

Development and Characterization of PI and PI-Based Composites for Industrial and Advanced Engineering Applications Using SPS Technique: A Mini Review

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Abstract

Recently, the application of spark plasma sintering (SPS) as a novel method in the fabrication of polymer and polymer-based composites have drawn the attention of researchers and industries. Owing to its advantages over the conventional polymer and polymer-based composites processing method. SPS is a sintering technique for producing components with high density at relatively low sintering temperature and short sintering time. However, as SPS allows good control of the sintering operation parameters and material microstructure compared to the traditional sintering technique, for example hot pressing, the review study focused on the fabrication of polyimide (PI) and PI-based composites using SPS technique. The unique characteristics ascertained from the SPS sintered PI-based micro/nanocomposites were discussed. Hence, it was observed that the mechanical, electrical, thermal stability, anti-wear, and corrosion resistance of PI-based composites could be improved using the SPS process favourable for industrial and advanced engineering applications. Furthermore, the authors ended the review with challenges and recommendations for future advancement of SPS applications practicable for commercial production. Thus, the review will pave the way for future research among polymer scientists, industrialist, and engineer.

Keywords

Spark plasma sintering, polyimide, polyimide composites, mechanical, and dielectric.

1. Introduction

With the rapid development of automobiles, aerospace, microelectronics, and energy storage technology, polymers and their adventures are urgently needed. Polymer matrices, such as polyethylene, epoxy, polyamide, and polyimides have been used in the design and fabrication of micro and nanocomposites for most engineering and technological applications. Owing to their specific characteristics, such as strength-to-weight ratio, low dielectric constant, anti-wear, chemical stability, and durability [Zang et al., 2017; Xu et al., 2021]. However, among these polymer matrices, polyimides (PIs) have gained much attention in recent years in the fabrication of polymer-based matrix composites because of their physiochemical characteristics, like mechanical strength, thermal stability, and chemical stability [Ogbonna et al., 2020]. And polyimide (PI) as one of the most high-performance polymers reportedly maintains these characteristics even under harsh environment [Rajan et al., 2022]. Thus, with the inherent properties of PI and its application as polymer matrix in fabricating both micro and nanocomposites, several studies have been conducted in obtaining through superior properties of PI-based material filled with inorganic fillers (e.g. Al₂O₃, TiO₂, SiO₂, BaTiO₃, and MgO) using different fabrication methods [Ai et al., 2020; Li et al., 2022; Ren et al., 2023]. Such as solution casting, in situ polymerization, hot pressing, sol-gel, and spark plasma sintering. However, of all the stated fabrication methods used in preparing PI and PI-based composite materials for engineering applications, spark plasma sintering (SPS) reportedly novel, cost effective, and easy to operate with low temperature sintering [Adesina et al., 2021; Tokita, 2021; Ogbonna et al., 2022]. Thus, the review study focuses on the fabrication and characterization of inorganic fillers

reinforced PI-based composites produced employing SPS manufacturing technique (Figure 1), as well as challenges and recommendations for future enhancement.

