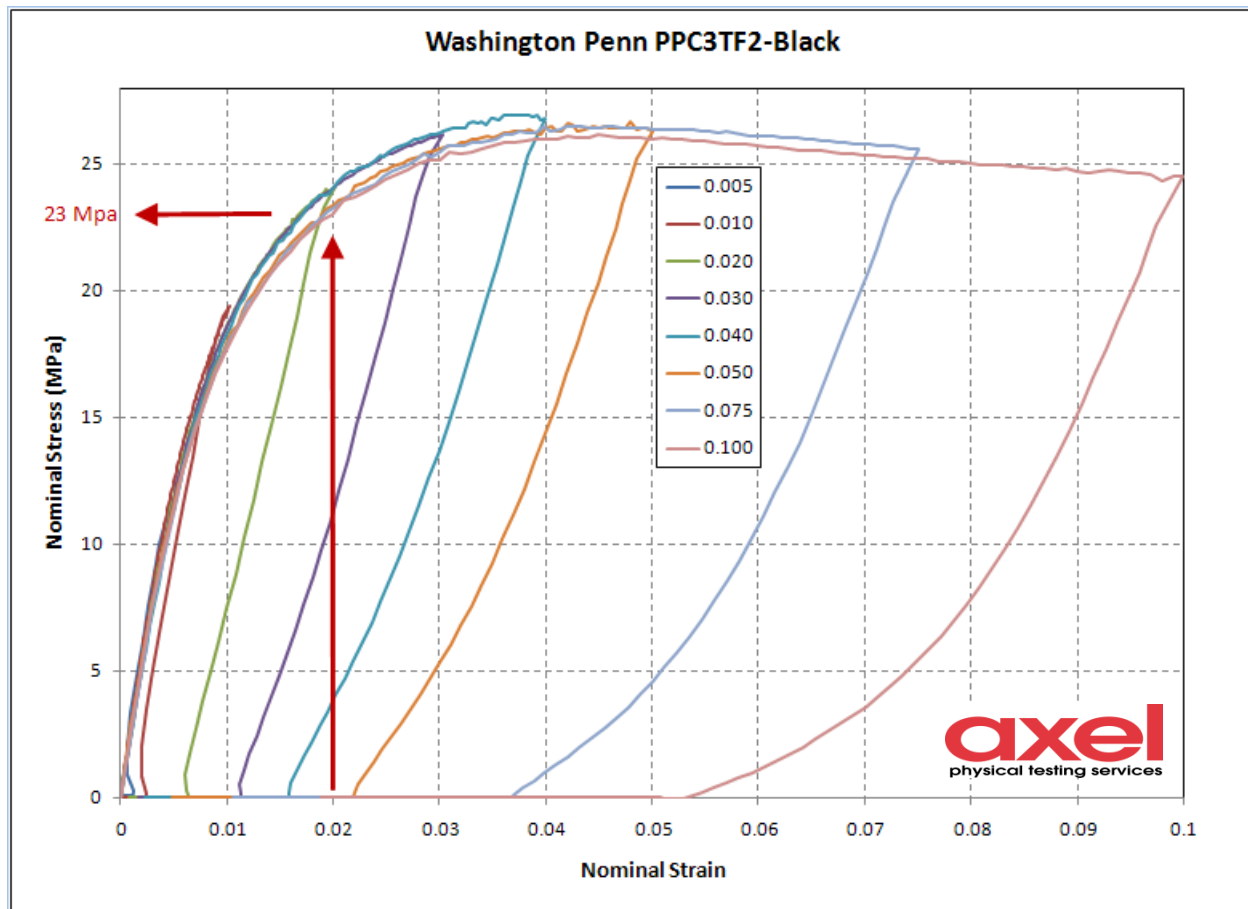# Calibration of Polypropylene

Washington Penn grade PPC3TF2-Black

Author: Tod Dalrymple   Date: August, 2014

Overview:

The purpose of this document is to describe a variety of test data that we have for a particular grade of polypropylene and demonstrate a calibration recipe that focuses on the nonlinear viscoelastic behavior of the material below yield. The Abaqus material model used to represent nonlinear viscoelasticity is the Parallel Rheological Framework (PRF) model. The samples of material for this testing were kindly donated by the Washington Penn company and the testing performed by Axel Products Physical Testing Services, Ann Arbor, Michigan.

Calibration of Polypropylene, Washington Penn grade PPC3TF2-Black.

Overview:

The purpose of this document is to describe a variety of test data that we have for a particular grade of polypropylene and demonstrate a calibration recipe that focuses on the nonlinear viscoelastic behavior of the material below yield. The Abaqus material model used to represent nonlinear viscoelasticity is the Parallel Rheological Framework (PRF) model. The samples of material for this testing were kindly donated by the Washington Penn company and the testing performed by Axel Products Physical Testing Services, Ann Arbor, Michigan. There were three types of tests performed:

1) Strain Recovery testing to determine the approximate yield point.
2) Uniaxial tension stress relaxation tests at 4 levels of strain (below yield).
3) Uniaxial tension tests at 4 strain rates.

The Strain Recovery test is also called by other names such as the load-unload-recover test. Identifying the yield point in polymers and plastics can be challenging because it may be masked by viscous effects. The point of this strain recovery test is to load and unload a specimen and allow enough time for the viscous strain to fully recover (dissipate) leaving only the permanent strain. Pages 4&5 of this document explain this test in a bit more detail, http://www.axelproducts.com/downloads/TestingPlasticForFEA.pdf.   For even more details, we can point to the following reference papers:

Lobo, H., J. A. Hurtado, *Characterization and modeling of non-linear behavior of plastics*, 2006 Abaqus User's Conference, available from DS SIMULIA

Quinson, R., J. Perez, M. Rink, A. Pavan, *Components of non-elastic deformation in amorphous glassy polymers*, JOURNAL OF MATERIALS SCIENCE 31 (1996) 4387-4394

Brusselle-Dupend, N., D. Lai, X. Feaugas, M. Guigon, M. Clavel, *Experimental characterization and modeling of the uniaxial mechanical behavior of polypropylene before necking*, Oil & Gas Science and Technology - Rev. IFP Volume 57, Number 2, March-April 2002

We have summarized the strain recovery testing in other documents and it is not our intention to get into all of the details of that test here, but simply to show the outcome for this polypropylene material. This test was performed in uniaxial tension on 8 separate virgin specimens, loaded to varying levels of imposed strain. Figure 1 shows the imposed strain and the timeframe over which it was applied. The legend shows the peak imposed strain. After reaching the peak imposed strain the load was quickly removed using a slip grip fixture. The load was removed to zero over about 0.26 seconds or less. The strain was allowed to recover for about 1200 secs, at which time the remaining strain was considered to be a permanent, or plastic, strain. Examination of the permanent strain versus imposed strain from these tests led us to conclude that yielding occurred at an imposed strain of about 1-2% strain. This is consistent with the papers by Brusselle-Dupend, who also tested a polypropylene and concluded that yielding occurred at about 1.5-3% imposed strain. As shown in Figure 2 below, this equates to a yield stress in uniaxial tension of about 20-23 MPa. Based on this information we decided to perform 4 simple tension stress relaxation tests at imposed strains of 0.5%, 0.75%, 1% and 1.5% strains. Our intention was to capture the viscous behavior in the absence of plastic yielding.
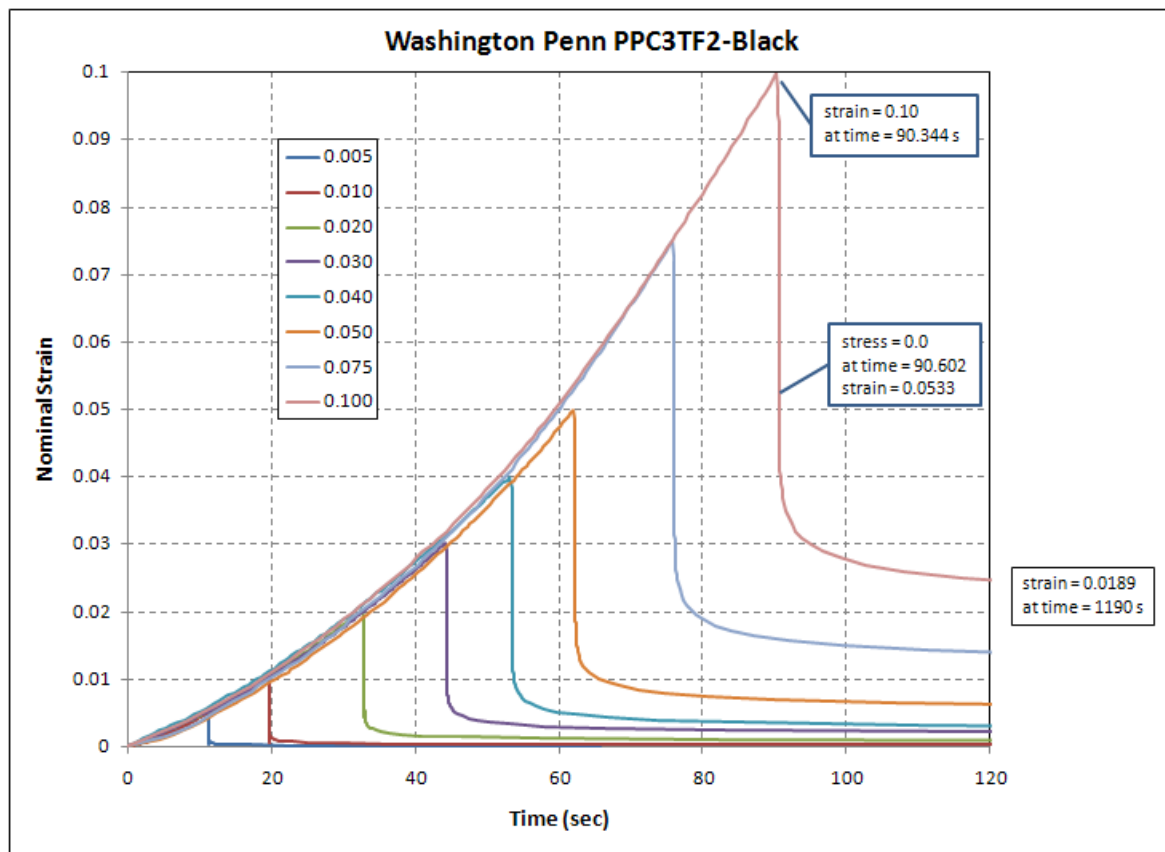
Figure 1, Strain history from the strain recovery test suite, 8 virgin specimens used
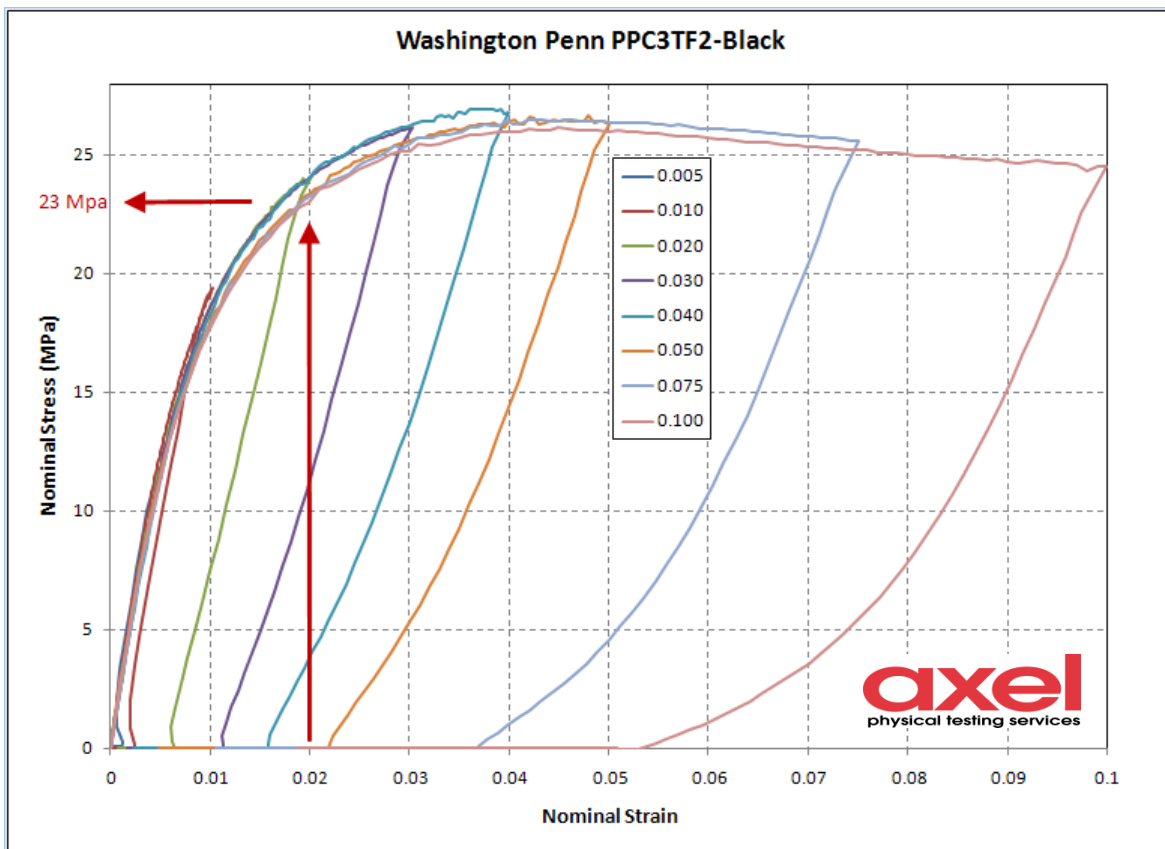

