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HEAT BUILD-UP AND BLOW-OUT OF RUBBER BLOCKS

by

A. N. Gent and M. Hindi

Institute of Polymer Science
The University of Akron
Akron, Ohio 44325

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Rubber blocks heat up under repeatedly-applied severe deformations and sometimes explode ("blow-out"). This phenomenon is shown to be due entirely to the high temperatures developed internally, about 200 °C, which cause decomposition of the vulcanizate, and generation of a volatile

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product. When the internal pressure is high enough, the surrounding rubber is torn apart and the sample bursts. The same process can be observed with a microwave oven, when the rubber sample is heated internally, without stress, to the same critical temperature T_c^{Γ}. Values of T_c^{Γ} are reported for different vulcanizates of several elastomers. They are found to be lower for softer materials, in accord with the theory of elastic expansion to burst of internal gas bubbles, and lower for materials having less stable crosslinks; for example, polysulfidic instead of monosulfidic or C-C cross-links. Maximum values of T_c^{Γ} were about 240 °C, significantly below the temperatures for rapid decomposition of the elastomers themselves. Possible reasons for this anomaly are discussed. Keywords: Explosive rupture; Fracture (Mechanics); Heating; Thermal decomposition.

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Heat-Buildup and Blow-out of Rubber Blocks

A.N.Gent and M.Hindi

Institute of Polymer Science and Polymer Engineering Center

The University of Akron , Akron , Ohio , 44325

1. 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 " blow-out ". It is an important mode of failure in thick rubber articles, such as tire treads and tank track pads, that are subjected to severe and frequently-applied loads. It is attributed here 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.

In spite of its obvious importance, there is surprisingly little published work dealing with blow-out. Many fundamental questions appear to be unanswered. Is the critical temperature at which blow-out occurs affected by the detailed chemistry of thermal decomposition, and, hence, is it different for different elastomers? Does it depend upon the physical properties of the rubber compound; for example, stiffness, extensibility, or resistance to tearing? Does repeated stressing play a direct role in causing the failure, as in mechanical fatigue of rubber, or is it merely a mechanism for raising the internal temperature to the level at which rapid decomposition takes place? In an attempt to answer some of these

questions an experimental study has now been carried out of heat build-up and blow-out in some selected rubber compounds. The results are reported here and compared with the predictions of a simple model of the blow-out process.

It should perhaps be pointed out that there is not at present a single, well-accepted, mechanism for blow-out. It was first thought to be a mechanical fatigue cracking process, aggravated by the high temperatures set up by cyclic stressing (1). 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 (2,3). In contrast, attention is focussed here on the pressure set up in the center of the block. 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). In the present case, pressure is thought to be generated by heating a volatile substance within the rubber compound; either an ingredient of the rubber mix, a byproduct of vulcanisation, or a product of thermal decomposition. This simple model is shown to account for a number of aspects of blow-out. In particular, it appears that externally-imposed stresses are not necessary to induce blow-out; the same type of failure can be brought about by the action of heat alone.

It may be asked: Why, then, is blow-out not also brought about by heating in a regular oven or autoclave ? In those cases, heat is conducted to the interior of the rubber from the surface and hence the hottest points, at which decomposition begins, are at the surface. Thus, volatile materials can readily escape. In contrast,

energy dissipated as heat as a result of mechanical working, and heat from microwave energy, is generated throughout the rubber. It is lost only from the surface. As a result, the highest temperature is developed at the center of the block.

As an illustration, if a thin layer, of thickness H, is uniformly heated at a rate Q ($J\ s^{-1}m^{-3}$) while the surfaces are maintained at a constant temperature T_o, the equilibrium temperature distribution within the layer will be parabolic in form, the maximum temperature T_m in the central plane being given by

$$T_m = T_o + (Q\ H^2/8\ K)$$

where K denotes the coefficient of thermal conductivity of the material. Because rubber is a rather poor conductor of heat the maximum temperature can reach high values, especially for thick layers. Moreover, rubber compounds that dissipate a high proportion of the energy of deformation are particularily liable to internal heating and hence to blow-out.

The experiments reported here were undertaken to find out whether simple heating was sufficient to cause blow-out or whether mechanical working was necessary for this type of failure to occur. In addition, marked differences between different rubbery materials and different compound formulations have been investigated.

2. Experimental details

Cylindrical rubber specimens, 25mm high and 17.5mm in diameter, and sheets of various thicknesses were made in a simple compression mold. The mix formulations and vulcanization conditions are given in Table 1. Values of Young's modulus for each material were determined from the initial slopes of measured stress-strain relations in compression. These measurements were made at ambient temperature and on blocks heated to elevated temperatures, close to the blow-out temperatures. The results are included in the Appendix.

Specimens were subjected to repeated compression using a Goodrich Flexometer, as described in ASTM D623 - 67 (5). The frequency of loading was 30 Hz and the stroke (double - amplitude) of imposed oscillation was 6.35mm. The severity of the test was adjusted by varying the static compressive load applied to the sample by means of a balance beam. Loads of either 14 or 19 kg were attached to the beam for this purpose. They correspond to compressive loads on the sample of 32 and 43 kg, respectively. The experiments were terminated when the sample failed by blow-out, typically within 15 min.

