Application Brief

Hitachi High-Tech HITACHI

Hitachi High-Tech Science Corporation
RBM Tsukiji Bldg., 15-5, Shintomi 2-chome, Chuo-ku, Tokyo 104-0041
TEL:+81-3-6280-0068 FAX:+81-3-6280-0075
http://www.hitachi-hitec-science.com

TA NO.10 MAY.1981 TG/DTA Measurements of Rubber

1. Introduction

Natural rubber and various types of synthetic rubbers are commonly used. The characteristics rubbers vary according to their types and the difference in the types and quantities of additives (i.e. vulcanizing agents, extenders and softeners).

Differences in characteristics due to different types of rubber and additives, the ratio between polymer content and additive content, and percentages of respective types of rubber in mixed rubbers can all be determined by measuring thermal decomposition behavior using a TG/DTA simultaneous measurement instrument.

In this brief, TG/DTA was used to measure the pyrolysis of SBR (styrene butadiene rubber), NR (natural rubber), CR (Chloroprene rubber), and SBR-CR mixed rubber to determine differences in composition ratio and thermal resistance between various types of rubber.

2. Methods and Data

2-1 SBR (styrene butadiene rubber)

Figure 1 shows that SBR lost 99% of its weight when it was heated up to 500℃ in the N_2 gas atmosphere. This fact indicates that 99% of the sample consisted of polymers and softeners. The residual 1% is considered to account for the vulcanizing agent, metallic compounds added as catalysts, and sulfides.

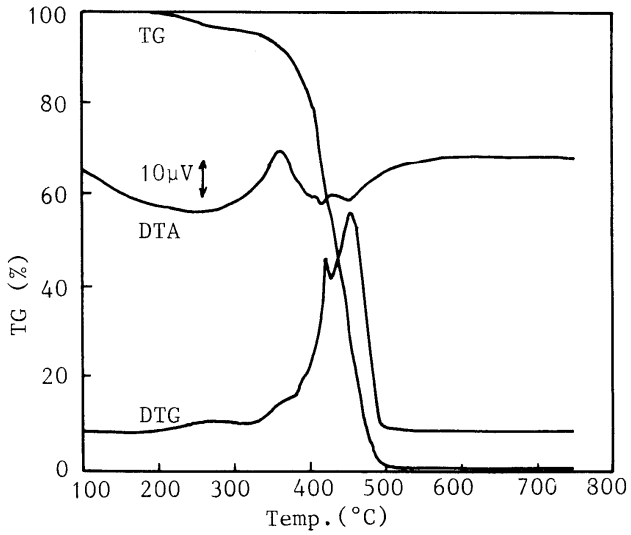

Figure 1 TG/DTA curve of SBR(styrene butadiene rubber)
Sample weight : 15mg
Heating rate : 10℃/min
Atmosphere : N_2, 250ml/min

The DTA curve shows an exothermic peak near 360℃ and an endothermic peak near 410 to 450℃. Prior to decomposition, the butadiene portion in the sample was recombined after generation and shifting of the radical due to severance of the principal chain and partially assumed the cyclic structure. This reaction is said to be observed as an exothermic peak[1)]. Thereafter, the endothermic peak appeared due to decomposition and gasification of the sample.

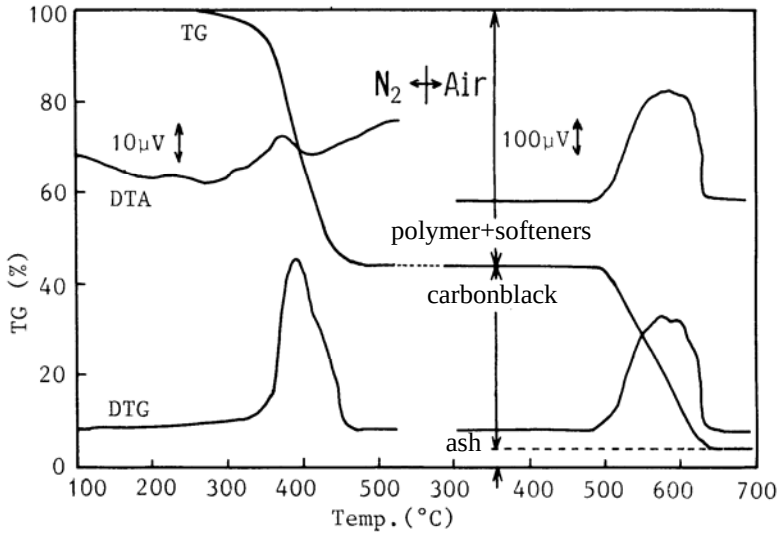

Figure 2 TG/DTA curve of NR (natural rubber)
Sample weight : 15mg
Heating rate : 20℃/min
Atmosphere : N_2(R.T.～550℃), 250ml/min
Air(300～700℃), 250ml/min

