Performance Comparison between Dreytek's Glass Flake, Standard Glass Spheres, Glass Fiber and Mica

4-21-19

Goals

- Compare physical properties between Dreytek's glass flake, standard glass spheres, mica, and glass fiber in natural PBT
  - Two grades of glass flake (FLEKA and Fineflake)
  - Single grade of mica (from Imerys)
  - Single grade of glass spheres (from Potters)
  - Single grade of glass fiber (from JohnsMannville)
- Identify best grade of glass flake and amount that can mimic the same molding behavior as mica, glass spheres, and glass fiber
- Once grade and amount has been identified, investigate other physical properties

Project Plan

- Compound PBT with 10 and 30 wt% of the following: **Mica, glass fiber, glass spheres, and glass flake**
  - IFC has a standard glass screw that is used to maintain the shape/size of glass but distributes the particles effectively in the polymer matrix

- Injection mold into standard specimens and measure mold shrinkage according to ASTM D955
  - Also look at impact, tensile strength, tensile modulus, weld line strength, creep, water absorption, heat deflection temperature, and spiral flow properties

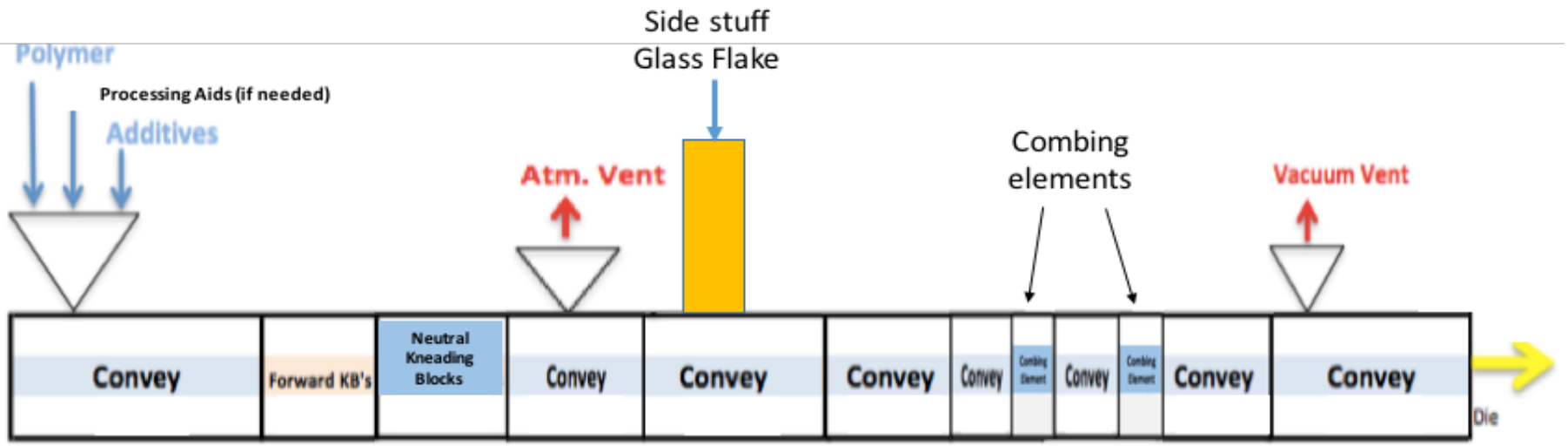
Materials

- Polybutylene terephthalate (PBT)
  - Supplied by IFC
  - Celanex Thermoplastic Polyester
- Glass Flake
  - Supplied by Dreytek
  - Two sizes: FLEKA® REFG-101, FINEFLAKE™ MEG160FY-M01
- Mica
  - Supplied by IFC
  - Standard grade: Imerys WG325 (34 micron)
- Glass spheres
  - Supplied by IFC
  - Standard grade: Potters Spheriglass 1922 w/CP-02 coating (~200 micron)
- Glass Fiber
  - Supplied by IFC
  - Standard grade: ThermoFlow 600+ (4 mm and 10 micron)

Formulations

Sample	PBT	FLEKA® REFG-101	FINEFLAKE™ MEG160FY-M01	Mica WG325	Spheriglass 1922 w/ CP-02 Coating	ThermoFlow 600+
1	100					
2	90	10				
3	70	30				
4	90		10			
5	70		30			
6	90			10		
7	70			30		
8	90				10	
9	70				30	
10	90					10
11	70					30
12	70	10				20
13	70		10			20
14	70			10		20
15	70				10	20

Extrusion Set-Up for Running Glass Flake (40:1 L/D)



Barrel 1: Deep flight forward convey elements
Barrel 2: Normal forward convey followed by forward kneading blocks (1 x 30 and 1 x 60)
Barrel 3/4: Neutral kneading blocks (7 x 90)
Barrel 4: Forward Convey, back vent
Barrel 5: Side stuffer with deep flight forward conveying elements
Barrel 6: Forward convey
Barrel 7: Forward convey followed by combing element at the end (2 x 30)
Barrel 8: One forward convey followed by combing element (2 x 30) followed by forward convey
Barrel 9-10: Forward convey
Die: 7 hole 3 or 4 mm die, no screen pack or through ring

Mold Shrinkage (ASTM D955 – 24 hr)

