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ARTICLE

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Rheological behavior of neat and carbon fiber-reinforced poly(ether ketone ketone) for extrusion deposition additive manufacturing

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Abstract

To develop new materials for extrusion additive manufacturing (AM) systems, a fundamental understanding of rheological properties is essential to correlate the effect of processing on material structure and its properties. In this work, the rheological properties of five different grades of neat and carbon fiber (CF)-reinforced poly(ether ketone ketone) are reported. Rheological properties are essential to understand the effect of reinforcing fibers and AM process parameters such as time, temperature, environment, and shear rate on flow behavior during processing. Small-amplitude oscillatory shear tests and steady shear tests indicated neat grades to exhibit less increase in viscosity over time when processed in air than the CF-filled grades. The filled grades showed greater shear thinning and lower sensitivity to temperature. Overall, this rheological analysis provides a broad framework for determining appropriate processing conditions for extrusion deposition AM of such high-temperature polymer systems.

1 | INTRODUCTION

Polymer additive manufacturing (AM) or the 3D printing process, such as material extrusion, has been used for printing a variety of thermoplastics and fiber-reinforced composites [1–4]. Scaling up of this process has been possible with the development of large-format extrusion deposition print platforms such as the Big Area Additive Manufacturing (BAAM) system. The BAAM system, with a build volume measuring 6 m x 2.4 m x 1.8 m and a

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temperature capability of up to 500°C, enables printing using pelletized feedstock of a variety of low- and high-temperature thermoplastics and composites at rates as high as 50 kg/hr, making the process faster and cheaper when compared to existing material extrusion systems [5–8]. These large-format systems not only offer the advantage of geometric flexibility but also enable the production of functionally graded components, thereby offering the potential to optimize cost and performance of 3D-printed components [8]. Some materials that have been used to print components on this system include unreinforced and fiber-reinforced composites of thermoplastics, such as acrylonitrile butadiene styrene (ABS) [6,7] and polylactic acid, as well as high-performance amorphous thermoplastics, such as poly (phenyl sulfone) (PPSU) [5,9], poly(ether imide) (PEI) [10,11], and semicrystalline matrices such as poly(phenylene sulfide) (PPS) [12]. In order to fully realize the potential of such large-format print systems to print components for newer applications, there is a need to develop high-performance materials that can be processed on these large-scale AM systems.

The development of new materials that are compatible with large-scale printing requires an understanding of the relationship between the process, polymer structure, and properties during deposition, as well as for postdeposition conditions. The BAAM process uses a single-screw extruder to melt and extrude the composite material feedstock in the form of precompounded pellets. The melt temperature is controlled by setting the temperatures at the three heating zones and the extruder tip. Some of the key parameters that can be controlled prior to and during the melt deposition process are processing temperature, processing environment (in the extruder), shear rates encountered during extrusion (by varying the screw speed, flow rate, and extruder nozzle geometry), and the intrinsic material properties of the chosen feed material, such as the chemical structure and filler composition. Prior to processing new materials, a systematic approach to understanding the effect of these parameters on the material's thermal and melt flow properties is essential. This would not only provide a guideline for identifying appropriate print conditions for new materials that have not previously been printed but also enables prediction of flow instabilities, chemical reactions, etc. that could possibly occur during printing. This approach, which is applicable to any extrusion deposition AM platform, is especially advantageous for large-scale systems to help lower the risk of system downtime and material waste.

The first step is to define the appropriate upper and lower bounds of the temperature window for deposition by characterizing the thermal properties of the feed material. The upper temperature limit is the degradation onset temperature (DOT), which can be identified using thermogravimetric analysis (TGA). The lower temperature limit is defined by the melting temperature, T_m, for semicrystalline polymers, which can be determined using the differential scanning calorimetry (DSC) technique. Upon determining an appropriate temperature range for processing, rheological characterization of feed materials is performed across various temperatures, shear rates, and processing environments (air or inert gas). Controlled rheological characterization enables the understanding of viscoelastic behavior of the chosen materials, which plays an important role in the ability to melt extrude and deposit these materials, as well as meet the postdeposition functional requirements. The aforementioned approach has been reported in previous efforts by the authors on polymers such as PPSU, PEI, and PPS [9,10,13]. The work discussed in this paper focuses on understanding the melt rheological characteristics of a semicrystalline high-performance thermoplastic, poly(ether ketone ketone) (PEKK) and its carbon fiber (CF)-reinforced composites for an extrusion deposition AM process. A comprehensive analysis to understand the effect of six different parameters: time, temperature, shear rate, environment, chemical structure, and filler loading on melt rheological properties of PEKK materials has been conducted and reported in this work.

