UPDATES ON AGING STUDY AND TRACKING RESISTANCE TESTS

Introduction

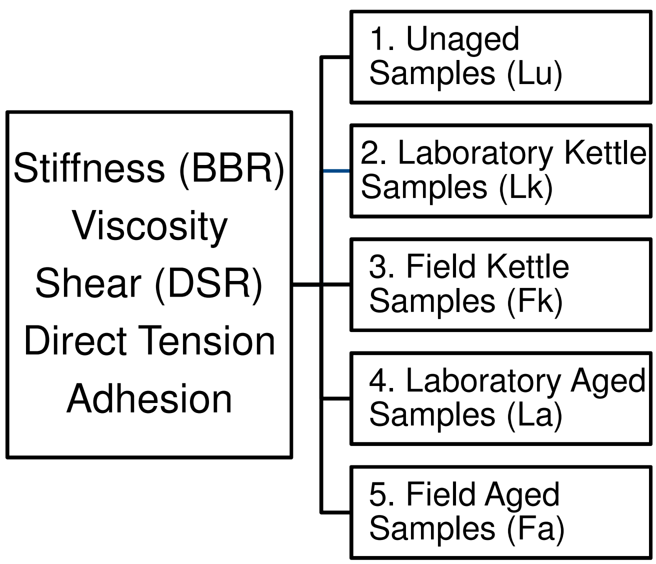
This document was prepared to briefly provide an update of the ongoing experimental program, which is part of the *TPF (5) 225 Pooled Fund study for Crack Sealant Field Validation*. The update includes the work on aging and tracking resistance. Hence, the following is presented:

1. Aging study
2. Development of track resistance test using DSR

Aging Study

Research Methodology

An experimental program was developed to inveistigate the field aging mechanisms and validate vacuum oven aging procedures. According to the program, tests are performed over a wide range of in service temperatures, as well as installation temperature. BBR, DSR, rotation viscosity, and adhesion are among the tests proposed. The tests are conducted on specimens aged in accordance with various laboratory aging protocols and field aged.



Sample Preparation

In this task, various aging methodologies were used to prepare test specimens. The crack sealant bending beam rheometer test (CSBBR) was utilized to assess low temperature performance of sealants under various aging conditions. In addition, the suitability of the vacuum oven aging process was evaluated. The following aging protocols were followed in the study:

1. Laboratory unaged (Lu) □ Samples are homogenized and prepared in accordance with ASTM D5167 (Practice for Melting of Hot-Applied Joint and Crack Sealant and Filler for Evaluation). The homogenized material is then poured into beams, individually wrapped in aluminum foil and stored for later use. The samples were stored in an air-tight container and placed in a freezer to avoid oxidation.
2. Laboratory aged (La) □ Samples prepared in accordance with the vacuum oven aging procedure. Samples were exposed to complete vacuum at 115°C for 16 hours.
3. Kettle aged (Lk) □ Sealant samples were collected during sealant installation at ATREL test site. Samples were obtained from the kettle at regular intervals: 2nd, 3rd, 4th, and 5th hour. The collected samples were poured into beams and stored in the freezer.
4. Laboratory melter aged (Ma) □ Sealant samples were left in the melter for 4 and 8 hours. The samples were then poured into beams and stored in the freezer for later use.
5. Field aged (Fa) □ Sealant samples were collected from ATREL and other test sites. The samples were further separated to “bottom” and “crust” to investigate differential aging.

<u>Aging Test Matrix</u>

An experimental program was developed to study aging mechanisms and validating laboratory vacuum aging procedure. The preliminary results from BBR and DSR tests are presented in this document. Nine materials were selected to conduct the experimental program. Table 2 presents the experimental program and reports progress update.

Table 1. Experimental Program to Study Aging Mechanisms and Progress Update

TEST	**EQUIPMENT**	**MEASUREMENT**	**Progress Update**
Apparent Viscosity	Rotational Viscometer (RV)	Rheological behavior of crack sealants at installation temperatures (160-180℃)	Nine materials were tested. Testing of a 1-year field-aged samples is pending
Flexural Creep	Bending Beam Rheometer (CSBBR)	Stiffness at service temperatures (-4 to -40℃)	Nine materials were tested (including various aging methods)
Complex Modulus	DSR	Modulus at intermediate and high in-service temperatures (28 to 82℃)	Two materials were tested. Other tests are underway

Viscosity Tests

Apparent viscosity tests were conducted on the samples collected from kettle during field sealant installation at ATREL. Figure 1 shows the results for nine of the products. The duration in the kettle clearly increases viscosity at installation temperatures. Viscosity testing is in progress to complete the study with field-aged materials (Fa) and laboratory unaged materials (Lu).

