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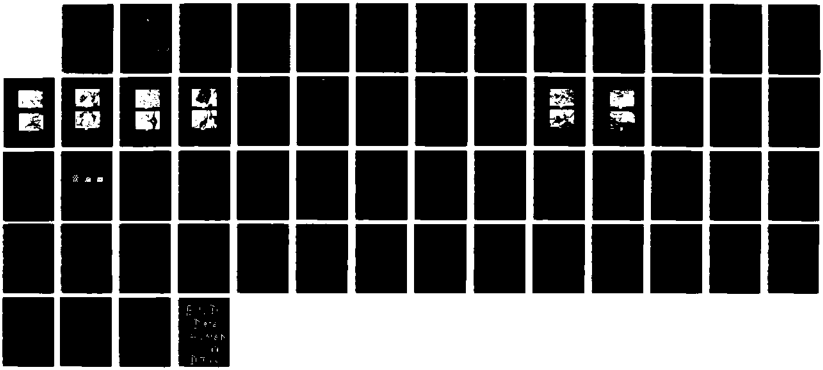

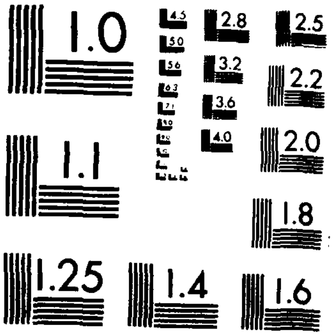
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REPORT NO. NADC-88026-60

THE INFLUENCE OF PARTICLE PROPERTIES ON THE ROOM TEMPERATURE COMPACTION OF POLYETHER-ETHERKETONE (PEEK) POWDERS

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This study deals with the characterization and cold compaction of polyether-etherketone (PEEK) powders. Four different types of PEEK powders which are commercially available from Imperial Chemical Industries (ICI) were characterized for density, crystallinity, particle size, particle size distribution and particle morphology. Fine and coarse size powders of a low viscosity grade (150PF and 150P) and similar powders of a high viscosity grade (450PF and 450P) were processed. Compaction was successful at room temperature using the 150 grade powders but not with the 450 grade powders. Compressibility curves were obtained at room temperature for the 150 grade powders and their post-compaction viscoelastic recoveries were measured. Both coarse and fine size powders of the 150 grade reached relative plateau densities of 95.8% and 95.1% when compacted at pressures exceeding 300 MPa. These high densification values at room temperature were associated with minimal post-compaction viscoelastic recoveries and as-compacted strengths as high as 8.9 MPa. A modified densification parameter (DP*) was developed based on the plastic deformation of the crystalline regions alone. This new dimensionless parameter, DP*, demonstrated a better fit of all the compaction data. The transverse rupture strength and the green density data are presented and explained in terms of this DP*. (AW)

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INTRODUCTION

High temperature thermoplastics like polytetra-fluoroethylene (PTFE), Upjohn polyimide 2080 (PI), poly-phenylene sulfide (PPS) and polyether-etherketone (PEEK) are relatively difficult to process by the conventional melt processing techniques for several reasons. PTFE is a semicrystalline thermoplastic with a glass transition temperature (Tg) of 126°C and a melting temperature (Tm) of 342°C [1]. The application of powder metallurgy (P/M) techniques to polymers started with the processing of PTFE because of its high melt viscosity and its high Tm [2]. PI-2080 is an amorphous polymer with a Tg in the range of 310-315°C [3,4]. Due to its high melt viscosity, compression molding is the primary technique used to form this polymer [3]. In addition, it is necessary to thermally cycle the mold to achieve quality components [4]. This latter molding requirement adversely affects the economics of using this technique in production because of its time and energy inefficiencies [4]. PPS and PEEK are also semicrystalline thermoplastics with Tg's of 85°C and 144°C, and Tm's of 285°C and 335°C, respectively [5-9]. Because both of these polymers have high melting temperatures, melt processing techniques require the use of high temperature molding equipment which results in high capital and energy costs. In particular, PEEK possesses a combination of high viscosity and low crystallization rates which make melt processing economically unattractive. Processing PEEK via conventional techniques such as injection molding and extrusion have been previously described [10-12]. An alternative processing technique is direct powder compaction and preform sintering. This approach eliminates the pelletizing step, reduces the need for recrystallization via annealing and is accommodating to the inclusion of hard fillers. However, this P/M approach suffers from a lengthy sintering step if conventional oven heating of the preform is utilized. Thermoplastics are good heat insulators and therefore, sintering times exceeding 30 minutes may be expected for parts in the one inch thickness range. Several possible alternatives [13] may be used to overcome this slow process including the use of induction heating of magnetic fillers [14-16], radio frequency or microwave heating of fillers with high dielectric loss properties [17,18] or particle fusion by ultrasonic compaction [18]. These approaches efficiently supply energy to the polymer particle surfaces where it is needed for consolidation and eliminates the need for melting and shearing the entire polymer mass. This is particularly significant for those polymers with narrow processing temperature ranges or ones which are sensitive to shear deformations during melt flow. The conservation of energy and the reduction of cycle time with these techniques make them very attractive for the processing of PEEK and other high performance thermoplastics. Additionally, a forging process may be used following sintering if necessary, to reshape and consolidate the sintered compacts while realizing the benefits of predesigned orientations.

Most studies [3,4,18-37] on polymer powder compaction have been performed using glassy polymers, thermosets, semicrystalline thermoplastics and polymer blends. The results of these investigations indicate that powder particle size, particle morphology, compaction rate, compaction pressure and dwell time at peak pressure are parameters which significantly influence the compaction process. This report deals with the compaction properties of a semicrystalline thermoplastic (PEEK) at temperatures significantly below its Tg. Compaction rate and dwell time at peak pressure are held constant in this study while the influences of crystallinity, particle size and particle surface properties on compaction efficiency are discussed.

The specific objectives of this report are to physically and morphologically characterize four different commercial PEEK powders and to explain their behavior when compacted at room temperature. Very little work has dealt with the compaction of semicrystalline polymers below their Tg. Bigg [30,31] tried to compact nylon 11 below its Tg. However, the compacted particles relaxed so much during sintering that they could not be handled or tested for strength. This type of behavior occurred because the nylon 11 particles were not plastically deformed sufficiently during compaction to form permanent interlocking junctions which provide green strength. Radhakrishnan and Nadkarni [36] compacted PPS below its Tg to study compaction-induced changes in crystallinity. However, they did not report green density or green strength values. The work involved in this report will show the advantages of cold compaction of a semi-crystalline polymer at temperatures significantly below its Tg. Currently, the authors are also extending this study of PEEK powder compaction to include mixtures of PEEK and nickel powders [38].

EXPERIMENTAL PROCEDURE

POWDER CHARACTERIZATION

Commercially available high viscosity (450PF, 450P) and low viscosity (150PF, 150P) grades of PEEK powders were obtained from Imperial Chemical Industries (ICI). Particle size and particle size distribution properties were determined using sieve analysis in accordance with ASTM Standard D1921-63 (reapproved 1975), Method A. Apparent densities were measured in accordance with ASTM Standard D1895-69 (reapproved 1979), Method A. The powder densities were measured with a Micromeritics (Norcross, GA) AutoPycnometer 1320 using helium. Weight percent crystallinity values were calculated from these density measurements using 1.401 g/cc and 1.263 g/cc as the respective 100% crystalline and amorphous densities [9]. Tap density tests were performed by transferring a weighed sample of powder into a graduated cylinder, tapping the cylinder from a height of one inch onto a padded surface until the volume became constant (usually about 100 times), and then measuring the volume. Scanning electron microscopy (SEM) was used to characterize particle surfaces. The SEM samples were prepared by dispersing the powder onto adhesive tape, sputtering the surface with a thin layer of gold and then examining under the SEM (AMR Model 1000A) at 20 kV.

Crystallinities of the as-received, annealed and melt recrystallized powders were measured using a DuPont 910 differential scanning calorimeter (DSC) controlled by a 1090 thermal analyzer. Each specimen was heated at a rate of 5°C/min. up to 500°C, then cooled to 100°C at a rate of 10°C/min., held at 100°C for 10 min. and then reheated to 500°C at a rate of 5°C/min. The temperatures and latent heats of crystallization and fusion were determined from the areas under the various peaks of the thermogram. This data was converted to weight percent crystallinity values using 130 J/g as the heat of fusion for 100% crystalline PEEK [18].

COMPACTION

The powders were compacted in a Tinius Olsen press using a Haller double-acting die with a one minute dwell time at peak pressure and without the use of a lubricant. The velocity of the punch at zero pressure was approximately 5 cm/min. Cylindrical (2.54 cm diameter) and rectangular (1.27 cm by 3.18 cm) dies were used to fabricate specimens. Green density was determined by measuring the weight and dimensions (24 hrs after compaction) of the samples. These values were used to evaluate compressibility as a function of compaction pressure. Viscoelastic recovery of the cylindrical powder compacts at room temperature was determined as a function of time after ejection. For this purpose, low melt viscosity PEEK specimens were compacted at room temperature using 400 MPa of pressure. Transverse rupture strengths of rectangular compacts (0.635 cm thick) were determined in accordance with ASTM Standard B312-82 at a crosshead speed of 0.5 cm/min. These green strength values were evaluated as a function of density. The SEM was also used to examine the fracture surfaces of these specimens.

RESULTS

POWDER CHARACTERIZATION

Powder density results determined using a gas pycnometer are listed in Table 1. Densities ranged from 1.281 g/cc to 1.318 g/cc for the as-received PEEK powders. Weight percent crystallinity values calculated from these density measurements are also included in Table 1. Both fine-sized powders (PF designated) had much lower densities and consequently significantly lower degrees of crystallinity than the coarse-sized powders. These values ranged from 14 to 42 percent crystallinity for the as-received powders. The last powder listed in Table 1 (annealed 450PF) was exposed to a temperature of 210°C for 2 hours and then oven-cooled to room temperature before testing. This annealed powder showed an increase of 15% in crystallinity compared to its as-received condition.