1.1 | Background on PEKK

PEKK is a semicrystalline thermoplastic in the family of poly(aryl ether ketones) (PAEKs), which are high-performance thermoplastics with applications in areas such as aerospace and automotive components, medical applications, electronics, and oil and gas exploration [14–18]. These polymers offer properties such as superior mechanical performance, chemical resistance, and high service temperatures, primarily determined by the regularity and rigidity of their backbone chain formed by aryl, keto, and ether linkages. The thermophysical properties

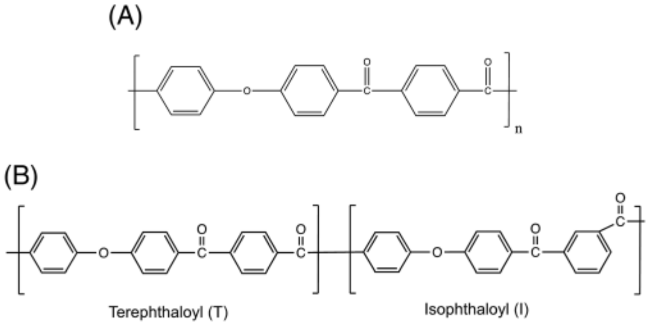

FIGURE 1 A, Structure of PEKK homopolymer. B, PEKK copolymer

of these polymers are determined by the ratio and sequence of keto to ether linkages. Increasing the keto linkages increases the rigidity of the monomer, leading to a higher glass transition temperature (T_g) [19,20]. In addition, this also increases the interaction and packing of the aryl groups, thereby increasing the crystal binding energy and melting temperature (T_m) [17,19]. Among the various polymers in the PAEK family, the PEKK homopolymer has a relatively high level (~ 67%) of keto linkages (structure as shown in Figure 1A with all paralinkages) and therefore exhibits a high melting temperature of about 400°C, which is in the vicinity of the degradation temperature of most PAEK polymers. This increases the difficulty of melt processing materials such as PEKK without degradation [17,19]. A typical approach to significantly lower the melting temperature of PEKK (and thereby the processing temperature) is to synthesize the material as a copolymer by introducing meta-linkages in the backbone chain, such as the structure shown in Figure 1B [21]. By varying the ratio of paralinkage (terephthaloyl) and metalinkage (isophthaloyl), known as the T/I ratio, thermophysical properties and crystallization behavior of PEKK can be significantly modified. Lowering the proportion of terephthaloyl groups lowers the rate of crystallization, as well as the melting point, and the grade with an appropriate T/I ratio can be chosen depending on the desired application [17]. In addition, reinforcing PEKK with fillers such as short CF can enhance the mechanical properties and lower the coefficient of thermal expansion, both of which are beneficial for printing such semicrystalline materials on BAAM with reduced warping [7].

2 | EXPERIMENTAL

2.1 | Materials

Five different grades of PEKK, procured from Arkema Inc., King of Prussia, Pennsylvania, USA were used in this work. The samples were obtained in the form of precompounded pellets for all analysis. The sample set comprised of three neat grades, with T/I ratios of 60/40, 70/30, and 80/20 and two short CF-reinforced grades (CF 30 wt.% and CF 40 wt.%) using the 80/20 matrix. Prior to all tests, the pellets were dried at 120°C for 6 to 8 hours in a vacuum oven. Table 1 lists some of the thermal properties of the selected grades. The DOT, T_g, and T_m values were obtained from previous TGA and DSC analysis by the authors [13,27–29].

TABLE 1 Feed materials properties

PEKK grade	Density at 23°C (g/cc)[22–26]	DOT (°C)	T_g (°C)	T_m (°C)
Neat 60/40	1.27	~ 500	158	288
Neat 70/30	1.29		161	335
Neat 80/20	1.29		158	361
80/20 30CF	1.39		159	360
80/20 40CF	1.45		158	358

2.2 | Rheological analysis

2.2.1 | Small-amplitude oscillatory shear (SAOS) tests

Small-amplitude oscillatory shear (SAOS) tests were performed on a TA Instruments Discovery Hybrid Rheometer (DHR-2) system, fitted with a parallel plates fixture (25 mm diameter). After drying, the pellets were melted between the plates directly using melt rings, followed by setting a gap in the range of 1.4 to 2 mm. Tests were conducted using a fresh sample each time at the chosen candidate test temperatures to determine the effect of temperature, environment, time, shear, and fillers on the viscoelastic properties of the melts. At least two tests were performed for each case to check for repeatability. The candidate test temperatures were 335°C and 350°C for the neat 60/40 grade, 350°C and 375°C for the neat 70/30 grade, and 375°C and 390°C for the three 80/20 grades (chosen to be at least 15°C above the identified T_m).

First, the linear viscoelastic regime (LVE), that is, oscillatory strain up to which there is no significant variation in storage modulus (G'), was determined with amplitude sweep tests in which the melted material was subjected to oscillatory shear of varying amplitudes (0.001-100%) at a constant frequency (ω) of 10 rad/s, and the changes in dynamic moduli with oscillatory strain were monitored. Upon selecting a suitable strain value in the LVE, isothermal time sweep tests were performed at a constant angular frequency (10 rad/s) for 10 minutes to determine the variation of dynamic moduli (storage modulus G' and loss modulus G'') and complex viscosity (η^*) with time. Variations in viscoelastic properties with time are indicative of possible structural changes taking place in the material and can be used to determine thermal stability. All amplitude and time sweep tests were performed with air as the test environment.

