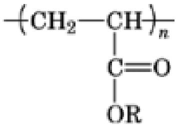
Kirk-Othmer Encyclopedia of Chemical Technology.

ACRYLIC ELASTOMERS

Acrylic elastomers have the ASTM designation ACM (1) for polymers of ethylacrylate and other acrylates. Conventionally, the M indicates a polymer having a saturated chain of the polymethylene type. The repeat structure of this polymer family shows both the presence of a saturated backbone, which is responsible for the high heat and oxidation resistance, and the ester side groups, which contribute to the marked polarity of this elastomer chain. These two main properties are not present in general-purpose rubbers. As a result, ACMs are designated as specialty elastomers, and are extensively used in highly demanding applications such as automotive underhood components.



$$\left(CH_2 - \underset{\underset{\underset{OR}{|}}{C=O}}{\underset{|}{CH}} \right)_n$$

The earliest study describing vulcanized polymers of esters of acrylic acid was carried out in Germany by Rohm (2) before World War I. The first commercial acrylic elastomers were produced in the United States in the 1940s (3–5). They were homopolymers and copolymers of ethyl acrylate and other alkyl acrylates, with a preference for poly(ethyl acrylate) [9003-32-1], due to its superior balance of properties. The main drawback of these products was the vulcanization. The fully saturated chemical structure of the polymeric backbone in fact is inactive toward the classical accelerators and curing systems. As a consequence they required the use of aggressive and not versatile compounds such as strong bases, eg, sodium metasilicate pentahydrate. To overcome this limitation, monomers containing a reactive moiety were incorporated in the polymer backbone by copolymerization with the usual alkyl acrylates.

1. Manufacture

Acrylate esters can be polymerized in a variety of ways. Among these is ionic polymerization, which although possible (6–9), has not found industrial application, and practically all commercial acrylic elastomers are produced by free-radical polymerization. Of the four methods available, ie, bulk, solution, suspension, and emulsion polymerization, only aqueous suspension and emulsion polymerization are used to produce the ACMs present in the market. Bulk polymerization of acrylate monomers is hazardous because it does not allow efficient heat exchange, required by the extremely exothermic reaction.

1.1. Solution Polymerization

This method is not commercially important, although it is convenient and practical, because it provides viscous cements that are difficult to handle. Also, the choice of the solvent is a key parameter due to the high solvent chain-transfer constants for acrylates.

1.2. Suspension Polymerization

This method (10) might be considered as a number of bulk polymerizations carried out simultaneously in the monomer droplets with water acting as a heat-transfer medium. A monomer-soluble initiator, eg, a peroxide or azo compound, and a protective colloid like poly(vinyl alcohol) or bentonite, are required. After completion of the polymerization, the excess of monomer(s) is steam stripped, and the beads of polymer are collected and washed on a centrifuge or filter and dried on a vibrating screen or by means of an expeller–extruder.

1.3. Emulsion Polymerization

In this method, polymerization is initiated by a water-soluble catalyst, eg, a persulfate or a redox system, within the micelles formed by an emulsifying agent (11). The choice of the emulsifier is important because acrylates are readily hydrolyzed under basic conditions (11). As a consequence, the commonly used salts of fatty acids (soaps) are preferably substituted by salts of long-chain sulfonic acids, since they operate well under neutral and acid conditions (12). After polymerization is complete the excess monomer is steam-stripped, and the polymer is coagulated with a salt solution; the crumbs are washed, dried, and finally baled.

Residual monomers exhibit a characteristic sharp odor even in subtoxic concentration, due to the very low olfactory threshold. Modern requirements in terms of environmental safeguard have led to significant improvements in the control of polymerization effluents, driving off gases, and residual monomer in the raw polymer. Consequently, the acrylic elastomers of the 1990s are practically odor-free, and represent a significant improvement over the products of the past.

2. Monomers

Two kinds of monomers are present in acrylic elastomers: backbone monomers and cure-site monomers. Backbone monomers are acrylic esters that constitute the majority of the polymer chain (up to 99%), and determine the physical and chemical properties of the polymer and the performance of the vulcanizates. Cure-site monomers simultaneously present a double bond available for polymerization with acrylates and a moiety reactive with specific compounds in order to facilitate the vulcanization process.