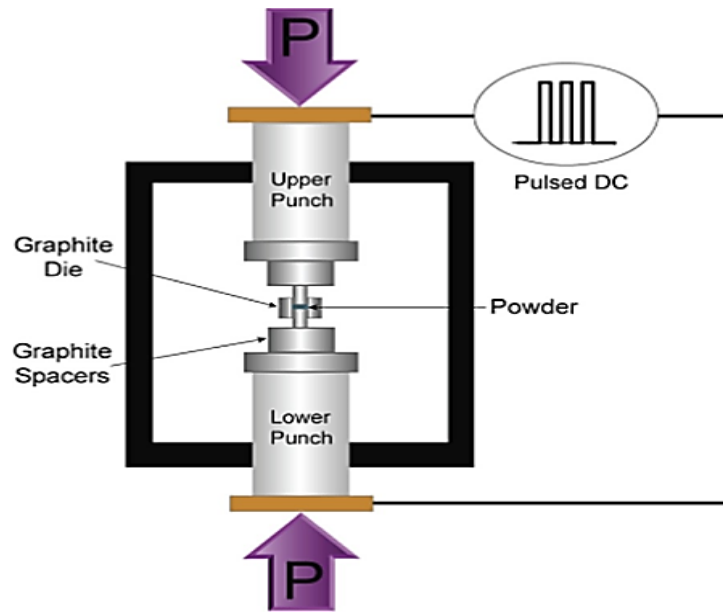


Figure 1. Schematic diagram of the SPS apparatus [Chuvildeev et al., 2015]

2. Literature Review

2.1 Literature review on PI-based composites filled with metal and inorganic fillers (micro and nanoparticles) using SPS method

SPS in the preparation of polymer-based composites reportedly overcome numerous challenges encountered while involving the traditional route, which are high temperature requirement and complete melting of polymer, limitation in the use of fillers, and longer time taken to achieve homogeneity in composites [Sebileau et al. [2017; Adesina et al., 2021]. In addition, more detail about SPS could be found in Matizamhuk (2016) work presented in the open literature.

However, positing that SPS produces composite component to near net shape with minimal defect and porosity (Matizamhuka, 2016), Ogbonna et al. (2022) studied the influence of electrical-corrosion-resistance (ECR) glass on the mechanical, tribological, and dielectric behaviour of PI composites at varying ECR content (0, 5, 10, 15, and 20 wt%) prepared using SPS technique. The microstructural evolution of the composites was examined using scanning electron microscope (SEM). Meanwhile, nanoindentation tests, wear tests, and LCR-meter tests were conducted to characterize the mechanical, tribological, and dielectric characteristics of the samples, respectively. The SEM results show uniform dispersion of the ECR particles within the PI matrix structural network. Addition of the ECR particles into the PI-based composites yielded improved hardness (~29%), elastic modulus (74.5%), wear resistance, and dielectric performances compared to the pristine PI. Here, optimum wear resistance was recorded at ECR/PI composites loaded with 15 wt% ECR. Hence, suggesting the candidature of ECR reinforced PI composite material for automotive and power applications demanding excellent hardness and elastic modulus, good wear resistance, and low dielectric constant.

Furthermore, Ogbonna, Popoola and Popoola, (2023) reported on the enhancement of mechanical and tribological behaviour of PI-based composites filled with TiO₂ nanoparticles. The PI-based nanocomposites were produced at 0, 4, 6, and 8 wt% TiO₂ nanoparticles employing SPS process. The microstructure, mechanical, and wear response of the resultant nanocomposites were examined by employing SEM, nanoindenter, and tribometer analyzer. The SEM results indicated that the TiO₂ nanofillers were homogeneously distributed into the PI matrix as could be seen in Figure 2. Although, few agglomerations occurred at the TiO₂/PI nanocomposites containing 8 wt% TiO₂ nanoparticles. The density of the PI reportedly increases with the increasing TiO₂ nanoparticles concentration. However, the hardness, yield strength, and modulus of the PI increased with the incorporation of the TiO₂ nanofiller particles by 314.8%,

313.3%, and 59.5%, respectively, at 8 wt% loading. Also, the experimental result depicted improved wear resistance of the TiO₂/PI nanocomposites in comparison to the virgin PI, especially on the 6 wt% TiO₂ particles loading. Increased wear rate of the nanocomposites beyond 6 wt%, though lower than that of the neat PI (See Figure 3) could be ascribed to the little agglomeration noticed in the TiO₂/PI nanocomposite containing 8 wt% TiO₂ (Figure 3d)