Sample	Pre-Annealed Transverse Direction (TD) %	Pre-Annealed Flow Direction (FD) %	Post-Annealed Transverse Direction (TD) %	Post-Annealed Flow Direction (FD) %	
1	1.42 +/- 0.02	2.09 +/- 0.41	1.56 +/- 0.02	2.22 +/- 0.31	
2	0.79 +/- 0.02	1.08 +/- 0.03	0.81 +/- 0.01	1.15 +/- 0.05	
3	0.03 +/- 0.01	0.39 +/- 0.09	0.10 +/- 0.01	0.43 +/- 0.10	← 30 wt% glass flake
4	1.05 +/- 0.02	1.26 +/- 0.03	1.23 +/- 0.02	1.34 +/- 0.08	
5	0.26 +/- 0.02	0.58 +/- 0.10	0.55 +/- 0.01	0.68 +/- 0.02	
6	0.94 +/- 0.01	1.38 +/- 0.17	1.05 +/- 0.01	1.50 +/- 0.03	
7	0.13 +/- 0.01	0.59 +/- 0.12	0.08 +/- 0.01	0.84 +/- 0.25	
8	1.42 +/- 0.01	1.99 +/- 0.05	1.44 +/- 0.01	2.07 +/-0.05	
9	1.00 +/- 0.01	1.79 +/- 0.17	1.10 +/- 0.01	1.87 +/- 0.17	
10	1.18 +/- 0.01	0.54 +/- 0.05	1.39 +/- 0.01	0.51 +/- 0.06	
11	0.94 +/- 0.02	0.02 +/- 0.02	1.10 +/- 0.02	0.08 +/- 0.04	← 30 wt% glass fiber
12	0.55 +/- 0.02	0.03 +/- 0.03	0.87 +/- 0.01	0.22 +/- 0.17	← 20 wt% glass fiber /10 % glass flake
13	0.84 +/- 0.02	0.11 +/- 0.04	0.97 +/- 0.01	0.31 +/- 0.20	
14	0.73 +/- 0.01	0.19 +/- 0.09	0.81 +/- 0.02	0.39 +/- 0.14	
15	0.92 +/- 0.01	0.10 +/- 0.02	1.15 +/- 0.01	0.17 +/- 0.07	

Shrinkage Testing Notes

- REFG-101 grade seems to perform better than MEG160-FY at 10 and 30 wt% loading
  - Much lower shrinkage in both TD and FD
- Glass flake shows much lower TD shrinkage than glass fiber (almost zero shrinkage at 30 wt% loading!)
  - FD shrinkage is lower for glass fiber due to fiber alignment
- REFG-101 grade shows lower shrinkage in both TD and FD compared to mica and glass beads
- Combination of glass fiber and glass flake can almost reach isotropic shrinkage in both TD an FD (potential to reach zero shrinkage with right combination?)
  - Big selling point as to why using combination of both fiber and flake!!!!!

Tensile Properties (ASTM D638)

Sample	Tensile Strength (psi)	Elongation at Break (%)	Tensile Modulus (psi)	
1	7,800 +/- 280	12.2 +/- 0.7	348,800 +/- 6200	
2	10,150 +/- 100	3.1 +/- 0.1	643,700 +/- 13,000	
3	13,030 +/- 180	2.2 +/- 0.1	1,284,200 +/- 20,300	
4	9,090 +/- 110	3.7 +/- 0.5	563,200 +/- 11,400	
5	10,250 +/- 60	2.2 +/- 0.1	1,022,000 +/- 11,700	
6	8,500 +/- 60	2.9 +/- 0.1	601,300 +/- 12,100	
7	9,320 +/- 140	1.4 +/- 0.1	1,252,800 +/- 30,700	
8	7,870 +/- 70	7.9 +/- 4.3	419,800 +/- 11,500	
9	6,740 +/- 10	1.8 +/- 0.1	576,300 +/- 9400	
10	11,900 +/- 100	3.4 +/- 0.3	634,600 +/- 16,800	
11	17,300 +/- 240	2.5 +/- 0.1	1,329,300 +/- 14,300	← 30 wt% glass fiber
12	16,930 +/- 40	2.5 +/- 0.1	1,385,400 +/- 13,500	← 20 wt% glass fiber /10 % glass flake
13	15,890 +/- 180	2.7 +/- 0.1	1,260,300 +/- 16,700	
14	14,420 +/- 180	2.4 +/- 0.1	1,290,800 +/- 21,100	
15	14,470 +/- 120	2.7 +/- 0.1	1,079,300 +/- 12,300	

Tensile Testing Notes

- Like impact, REFG-101 grade seems to perform better than MEG160-FY at 10 and 30 wt% loading
- REG-101 grade shows comparable results to glass fiber in terms of modulus and elongation at break
  - Lower tensile strength than glass fiber but much better performance over mica and glass spheres
- Combination of glass fiber and glass flake is better than with glass fiber/mica or glass fiber/glass spheres
- Overall, depending on the other measured properties, there could be a synergy of using flake and fiber to achieve optimal performance/cost

Flexural Properties (ASTM D790)

Sample	Flexural Strength (psi)	Flexural Modulus (psi)
1	12,070 +/- 290	346,600 +/- 7,300
2	16,680 +/- 330	565,000 +/- 14,300
3	20,190 +/- 150	1,078,500 +/- 9,700
4	15,000 +/- 170	499,400 +/- 3,700
5	17,190 +/- 110	878,100 +/- 7,500
6	14,410 +/- 110	576,800 +/- 5,800
7	14,090 +/- 200	1,110,200 +/- 16,100
8	13,570 +/- 160	423,800 +/- 5,400
9	12,340 +/- 110	563,900 +/- 6,200
10	18,000 +/- 200	529,300 +/- 9,600
11	26,260 +/- 490	1,143,500 +/- 29,100
12	24,930 +/- 350	1,122,400 +/- 32,000
13	23,780 +/- 400	1,040,400 +/- 24,100
14	21,710 +/- 180	1,079,500 +/- 27,000
15	22,400 +/- 140	919,000 +/- 10,400

← 30 wt% glass fiber

← 20 wt% glass fiber /10 wt % glass flake

Flexural Testing Notes

- Like impact, REFG-101 grade seems to perform better than MEG160-FY at 10 and 30 wt% loading
- REG-101 grade shows slightly lower results to glass fiber in terms of modulus and strength
  - However, still shows better performance over mica and glass spheres
- Combination of glass fiber and glass flake is better than with glass fiber/mica or glass fiber/glass spheres
  - Properties comparable to glass fiber by itself

Notched and Unnotched Izod (ASTM D256)