2.1. Backbone Monomers

The most important monomer both from a historical and commercial point of view is ethyl acrylate [140-88-5]. Substantially, homopolymers of ethyl acrylate with little amounts of proper cure-site monomer(s) constitute the base grade of the ACMs present on the market (13). These grades offer excellent oil resistance and a very satisfactory thermal resistance, but limited low temperature behavior, because the glass-transition temperature (T_g) is about $-15°C$ (14). Alkyl acrylates with longer side chains exhibit lower T_g (15). On the other hand, the longer alkyl group reduces oil resistance (16) so that a proper balance has to be found. Experimental and theoretical considerations based on the Group Contribution Theory (GCT) (17) are useful to predict both the low temperature flexibility, expressed as T_g, and the most important oil resistance related parameter δ as a function of the side-group chain length of poly(n-alkyl acrylates) (18). Figure 1, which correlates the predicted glass-transition temperatures (T_g) and the solubility parameters (δ) indicates that apart from

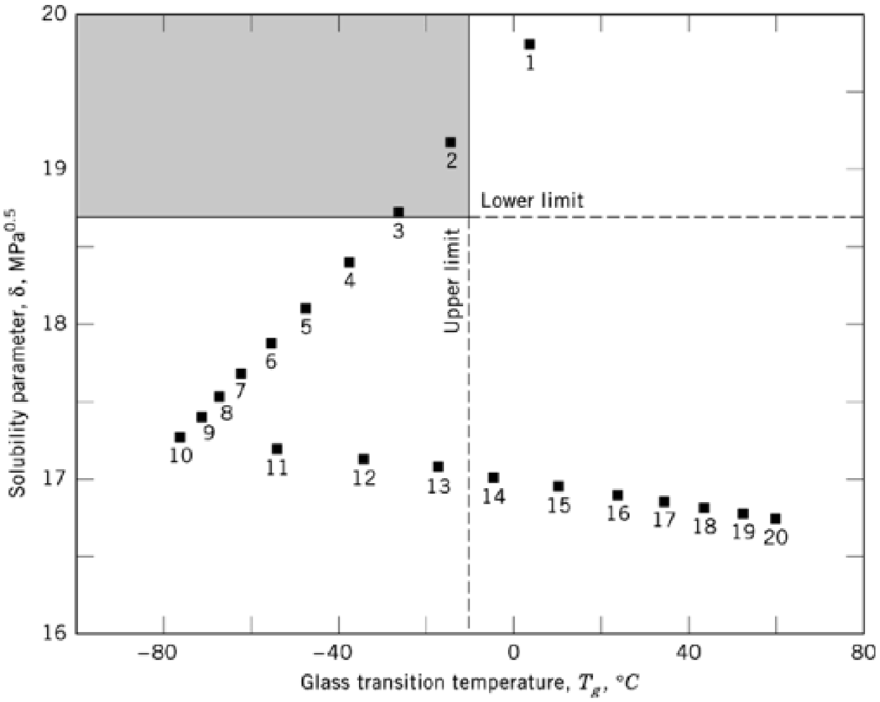
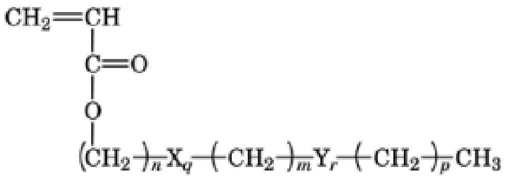


Fig. 1. T_g vs δ for poly(alkyl acrylates). ■=number of C atoms in alkyl side chain. To convert $\mathrm{MPa}^{0.5}$ to $(\mathrm{cal/cm}^3)^{0.5}$, divide by 2.05.

poly(ethyl acrylate) and poly(propyl acrylate) [24979-82-6] no other poly(alkyl acrylate) reaches a satisfactory balance of property requirements. Thus butyl acrylate [141-32-2] is only used as a comonomer, usually with ethyl acrylate.

To improve the performance of acrylic elastomers, side chains are required where the δ value is higher than with n-alkyl groups. Thus the use of polar groups, for instance heteroatoms, is suggested. The general formula for these acrylate monomers may be portrayed as follows:



$$CH_2{=}CH{-}C({=}O){-}O{-}(CH_2)_n X_q {-} (CH_2)_m Y_r {-} (CH_2)_p CH_3$$

If $q = 1$, r and $p = 0$ and X = oxygen, the family of alkoxyalkyl acrylates is obtained. The improvement of the solubility parameter for this family compared to poly(n-alkyl acrylates) is shown in Figure 2.

