Reprocessing of polymeric materials for reintegration into moulded components

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HS Polymer Reprocessing Ltd is a small company that specialises in ‘de-vulcanising’ high-value rubber materials for incorporation back into virgin compound and thus new parts. It has been operating since 2006, with most of its customer base in Germany. Test results have shown that material properties are maintained within product specification. This process can be of particular benefit for high-value specialist elastomers.

It has long been considered that once a polymeric material has been cured or vulcanised into shape then it is not possible to transform it back into its original state. However, this is one of the rubber industry’s myths.

HS Polymer Reprocessing has developed a method for ‘de-vulcanising’ factory scrap into a form that is clean and easy to incorporate into a process at no extra cost and, once incorporated, shows little or no loss in the physical properties of the product. This feature introduces the recycling of high-value polymeric materials and hence the potential for cost savings.

Misconceptions

These can be listed as follows:

• **Perception:** There is a common perception that products incorporating recycled materials are somehow inferior.
• **Implementation costs:** There is a cost connected to keeping your factory scrap segregated and clean. There is also a cost in reincorporating the materials once reprocessed.
• **Headaches:** Product re-certification and the inevitable product failure because of the inconsistency of the reprocessed material.

Advantages

HS Polymer Reprocessing considers that the advantages of reprocessing high-value materials far outweigh the problems and myths listed above for the following reasons:

• **Value of investment:** A typical elastomer moulding process produces 10% or more of cross-linked rubber scrap in the form of mould flash and product off-cuts. This represents money that has been invested in materials and processing costs. Put simply, it is money that is being thrown away. Up to the point of manufacture this mould flash and off-cut has been carefully monitored and has passed all the same rigorous quality-control criteria as the final product. The only difference between the two is that the scrap material was in the wrong part of the mould at the final moment.
  • **Disposal costs:** Legislation is increasing the costs of disposal of these materials, and it would be foolish to assume that the costs will not continue to escalate.
  • **Raw material costs:** Most of the raw polymers that are used in high performance applications are linked in price to the petrochemical industry. We have seen the effects of the oil price in the polymer industry and increases are not likely to slow down in the future. At best a temporary levelling in pricing could be hoped for, but long-term future price rises should be anticipated.
  • **Green credentials:** This is not simply a nice thing to have. In time it will become a necessity.

How does it work?

The cross-linked material is placed under a controlled amount of strain and stress, focusing on the relatively inflexible cross-link within the polymer matrix rather than the carbon–carbon backbone of the polymer chain – a process that has been called “mechanical de-vulcanisation”.

An analogy would be a storm passing over a corn field. Once the storm has passed the corn is still standing, but the fence has been blown over. The process involves getting the ‘storm’ conditions right for each individual compound. Knocking down the fence without damaging the corn is a careful balance. Once the cross-link is broken the radical ends are simply capped off by air.

No chemicals or additives

The process does not use chemicals or additives. We distinguish between chemicals – those things that could be added to the compound to reverse, or alter, some of the chemistry within the compound – and additives, which serve a secondary purpose, for example, anti-tack dusting. Neither of these is used in the process.

Whatever is supplied for recycling is returned to the customer. Because of this the reprocessed material is effectively identical to the virgin material in chemical nature, and so product re-certification may not be essential.

Virgin material

The result of the process is that the material begins to flow again in the same way that the virgin, uncured, compound flows.

The product from the recycling process looks like a milled sheet, with no powders or dust or any other handling nightmares. It can be weighed out simply by either tearing a section of sheet by hand, or if necessary by cutting it with a knife or scissors.

Because everything is incorporated or encapsulated in the original polymer compound there is no need for extensive MSDS/COSHH paperwork – the material is clean in both handling and in paperwork.

Blended

Because the material flows like a polymeric material, which of course it is, incorporation with the virgin material is easy. Alternatively, it can be blended with other compatible materials. This can be carried out with ease in either a mixer or a mill, both of which are standard pieces of
equipment in the compounding plant where the material was originally made. The reprocessed material simply re-enters the process at this point and does not require any extra investment in equipment or, for that matter, additional processing time.