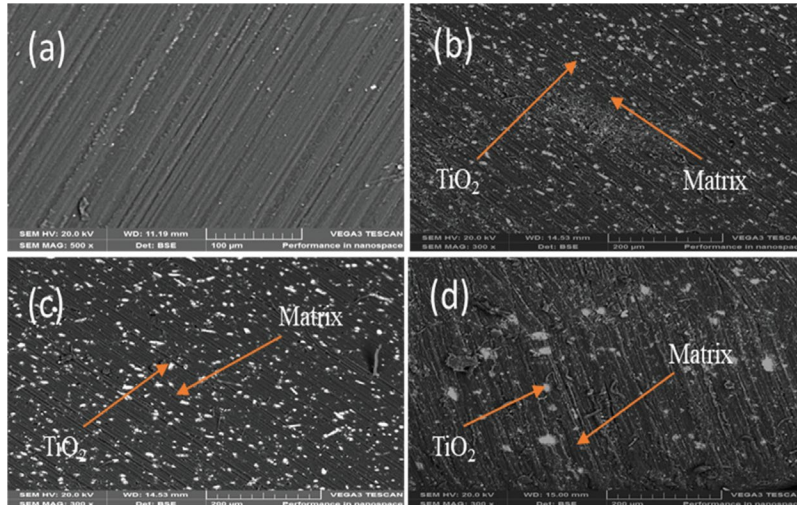


Figure 2. The morphological image of the SPS sintered TiO₂/PI nanocomposites; (a) 0 wt% TiO₂, (b) 4 wt% TiO₂, (c) 6 wt% TiO₂, and (d) 8 wt% TiO₂ (Ogbonna, Popoola and Popoola, 2023)

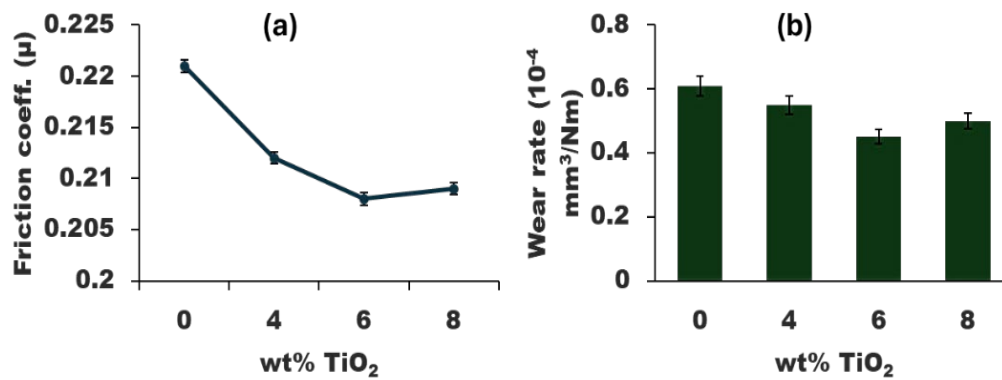


Figure 3. Tribological response of the SPS sintered TiO₂/PI nanocomposites sliding against stainless steel ball under 20N load, 150rpm sliding speed, and 20min duration (Ogbonna, Popoola and Popoola, 2023)

In another study, Schwertz et al. (2015) observing that SPS is efficient for sintering and consolidating dissimilar materials in different domains for structural as well as biomedical application (Watanabe et al., 2011), studied the influence of aluminium powder on the mechanical performance of PI composites for structural applications using SPS method. In the study, for sintering temperature as low as 350 °C, results show homogeneous mechanical characteristics. Increase in density of PI by 17.9% was recorded with the addition of the aluminium (Al) powder particles. Maximum compression strength (596 MPa) and elastic modulus (4.06 GPa) were noted in the Al/PI composites containing 25 wt% Al compared with the pure PI and other reinforcement.

