MVR (Mooney　Viscometer) vs FDR (Flat Die Rheometr)

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1. History

Natural rubber contains a very large molecular weight component (Gel) which makes it unsuitable for processing in its original form. Strong shearing forces are applied using a Banbury Mixer, Kneader, or Open mill to break down the gel and reduce its molecular weight. This operation is called mastication. A plastometer is used to express the degree of mastication, the most representative being the “Williams Plastometer”, which compresses rubber between two parallel plates. The Williams Plastometer is still used in the field of silicone rubber.

In 1934, Dr. Melvin Mooney of U.S. Rubber developed a rotating disc apparatus, which he named the “Viscometer”.

When World War II began, American tire companies were using natural rubber from Southeast Asia and synthetic rubber (SBR) from Germany, but both became difficult to obtain. The demand for rubber surged due to the war, leading the U.S. to research synthetic rubber made from petrochemicals and develop GR-S (SBR), GR-A (NBR), and Neoprene. The early synthetic rubbers were not only inadequate in quality performance but also often difficult to process with natural rubber equipment. At this time, Mooney Viscosity proved effective as a processability index, and the Mooney Viscometer began to be used not only for synthetic rubber but also for compounds (rubber compounds　mixed with carbon black and others).　 The Mooney Viscometer became an essential item for rubber companies.

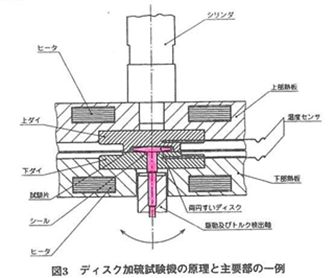
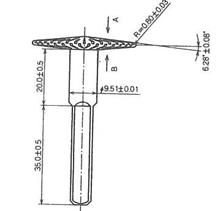
Synthetic rubber manufacturers' catalogs invariably list Mooney Viscosity, allowing compounders to refer to it when selecting rubber grades.

　　The Mooney Viscometer typically measures at 100°C for 5 minutes. At this temperature, rubber vulcanization does not occur, but when the temperature is raised to 125°C, vulcanization begins. The time taken to reach the start of vulcanization at 125°C is called the “scorch time”, and as one of the vulcanization characteristics, compounders place importance on scorch time in the same way as Mooney viscosity, as a guideline for processing without early vulcanization.

Before World War II, natural rubber was predominantly used, and the number of vulcanization accelerators was limited. However, with the emergence of various synthetic rubbers, the variety of accelerators increased, and as the formulation technology diversified, there arose a need for vulcanization testing machines to analyze the behavior of vulcanization.

The Mooney Viscometer has a disk-shaped rotor that rotates at 2 rpm within a sealed circular chamber (dies), detecting torque. By raising the temperature above 125°C to 150°C or 160°C, it should be possible to observe the situation of rubber vulcanization, but slipping occurs between the rotor and the rubber, making it impossible. It is also mechanically infeasible.

Therefore, devices specifically for vulcanization have been developed. The first generation consists of reciprocating vibration types like the “Vulkameter” and “Curometer” created in the 1950s, while the second generation is the “Oscillating Disk Rheometer” (ODR) which is a sealed torsional vibration type from the 1960s. The Mooney viscometer rotates the rotor in one direction, but it detects torque by applying a small angle (±1 deg or ±3 deg) of reciprocating torsional vibration to an umbrella-shaped rotor. The performance of vulcanization testing machines improved dramatically with the ODR, and Monsanto's “ODR100” model and similar products became widely popular. However, since heat dissipation occurs through the rotor's axis, temperature control was inadequate.

Rotor-type sealed torsional oscillation rheometer Oscillating Disk Rheometer (ODR)

The third generation, developed in the 1980s, is a rotor-less sealed torsional vibration type that solves the heat dissipation problem by applying reciprocating torsional vibrations to the dies without using a rotor. The pioneer was Monsanto's Moving Die Rheometer (MDR), which effectively became the standard for subsequent manufacturers. Additionally, it has been digitized, leading to the present day.

In Japan, during the 1960s, JSR (now ENEOS) developed a rotorless vulcanization testing machine "Curastometer," which was very widespread due to its low price, while the expensive MDR was only used by some synthetic rubber manufacturers and tire manufacturers. The Curastometer evolved into types II, III, V, W, and VII, but from type III onwards, it adopted parallel disc dies (flat dies), and FDR (Ueshima) and RLR (Toyo-Seiki) of the same specifications are also produced, meaning that there are currently three companies manufacturing them. Flat dies are mainstream in Japan, and domestic rubber companies possess vulcanization testing machines as essential equipment, regardless of whether they are tire manufacturers, industrial product manufacturers, large corporations, or small companies. However, the export to foreign countries is not very common, and in the European and North American markets, including Asia, Monsanto (now Alfa-Technologies)'s MDR or its imitations are used.

Among these, there are over 10 companies in China producing MDR imitations, which I’ve heard are priced around 10,000 US$, but I think their performance, quality, and durability are completely unreliable.

