

Characterization of Extent of Cure of Form-in-Place Compounds

Hui Ye, Claudine Lumibao, Douglas S. McBain
Laird Technologies
4707 Detroit Avenue
Cleveland, Ohio, 44102
USA

216-939-2300 (Phone), 216-939-2310 (Fax), Hui.Ye@lairdtech.com

Abstract

Form-In-Place (FIP) gasketing is particularly well suited to provide effective EMI shielding for wireless handsets, PDAs, PC cards, telecom base stations, and many other compartmentalized electronic assemblies. This method of gasketing provides effective cost savings in the form of raw materials, labor and assembly time. The elastomeric compounds can be engineered to have advanced electrical and/or thermal performance in addition to environmental sealing. The performance of FIP gaskets is highly dependent on the cure rate and cure state of the silicone-based matrix. However, it is sometimes difficult to use traditional methods to characterize the extent of cure of such compounds due to application differences and cure chemistry. In this study, we present a simple and practical method to characterize the extent of cure of FIP compounds. The effect of temperature, humidity and time on the extent of cure has been extensively studied with this method using high performance electrically conductive FIP electrical conductive elastomer gaskets as an example.

Keywords: Form-in-place Gasketing, Electrically conductive Elastomer, cure rate test, vulcanization.

1. Introduction

Form-in-Place (FIP) gasketing is an automated system for dispensing conductive elastomer EMI shielding, grounding, and environmental gaskets onto metal or plastic substrates. FIP gasketing is particularly well suited for wireless handsets, PDAs, PC cards, telecom base stations, and many other compartmentalized electronic assemblies. This method of gasketing provides significant cost savings in the form of raw materials, labor and assembly time. The elastomeric compounds can be engineered to have advanced electrical and/or thermal performance in addition to environmental sealing.

The elastomeric FIP compounds are generally made from un-cured polymer filled with conductive filler, commonly referred to as "paste". The paste is dispensed directly on the metal fixture in the required bead shape and position using robotic equipment. The bead becomes an elastomeric gasket after cure with humidity or heat. Figure 1 demonstrates an electrical device substrate with FIP gasket applied. The two most popular elastomer

materials used for FIP are moisture-cure silicone (RTV) and heat-cure silicone (HTV). RTV silicone compounds slowly cure in atmospheric moisture to a soft silicone elastomer, usually with the release of a low-molecular hydrolysis product, while HTV silicone compounds cure quickly at higher temperatures to a relatively high mechanical strength without the release of low molecular weight by-products [1].



Fig. 1. Demonstration of FIP Gasket technology

The performance of FIP gaskets is highly dependent on the cure rate and cure status of the silicone compounds. However, it is difficult to use traditional methods to characterize the extent of cure of such compounds due to the

specifics of application and cure chemistry, especially for the FIP based on moisture-cure elastomers. Common methods, such as an oscillating disk rheometer or rotorless curemeter work well for the characterization of bulk elastomers. However, for FIP gasket applications, RTV compounds are dispensed as a bead and can only cure from outside to inside and from top to bottom. The rate of RTV cure decreases with increasing depth, leading to less homogenous networks. For HTV compounds, during the initial stages of cure the metal substrate functions as a heat sink, delaying cure and similarly leading to non-homogeneous networks. Physical properties thus vary considerably depending on the cure process and the extent of cure. Cure can be characterized by determining properties based on physical testing, but standard methods such as an ASTM-type of tensile test, are impractical to apply and may not correspond well to the real-world cure of the FIP beads.

In this study, we present a simple and practical method to characterize the extent of cure of FIP compounds. The effect of temperature, humidity and time on the extent of cure has been extensively studied with this method using high performance FIP electrical conductive elastomer gaskets as an example.

