

Run 553 log

OK#1: ice I about 100:0 by volume, notes in lab book. Sample was prepared as a twin to a 95:5 sample coming up, then left open to room air since 12/3/07. Clocks synchronized.

New this run: My own two 180-L LN tanks, filled from other tanks from Airgas

3/4/08 1430 Ts near 223 K, still awaiting steady. Sample P to 4 MPa slowly over ~ 4 min. Just checked notes for run 550, where I did first P before running impact wrench. Here I ran the impact wrench full tilt at P = 0.

1452 Pist contact at 20 MPa: 2.6437 V. Looked pretty solid.

1501 Pist contact at 30 MPa: 2.7086 V.

1505 Pist contact at 40 MPa: 2.7516 V. Really leaky.

1511 Pist contact at 50 MPa: 2.8046 V. Just tightened up really a lot.

1517 Pist contact at 60 MPa: 2.8668

~1700 Leak just won't go away. Dismantle, tighten nut one hydraulic stroke, reassemble, still not great. Station keep o'nite, let P drift.

3/5/08 0817 Dropped to ~30 MPa o'nite, but definitely tighter this morning Set up for run, wait a while.

0849 Unloaded s gage at 60 MPa: -0.009138/-0.009132/9135.

0851 Pist contact at 60 MPa: 2.8825 V.
59.9 MPa digital Heise = 59.6 MPa analog.
Length calc: starting length at P = 0 is 2.895". Green trace disp from 20 to 60 MPa = $0.243 \text{ V} \times 0.172''/\text{V} = 0.04''$, add some for vessel modulus, subtract some for 0-20 MPa, est L0 = 2.84".

0905 (1) s set point to 24 mV (30 MPa); 223 K, P = 60 MPa.

1039 Record interval to 60 s.

1501 (from data file) Intensifier tops out.

1603 Recovering from drop in P to ~ 50 MPa when intensifier topped out and hour ago. Restroked and back up to P, but s dropped 10-15% because of contraction of vessel, overloading, and overdeforming sample. Run motot at V0.09 (was running at ~V0.01) until s comes back up, then can determine how much strain the sample suffered in the overload.

1638 s approaching setpt. From periodic e readings noted on stickies, looks like overdeformation was about 0.1%, which would have taken ~4 hrs at edot running before incident.

1743 Stop, unload.

1744 (67 s after last unload) Pist contact: 3.5590 V

1747 Unloaded s gage at 60 MPa -0.008583/-0.008572/8578.

1801 (18'15" after) Pist contact 3.5509 V
Back off piston, let P drift o'nite.

3/6/08 0928 Been back up at 60 MPa for ~55 mins.
Pist contact 3.5336 V.
0940 Piston withdrawn. Do quick leak check. At 60.0 MPa, close main
(V11).
1002 Open V11. P drops from 56.98 to 56.78. Close V11.
1053 Again: 49.69->49.65. Leave V11 open.
1059 Remove s gage, insulators, peek in. P inlet bubbling like mad.
Dumb shit. P still dropping a hair with V11 open. Close it. 49.44
MPa. Pretty much stays there.

3/5/08 0949 hrs 0.131542"/0.046318 (1)
3/5/08 1037 hrs 0.132714"/0.046730
3/5/08 1245 hrs 0.134282"/0.047282
3/5/08 1640 hrs 0.138826"/0.048883
3/5/08 1737 hrs 0.140533"/0.049484