Sample	Notched Izod (ft-lb/in)	Unnotched Izod (ft-lb/in)	
1	0.45 +/- 0.01	> 16	
2	0.60 +/- 0.16	11.4 +/- 0.27	
3	0.57 +/- 0.02	8.52 +/- 0.41	
4	0.46 +/- 0.01	3.92 +/- 0.16	
5	0.52 +/- 0.01	6.21 +/- 0.25	
6	0.47 +/- 0.01	6.11 +/- 0.22	
7	0.43 +/- 0.03	3.42 +/- 0.24	
8	0.45 +/- 0.01	11.9 +/- 0.86	
9	0.49 +/- 0.01	4.22 +/- 0.07	
10	0.67 +/- 0.06	6.40 +/- 0.23	
11	1.4 +/- 0.10	11.6 +/- 0.39	← 30 wt% glass fiber
12	1.2 +/- 0.07	10.5 +/- 0.53	← 20 wt% glass fiber /10 wt % glass flake
13	1.2 +/- 0.06	10.6 +/- 0.41	
14	1.0 +/- 0.07	9.21 +/- 0.26	
15	1.1 +/- 0.04	10.0 +/- 0.50	

Impact Testing Notes

- REFG-101 grade seems to perform better than MEG160-FY at 10 and 30 wt% loading
- Glass flake shows better impact than both mica and glass spheres at higher loadings
- Glass fiber still shows best impact performance at 30 wt% loading
  - Combination of glass fiber and glass flake is better than with glass fiber/mica or glass fiber/glass spheres
- Overall, depending on the other measured properties, there could be a synergy of using flake and fiber to achieve optimal performance/cost

Heat Deflection Temperature (ASTM D648)

Sample	0.45 MPa (oC)	1.8 MPa (oC)
1	108.4 +/- 6.2	47.6 +/- 1.1
2	131.4 +/- 10.7	79.6 +/- 2.9
3	190.6 +/- 4.8	150.1 +/- 10.5
4	155.8 +/- 0.4	104.5 +/- 0.2
5	178.2 +/- 6.5	138.7 +/- 10.2
6	149.0 +/- 7.4	63.7 +/- 1.2
7	185.4 +/- 1.3	126.8 +/- 2.2
8	144.5 +/- 11.0	64.6 +/- 7.1
9	149.9 +/- 8.8	99.3 +/- 0.1
10	192.4 +/- 10.7	140.5 +/- 10.1
11	>200	196.4 +/- 6.0
12	>200	188.8 +/- 0.4
13	>200	173.1 +/- 13.9
14	>200	180.3 +/- 4.2
15	>200	196.1 +/- 7.6

← 30 wt% glass fiber

← 20 wt% glass fiber /10 wt % glass flake

Heat Deflection Notes

- Like all mechanical properties , REFG-101 grade seems to perform better than MEG160-FY at 10 and 30 wt% loading

- REG-101 grade shows comparable results to mica at both pressures
  - Glass beads has very little improvement in HDT of PBT

- Glass fiber shows best HDT performance but combination of glass fiber with mica and/or glass flake shows comparable performance

Weld Strength

Sample	Tensile Modulus (psi)	Tensile Strength (psi)	Elongation at Break (%)	
1	15,900 +/- 1840	170 +/- 80		
2	29,000 +/- 1,840	280 +/- 80	1.1 +/- 0.3	
3	50,670 +/- 9,060	230 +/- 170	0.5 +/- 0.3	← 30 wt% glass flake
4	23,410 +/- 1,870	190 +/- 60	0.8 +/- 0.2	
5	40,930 +/- 2,950	260 +/- 70	0.7 +/- 0.2	
6	25,220 +/- 2,920	240 +/- 40	1.1 +/- 0.2	
7	42,160 +/- 22,320	170 +/- 110	0.4 +/- 0.1	
8	18,110 +/- 2,430	140 +/- 70	0.8 +/- 0.3	
9	24,660 +/- 1,150	180 +/- 50	0.9 +/- 0.3	
10	29,680 +/- 1,260	360 +/- 30	1.3 +/- 0.2	
11	54,790 +/- 1,110	250 +/- 60	0.5 +/- 0.1	← 30 wt% glass fiber
12	56,930 +/- 2,180	350 +/- 60	0.7 +/- 0.1	← 20 wt% glass fiber /10 wt % glass flake
13	65,460 +/- 2,153	600 +/- 40	1.3 +/- 0.1	←
14	42,470 +/- 7,250	280 +/- 100	0.8 +/- 0.2	
15	46,820 +/- 3,240	410 +/- 30	1.0 +/- 0.1	

Weld Strength Notes

- Appears to be a synergistic effect of using the combination of glass fiber with glass flake in terms of weld strength (compare samples 3, 5 and 11,12, and 13)

Melt Flow Index (ASTM D1238)

Sample	Melt Flow Index (g/10 min @ 260 °C)	
1	50.1	
2	34.6	
3	31.7	← 30 wt% glass flake
4	40.6	
5	29.6	
6	36.3	
7	27.8	
8	47.3	
9	20.1	
10	28.3	
11	20.9	← 30 wt% glass fiber
12	19.2	← 20 wt% glass fiber /10 wt % glass flake
13	18.0	
14	22.9	
15	22.1	

