We are the Elastomeric Compounding Specialists
FROM FORMULATION TO APPLICATION

CASE STUDY

Study by M. Winrow - R. Johnson - E. Buckley of Clwyd Compounders, UK.

Updated Silicone Aerospace Specifications

Issues for Compounders and Downstream Users

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Silicone compounds are heavily linked with several highly detailed specifications. This is because the aerospace industry has high levels of liability for the quality of the product and these quality requirements are second to none.

Compounders are currently left questioning the relevance of these silicone specifications that came into use several years ago. BS F 152 and 153 are the current specifications which replace DTD 5531 and 5582 which were made obsolete in 1999.

The new specifications appear to be a direct copy over of the obsolete specifications. They were originally developed for what we can now refer to as 'old technology polymer grades', as technology evolves we are offered newer technology grades that give much superior initial physical results.

The problem that we as compounders face, is that the specifications do not take the properties of the new grades into consideration.

Introduction

Silicone in Aerospace

Any material used in the aircraft industry has to be elite with no room for failure and they typically have highly detailed specifications to pass. Silicone rubber is often selected as a material of choice in this industry due to its excellent properties. They have characteristic properties of both organic and inorganic compounds and are able to overcome some disadvantages that organic polymers have. This is due to their siloxane backbone making it much more stable than typical organic backbones, as seen in figure 1. Silicone rubbers therefore have a higher chemical stability and superior electrical and thermal properties which are all essential in aerospace parts. The research we have done is based on heat curable silicones with a peroxide cure package.

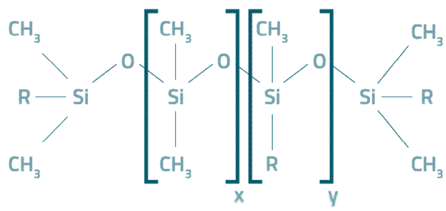

Figure 1 | Siloxane Backbone Structure

Obsolete Specifications Against Current Specifications

Silicone for aerospace specifications came into use over 40 years ago and were known as DTD 5531 and 5582. These specifications were geared around the polymer technology at the time. On April 1st 1999 these specifications were made obsolete and were replaced with new specifications BS F152 and BS F153. The strange thing we as compounders have noticed is that they are exactly the same as the old respective DTD specifications (as seen below), but the polymer technology has somewhat evolved and hence improved.

Property	Units	Spec requirement DTD 5531A Gd 60	Spec requirement DTD 5582A Gd 60
Hardness	IRHD	56 - 65	56 -65
Density	Mg / m³	documentation 0.05	documentation 0.05
Tensile strength	Mpa	5.5 min	4.8 min
Elongation at break	%	250 min	140 min
Tear strength	N/mm	30 min	25 min
Compression set	%	25 max	15 max
After heat ageing 336h @200 ° C			
Hardness change	IRHD	-3 to +7	-3 to +5
Change in TS	%	-20 max	-20 max
Change in E@B	%	-30 max	-30 max

Property	Units	Spec requirement BS F152:2006 Gd 60	Spec requirement BS F153:2006 Gd 60
Hardness	IRHD	56 - 65	56 -65
Density	Mg / m³	documentation 0.05	documentation 0.05
Tensile strength	MPa	5.5 min	4.8 min
Elongation at break	%	250 min	140 min
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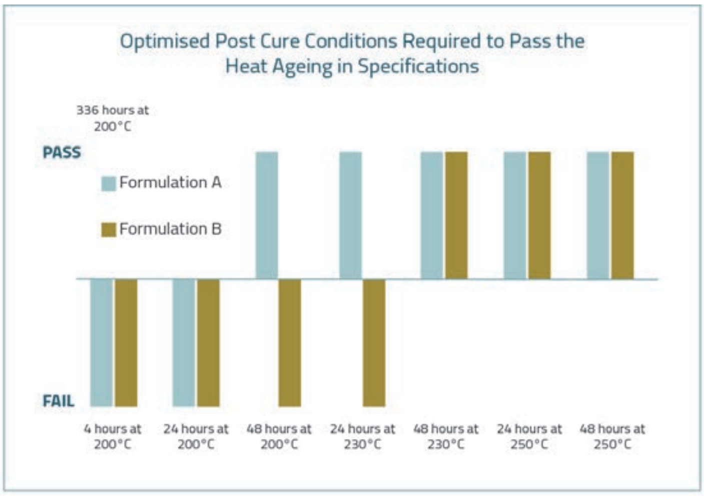
Research