Measurements of the temperature of the sample were made initially by means of a thermocouple attached to the surface on which the sample rests. Due to the pronounced thermal gradients set up in the sample by heat loss from the exposed surface, this temperature was generally far below that existing at the center of the rubber cylinder, Table 1. In order to determine the real blow-out temperature, a probe thermocouple ("Piercing Probe, Type T" with "Digi-Sense J,K,T Thermometer", Cole-Palmer Instrument Company) was inserted into the sample, as close as possible to its center, immediately after blow-out had occurred. It is these

temperatures that are reported below as blow-out temperatures.

In order to duplicate the internal heating brought about by repeated compressions without imposing any deformation, other samples were heated in a small microwave oven (Sears Kenmore model 87213/4, 900W, 2450MHz). Blow-outs occurred similar to those obtained with the Goodrich Flexometer. Visual comparisons are given in Figures 1 and 2. The times required for blow-out were much shorter, however, being typically less than 1 min.

Difficulties were experienced with soft rubber compounds in both experiments. In the Goodrich Flexometer they tended to spring out of position during cyclic compression. And materials containing only small amounts of carbon black, less than about 20 parts by weight per 100 parts of rubber, did not heat up sufficiently rapidly in the microwave oven to undergo blow-out. Thus, results are given only for rubber compounds containing 25 or more parts of carbon black.

Some of the rubber compounds were examined with a DuPont Thermogravimetric Analyzer (Model 951) for the amount of weight loss that they experienced on heating in an inert atmosphere (nitrogen) at about 10°C per min.

3. Experimental results and discussion

(i) Blow-out due to repeated compression

Blow-out experiments were carried out on selected samples, using the Goodrich flexometer. Temperatures in the center of the rubber blocks, measured immediately after blow-out, are given in Table 2. They were considerably higher than those measured at the platen surface, by as much as 50°C. This difference indicates the severity of the temperature gradients set up in Goodrich flexometer samples, presumably due to cooling from the surfaces.

As the results in Table 2 show, the actual temperatures reached in the interior of the rubber at blow-out were about 200°C , similar to polymer decomposition temperatures. This suggests that blow-out is, in fact, a consequence of thermal decomposition of the polymer. In order to test this hypothesis, some experiments were carried out with a microwave oven, so that samples could be heated without being subjected to mechanical working.

(ii) Blow-out due only to heating

Samples of an SBR compound (SBR2), a natural rubber compound (NR2), and two butyl rubber compounds (IIR1 and IIR2), were subjected to blow-out both by repeated compression using the Goodrich flexometer and by direct heating in the microwave oven. As the results given in Table 2 show, the measured temperatures at which blow-out occurred were approximately the same in both experiments, although the times taken to reach blow-out were quite different ; about 10 - 15 min in the flexometer experiments but only about 1 min in the microwave oven. And the appearance of the failed samples was quite similar (Figures 1 and 2). Thus, the criterion for blow-out seems to be a relatively simple one : the center of the sample must reach a certain critical temperature. Mechanical working

is only a means of generating heat internally and reaching this critical condition at the center of the sample; it does not appear to play any other significant role in causing blow-out.

Although blow-out by flexing and by microwave heating were found to be substantially the same, there were significant differences between the appearance of samples. One obvious feature of flexed samples was their permanent set, shown by the barrel shape and retained compression evident in Figures 1 and 2. Another characteristic feature was the concentration of decomposition in the center of the sample, Figures 3 and 4, reflecting the development of the highest temperatures here. In contrast, samples that had undergone microwave heating were found to decompose over larger internal regions, wherever particularly severe heating had occurred. These zones were often not in the center of the sample because the microwave heating was not especially uniform.

Apart from these differences, the two processes were remarkably similar, taking place at the same temperature and resulting in materials with the same physical appearance. But the actual blow-out temperatures and the character of the decomposition products were found to be strikingly different for different elastomers and, to some extent, for different formulations of the same elastomer. Results for some typical compounds are presented below.

(iii) Blow-out temperatures of typical rubber compounds

The amounts of sulfur and accelerator were adjusted to give two SBR compounds of markedly different elastic modulus (SBR2 and SBR3). Blow-out was found to take place at significantly different temperatures, the lower modulus compound failing at a lower temperature, Table 3. Similarly, when the amount of carbon black in the compound formulation was varied (SBR1 and SBR3), the lower modulus material suffered blow-out at a lower temperature than the higher modulus one, Table 3. In general, higher modulus materials appeared to withstand higher temperatures than softer ones before blow-out.

Of course, it is the modulus at high temperatures that is relevant in this comparison. Measurements of modulus at temperatures close to the blow-out temperature are included in Table 3; they confirm the general conclusion reached above that materials that are harder at operating temperatures can withstand higher temperatures before blow-out.

This feature is consistent with the proposed mechanism of blow-out. The critical pressure at which a small internal cavity in a rubber block will expand indefinitely is given approximately by $5E/6$, where E is Young's modulus (4,6,7). Thus, a harder material would be expected to withstand higher internal pressures without rupturing.

Some compounds appear to soften markedly at high temperatures and undergo blow-out immediately. The butyl rubber materials are a notable example, Table 3. Indeed, it seems likely that blow-out occurs when these compounds have softened to such an extent that they are unable to withstand even slight pressures generated by a volatile constituent within them.

