



A new method to prepare composite powders customized for high temperature laser sintering

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ABSTRACT

Composites have the potential to enhance the mechanical properties of components fabricated by additive manufacturing; however, the bottleneck is the limited number of polymeric composite powders available for this manufacturing process. This paper describes a generically new method to create composite powders that are suitable for High Temperature Laser Sintering (HT-LS). C-coated Inorganic Fullerene-like WS₂ (IF-WS₂) nanoparticles and graphene nanoplatelets (GNPs) have been chosen to demonstrate their incorporation into a high performance polymer matrix: Poly Ether Ether Ketone (PEEK). The morphological and physical property investigations have confirmed that the resulting composite powders exhibit the desired particle morphology, size, distribution and flowability for HT-LS applications. Further preliminary sintering results have demonstrated that they are comparable to the currently available commercial grade of PEEK powder HT-LS applications in terms of powder packing properties and flow ability. The new strategy reported here brings in great potential for the additive layer manufacturing of high performance polymeric composite components with improved mechanical and added functionalities by choosing the proper matrix and filler combination.

1. Introduction

Laser Sintering (LS) can produce complex parts directly from computer-aided design data, without the involvement of any expensive mould tooling [1], thus this technology has attracted great research attention. One of the key bottlenecks for the wider development and application of this technology is the lack of available powder materials. For a long time, polyamides (PA11 and PA12) have predominantly been used for a standard LS process, due to their excellent processability. The incorporation of reinforcement to polyamides can lead to composite components with improved performance and widened applications [2–4]. However, for harsh environments such as aerospace, defence and gas applications, the currently available polymers for LS cannot meet these requirements. High performance polymeric materials that are suitable for LS processing need to be developed.

PEEK is indeed one of such best candidates, due to its high melting point, good biocompatibility, high mechanical properties and chemical resistance [5–7]. However, due to the high melting temperature, it was not possible to process PEEK with standard LS systems with operating temperature only up to 200 °C until the High Temperature Laser Sintering (HT-LS) system, EOSINT P800 was introduced to the market. Berretta et al. have successfully applied the commercial grade PEEK (450PF Victrex) to the HT-LS system [8].

Incorporating various nanofillers into the PEEK matrix during HT-LS could further improve the mechanical properties and introduce crucial functionalities for different advanced applications. Yuan et al. physically added 5 wt% of graphite platelets into PEEK150PF powder and achieved a 40% increase in tensile strength, however for concentrations above 5%, in this case 7.5 wt%, the Micro-CT analysis revealed porosity in the microstructure [9], which was related to the poor powders flow and uneven heating absorption between the graphite platelets and PEEK powder during the HT-LS. One of the conventional ways to incorporate fillers into a polymer matrix is direct melt-compounding [10,11], however the high cohesive forces between nanofillers cause their severe agglomeration and poor dispersion inside the polymer matrix [12], which significantly limits the full potential of the nanofillers. Furthermore, modifying the melt compounded polymer into powder form with appropriate particle size and morphology requires following up milling or other size reduction processes that are very energy consuming and costly, needless to say the huge challenges for obtaining powders of round shape and proper size distribution by milling, especially for high performance polymers, such as PEEK [13]. Therefore, investigation to develop new strategies for making composite powder in a suitable morphology and cost-effective way is of highly importance.

Graphene nanoplatelet (GNP) is one of the best nanofillers in

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polymer-based composites, because of its excellent mechanical, electrical and thermal properties. Inorganic fullerene-like tungsten disulphide (IF-WS₂) is another interesting nanoparticles with hollow core and layered shell morphology. Such a unique microstructure brings in exceptional lubricating and wear resistant properties, and extraordinary shock-absorbing properties for ultimate protections, triggered us to incorporate it as nanofiller into the PEEK matrix. Previous studies have shown that adding 5 nm carbon coating on the surface of IF-WS₂ nanoparticles (C-coated IF-WS₂) can improve their thermal stability against oxidation [14].

We herein describe a completely new and cost-effective method for the preparation of composite PEEK powders using GNPs and C-coated IF-WS₂ as the nanofillers. We used salt template as a spacer and adapted a partial melting process to purposely create a ‘weak’ porous composite block. After leaching out the salt, the porous block can be easily broken into powder form. A number of researchers have used salt to produce porous PEEK structure for PEEK bioactivity improvement and better implant-bone contact [15,16]. However, to the best of our knowledge, this is the first time that this method is used to prepare composite powders suitable for Laser Sintering. Several types of near spherical-shaped composite powders with desirable particle size, distribution and flowability have been achieved. Finally, we will further demonstrate their suitability for HT-LS.

2. Experimental

2.1. Materials

PEEK 450PF was supplied by Victrex. Graphene nanoplatelets (GNPs, 6–8 nm thick × 5 μm wide) were purchased from ABCR GmbH & Co, Germany. NaCl (BioXtra grade with 99.5% purity) was purchased from Sigma Aldrich, UK. Full preparation details of the C-coated IF-WS₂ have been described in our previous papers [17,18].

2.2. Mixing the PEEK with nanofillers

C-coated IF-WS₂ particles were dispersed in water/ethanol solvent and probe sonicated for 5 min to obtain a stable suspension. 1 and 5 wt % of C-coated IF-WS₂ nanoparticles were further mixed with PEEK 450PF in the suspension, and subjected to another 10 min sonication a well-dispersed composite suspension was obtained. The mixture suspension was placed on a hot plate at 150 °C, to dry under continuous magnetic stirring. The PEEK-GNP composite powder was prepared by following the same process, using the same GNP nanofiller amounts of 1 and 5 wt%.