Figure 2, Stress-Strain responses from the strain recovery test suite

The stress relaxation testing is summarized here in Figure 3. Four separate tests were performed.
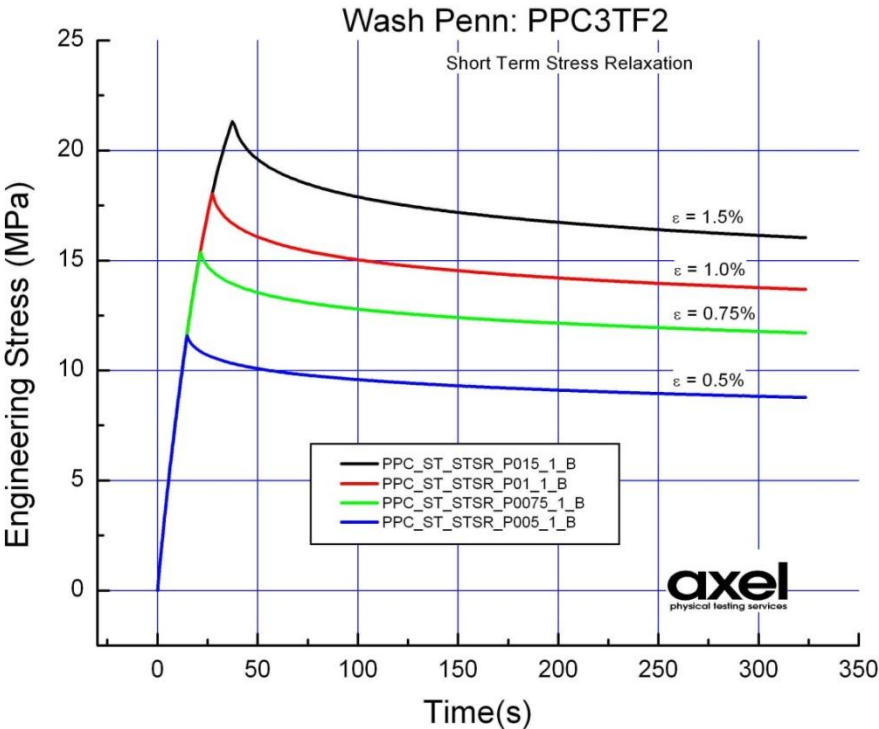

Figure 3, Stress relaxation test suite responses, 4 tests

Uniaxial testing at 4 different strain rates was also performed and summarized here in Figure 4:
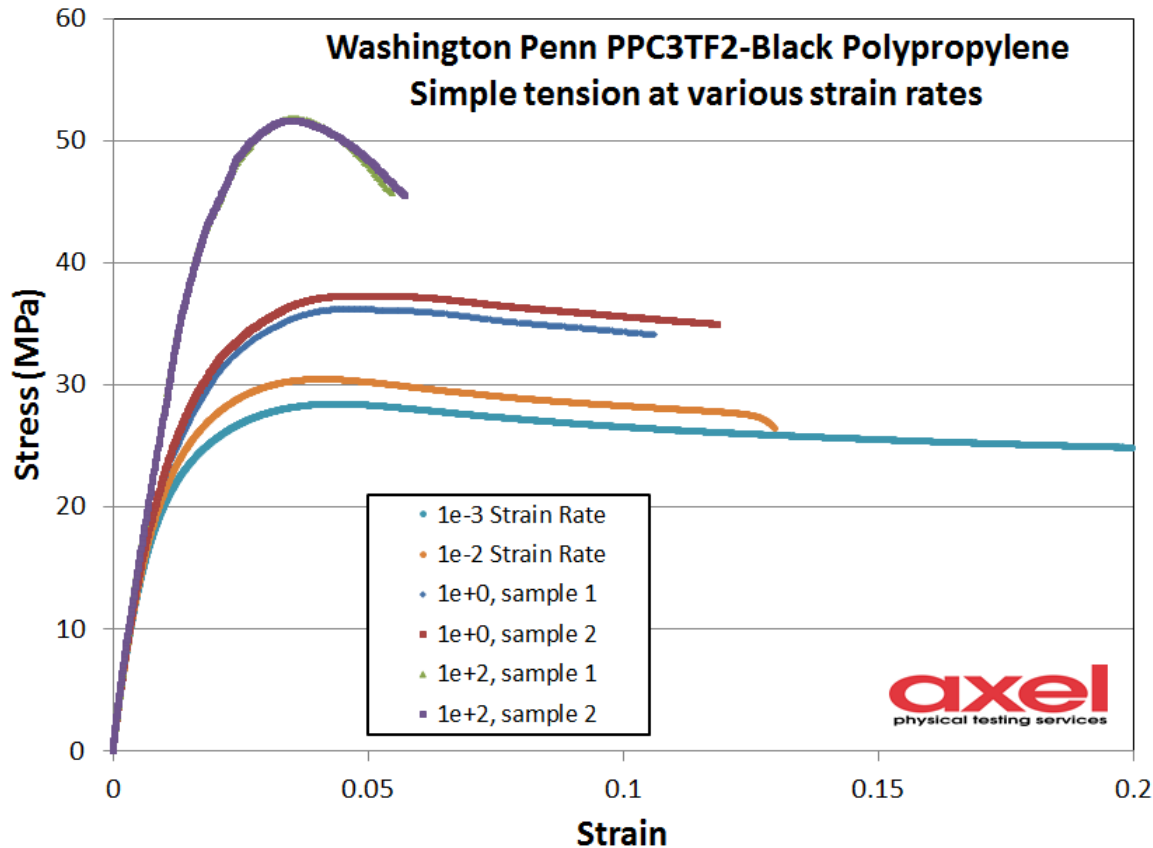