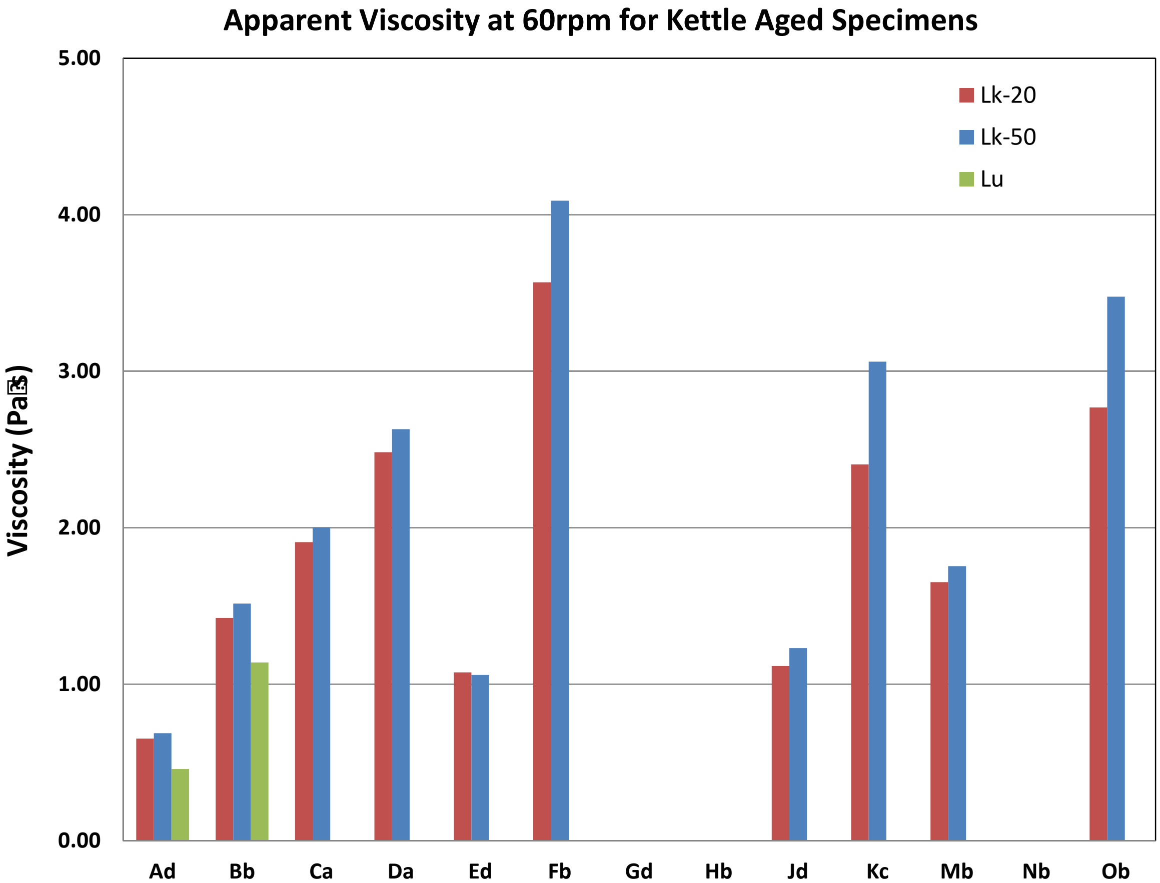


Figure 1. Apparent viscosity results to illustrate kettle aging.

<u>CSBBR Tests</u>

Crack sealant bending beam rheometer tests were conducted for nine of the materials at their corresponding low temperature grade. Stiffness at 240s, average creep rate, and master curves were investigated to evaluate the influence of aging at low temperatures. The tests were conducted at a temperature range of □4 to □40℃. Table 2 illustrates the testing program. Field-aged samples collected from various test sites were also added to the experimental program.

Table 2. Testing Matrix for Evaluating Aging Methods using the CSBBR Test.