DSC was also used to determine weight percent crystallinity values by measuring the enthalpy of fusion. These results are shown in Table 2. This technique confirms that the 450PF material is not fully crystallized in the as-received condition and therefore, will continue to do so when its temperature is raised above its Tg as shown in Fig. 1. This plot is a typical thermogram from a DSC run on a sample of 450PF powder. A pre-melt

TABLE 1: POWDER DENSITY RESULTS

MATERIAL	DENSITY (g/cc)	PERCENT CRYSTAL-LINITY
150PF	1.301	30
150P	1.318	42
450PF	1.281	14
450P	1.313	39
*450PF	1.300	29

* Annealed at 210°C for 2 hours.

TABLE 2: DSC RESULTS

MATERIAL	CONDITION	HEAT OF TRANSITION (J/g)	PEAK TRANSITION TEMPERATURE (°C)	PERCENT CRYSTAL-LINITY
150PF	AS-RECEIVED	50	342	38
	RECRYSTALLIZED FROM MELT	32	249	25
	RE-MELTED	28	326	21
150P	AS-RECEIVED	56	343	43
	RECRYSTALLIZED FROM MELT	56	297	43
	RE-MELTED	37	338	28
450PF	PRE-MELT CRYSTALLIZATION	16	168	12
	AS-RECEIVED (ANNEALED)	44	338	34
	AS-RECEIVED (CALCULATED)	29		22
	RECRYSTALLIZED FROM MELT	41	255	32
	RE-MELTED	38	332	29
450P	AS-RECEIVED	52	334	40
	RECRYSTALLIZED FROM MELT	25	268	19
	RE-MELTED	28	331	22

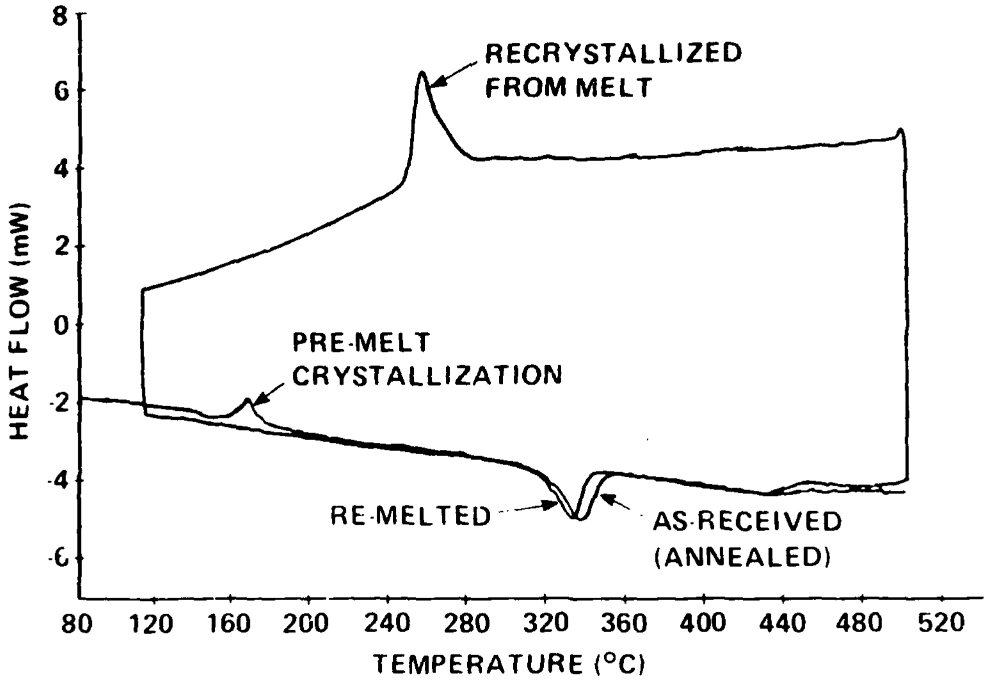


Figure 1 - DSC Thermogram of 450PF Powder

crystallization peak which occurs at about 168°C is apparent in Fig. 1. The heat of pre-melt crystallization at 168°C was subtracted from the heat of fusion peak at the normal melting point and the difference is listed as the as-received (calculated) crystallinity for the 450PF powder.

The peak crystalline melting point of the powders ranged from 334°C to 342°C. The percent crystallinity values determined for the as-received coarse powders (150P and 450P) using density data were within one percent of their respective values as determined by the heat of fusion technique. However, a similar comparison of the two techniques for the fine particles (150PF and 450PF) revealed differences of about 8 percent. Several reasons may be put forth to explain these differences. The DSC technique is most effectively used to determine the percent crystallinity of highly ordered materials with sharp, well-defined melting peaks [39]. The PEEK powders studied in this investigation (especially the fine-sized powders) did not have the above characteristics so the baseline was extrapolated across these broad peaks, which may have introduced additional error. However, the relative values of crystallinity are consistent within each of the above techniques.

After heating the powders through the melting point and recrystallizing, the crystallinities determined from a second melting peak decreased by 5 to 18 percent compared to those obtained during the first melt of each powder. The peak melting points decreased by 3-16°C upon remelting which also indicates a reduction in order when PEEK is cooled from the melt at a rate of 10°C/min. The heats of recrystallization and second melting peaks have fairly close values but some differences are to be expected due to the temperature dependence of the 100% crystalline heat of fusion value used in these calculations. In some instances, the recrystallization process produced a complex peak which appeared to be a combination of two peaks. This observation is in accord with the dual crystallization mechanism for PEEK proposed by Velisaris and Seferis [40].

SEM photographs of the PEEK particles are shown in Figs. 2-5. Fig. 2 is a photograph of the 150P powder which is very similar in appearance to the 450P powder. Both of these powders appear to be almost equiaxed with relatively smooth surfaces. Figs. 3 and 4 show the 150PF and 450PF powders, respectively to be much smaller in size compared to the coarse-sized powders. These fine-sized powders are also seen to be irregularly shaped with fairly rough surfaces. This is particularly evident in Fig. 4 for the 450PF powder where it appears as though some of the particles have been deformed to the extent of fiber formation. Figs. 4 and 5 reveal that the as-received and the annealed 450PF powders are similar. This is indicative of plastic deformation in the as-received 450PF powder which will not be significantly affected by annealing.

Several powder characteristics are summarized in Table 3. The apparent or bulk density provides a measure of the volume the powder will occupy at zero pressure. The primary importance of this parameter is realized when storage volume or tooling size needs to be determined. Apparent densities ranged from 0.37 to 0.49 g/cc. The 150PF and 150P powders have compression ratios (green density/apparent density) of 3.0 and 3.3, respectively at their maximum green densification levels.