Purpose

This research highlights the challenges that compounders like ourselves can be faced with when requested to formulate compounds to meet these specifications. In particular, the issues faced in meeting the heat ageing aspect of the specifications. Polymer technology has advanced greatly since the DTD specifications were created. Today we have access to polymers that have initial physical properties far superior to the ones 40 years ago. The problem with this is that as the specifications have the same drop off limits after ageing as the obsolete specifications, it means these superior initial physicals haven't been taken into account. Consequently, whilst compounders like ourselves can meet these specifications using older grades or grades with poorer initial results, we feel we are unable to take full advantage of these superior modern grades without having to excessively post cure the compound to lower these initial results. This research focuses on what compounders have to currently do to compounds to meet essentially out of date specifications.

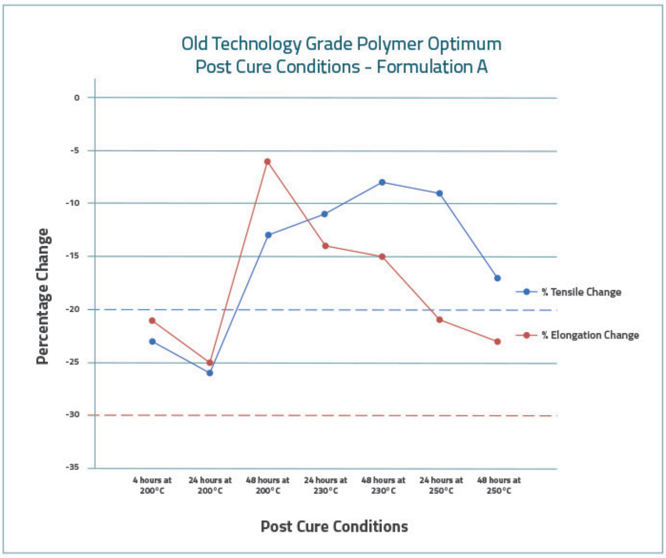
Analysis of 60 Grade Old Technology vs. Modern Technology

For this research, we looked at the 60 hardness range of polymers. We believe that the findings for other hardness grades of polymers would have had similar findings. Formulations were derived using two grades of polymer. The first based on an older technology—Formulation A (MF 960U) and the second based on a more modern grade—Formulation B (MF 660U). Both of the grades were chosen to have similar initial properties to make for a better comparison. These grades were chosen to show that we are still able to formulate using some of the modern grades but when we do, we have to essentially condition the polymer by optimising the post cure of the compounds. Different post cure conditions were tested on both formulations to find the optimum conditions for both grades of polymer and to analyse any differences between the older technology grades and the modern technology grades.

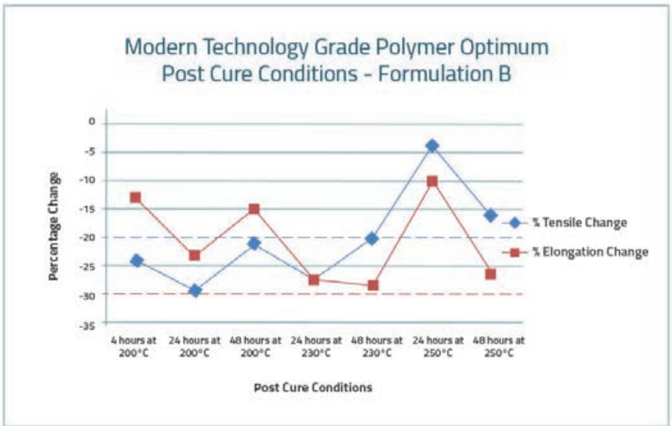


Graph 1

The graph above shows the effects of different post cure conditions on the two formulations. The graph documents at what post cure conditions the formulations will pass the 336 hours at 200°C heat ageing aspect of the specifications. We can see from the graph that formulation A using the older technology grade needs to have an initial post cure of at least 48 hours at 200°C. For an older technology grade polymer, MF 960U has respectfully high initial physical results. Due to this, these initial results have had to be lowered by using a slightly extended post cure in order for the values to remain within the acceptable drop off limit after heat ageing. Formulation B had to have even further extended post cure conditions as the modern technology grade has initial results even higher than the polymer in formulation A, even though this grade was chosen as it had the most comparable initial values to that of the older technology grade. A post cure minimum of 48 hours at 230°C is needed in order to sufficiently condition the initial values of the physical results, so that the drop off percentage after ageing would remain within the required limit.



