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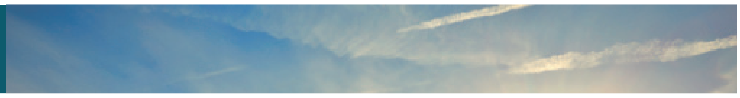

CASE STUDY

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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 F152 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. 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.

Property	Units	Spec Requriement DTD 5531A Gd 60	Spec Requriement DTD 5582A Gd 60	Spec Requriement BS F152: 2006 Gd 60	Spec Requirement BS F153: 2006 Gd 60
Hardness	IRHD	56-65	56-65	56-65	56-65
Tensile strength	Mpa	5.5 min	4.8 min	5.5 min	4.8 min
Elongation at break	%	250 min	140 min	250 min	140 min
Tear Strength	N/mm	15 min	12.5 min	15 min	10 min
Compression set	%	25 max	15 max	25 max	15 max
After Heat Ageing 336h @ 200°C					
Hardness Change	IRHD	-3 to +7	-3 to +5	-3 to +7	-3 to +5
Change in TS	%	-20 max	-20 max	-20 max	-20 max
Change in E@B	%	-30 max	-30 max	-30 max	-30 max

Figure 1: Comparison between DTD and BS F specifications.

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 (refer Figure 1), but the polymer technology has somewhat evolved and hence improved.

Purpose

This research highlights the challenges that compounders can face when requested to formulate a compound to meet these specifications. Today we have access to polymers that have far higher initial properties to the ones that was available 40 years ago. The aged properties of these modern materials exhibits a higher drop off in tensile properties, often exceeding the limits set by the specification. Consequently, whilst compounders like us can meet these specifications using older grades or grades with poorer initial results, we feel we are unable to take full advantage of these modern grades without excessive post cure. This research focuses on what compounders have to currently do, to meet the essentially out of date specifications.

Analysis of 60 Grade Old Technology vs. Modern Technology

In this investigation, we have selected 60 hard grade polymers of the older and modern technology. The first —Formulation A (MF 960U) which is an older grade polymer and the second based on a more modern grade— Formulation B (MF 660U). Both the grades were chosen to have similar initial properties to make a good comparison. The compounds were subject to different post cure conditions as given below and were tested against the specification to analyse the results. Further the polymers were analysed under a modern MFR (Multi-Function Rheometer) to understand the polymer chemistry.