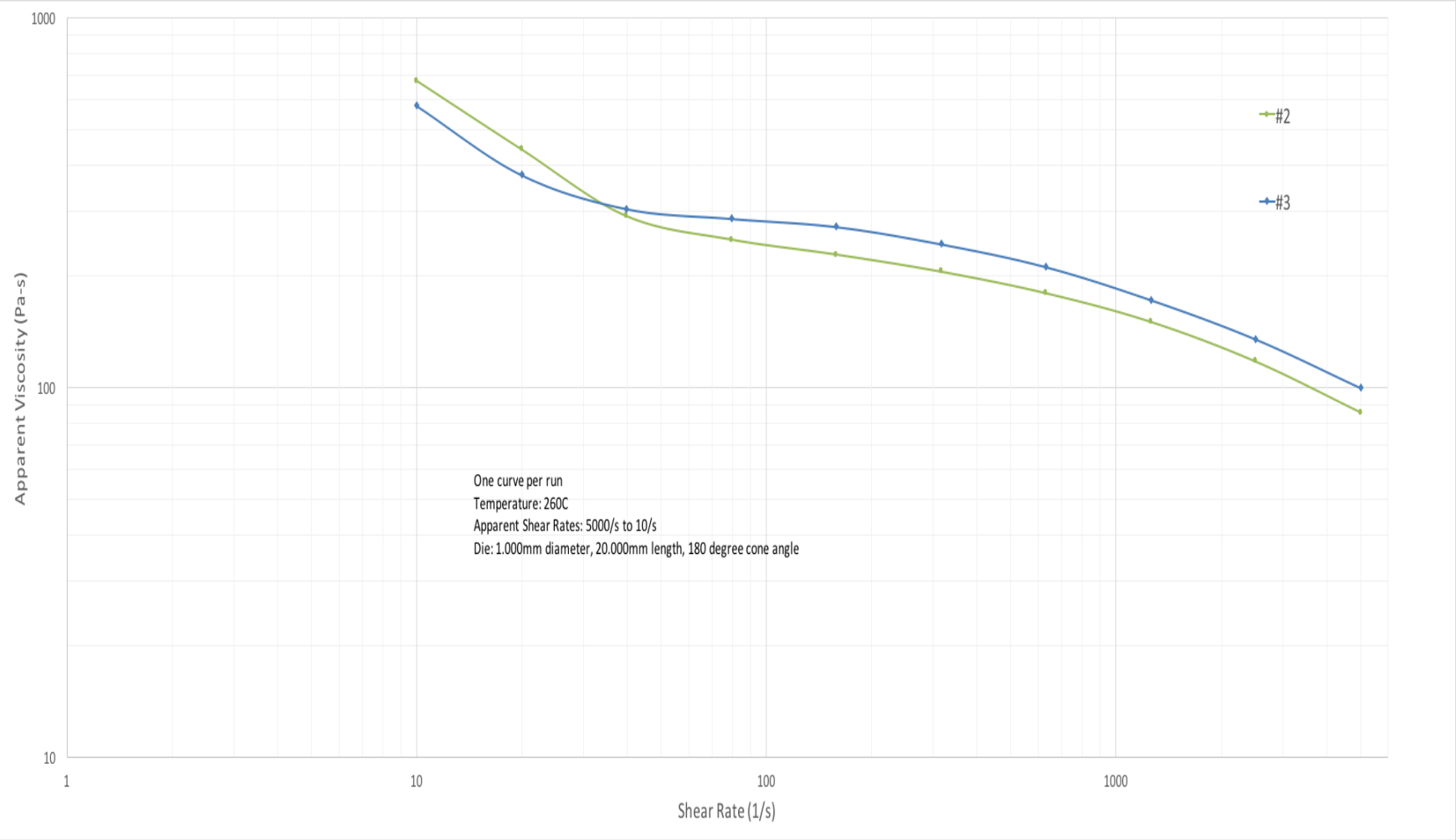
Melt Flow Notes

- See improved flow in glass flake samples over glass fiber
  - Glass spheres show best flow at lower loadings while glass flake shows best flow at higher loadings
- Addition of glass flake/fiber (sample #12) shows slightly lower MFI to glass fiber (sample #11)
  - Rheology data suggests that glass flake samples help flow at high shear rates (see rheology curves)