CSBBR TESTING MATRIX												
ID		Lu	LK		MA		FK	LA	FA		ASTM Type	Temp (°C)
			2 hr	5 hr	4 hr	8 hr			crust	bottom		
		✓						✓				-40
1	Ad	✓	✓	✓	✓	✓	✓	✓			IV	-34
		✓										-28
		✓	✓	✓	✓	✓	✓	✓				-16
2	Bb	✓						✓			II	-22
		✓						✓				-28
		✓	✓	✓	✓	✓		✓				-4
3	Ca	✓		✓				✓	✓	✓	I	-10
		✓						✓	✓	✓		-16
		✓	✓	✓	✓	✓		✓				-16
4	Da	✓		✓				✓	✓	✓	I	-22
		✓						✓				-28
		✓	✓	✓	✓	✓		✓				-22
5	Ed	✓		✓				✓	✓	✓	IV	-28
		✓						✓				-34
		✓		✓				✓	✓	✓		-22
6	Fb	✓						✓			II	-28
		✓						✓				-34
7	Jd	✓						✓			IV	-34
		✓	✓	✓				✓	✓	✓		-40
		✓	✓	✓			✓	✓	✓	✓		-28
8	Mb	✓						✓			II	-34
		✓						✓				-40
		✓						✓				-22
9	Ob	✓	✓	✓				✓	✓	✓	II	-28
		✓						✓				-34

Figure 2 illustrates sealant stiffness results for laboratory unaged (Lu) and laboratory aged (La) specimens. The effect of vacuum aging on sealant low temperature stiffness is evident. In general, vacuum aging increases sealant low temperature stiffness.

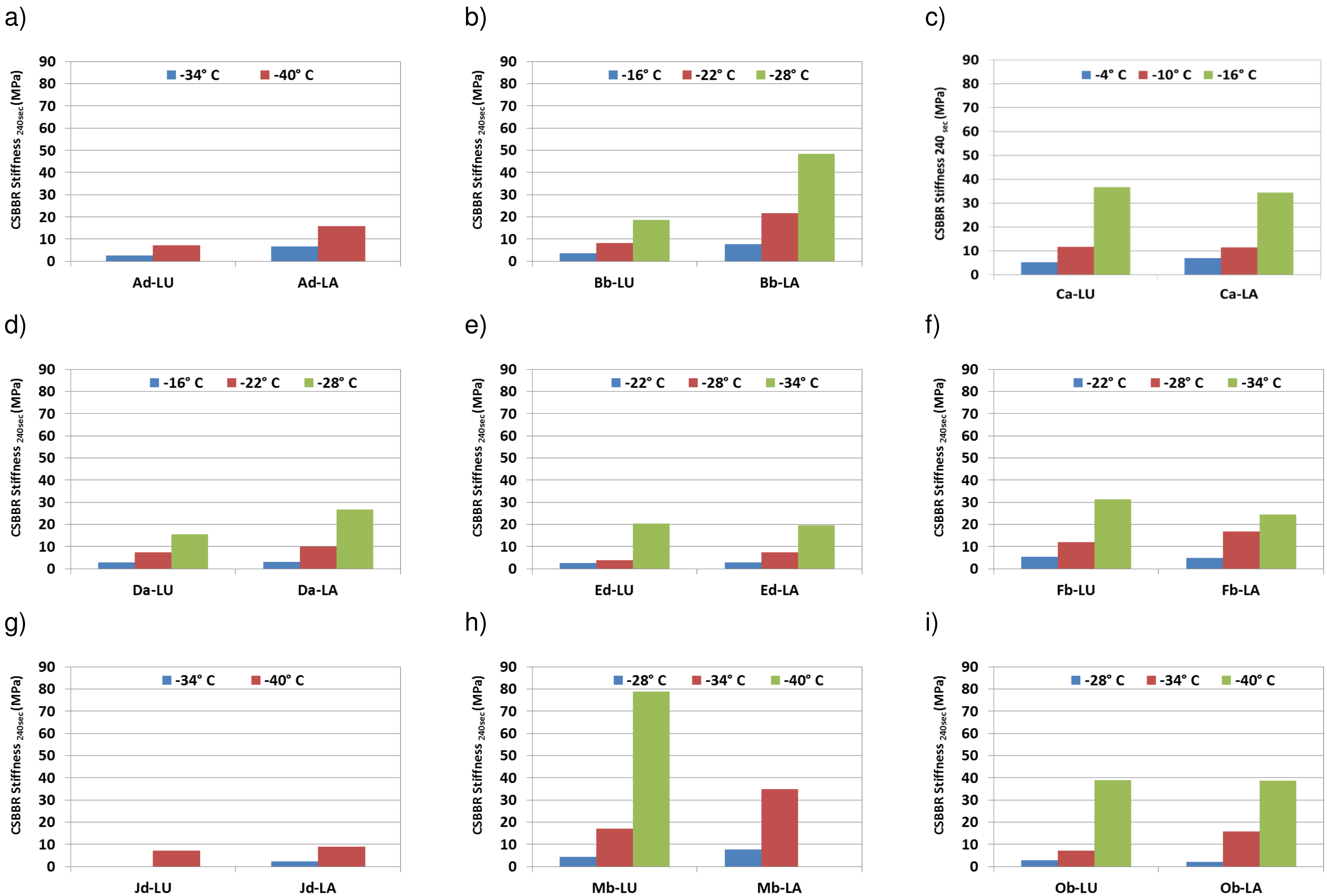


Figure 2. Effect of vacuum aging on CSBBR stiffness (@ 240s) at various temperatures.