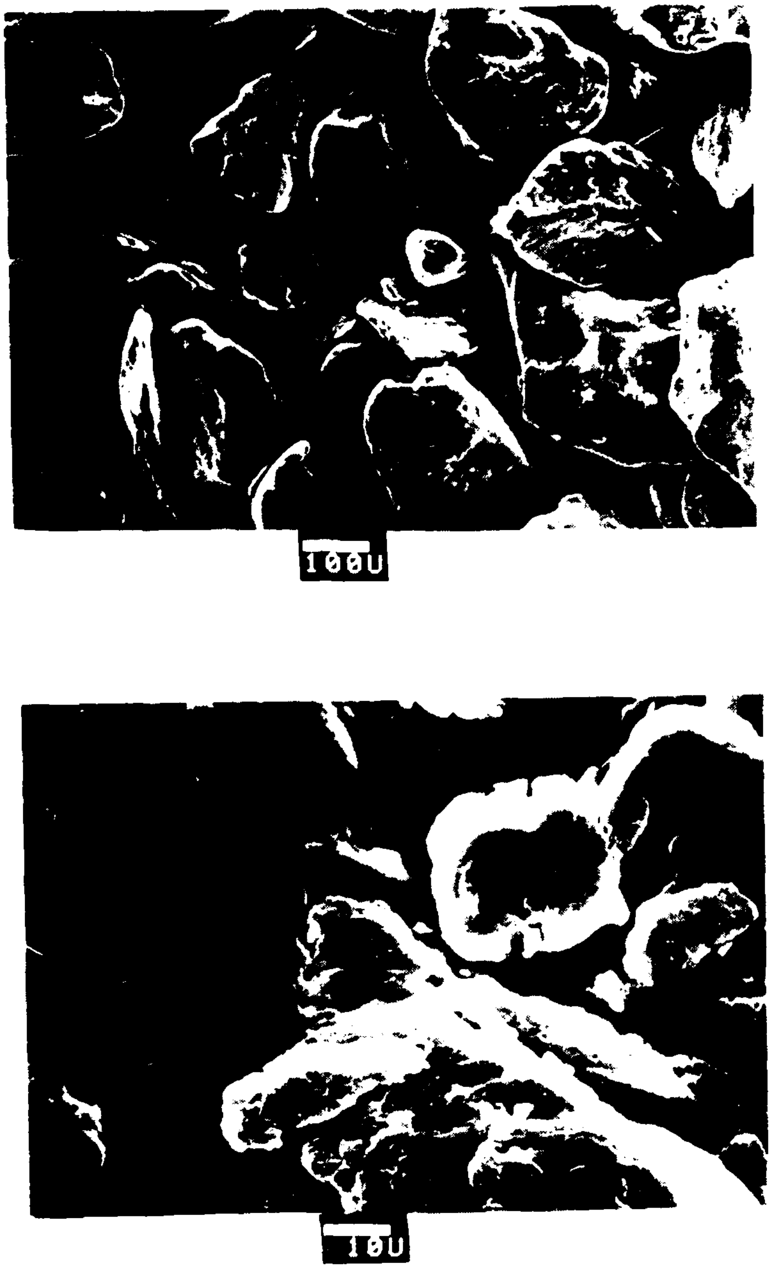


Figure 2 - SEM Photomicrographs of 150P Powder

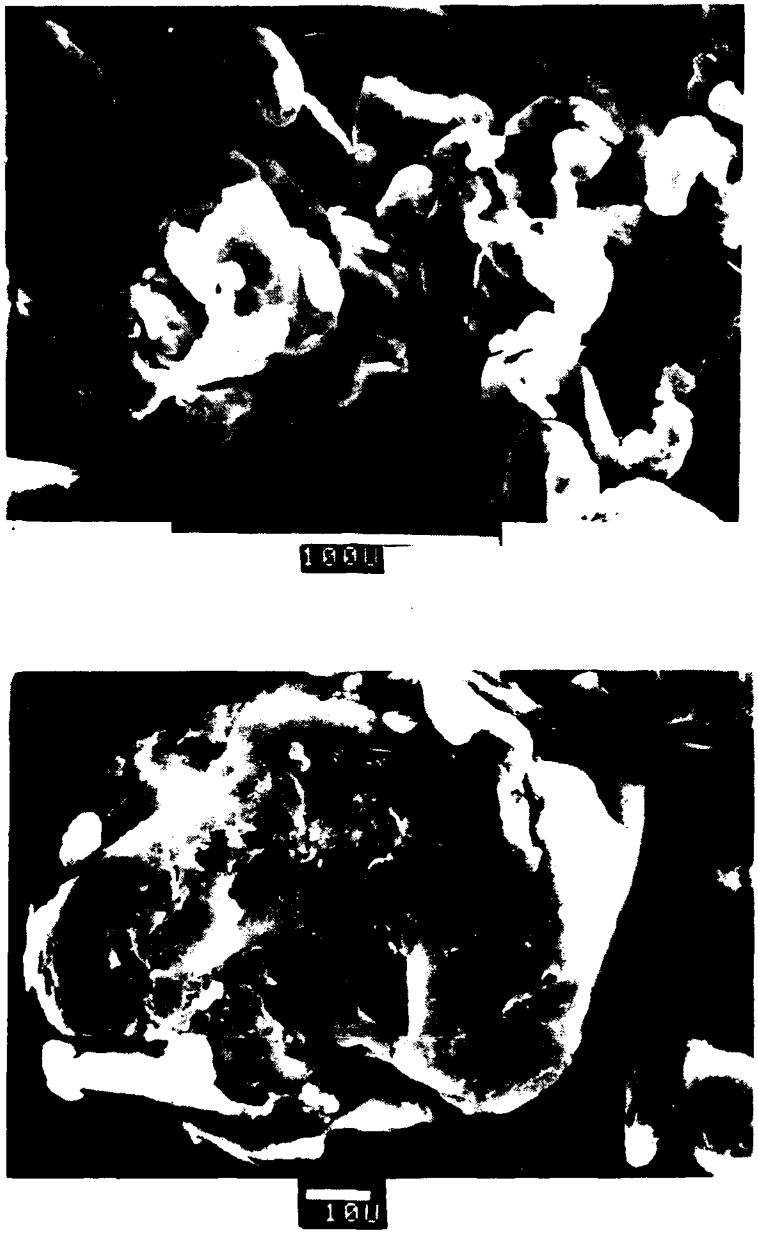


Figure 3 - SEM Photomicrographs of 150PF Powder

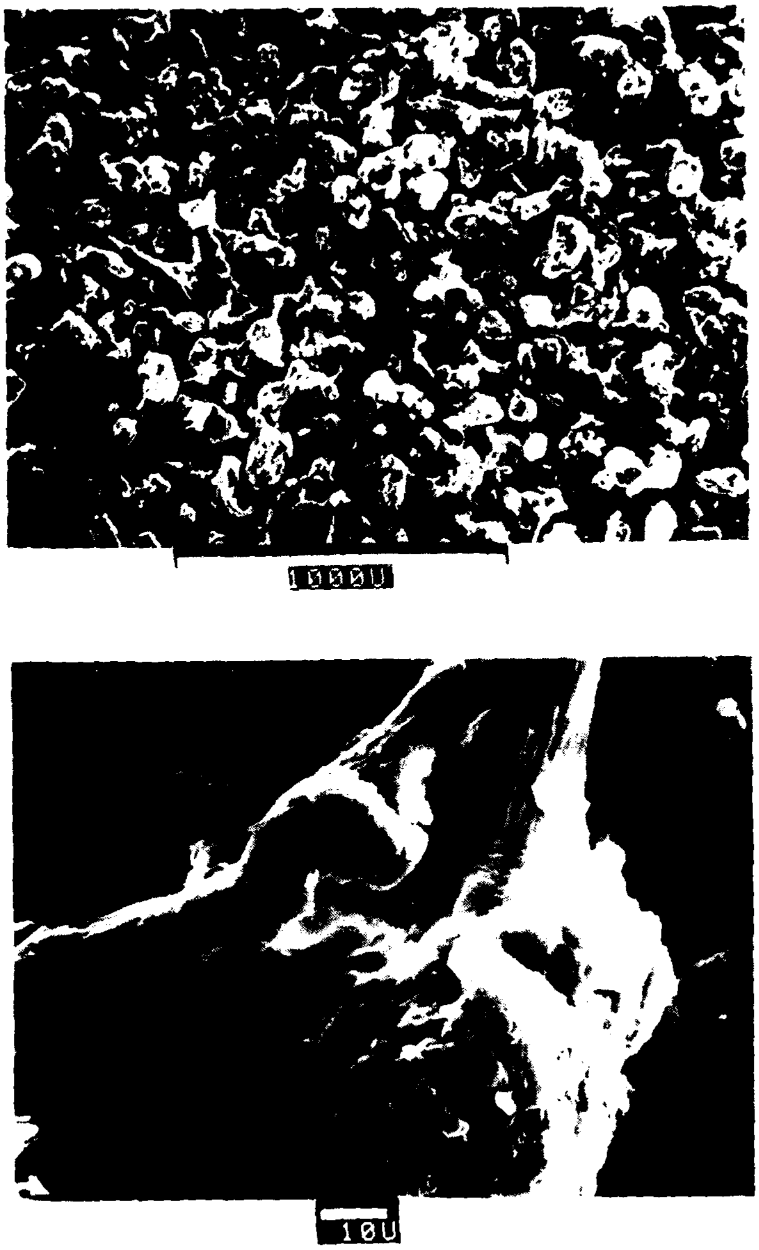


Figure 4 - SEM Photomicrographs of 450PF Powder

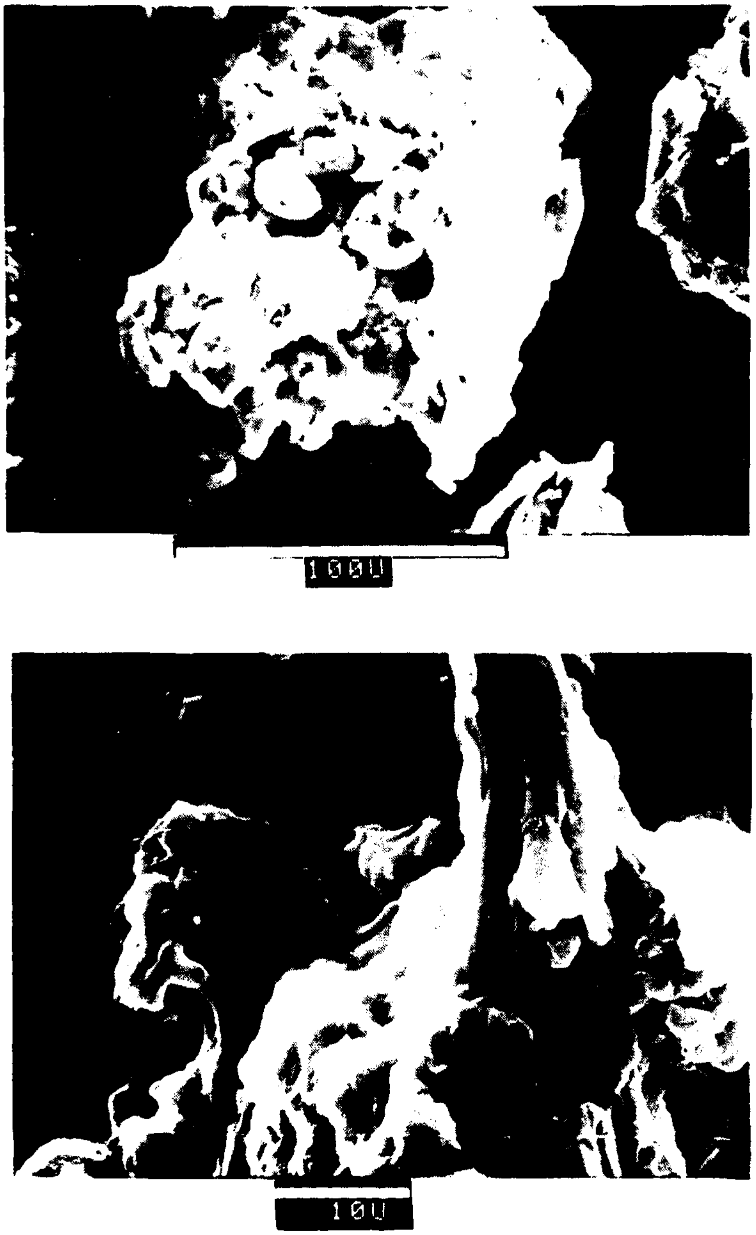


Figure 5 - SEM Photomicrographs of Annealed 450 PF Powder

TABLE 3: POWDER CHARACTERISTICS

MATERIAL	APPARENT DENSITY (g/cc)	TAP DENSITY (g/cc)	MEAN PARTICLE DIAMETER (μm)
150PF	0.42	0.57	145
150P	0.38	0.45	530
450PF	0.37	0.52	170
450P	0.49	0.53	680

The tap densities varied from 0.45 to 0.57 g/cc. The percent increase in apparent density due to tapping is attributable to interparticle arrangements. These percent increase values for the 150PF, 150P, 450PF and 450P powders (calculated from Table 3) are 36, 18, 41 and 8, respectively. Note that the fine-sized powders of each grade have higher percent increases in apparent density after tapping. This is in part due to the rough irregular surfaces of the 150PF and 450PF powders (Figs. 3 and 4). These rough surfaces cause high interparticle friction levels compared to the smoother surfaces of the coarse-sized particles (Fig. 2). The tapping action provides enough energy to overcome the barriers to interparticle motion and allows the fine-sized powders to pack efficiently.