Depending on the thermal stability of the samples in air, frequency sweep tests were performed in either an air or nitrogen environment at all the indicated test temperatures to determine the variation in dynamic rheological properties with frequency. In order to minimize the thermal effects of test duration on the studied properties, frequency sweep tests were performed twice with fresh samples, once sweeping from 628 to 0.1 rad/s (high-to-low) and then from 0.1 to 628 rad/s (low-to-high), and the average values are reported.

2.2.2 | Steady shear tests

Steady shear tests were performed on a Dynisco LCR 7001 capillary rheometer to understand the effect of high shear rates on melt viscosity of the chosen materials. The test setup included a die with an orifice diameter of 0.75 mm, L/D of 20, and entrance angle of 120°. Each test involved heating about 10 g of the pellets in the barrel for ~ 10 minutes to ensure complete melting followed by steady shear testing in the range of 5000 to 5/seconds in air. For each test condition, at least two tests were performed to check for repeatability. Steady shear tests are more representative of extrusion deposition in systems such as the BAAM as they not only cover the shear rate ranges encountered in this system but also account for the effects of orienting polymer chains and filler materials in the flow direction.

3 | RESULTS AND DISCUSSION

3.1 | Thermal stability tests

Figure 2A-C represents the variation in G' and loss modulus (G'') with time for the three neat grades at the candidate test temperatures. The variation in η^* with time is shown in Table 2. Analysis of Figure 2 shows that all three grades of PEKK exhibit higher G'' than G' across all test temperatures, indicating that the material in the melt state is more viscous than elastic at the chosen processing temperatures. An increase in temperature lowers both the moduli and viscosity, as expected. However, with

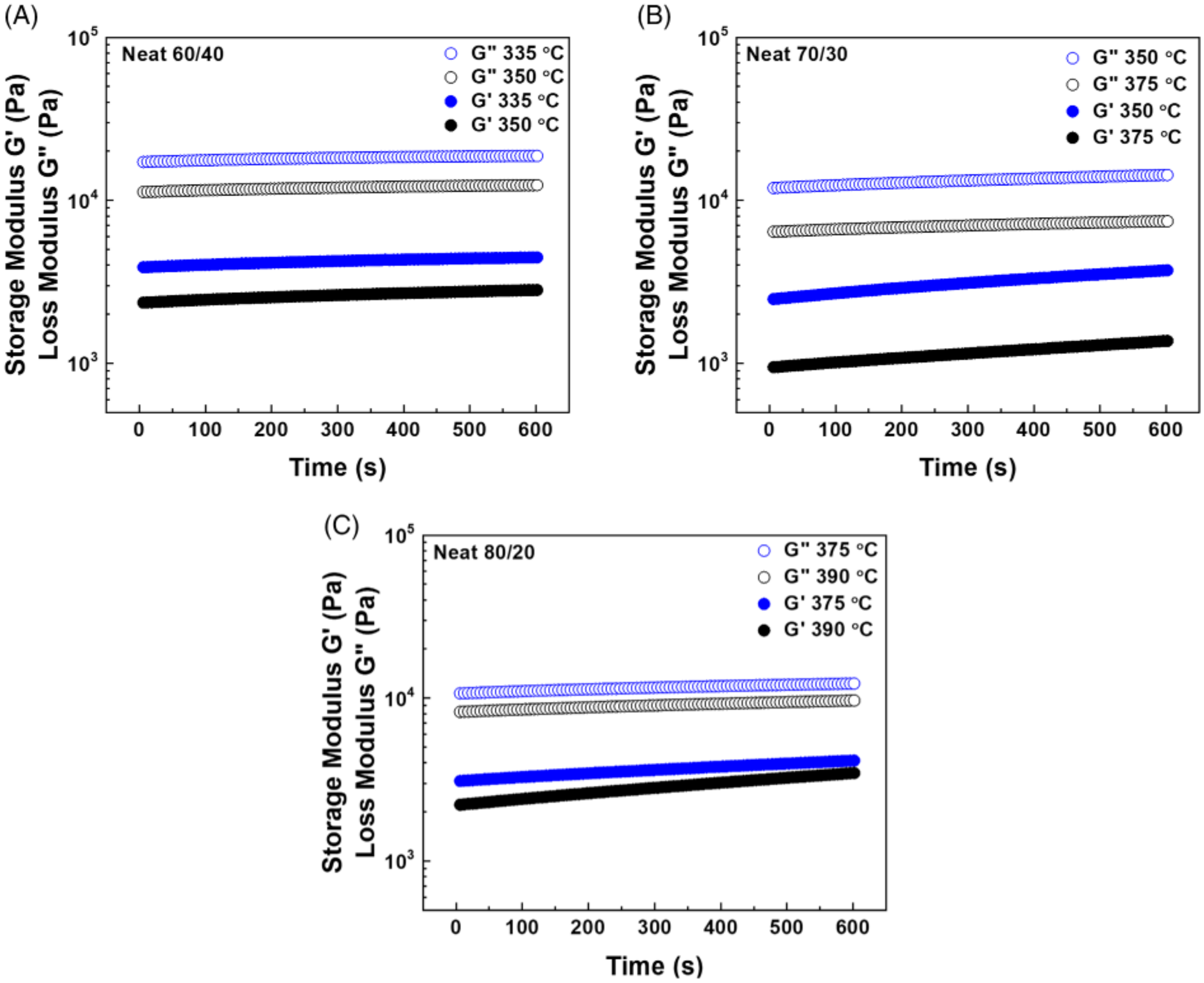

FIGURE 2 Time sweep tests at candidate processing temperatures in air for;(A) 60/40, (B) 70/30, and (C) 80/20 neat grades (LVE strain: 1%) [Color figure can be viewed at wileyonlinelibrary.com]