Figure 4, Simple tension pull tests to failure at 4 different strain rates

It is always a good idea to look over your test data, looking for consistent data. We have done that with this data, looking for zero shift errors, etc. It is also a good idea to compare test data from the different test suites against each other. In Figure 5 below, we show the 4 stress relaxation tests compared to one of the strain recovery tests.
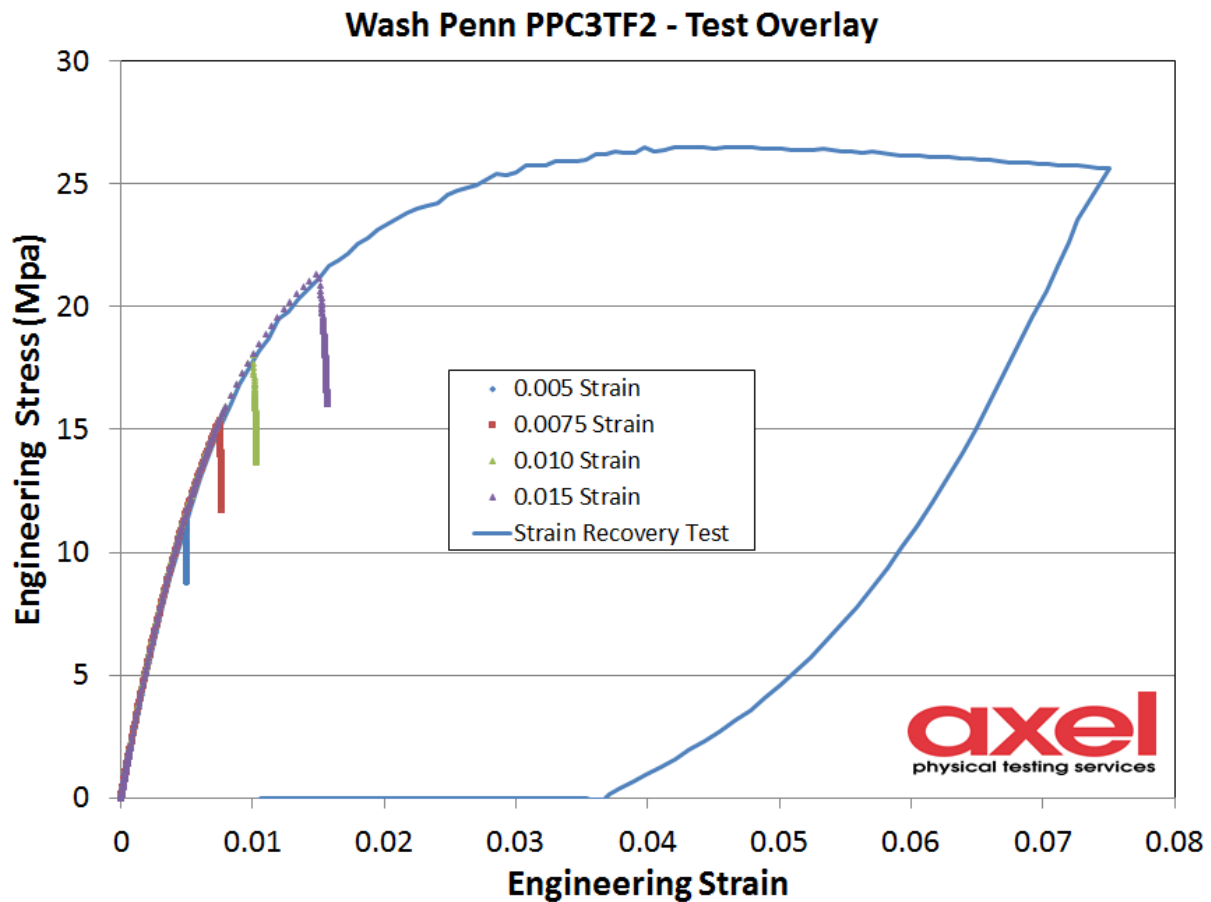

Figure 5, Comparison of various test data for consistency checking

One slight thing to note and this is somewhat common, in the stress relaxation testing the crosshead displacement was held fixed, and the strain measured in the narrow region of the ISO5286 dogbone specimen grew slightly over time. For instance, in the above plot of the 1.5% strain test (purple symbols), as the stress decayed from about 21 MPa down to about 16 MPa, the laser extensometer measured strain went from 1.5% strain to 1.56% strain at the end of the test. We have used this laser extensometer measured strain as the strain loading in all of our calibration activity.

Now that we have reviewed the test data, we will describe a step-by-step process of calibration. This process will be described as follows:

1) Determine elastic modulus from the highest strain rate test data (100 sec^{-1} rate).
2) Calculate a Prony series viscoelasticity from only the lowest stress relaxation data.
3) Show the Prony series for all relaxation data – note that nonlinear viscoelasticity is needed.
4) Convert Prony parameters to degenerate PRF model parameters
5) Show the degenerate PRF model response
6) Optimize the PRF model parameters to 4 pieces of test data (3 relaxation + 1 high rate).

Calibration Process:

As stated earlier, our goal is to determine the parameters, or coefficients, needed for a PRF model to describe the nonlinear viscoelastic behavior prior to yielding. We will approach this in a step-by-step methodology, developing a recipe that one can follow for other materials.

1) Use the highest rate stress-strain curve to determine the elastic modulus. Using the early part of the stress-strain curve we determine an elastic modulus of 3300 MPa. Since our goal is to determine a nonlinear viscoelastic PRF model, which uses hyperelasticity for the elastic behavior, we convert E=3300 MPa into a NeoHookean C10 = 550 (assuming incompressibility, E=6*C10).   Note that the highest rate test reached a strain of 0.003 at 0.066 milliseconds. Remember that for later.
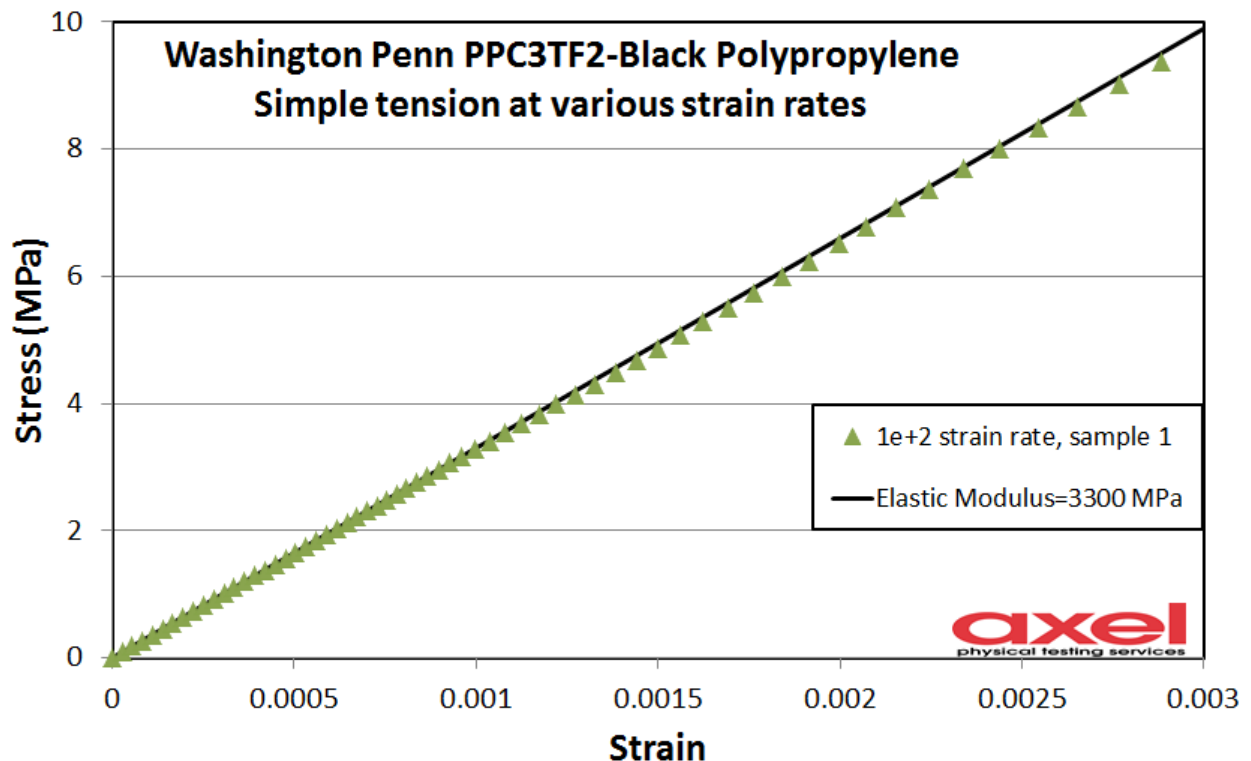

Figure 6, Calculation of elastic modulus from the highest rate simple pull test

   a) Estimate the decay ; move to subdirectory a_estimate_decay_to_lowest_relax_data

Now that we have estimated the elastic modulus, we will look at the instantaneous elastic response versus the relaxation test data to understand how much relaxation needs to takes place. This will allow us to make an initial estimate of the Prony series parameters. In the Excel file that we have used to archive all of our relaxation test data, we simply add a column of elastic stress response (calculated at all the test time points / strain points).

This is so we can see how high the elastic response is and then estimate the Prony Σ g's

From the plot below, we estimate the Σ g's ~ 0.46   (46% decay from about 16.25 MPa down to 8.75)
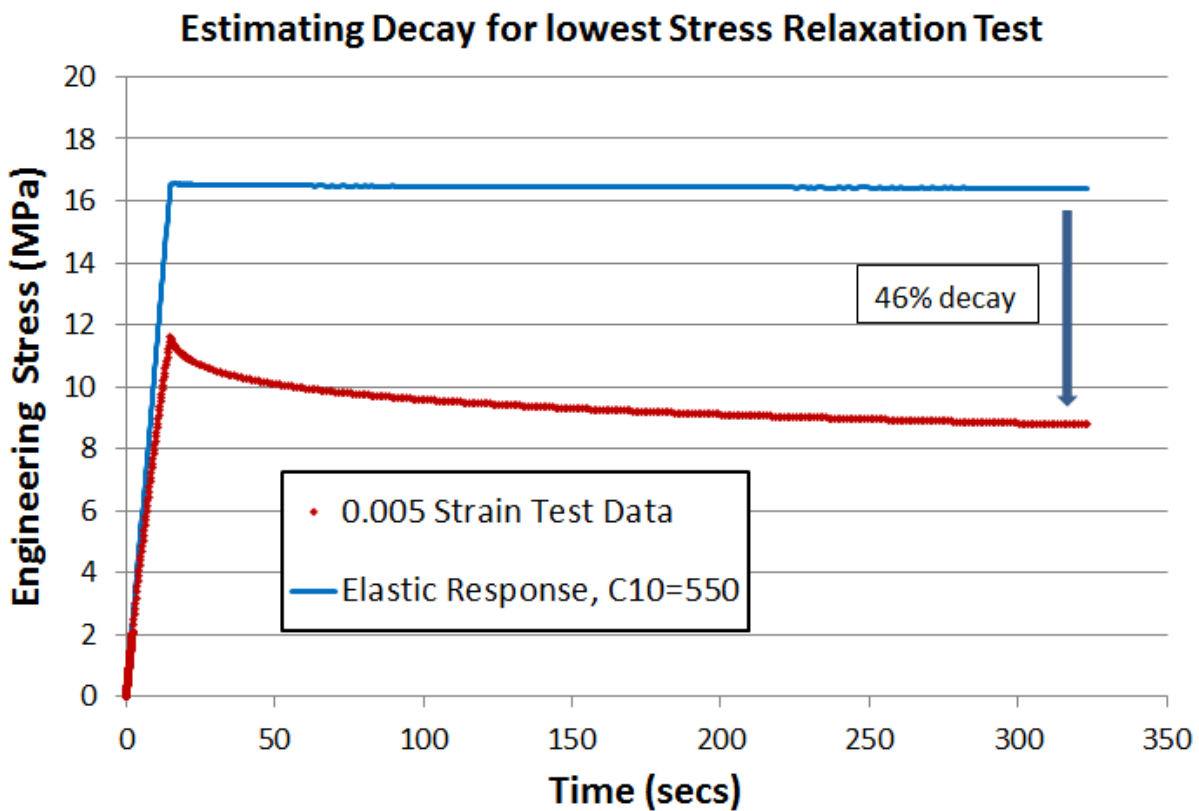
Figure 7, Estimate decay from instantaneous elastic response to longer time

