vulcanizates is more complex. These results are briefly discussed.

Résumé

Une méthode autographe est décrite, pour obtenir des courbes charge-extension d'échantillons annulaires, soumis à extension à différentes vitesses, variant de 0,1 jusqu'à 2000%/sec. Les résultats, montrant l'effet de la vitesse d'extension sur la résistance à la tension et l'extension limite et sur la courbe poids-extension, sont donnés pour une

gomme GR-S vulcanisée et pour deux gommes GR-S vulcanisées contenant du noir actif. La résistance à la tension de la gomme vulcanisée, aux températures auxquelles les expériences ont été réalisées, augmente uniformément avec la vitesse d'extension; mais la résistance à la tension des gommes vulcanisées contenant du noir actif, passe par un maximum. L'effet de la vitesse d'extension sur la courbe poids-extension, dans le cas de la gomme vulcanisée, est bien celui que l'on pourrait attendre à partir des propriétés visco-élastiques de cette substance; cet effet est plus complexe dans le cas des gommes vulcanisées contenant du noir actif. Ces résultats sont discutés brièvement.

Zusammenfassung

Es wird eine autographische Methode zur Bestimmung der Last-Dehnungskurven von ringförmigen Proben beschrieben,

die mit Geschwindigkeiten von etwa 0,1 bis 2000%/sek gedehnt werden. Ergebnisse, die den Einfluss der Dehnungsgeschwindigkeit auf die Zugfestigkeit und Bruchdehnung sowie auf die Last-Dehnungskurve erkennen lassen, werden für ein GR-S-Gummivulkanisat und für zwei russgefüllte GR-S-Vulkanisate angegeben. Die Zugfestigkeit des Gummivulkanisats nimmt bei den Prüfungstemperaturen einheitlich mit der Dehnungsgeschwindigkeit zu, die Zugfestigkeit der gefüllten Vulkanisate geht jedoch durch ein Maximum. Der Einfluss der Dehnungsgeschwindigkeit auf die Last-Dehnungskurve des Gummivulkanisats ist ein solcher, wie er nach den visko-elastischen Eigenschaften des Materials erwartet werden kann; im Falle des gefüllten Vulkanisats liegen die Verhältnisse komplizierter. Eine kurze Diskussion der Ergebnisse wird durchgeführt.

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