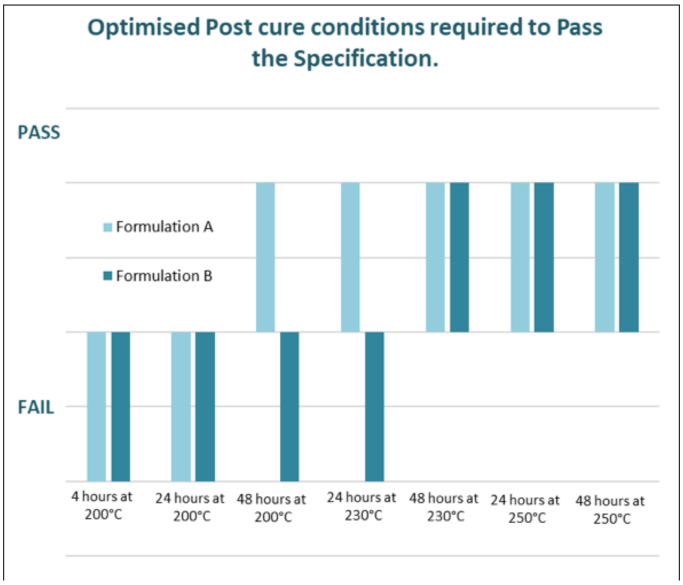
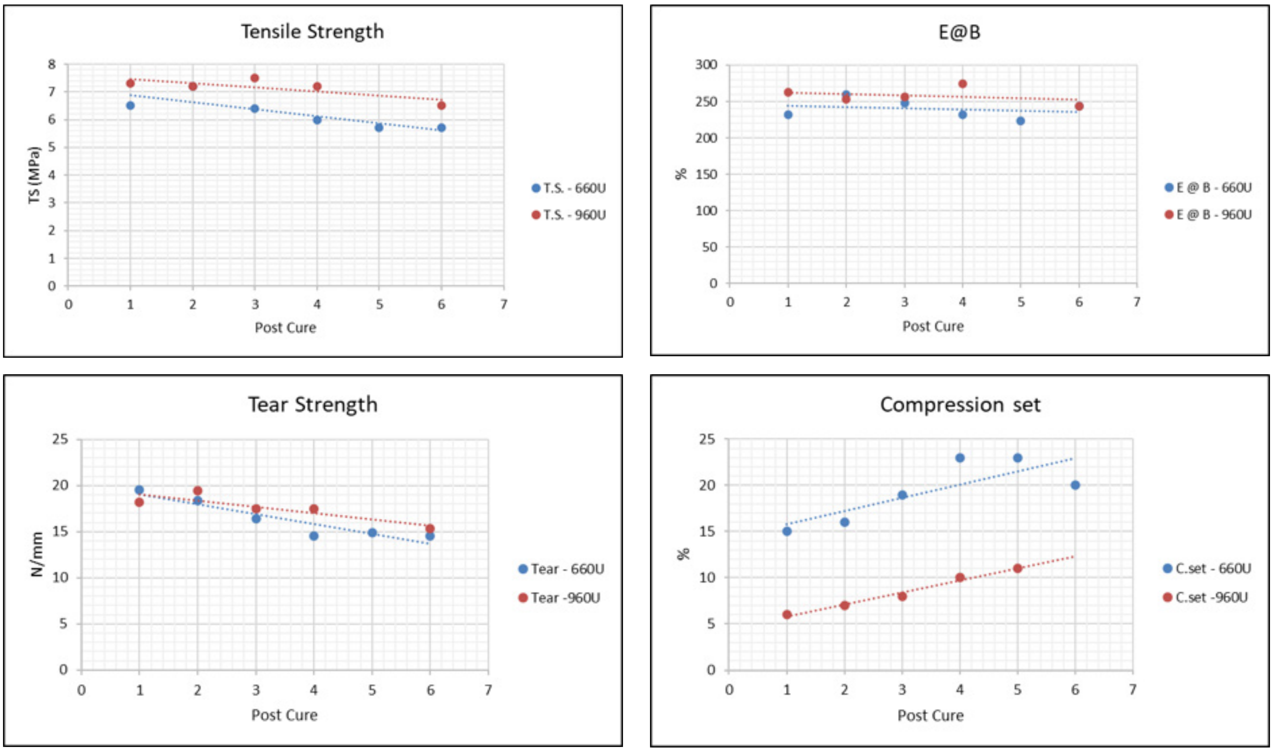


Figure 2: Post cure conditions for the compounds against specification.

Figure 2 shows the effects of different post cure conditions on the two formulations. It describes the condition at which the formulations satisfies the 336 hours (at 200°C) aged test of the specification. It can be observed that formulation A using the older technology grade satisfies the spec at a post cure of 48 hours at 200°C. As observed from figure 3, the older technology grade polymer - MF 960U has respectfully high initial physical results and superior compression set. Modern polymer MF 660U on that other hand is found to stabilise after a post cure of 48h at 230°C.



Post cure condition	1	2	3	4	5	6
	24h@ 200°	48h@ 250°	24h@ 230°	48h@ 230°	24h@ 250°	48h@ 250°

Figure 3: Physical properties of the compounds upon aging.

Aged tensile properties of the compounds against 336 hrs as described in Figure 4 & 5, indicate that MF 960U is found to perform better by passing the specification at a less severe post cure condition of 48hrs at 200°C. Whereas the modern polymer MF 660U has to undergo a more severe condition of 48h at 230°C to satisfy the specification. The aged properties also indicate a stabilisation of the properties at increasing post cure conditions indicative of an artificial aging of the polymers.
Figure 4 & 5 show in detail the effect of longer post cure conditions on the tensile change and elongation change of the two grade polymers. It can be seen that the tensile strength change after heat ageing for 660U is in this case the aspect which is harder to meet than the elongation at break change.