The mean particle diameter for each of these powders is also shown in Table 3. These values ranged from 145 μm to 680 μm. The fine and coarse 450 grade powders are slightly larger than the similar 150 grade powders. The coarse-sized powders of each grade are approximately four times the size of their respective fine-sized powders. The particle size distributions for these powders are shown in Fig. 6. The coarse-sized particles of each grade have broader distributions than the fine-sized particles. Each coarse-sized powder has a significant weight fraction of particles greater than the largest sieve size used.

COMPACTION

Compaction of each of the four different PEEK powders was attempted at room temperature. The 150 grade powders were pressed into quality compacts of good integrity that were easy to handle while both 450 grade powders crumbled when attempting to remove them from the die. Relative green density (green density/theoretical powder density) versus compaction pressure is plotted in Fig. 7 for 150P and 150PF powders. These powders densified with increasing compaction pressure until a constant densification level was reached. The plateau densification level for these compacts is reached at about 300 MPa (plateau pressure). The relative plateau densities (plateau density/theoretical powder density) of the 150PF and 150P powders were 95.8% and 95.1%, respectively. It is seen

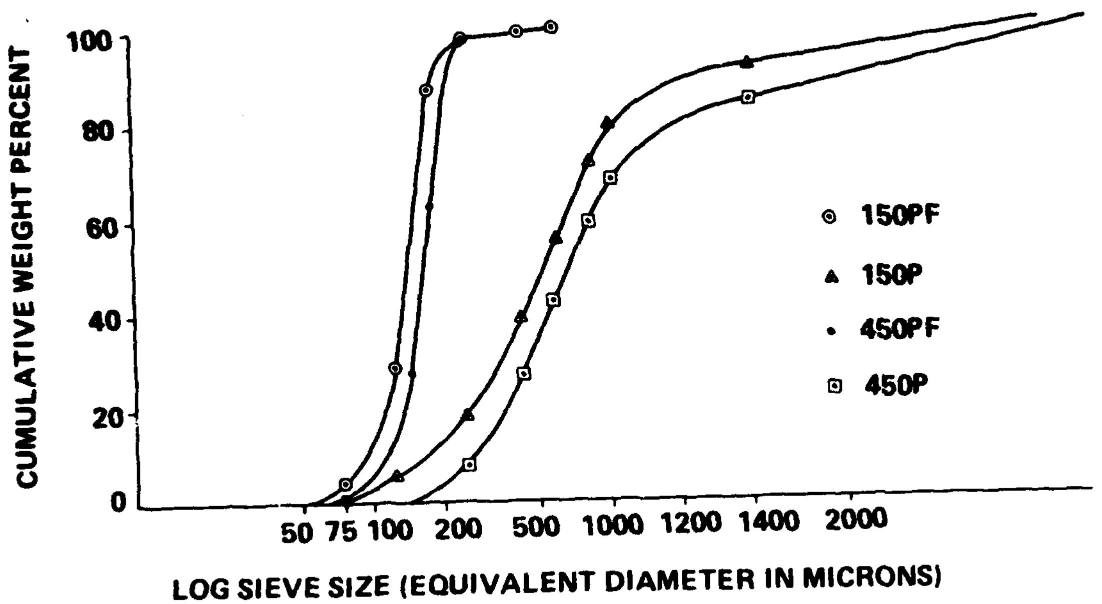


Figure 6 - Particle Size Distributions for Commercial PEEK Powders

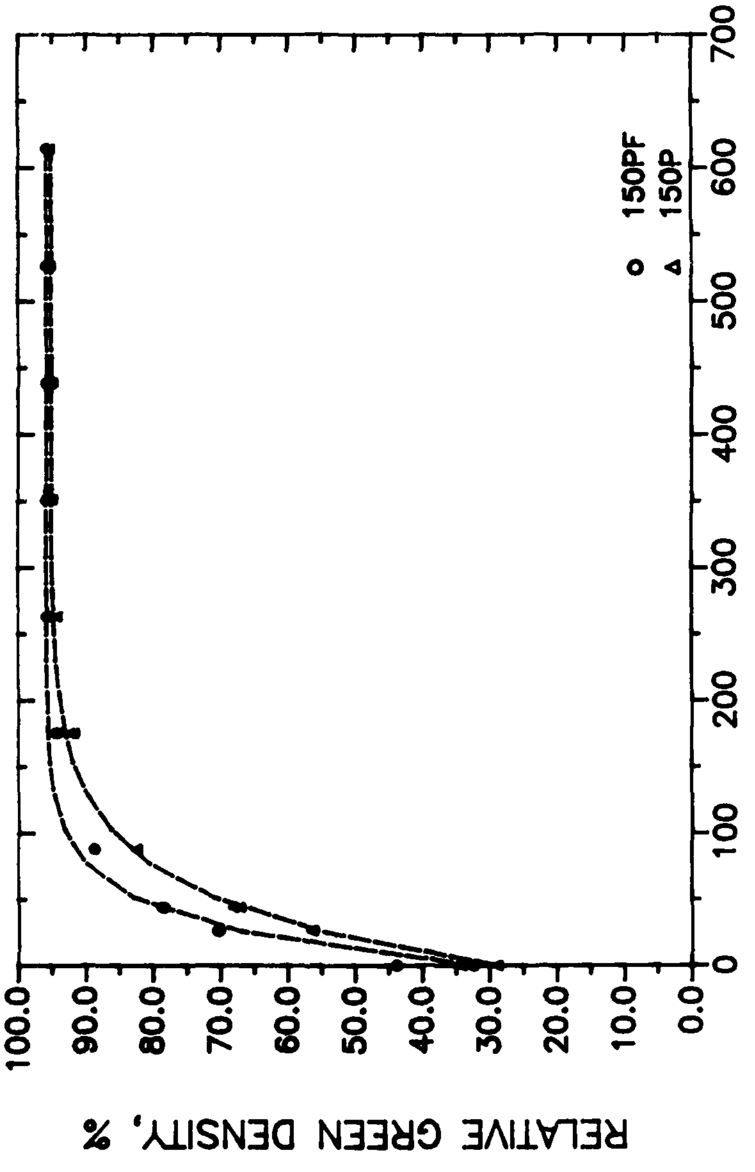


Figure 7 - Relative Green Density of 150 Grade PEEK versus Compaction Pressure

from Fig. 7 that the fine-sized powder (150PF) achieved a higher relative plateau density than the coarse-sized powder (150P), respectively. This observation will be discussed in the next section.

A graph of transverse rupture strength as a function of relative green density is shown in Fig. 8. Both 150 grade compacts displayed comparable strength versus density with the 150P material being slightly stronger. Strength values as high as 8.9 MPa and 7.6 MPa were measured for the 150P and 150PF samples, respectively. Compacts which had a relative green density less than 55 percent crumbled quite readily even with delicate handling. The fracture surfaces of 150PF and 150P specimens which were compacted at a pressure of 614 MPa are shown in Figs. 9 and 10, respectively. The individual particle boundaries are visible in both these figures and the mode of fracture appears to be along these boundaries. Figs. 9 and 10 both show a large amount of plastic deformation. A comparison of these figures reveals that the 150P material experienced a greater degree of plastic deformation which resulted in better mechanical interlocking of particles and therefore, higher strengths.

Room temperature viscoelastic recovery data for the 150 grade specimens in the axial and radial directions expressed as a function of time after compaction at 400 MPa are shown in Fig. 11. The dimensional changes in the 150P compacts are slightly greater than that of the 150PF compacts. However, on a volume change basis, they exhibit almost identical behavior (0.16% versus 0.15%). The 150PF and 150P compacts displayed equilibrium axial expansions of about 0.24% and 0.36% relative to their thicknesses 24 hours after compaction, and radial contractions of 0.28% and 0.31% relative to the die diameter, respectively. The elastic portion of the recovery in the radial direction for the 150PF and 150P specimens was -0.06% and -0.09%, respectively. The dimensions in the radial and axial directions are almost completely stable within 24 hours after ejection from the die.