Thus, there appear to be two factors governing the susceptibility of rubber compounds to blow-out. The first is their propensity to generate volatile substances at high temperatures. The second is their tendency to soften on heating and thus lose resistance to expansion of a pressurized internal cavity. Although these two processes are directly connected in some instances, they are not inevitably related. For example, one could envisage a rubbery polymer that decomposed on heating to yield a highly-volatile gaseous product such as CO_2 or H_2O, but did not undergo main-chain or crosslink rupture and hence did not soften significantly. On the other hand, some vulcanizates might undergo thermal decomposition of crosslinks, and soften appreciably, without generating substantial quantities of volatile matter.

Of the materials examined here, the butyl rubber compounds showed a striking degree of softening as a result of thermal decomposition. They were transformed into soft, fluid materials, resembling butter in consistency, at blow-out temperatures, Figure 5. Thus, only a slight internal pressure would be necessary to cause blow-out of these materials. In contrast, NR and SBR compounds were found to be stiffer than others initially and to retain their stiffness at high temperatures, as the values of Young's modulus given in Table 3 show. For these materials, therefore, a high internal pressure would be necessary to cause unbounded expansion of an internal cavity. Indeed, they were found to have higher blow-out temperatures than other materials. SBR failed more by explosive rupture than by profuse evolution of gas. A cross-section of an SBR sample is shown in Figure 6. The fracture plane where the rubber has been torn apart is evident but there are

only slight signs of the gas-filled cavities that are assumed to be the origin of fracture in this material, as in the others.

Natural rubber compounds became soft and sticky as a result of thermal decomposition, whereas the SBR compounds remained relatively dry. In both cases, however, blow-out took place explosively, especially for the NR materials with C-C or monosulfide cross-links (NR2, NR3, NR4) which blew out at relatively high temperatures, Table 3. On the other hand, polybutadiene compounds did not undergo blow-out, even after long periods of heating in the microwave oven, and whatever the amount of carbon black incorporated. Instead, the samples softened, swelled, and became porous in the interior, Figure 7 without actually exploding. As they became hotter and hotter they gave off a dense white smoke but still did not burst open as the other rubber samples did. Apparently, the amount of volatile material produced by this polymer is significantly smaller, or the vapor pressure of the products of decomposition is significantly lower. "Blow-out" temperatures for polybutadiene samples, given in Table 3, were taken as the temperatures measured in the center of samples immediately after they exhibited pronounced swelling.

Possible origins of the volatile substances that cause blow-out are discussed in the following section.

(iv) Possible causes of blow-out

Certain ingredients in rubber formulations might be expected to volatilise at high temperatures and thereby create high internal pressure, causing blow-out in extreme cases. If a significant amount of water is present, for example, then high internal pressures would be generated at temperatures much above 100^{o}C. In order to see whether this is an important mechanism of blow-out, a small amount of salt was included in several mix formulations and the samples were soaked in water for several days before testing, so that they absorbed 1-2% of water by weight. They were found to undergo blow-out at virtually the same temperatures as dry samples, indicating that trapped water is not the principal cause of blow-out in rubber compounds.

Another possible volatile ingredient of many of the present compounds is a hydrocarbon processing oil. However, when an otherwise identical formulation was employed with processing oil omitted (SBR1 and IIR2), the vulcanised samples were found to blow-out at about the same temperatures as before, and in the same way, Table 3. Thus, it does not appear that the processing oil itself was responsible for blow-out.

There remains the possibility that some other ingredient of the mix formulations, or some product of the vulcanization reactions, is the important volatile substance. While this possibility cannot be completely discounted, it should be noted that the observed blow-out temperatures were quite different for different polymers, even when their mix formulations and vulcanization reactions were rather similar. On the other hand, the blow-out temperatures were very different for different vulcanization recipes with the same polymer. It is therefore thought that the vulcanizates themselves were

responsible both for the volatile products and for the lowered resistance to containing them that together resulted in blow-out.

They are known to differ substantially in their susceptibility to thermal decomposition. Vulcanizates with polysulfide crosslinks will soften at lower temperatures than those with monosulfide or with carbon-carbon crosslinks because of the lower strength of such linkages (8,9). If crosslink rupture is also accompanied by the production of a volatile substance, then the occurrence of blow-out at temperatures at which a significant extent of crosslink rupture takes place is to be expected.

The differences observed between different crosslinking systems with the same polymer are in accord with this hypothesis. Peroxide recipes, yielding C-C crosslinks, and monosulfide ("sulfurless") recipes gave the highest blow-out temperatures, Table 3, approaching the temperatures at which polymer decomposition would be expected. And the differences observed between the different elastomers can be accounted for by differences in stability of the crosslinks formed within them, even when the recipes employed were rather similar.

Thus, although the exact nature of the volatile constituent is still not clear, the hypothesis that it is a gaseous product of the thermally-excited decomposition of crosslinks in vulcanized rubber accounts reasonably well for the main features of blow-out, namely, the general temperature range in which it occurs, its close relationship with softening temperatures, the lower values of blow-out temperature found for materials containing polysulfidic crosslinks in comparison with C-C crosslinks, and the marked differences observed between different polymers.