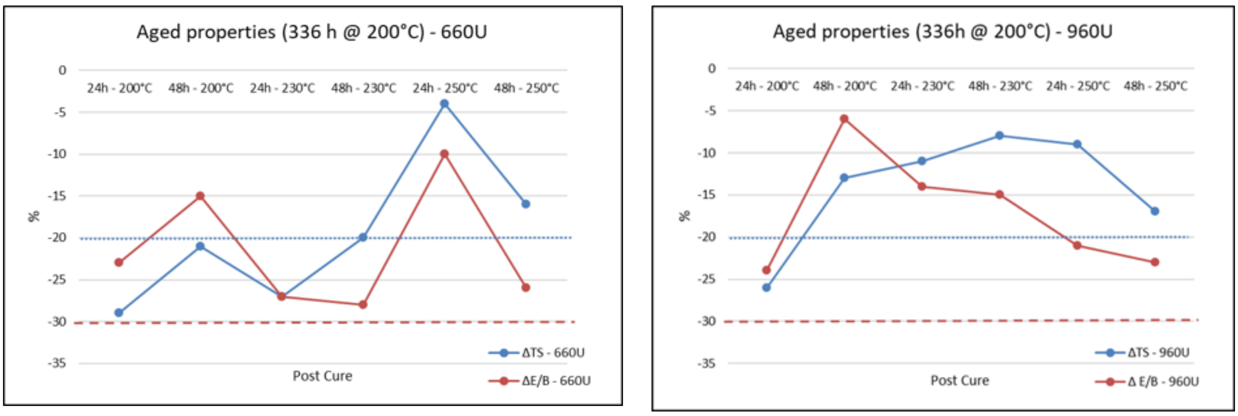


Figure 4 & 5: Percentage change in TS and EB on aging.

Other Available Grades

Having chosen a modern technology grade polymer with comparable initial physical results to an older technology grade polymer, It is also important to evaluate other modern grades available to compounders with even higher tensile properties. We have evaluated Elastosil R401/60 S against the specification and have observed that the initial tensile properties of the grade to be significantly higher than the other grades. More interestingly it was also observed as described in Figure 6 & 7, that the final Tensile strength of R401/60 S after aging was similar to the initial pre aging values from MF grades. Although this does not pass the BS F specification as the % change is observed to be -24% against the set limit of -20%, the final absolute value is found to be much higher. Similar holds true for elongation at break.

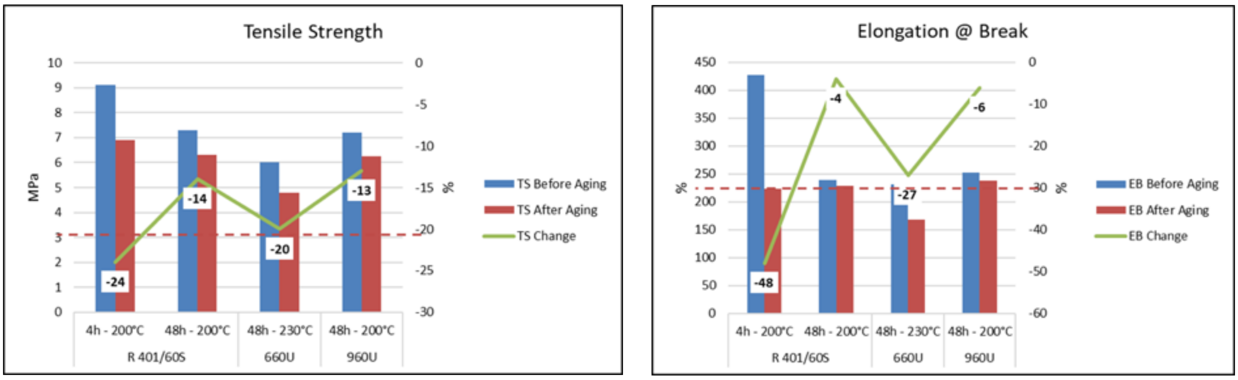


Figure 6 & 7: Change in TS and EB for R401/60 S, MF 660U & MF 960U.

The ability to use grades like these with higher physical results would be highly beneficial to the overall properties of the compound, but the % change in tensile properties set by the 40 year old specification often limits its application for this specification. This highlights the oversight that has been made when transitioning from the DTD specifications to the BS F specifications. There has been little consideration given to these modern grades due to its unavailability at the time of the specification's drafting and it poses the need for these specifications to be reviewed, to enable the use of these superior grades that the industry now has access to.