2.3. Fabrication of PEEK-IF-WS₂ and PEEK-GNP composite powders for HT-LS

All composite powders were dry mixed with NaCl powder, with an optimised NaCl/powder ratio of 6/1, and the mixture was then placed in a mould for cold press. After pressing, the mould was heated up in a cubic furnace at 400 °C, just above the melting point of PEEK, for a given time depending on the mould size to allow for the PEEK to melt and mix with the nanofillers. The salt particles were used as a spacer to generate and regulate the porous structure in the sintered blocks. After cooling, the salt was leached away by immersing the blocks in water under magnetic stirring for 3 h, leaving behind porous composite blocks.

The porous blocks were crushed using a simple food blender in distilled water for 5 and 10 min, to assess any effects of milling time on the particle size and morphology. After milling, the suspension was filtered and washed with water to remove any trace of salt, and the resulting composite powder was left for 24 h in an oven for drying. The dried powder was sieved using 212 μm and 125 μm meshes, separately. In each sample, around 80% of the total powder passed through the

125 μm mesh and it was collected for further characterization.

2.4. Fabrication of PEEK-IF-WS₂ and PEEK-GNP composites by HT-LS

The HT-LS process was carried out in a reduced build mode using the EOSINT P800 system [8]. Four different composites including two PEEK-IF-WS₂ composites with 1 and 5 wt% IF-WS₂ nanofiller and two PEEK-GNP composites with 1 and 5 wt% GNPs were synthesized. The samples are one layer thick and each sample was repeated three times with 1 exposure, 2 exposures and 3 exposures of laser beam with the laser power of 15 W, using a scan speed of 2550 mm/s and scanning interval of 0.2 mm in order to get a robust sample.

2.5. Structure and morphology characterization

Micro-CT (X-TEC Bench top CT 160 XI) was used to analyse the 3D structure of the porous blocks. An SEM (NOVALAB 600) was used to investigate the morphology and size of the powders, and the fracture surfaces of the sintered samples. To visualise the dispersion of nanofillers inside the PEEK matrix before and after the melting process, a JEM2100 TEM (operated at 200 kV) was used. TEM samples were prepared by dispersing a small amount of powder into acetone and drop casting on a holey carbon Cu grid.

2.6. Particle size distribution

A Saturn DigiSizer 5200 Micrometrics was used to measure the particle size distribution. To carry out the measurement, the target particles were dispersed in deionized water to form a suspension, which will be sampled at certain concentration to obtain the particle size distribution.

2.7. Powder rheology

To characterise the flow properties of the composite powder, a Freeman FT4 powder Rheometer was used to measure powder stability and flowability. The powder is placed inside a standard 25 ml split vessel and a 22 mm diameter twisted blade rotates and moves simultaneously into the powder sample, firstly to condition the environment, which aims to remove any undesired air trap and powder agglomeration, then to perform the test. The parameters are measured by means of torque, which is applied by the simultaneous counter-clockwise rotation of the blade at 100 mm/s whilst moving downwards. The energy necessary to displace, lift, promote stability or flow of the powder are respectively associated with Basic Flow Energy (BFE), Specific Energy (SE), Stability Index (SI) and Flow Rate Index (FRI); these values are obtained from the stability and flow rate test using the following equations:

$$SI = \frac{\text{Energy Test 7}}{\text{Energy Test 1}} \quad (1)$$

$$BFE = \text{Energy Test 7} \quad (mJ) \quad (2)$$

$$SE = \frac{\text{Up Energy Test 6} + \text{Up Energy Test 7}}{2 \text{ Split Mass}} \quad (mJ/g) \quad (3)$$

$$FRI = \frac{\text{Energy Test 4}}{\text{Energy Test 1}} \quad (4)$$

In the Stability Test, the blade was performed seven successive cycles with a constant speed of 100 mm/s throughout the pre-conditioned powder and by analysing the difference in the energy required for each cycle by means of SI, the Stability Test data were obtained.

The difference between BFE and SE is that the BFE refers to energy measured when the blade is rotating anti-clockwise into the pre-conditioned powder from the top of the vessel to the bottom, whilst the SE