(v) Effect of sample thickness

In order to see whether the thickness of the sample had any effect on its propensity to blow-out, specimens were prepared from the same material, an SBR compound, with widely different dimensions. As the results given in Table 4 show, they all suffered blow-out at about the same temperature, and after about the same heating time in the microwave oven. Thus, it appears that any sample will blow out if the critical temperature is reached at its center. It should be noted, however, that the thinner sheets, only about 3mm in thickness or less, did not really explode but instead developed a pronounced swelling to form a large blister or aneurysm, Figure 8, resembling the behavior discussed above of polybutadiene compounds.

This feature suggests that the principal factor in deciding whether a specimen will explode or merely swell is the amount of volatile material produced. Thin sheets will have a smaller volume of material at decomposition temperatures than thicker ones and will therefore generate a correspondingly smaller amount of volatiles. It may be surmised that polybutadiene compounds produce a smaller quantity of gaseous products on thermal decomposition, possibly reflecting the different paths that free-radical reactions tend to take in this polymer, resulting in addition rather than molecular scission (10).

(vi) Unresolved issues

Two features remain unexplained.

Even for simple rubber compounds with C-C crosslinks, the measured blow-out temperatures were surprisingly low, at most 250°C, whereas rapid thermal decomposition of the same materials was found by TGA not to take place below about 300°C. While it is possible that the measured blow-out temperatures were systematically in error, because of delays in inserting the thermocouples and difficulties in obtaining good thermal contact with the fractured rubber surfaces, the results were consistent and reproducible. Improved experimental techniques are needed to determine exactly what the blow-out temperatures are. The present measurements should probably be regarded as giving correct relative values but an underestimate of the real temperatures.

The second unknown is the actual volatile material or materials responsible for blow-out. Several attempts were made to collect and analyze the vapors emitted by samples undergoing thermal decomposition. They were found to consist of hydrocarbons, probably including the elastomer monomer, but no substance characteristic of the particular crosslinking system used could be detected. And yet large differences were found in blow-out temperatures for different vulcanizates of the same polymer. Again, improved experimental techniques are needed to determine the decomposition products completely.

4. Conclusions

The phenomenon of explosive "blow-out" of thick rubber blocks, under repeatedly-applied, severe compressive loads, is due entirely to the development of high internal temperatures. If the compound is electrically conductive, the phenomenon can be duplicated in a microwave oven without imposing any mechanical loads.

Blow-out appears to consist of the expansion to burst of pressurized cavities within the rubber compound. Pressure appears to be generated internally by a volatile constituent or decomposition product of the rubber. Expansion is restrained by elastic stresses set up in the rubber as the cavity expands. Bursting is made easier in some compounds because they soften markedly at high temperatures and thus lose resistance to cavity expansion.

Different elastomers have strikingly different blow-out temperatures. Butyl rubber compounds blow out at relatively low temperatures, about 180°C, whereas NR and SBR compounds blow out at temperatures of about 200°C or higher. Also, different vulcanizate structures have different blow-out temperatures. For example, materials with C-C or monosulfidic crosslinks show higher blow-out temperatures than those with polysulfidic crosslinks.

Polybutadiene compounds did not blow out at all. Instead, they developed internal cavities that grew in size and number but never burst open to the exterior, probably because the internal pressure never reached sufficiently high levels.

It is concluded that the principal cause of blow-out is the generation at high temperatures of sufficient quantities of a volatile decomposition product, but the reaction is specific to the particular elastomer and crosslinking system employed. It does not appear to consist of simple thermal decomposition of the hydrocarbon

elastomer, which would require higher temperatures than those observed at blow-out.

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Table 1.

Mix formulations, in parts by weight, and vulcanization conditions employed in preparing test samples, are given below. Values of Young's (tensile) modulus E, measured in compression at 25°C on vulcanized samples, are appended to the table.

	Compound Formulation											
	SBR1	SBR2	SBR3	SBR4	NR1	NR2	NR3	NR4	BR1	BR2	IIR1	IIR2
SBR 1502	100	100	100	100								
Natural rubber (Pale crepe)					100	100	100	100				
Polybutadiene (Diene 35 NFA, Firestone)									100	100		
Butyl rubber (365, Exxon)											100	100
Carbon black (Vulcan 3, HAF N330, Cabot)	25	50	50	50	50	50	50	50	30	50	40	40
Zinc oxide	5	5	5	0	5	5	0	0	5	5	5	5
Stearic acid	2	2	2	0	2	2	0	0	2	2	2	2
Processing oil	0	5(A)	5(A)	5(A)	5(B)	5(B)	5(B)	5(B)	5(B)	5(B)	5(C)	0
Sulfur	2	1	2	0	2.5	0.25	0	0	1	1	2	2
Dicumyl peroxide	0	0	0	1	0	0	2.5	3	0	0	0	0
Accelerator	1(D)	0.5(D)	1(D)	0	0.6(D)	4(E)	2(F)	0	0.5(D)	0.5(D)	1(G)	1(G)
Salt (NaCl)	0	0.5	0.5	0	0	0	0	0	0	0	0	0
Vulcanization time (min)	50	50	50	50	50	50	50	50	50	50	60	60
Vulcanization temp.(°C)	150	150	150	150	150	155	155	155	150	150	150	150
E (MPa)	4.5	3.6	8.1	4.5	4.5	6.1	3.2	4.5	1.3	2.0	7.7	6.8

A: Sundex 790T (Sun Petroleum Company)
B: Sunflex 115 (Sun Petroleum Company)
C: Sunpar 2280 (Sun Petroleum Company)
D: Santocure MOR (Monsanto)
E: Tetramethylthiuram disulfide (TMTD)
F: Zinc dimethyldithiocarbamate (ZMDC)
G: Tetramethylthiuram monosulfide (TMTM)

Table 2: Blow-Out Temperatures T_b and Times t in Flexometer and Microwave Experiments.