Additionally, Schwertz et al. (2014) examined the mechanical properties of spark plasma sintered PI-based material at 300 °C and 320 °C sintering temperature. In the study, relative densities higher than 99% were achieved after the PI material consolidation. From the in-situ compression and hardness test, maximum hardness (88 shore D), compressive

strength (736 MPa), and elastic modulus (3.30 GPa) were reported at the 320 °C sintering temperature, 40 MPa pressure, 10 °C/mm heating rate, and 5 min dwell time. And such improved mechanical strength parameters refer to the uniform grain structure of the sintered PI.

Again, Schwertz et al. (2015) carried out a study on the optimization of SPS processing parameters affecting the characteristics of PI. In the study, employing a technique incorporating design of experiments with physical, structural, and mechanical properties evaluation. The influence of SPS parameters including temperature, pressure, holding time, and cooling rate on density, mechanical characteristics, and microstructure of PI were evaluated. The results showed that the mechanical characteristics of the PI material were enhanced through increasing the sintering temperature up to 350 °C. Here, the optimal SPS parameters were noticed at a sintering temperature of 350 °C with pressure of 40 MPa and holding time of 5 minutes. And under these conditions, the relative density, shore hardness, compressive strength, and Young's modulus were measured to be 99.6%, 87.3, 738 MPa, and 3.43 GPa, respectively. Meanwhile, when sintered at 320 °C under a pressure of 100 MPa, an increase in holding time above 5 min was crucial to improve the mechanical characteristics.

Ogbonna et al. (2024) investigated the enhancement of PI matrix mechanical, thermal stability, and dielectric characteristics with the incorporation of boron-free E-glass reinforcement particles (0, 5, 10, and 15 wt%) using SPS technique. Incorporation of the boron-free E-glass into the PI remarkably improved its mechanical (harness by 9.4% and modulus by 43. 6%), thermal stability, and dielectric characteristics. Boron-free glass reinforced PI composites containing 15 wt% boron-free E-glass exhibited the lowest dielectric constant and dissipation factor with reduced electrical conductivity (Figure 4). Hence, in consideration, the exhibited properties of the composites are suggested for mechanical loadbearing, thermal operation, and microelectronics applications.

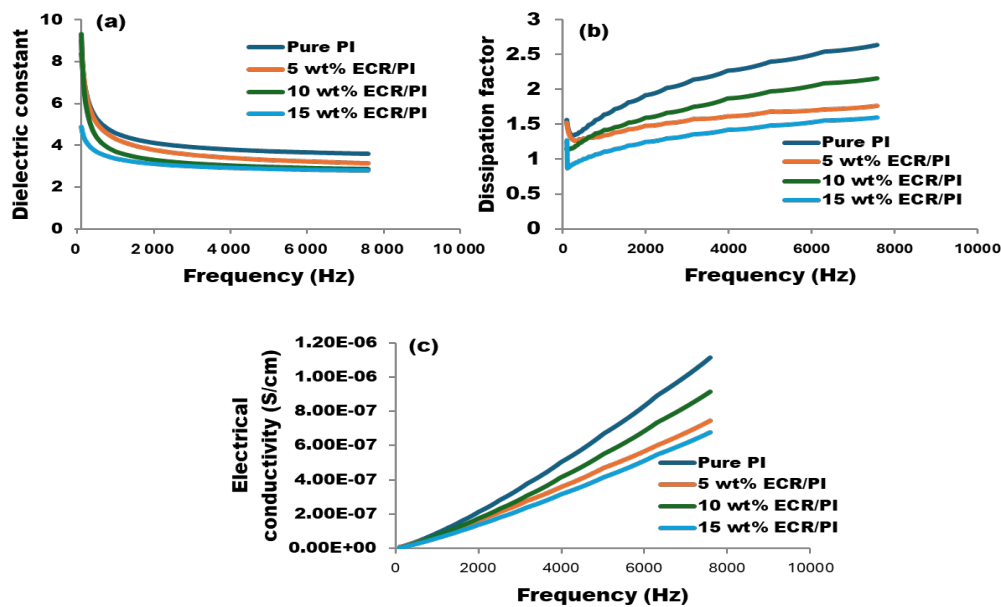


Figure 4. Electrical/dielectric behavior of spark plasma sintered ECR/PI composites (Ogbonna et al., 2024)