As a general practice, when estimating the number of (g, tau) pairs needed in a Prony series representation, a good rule of thumb is to have one (g, tau) pair per decade of time. When I first went through this exercise, I chose to define 4 pairs g's and tau's:

Estimate 4 taus's as 0.4, 4, 40, 400

Estimate 4 g's as 0.2, 0.15, 0.10, 0.05   (sum is 0.50)

Things change a bit when our target is to convert from a Prony series to the PRF model with the Bailey-Norton creep law. The "m" parameter in the Bailey-Norton creep law allows this functional form to modify (shift) the relaxation response over time. So as we convert from Prony series to the PRF model, we know that we will need fewer PRF terms. Knowing this, we select fewer Prony terms in our initial guess:

Estimate 3 taus's as 0.4, 10, 200

Estimate 3 g's as 0.2, 0.15, 0.10   (sum is 0.45)

Specification in MCalibration looks like:

```
*Hyperelastic, Neo Hookean, Moduli=instant
550.0, 0.0
*Viscoelastic, Time=Prony
** g_i, kappa_i, tau_i
0.200,  0,    0.4
0.150,  0,   10.0
0.100,  0,  200.0
**
```

2) Move to subdirectory 2_C10_and_Prony.

Our first guess at Prony terms is pretty close. Results from unit cube analysis, file:
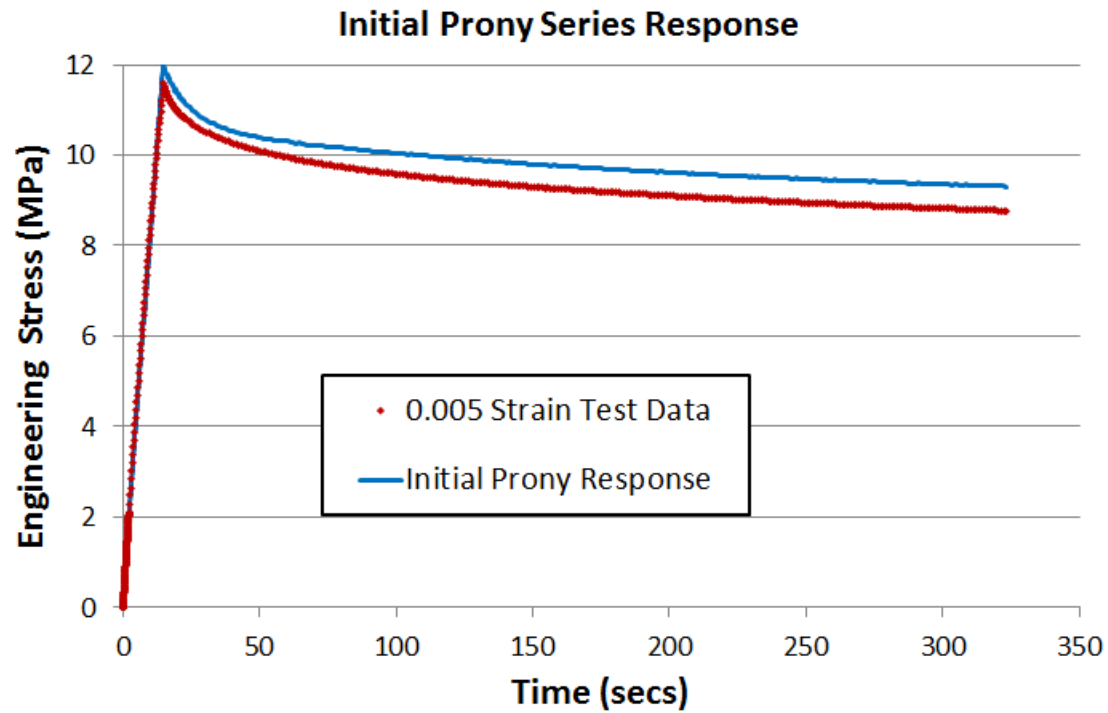STSR_unit_cube_initial_Prony_0050_strain

Figure 8, Prony series response to lowest stress relaxation data

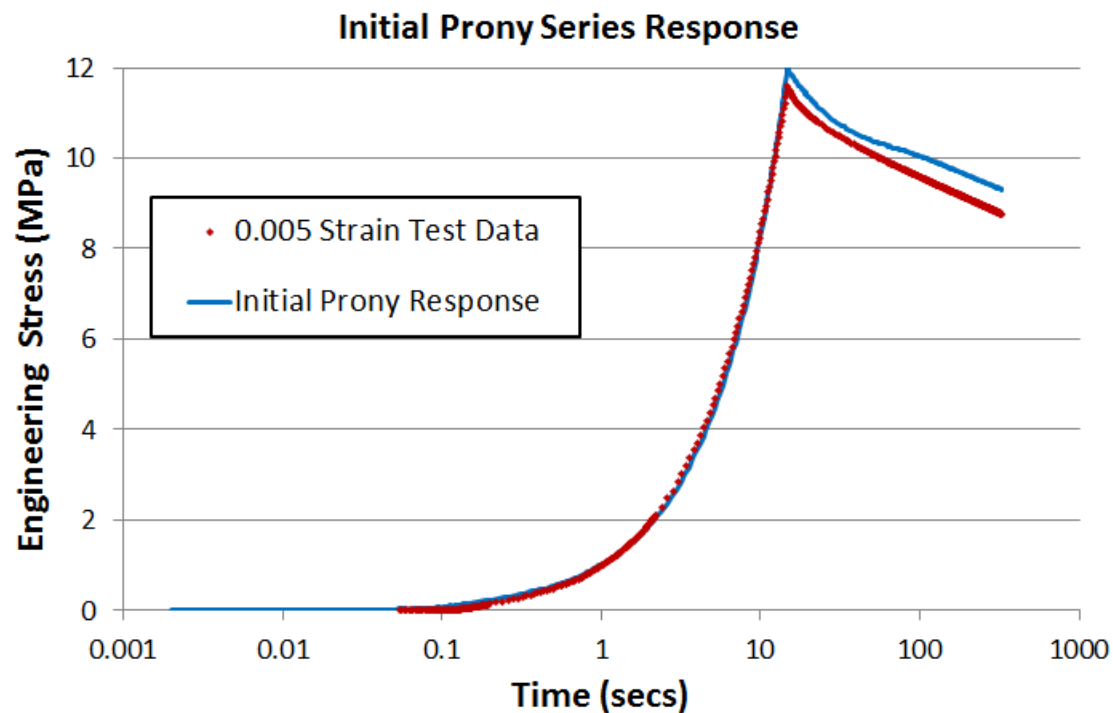Alternate view, with log time (to better see the initial strain ramp up):

Figure 9, Prony series response to lowest stress relaxation data, log(time)

Let's refine the Prony coefficients. Looking at the Figure 2a, we can see that the first Prony g term needs to be a little larger, change 0.2 to 0.21. And let's change the 2nd g from 0.15 to 0.16
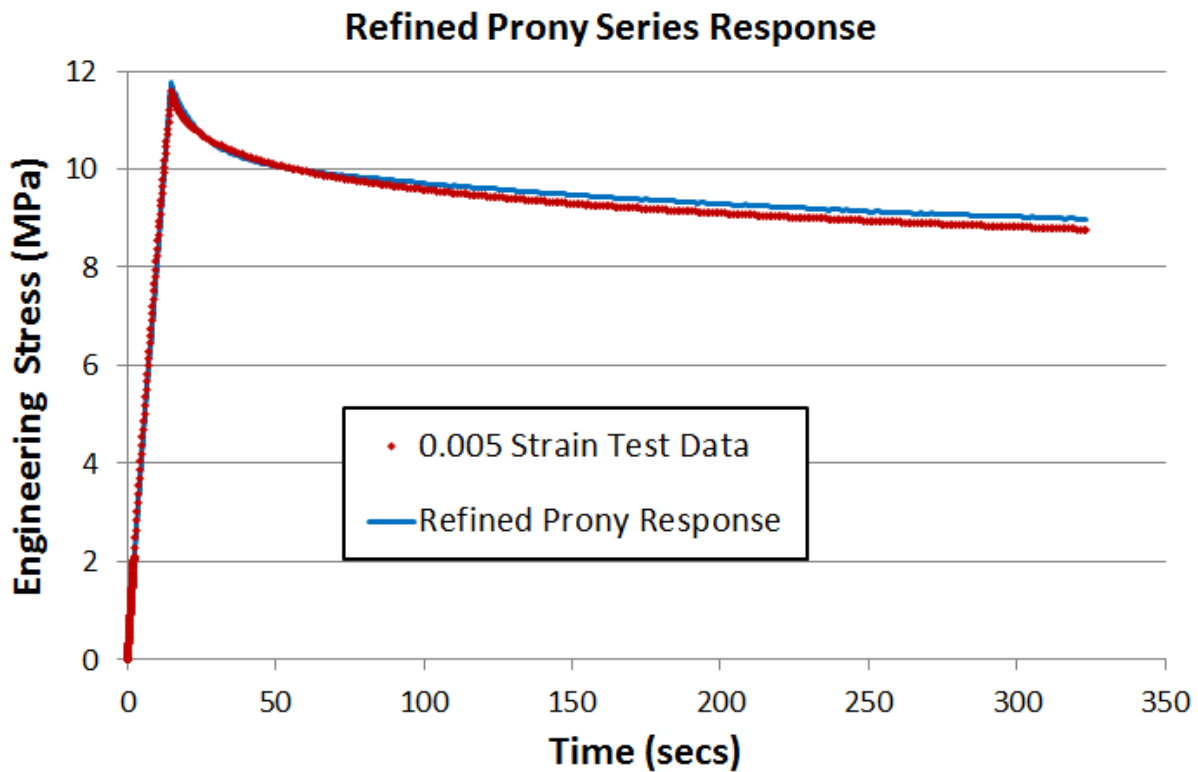
Refined Prony Series Response