2. Experimental

2.1 Sample preparation

The potential substrates with which FIP gaskets can be utilized are many; however, the cross-section shape of different FIP gaskets is in practice limited to several common types. The "D-shape" cross-section is the most common type of FIP bead. Figure 2 shows the FIP sample beads for this study, which vary only in size for many applications, as shown in Figure 1. Multiple straight-line beads are dispensed on an aluminum platen. The bead size is about 1.2mm in width x 1.0mm in height.

To get comparable data, all RTV samples must have a similar bead size, and are cured in a controlled chamber to achieve reproducible conditions of temperature and humidity. For this study, 25°C and 50% RH are standard except where specifically mentioned. Testing was conducted in the lab with a temperature of 22°C and relative humidity around 50% RH. Three different kinds of FIP compounds have been used for comparison, namely RTV-1, RTV-2,

and RTV-3, which represent different fillers and silicone-base compositions. For the compression set test and adhesion tests, a different fixture has been used but the bead cross-section size is close to 1.2mm x 1.0 mm with the standard D-shape.

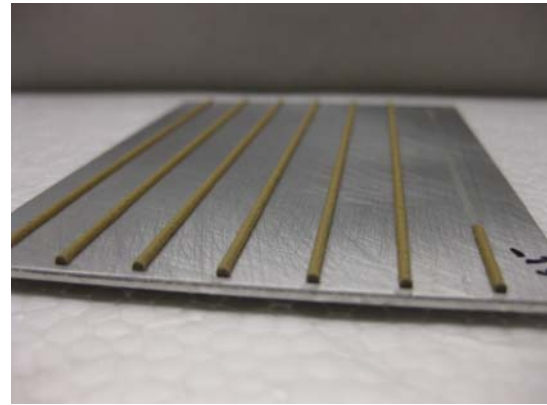


Figure 2. FIP sample beads on the Aluminum platen

2.2 Testing

Three parameters have been used to characterize the extent of cure of FIP compounds, i.e., tack-free time, handling time and cure time.

A **touch test** is used to get tack-free time of the FIP beads. After dispensing is done, touch the FIP bead with a clean, dry finger every 5 minutes. When the FIP strip is no longer tacky, the time elapsed since dispensing is called the **tack-free time**. A short tack-free time reflects a fast initial cure but also means limited time to modify the bead if necessary.

A **denting test** is used to get the handling time. A special denting fixture has been designed to check the handling time. Once the FIP gasket has become tack-free, the fixture is used to dent the FIP gasket bead every 15 minutes. An estimate of the depth of the dent in the FIP bead relative bead height is made, and recorded along with time as the depth percentage. The time between dispensing and the time when the dent depth percentage is around 10% is defined herein as the **handling time**. At this stage assemblies can be moved and minor contact will not damage the bead; however, the physical properties of the FIP beads are not developed sufficiently for compression or end-use application. Figure 3 shows that the head of the denting fixture is pressed on the FIP bead. There are three apparent dents in the most front bead; the dent

depth percentages are approximately 50%, 100%, and 30%, respectively.

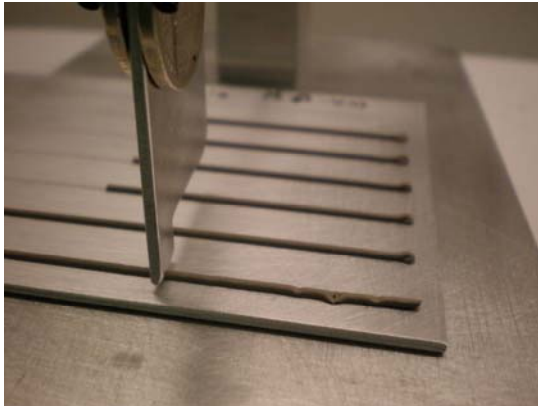


Figure 3. Demonstration of denting the FIP beads using the denting fixture

A simple **tensile test** is used to evaluate the cure state of the FIP bead. Using a common force gauge, a caliper and special clips, a test stand is used to obtain the maximum tensile force and elongation at failure of the FIP beads. Figure 4 depicts the simple tensile test platform.