There are separate documents regarding the issues with the Moving Die Rheometer (MDR), but:

(1) The profile and dimensions of the dies are not published. There are many companies that create imitations, but there is no standard, making it unclear whether they are the same as the alfa-technologies MDR. It is obvious that if the profile and dimensions of the dies differ, the measurement results will not be the same. I have also heard that there are machines that imitate the initial MDR, such as those with different torsion angles.

(2) The bicornical dies of the MDR are thin in the center and thick at the edges, so the center cures quickly while the edges cure slowly. Additionally, while the MDR has heaters on the top and bottom, there are no heaters on the sides, which causes insufficient curing at the edges due to heat dissipation.

It is entirely strange for a device that measures the curing behavior to have non-uniform curing conditions.

(3)The dies of the MDR have radial grooves which do not prevent slippage.

There have been no visible efforts from alfa-technologies to improve these issues.

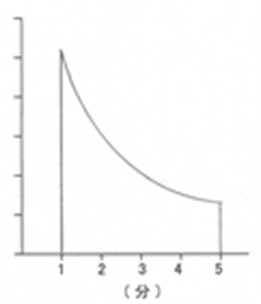
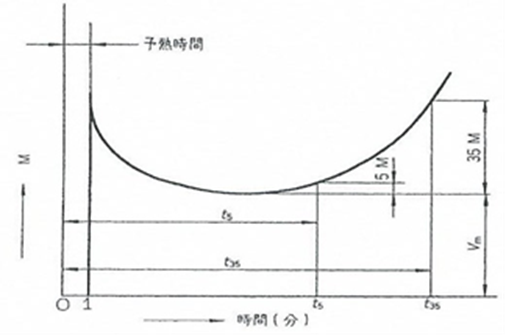
2. Purpose of MVR (Mooney Viscometer)

Two types of tests are conducted with the Mooney Viscometer.

Test 1: A non-vulcanized rubber sample is inserted and pressurized at a die temperature of 100°C. After preheating for 1 minute, the rotor is rotated, and the value is read after 4 minutes. This is generally referred to as the “Mooney Viscosity Test” and is denoted as ML1+4@100°C.

Test 2: A non-vulcanized rubber sample is inserted and pressurized at a die temperature of 125°C. After preheating for 1 minute, the rotor is rotated, and the test is concluded when the indicated value rises by 5 points (Vm + 5) after reaching the minimum value (Vm). The duration from when the sample is inserted and pressurized to the conclusion is referred to as “Mooney Scorch Time (t5 up time)”.

　 Mooney　Viscosity　Test　　　　　　　　Mooney　Scorch　Test

Test 1 is performed on all rubber compounds mixed with raw materials such as natural rubber, synthetic rubber, and carbon black, and it represents the hardness/softness of the rubber. High Mooney Viscosity natural rubber or synthetic rubber are hard rubbers, which also exhibit high Mooney Viscosity when compounded. Natural rubber and synthetic rubber with low Mooney Viscosity are soft rubbers, showing low Mooney Viscosity when compounded.

*Here, 'hard/soft' refers to the tactile hardness (toughness) during processing such as Mixing or Building, and is unrelated to the hardness of the vulcanized product (Durometer Hardness).*

Generally, natural rubber and synthetic rubber with high Mooney Viscosity are hard and take longer to process in a Banbury Mixer or Roll Mill, consuming more power. Conversely, natural rubber and synthetic rubber with low Mooney Viscosity are soft and easy to process.

In that case, it is not necessarily true that rubber with low Mooney Viscosity is superior. This is because the actual compounds that become rubber products contain various additives such as natural rubber, synthetic rubber, carbon black, softeners, plasticizers, mineral fillers, antioxidants, sulfur, accelerators, and cure activators, which also change the Mooney Viscosity. The tread rubber of large tires must be hard because strength and abrasion resistance are important. When carbon black is added to increase strength, the compound's Mooney Viscosity also increases, leading to poor processability in extrusion and building, so natural rubber and synthetic rubber with low Mooney Viscosity are chosen. Although rubber for the carcass of the same tire does not require abrasion resistance, it needs to be soft and flexible, so more softener is added. Adding more softener makes the compound soft, but if it becomes excessively soft, it can stick to machinery or deform during forming or building, which is why natural rubber and synthetic rubber with high Mooney Viscosity are used. Thus, depending on the product and the processing process, there is an optimal Mooney Viscosity, and the compounder designs the formulation while considering these factors.

The Mooney Viscometer is a testing device for processability.

1. Purpose of FDR (Flat Die Rheometer)

On the other hand, rheometers represented by FDR or MDR are devices used to measure vulcanization. Instead of using rotors, a reciprocating torsional vibration (±1 degree) is applied to one of the upper and lower dies, and the torque transmitted to the other die through the rubber is measured.