Quality control
Because the material supplied has been subject to the manufacturer's quality control through to the point of manufacture, the material provided to HS Polymer Reprocessing is consistent with that standard. It has all the correct ingredients, in the correct proportions, and has seen a consistent process throughout its history. At the point of curing, the majority of its physical properties are fixed and there will be little variation from this point up to when it is received for reprocessing in our plant. Once the material enters the process, careful monitoring and process control is employed. The heat history of the material throughout our process is controlled, and the material that is returned to the customer will be as consistent as the material supplied.

Effect on material properties
The experimental results presented here have been supplied by customers of this process, and the cooperation of these organisations in permitting publication is gratefully acknowledged. For obvious reasons the materials and customers must remain anonymous. These results must be used with some caution, but are a valuable indication of the potential results that can be obtained using this process. It is not suggested that any two materials will behave in the same way, or that the results shown will be in any way typical of what may be experienced with any other

<table>
<thead>
<tr>
<th>FKM Gasket Material – Compound A</th>
<th>% Reprocessed Material Added</th>
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<tbody>
<tr>
<td></td>
<td>0%</td>
</tr>
<tr>
<td>Rheology</td>
<td></td>
</tr>
<tr>
<td>ODR</td>
<td></td>
</tr>
<tr>
<td>6 min @ 180°C, 3°arc</td>
<td>16</td>
</tr>
<tr>
<td>Min Torque (in lbs)</td>
<td>135</td>
</tr>
<tr>
<td>Max Torque (in lbs)</td>
<td>59</td>
</tr>
<tr>
<td>ts4 (s)</td>
<td>132</td>
</tr>
<tr>
<td>tc90 (s)</td>
<td></td>
</tr>
<tr>
<td>Physical Properties</td>
<td></td>
</tr>
<tr>
<td>Press @ 8°C @ 165°C</td>
<td>82</td>
</tr>
<tr>
<td>post cure 24 hrs @ 230°C</td>
<td>22.1</td>
</tr>
<tr>
<td>Hardness (IRHD) Tensile Strength (MPa) Compression</td>
<td>1.89</td>
</tr>
<tr>
<td>Set 24 hrs @ 150°C</td>
<td></td>
</tr>
<tr>
<td>Specific Gravity</td>
<td></td>
</tr>
</tbody>
</table>

Table 1. The effects on both rheological and physical properties with the addition of different percentages of reprocessed material.

Figure 1. The change in rheological results with the addition of reprocessed material for FKM Material A.

Figure 2. A comparison of the change in rheological results with the addition of reprocessed material for FKM gasket compounds A and B.
compound, however, it is considered that some fundamental factors exist throughout. It is believed that the cure system has already been used up in the original moulding process and broken in the recycling, and therefore there is little risk of scorching the resulting blend. This is demonstrated in the rheological data for compound A, an FKM gasket material. In this material it can be seen that there is little change in the scorch characteristics, as measured by T5 or T95. In fact, some of our customers have found that it is necessary to modify the curative levels to compensate for the lost components from the reprocessed material. This compound 'tweaking' is something that can be anticipated and should not be too complex for an experienced formulator if the materials are understood.

**Material property data**

Table 1 shows material property data, both from processing rheology tests and the final physical properties, for an FKM gasket compound that includes up to 40% reprocessed material.

The property that shows the most noticeable shift is the minimum torque (M1), as measured on a rheometer, which is much greater than the corresponding maximum torque (M1H). This is shown graphically in Figure 1. It is believed that this is an effect of the loss of volatiles in the curing process that acted as process aids, and that the increase is therefore of less significance than might be expected, particularly as the increase is not reflected to the same degree in the M1H or, indeed, the hardness figures. This is supported by injection moulding trials where, despite the high M1, no problems were encountered.