After a specific time, a blade or knife is used to peel off one piece of FIP bead from the aluminum platen. The FIP bead is cut to 40 mm in length, and the bead carefully fastened between two binder clips. The tail loop of the top clip is connected to the force gauge. The tail loop of the bottom clip is fixed to the bolt on the base of the platform. Stretching the FIP bead until it breaks allows a measurement of the tensile force at breaking. The caliper connected to the force gauge can be used to record the extension length of the FIP bead at break. Two FIP beads are tested for each condition and the average value calculated. If the beads break dissimilarly, more beads will be tested. The test is repeated every 30 minutes after handling time is achieved. After six hours from after, the test may be discontinued or done at 12hrs, 24hrs, and 48hrs or longer. The width and height of the cured FIP bead is measured using an optical instrument and the dimensions used to calculate the area of the initial bead cross-section. A simple tensile strength is obtained by dividing bead breaking force with the cross-sectional area.

To evaluate the feasibility of using this simple tensile test to characterize the extent of cure of FIP compounds, two important properties of FIP compounds, i.e., compression set and adhesion strength have been measured for comparison. Compression set vs. time and

Adhesion strength vs. time has been measured for the FIP beads after same condition of curing for tensile test. The compression set test has been conducted at 85°C for 22hrs following ASTM D395 [2]. Adhesion strength was measured using an internal Laird Technologies test method.



Figure 4. The simple tensile test platform

3 Results and Discussion

3.1 Tack-free Time

RTV-1, RTV-2, and RTV-3 show similar tack-free times of approximately 10-15 minutes. The measurement of tack-free time is somewhat subjective, but different individuals typically report values within a 5 minute range when evaluating the same fast-cure FIP compounds.

3.2 Handling Time

The dent test for FIP cure indicates that the penetration depth of the fixture into the bead will be less than 10% after 1 hour for RTV-1 and RTV-2, and for RTV-3 to develop the same strength requires approximately 1.5 hours. Adhesion of the beads of the three materials to an aluminum substrate begins to develop in a similar time frame. Accordingly, the handling time for RTV-1 and RTV-2 is one hour, but RTV-3 requires 1.5 hours.

3.3 Simple tensile forces vs. curing time

Fig. 5 shows the simple tensile force of FIP beads vs. cure time. As seen in Fig. 5, the simple tensile force required for failure of an FIP bead during extension has been shown to increase with time. Tensile force of FIP beads increases rapidly at first and then levels off. These plots are very similar to the torque vs. time plots (after scorching time) obtained with an oscillating disk rheometer. It is reasonable that both the tensile strength and torque reflect the extent of elastomer cure. The tensile test described herein is easily performed and the test sample size can be identical to a real application and cured under any application specific conditions.

The tensile force for bead failure of both RTV-1 and RTV-2 achieves a nearly constant value within 24 hours. The comparable tensile force for RTV-3 continues to increase over a period of one week. Both RTV-1 and RTV-2 reach about 90% maximum tensile strength after 4 hours and reach about 95% maximum tensile force within a 48 hour period. However, RTV-3 takes 48 hours to reach about 90% maximum tensile force and requires more than 72 hours to reach 95% maximum tensile force. The optimum properties of an FIP will be developed when the tensile strength reaches around 95% of its maximum as indicated from compression set and adhesion below.