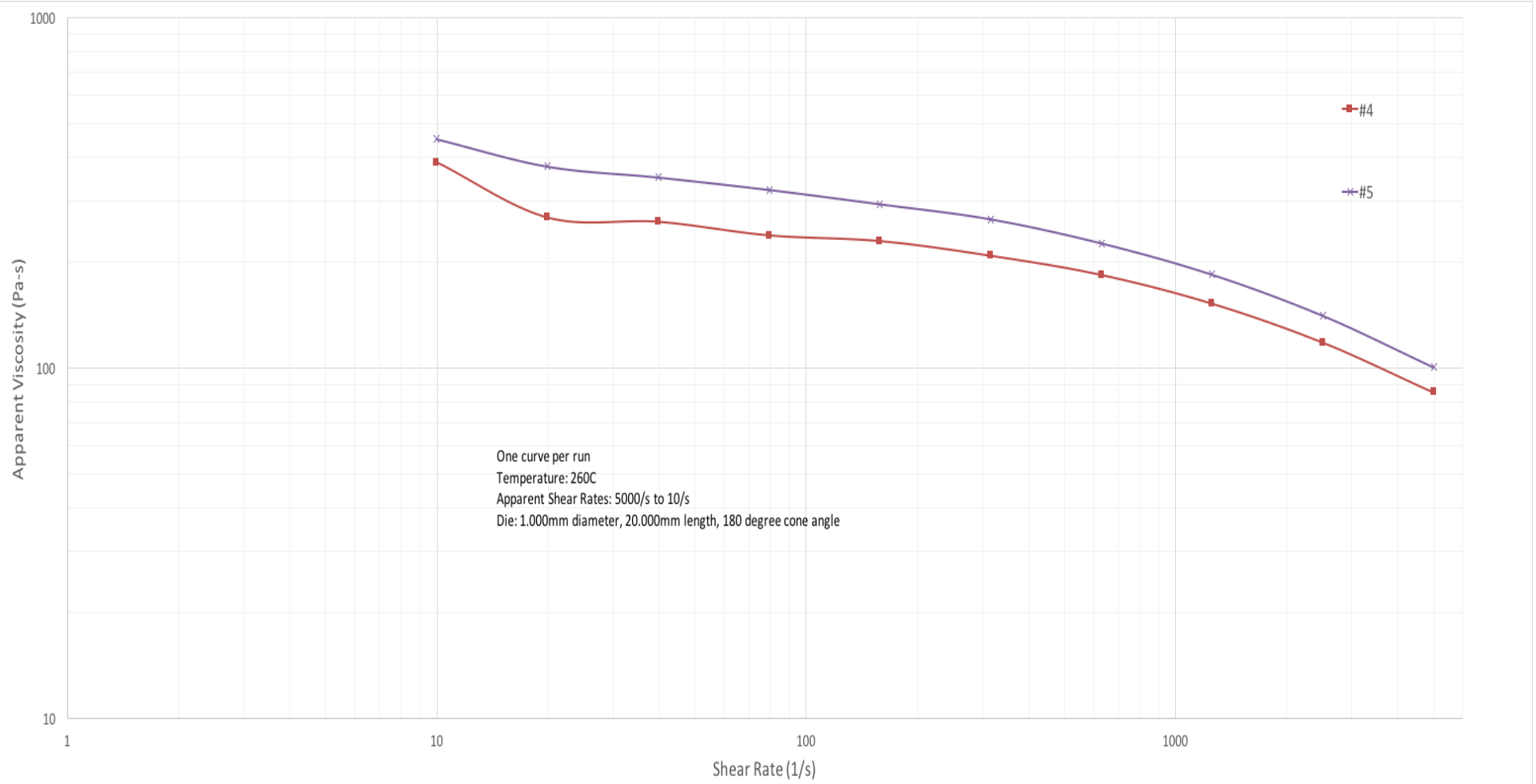
Shear Rate Viscosity

Sample	Low shear (30 1/s)	High Shear (5000 1/s)	
1	226	86	
2	391	99	
3	384	111	← 30 wt% glass flake
4	295	93	
5	383	107	
6	307	92	
7	401	105	
8	242	100	
9	547	136	
10	408	92	
11	535	104	← 30 wt% glass fiber
12	577	100	← 20 wt% glass fiber /10 wt % glass flake
13	603	110	
14	487	107	
15	506	113	

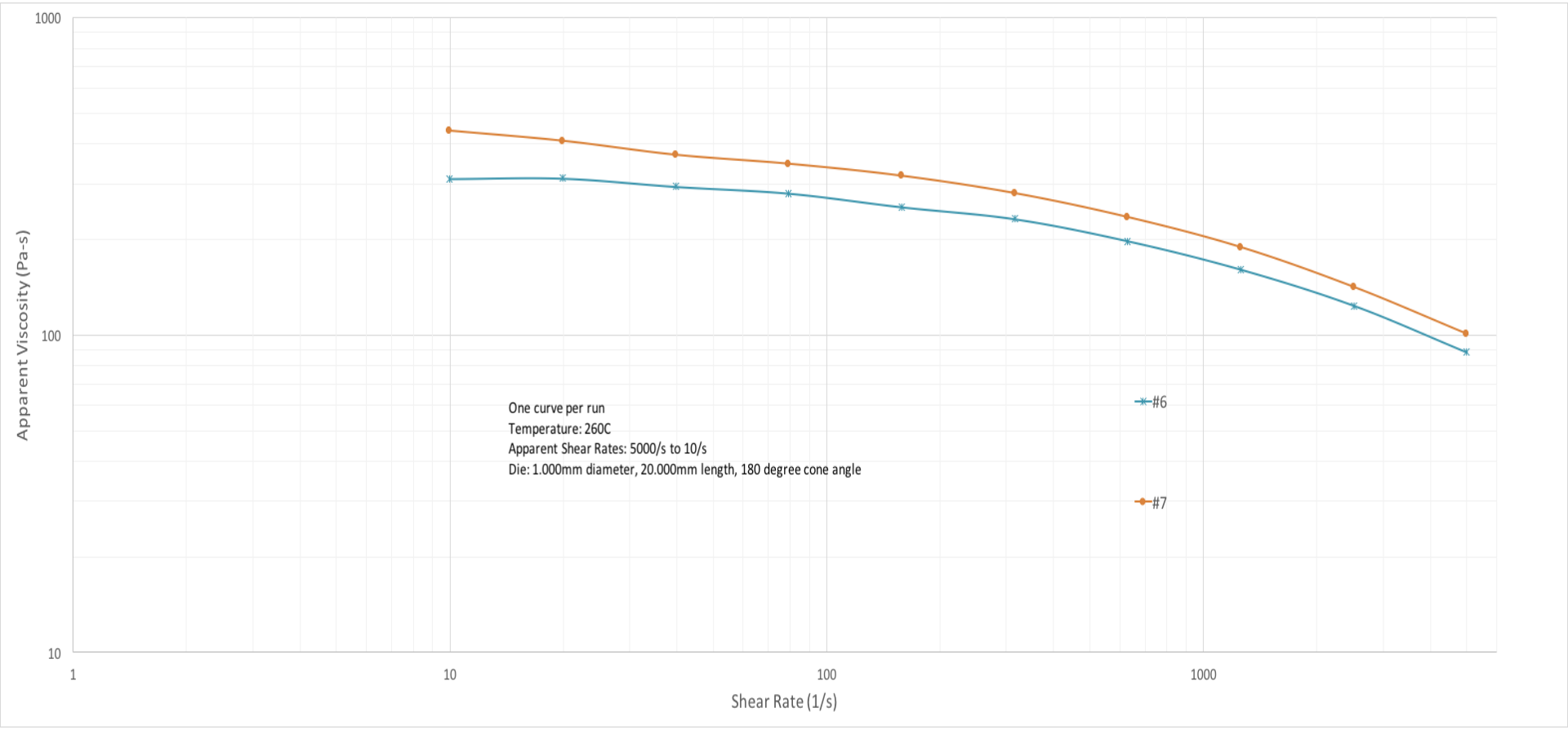
Rheology Curves (Samples 2 and 3)