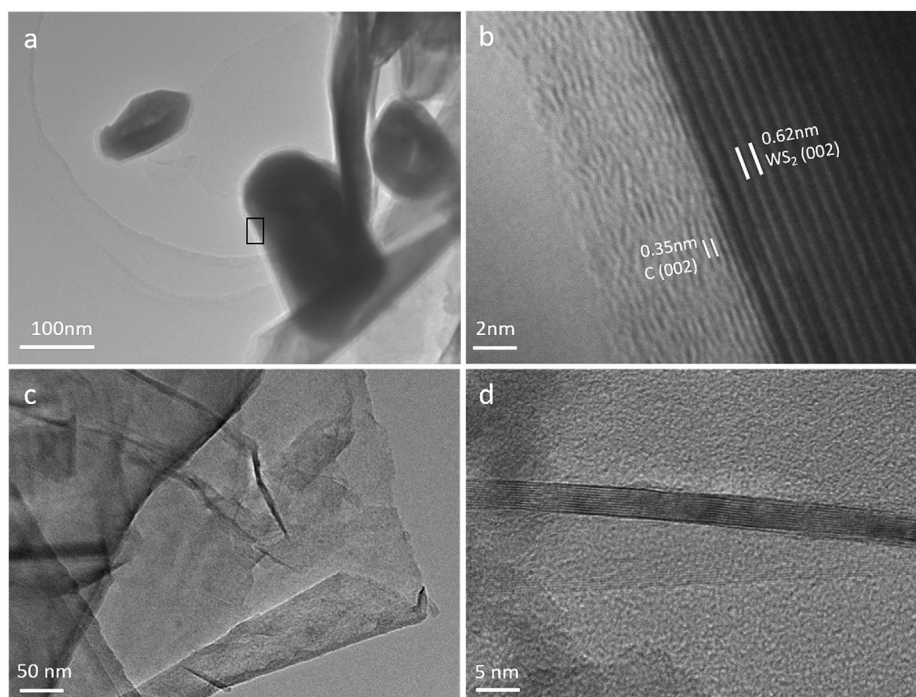


Fig. 1. TEM images. a) C-coated IF-WS₂ synthesized by the rotary CVD furnace, b) Higher magnification image from the marked area in Fig. 1a, c) Graphene nanoplatelet (GNP), and d) Higher magnification image from Fig. 1c showing a piece of GNP with a stack of 10 graphene layers.

is calculated during the upward clockwise travel of the rotating blade, therefore neglecting the powder packing factor.

Following the seven successive cycles at a constant speed to compute SI, the blade now rotates at four different speeds, hence creating an energy profile directly associated with the effect of flow rate on powder properties. The FRI is a measurement of flow capability and is calculated by the ratio of the energy required to displace the powder at the lowest speed (10 mm/s) and at the highest speed (100 mm/s).

3. Results and discussion

3.1. TEM study of the synthesized GNP and C-coated IF-WS₂

TEM images from the C-coated IF-WS₂ synthesized by the rotary CVD furnace are presented in Fig. 1a and b. As previously discussed by Xu et al., the sample contains both multi-layered hollow core particles with and a minute amount of nanoplatelets [17].

The higher resolution TEM image in Fig. 1b shows the well-crystallized IF-WS₂ particles with an interfacial layer spacing of 0.62 nm between the (002) plane. The smooth, continuous and multiple graphitic-layered C-coating of about 5 nm thick was grown by the rotary CVD technique. The carbon layers are separated by *ca.* 0.33–0.36 nm, which is very close to that of graphite ($d_{002} = 0.336$ nm). The reason for using C-coating on the IF-WS₂ nanoparticles are two-fold. Firstly, it improves the thermal stability and thermal conductivity of the IF-WS₂ nanoparticles; secondly, it offers stronger cohesion with the PEEK matrix than IF-WS₂. The TEM images in Fig. 1c and d confirm that the GNPs are stacks of 8–12 graphene sheets with an average thickness of 6–8 nm.

3.2. Morphological studies of the composite powders

We have chosen the 1 wt% PEEK-IF-WS₂ as an example to demonstrate the morphological features of the composites, and the results of other physical properties such as particle size distribution and powder rheology will be presented for all four composite powders.

Fig. 2 shows the digital and Micro-CT images of the porous PEEK

block after leaching out the NaCl. These images have confirmed that both pure (Fig. 2a) and the composite (Fig. 2b) samples have been prepared successfully by this technique. The micro-CT images (Fig. 2c–d) have shown that there are no cracks or any signs of degradation inside the block sample, and that the pores almost have the same size, as a result of the homogenous distribution of the salt particles in the PEEK matrix. The optimal 6:1 ratio of salt/PEEK allowed for a good framework formation for the PEEK, whilst maintained an open channelled feature for the easy salt leaching process. The colour difference between Fig. 2a and b is due to the addition of 1 wt% IF-WS₂, which makes the block look darker. This colour change with only 1 wt% IF-WS₂ addition indicates that the IF-WS₂ nanoparticles are properly mixed with the PEEK during melting, otherwise they would be leached out and the colour would be the same to the plain PEEK after washing. Direct evidence will be presented later to confirm the good nanofiller inclusion in the PEEK.

Fig. 2d shows the SEM image of the PEEK-1 wt% IF-WS₂ composite particles that were produced by milling the porous blocks into powders and after sieving. The particles have round and smooth surface without any sharp edges. Further details of the physical properties of the composite powders, as well as their comparison with commercial grade powders for HT-LS, will be presented in section 3.3.

To confirm that composite powders having been successfully created using this technique, TEM analyses were carried out on the PEEK-1 wt% IF-WS₂ and PEEK-1 wt% GNP mixture, before and after the melting process, and the results are shown in Figs. 3 and 4, respectively.

Prior to the melting process, the IF-WS₂ nanoparticles are only partially and loosely attached to the large PEEK powder during the mixing, as shown in Fig. 3a, owing to the Van der Waals forces. After the melting process, the IF-WS₂ nanoparticles are encapsulated inside the PEEK powder and their distribution is uniform, as shown in Fig. 3b. Fig. 3c shows a partially exposed IF-WS₂ particle from the PEEK matrix with the characteristics of a layered IF-WS₂ structure. The EDX line scan result from the selected black area in Fig. 3d shows the intensity of the W and S elements increases from the edge to the centre, which confirms that the black area is IF-WS₂.