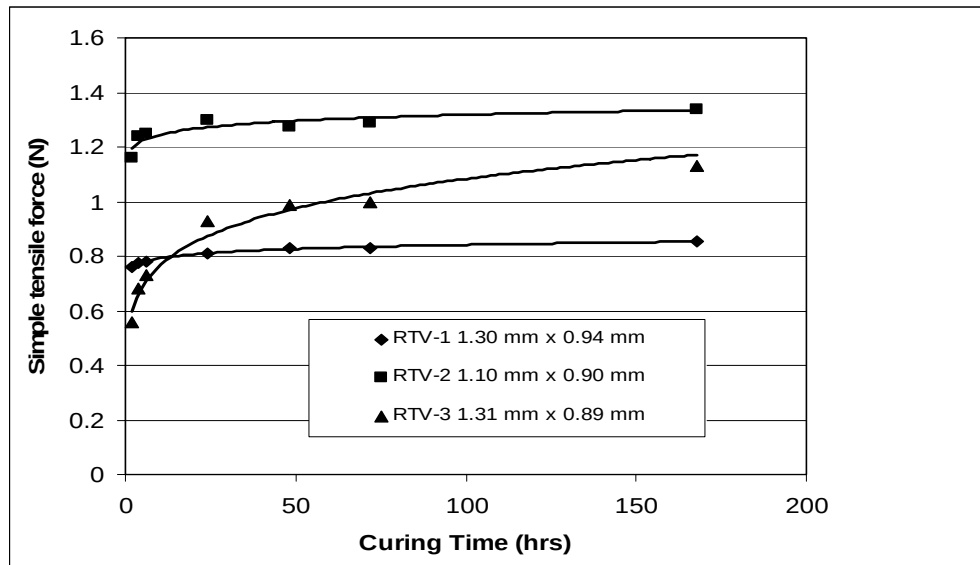


Figure 5. Tensile Force of FIP beads vs. Cure Time

3.4 Elongation vs. curing time

Figure 6 shows the elongation vs. curing time of different FIP compounds. There is a similar trend of the elongation rate vs. time but with a very large variation. There is also a large variation among the elongation data of different specimens for the same compound and after the same controlled curing condition. In contrast, the difference on tensile strength of different specimens is not significant, less than 5%. This

reflects that the elongation of FIP beads is sensitive to other factors besides the curing time. Elongation vs. time may be not a good characteristic to evaluate the cure rate and cure state. As shown in Fig. 6, the tensile elongation of beads of the RTV-1 and RTV-2 materials increases rapidly with cure time at beginning, followed by a decrease with further curing to a nearly constant value, which is common of rubber materials. The decrease of elongation of RTV-3 may be a result of experimental error.

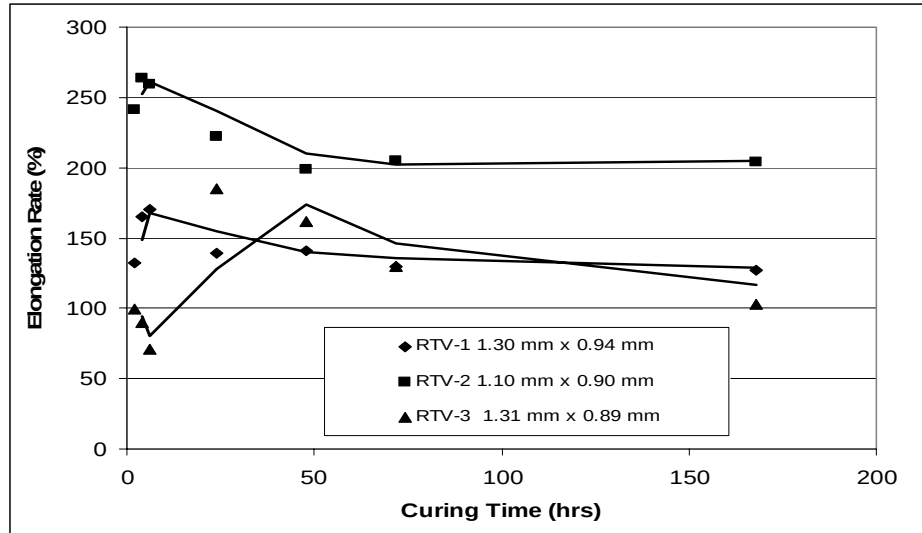


Figure 6. Elongation of FIP beads vs. Cure Time

3.5 Compression set vs. curing time

Compression set is defined as the tendency of soft materials, such as elastomers, to undergo permanent deformation, as well as the tendency of such materials to lose their ability to recover in a completely elastic manner [2]. FIP compounds show high compression set if not cured well because cross-linking continues under compression, and such structure will not rebound after the compression force is removed.