Material	Flexometer			Microwave	
	T_b (°C) Sample base	T_b (°C) Sample center	t(s)	T_b (°C) Sample center	t(s)
NR1	164	181	510	176 ± 14	53
SBR3	145 ± 7	215	420	227 ± 9	47
IIR1	144	182 ± 2	1260	182 ± 4	27
IIR2	146	184 ± 5	1200	185 ± 5	26

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IIR2	146	184 ± 5	1200	185 ± 5	26

Table 3: Effect of Young's Modulus E on Microwave Blow-Out Temperatures T_b for Various Rubber Compounds.

Material	E (MPa) at 25°C	E (MPa) at 190°C	T_b (°C)	Blow-Out Time t(s)
NR1	4.5	2.2	176 ± 14	53
NR2	6.1	4.5	215 ± 7	45
NR3	3.2	2.9	213 ± 5	58
NR4	4.5	4.3	222 ± 7	93
SBR1	4.5	3.4	196 ± 8	180
SBR2	3.6	---	173 ± 10	43
SBR3	8.1	4.5	227 ± 9	47
SBR4	4.5	5.1	252 ± 5	75
BR1	1.3	---	200 ± 3	56
BR2	2.0	---	192	76
IIR1	7.7	1.2	182 ± 4	27
IIR2	6.8	0.8	185 ± 5	26

Table 4: Effect of Sample Thickness on Microwave Blow-Out Temperature (Compound SBR3).

Sample Thickness (mm)	Blow-Out Temperature (°C)
(Cylinder, diameter 17.5 mm, height 25 mm)	227 ± 9
12.7	232 ± 7
7	230 ± 8
4	232 ± 6*
2	228 ± 10*

Note: Results marked with an asterisk denote temperatures of pronounced swelling of the sample (see Figure 8). Explosive blow-out did not take place in these cases.

Figure Legends

Figure 1. Blow-out samples of NR1. (a) Flexometer. (b) Microwave.

Figure 2. Blow-out samples of SBR3. (a) Flexometer. (b) Microwave.

Figure 3. Cross-section of NR1 samples cut open shortly before blow-out. (a) Flexometer. (b) Microwave.

Figure 4. Cross-section of SBR3 samples cut open shortly before blow-out. (a) Flexometer. (b) Microwave.

Figure 5. Cross-section of a butyl rubber sample (IIR1), cut open after blow-out in a microwave oven.

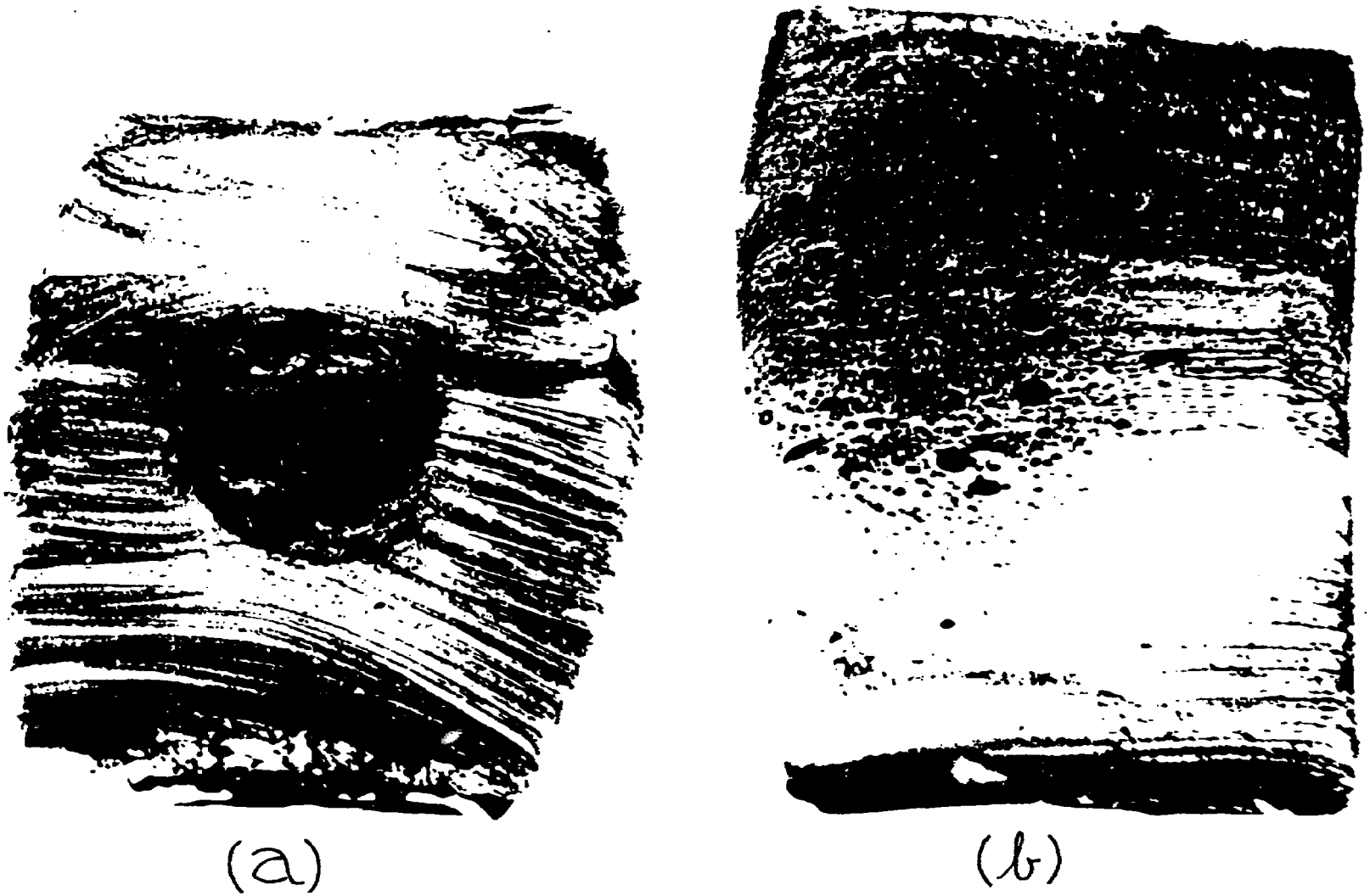
Figure 6. (a) Cross-section of an SBR1 sample, cut open shortly before blow-out in a microwave oven.
(b) View of a similar sample after blow-out.