Figure 3 illustrates the results of sealants aged utilizing various aging mechanisms. The influence of kettle, melter, and vacuum oven aging is summarized in Figure 3. An aging index is introduced, which is the ratio of aged to unaged sealant's stiffness. For five of the materials, vacuum oven-aged specimens clearly demonstrated an increase in stiffness. Kettle-aged specimens exhibited an increase in stiffness generally; however, no significant change after two hours of aging. It appears that excessive duration of melter aging could cause sealant stiffness degradation.

	Aging Index						
Material	Lu	LK-2h	LK-5h	MA-4h	MA-8h	LA	Temp
Ad	1.00	1.40	1.25	1.70	2.60	2.48	-10° C
Bb	1.00	1.11	1.28	0.78	1.11	1.98	-22° C
Ca	1.00	0.80	2.27	0.62	0.59	4.29	-28° C
Da	1.00	1.31	1.18	1.02	0.81	1.14	-22° C
Ed	1.00	1.69	1.48	2.51	2.47	1.18	-40° C

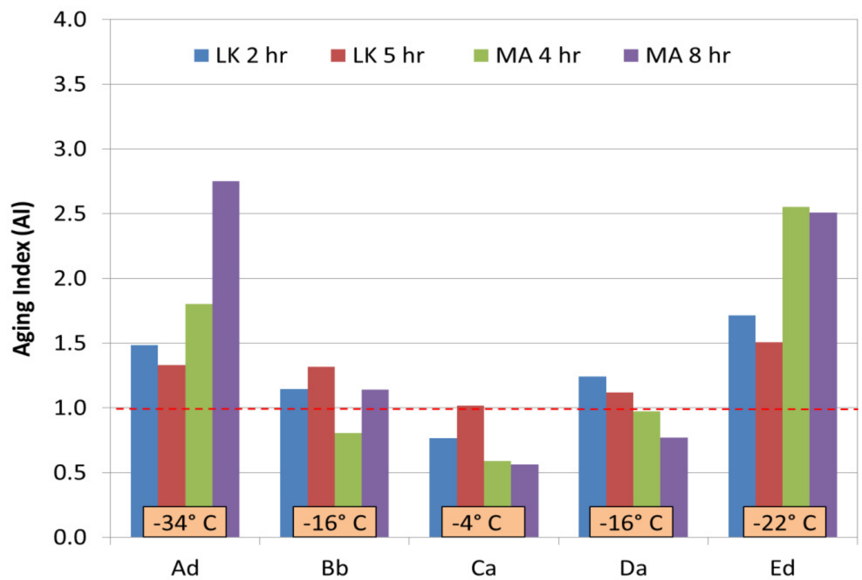


Figure 3. CSBBR sealant stiffness results illustrating the effect of various aging mechanisms

CSBBR tests were also conducted on sealant samples collected from the ATREL test site. The sealant samples were collected after six and 12 months of installation. The behavior of various sealants after 11 months of installation is presented in Figure 4. The effect of aging varies for from one sealant to another. Most sealants developed block cracking and surface deformations; the surface pattern of sealants Jd and Kc is distinctive. The two sealants are produced by the same manufacturer and are recommended for application in cold climates.