Figure 10, Refined Prony series response compared to the lowest relaxation test data

So far we have used two pieces of test data to define the Neo-Hookean elasticity and the Prony series. Let's take a look at both responses.
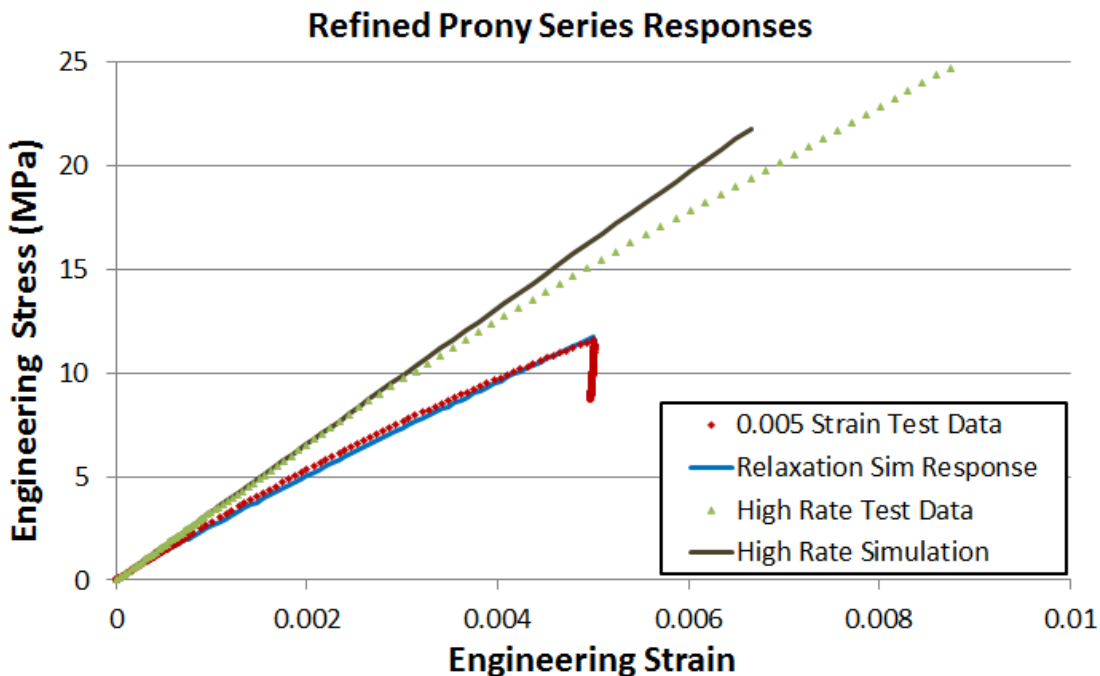
Refined Prony Series Responses

Figure 11, Refined Prony series response, showing both high-rate test and lowest relaxation test data

So far, the steps we've taken to estimate the elastic modulus and the Prony series have not used any optimization or calibration tool. We have made "by hand" estimates and shown responses generated by running a unit cube simulation. For simplicity and convenience, beginning in step 3 we will use a calibration tool called MCalibration (from Veryst Corp.).

3) Now take a look at how our existing Prony model responses compare to all 4 stress relaxation tests.
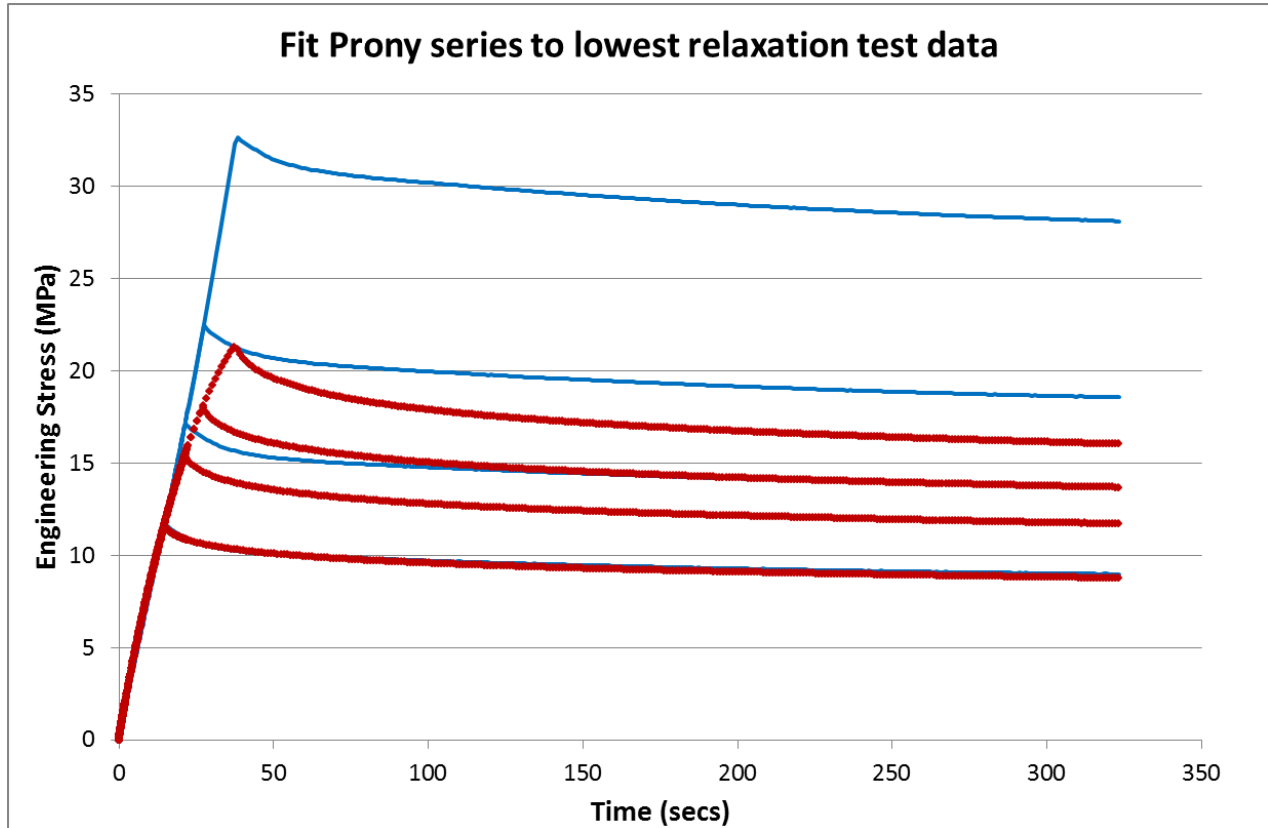

Figure 12, Prony model response to all 4 stress relaxation tests

This shows us that the polypropylene material behaves in a nonlinear viscoelastic manner. That is, at higher and higher strain excitations, the real material responds with more viscous flow. We need to move to a nonlinear viscoelastic model.

4) Convert Prony to PRF ; change to directory **4_convert_Prony_to_Norton**

In our directory structure, we will move to directory **4_convert_Prony_to_Norton** and use the Excel spreadsheet named "Prony to Norton Conversion_3gs" to convert the NeoHookean+Prony series model to an equivalent PRF model. This directory contains only the Excel spreadsheet. The PRF model can be calibrated in MCalibration using an Abaqus template version, which will run an Abaqus unit cube analysis to get the PRF responses, or MCalibration includes special built-in (equation driven) solver for the PRF model. Since the built-in MCalibration solver uses different coefficient terminology, the Excel spreadsheet calculates both the standard Abaqus terms and the terms required to use the special MCalibration version. The maximum number of viscous terms allowed (version 2.5.2) in the special MCalibration version of the PRF model is limited to 3. In the figure below we shoe how the Excel spreadsheet converts between our NeoHookean+Prony material model to a 3 term PRF model.

This conversion assumes n=1 and m=0				
Prony Series			Power Law Creep	
			Sratio_i= g_i	
			A_i= 1/(6*g_i*tau_i*C10)	
C10	550			
D1	0			
g1	0.21		SR1	0.21
tau1	0.4		A1	3.6075E-03
g2	0.16		SR2	0.16
tau2	10		A2	1.8939E-04
g3	0.1		SR3	0.1
tau3	200		A3	1.5152E-05

Figure 13, Excel spreadsheet conversion from Prony coefficients to degenerate PRF model parameters.