Figure 7. Cross-sections of BR1 samples, cut open after various periods of heating in a microwave oven. (a) 2 min, (b) 6 min, (c) 10 min.

Figure 8. Cross-sections of thin samples of SBR3, cut open after heating in a microwave oven for 40 s. Sheet thickness: (a) 4 mm, (b) 2 mm.

Figure 1

Figure 2



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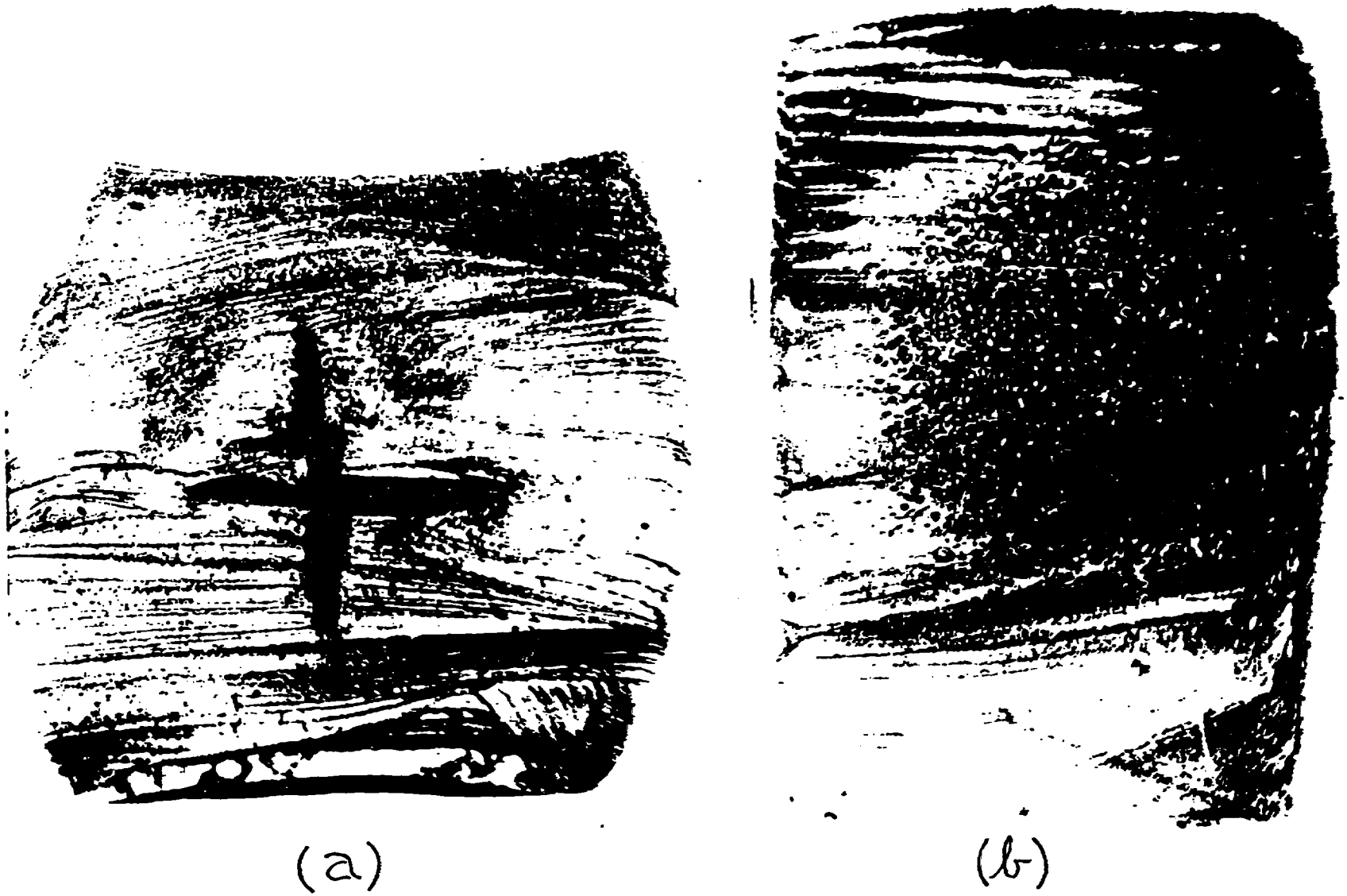