In addition, Ogbonna et al. (2023) investigated the improvement of mechanical and electrical characteristics of ECR-glass/PI composites with the addition of varying TiO₂ nanoparticles (0, 2, 4, and 6 wt%) for insulation applications. The resultant nanocomposites were produced at sintering temperature of 320 °C, pressure of 30 MPa, 5 °C/min heating rate, and 9 min dwell time. Characterizing the morphological evolution of the resultant nanocomposites using SEM, even dispersion of the TiO₂ nanoparticles in the ECR/PI composites was reported. Optimal hardness and Young's modulus values of approximately 2.20 GPa and 14.1 GPa, respectively, was recorded in the ECR/PI composite containing 6 wt% TiO₂ nanoparticles (Figure 5). ECR acts against plastic deformation, in turn improving the composite mechanical properties. Further, ECR-glass/PI composites filled with 6 wt% TiO₂ possessed the lowest dielectric constant of 1.18 and electrical conductivity of 3.16 x 10⁻⁶ S.cm⁻¹. And this could be ascribed to PI molecular

chain impediment (Dong et al., 2022). The findings demonstrate the facile processability of three-component PI nanocomposites and their promise for mechanical and electrical insulation applications.

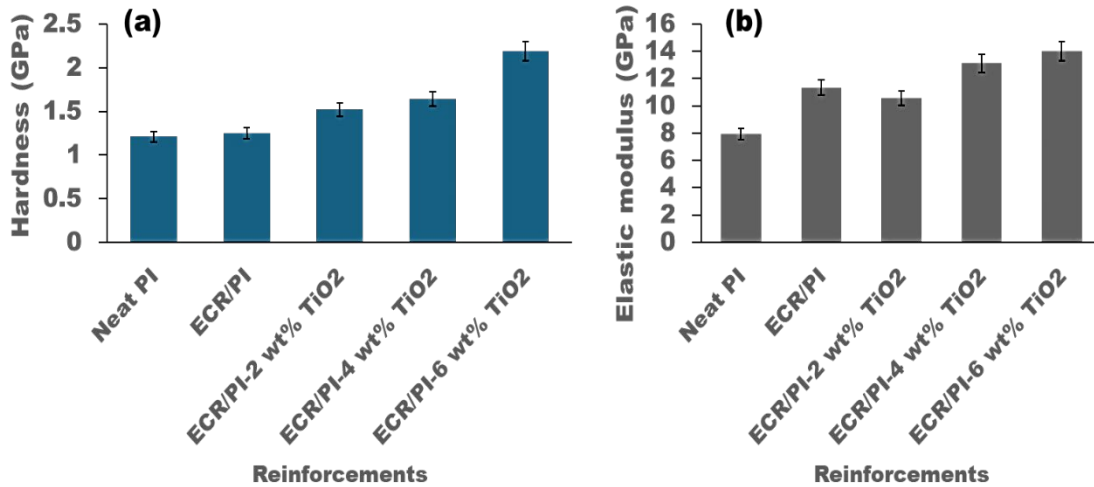


Figure 5. Mechanical response of the SPS sintered ECR/PI-composites (Ogbonna et al., 2023)

Additionally, Ogbonna et al. (2023) reported on the effects of ECR-glass fiber on the corrosion and mechanical behaviour of PI matrix composites produced employing SPS method. The samples morphology, corrosion, and mechanical characteristics were evaluated using scanning electron microscopy, immersion, and polarization tests, and nanoindentation tests, respectively. The result demonstrates even distribution of the ECR fiber particulates into PI matrix structural network. Also, the resulting analysis illustrated that the incorporation of the ECR particulates concentrations (5, 10, and 15 wt%) into the PI enhanced its hardness and elastic modulus both before and after exposure of the samples in a corrosive medium (HCl). Herein, the corrosion rate of the pristine PI reportedly reduced by 61.7%, 50.3%, and 39.0%, respectively, at 5, 10, and 15 wt% ECR-glass fiber additions. This evidenced that the anti-corrosion behaviour of PI could be improved with ECR particles incorporation. And this corroborates the potential of ECR filler materials in the development of novel PI-based composites for corrosion resistance and structural applications (Wang et al., 2024).