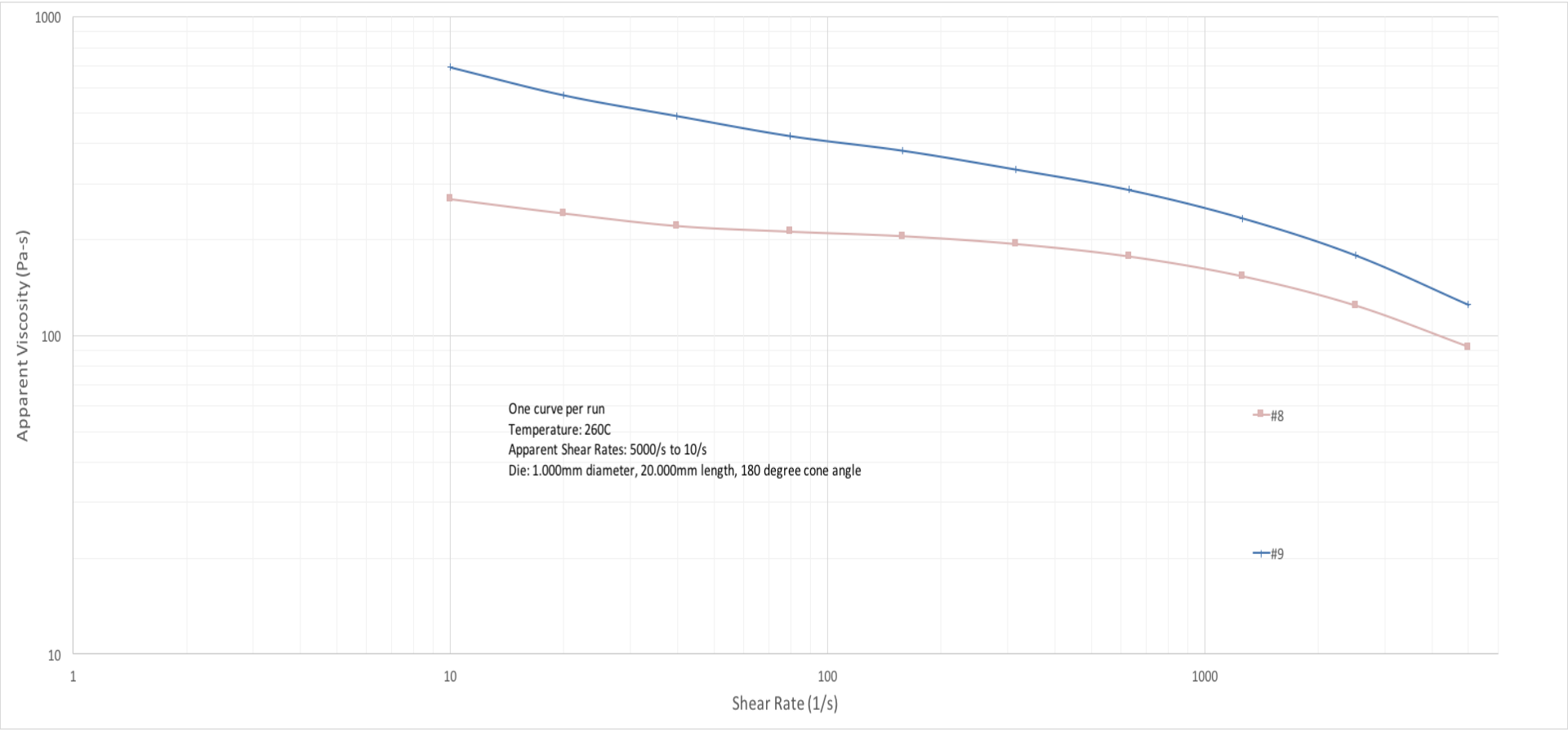
Rheology Curves (Samples 4 and 5)



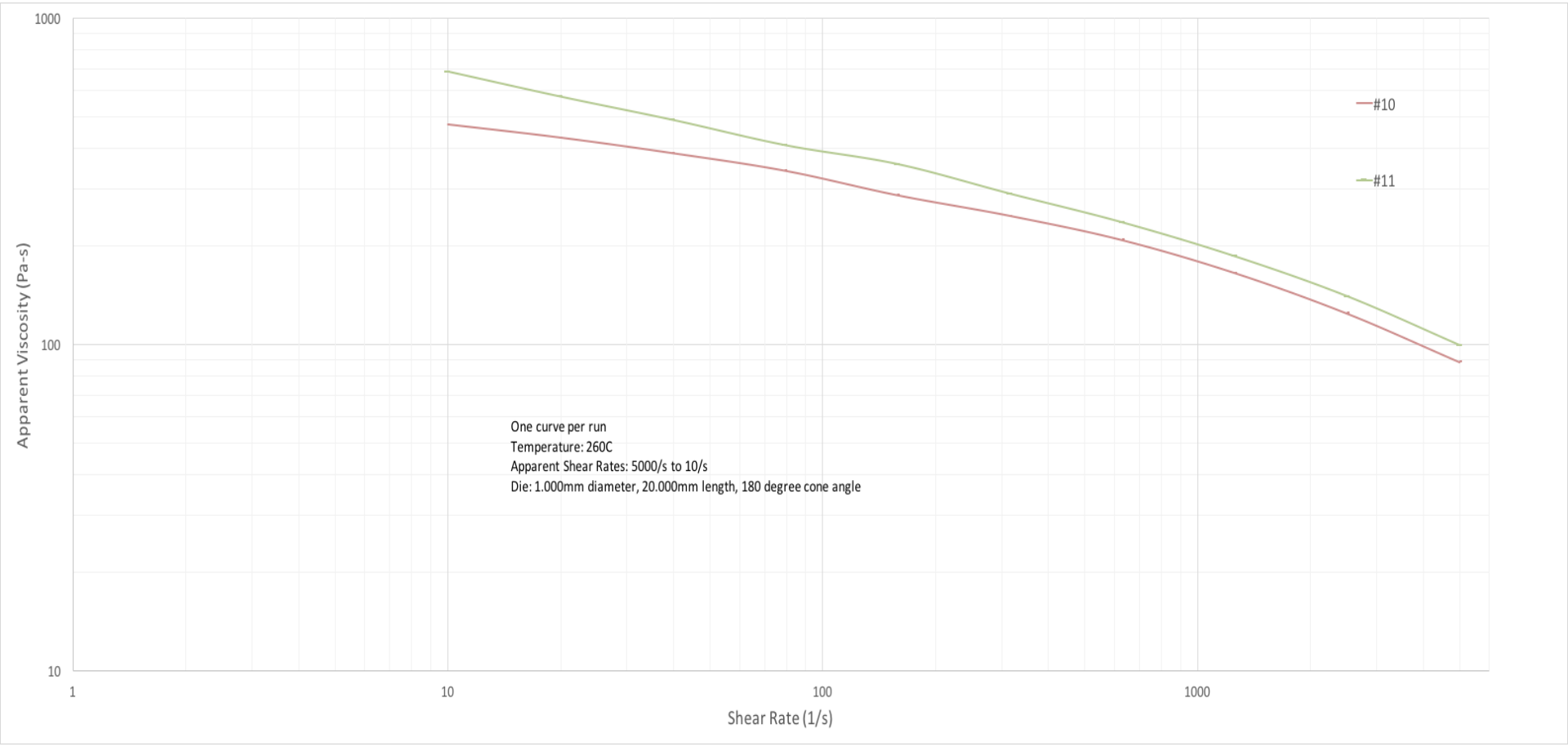
Rheology Curves (Samples 6 and 7)