The compression set of FIP beads was determined for beads aged uncompressed for a period of time, followed by compression to 25% and aging at 85°C for 22 hours. Figure 7 shows the compression set vs. time of different FIP compounds. As shown in Figure 7, the

compression set of all FIP compounds decrease quickly at the beginning and then levels off. This is reasonable because better developed cross-linking structure of the elastomer leads to lower compression set. The compression set of RTV-1 and RTV-2 achieves a nearly constant value within 48 hours, which means the elastomeric structure of FIP compounds has been well developed. A comparable bead of RTV-3 also shows decreasing compression set with increasing cure time, but the value is still declining after one week (168 hours). Comparing Fig. 7 to Fig. 5, the trends are consistent, and of the three compositions RTV-3 cures the slowest.

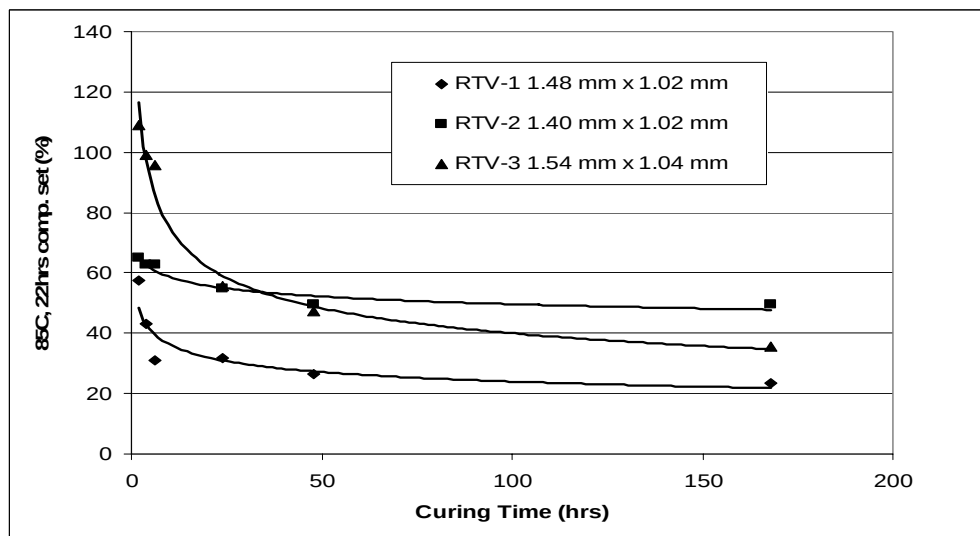


Figure 7. Compression Set of FIP beads vs. Cure Time

3.6 Adhesion strength vs. curing time

Fig. 8 shows the adhesion strength vs. cure time of different FIP compounds. The adhesion strength of both RTV-1 and RTV-2 increases with cure time and achieves a nearly constant level within 48 hours, as shown in Figure 8. The adhesion strength of RTV-3 also increases with cure time, with rapid development within 48 hours and a slow increase over the one week period. The trend of adhesion strength vs. time is the same as the tensile strength vs. time shown above. Moreover, the pattern of Fig. 8 is very close to those of Fig. 5, indicating the feasibility of using simple tensile test to

characterize the extent of cure of FIP compounds. During the adhesion test, a tear force is applied on the FIP beads parallel to the bottom face. The length of contact area is up to 10mm. High adhesion strength reflects not only higher tear strength but also higher tensile strength of the FIP compounds since most FIP bead failure results from cohesive failure. An exception is during the initial curing time when the bond between FIP bead and the substrate is still weak. Both mechanical properties can be used to evaluate the extent of the cure of FIP compounds. And the results of our simple tensile test can be used to predict the adhesion strength of FIP compounds too.