time, both moduli and η^* show some increase, which has been observed to be greater for 70/30 and 80/20 grades than for 60/40 in air. One of the possible factors causing this increase could be structural changes taking place due to branching and/or crosslinking reactions taking place in air with time (thereby increasing in elastic behavior), which is observed to further increase with increasing temperatures. Such crosslinking reactions in air have been observed and reported for other PAEKs such as PEEK at elevated temperatures [30]. Changes to viscoelastic properties due to thermal effects on the polymer structure, especially during processing in oxidizing environment, has been commonly reported for other high-performance materials, as well as polymers such as low-density polyethylene (LDPE) [19,31,32]. It should be noted that, in this work, any increase in viscosity in the range of 20% or lower in isothermal time sweep tests is considered to be of low significance and in the acceptable range for AM processing as the residence time on AM systems is much lower (~ 60 seconds), and the material also experiences higher shear rates (~100-200 seconds^{-1} at the nozzle), which can possibly delay the crosslinking

TABLE 2 Variation of complex viscosity with time for neat grades

Neat grade	Test temperature (°C)	Complex viscosity at t = 6 s (Pa.s)	Complex viscosity at t = 600 s (Pa.s)	% Increase in complex viscosity
60/40	335	1761	1926	9.3
	350	1149	1271	10.6
70/30	350	1215	1478	21.6
	375	647	757	17
80/20	375	1111	1294	16.5
	390	851	1025	20.4

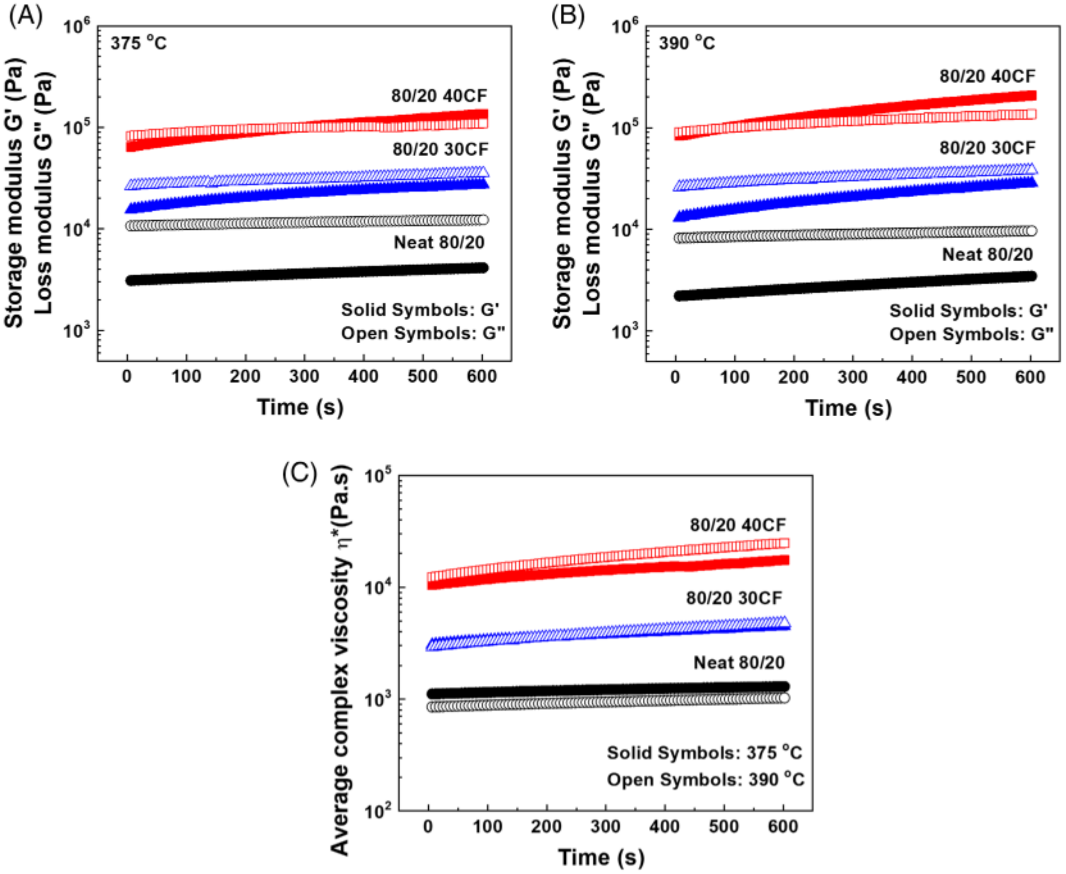

FIGURE 3 Time sweep tests in air for the 80/20 grades; (A) dynamic moduli at 375°C, (B) dynamic moduli at 390°C, and (C) complex viscosity at 375°C and 390°C [Color figure can be viewed at wileyonlinelibrary.com]

process. Hence, the three neat grades of PEKK studied here exhibit low viscosity buildup (increase in viscosity) and good thermal stability that make them suitable for AM processing for short residence time in air.