Fig. 4a and b shows TEM images from the PEEK-1 wt% GNP mixture

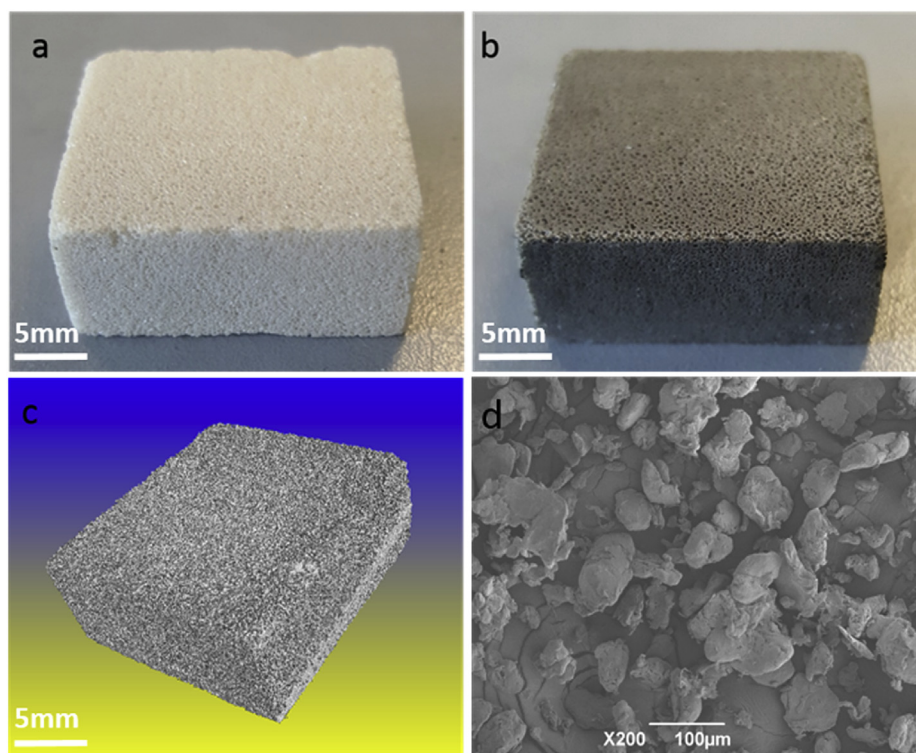


Fig. 2. (a) Plain porous PEEK block, (b) Porous nanocomposite block of 1 wt% IF-WS₂ in PEEK, (c) Micro-CT image of the composite PEEK block, and (d) SEM image of PEEK-1 wt% IF-WS₂ composite particles obtained from milling the block shown in (b).

before and after the melting process, respectively. As shown in Fig. 4a, prior to the melting process, GNPs are separated from the PEEK particle and attached to the TEM grit. However, Fig. 4b shows that the GNPs are encapsulated inside the PEEK matrix after the melting process. High resolution TEM image taken from the selected area in Fig. 4b shows the GNP characteristics of the layered structure, which confirms the

partially exposed area being graphene nanoplatelet. Therefore, both composite powders containing IF-WS₂ or GNP fillers have been generated successfully by this technique.

As shown in Fig. 3a, prior to melting, the mixture is a dry blend. This type of mixing has been proven by others that it is not suitable for the LS process to make high quality composite components, because the

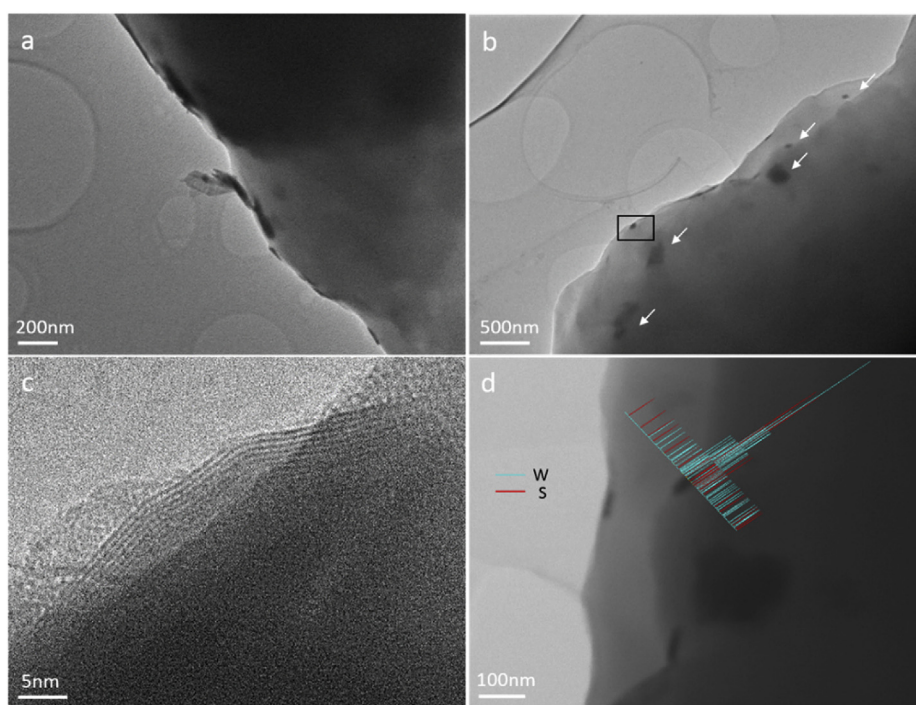


Fig. 3. TEM images of the PEEK with 1 wt% IF-WS₂. (a) Before the melting process, (b) After the melting process, (c) Higher resolution image from a selected area in Fig. 3b showing the layered IF-WS₂ microstructure, and (d) EDX line scan from a single particle inside the PEEK matrix.

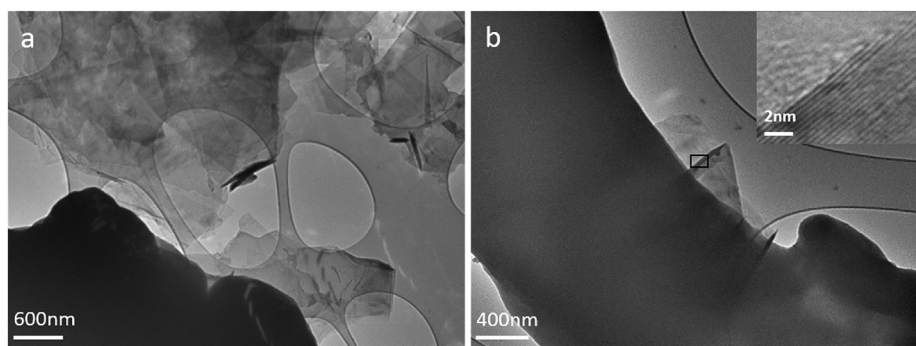


Fig. 4. TEM images from the milled PEEK-1 wt% GNP composite powder. (a) Before the melting process, and (b) After the melting process.