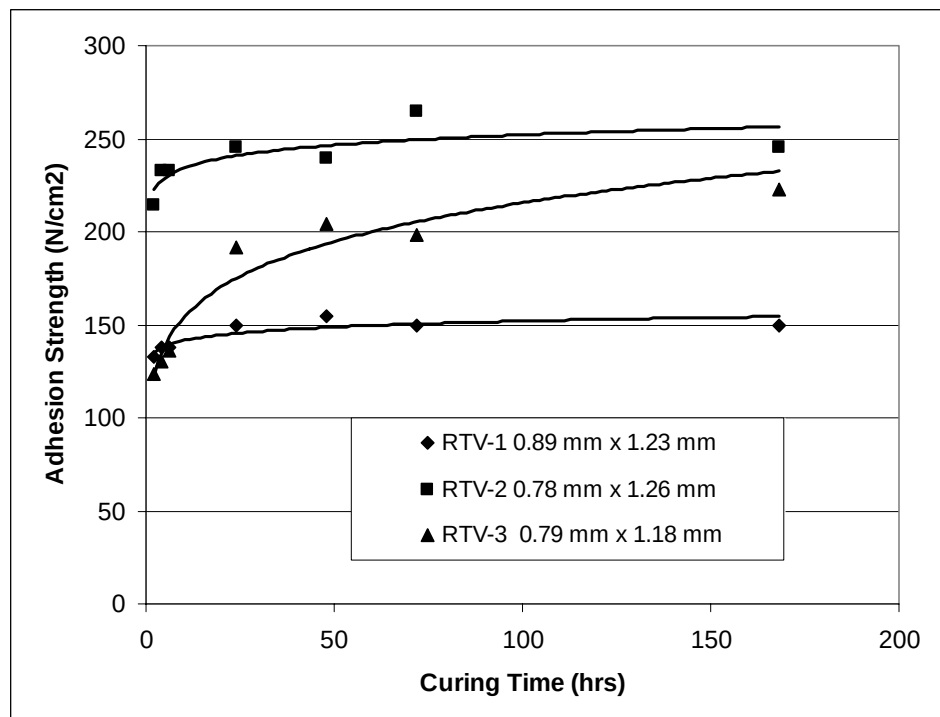


Figure 8. Adhesion strength of FIP beads vs. curing time

3.7 Effect of temperature and humidity on the extent of cure of moisture cure FIP compounds

Relative humidity will affect the cure rate of moisture-cure FIP compounds. High temperature may also facilitate the cure process of moisture-cure FIP compounds if there is a thermal activation reaction going on. It is desirable to understand the performance of an RTV system in order to best support applications and customers. Hereafter, three parameters, i.e., tack-free time, handling time and cure time will be used to quantitatively evaluate the effect of temperature and humidity on the extent of cure

of a typical FIP product, RTV-1. Two temperatures and three relative humidity conditions were selected for study.

Table 1 lists the tack-free time and handling time of RTV-1 under different temperatures and humidity conditions. As seen from Table 1, the initial cure speed of FIP beads is significantly dependent on humidity, especially for tack-free time and handling time. However, increasing the cure temperature from 25°C to 35°C does not dramatically change the cure rate of FIP compounds under same relative humidity.

There are two processes going on during the cure of moisture-cure FIP compounds:

hydrolysis and polymerization. Hydrolysis is highly dependent on the effective concentration at the reaction site. High humidity sensitivity but no temperature sensitivity for the initial cure reflects that the initial cure of moisture-cure FIP compounds is controlled by the diffusion of water, as would be expected. If moisture-cure FIP compounds are placed in a high temperature but without humidity control, it may not be helpful to facilitate cure. High humidity is critical to obtain fast cure rate of the moisture-cure FIP compounds. As indicated in the Table 1, below 25%RH, the FIP beads cure very slowly and cannot be handled within 4 hrs. To obtain an acceptable handling time (1 hour or less), 50%RH or higher humidity is necessary.