Figure 3A-C represents the variation of dynamic moduli and complex viscosity with time for the three 80/20 grades (unreinforced and two CF-reinforced grades) at the two candidate test temperatures of 375°C and 390°C. To remain in the LVE, a strain amplitude of 1% was used for neat and 0.04 to 0.06% for the two filled grades. The moduli data show an increase in both G' and G'' with increasing filler content at both temperatures. However, unlike the behavior observed for neat grades, the filled materials exhibit increasingly elastic behavior ($G' > G''$) with time. For 80/20 40CF, the crossover point (where $G'' = G'$) occurs at ~300 seconds at 375°C and at ~100 seconds at 390°C. The increase in G' with time can be attributed to possible structural changes in the form of crosslinking, which could be accelerated with increasing temperature and due to the presence of sizing (functional groups applied as coatings to CFs to enhance filler-matrix interactions). A similar effect is also observed as an increase in complex viscosity by 47% and 64%, respectively, at 375°C and 390°C after 10 minutes for 80/20 30CF and of 67% and 102% for 80/20 40CF under similar conditions. This indicates that increasing the filler content and the operating temperature reduces thermal stability of these materials over time. In addition, higher fiber loading can also lead to a higher viscosity at elevated temperatures (as observed for 80/20 40CF).

As the viscosity buildup is notably faster and more significant for the filled grades, all the subsequent frequency sweep tests were conducted in a nitrogen test environment. Based on this, the use of an inert cover gas in the extruder for systems such as the BAAM is recommended while processing these materials.

3.2 | Frequency sweep tests

The variation of dynamic moduli and complex viscosity with frequency at the candidate test temperatures for the neat grades are shown in Figure 4A-D. The charts indicate average values of these quantities obtained from frequency sweep tests from high-to-low range and vice versa. Note that standard deviations were very low to be