A DENSIFICATION PARAMETER FOR PEEK POWDERS COMPACTED AT ROOM TEMPERATURE

It was observed from Fig. 7 that the 150PF (fine-sized) compacts achieved higher relative plateau densities than the 150P (coarse-sized) compacts. However, it was observed from Fig. 8 that the 150PF compacts were not as strong as the 150P compacts. The combination of the above observations was unexpected for a couple of different reasons. First, previous work [19,26,28,35] has indicated that the higher the relative plateau density of a particular polymer, the stronger the resultant compact. This seems logical if it is assumed that a uniform void size distribution is achieved in the resultant compact. Secondly, it has been reported [3,18-20,26] that the smaller the average particle size of a specific polymer, the higher the resultant green strength. This trend was reasoned in one paper [19] based on the number of interparticle contact points. The present results with the 150 grade PEEK compacts contradict

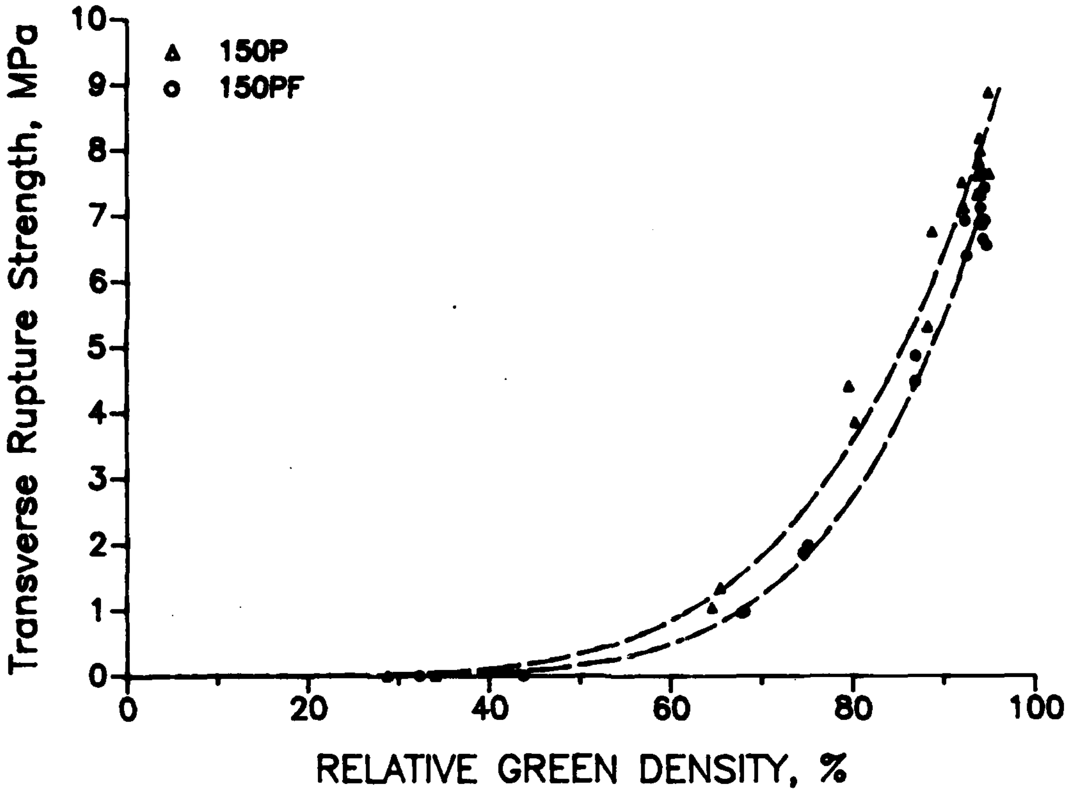


Figure 8 - Transverse Rupture Strength versus Relative Green Density for 150 Grade PEEK