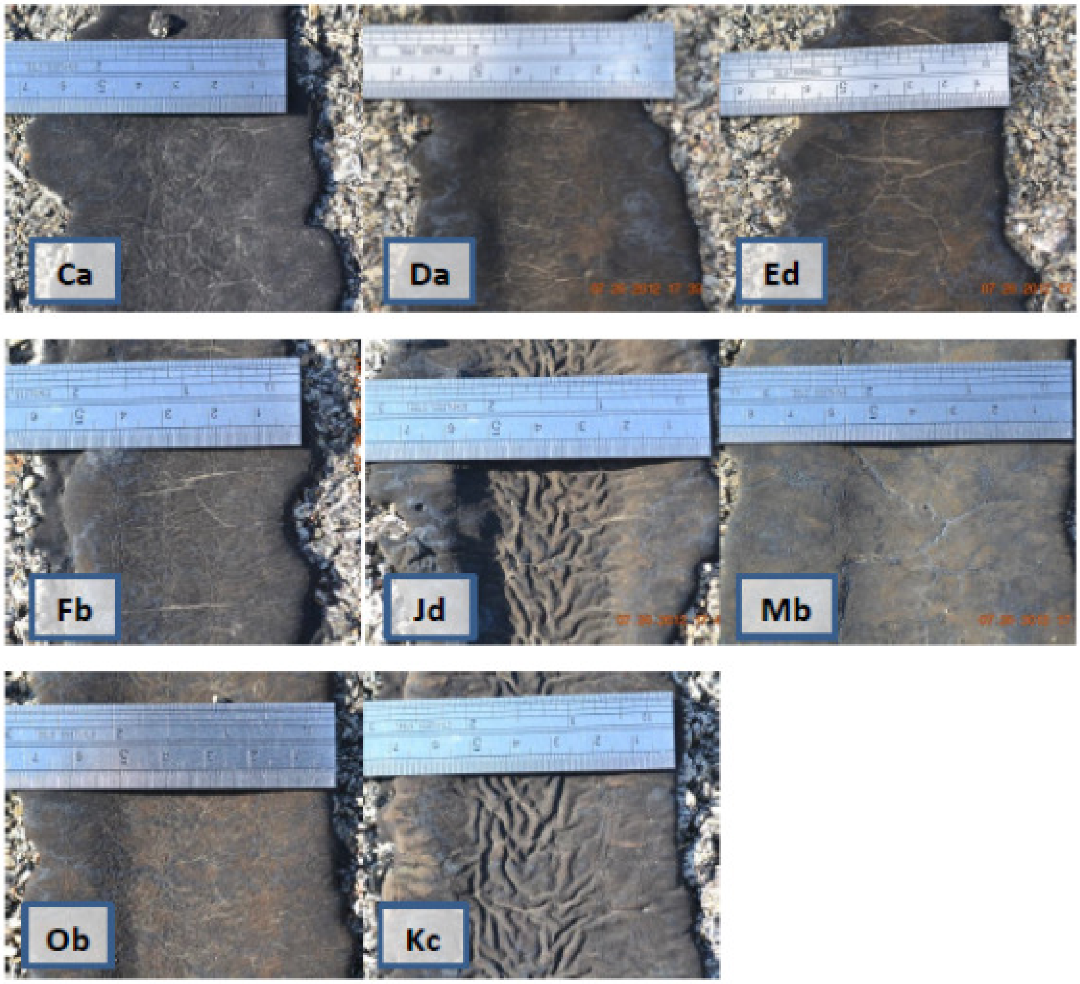


Figure 4. Pictures taken from routed and sealed cracks at ATREL test site to illustrate surface aging of various sealants under same weathering conditions without vehicular loading.

Since the ATREL testing site is protected from vehicular traffic, the samples collected were fairly clean without any debris or incompressible materials. After cleaning the samples as needed, the remaining thickness of the sealant is approximately 20mm. To assess differential aging through the specimen's thickness, each specimen was cut into two parts: Top 5mm of the sample, exposed to weathering, which is designated as "crust"; and the remaining 15mm, which is designated as "bottom". These two parts were tested independently to evaluate their apparent viscosity, flexural stiffness, and adhesion strength.

Figure 5 shows the stiffness evolution from six to 12 months for each sealant crust and bottom parts. It is evident that the sealant stiffness has increased significantly over 12 months of exposure to normal weathering. The stiffness increase of the crust part is more than double that of the bottom.