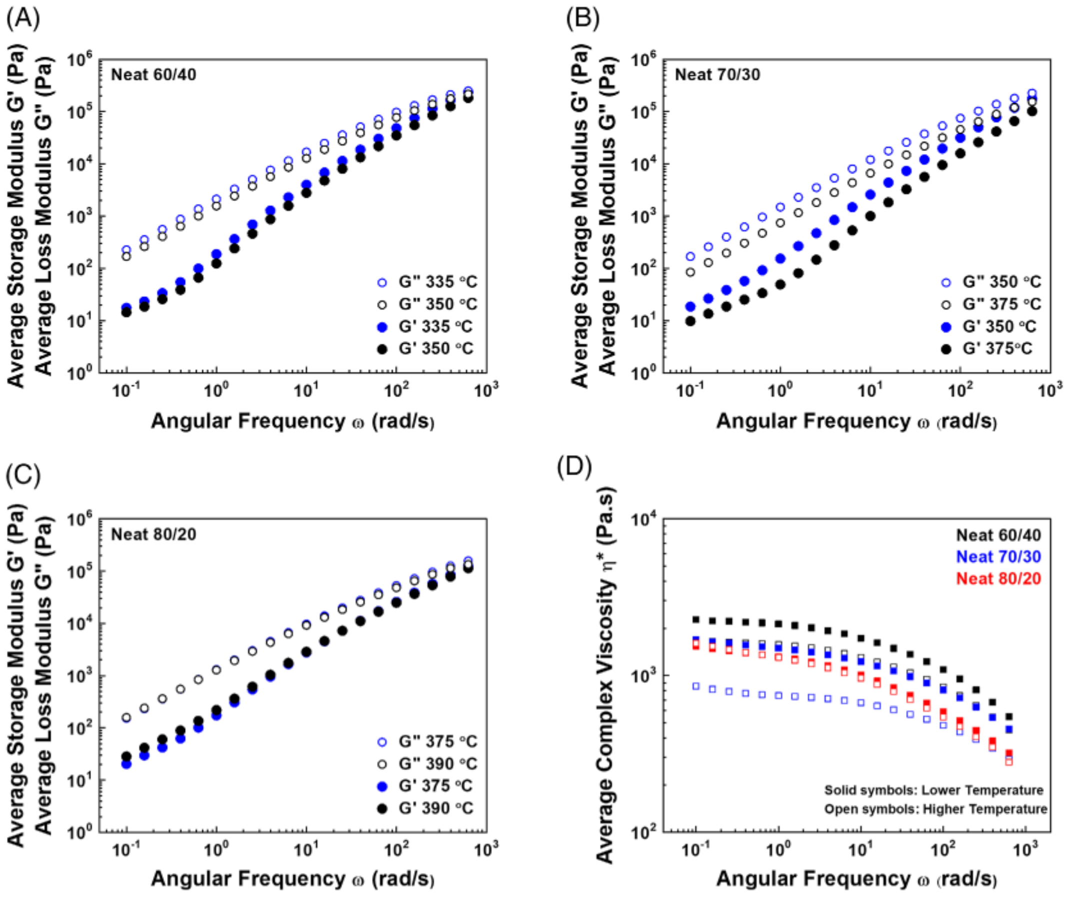

FIGURE 4 Frequency sweep tests indicating variations in (A) dynamic moduli for 60/40, (B) dynamic moduli for 70/30, (C) dynamic moduli for 80/20, and (D) complex viscosity for the three neat grades in air [Color figure can be viewed at wileyonlinelibrary.com]

clearly indicated on these plots (typically less than 10%). For all the three neat grades, G' and G'' increase with frequency (Figure 4A-C), which is typical behavior of most polymer systems. At relatively low frequencies, the test time is large compared to the relaxation time of the polymer chains, which allows the chains to recover to their original state under low strain conditions. As the frequency increases, the test time shortens, and the chains might not recover to their original state, thereby exhibiting greater stiffness [33]. For all the three grades, a crossover point ($G''/G' = 1$) was not observed in the frequency range tested, indicating that these unreinforced grades show primarily viscous behavior at all tested frequencies. The variation of complex viscosity with frequency (Figure 4D) shows these neat grades to exhibit a fairly Newtonian behavior at low frequencies and shear thinning behavior only at frequencies greater than ~ 10 rad/s. As the shear rates experienced in the nozzle region of the BAAM system is in the range of 100 to 200 seconds^{-1}, and is greater than 1000 seconds^{-1} in the screw region, the shear thinning behavior of these materials at higher frequencies (or shear rates) is beneficial to enable easier processing. Shear thinning behavior of many polymers can be described by Equation 1, where η is the shear viscosity, $\dot{\gamma}$ is shear rate, k is consistency index, and n is the power law index. Table 3 lists the power law indices for the three grades of PEKK calculated between 10 and 628 rad/s. The 80/20 grade exhibited greater shear thinning than the other two neat grades. The effect of temperature was least pronounced for the 80/20 grade, but an increase in temperature was shown to significantly decrease viscosity for the 60/40

TABLE 3 Shear thinning exponent for neat grades

Neat grade	Test temperature (°C)	Shear thinning exponent '*n*'
60/40	335	0.73
	350	0.75
70/30	350	0.76
	375	0.80
80/20	375	0.73
	390	0.71