**Physical properties**

Figure 3 shows the variation in physical properties of FKM compound A as the quantity of reprocessed material is increased. The shift in physical properties away from the virgin material never exceeded 20% even at levels of reincorporation above 20%.

**Table 2. The effects on physical properties with the addition of different percentages of reprocessed material to an FKM fuel-line material.**

<table>
<thead>
<tr>
<th>Physical Properties</th>
<th>Specification</th>
<th>% Reprocessed Material Added</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>0%</td>
</tr>
<tr>
<td>Hardness (Shore A)</td>
<td>49 – 55</td>
<td>52</td>
</tr>
<tr>
<td>Tensile Strength (N/mm²)</td>
<td>≥ 8</td>
<td>8.4</td>
</tr>
<tr>
<td>Elongation at Break (%)</td>
<td>≥ 300</td>
<td>458.0</td>
</tr>
<tr>
<td>Tear Resistance (N/mm)</td>
<td>≥ 4</td>
<td>7.4</td>
</tr>
<tr>
<td>Specific Gravity</td>
<td>1.88 – 1.92</td>
<td>1.91</td>
</tr>
</tbody>
</table>

Figure 2 illustrates the rheology data for a second FKM compound, Compound B. The same trend can be seen in this compound and this time the effects are much greater, but still predictable.

**Fluorosilicone**

Most of the graphs shown have been for FKM materials. Similar success has been achieved with other material types, including HNBR, fluorosilicone and perfluoroelastomer, but also the humble tyre crumb. However cost-effectiveness must still play an important part in material selection. Figure 5 illustrates some results obtained for reprocessing a fluorosilicone O-ring compound.

There is a potential for the reprocessed material to be used in a ‘secondary’ application, that is, not the application of the original material but one which will benefit from the material properties without the material cost. Here, it may be quite acceptable to use much higher percentages of reprocessed material than in the manufacture of the original parts.

**Logistics**

The view of this company is that it is important to recognise the value of ‘scrap’. If it is considered as a material that is still in the company process flow – to be reincorporated several steps back in the manufacturing process – then it would be foolish to regard this as recycling.

The reprocessing process does not take waste. The scrap material supplied should have a high intrinsic value. The highest possible amount of this material is then reprocessed.
Currently a loss of below 0.5% by weight is recorded in the return, as reprocessed material, and the loss is returned separately. When possible, the boxes and pallets that are used for the material delivery to the plant are used for the return journey. This enables the reprocessing to run a clean plant without the need of a waste management license. There are no stock materials requiring disposal from the reprocessing plant.

Customers of the process are encouraged to arrange their own transportation, both for delivery and collection. In this way the correct paperwork can be maintained for each shipment, to tally with an individual customer's internal requirements. This has been found to be mutually beneficial for both customers and the reprocessing plant. The processing plant is certified to BS EN ISO 9001:2008. These measures make it possible to keep the operating costs to a minimum by letting the customers control those parts of the process that are within their control. In this way the specialist experience of the moulding company and the reprocessing company are used most effectively to make the process as cost-effective as possible.

To take advantage of this process it is not necessary for HS Polymers to know the chemical make-up of the materials being processed or the logistics of the moulding company operations.

HS Polymer Reprocessing is currently looking to licence its technology in the USA and Far East, and is also looking for sales agents throughout Europe.

Conclusions

A process has been developed that can mechanically ‘de-vulcanise’ moulded elastomers to effectively convert them back to uncured material. Test results show that this recovered material can be incorporated into the moulding process with minimal effect on material properties—generally these will still be within the product specification.

As may be expected, the higher the value of a material the more cost-effective the process can be for the moulding company; but there is a minimum order volume below which it may not be worthwhile.

It is company policy to concentrate on the specialist task of reprocessing, and allow customers to control as much of the supply chain as possible in order to ensure an economic and viable operation. By mutually minimising costs with customers it is the company’s aim to grow as partners in a greener world.