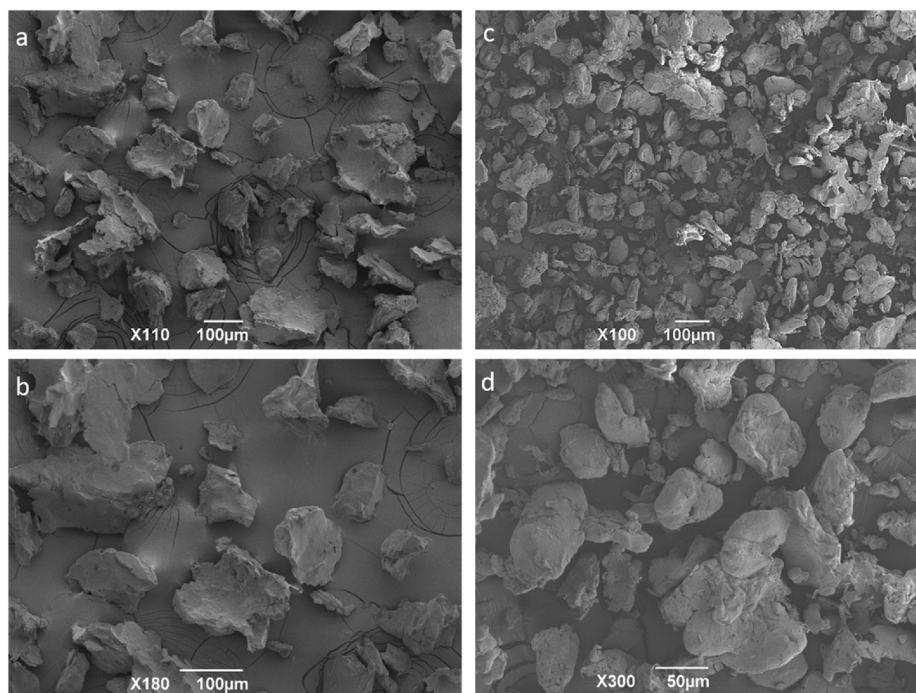


Fig. 5. SEM images of the milled and sieved composite powders with 1 wt% IF-WS₂ addition of various holding time. (a–b) 45 min and (c–d) 35 min.

different phases cause friction between these particles which results in poor powder flowability and therefore poor final density of the component. Additionally, the different thermal conductivities of the filler and PEEK would lead to inhomogeneous heat absorption/dissipation when exposed to the laser beam. Therefore, a complete sintering state might not be achieved for each deposited layer by using dry powder mixtures [9].

In contrast to the dry blend, the incorporation of nanoparticles into the polymer particles has several key advantages. Firstly, there will be no inhomogeneous heat absorption/dissipation during the HT-LS, since the filler nanoparticles are encapsulated inside the matrix powder. Secondly, for the same reason of encapsulation, there will be no second phase of different sizes, hence would enhance the powder flowability. Finally, as a composite powder with filler entrapped inside the PEEK, its exposed surface is as smooth as the virgin PEEK. PEEK particle surface is very important for HT-LS, because it effectively avoids the critical interfacial reaction between the filler and matrix during the rapid sintering process (which took place during the melting stage when producing the powder), eliminating the uncertainty and high demand for interfacial reaction between different phases during rapid laser sintering stage.

Having successfully created composite powders, we investigated their fundamental particle characteristics, optimise protocols for

achieving ideal powder shape, size and distribution, and finally assessed their suitability for HT-LS, in comparison to the plain PEEK powder.

Firstly, the study was concentrated on the influence of different furnace holding time to the melting process during the powder manufacturing. After milling the porous blocks into powders and after sieving, we visualised the particle morphology using SEM, and the results are shown in Fig. 5a–d. For the longer holding time of 45 min, Fig. 5a–b, the powder exhibit irregular shapes with larger particle sizes than those samples produced by shorter holding time of 35 min. The powders of shorter holding time as shown in Fig. 5c–d show features of smaller, rounder, free from sharp edges and more uniform than those shown in Fig. 5a–b.

The different holding time promoted different degrees of melting, mixing and crystallisation of the PEEK in the composite blocks, hence leading to different mechanical properties. The longer holding time enhanced the strength of the blocks and made them difficult to break and to obtain fine powders. As the main purpose of this investigation is to create composite powders, not to produce strong porous blocks, the partially melting condition, which resulted in easy-to-break porous blocks with round particles (Fig. 5c and d), was chosen as suitable for the rest of our study.