MFR Analysis

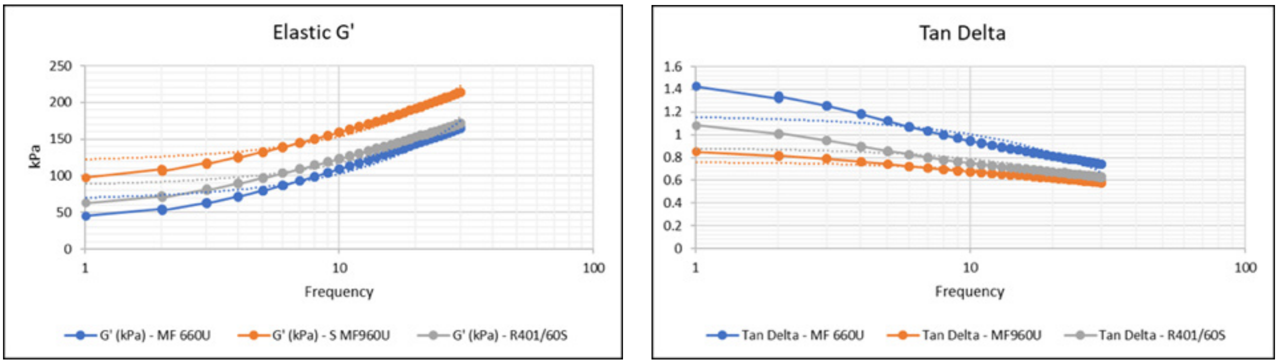


Figure 8 & 9: Elastic modulus G' and Tan D from Frequency sweep.

To understand these observations, the raw polymers were subject to a frequency sweep and a strain sweep under a modern Multi-Function Rheometer. A strain sweep from 0-200% was done at 0.5 Hz frequency and 100°C while a frequency sweep was done from 0-30 Hz at 10% strain and 100°C. From figures 8 & 9, the higher elastic modulus G' of MF 960U at low frequency throws light on the higher entanglements of the polymers, which can be correlated to the higher tear strength and the higher molecular weight as also observed from its viscous modulus. MF660U is found to have the highest change in tan delta, followed by R401/ 60S and 960U, indicative of its molecular weight distribution in the same order respectively.

Figures 10 & 11 from the strain sweep, show a rapid decrease of the elastic modulus (G') of 960U followed by R401/60S and then 660U which indicated the ability of the entanglements in the polymer to disentangle at high strain. This is also showing the stability of the polymers and can be correlated to the heat aging results observed earlier for these materials. At higher strain, the entanglements pull apart and the elastic reaction is lowered, here we even observe the loss of the reaction at higher strains.

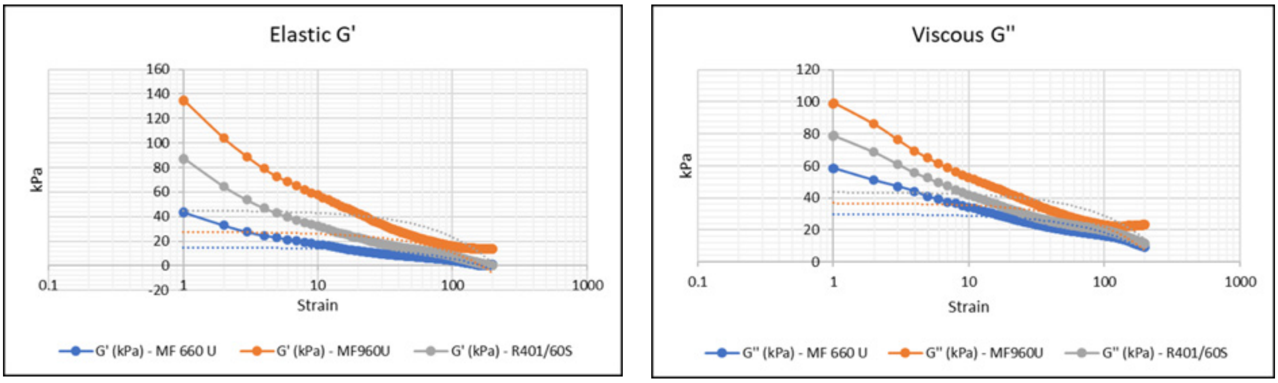


Figure 10 & 11: Elastic modulus (G') and Viscous modulus(G'') from strain sweep.

Conclusions

The results we have observed indicate of the need for a lengthy and high temperature post cure prior to heat ageing to satisfy the limits of the specification. We have also observed that the modern technology polymers have higher initial properties and thus due to the molecular nature of the material as analysed from the MFR, it would require a more severe post cure condition to satisfy the aged tensile limits. Although this technique can be utilised, it is highly unlikely that the downstream users will alter their processes to the same conditions. The problem we therefore anticipate is that, as more modern grades like R401/ 60S will become prevalent in the future, there will be a limitation of utilising them for their superior physical properties. Thus, there would be a need to considerably condition these compounds prior to ageing, which would affect the productivity at the customer's end. This limits the industry to utilise these materials, as the improvement in the properties of these grades hamper the application on the end part, leaving us to question the relevance of the current specifications. Is it time that these specifications are re-evaluated with the new polymers in mind? This project has highlighted the importance for current and upcoming materials to be considered and of the need to reconsider the specification and its relevance, for being future ready to new materials and manufacture superior products for the industry.

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