In-mold crosslinking of rubber compounds using an IM12

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Besides its wide range of thermoplastic processing applications, the Xplore MC 15 HT high torque laboratory micro-compounder now also offers high-viscous rubber processing capabilities. Thanks to the small volume and high-shear intensity of the MC15HT, preparing rubber formulations is quick and cost-effective compared to conventional techniques.

In this application note, we aim to demonstrate the use of the MC15HT in rubber compounding and subsequent crosslinking using an IM 12 injection moulding machine.

Two types of EPDM recipes (low and high viscosity) with 50 phr carbon black, 3 phr sulfur, and other processing and vulcanization agents were compounded in the MC15HT at 100°C barrel temperature and 100 rpm screw speed for 3 min:

- EPDM-LV (25 MU – ML 1+4, 125°C)
- EPDM-HV (80 MU – ML 1+4, 125°C)

The rubber compound was collected with the transfer unit of the IM 12 and injection molded with an IM12 Xplore Injection molding machine to obtain tensile test specimens (Fig. 1) for subsequent tensile testing.

The mold temperature was set to 160°C and the melt temp to 100°C. The injection and holding air pressure was set at 5 bar. The cycle time was set at 4 min and 10 min to observe the effect of crosslinking time on the sample's mechanical properties.

One should note that the mould temperature of 160°C was determined from a previously performed MDR (moving to die rheometer) and differential scanning calorimeter (DSC) tests to ensure the crosslinking of the rubber sample.

It is essential to know that both low- and high-viscosity rubber compounds were successfully crosslinked using an IM12 under the given conditions. Table 1 shows that the curing time significantly affects the mechanical properties of EPDM vulcanizates. Tensile strength, modulus, and elongation at break of the cross-linked samples for 4 minutes are lower than those of the cross-linked samples for 10 minutes, regardless of the viscosity. This can be attributed to the low cross-linking density in samples that are cross-linked for a shorter time. The crosslinking reaction starts when the temperature of the compound reaches the crosslinking initiation temperature and spreads through the compound with the evolution of the temperature gradient. By the time, reactive species are transported to the reaction sites on the rubber chain by diffusion, and thus, the reaction continues. In other words, sufficient time is needed to achieve a high degree of crosslinking and crosslinking density.

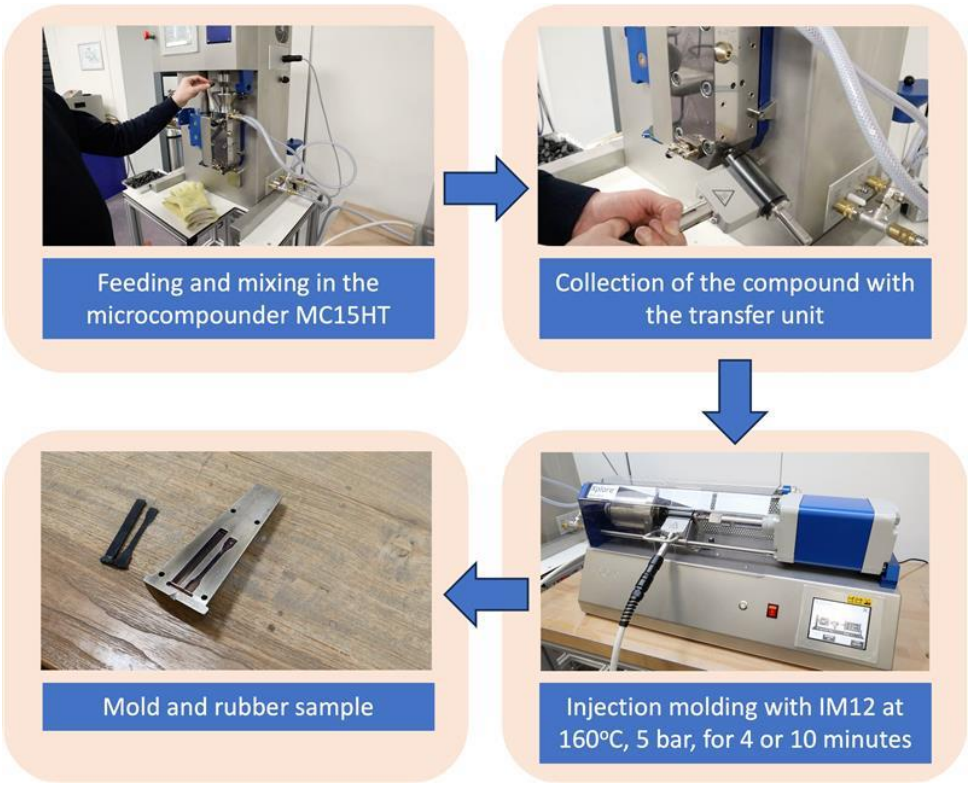

Figure 1. Injection molding using Xplore MC15HT and IM12

Rubber compounding and subsequent crosslinking can be performed with an Xplore MC15HT and an IM12 more effectively and quickly than conventional manufacturing methods like a two-roll mill Banbury combination. This availability enables the researchers to have multiple trials in a shorter time and with fewer raw materials.

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Table 1. Tensile test and hardness test results of compounds.

Compounds	Tensile Strength (MPa)	Elongation at Break (%)	Modulus at 100% elongation (MPa)	Shore D
EPDM-LV (4 min)	6.1 ± 1.2	402 ± 96	1.10 ± 0.08	48.0 ± 0.2
EPDM-LV (10 min)	10.0 ± 1.1	1161 ± 91	1.75 ± 0.07	60.0 ± 0.1
EPDM-HV (4 min)	7.4 ± 1.1	540 ± 101	1.04 ± 0.14	51.0 ± 0.2
EPDM-HV (10 min)	11.4 ± 0.9	1030 ± 71	1.62 ± 0.02	57.8 ± 0.5