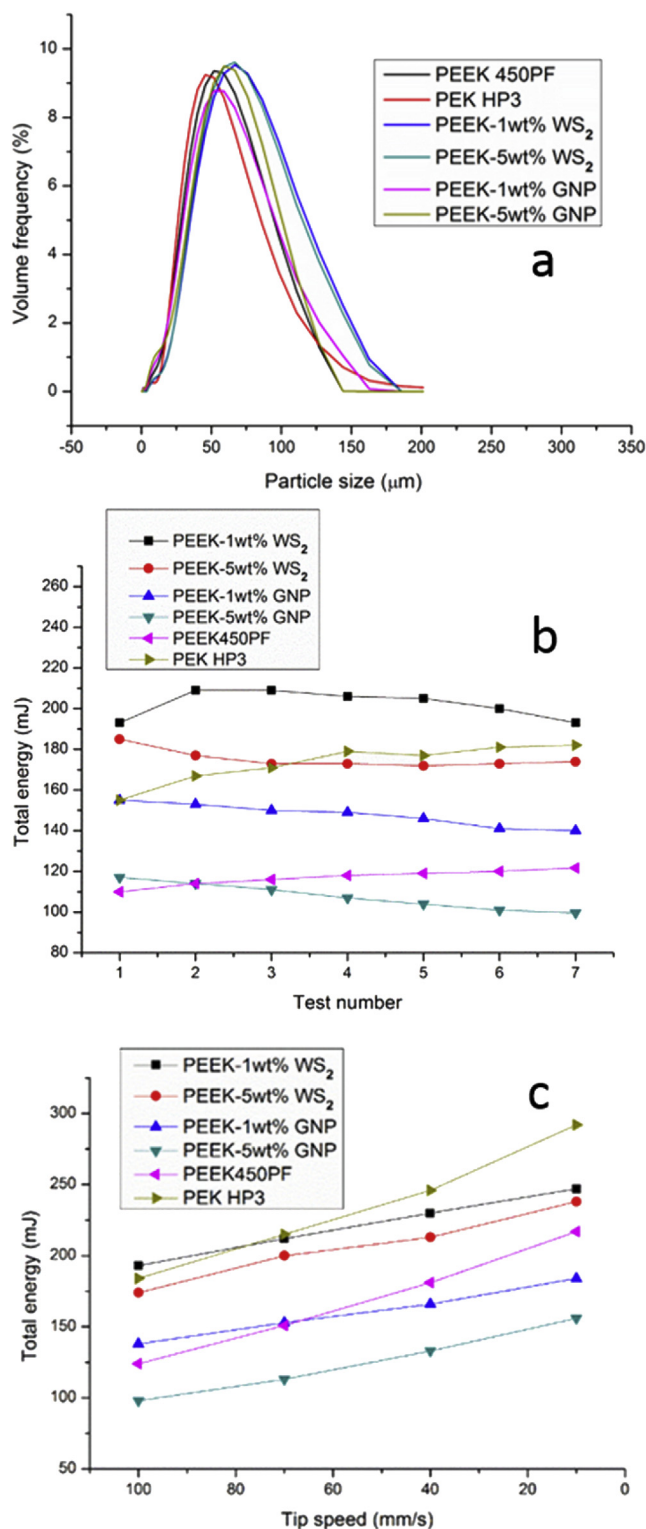


Fig. 6. a) Particle size distribution of commercial plain PEEK 450PF, and various nanocomposite powders, b) The energy profile of the samples obtained using a constant flow rate of 100 mm/s; and c) The energy profile of the samples using variable flow rates.

3.3. Particle size distribution (PSD) and rheological properties of the powders

To evaluate the PSD of the resulting nanocomposite powders, we compared them with the commercial plain PEEK 450PF powder and

Table 1
Stability Index (SI) and Flow Rate Index (FRI) of PEEK composite samples compared with PEEK 450PF and PEK HP3.

	Stability Index (SI)	Flow Rate Index (FRI)
PEEK-1wt% WS ₂	0.994	1.286
PEEK-5wt% WS ₂	0.942	1.36
PEEK-1wt% GNP	0.908	1.33
PEEK-5wt% GNP	0.852	1.59
PEEK 450PF	1.11	1.75
PEK HP3	1.17	1.58

EOS PEK HP3 the only commercial grade powder available for HT-LS. The results are presented in Fig. 6a. As shown in the PSD profiles, PEK HP3 and PEEK 450PF exhibit a narrow range of PSD pattern between 0 and 150 μm that is centred near to 50 μm; and the 1 wt% PEEK-GNP PSD is also centred at 50 μm, with a slightly wider distribution (1–170 μm) than the PEEK 450PF. The PSD profiles of the other three nanocomposite powders, 1 wt% IF-WS₂, 5 wt% IF-WS₂ and GNPs, are all centred at around 70 μm, with a similar distribution between 0 and 180 μm. These PSD results are close in distribution to the PEK HP3 and plain PEEK 450PF, and therefore these powders are deemed as suitable for laser sintering [19].

Fig. 6b and c presents the results of the stability and variable flow rate tests for the composite powders, against the plain PEEK 450PF. Fig. 6b shows that all powders display a smooth and nearly flat energy profile, which indicates that the powder was not affected by being made to flow, and that there was no sign of agglomeration, moisture uptake or electrostatic charges during the 7 cycles of test. Table 1 summarises the Stability Index (SI) results obtained from Eq. (1). In an ideal situation where there is no difference between test 7 and test 1, the SI is equal to 1; whilst in reality minor variations occur. For the composite samples, all their SI obtained are within $0.9 < SI < 1.1$, suggesting that the composite powders are highly stable (Table 1).

The variable flow rate test was carried out by recording the energy profile for each sample at four different flow rates (Fig. 6c). The Flow Rate Index (FRI) was obtained by inputting the total energy values at the lowest and the highest flow rate from Fig. 6c into Eq. (4) and the results were summarised in Table 1. In general, cohesive powders are expected to be more sensitive to the flow rate and non-cohesive granular powders would be less sensitive to the flow rate. The granular powders with less sensitivity to flow rate are expected to reveal almost same total energy when altering the flow rate and the FRI is close to one. As shown in Fig. 6c, the total energy has slightly increased with reducing the flow rate in all samples. However, the FRI data show the detailed difference among samples. For example, the FRI value for PEEK 450PF is 1.75, which is higher than other samples indicating more sensitivity of this powder to the flow rate (Table 1).