In another work, friction and wear characteristics of PI-based nanocomposites containing single wall carbon nano-horn particles fabricated by SPS route (Figure 6) were presented by Tanaka et al. (2005). In the study, carbon nanotube and graphite reinforced PI-based composites were also produced for comparison. From the experimental results, the carbon nano-horn markedly reduced the wear of the PI composites. The specific wear rate of the PI-based containing only 5 wt% carbon nano-horn was of the order of 10^{-8} mm³/Nm that was two orders of magnitude lower than those of the pure PI. Meanwhile, the PI-based composites with carbon nanotube depict to be slightly inferior to the carbon nano-horn/PI composite, though was greatly better than that of the graphite particles reinforced PI-based composites. Therein, better transferred films in the PI-based composites filled carbon nano-horn ascribed to their low friction and wear characteristics reported in the study.

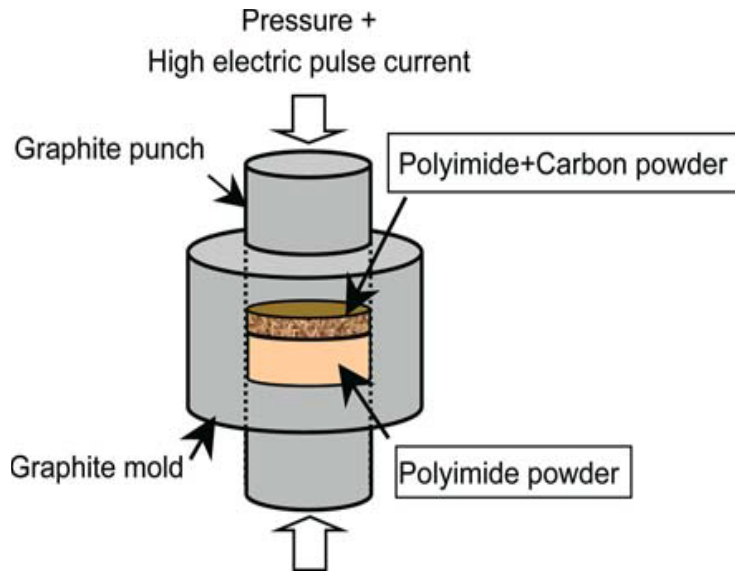


Figure 6. Major part of SPS process (Tanaka et al., 2005)

2.2 Potential applications of polyimide micro and nanocomposites

In the discussion above, processing of PI and PI-based composites with SPS technique for industrial and engineering applications depict some prospect, knowing that polymers and polymer-based materials are used in the design and development of material components for automobile, aerospace, microelectronics, and energy storage. PI-based materials do display sustainable characteristics even at low quantity of filler particles (metal oxide, carbide, and nitride) for multipurpose application. Besides, it was noticed that introducing reinforcement glass fiber powder, titanium dioxide powder, aluminium powder, and carbon-based fillers into the PI network using SPS results in improving the performance of the composites (Ogbonna et al., 2024; Schwertz et al., 2015). Improved PI-based composites property, such as hardness, yield strength, elastic modulus, breakdown strength, thermal stability, anti-wear, and chemical stability are worthy for aircraft, automotive, electronics, composite insulators, and high-temperature dielectric capacitors (Fazil et al., 2018). Furthermore, because of the lightweight, mechanical strength to weight ratio, and stability of behaviour of PI-based material, reinforced PI composites have reportedly been utilized for structural, tribological, and biomedical applications (Schwertz et al., 2015; Chen et al., 2017). More so, spark plasma sintered PI-based materials as selected functionally graded material have become newest research interest among researchers and industries for advanced engineering applications. However, there are some issues associated with the fabrication of PI-based materials employing SPS method. As such, in this review, such challenges were presented with recommendations for better advancements.