The most important member of this class is 2-methoxyethyl acrylate (MEA) [3121-61-7] (16), which along with ethyl acrylate and butyl acrylate constitutes the building blocks of current acrylic elastomers.

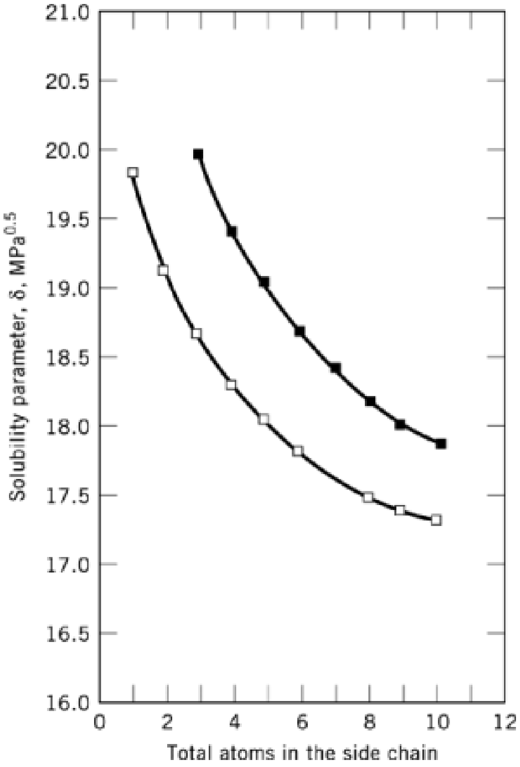


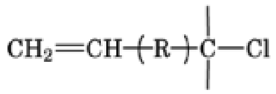
Fig. 2. Improvement in δ achieved by alkoxy substituents. □= only carbon atoms; ■= with one oxygen atom. To convert $MPa^{0.5}$ to $(cal/cm^3)^{0.5}$, divide by 2.05.

2.2. Cure-Site Monomers

A large variety of cure-site monomers has been proposed, but only a few have achieved commercial significance. Two of the most important classes are labile chlorine containing monomers and epoxy/carboxyl containing monomers.

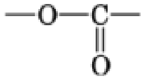
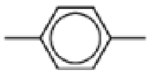
2.2.1. Labile Chlorine Containing Monomers

Chlorine is introduced in the acrylic elastomer chain by analogy to polychloroprene (19). The monomers are characterized by the simultaneous presence of a double bond available for polymerization with acrylates and a chlorine atom ready to react easily during the vulcanization step. The general formula is as follows where R is a group that might enhance the reactivity of the double bond and/or of the vicinal chlorine atom.



$$\mathrm{CH_2{=}CH{-}\!\left(R\right)\!{-}\overset{|}{\underset{|}{C}}{-}Cl}$$

The historical evolution of R producing ever more reactive chlorine and/or double bonds is summarized in the following by way of examples of representative monomers.

Year	R	Monomer	CAS Registry Number	Reference
1950	$—O—CH_2—$	2-chloroethyl vinyl ether	[110-75-8]	19
1960	 $—O—\underset{\underset{O}{\Vert}}{C}—$	vinyl chloroacetate	[2549-51-1]	20
1970		*p*-vinylbenzyl chloride	[30030-25-2]	21

In order to enhance the reactivity of the chlorine atom, a second reactive monomer can be adopted giving dual cure sites. According to the literature, the second monomer can contain carboxyl (22–24), cyanoalkyl (25), hydroxypropyl (26), or epoxy groups (27, 28).

2.2.2. *Epoxy/Carboxy Cure Sites*

Epoxy/carboxy cure sites probably represent the most important alternative to labile chlorine containing monomers. There has been increasing interest in them due to the discovery of the highly efficient quaternary ammonium salt-based accelerators (29–34). The reaction between the epoxy ring and carboxylic acid can happen in the following three ways:

Reactive sites in the polymer	Curing system
epoxy containing reactive monomer	poly(carboxylic acid) + catalyst
carboxy containing reactive monomer	polyepoxide compound + catalyst
carboxy containing reactive monomer + epoxy containing reactive monomtabuer	catalyst

2.2.3. *Double-Bond Cure Sites*

The effectiveness of this kind of reactive site is obvious. It allows vulcanization with conventional organic accelerators and sulfur-based curing systems, besides vulcanization by peroxides. Fast and controllable vulcanizations are expected so double-bond cure sites represent a chance to avoid post-curing. Furthermore, blending with other diene elastomers, such as nitrile rubber [9003-18-3], is greatly facilitated.