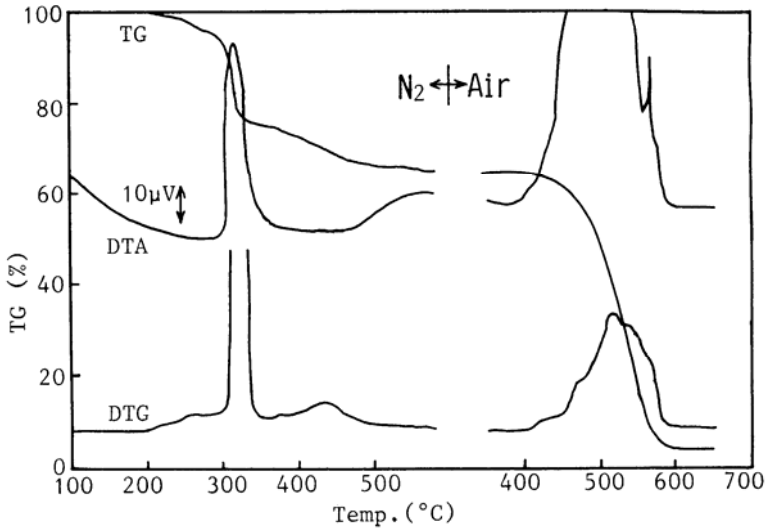

Figure 3 TG/DTA curve of CR (Chloroprene rubber)
Sample weight : 15mg
Heating rate : 10℃/min
Atmosphere : N_2(R.T.～550℃), 250ml/min
Air(350～650℃), 250ml/min

2-2 NR (natural rubber)

Figure 2 shows that NR lost 56% of its weight due to decomposition and gasification of the polymers and softener when heated to 550℃ in N_2 gas. Then the temperature was dropped to 300℃ and the gas flow was changed from N_2 to Air. Since this TG/DTA module is of a horizontal differential type, no fluctuation in apparent weight due to gas change-over was observed.

When heated in air, NR lost 40% of its weight accompanied by significant heat production within the range of 500 to 630℃. This is considered to have been caused by oxidation and decomposition of carbon black which was added to NR as an extender.

2-3 CR (Chloroprene rubber)

Figure 3 shows that CR suddenly lost most of its weight accompanied by an exothermic reaction in the vicinity of 330℃. This seems to have resulted from dehydrochrorination from CR. Later decomposition and oxidation of carbon black was observed, the same as in the cases of SBR and NR.

2-4 SBR-CR Mixed Rubber

In order to evaluate SBR-CR mixed rubber, the CR content of SBR-CR rubber was calculated on the basis of the weight loss accompanying dehydrochrorination caused by pyrolysis of CR. Figure 4 shows that the weight reduction ratio due to de-HCl was 26.9%, meaning that the CR content was 54.7%.

References

1) M.A.Golub and R.J.Gargiulo, Polymer Lett., **10**, 41 (1972)

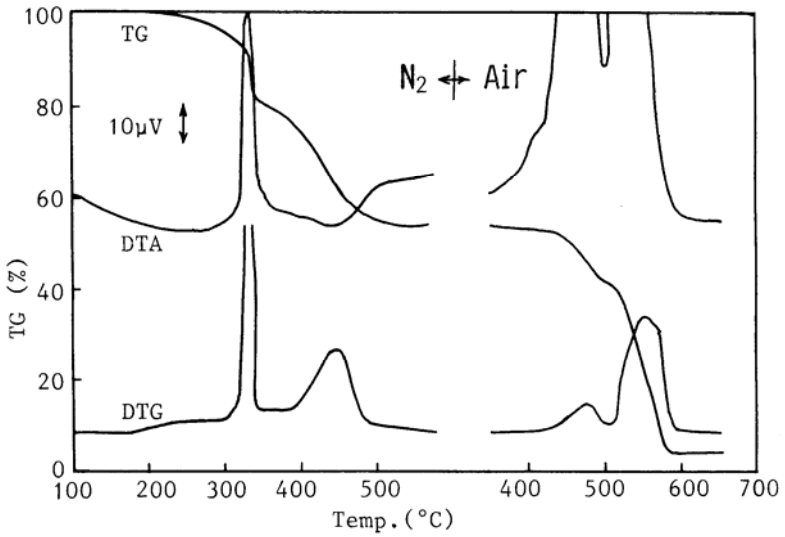

Figure 4 TG/DTA curve of SBR-CR Mixed Rubber
Sample weight : 15mg
Heating rate : 10℃/min
Atmosphere : N_2(R.T.～550℃), 250ml/min
Air(350～650℃), 250ml/min