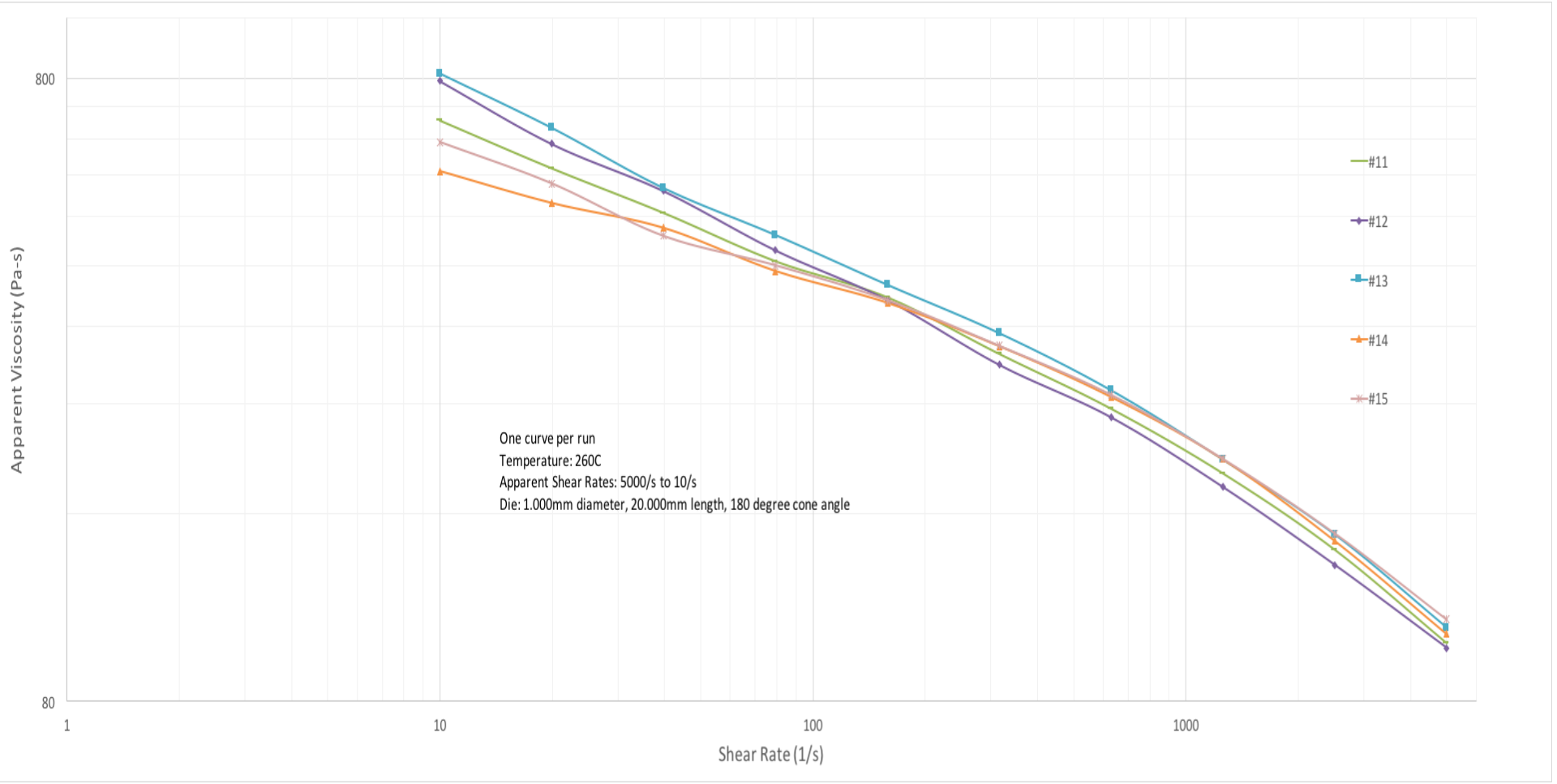
Rheology Curves (Samples 8 and 9)