Graph 2



Graph 3

Graph 2 and 3 show in more detail the effect that the different post cure conditions have on the tensile change and elongation change of old and modern technology grade polymers, such as in formulation A + B as shown above. It can be seen that the tensile strength change after heat ageing for formulation B is in this case the aspect which is harder to meet than the elongation at break change. It is important to note that the initial elongation at break of this polymer grade is not particularly high and as such doesn't cause a problem in this instance to meeting these specifications. In grades where the initial elongation at break and tensile values are high, a more extended post cure would be required to lower the initial values in order to meet the specification limits.

Other Available Grades

Having chosen a modern technology grade polymer with comparable initial physical results to an older technology grade polymer and highlighted the need to condition the polymer somewhat for it to meet the ageing specifications. It is important to show that there are many more modern technology grades available to compounders with even higher initial physical results. This is a significant development in polymer technology and poses vast advantages. However, it can also make it very difficult to achieve a formulation that meets these specifications using the modern technology grades of polymers that are readily available. For example, according to the datasheet for Elastosil R 401/60, it has an initial tensile strength of 11 MPa and elongation at break of 440%, as seen in the table below. These values are significantly greater than the old technology grades and some of the modern technology grades also, such as the ones used in formulations A and B.

The ability to use grades like these with the higher physical results would be highly beneficial to the overall properties of the compound. However using R401/60 as an example, with the initial tensile strength being higher at 11 MPa, it means that it would only need to lose 2.3 MPa for it to fail the ageing section of the specifications. Even when the tensile strength after ageing could be higher than some polymers' initial values. This highlights the oversight that has been made when transitioning from the DTD specifications to the BS F specifications. No consideration for these modern grades has been made and it poses the question as to whether it is time these specifications were modified to enable the use of these superior grades that compounders have access to.

	MF960U (Old Technology)	MF660U (Modern Technology)	R401/60 (Modern Technology)
Tensile Strength (MPa)	7	6.9	11
Elongation @ Break (%)	250	320	440

Conclusions

The results we have seen have shown that a lengthy and/or high temperature post cure prior to heat ageing is required in order for the specifications to be passed. We have noticed that due to the polymer grades based on the new technology having higher initial properties, the required post cure conditions have to be extended further. Whilst we are able to do this, it is highly unlikely that downstream users will alter their process to the same conditions. The problem we can therefore anticipate is that as more modern grades will be more prevalent in the future such as R401/60, we will be faced with the problem of formulating with them to utilise their higher physical properties. We envisage having to considerably condition the compounds prior to ageing. This is a huge shame for the industry as the improvement in properties of these grades should be welcomed as they could in turn give benefits to the end part. This leaves us questioning the relevance of the current specifications, as at the time that they were created, these modern technology grades wouldn't have been available. Is it time that these specifications were re-evaluated with these polymers in mind? This project has highlighted the importance for current and upcoming materials to be considered before the changing of specifications in the future.

Future Work

Rather than being able to draw a definitive conclusion from our findings, we have paved the way for future work and investigations to be carried out. It makes little sense to essentially lose the excellent properties of a polymer based on specifications created over 40 years ago. Would it make more sense to have an absolute minimum value for the tensile strength and elongation at break after heat ageing instead of the current percentage drop off limits? This would allow for example, a polymer with a high initial tensile strength and/or elongation at break to drop to values often higher than some polymers' initial results and still pass rather than under the current limits this could fail due to the percentage drop off being outside these limits. Something we have found from this work is that the results are often on the cusp of the specification limits and due to the required post cure conditions, they are often unreproducible. The heat ageing tests at 336 hours are in themselves a lengthy procedure so to have to optimise post cure conditions for compounds can take a considerable amount of time and hence increase lead times that we can offer customers. This has also presented us with the need to understand how to better stabilise these polymers with higher physical properties so that they remain within the current percentage drop off limits. This is something we feel needs much more investigation within the industry, and will become key if the specifications were to remain unchanged. Finally, we feel that this is a topic of high importance to both the rubber and aerospace industry and needs addressing promptly, especially as we don't know how long the older grades that meet the specifications more easily will be available for.

Download our research in full:
https://www.clwydcompounders.com/quality-technical-information/case-studies.asp