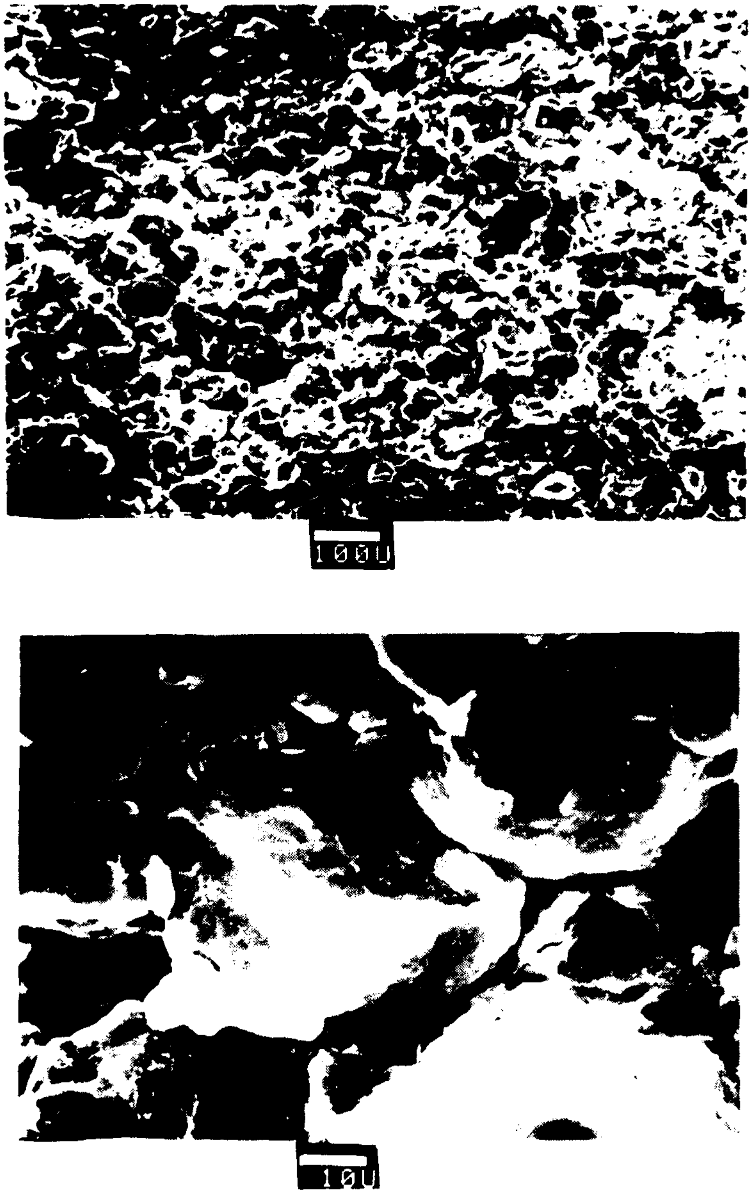


Figure 9 - SEM Photomicrographs of 150PF Fracture Surface

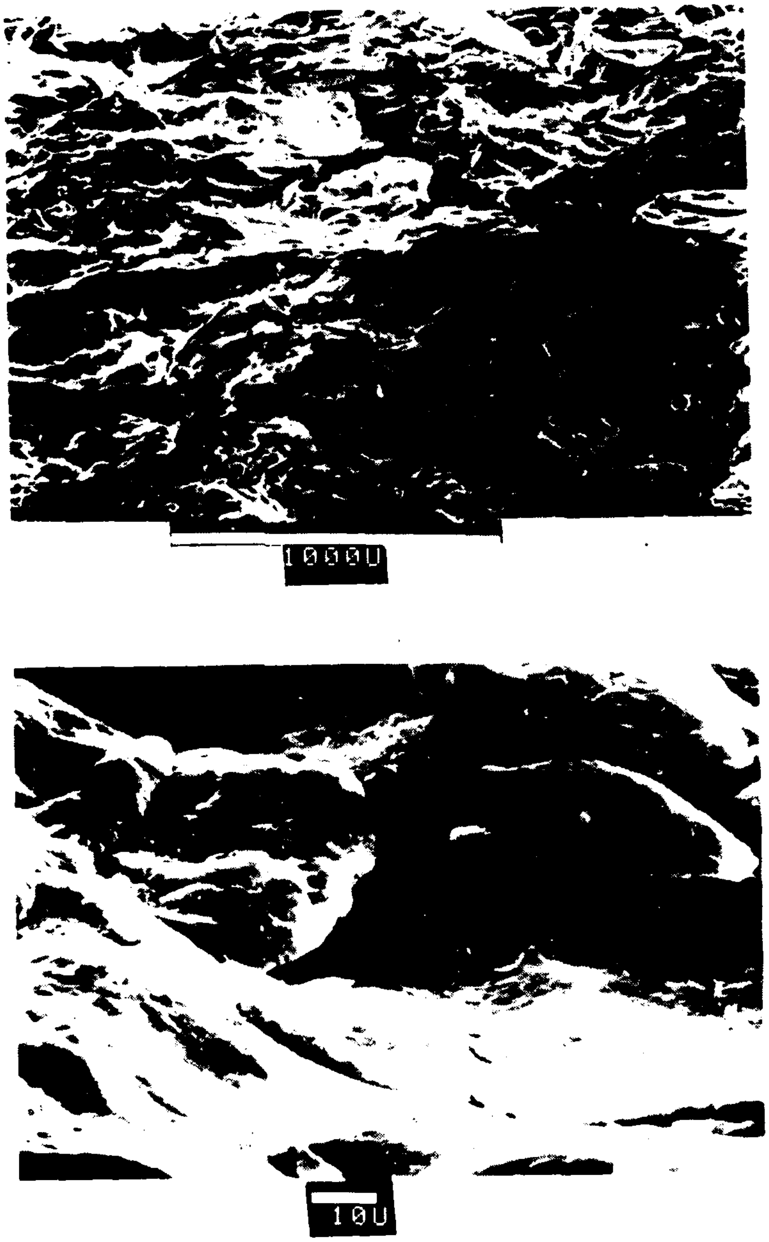


Figure 10 - SEM Photomicrographs of 150P Fracture Surface

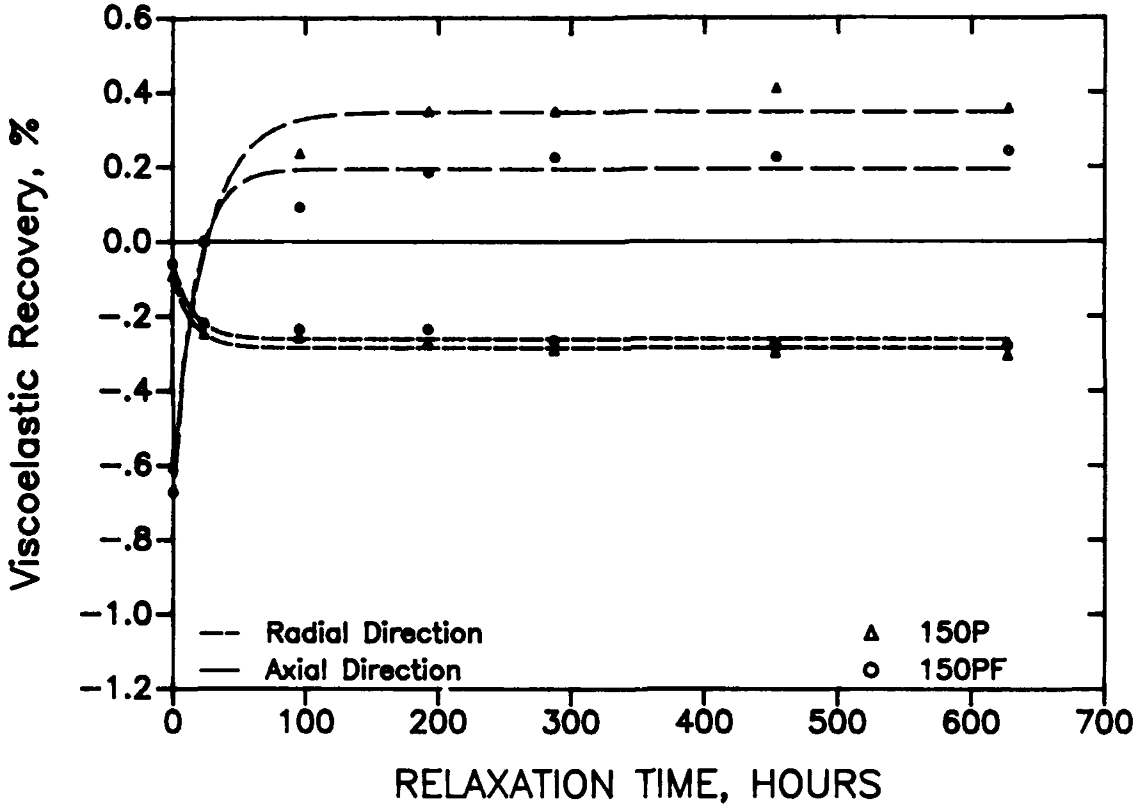


Figure 11 - Viscoelastic Recovery as a Function of Relaxation Time for 150 Grade PEEK

both of the above statements which were gleaned from previous investigations.

These disagreements could be due to differences in the apparent densities. Therefore, compressibility curves have been normalized using a densification parameter (DP) [28,32] which eliminates the initial differences between powders due to variations in apparent density. The DP represents the fractional densification and is defined by:

$$DP = (Dg - Da)/(Dt - Da) \qquad (1)$$

where, Dg = green density
Da = apparent density
Dt = theoretical powder density

A plot of the compressibility curves for the 150 grade materials using this DP is shown in Fig. 12. However, this is the same relative order of densification that was seen in Fig. 7. Similarly a graph of transverse rupture strength versus DP for these materials is shown in Fig. 13. Again it is seen that Fig. 13 is very similar to Fig. 8. Hence, the differences in the apparent densities are not the cause of the above disagreements.

Another possible reason for these contradictions is described as follows. Upon examination it can be seen that an assumption is made in using this DP to measure compaction efficiency. This assumption is that the entire polymer (both the crystalline and amorphous domains) plastically deform uniformly. This assumption may be valid for semicrystalline polymers compacted above Tg but not for ones compacted significantly below Tg as in the present case with PEEK. In order to modify this DP for semicrystalline polymers compacted below Tg, new assumptions need to be introduced.

Fig. 14 illustrates the current polymer powder compaction process. Initially the die is filled with powder which has a volume, Va, based on its apparent density (Fig. 14a). The apparent density is equal to the mass of the powder (M) divided by the apparent volume (Va). This apparent volume is comprised of the volume occupied by the powder (Vt) plus the volume of voids (Vv). The specimen is then compacted in a die using a pressure in the plateau region of the compressibility curve (Fig. 14b). While under this high pressure, the volume of porosity is approximately zero. Hence, the specimen under pressure has about the same density as the as-received powder, and occupies a volume, Vt. Upon ejection from the die, the green compact elastically recovers. The volume of the compact 24 hours after removal from the die (Fig. 14c) is named the green volume (Vg). This Vg is comprised of the powder volume (Vt) plus the volume of voids (Vv') introduced by the elastic and viscoelastic recovery processes.

The above description of the compaction process is valid for all classes of polymers. In the following discussion several assumptions are made based on applicable literature and on intuition in order to explain the behavior of PEEK powders compacted at room temperature. Previous findings [41] indicate that the deformation in most semicrystalline thermoplastics is initiated in the amorphous domains of the polymer. This

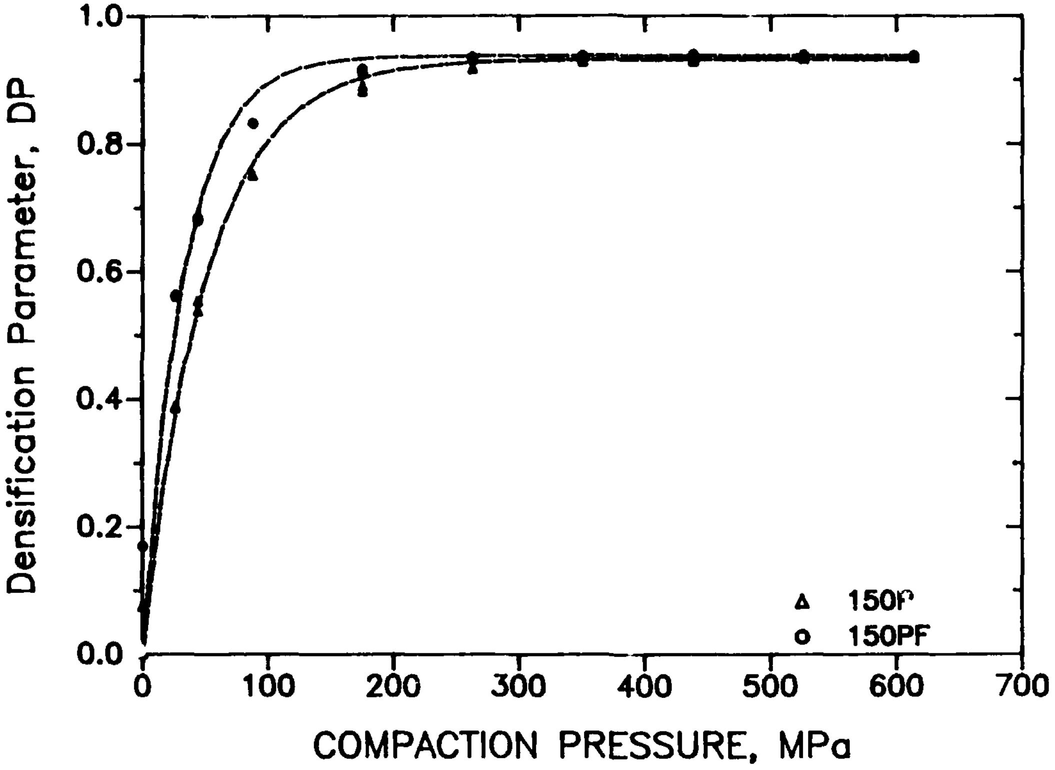


Figure 12 - Densification Parameter versus Compaction Pressure for 150 Grade PEEK Samples

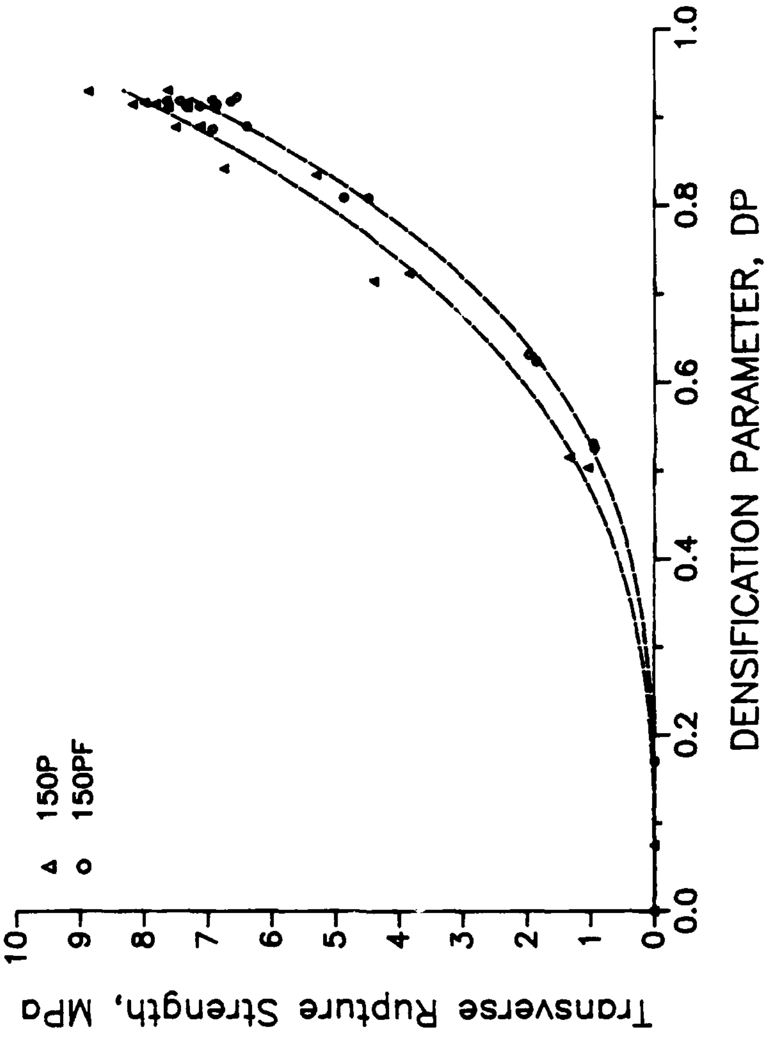
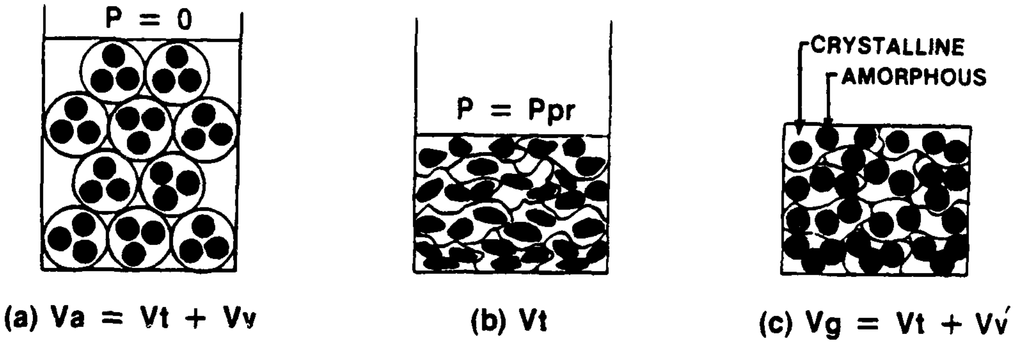