ダイアグラム, 設計図

AI 生成コンテンツは誤りを含む可能性があります。

The test temperature is conducted at the actual vulcanization temperature (usually 140 to 200 °C). The Mooney viscometer preheats for 1 minute, while the rheometer does not preheat. After inserting the sample and closing the dies under pressure, the motor is turned to start the test. Immediately after the start, vulcanization has not yet begun, and the rubber becomes soft due to heat, resulting in a decrease in torque. However, over time, the vulcanization reaction occurs due to sulfur, and as the rubber vulcanizes, it becomes hard, causing the transmitted torque to increase rapidly. When sulfur is consumed, the vulcanization rate slows down, the torque increase becomes gradual, and eventually, it reaches equilibrium where the torque levels off. The vulcanization curve obtained in this way has a shape unique to its formulation, so by conducting a rheometer test, if the curve is the same as usual, it is judged as a normal compound (OK), and if it is not the same, it can be determined as abnormal (NG).

Many of the uses of the rheometer are for mixed compound inspection.

Generally, the minimum torque (ML), maximum torque (MH), 10% curing time (tc10), and 90% curing time (tc90) are considered as check gates.

*ML represents the hardness or stiffness of uncured rubber, while MH represents the hardness or stiffness of cured rubber. The difference MH - ML (ME) is the increase in hardness due to curing. The time corresponding to 10% of ME is referred to as 'curing start time', and the time corresponding to 90% of ME is referred to as 'optimal curing time', which are denoted as tc10 and tc90, respectively.*

Another purpose of the FDR (Rheometer) is in the field of R&D. The FDR does not measure the chemical reactions of sulfur itself. Instead, it measures the visco-elastic changes caused by sulfur cross-linking. Specifically, it measures parameters such as M' (dynamic modulus), M'' (elastic modulus), M\* (complexed modulus), and tan δ, and graphs these results. This is a type of DMA. While conventional DMA measures cured rubber, it cannot measure uncured rubber; however, FDR can measure visco-elastic changes from the uncured state to the cured state, and even into the over-cured region, serving as a means to elucidate the mechanism of cross-linking.

1. Relationship between MVR (Mooney Viscometer) and FDR Cure-curve

The principles of MVR and FDR detect resistance as torque by applying a constant deformation to rubber. The difference lies in whether the deformation is due to the rotation of the rotor or the torsional vibration of the dies, but the results obtained are complex elasticity, which is a combination of dynamic elasticity and dynamic viscosity, indicating that the same properties are being observed. The blue circle on the Rheometer Graph represents the section where the rubber softens after the start of the test and reaches the minimum value (ML), which behaves the same as the Mooney Viscosity (ML1+4) measured by the Mooney Viscometer. The red circle on the Rheometer Graph indicates the section where the vulcanization of the rubber begins and the increase in elasticity starts (tc10), which has the same meaning as the Mooney Scorch Test measured by the Mooney Viscometer.

In other words, the Mooney Viscosity (ML1+4) and Mooney Scorch Time (t5 up time) measured by the Mooney Viscometer show the same changes as the initial parts of the FDR Cure curve, which are ML and tc10. In fact, it has been confirmed that there is a correlation between Mooney (ML1+4) and FDR(ML), as well as between Mooney scorch (t5 up time) and FDR(tc10). Some people argue that the Mooney Viscometer may not be necessary if FDR is available. In factories, like tire companies that consume large amounts of compound daily, it is not uncommon to conduct inspections in a short time without Mooney Viscometer Tests, only performing Rheometer Tests to determine OK/NG.

**So, is the Mooney Viscometer a relic of the past and a futile test?**

**The answer is “NO ! ”.**

While there are rubber companies that have discontinued the Mooney Viscometer and rely solely on rheometers, many rubber companies possess both rheometers and Mooney Viscometers and actually use both. The reason is that...

(1) The rheometer measures at temperatures between 140°C and 200°C, whereas the Mooney viscometer measures at either 100°C or 125°C. Processing in rubber factories often occurs at around 80°C to 120°C. For example, the extrusion temperature for tire tread rubber is around 80°C to 110°C, and the extrusion temperature for rubber hoses is also similar. Tire carcasses are coated with rubber on woven polyester fiber using a calender machine at temperatures between 70°C and 100°C. Small rubber parts like O-rings and diaphragms are produced using injection molding, where the flow characteristics of rubber at 100°C to 150°C are important. In this way, the Mooney viscometer is suitable for evaluating characteristics in processing before vulcanization, whereas the rheometer is a testing machine for evaluating characteristics during vulcanization, serving a different purpose.

(2) The rheometer has a high test temperature, so LM and tc(10) pass through quickly, resulting in low torque values. It is not suitable for detecting subtle differences in rubber. The Mooney viscometer can detect subtle differences that are difficult to identify with the rheometer. It can be understood by considering that we are magnifying the blue or red dot areas of the rheometer graph.

(3) Many rubber engineers know from experience the range of Mooney Viscosity that is suitable for their company's processing equipment, processing methods, and processing conditions. This knowledge is a technical know-how built through countless failures and trial and error, and because rubber engineers value it, they likely will not doubt the usefulness of the Mooney Viscometer, even if similar data can be obtained from a Rheometer.