The idea of using polyunsaturated monomers is rooted in the early history of acrylic elastomers. The first monomers used were butadiene [106-99-0] (35), isoprene [78-79-5] (36), and allyl maleate [999-21-3] (37), but they did not find commercial success because during polymerization large portions of polymer were cross-linked. Other monomers have been proposed more recently: tetrahydrobenzyl acrylate (38), dicyclopentenyl acrylate [2542-30-2] (39), and 5-ethylidene-2-norbornene [16219-75-3] (40). The market potential, at least for the more recent ones, is still to be determined.

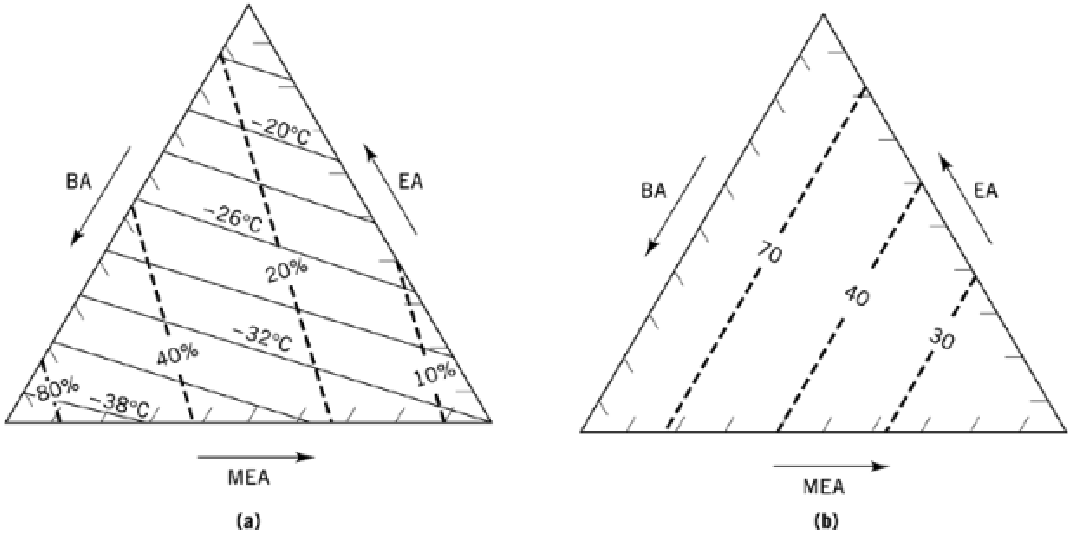


Fig. 3. Elastomer properties as a function of monomer composition, butyl acrylate (BA), ethyl acrylate (EA), and methoxyethyl acrylate (MEA). (**a**), (—) glass-transition temperature; (___) swelling in ASTM No. 3 oil; (**b**) (_ _ _) residual elongation at break, %, after heat aging.

2.3. Structure–Property Relationships

The modern approach to the development of new elastomers is to satisfy specific application requirements. Acrylic elastomers are very powerful in this respect, because they can be tailor-made to meet certain performance requirements. Even though the structure–property studies are proprietary knowledge of each acrylic elastomer manufacturer, some significant information can be found in the literature (18, 41). Figure 3**a** shows the predicted T_g, according to GCT, and the volume swell in reference fluid, ASTM No. 3 oil (42), related to each monomer composition. Figure 3**b** shows thermal aging resistance of acrylic elastomers as a function of backbone monomer composition.

The cure sites present in the polymer also significantly influence the expected properties. Labile chlorine cure sites generally give good elongation at break retention after heat aging, and show low sensitivity to acidic compounds in the mix recipe. Epoxy/carboxyl cure sites give good compression set and good hydrolysis resistance. Dual cure site-based polymers vulcanized by quaternary ammonium salts may behave in different ways (43, 44) compared to conventional cure sites. These considerations and Figure 3**a** and 3**b** taken together offer an example of a molecular engineering approach to the design of acrylic elastomers tailor-made with specific requirements in mind.