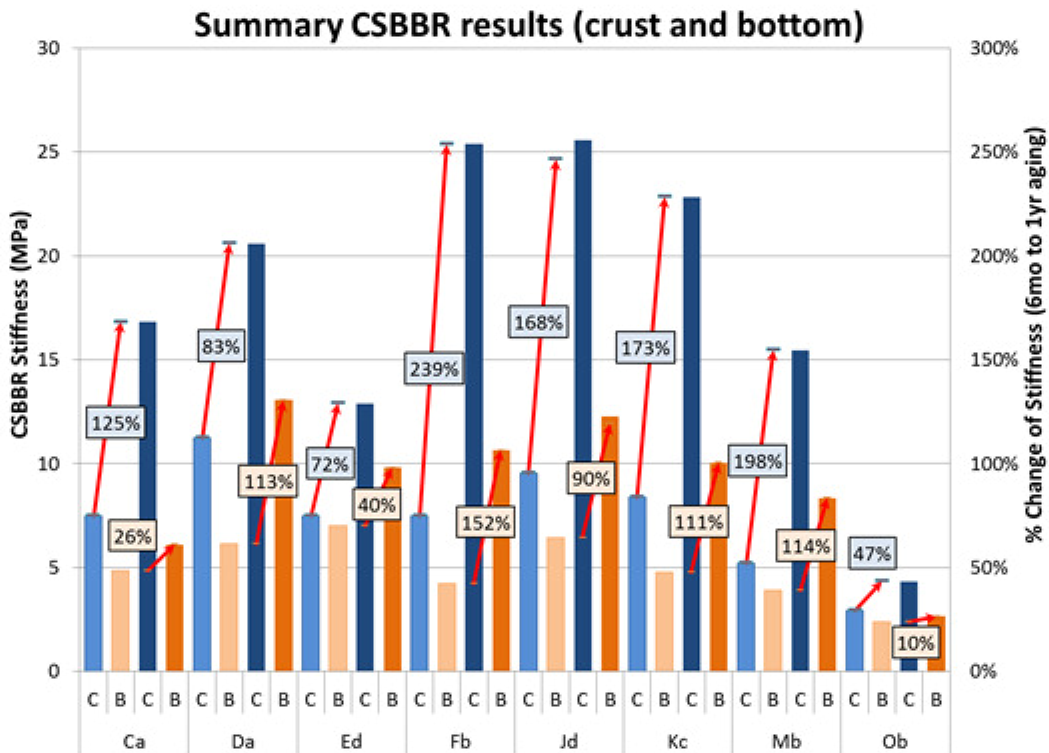


Figure 5. CSBBR results of sealants collected from ATREL test site.

DSR Tests

Preliminarily, three sealants were selected to investigate the effect of field aging on their viscoelastic properties at intermediate and high temperatures using DSR. A frequency sweep test has been used to measure the complex shear moduli at various loading frequencies. Both bottom and crust parts of ATREL test site sealants were tested at 10% constant strain loading and angular frequency range of 0.1 to 100 rad/s. The test was conducted at four temperatures; 28℃, 46℃, 64℃, and 82℃. This allows characterizing the sealant at intermediate and high temperatures. Test results are presented as a master curve calculated at 64℃; Figure 6 presents results for two sealants. Each master curve presents the average of three replicates.

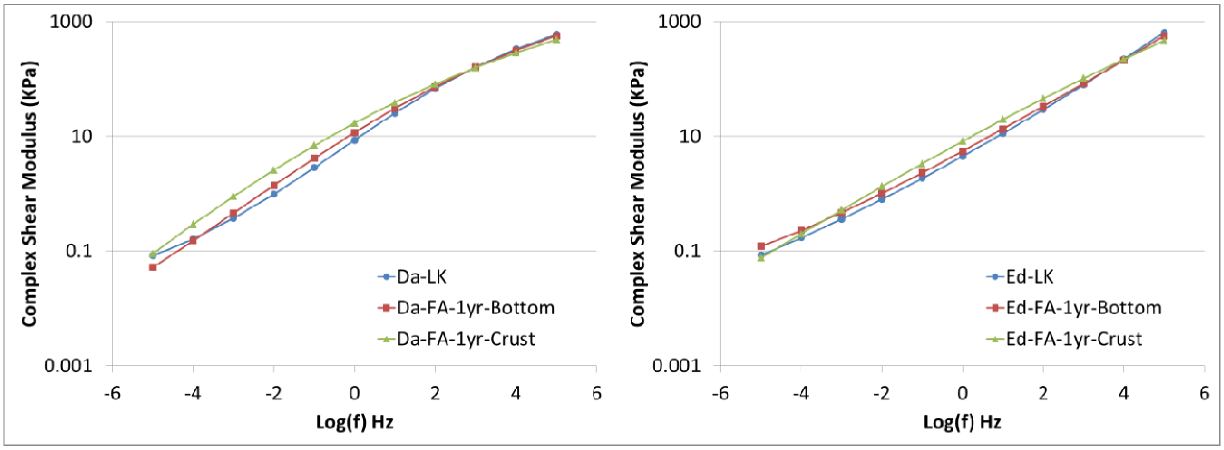


Figure 6. Complex shear modulus master curve at 64℃ for two sealants exposed to different aging conditions