Such results are expected, as PEEK 450PF is not a powder grade designed for the LS. However, the flow sensitivity of the composite powders is much lower than that of the plain PEEK 450PF. For example, the powders with 1 and 5 wt% IF-WS₂, and 1 wt% GNP addition have the FRI value of 1.28, 1.36 and 1.33, respectively, which is lower than PEEK 450PF and even lower than that of the EOS PEK HP3 powder (1.58). This excellent low sensitivity to the flow rate is beneficial for the powder application in HT-LS, and such feature could be originated from the round powder shape and the partially exposed nanofillers of WS₂ and GNP that both have excellent lubricating properties, as shown early in Fig. 5. The increased FRI of 1.59 in the 5 wt% GNP sample indicated more inter-particle cohesion compared with other powders.

The Basic Flow Energy (BFE) and Specific Energy (SE) of different powders have been calculated from Eqs. (2) and (4), and the results are shown in Fig. 7. All composite powders except the 5 wt% GNP sample exhibit higher BFE than the plain PEEK 450PF, and the 1 wt% IF-WS₂ sample shows an even higher BFE value than that of the EOS PEK HP3 powder. The higher the BFE values, the better packing properties of the

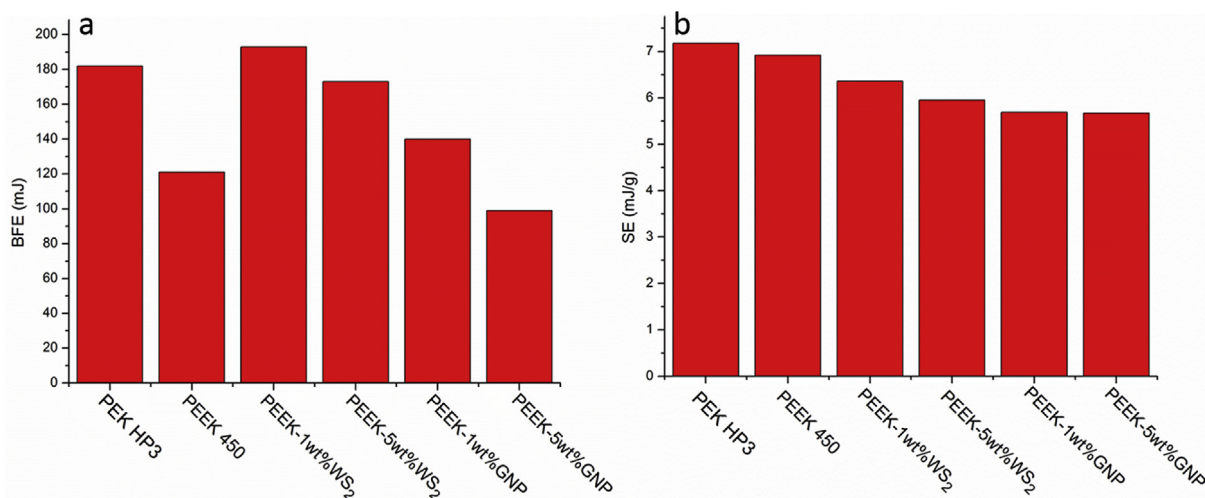


Fig. 7. (a) Basic Flow Energy (BFE), and (b) Specific Energy (SE) of PEK HP3, PEEK 450PF and composite powders.

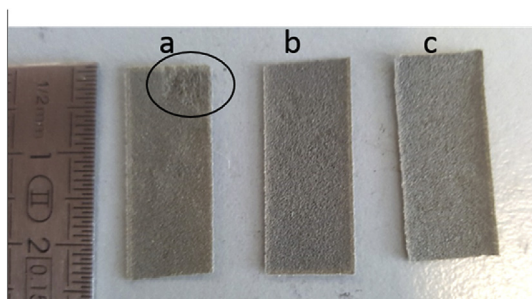


Fig. 8. Laser Sintered PEEK-5 wt% IF-WS₂ composites, with 1, 2, and 3 laser exposures from a to c, respectively.

powder, which is translated as the increased resistance to the downward travel of the blade. The good packing property of the composite powders is likely to result in high sintering density for components, which is directly related to mechanical properties.

The SE, measuring the energy required during the upward travel of the blade, is only dependant on the shear forces between the particles due to low environmental stress. In this context, all tested powders present very similar SE values (Fig. 7b), indicating a comparable level of mechanical interlocking and friction between the composite powders and the two commercial contrast powders. The very good powder rheology properties of the nanocomposite powders, better than the plain PEEK 450PF and comparable to the EOS HP3, is believed to arise from the overall powder shape, size, distribution and surface morphology. In this context, we have shown that the key morphological characteristics of the new composite powders, including round particles free of sharp edges, smooth surface, good size distribution, have led to the high stability, low sensitivity to flow rate, and good rheology features that are highly desirable for HT-LS applications.

3.4. HT-LS validation

Fig. 8 shows one layer thick of PEEK-5 wt% IF-WS₂ composite laser-sintered using single, double and triple exposures of laser beam. Sample (a) has an unsmooth surface with some chipped parts (circled) indicating 1 exposure of laser is not sufficient to obtain a completely melted sample and some parts remained powder. Samples b and c show a coherent fully melted appearance with no signs of polymer degradation.