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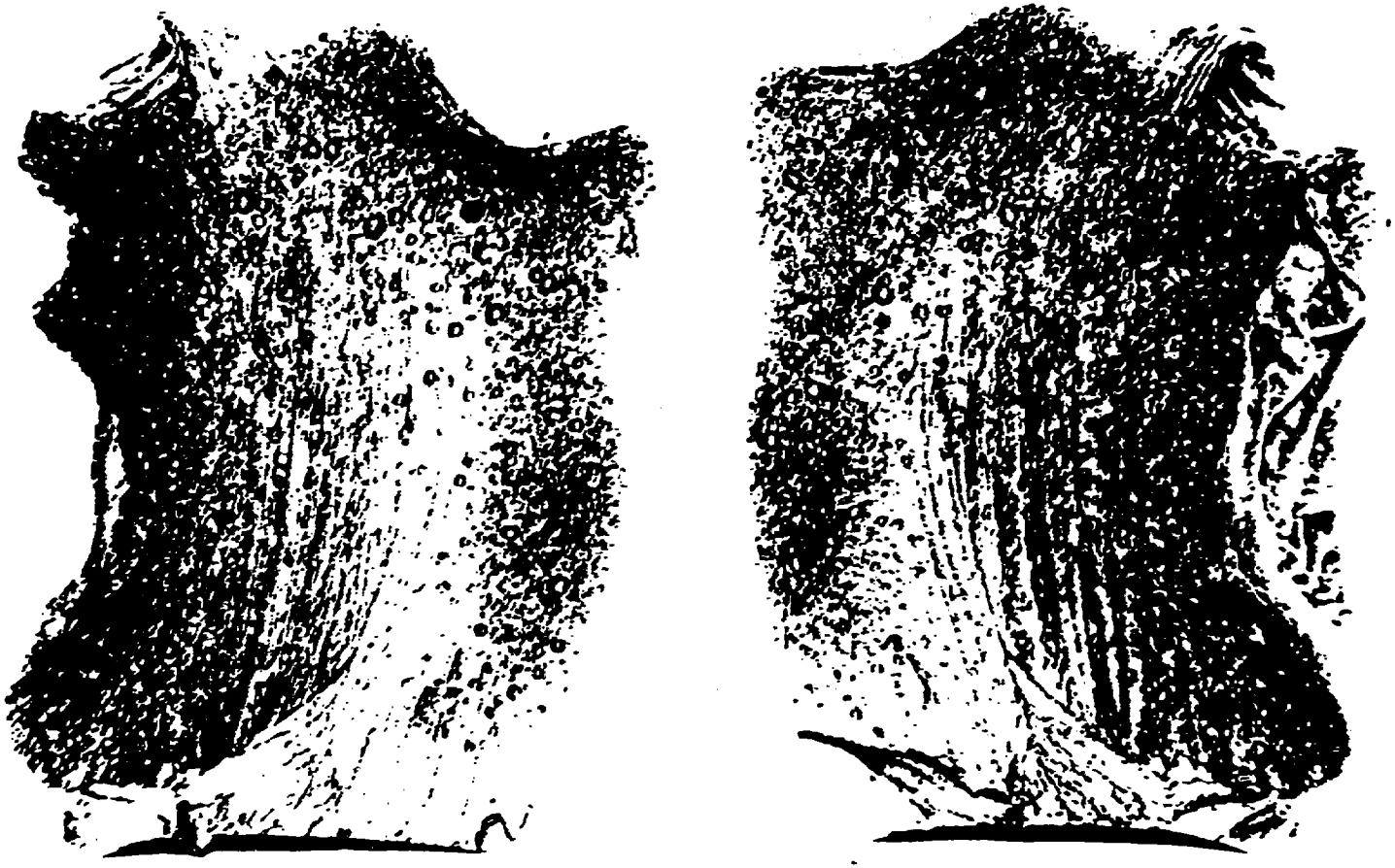