Development of Track Resistance Test Using DSR

Sealant tracking failure results from shear loading applied by passing vehicles. Several factors contribute to sealant tracking including improper selection of sealant type, early traffic opening, improper sealant installation, and high temperatures. Sealant tracking is an important failure; hence, a parameter related to tracking will be included in the sealant grading system. In the first phase of the crack sealant study, multiple stress creep recovery (MSCR) test was developed using the DSR to determine the high temperature grading and tracking resistance. However, the MSCR test procedure is complex and time consuming. Therefore, a more practical test is considered. The following test procedure is proposed:

- Monotonically increase shear strain at a constant shear rate until sealant failure and identify yield stress (shear strains may reach 600%);
- A shear rate of 0.01 1/sec is used; and
- Testing temperature range is 46 to 82℃ at 6℃ increments.
- A threshold shear stress value will be determined for consideration in the sealant grading system.

The following testing protocol was applied on five sealants. The specimens were prepared according to the ASTM procedures (1hr melting and homogenization - Lu).

Table 3. The Experimental Program for High Temperature Grading.

Test	Parameter	Objective
MSCR	C and P	Identify high temperature grade
Complex Modulus	G* and phase angle	Develop master curve
Yield	Yield stress	A potential new approach for high temperature grading

Preliminary Test Results

The samples used in the pooled fund (Ad, Bb, Ed) and two samples from Michigan test deck were used. The MSCR test results for sealant Ad is shown in Figure 7.**Error! Reference source not found.** A summary of MSCR test results for five sealants is presented in Table 4. According to the MSCR procedure, the sealant high temperature grade is highlighted where C is greater than 4 kPa and P is greater than 0.7.

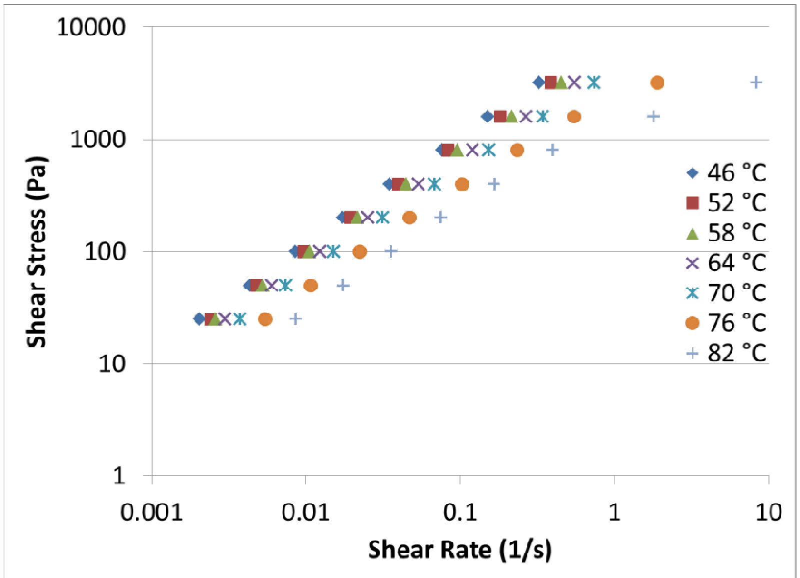


Figure 7. MSCR test results, shear stress vs. shear rate at various temperatures.

Table 4. A Summary of MSCR Parameters for Five Sealants.

Sample	Temperature (°C)	MSCR Parameter	
		C (kPa)	P
MI-Sec 7 Lu	82	5.943	0.7949
MI-Sec 16 Lu	70	11.204	0.8924
Ad-Lu	70	4.381	0.9105
Bb-Lu	64	4.807	0.9187
Ed-Lu	76	4.157	0.8642

The yield test results for Ad and Bb samples at three different temperatures are presented in Figure 8. The tests were conducted at three temperatures initially. As temperature increases, the capacity of the material sustaining shear loads decreases. Since sealants do not exhibit a clear yielding point, yield stress will be selected at specific strain levels (50%, 100%, and 200% etc.). The yield test was repeated for the five materials whose MSCR parameters were presented in Table 4.