Figure 9 displays the plots of simple tensile strength vs. curing time under different temperature and humidity for RTV-1. Similar to Fig. 5, the tensile strength of FIP beads will increase gradually, initially quickly and then

leveling off. Again, high relative humidity facilitates cure. When the humidity level is greater than 50% RH, no significant effect on the curing time is observed with temperature.

Table 1 characterizes the cure of RTV-1. As shown above, different physical properties can develop at different rates during cure. Although cure time would be best expressed as a range, a minimum cure time consistent with good properties development is shown.

It is also worth noting that higher cure temperature may also lead to higher tensile strength. Higher temperature may improve the movement of monomers or oligomers and short polymer chains, thus leading to a better internal cross-linking structure. As described in the preceding section, high tensile strength will lead to high adhesion strength. In some cases it may be desirable to cure FIP beads at elevated temperature as well as high humidity.

Table 1. Tack-Free, Handling, and Cure Times of RTV-1 at conditions of varying temperature and humidity.

	Tack-free Time (mins)	Handling Time (mins)	Cure time (hrs)
25 °C, 25%RH	60	240	48-72
25 °C, 50%RH	15	60	24-48
25 °C, 75%RH	10	45	24-48
35 °C, 25%RH	45	210	48-72
35 °C, 50%RH	15	60	24-48
35 °C, 75%RH	10	45	24-48

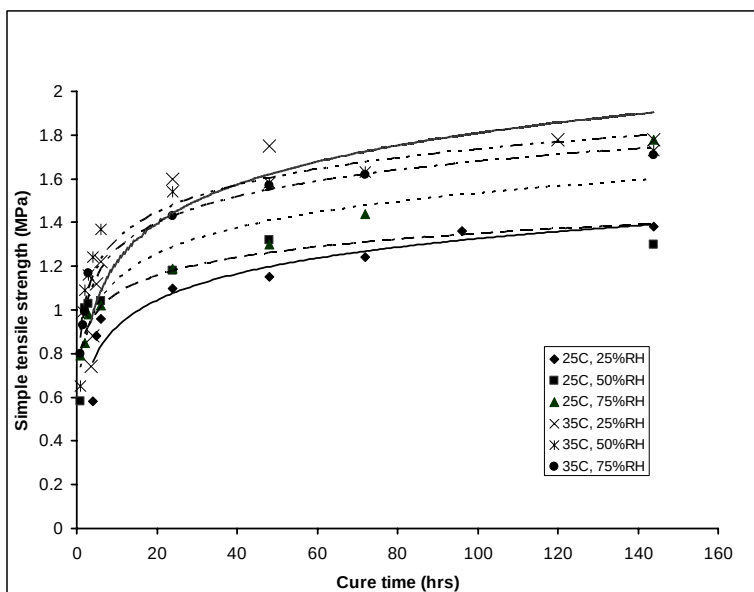


Figure 9. Simple tensile strength vs. cure time of RTV-1 at conditions of varying temperature and humidity.

4 Conclusions

A simple and practical method to characterize the extent of cure of FIP compounds has been described. Using a simple experimental setup, tack-free time, handling time, and cure time can be quantitatively defined, and the cure state of the FIP compounds described. The method has been successfully to study the moisture cure FIP compounds, and demonstrates the importance of high humidity to the cure of such FIP compounds. Slightly increasing the cure temperature will not dramatically increase the cure rate, but may lead to better mechanical properties of the cured FIP gasket. To reach optimum properties of FIP gaskets, a thorough understanding of FIP cure is required.

References

- [1] Bernd Pachaly, Frank Achenbach, Christian Herzig, and Konrad Mautner, "Silicones", Wiley-VCH, Weinheim, 2005.
- [2] "Standard Test Methods for Rubber Property – Compression Set", ASTM D395.