3. Applications

Specialty rubbers, to which ACM belongs, are selected on the basis of their performance. As a consequence, many elastomeric families may compete for the same application. A first selection can be carried out on the basis of oil and temperature resistance, according to ASTM D2000 (Fig. 4).

Selection of the right elastomer has to take into account not only the low temperature resistance, the elastic properties, and mechanical properties, but also price, which can vary widely in specialty elastomers.

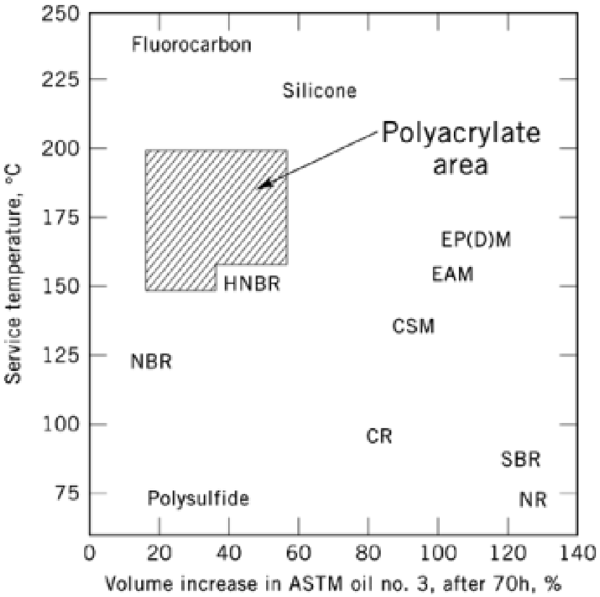


Fig. 4. Oil and temperature resistance of various elastomers.

On these bases ACMs have found commercial applications mainly in automotive nontire applications. Some significant examples are as seals: lip, pinion, shaft, transmission, clutch, O-rings, bearing dust seals; as gaskets: oil pan, emission control, cover valve, cork-bound, sponge gaskets; in electrical insulation (ignition cable, spark plug boots); valve stem oil deflectors; hose and hose linings; miscellaneous mechanical goods; wire and cable jacketing; noise control and vibration damping; and plastic modification.

4. Compounding

A rubber compound typically consists of many additives and ingredients, although in the case of acrylic rubber compound, the recipes are usually simpler. General recipes include the following:

Ingredient	phr
polymer	100
stearic acid	1–3
processing aid	1–3
antioxidant	1–2
filler (black and/or white)	20–100
plasticizer	0–5
cure system	0.5–8

The compound recipe is a function both of cure site(s) present in the polymer and of application requirements.

As a general rule zinc oxide is not used because it is not involved in the vulcanization mechanism of acrylic rubber. Zinc salts, eg, zinc stearate, have to be avoided because they may adversely affect the performance of the compound and/or the vulcanizate.

Plasticizers (qv) are usually present at lower concentrations compared to general-purpose rubber-based compounds, because of their volatility at typical ACM service temperatures and/or their partial extractability by the aggressive fluids where acrylic elastomers are employed. Other additives are therefore required to improve processibility. These processing aids act as lubricating agents and enhance the release characteristics of the acrylic compound and/or reduce compound viscosity.

Antioxidants (qv) usually provide only a marginal improvement in heat aging resistance because of the inherent oxidation resistance of the saturated backbone of ACM. The antioxidants most frequently used are nonvolatile amine compounds.

Carbon blacks are the preferred reinforcing fillers because they provide a better overall balance of vulcanizate properties than the mineral white fillers. As with other elastomers, smaller particle size blacks provide the highest reinforcement, whereas higher structure types contribute to processing characteristics. As a general rule, relatively low amounts of the ASTM N300 series of blacks are suggested for injection molding applications, and higher amounts of medium large particle size blacks (N500 and N700) are preferred in extrusion (see Carbon, carbon black).

Where the application requires effective electrical insulation or color coding, white filler reinforcing agents have to be used. High reinforcing silica tends to give compounds that harden during storage. Aluminum silicate and silicoaluminate are usually preferred. To improve filler–polymer interaction and vulcanization behavior silane-modified fillers are often employed.

Graphites (natural or synthetic) are black semireinforcing fillers used almost exclusively in rotary shaft seal applications where surface lubricity and abrasion resistance must be enhanced (see Carbon, natural graphite).