5) Equivalent PRF model response ; change to directory **5_initial_PRF_model**

Using all the coefficients from the Excel spreadsheet, here is the equivalent PRF model response. Just as we expected, it shows the same response as the Prony model. This is because n=1 and m=0 makes this PRF material model a linear viscoelastic model.
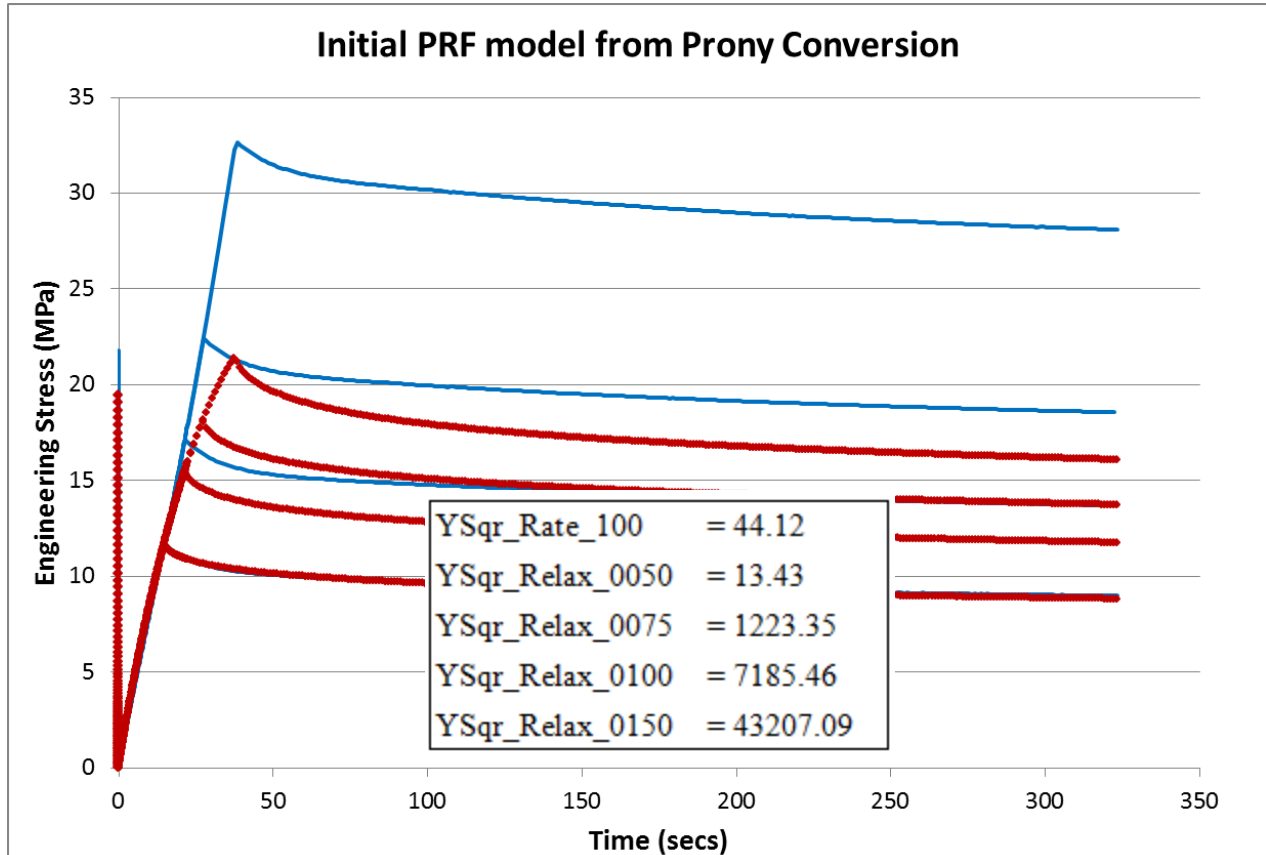

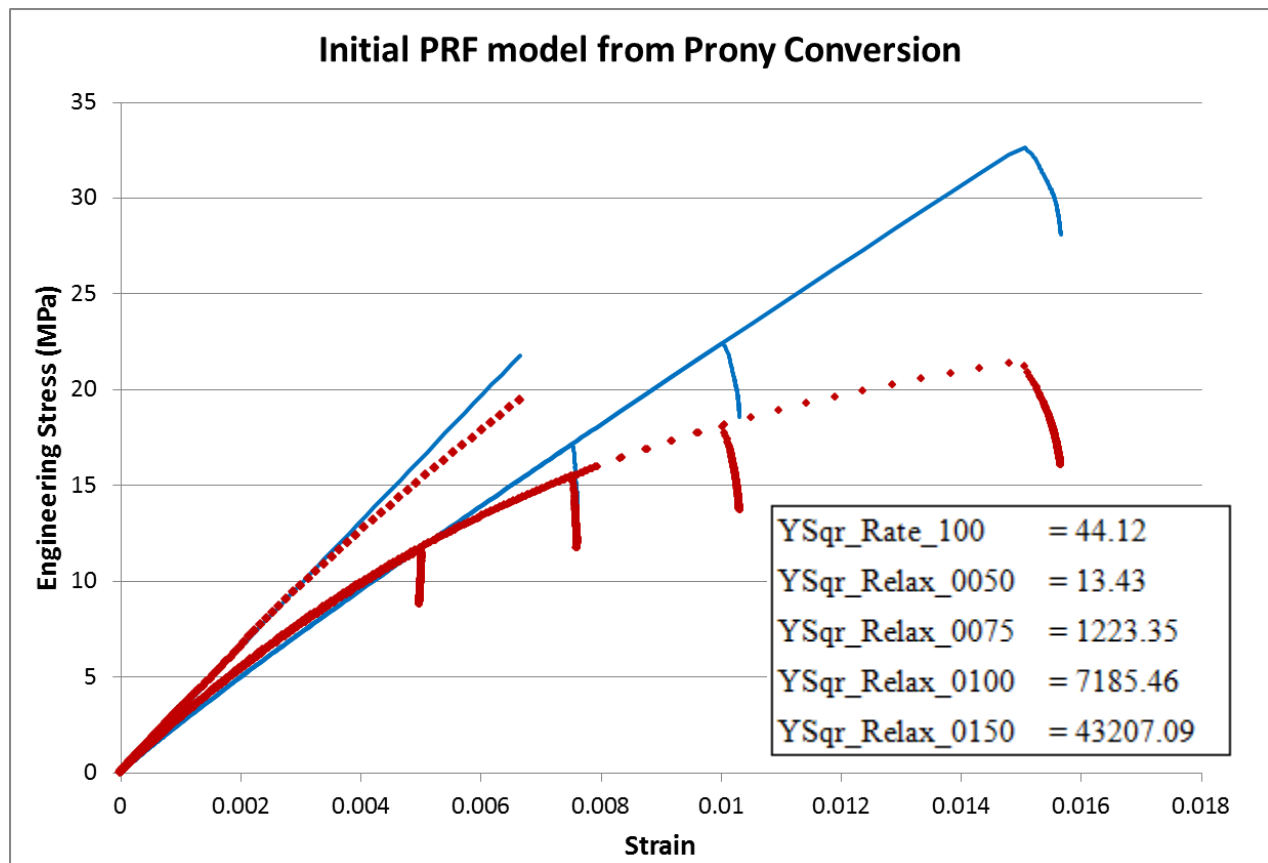Figure 14, Equivalent PRF model responds just like the Prony model

Figure 15, Equivalent PRF model responds just like the Prony model

The PRF material model used in the simulations for Figure 14 & 15 is given here:

*Material, name=PRF_mat
*HYPERELASTIC,NEO HOOKEAN, moduli=instantaneous
550.0, 0.0
**
*Viscoelastic, Nonlinear, NetworkId=1, SRatio=0.21, Law=strain
** A1,   n1, m1
0.0036075, 1.0, 0.0
**
*Viscoelastic, Nonlinear, NetworkId=2, SRatio=0.16, Law=strain
0.00018939, 1.0, 0.0
**
*Viscoelastic, Nonlinear, NetworkId=3, SRatio=0.10, Law=strain
1.5152e-5, 1.0, 0.0

The YSqrDiff values shown in Figures 14&15 are the standard "Sum of the Squared Difference" error measure from the Isight Data Matching component.   We already understand that the PRF model needs n>1. This change will make the relaxation happen faster at higher strains.   We also know that if we change m<0 this will accentuate relaxation at early times.

6) Optimization of the PRF model with Isight; change to directory **6_optimize_PRF_model**

To calibrate, or optimize, the PRF material model parameters we will make use of Isight. The Isight workflow is shown below in Figure 16. We are a little concerned that the highest stress relaxation test (file relax_data_0150) may have loaded a bit beyond yield. Because of that, we will use just the lowest 3 stress relaxation tests (plus the highest rate test) to do the next bit of calibration. The Isight workflow uses 4 Abaqus components; each component runs a single unit cube analysis. The strain from each test is used to drive the unit cube displacement. Each of the four Abaqus unit cube input files references (via *include) the same material model definition (file named PRF_mat.inp). The Data Matching component is used to compare each of the 4 simulation responses to their respective test data. We have used the "Sum of the Squared Difference" error measure. The Optimization driver is used to perform the optimization. The error measures from the Data Matching component are fed to the optimizer and the optimizer in turn attempts to minimize the error responses by changing the material model parameters. Once the optimization process is complete, the final 4 simulation responses (time, strain, stress) are written to an Excel file. This Excel file also holds the original test data and we have generated most of the test – simulation comparison plots from this Excel file.
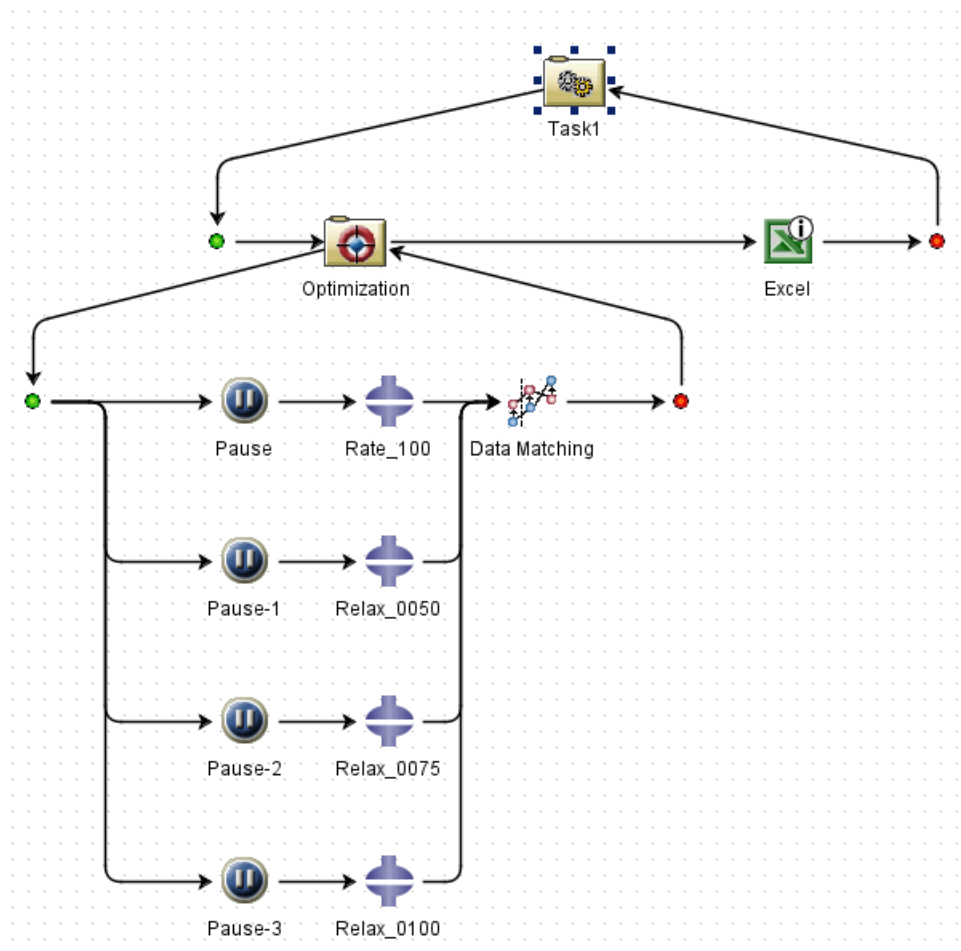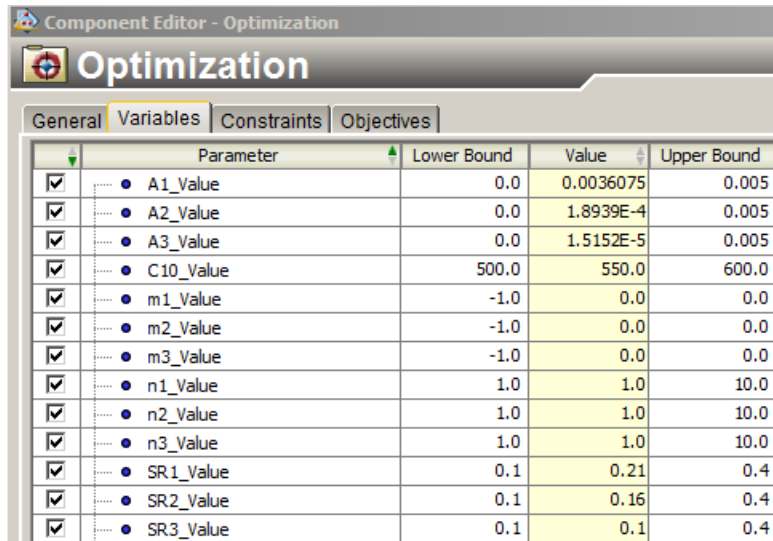

