

€ TUBALL™ MATRIX

PROCESSING GUIDELINES

for TUBALL™ MATRIX 618 beta, 620 beta

(for non-marking anti-static compounds, e.g. solid tires)

RECOMMENDATIONS ON THE USE OF TUBALL™ MATRIX

MIXING EQUIPMENT

Figure 1.

Internal mixer

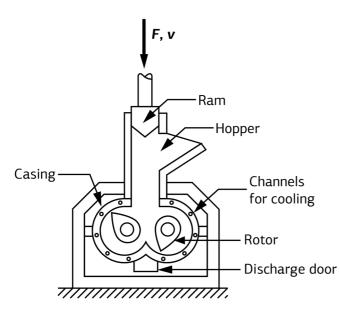
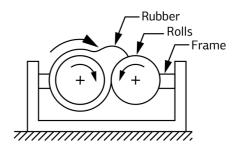


Figure 2.

2-roll mill



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DILUTION PRINCIPLES

Recommended dosage

A typical loading level to test is between 2 and 5 wt.% of TUBALL™ MATRIX.

Examples of calculating the concentration of TUBALL™ MATRIX in a rubber compound are shown below.

| Compound | Reference, phr | TUBALL™ MATRIX 618 beta, 3 wt.%, phr | TUBALL™ MATRIX 620 beta, 4.4 wt.%, phr | | | |
|-------------------------|-------------------|---|---|--|--|--|
| Natural rubber | 90 | 90 | 90 | | | |
| Butadiene rubber | 10 | 10 | 10 | | | |
| STAGE 1 | | | | | | |
| Precipitated silica | 50 | 50 | 50 | | | |
| Silane TESPT/Si 69 | 4 | 4 | 4 | | | |
| TiO ₂ | 2.75 | 2.75 | 2.75 | | | |
| Zinc oxide | 8 | 8 | 8 | | | |
| Stearic acid | 3 | 3 | 3 | | | |
| Wax | 1 | 1 | 1 | | | |
| PEG | 1 | 1 | 1 | | | |
| Anti-aging agent | 1 | 1 | 1 | | | |
| STAGE 2 | | | | | | |
| TUBALL™ MATRIX 618 beta | - | 5.45 | - | | | |
| TUBALL™ MATRIX 620 beta | - | - | 8 | | | |
| CTP | 0.2 | 0.2 | 0.2 | | | |

| NOBS | 1.25 | 1.25 | 1.25 | |
|--------|-------|--------|-------|--|
| DPG-80 | 1 | 1 | 1 | |
| Sulfur | 2.5 | 2.5 | 2.5 | |
| Total | 175.7 | 181.15 | 183.7 | |

It is recommended that laboratory tests be carried out to study the most efficient working concentration of TUBALLTM MATRIX for the particular compound to balance the properties, as it depends on the formulation type, raw materials and on the processing.

Introduction sequence

It is recommended to add TUBALL[™] MATRIX at the second/last mixing stage with curatives.

COMPOUNDING

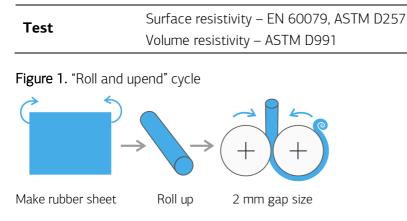
Add TUBALL™ MATRIX at the second mixing stage at the same time as curatives.

An example of a two-stage compounding process for internal mixer (<u>Banbury type, tangential</u> 1.6L) and 2-roll rubber mill (10-inch) is shown below.

Rotor speed of Banbury internal mixer is 50 rpm. Roll friction of 2-roll mill is 1:1.1, slow roll speed is 14.3 rpm. 2-roll mill is connected with cooling water. Roll surface temperature is close to room temperature.

NOTE! For other types of internal mixer (intermix type – intermeshing rotors, kneader) parameters such as rotor speed and mixing time should be adjusted accordingly.

| | Process step | Time, min |
|--------------|--|-----------|
| Plastication | Plasticize NR and BR on 2-roll mill, 2-3 mm gap size | 5 |
| Stage 1 | Add plasticated NR and BR into internal mixer | 1 |
| | Add ¾ parts of silica, zinc oxide, wax, PEG and stearic acid | 1.3 |
| | Add ¼ parts silica and Si 69. Keep 5 g of silica for further feeding of MATRIX at Stage 2 | 1 |
| | Mixing | 2 |
| | Raise the Ram and clean any powder from the sides, continue to mix, temperature <140°C | 2 |
| | Discharge from internal mixer | |
| | "Roll and upend" mix 6 times on 2-roll mill, 2 mm gap size | |
| | Make sheet and conditioning | ≥60 |
| Stage 2 | Add half of compound into internal mixer | |
| | Add TUBALL™ MATRIX, silica kept from Stage 1, curing agent, additives and the other half of the compound into internal mixer | |
| | Mix, temperature 90–120°C | 4 |
| | "Roll and upend" mix 7 times on 2-roll mill, 2 mm gap size | |
| | Make sheet (no narrow gap) | |
| Curing | Temperature 160°C (May vary based on formulation) | 8 |



MIXING AND CURING CONDITIONS

No special steps to control the mixing temperature and curing conditions are required with TUBALL[™] MATRIX. The nanotubes can resist much higher temperatures than polymers, so the usual steps to avoid polymer degradation are sufficient.

DETERMINATION OF PERFORMANCE WITH TUBALL™ MATRIX

Rubber properties can be determined according to the following international standards:

ASTM D 412 – Strength indices;

ASTM D 2240 - Shore A hardness;

ASTM D 395 – Compression set;

ASTM D 1646 – Mooney viscosity;

ASTM D 257, D 991, ISO 14309 - Electrical resistance;

ASTM D 5289 - Rheometric data;

ASTM D 5963 - Abrasion indices, etc.

If there are special requirements for the rubber, or other operational needs, other tests as defined by the user may need to be conducted.

ELECTRICAL RESISTIVITY MEASUREMENTS

It is recommended to follow international standards for measurement of electrical properties in the laboratory and for molded parts. Non-standard methods, accuracy of handheld devices, and the surface quality of samples can affect the data.

OCSiAl supports customers to obtain qualified electrical resistivity measurements. To perform the correct measurements, please refer to the <u>Electrical resistivity guidelines</u> on our YouTube channel. Alternatively, contact our regional offices and technical support centers to receive a hard copy of the guidelines or request measurements of your samples.

NOTE. Adjustment of plasticizer content in formulation

The total plasticizer content will be increased with TUBALL[™] MATRIX addition compared to the reference compound. Depending on the dosage of TUBALL[™] MATRIX, the plasticizer content may be adjusted in order to avoid a significant impact on viscosity and stiffness and to optimize the final properties.

Adjustment of pigment content in formulation to reach the required color

To make compounds colored it is required to use pigments. Graphene nanotubes have an inherent black color and TUBALL^M MATRIX will affect colors, making them darker. The extremely low effective dosage of TUBALL^M allows the use about 5–8 phr of color pigment (TiO₂, etc.) to produce an anti-static, non-marking colored compounds. The exact dosage of pigment and color depends on compound and pigment type and should be investigated experimentally for each particular formulation.

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