5. Vulcanization Systems systems

Because of the different vulcanization chemistry involved in each commercial ACM, a vulcanization system specific to the cure site present has to be adopted. Many cure systems for labile chlorine containing ACM have been proposed (45). Among these the alkali metal carboxylate–sulfur cure system, or soap–sulfur as it is called in the United States, became the mainstay of acrylic elastomer technology in the early 1960s (46), and continues to be widely used.

New efficient vulcanization systems have been introduced in the market based on quaternary ammonium salts initially developed in Italy (29–33) and later adopted in Japan (34) to vulcanize epoxy/carboxyl cure sites. They have been found effective in chlorine containing ACM dual cure site with carboxyl monomer (43). This accelerator system together with a retarder (or scorch inhibitor) based on stearic acid (43) and/or guanidine (29–31, 33) can eliminate post-curing. More recently (47, 48), in the United States a proprietary vulcanization package based on zinc diethyldithiocarbamate [14324-55-1] (49) has been reported that offers a superior balance between cure rate at molding temperatures and safety at process temperatures. It is proposed as a curative system that does not require post-vulcanization.

More detailed compounding suggestions and additives information can be found in the technical bulletins published by ACM and additives suppliers (50–54).

6. Economic Aspects

Acrylic rubbers, as is the case for most specialty elastomers, are characterized by higher price and smaller consumption compared to general-purpose rubbers. The total rubber consumption in 1991 was forecast (55) at 15.7 million t worldwide with a 66% share for synthetic elastomers (10.4×10^6 t). Acrylic elastomers consumption, as a minor amount of the total synthetic rubbers consumption, can hardly be estimated. As a first approximation, the ACM consumption is estimated to be 7000 t distributed among the United States, Western Europe, and Japan/Far East, where automotive production is significantly present.

The price of ACM is approximately four to five times greater than that of general-purpose rubbers like SBR or NR, and about three times the price of standard engineering rubbers such as NBR and EPDM. However, they cost about four to six times less than other specialty elastomers like fluorocarbon elastomers FKM [25790-89-0] or HNBR.

Some significant information about trade names, producers, plant location, and capacity are reported in Table 1.

Table 1. Production Capacities of Acrylic Elastomers

Company	Trade name	Location	Capacity, t/yr
EniChem Elastomeri	Europrene AR Cyanacryl[a]	Ravenna (Italy)	2000
Nippon Zeon	HyTemp[b]	Louisville, Ky.	4000
Nippon Zeon	Nipol	Kawasaki (Japan)	500[c]
Toa Paint	Acron	Osaka (Japan)	1300
NOK	Noxtite	na (Japan)	na
Toa Resin	Acron	Taipei (Taiwan)	600 0.0pt1,80.0pt

[a] Formerly produced by American Cyanamid in Bound Brook, N.J. (U.S.).

[b] Formerly trademark Hycar, produced by B.F. Goodrich.

[c] Estimated capacity.

7. Health and Safety

Acrylic elastomers are normally stable and not reactive with water. The material must be preheated before ignition can occur, and fire conditions offer no hazard beyond that of ordinary combustible material (56). Above 300°C these elastomers may pyrolize to release ethyl acrylate and other alkyl acrylates. Otherwise, thermal decomposition or combustion may produce carbon monoxide, carbon dioxide, and hydrogen chloride, and/or other chlorinated compounds if chlorine containing monomers are present in the polymer.

Overexposure to acrylic rubbers is not likely to cause significant acute toxic effects. ACM however may contain residual monomers, mainly acrylate monomers, vapors of which are known to cause eye and/or skin irritation.

According to ACGIH (57) the Threshold Limit Values (TLV) for a normal 8-h workday and 40-h workweek exposure (TWA) are 5 ppm (20 mg/m^3) for ethyl acrylate, and 10 ppm (55 mg/m^3) for butyl acrylate. Furthermore these monomers are codified A2 (Suspect Human Carcinogen). Therefore, according to good practice of industrial hygiene, these types of rubber should be used in a well-ventilated area. The use of eye protection and gloves is recommended when handling acrylic elastomers.

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Related Articles

Elastomers, Synthetic, Survey; Butyl Rubber; Chlorosulfonated Polyethylene; Ethylene–Acrylic Elastomers; Ethylene–Propylene–Diene Rubber; Fluorocarbon Elastomers; Nitrile Rubber; Phosphazenes; Polybutadiene; Polychloroprene; Polyethers; Polyisoprene; Thermoplastic Elastomers