Figure 16, Isight workflow for PRF model parameter optimization

Side Note: Why have we used the "Pause" component? With the 4 Abaqus analysis jobs started simultaneously on a Windows laptop, there was some intermittent difficulty with file cleanup occurring at the end of the simulations. Attempted deletion of some files in the Windows TMP$DIR was raising a

Windows error that would stop the Isight execution. Inserting the Pause component with a 1 second delay between simulation launches circumvented this glitch.

Using the Isight workflow shown in Figure 16, we initialize the Optimization component with the design variables as shown below (Fig. 17). There are no optimization constraints, and the optimizer attempts to minimize the sum of the four error norms.

	Parameter	Lower Bound	Value	Upper Bound
☑	● A1_Value	0.0	0.0036075	0.005
☑	● A2_Value	0.0	1.8939E-4	0.005
☑	● A3_Value	0.0	1.5152E-5	0.005
☑	● C10_Value	500.0	550.0	600.0
☑	● m1_Value	-1.0	0.0	0.0
☑	● m2_Value	-1.0	0.0	0.0
☑	● m3_Value	-1.0	0.0	0.0
☑	● n1_Value	1.0	1.0	10.0
☑	● n2_Value	1.0	1.0	10.0
☑	● n3_Value	1.0	1.0	10.0
☑	● SR1_Value	0.1	0.21	0.4
☑	● SR2_Value	0.1	0.16	0.4
☑	● SR3_Value	0.1	0.1	0.4

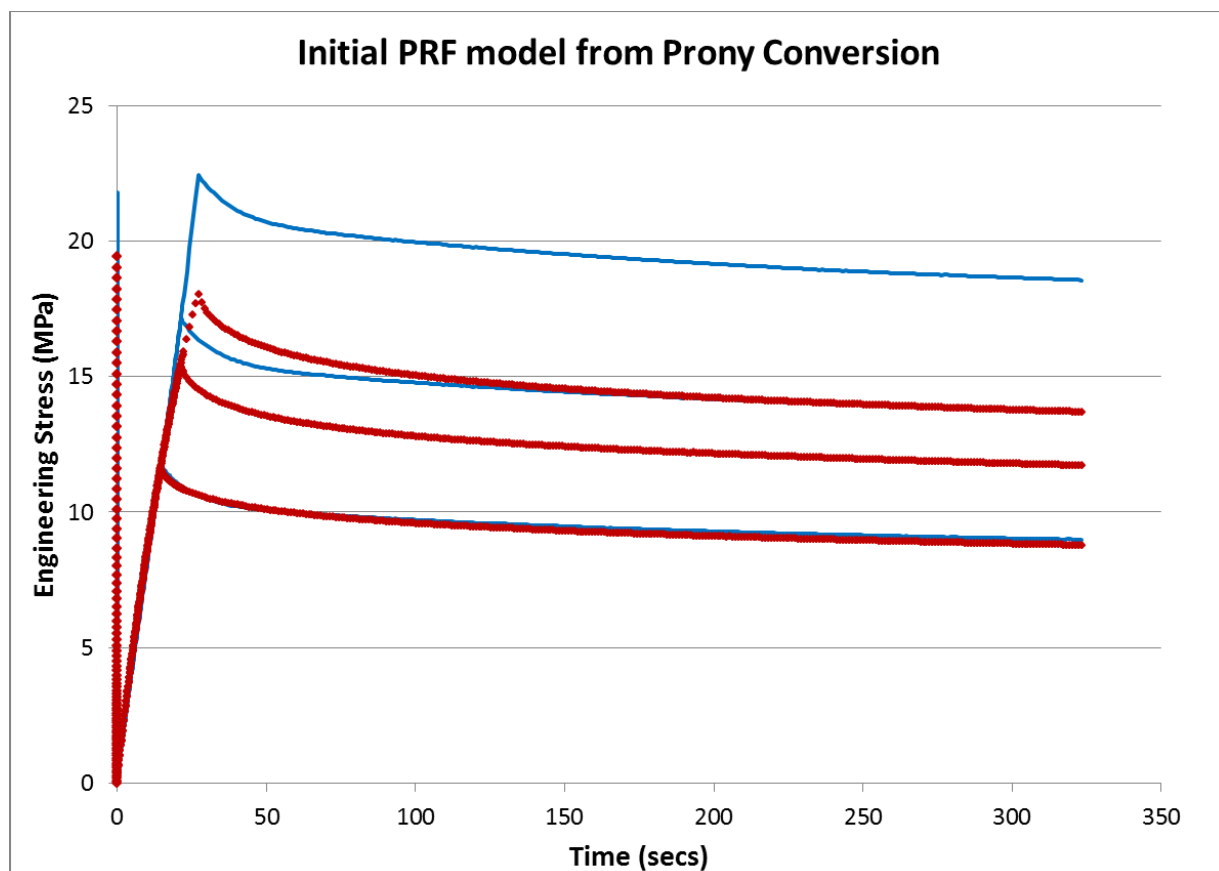Figure 17, Isight Optimizer, initial PRF model parameters (design variables)

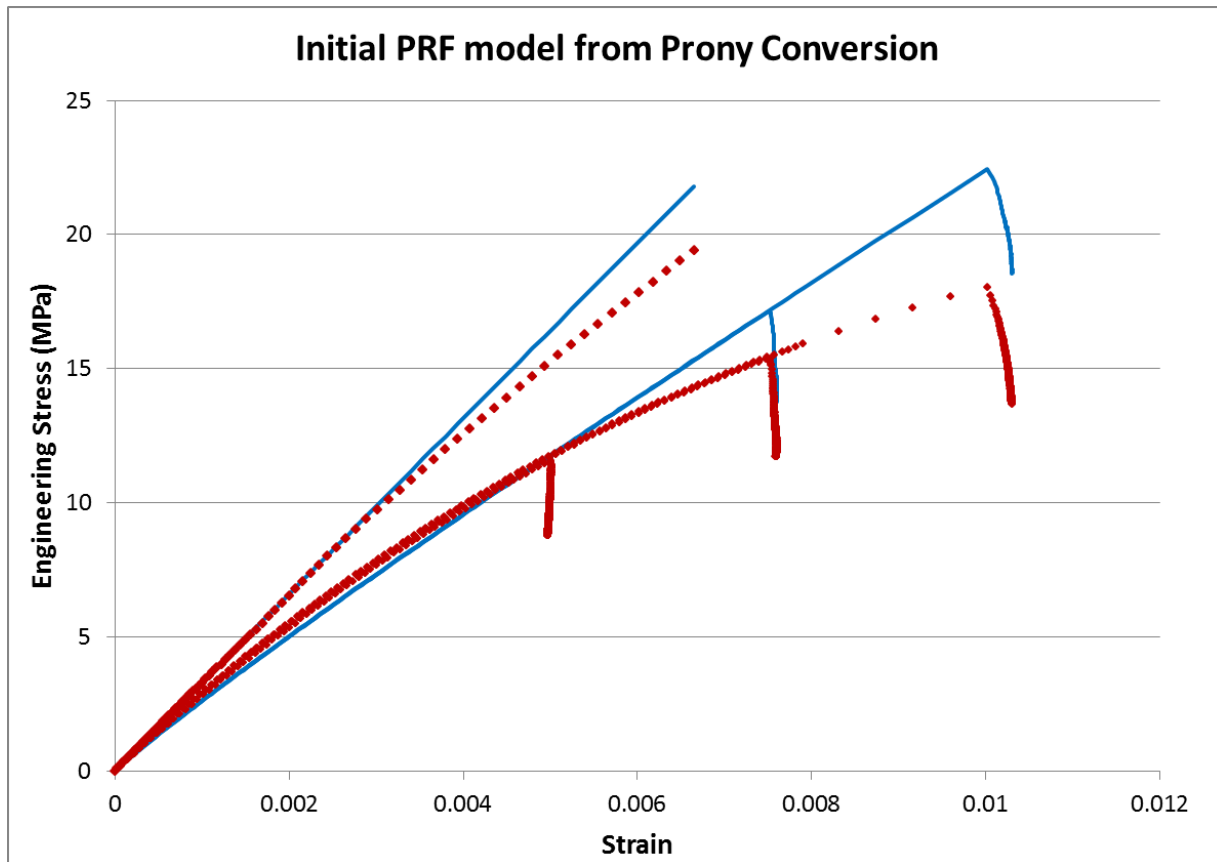Figure 18, Baseline PRF response, n=1, m=0, using 3 lowest relaxation tests + high rate test