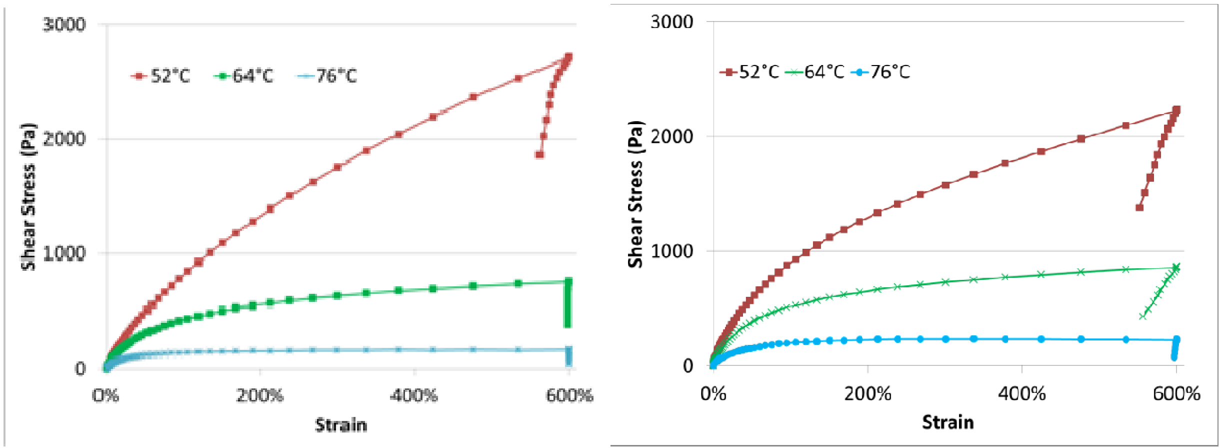


Figure 8. Shear stress vs. strain for two sealants at three different temperatures

The results of yield tests are shown in Figure 9. These are preliminary results and further analysis is underway. Preliminary results suggest that the yield test may potentially group sealants in stress range based on strain level (50, 100, or 200%). For example, if a sealant is tested at a specific temperature, and it has a shear stress higher than 250kPa at 200% strain level, then the sealant will pass that criterion.

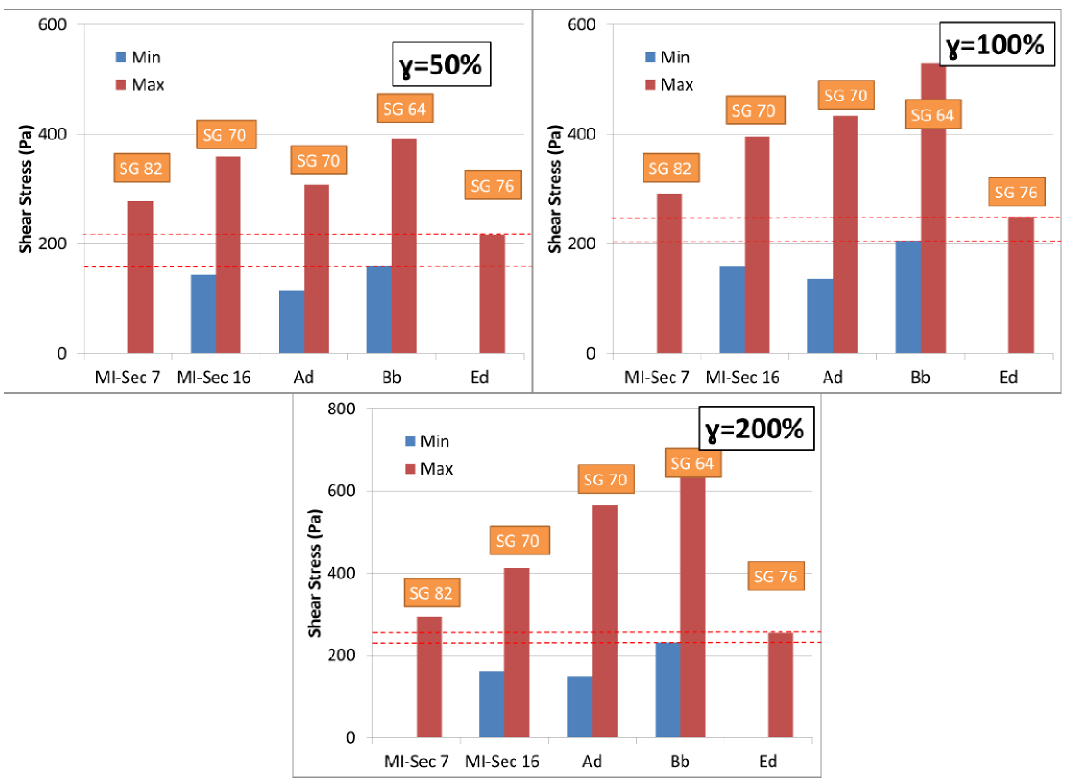


Figure 9. Preliminary investigation results using yield test.