Figure 13 - Transverse Rupture Strength versus Densification Parameter for 150 Grade Compacts



(a) Va = Vt + Vv (b) Vt (c) Vg = Vt + Vv′

Figure 14 - The Compaction Process (a) Apparent Volume (Va) Before Compaction in the Die, (b) Approximate Theoretical Powder Volume (Vt) When Under Pressure in the Plateau Region (Ppr) and (c) the Green Volume of the Compact 24 hrs. After Ejection From the Die

is because the amorphous domains are usually weaker than the highly ordered crystalline domains. This generalization also appears to be true for PEEK based on data reported by Kemmish and Hay [42]. Their data indicates that increases in the degree of crystallinity results in marked increases in the yield strength and slight increases in the yield strain. The first assumption is based on these statements. This assumption is that during compaction, the amorphous domains are initially deformed elastically but are restricted in deformation by the crystalline domains and by the limited chain mobility when the temperature is below Tg. Following this initial deformation, further elastic deformation occurs in both the amorphous and crystalline domains until the elastic limit is reached. Once this elastic limit is exceeded, plastic deformation occurs.

It is readily apparent from the microscopy and green density results that the PEEK powders do undergo plastic deformation during compaction. However, it is not clear as to where within the polymer (i.e. amorphous or crystalline domains) this deformation occurs. Karbach [43] has investigated the plastic deformation of thin films of melt-spun PEEK below their Tg. He found that by deforming the polymer perpendicular to the initial chain axes that a 90^o rotation of the chain orientation occurred. He also reported that by deforming these PEEK films parallel to the initial axes that an extended lamellar morphology resulted.

It has also been reported [44,45] that entanglements in the amorphous domains of UHMWPE prevented it from fully crystallizing. This statement was deduced from the following. UHMWPE was exposed to low doses of radiation which caused chain scissions. These scissions were believed to have occurred on chains at the points of these high energy entanglements. Once the scissions occurred and these entanglements were removed, an increase in the degree of crystallinity was observed. This indicated that the segments at the interfaces between the crystalline and amorphous domains were under strain due to these entanglements and were prevented from crystallizing. The relatively low degrees of crystallinity for these PEEK powders compared to other semicrystalline polymers [2,5,28,36,39] may be attributed in part to entanglements and to chain stiffness due to the bulky phenyl groups positioned along the backbone of these chains. These restrictions on chain rotation and/or translation are more pronounced at temperatures below Tg. This is because the chains are associated with a minimal amount of free volume below Tg which severely hinders their segmental mobility.

The second assumption is now put forward based on the above discussion. It is believed that due to entanglements, the interlamellar amorphous regions in PEEK deforms elastically but that further deformation in this region is prohibited by entanglements and restrictions on the mobility of the chains. Because of these restrictions on the deformation of the interlamellar layer, it is felt that the crystalline lamellae must deform under the influence of further stress. Crystals are then pulled out and aligned in the direction of the applied shear, plastically deforming the crystalline regions while negligible flow occurs in the amorphous domains.

This second assumption implies that the rigid amorphous domains are already under significant strain due to crystallinity which leaves the crystalline domains alone to plastically deform in order to relieve the stress concentrations at the particle-particle interfaces. Since the elastic and viscoelastic deformations are quickly recovered within 24 hours after compaction, it is inferred from this assumption that the amorphous domains do not play a significant role in compact densification. This phenomenon is dramatically depicted in Fig. 14 by the shaded amorphous domains within the particles. Therefore, the amorphous contributions should be excluded from the DP expression (equation (1)) which determines the compaction efficiency. This modification is explained below. Also note that the particle surface indentations and the interparticle voids shown in Figs. 9 and 10 substantiate that more plastic deformation occurs in the powders of each grade which contain a higher degree of crystallinity. These observations give credence to this second assumption.

The third assumption is that due to the limited overall strain during the compaction process, the crystalline domains may be plastically deformed near the particle surfaces only without significantly reducing the degree of crystallinity of the bulk powder. This assumption was checked experimentally by performing DSC tests on shavings from the 150PF and 150P compacts, and negligible reductions in crystallinity were measured compared to the as-received powders.

The above discussion has led to the formulation of a crystalline domain based *densification parameter* to explain the compaction behavior of PEEK powders at room temperature. This new expression is constructed in the following paragraphs.

In order to mathematically exclude the amorphous domains from the measurement of compaction efficiency, a modified densification parameter (DP^*) is defined by:

$$DP^* = (Dg^* - Da^*)/(Dt^* - Da^*) \quad (2)$$

where, Dg^* = modified green density

$$= (M - Mam)/(Vg - Vam) = Mx/(Vx + Vv') \quad (3)$$

Da^* = modified apparent density

$$= (M - Mam)/(Vt - Vam) = Mx/(Vx + Vv) \quad (4)$$

Dt^* = modified theoretical powder density

$$= (M - Mam)/(Vt - Vam) = Mx/Vx \quad (5)$$

Mam = mass of amorphous domains
Mx = mass of crystalline domains
Vam = volume of amorphous domains
Vx = volume of crystalline domains

and where M, Vg, Va, Vv, Vv' and Vt are the same as previously defined.

Note that Dt^* is the same as the 100% crystalline density and that:

$$M = Mx + Mam \qquad (6)$$

$$Vg = M/Dg \qquad (7)$$

$$Va = M/Da \qquad (8)$$

$$Vt = M/Dt \qquad (9)$$

$$Mam = (1 - X)(M) \qquad (10)$$

$$Vam = Mam/Dam \qquad (11)$$

where, X = weight fraction crystallinity (Table 1)
Dam = amorphous density = 1.263 g/cc [9]

A normalized compressibility curve using the modified densification parameter for the 150PF and 150P PEEK compacts is shown in Fig. 15. This figure shows that the 150P powder has a higher compaction efficiency than the 150PF powder at high pressures and a lower one at low pressures. In the plateau region this is a reversal in the relative densification order compared to Figs. 7 and 12. Based on the DP^* model described above, the higher degree of compaction efficiency in the 150P powder at high pressures is primarily attributable to its higher degree of crystallinity (Table 1). At low pressures, particle shape and particle size distribution are the predominant parameters which influence compressibility.

The green strength results normalized in terms of the modified densification parameter are graphed in Fig. 16. This figure shows that the slightly higher strength values of the 150P compacts are consistent with its higher DP^* and higher degree of crystallinity compared to the 150PF compacts. This subtle reversal in the densification order gives strong support to the role of crystallinity and plastic deformation during the compaction of PEEK.