Fig. 9 shows the SEM images from four different composites sintered using the triple laser exposure. All samples exhibit a non-smooth

surface with cavities. It is evident that sintering of just one layer is not enough to remove the irregularities related with the powder bed underneath, and inhomogeneity will appear in single layer samples even at optimal laser power conditions. However, PEEK-IF-WS₂ composites in Fig. 9c and d show a more homogeneous surface compared to the PEEK-GNP composites (Fig. 9a and b). Different times of laser exposure did not result in much difference in the surface morphology for the PEEK-1 wt% GNP composites, however a much smoother surface was evident for the PEEK-1wt% IF-WS₂ composites.

This result is consistent with the powder rheology data. PEEK-IF-WS₂ composites showed higher BFE value than the PEEK-GNP composites (Fig. 7a). Higher BFE in the PEEK-IF-WS₂ composites indicates the better packing properties of the powder, which probably leads to smoother surface of the laser-sintered samples (Fig. 9c and d). The microstructure of the PEEK-5 wt% GNP composite showed unsmooth and flaky surface (Fig. 9b). This is probably the result of these weaker packing parameters in PEEK-5 wt% GNP composite compared with other samples, such as BFE, SE and FRI (Table 1 and Fig. 7).

The process of mixing, melting the blend into a porous structure and the milling to create the nanocomposite powder is an effective way to achieve good nanofiller dispersion in the powders that are suitable for HT-LS applications. Further studies on the mechanical behaviour of these materials will be presented in a follow on paper.

4. Conclusion

A new, cost-effective and environmentally friendly technique has been developed for the creation of various novel composite powders that are suitable for HT-LS additive layer manufacturing. The composite powders were produced via the partial melting of porous PEEK blocks following by grinding, without changing the physical properties of each constituent. Microstructural characterizations have confirmed that the IF-WS₂ and GNP nanofillers were encapsulated inside the PEEK matrix, and that the surface of the composite particles was smooth and round, which makes them potentially suitable for HT-LS applications. Further powder rheology studies have shown that the nanocomposite powders exhibited improved powder rheology when compared with the plain PEEK 450PF, comparable to the EOS HP3 PEEK. Using single layered samples fabricated by HT-LS, we have finally validated that the resulting composite powders are suitable for HT-LS applications. The PEEK-IF-WS₂ thin film showed less cavity and exhibited a smoother surface than those of PEEK-GNP. This new strategy can be extended to various micro- and nano-fillers, in countless combinations with diverse polymeric matrices, for new and functional composite powders design. It is expected that the new composite powders could truly open new

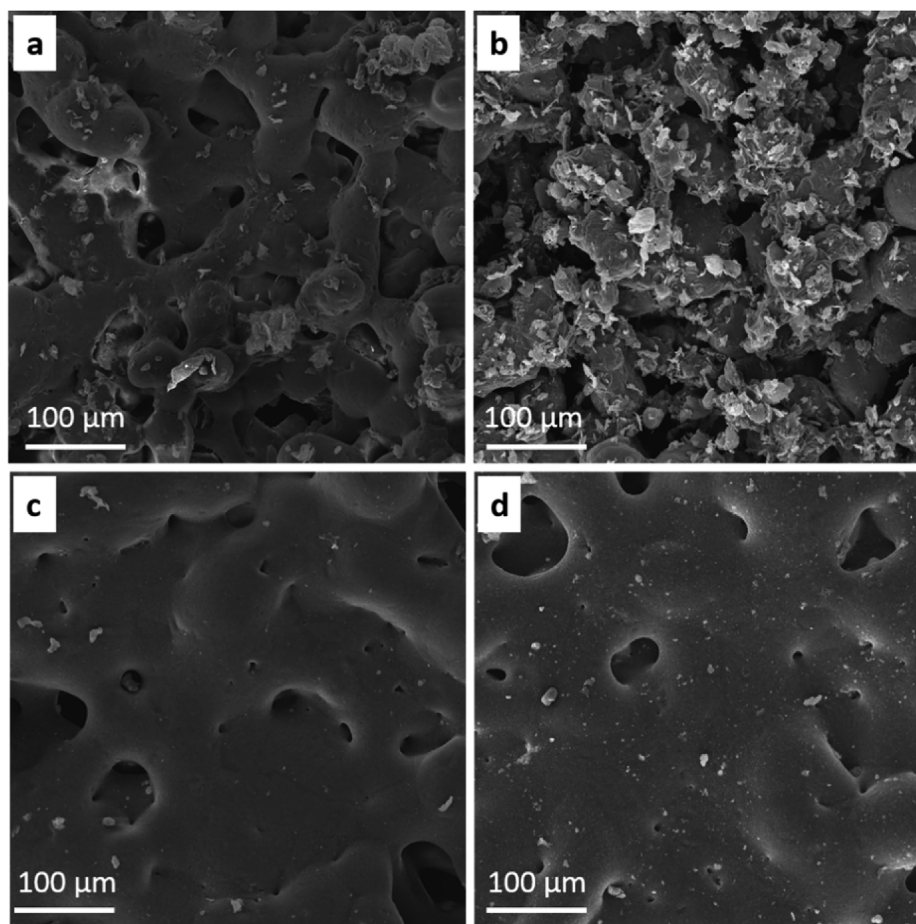


Fig. 9. SEM images of surface morphology of HT-LS fabricated composites after 3 times laser exposures. (a) 1 wt% GNP and (b) 5 wt% GNP; (c) 1 wt% IF-WS₂ and (d) 5 wt% IF-WS₂.

revenue for the manufacturing of high performance multifunctional composite components by LS and HT-LS.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at <https://doi.org/10.1016/j.compscitech.2018.08.006>.

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