Figure 19, Baseline PRF response, n=1, m=0, using 3 lowest relaxation tests + high rate test

Here are the error measures and the sum of the errors is the Objective Function:

YSqrDiff_Rate_100	YSqrDiff_Relax_0050	YSqrDiff_Relax_0075	YSqrDiff_Relax_0100	Objective Function
44.11825481	13.08320751	1222.348854	7183.832553	8463.382869

For material model calibration/optimization in the past I have found both the Hookes-Jeeve (HJ) and Pointer (Ptr) algorithms fairly effective. I ran a series of 3 successive HJ optimizations and found a very reasonable match of the simulation results to the test data. In the HJ optimization, the Relative Step Size (RSS) is set to 0.2 (default value is 0.02) and the number of iterations is set to 1,000.

```
1)  Run the file PP_PRF_HJ3_0_Initial.zmf   This file has parameter values
    initialized to the Prony conversion numbers.   Initial sum of YSqrDiff
    is 8463.38
2)  Run the file PP_PRF_HJ3_1_1000.zmf. Final sum of YSqrDiff = 316.99
3)  Run the file PP_PRF_HJ3_2_1000.zmf. Final sum of YSqrDiff = 45.208
4)  Run the file PP_PRF_HJ3_3_1000.zmf. Final sum of YSqrDiff = 24.000

    Final error norms are:
```

$$
\begin{aligned}
\text{Objective Function} &= 24.000 \\
\text{DiffYSqr\_Rate\_100} &= 0.180 \\
\text{DiffYSqr\_Relax\_0050} &= 7.861 \\
\text{DiffYSqr\_Relax\_0075} &= 9.820 \\
\text{DiffYSqr\_Relax\_0100} &= 6.139
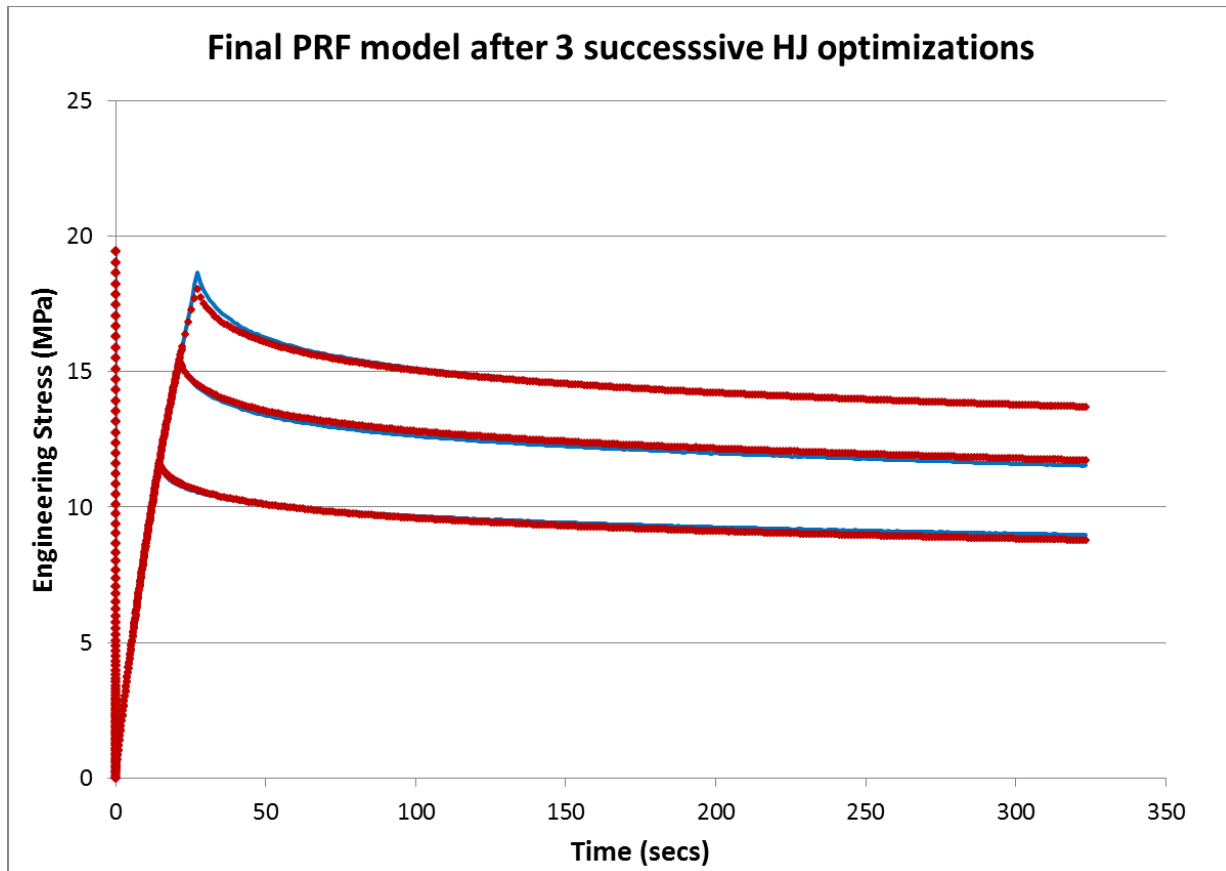\end{aligned}
$$

Figure 20, High rate and stress relaxation responses after optimization of parameters
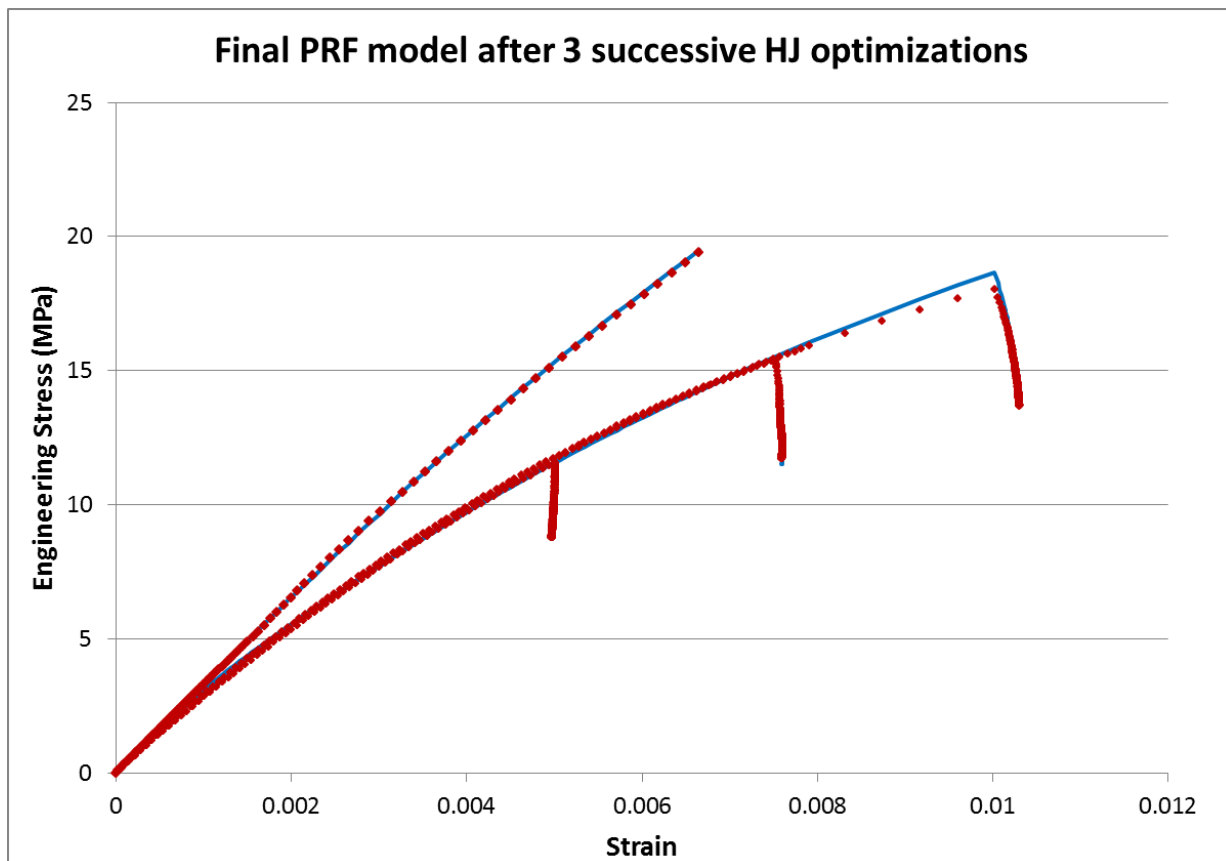

Figure 21, High rate and stress relaxation responses after optimization of parameters

Figure 20 shows the stress responses as a function of time – this is very useful to see the stress relaxation responses, but the high rate test & simulation response lie along the y-axis. Figure 21 plots the same 8 curves, but plots stress as a function of strain.

Each of the HJ optimizations took on the order of 6.5 ~ 7.5 hours of computation on a laptop computer. From the Isight job log :

Status	User	Job Name	Started	Duration	Job Size
🏁	TDK	PP_PRF_HJ3_3_1000 - 2014.08.08 15:14:18	August 8, 2014 3:14:31 PM EDT	07:33:50	201 MB
🏁	TDK	PP_PRF_HJ3_2_1000 - 2014.08.08 07:40:13	August 8, 2014 7:40:26 AM EDT	07:15:53	187 MB
🏁	TDK	PP_PRF_HJ3_1_1000 - 2014.08.07 20:27:56	August 7, 2014 8:28:06 PM EDT	06:31:30	182 MB

The resulting PRF material model looks like this:

*Material, name=PRF_model
**
*Hyperelastic, NeoHooke, Moduli=instantaneous
 549.6,   0.0
**
*Viscoelastic, Nonlinear, NetworkId=1, SRatio=0.337060, Law=strain
4.8828e-7, 3.13398, -0.551660
**
*Viscoelastic, Nonlinear, NetworkId=2, SRatio=0.150654, Law=strain
2.4414e-6, 4.91992, -0.746191
**
*Viscoelastic, Nonlinear, NetworkId=3, SRatio=0.372607, Law=strain
7.81403e-5, 3.801953, -0.616699