3. Challenges and Recommendation

Herein, the application of SPS technology in the fabrication and characterization of PI-based composites were reviewed. With the available literature, PI-based composites processed using SPS fabrication method happened to possess good mechanical, electrical, thermal stability, wear resistance, and corrosion resistance properties. The possibility of sintering powder particles to near full densification and better diffusion of particles with little grain growth using SPS sintering ascribed to the noted improved characteristics of PI and PI-based composites (Adesina et al., 2021; Ogbonna et al., 2022). Observation from the reported data, the consolidation optimization in the SPS sintering enables the production of PI-based high-density composites with uniform microstructure and enhanced physical and mechanical characteristics (Schwertz et al., 2015; Ogbonna et al., 2022). Though, low cohesion between the reinforcement fillers and PI matrix remains one of the challenges facing the use of SPS in producing PI-based composites. However, to address the low cohesion in SPS sintered PI-based micro/nanocomposites, the authors recommend surface functionalization and modification of the fillers using coupling agent like saline. Knowing that coupling agent is used to remove moisture and improve the distribution of fillers in polymer matrices and dimensional stability of the resultant composite (Taib and Julkapli, 2019). Although, proper care should be put into consideration with the type of saline to apply as SPS is powder metallurgy technique involving solid state fabrication and consolidation of powder materials (Alojaly et al., 2023). Optimization in dispersion parameters, such as speed and

time using 3D turbula dispersion and other advanced dispersion methods should also be incorporated in the design and fabrication of PI-based composites employing SPS sintering. As to gain proper understanding and exploring of SPS technologies route in solving many material production challenges.

4. Conclusions

The processing of PI-based composites using the SPS sintering method was reviewed. The review demonstrated the practicability of involving the novel SPS method in fabricating PI and its micro/nanocomposites. From the review, SPS sintered PI-based composites remain a promising material in the composition and development of components for mechanical load bearing and mechanical friction, as well as insulation applications. Owing to the mechanical, wear, electrical, thermal stability, and corrosion behaviour of the PI-based composites reported in the study. Thus, SPS technique could be used in mitigating issues of PI and PI-based composites degradation arising from fabricating polyimide-based material at high-temperatures due to the practicability of SPS sintered polymers at low sintering temperature when compared to the conventional method such as melt processing method. However, as SPS PI-based materials are generally characterized with high mechanical hardness and elastic modulus, challenges associated with PI-based composites using SPS sintering were also presented. Thus, more suitable optimization of the SPS and dispersion parameters should be considered in achieving more superior properties of the spark plasma sintered PI and PI-based composites.

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Biographies

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OM Popoola is an Engineer/technologist/lecturer/author with diverse experience in the Electrical and Power Industry, Oil and Gas Sector, Shipping Building Industry, Education and Quality Management field. A Certified Measurement and Verification Professional (CMVP) with a proven record and extensive knowledge of the energy industry. An author & co-author of numerous scientific publications, as well as a reviewer for different journals. His research interest includes Energy Management, Energy and Behaviour, Renewable and Sustainable Energy, Quality Management, Power electronics application in Power Systems, New Materials as well as Laser Applications. He has made several presentations both locally and internationally. Prof Popoola has received awards, support funding and accolades for his knack and drives for research interest that contributes to sustainable energy/society, especially with a multidisciplinary approach. Furthermore, innovative product development in new materials and load management with emphasis on proficient operation/utilization, effective management, affordability, cost savings, etc. are some high points in his career.