DISCUSSION

From communication with the manufacturer [46] it was learned that the fine-sized powders of each grade are produced by mechanically grinding the respective coarse-sized powders. The powder density results show that the fine-sized powders have substantially lower densities and hence lower crystallinity values than the coarse-sized powders. The DSC results also show this same trend in the degree of crystallinity but the differences are not as pronounced as they are from the density results. However, these techniques do show that there is a reduction in crystallinity due to the mechanical grinding process. Furthermore, it is clearly evident from both DSC and density results that the 450PF powder is not fully crystallized in its as-received form. Consequently, the DSC results

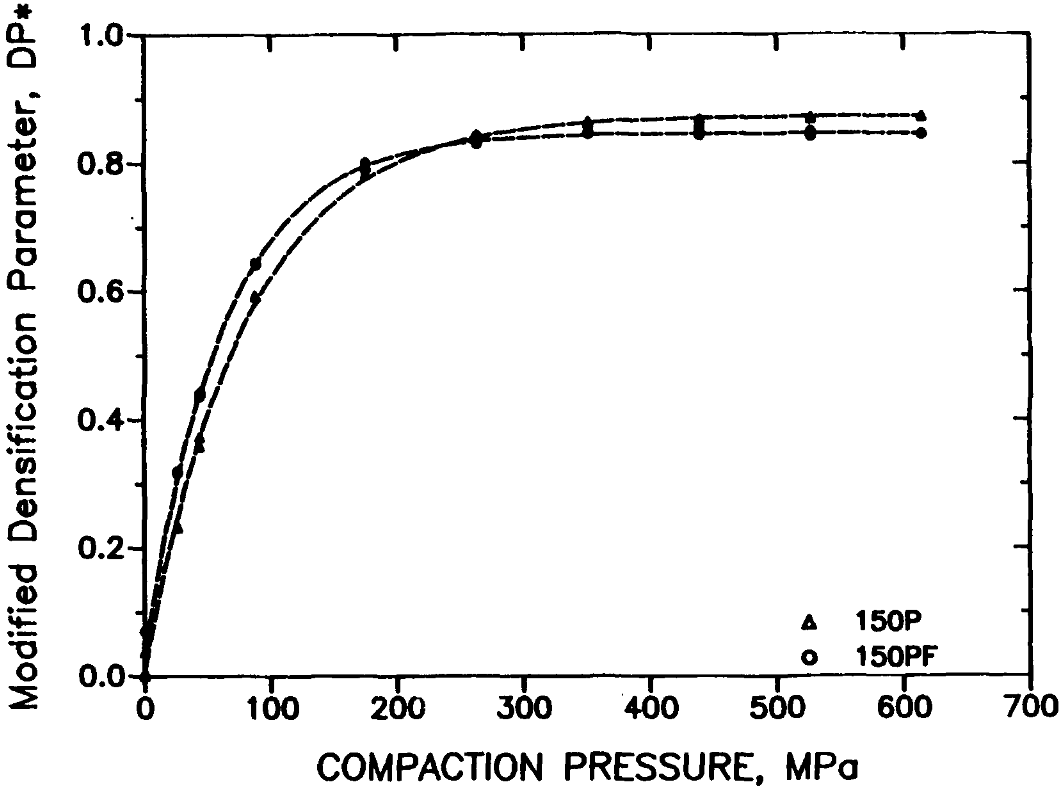


Figure 15 - Modified Densification Parameter versus Compaction Pressure for 150 Grade PEEK Samples

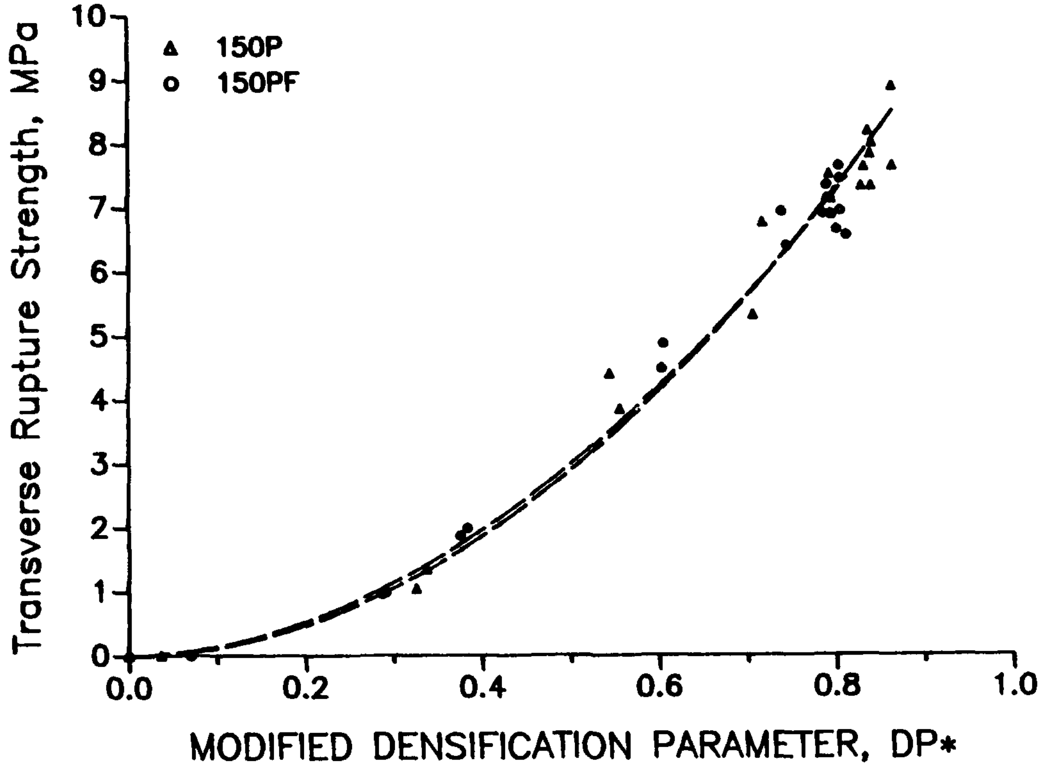


Figure 16 - Transverse Rupture Strength versus Modified Densification Parameter for 150 Grade PEEK Samples

reveal that it is the only material which has a higher degree of crystallinity after melting and recrystallizing than it does in its as-received condition.

The compaction results indicate that there are dramatic differences between the 150 grade and 450 grade of PEEK powders. According to the manufacturer [47], the 150 grade has a melt viscosity of about 1200 P at a temperature and shear rate of 380°C and 1000/s, respectively while the 450 grade has a viscosity of about 3000 P under the same conditions. This is due to its higher molecular weight compared to the 150 grade [48]. The 450 grade also has a slightly higher yield strain than the 150 grade [47]. The 450 grade powders when compacted at pressures up to 614 MPa were not plastically deformed enough to hold the specimens together when ejected from the die. However, the 150 grade compacted quite nicely under the same conditions. Given the limited deformation during compaction, the 450 grade powders probably were not deformed sufficiently beyond their yield strain. When removed from the die, the 450 powders recovered this elastic deformation leaving the compacts with insufficient strength for handling. Since the 450 grade could not be compacted at room temperature, compaction at higher temperatures was attempted. It was reasoned that quality compacts could be obtained by compressing these powders at a temperature above their Tg. Therefore, 450 grade powders were heated to 210°C and successfully compacted in a cold die. Another technique for enhancing the plastic deformation was also implemented with the 450 grade powders by mixing them with a hard filler (nickel powder). This technique was also effective in providing good particle interlocking necessary to achieve a quality compact at room temperature [38].

The compressibility curves for the 150 grade PEEK powders are similar to those of UHMWPE [28] and other polymers [32] in that a maximum or plateau density level is reached which is below the theoretical compact density. The compacts formed under high pressures (>300 MPa) are at or very close to the theoretical density in the die. However, the elastic component of the strain is recovered when the load is removed. This recovery results in a green density which is below the theoretical powder density. The relative plateau density level of the 150 grade PEEK compacts however, ranked relatively high compared to UHMWPE [28] and other thermoplastics [32]. These high densification levels with PEEK indicate that most of the particle deformation during consolidation is attributable to plastic deformation.

Introducing the role of crystallinity into the densification parameter (DP) appears to be logical in the present PEEK systems particularly because the amorphous region is below Tg during compaction. Calculation of the DP on the basis of the crystalline content yielded the modified densification parameter (DP^*). This DP^* is based on the assumption that the plastic deformation during compaction occurs in the crystalline domains alone. Using this DP^*, the relationship between the relative plateau densities and the green strength values appears to be consistent and in agreement with other systems in the literature.

If plastic deformation is accepted as the reason for the quality of these PEEK compacts then it is easy to see that increasing the degree of plastic deformation and mechanical interlocking can dramatically increase the green strength.

CONCLUSIONS

1) Semicrystalline PEEK powders were compacted at temperatures below their glass transition temperature to produce high density preforms which have enough strength for processing provided the particles can undergo sufficient plastic deformation.

2) Commercial 150 grade PEEK powders were compacted at room temperature to yield high quality specimens with adequate strength for handling and further processing, while the 450 grade did not undergo sufficient plastic deformation to be consolidated under the same conditions.

3) The fine powders (PF designated) of each grade of PEEK had markedly lower crystallinity values than their respective coarse powders. Additionally, the as-received 450PF powder underwent further crystallization when heated above the glass transition temperature.

4) Both 150 grade PEEK compacts reached about the same plateau or maximum density level despite differences in particle characteristics. These relative plateau density levels were 95.8% and 95.1% of the 150PF and 150P powder densities, respectively.

5) PEEK compact densities in the plateau region of the compressibility curve had only a slight dependence on post-compaction time since the axial dimension was expanding while the radius was contracting at nearly the same rate.

6) Most of the viscoelastic recovery took place within 24 hours and the total viscoelastic recovery in the radial direction was less than one half of one percent. Based on the 24 hour thickness, the axial viscoelastic recovery was also less than one half of one percent.

7) PEEK deformation during compaction is mainly due to plastic deformation of the crystalline regions of the polymer. The amorphous regions when below Tg are too restricted in mobility to be an important contributor to the consolidation process.

8) A new densification parameter, DP^*, based on the crystalline component of the polymer alone, was derived and appears to describe the compressibility and strength data consistently.

RECOMMENDATIONS FOR FUTURE WORK

The present investigation systematically exploited some of the fundamentals involved with fabricating PEEK compacts using P/M technology. Since no previous work has been reported on the cold compaction of PEEK powders, there is much need to extend this research. Additional areas which may significantly advance the state-of-the-art using this high performance polymer are suggested below.

1) Investigate the sintering behavior of PEEK compacts and characterize the resultant properties and the dimensional stability of the sintered product.

2) Exploit the use of ultrasonic compaction to fuse the polymer particles together. This may eliminate the need for a sintering step.

3) Utilize a dynamic (high rate) compaction technique to achieve higher green densities and to enhance plastic deformation.

4) Study the effect of adding conductive metal powders to the PEEK compacts to demonstrate the feasibility of fabricating conductive composites using P/M technology.

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