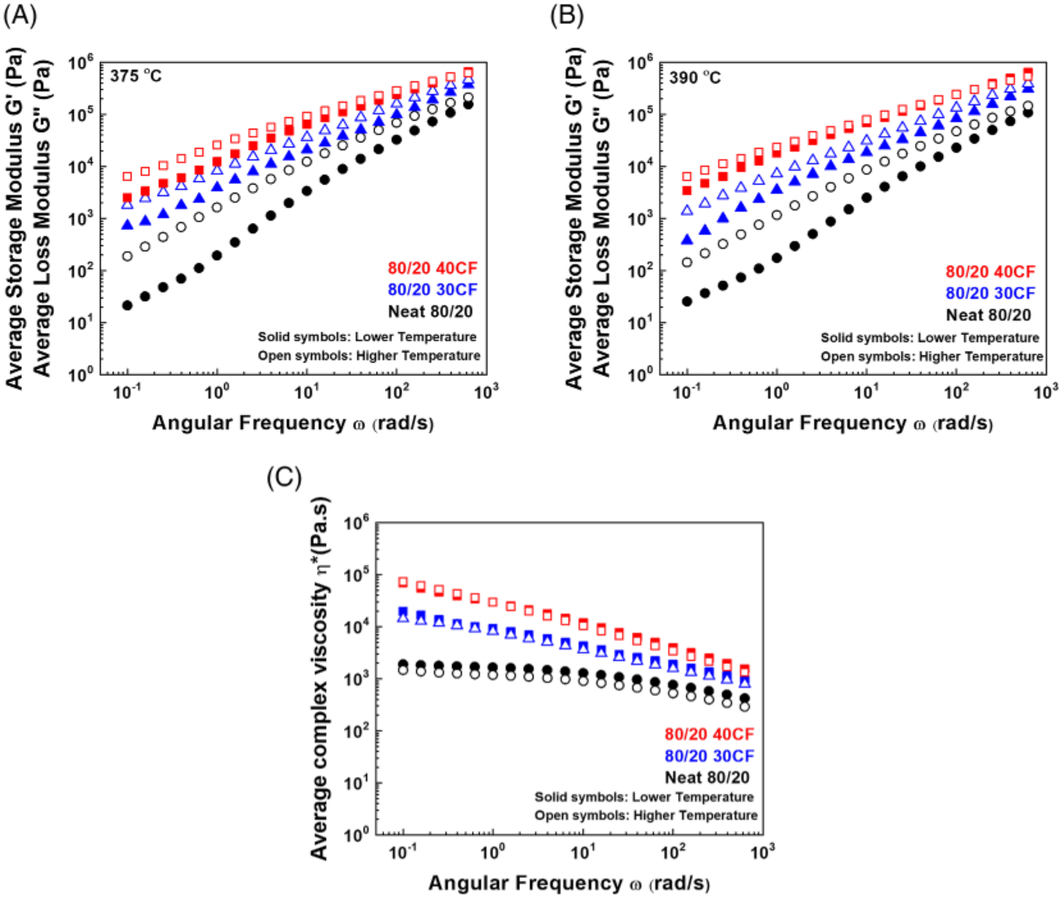

FIGURE 5 Frequency sweep tests for the three 80/20 grades indicating variations in (A) dynamic moduli at 375°C, (B) dynamic moduli at 390°C, and (C) complex viscosity [Color figure can be viewed at wileyonlinelibrary.com]

and 70/20 grades. In terms of setting processing conditions for AM, this behavior indicates that varying the shear rate (by changing screw speed, nozzle geometry) may be a more effective method for altering the effective viscosity than changing temperature for the 80/20 grade.

$$\eta = k(\dot{\gamma})^{n-1} \tag{1}$$

The frequency sweep data for the three 80/20 grades (in nitrogen) at the two candidate test temperatures are represented in Figure 5A-C. Both G' and G'' increased with increasing frequencies (similar to the neat grades) and increasing filler content. In addition, the filled materials exhibited a crossover point that shifted to lower frequencies with an increase in both filler content and temperature, indicating an increase in elastic behavior. As expected, the complex viscosity data shown in Figure 5C indicated an increase in viscosity with the addition of fillers as fillers obstruct the mobility of the polymer chain segments. However, the increase was observed to be greater at lower frequencies, a behavior that has been reported for other filled systems such as carbon nanotubes in polycarbonate, glass fiber suspensions in LLDPE, and other polypropylene composites as well [33–36]. This behavior can be explained in terms of the extent of movement of fillers with changing polymer viscosity (or with changing shear rates for shear thinning materials). For the SAOS tests, the fibers were randomly oriented as the pellets were melted between the parallel plates. At low shear rates (frequencies), the deformation is low, and the fibers tend to partially align along the flow direction but do not fully reorient. Because the viscosity of the matrix is higher at lower shear rates, the resistance to fiber movement is also greater, which gives rise to increased stress to maintain the same shear rate, thereby increasing viscosity. However, for shear rates relevant to AM processing (100-200/seconds), this behavior favors the processing of even highly filled systems as the viscosities at the nozzle would not be as significantly affected by filler content. However, it

TABLE 4 Shear thinning exponent for 80/20 grades

80/20 grade	Test temperature (°C)	Shear thinning exponent '*n*'
Neat	375	0.73
	390	0.73
30 wt.% CF	375	0.65
	390	0.64
40 wt.% CF	375	0.51
	390	0.50