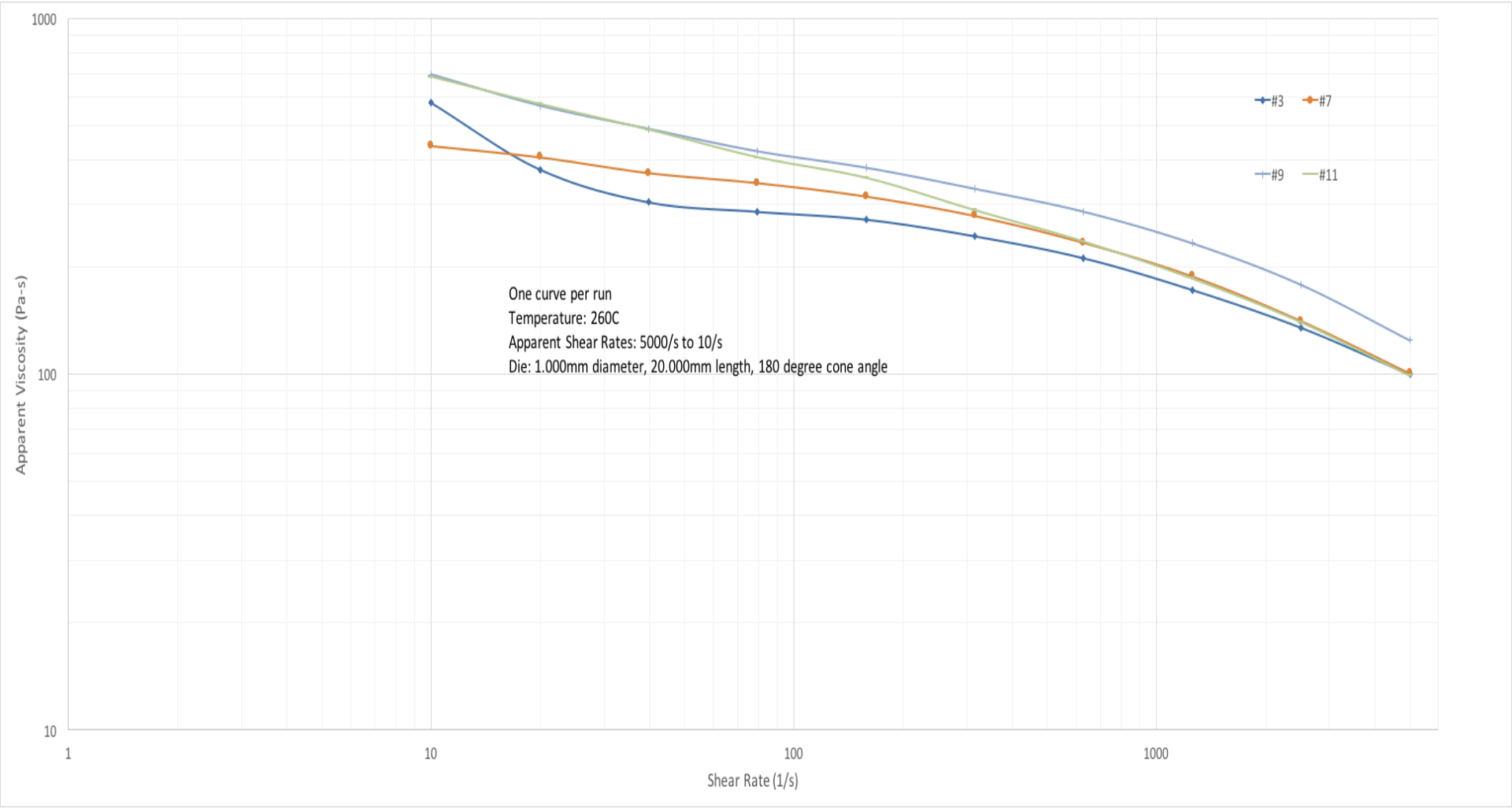
Rheology Curves (Samples 10 and 11)



Rheology Curves (Samples 11,12,13,14,15)



Rheology Curves (Samples 3,7,9,11)



Rheology Notes

- Viscosity flow of glass flake filled systems is very irregular at low shear rates
  - High shear rates, flow is very good and looks to exhibit highest shear thinning behavior

- Addition of glass flake to glass fiber (sample #12) shows higher shear thinning behavior than glass filled PBT (sample #11)
  - Suggest samples should flow easier in an injection molding application

Water Absorption (ASTM D570)

Sample	24 hours (% wt)	48 hours (% wt)	168 hours (% wt)
1	0.15 +/- 0.07	0.15 +/- 0.02	0.23 +/- 0.11
2	0.25 +/- 0.10	0.15 +/- 0.08	0.29 +/- 0.09
3	0.12 +/- 0.24	0.08 +/- 0.19	0.10 +/- 0.13
4	0.22 +/- 0.16	0.17 +/- 0.05	0.19 +/- 0.04
5	0.19 +/- 0.11	0.06 +/- 0.08	0.17 +/- 0.10
6	0.15 +/- 0.12	0.07 +/- 0.08	0.12 +/- 0.03
7	0.10 +/- 0.14	0.06 +/- 0.08	0.22 +/- 0.04
8	0.00 +/- 0.22	0.04 +/- 0.22	0.09 +/- 0.23
9	0.00 +/- 0.03	0.08 +/- 0.04	0.02 +/- 0.10
10	0.07 +/- 0.09	0.07 +/- 0.05	0.06 +/- 0.04
11	0.07 +/- 0.11	0.00 +/- 0.07	0.10 +/- 0.15
12	0.00 +/- 0.23	0.00 +/- 0.13	0.00 +/- 0.20
13	0.25 +/- 0.22	0.09 +/- 0.06	0.21 +/- 0.06
14	0.16 +/- 0.06	0.13 +/- 0.06	0.14 +/- 0.07
15	0.11 +/- 0.13	0.00 +/- 0.10	0.01 +/- 0.21

← 30 wt% glass fiber

← 20 wt% glass fiber /10 wt % glass flake

Water Absorption Notes

- Essentially very little to no water uptake in all these samples over 24, 48, and 168 hours

Density Measurements (ASTM D792)

Sample	Density (g/cm^3)	
1	1.312 +/- 0.002	
2	1.381 +/- 0.001	
3	1.532 +/- 0.002	
4	1.382 +/- 0.001	
5	1.537 +/- 0.005	
6	1.389 +/- 0.001	
7	1.560 +/- 0.002	
8	1.379 +/- 0.001	
9	1.532 +/- 0.001	
10	1.384 +/- 0.001	
11	1.544 +/- 0.001	← 30 wt% glass fiber
12	1.546 +/- 0.004	← 20 wt% glass fiber /10 wt % glass flake
13	1.547 +/- 0.001	
14	1.550 +/- 0.001	
15	1.536 +/- 0.001	

Density

- Density are within standard deviation of 10 and 30 wt% loading levels