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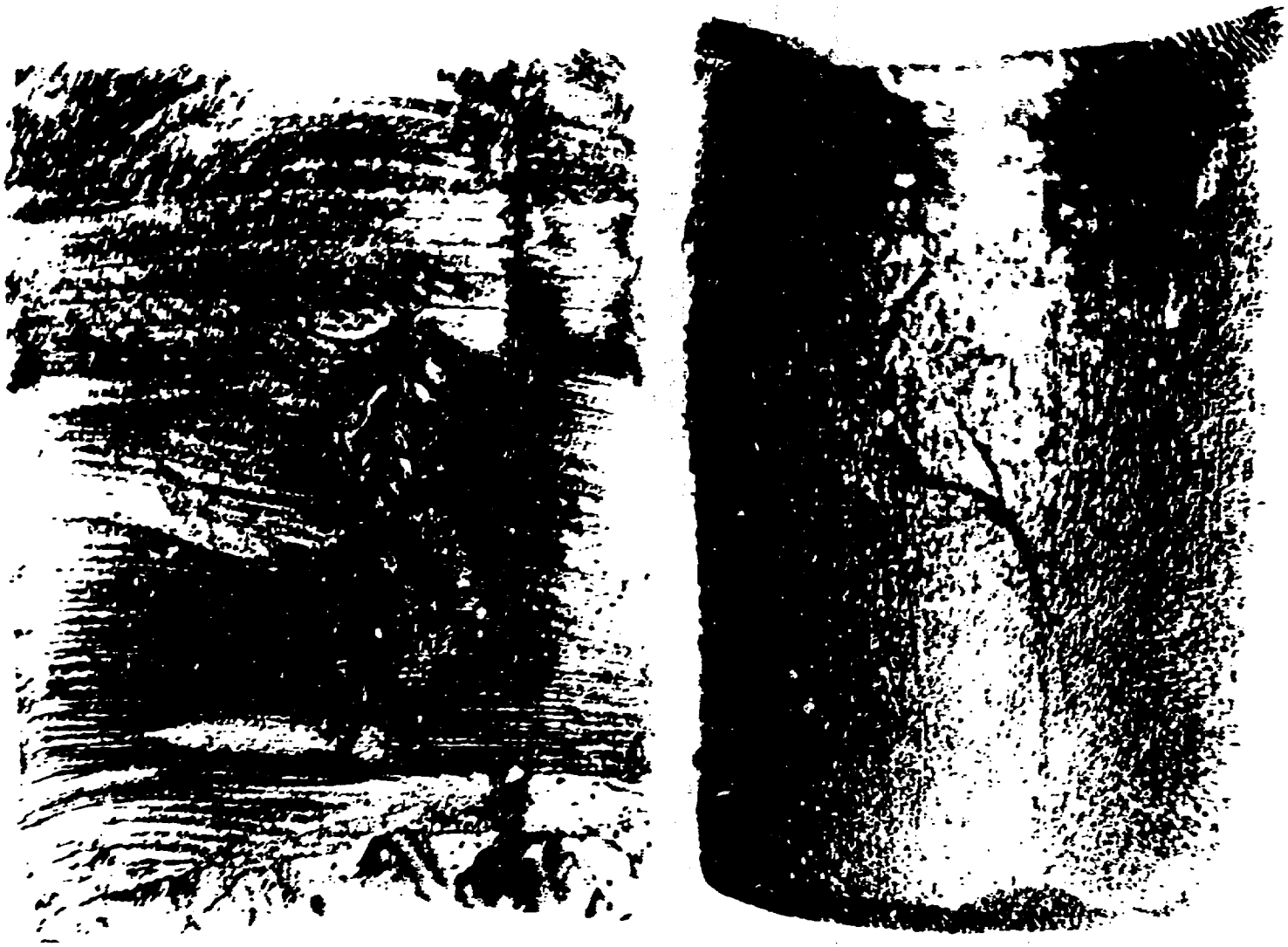
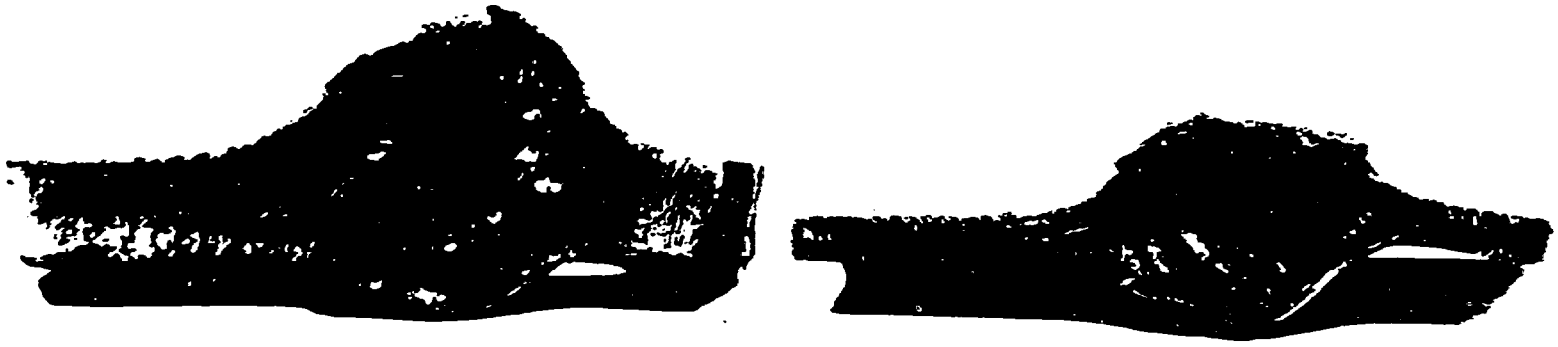


Figure 6

Figure 7



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