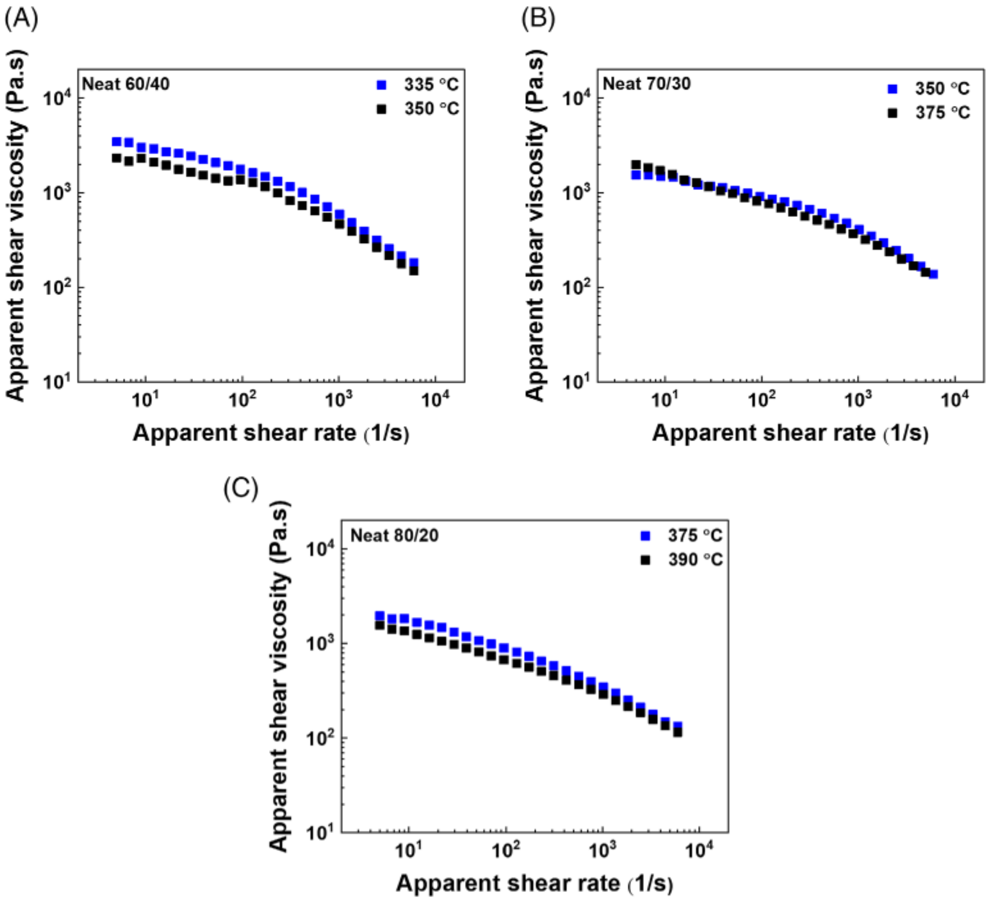

FIGURE 6 Variation of apparent shear viscosity with apparent shear rate for (A) 60/40 grade, (B) 70/30 grade, and (C) 80/20 grade [Color figure can be viewed at wileyonlinelibrary.com]

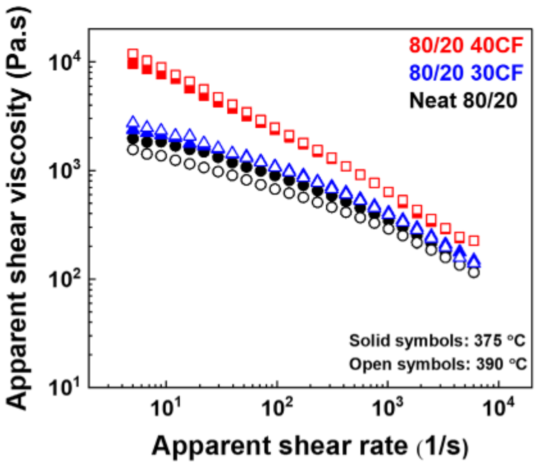

FIGURE 7 Variation of apparent shear viscosity with apparent shear rate for the three 80/20 grades [Color figure can be viewed at wileyonlinelibrary.com]

should be noted that the zero shear viscosities of the filled systems are higher and would need to be overcome to initiate flow of these materials during extrusion. This can be observed as an increased dependence of viscosity on filler concentration at lower frequencies. More specifically, there is a shift in the Newtonian plateau to lower frequencies with increasing filler loading, although the plateau region is not directly observed in the frequency range of testing for filled grades. A high zero shear viscosity would have to be overcome by enough driving pressure in the AM system used. In addition, the extent of shear thinning is also enhanced by the addition of fillers (lower shear thinning exponent, *n*, observed with increasing filler loading, as indicated in Table 4). This behavior has been reported for other filled polymer systems as well, such as carbon nanofiber- and glass fiber-reinforced polyethylene composites [33,37]. It is notable that there is no significant effect of temperature on viscosity for the reinforced grades across all frequencies tested. This enables a wider processing temperature window to enable the most suitable processing conditions when processing for AM applications.

3.3 | Steady shear tests

To evaluate the performance of the PEKK grades at shear rates more relevant to the BAAM extrusion conditions, the samples were tested in a capillary rheometer at given shear rates. Figures 6 and 7 represent the variation of apparent shear viscosity with apparent shear rate for all the grades of PEKK. From Figure 6, it is evident that all the three neat grades exhibit considerable shear thinning, which follows the trend observed at the higher frequency range of the SAOS tests (Figure 4D). For the CF-reinforced systems (Figure 7), increasing the filler loading increased the shear viscosity, as expected, although the effect of filler loading on viscosity is less significant at higher shear rates (again, following the trends observed in SAOS tests). This can be attributed to the orientation of polymer chains and fillers in the flow direction at such high deformation rates, thereby lowering the viscosity.

Rheological characterization of the five PEKK grades demonstrated the effect of various process parameters on the viscoelastic behavior and thereby provided guidelines for optimizing processing conditions and determining printing success of materials new to the 3D printing domain. It has been possible to determine the thermal stability of these materials and variations in dynamic and steady shear viscoelastic properties under different processing conditions, which provided an improved understanding of melt behavior under various processing conditions that may be encountered in extrusion deposition AM platforms.

4 | CONCLUSIONS

This work investigated the rheological properties of five different grades of PEKK for use as a feedstock material in extrusion deposition AM process using dynamic rotational rheological studies and shear analysis. Results indicated that the unreinforced grades had lower viscosity buildup (and structural change) with time in air than the CF-reinforced grades. Thus, the use of cover gas while processing such materials on large-scale systems is highly recommended. The effect of temperature on rheological properties, mainly complex viscosity, was more evident for the neat 60/40 and 70/30 grades than the three 80/20 grades. All five grades exhibited a degree of shear thinning, with the extent of shear thinning being greater for the reinforced grades. Furthermore, fillers were observed to increase the dynamic moduli and viscosity. Such investigations into the rheological properties of high-performance thermoplastics are very beneficial in defining the appropriate processing conditions for extrusion-based AM.

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