Investigation of the Influence of Al₂O₃ Particles on the Microhardness and Tensile Strength of PA6 Composites

Song Jeng-Huang¹, Angelo Geo¹, Sathiyalingam Kannaiyan^{1,*} and Yopi Yusuf Tanoto^{1,2,*}

¹Department of Mechanical Engineering, National Taiwan University of Science and Technology, Taipei 10607, Taiwan

²Department of Mechanical Engineering, Petra Christian University, Surabaya 60236, Indonesia

Abstract: Polyamide is a high-performance synthetic plastic known for its strength, durability, flexibility, chemical resistance, and low cost, making it widely used in engineering, automotive, and electrical. However, the surface and mechanical properties can be further enhanced to meet the growing demands of advanced engineering applications. This study aims to investigate the influence of Al_2O_3 particles on the hardness of polyamide 6 (PA6). The Al_2O_3 was mixed with PA6 at weight percentages (wt.%) of 0.3% and 1.5% then were fabricated into composite plates using compression molding and subsequently. As a result, the composites achieved higher microhardness and tensile strength compared to the matrix with increases of 13.3% and 7.3% achieved by incorporating 0.3 wt.% of reinforcement, respectively. This result suggests that Al_2O_3 has the potential to improve the surface properties and mechanical strength of the matrix material.

Keywords: Al_2O_3 , compression molding, microhardness, polyamide 6, tensile strength.

1. INTRODUCTION

Polyamide 6 (PA6), commonly known as nylon 6, is a widely used thermoplastic polymer known for its excellent mechanical properties, thermal stability, and resistance to chemicals and rust. PA6 is a semicrystalline thermoplastic polymer widely utilized in engineering applications [1, 2]. It is highly regarded for its excellent processability, strong tensile properties, and outstanding resistance to abrasion and chemical agents [3]. These properties make it a material of choice for various industrial applications, including automotive components, electrical and electronic devices, and consumer goods [4]. However, the inherent mechanical properties of PA6 can be further enhanced to meet the growing demands for highperformance materials in advanced engineering applications [5].

One effective strategy to improve the mechanical properties of polymers is the incorporation of particulate reinforcement. PA6 properties can be improved by incorporating reinforcement. Bragraglia et al. utilized boron nitride (BN) as a reinforcement. The addition of 45 wt.% BN to PA6 results in a 153% increase in elastic modulus. However, when compared to pure PA6, both the strain at rupture and ultimate tensile strength (UTS) decrease by as much as 50.7% [6]. The most recent research aimed at enhancing the PA6 matrix with microparticles was conducted by Tanoto and Huang, who utilized AZ61 particles as reinforcement within the PA6 matrix. The maximum values achieved for tensile strength and hardness

were 58 MPa and 21.1 HV, respectively. These peak values were attained by incorporating 3 wt.% and 5 wt.% of AZ61 into the matrix [7].

Among the various particles studied, alumina (Al₂O₃) stands out due to its exceptional hardness, high thermal conductivity, and stability. Al₂O₃ particles, being a type of metal oxide, offer excellent properties that can greatly enhance and improve various characteristics of polymer composites, making them a promising option for polymer reinforcement [8]. The tribological analysis, studied by Ain et al. shows that increasing Al₂O₃ content reduces the friction coefficient by enhancing load distribution across the interface of the Polydimethylsiloxane (PDMS) matrix [9]. Coban et al. report that the high modulus of Al₂O₃ and its strong interaction with the Polyphenylene sulfide (PPS) matrix enhance dynamic mechanical properties [10]. Rawat et al. reported that alumina micro powder effectively increased the hardness of glass fiber by as much as 10% [11]. However, no one paper discussed Al2O3 as a reinforcement for PA6.

The literature review indicates that the incorporation of polymer and Al_2O_3 presents significant potential for mixing as a polymer matrix composite (PMC). The Al_2O_3 /PA6 composite is promising as a lightweight material with excellent surface hardness and mechanical properties. It is important to note that this study specifically focuses on the impact of Al_2O_3 as a reinforcement material to enhance the properties of the PA6 matrix.

2. EXPERIMENTAL

2.1. Material

In this study, the same PA6 powder from the previous study was utilized as the matrix material [7].

^{*}Address correspondence to these authors at the Department of Mechanical Engineering, National Taiwan University of Science and Technology, Taipei 10607, Taiwan; E-mail: sathiaerospace@gmail.com

Department of Mechanical Engineering, Petra Christian University, Surabaya 60236, Indonesia; E-mail: yopi.tanoto@petra.ac.id

For reinforcement, Al_2O_3 micro powder with an average particle size of 7.5 µm (D_{10} = 0.24 µm, and D_{90} = 17.5 µm) was employed. Al_2O_3 was incorporated into the matrix at various weight percentages, specifically 0.3 wt.%, and 1.5 wt.%. These two values were selected based on the results of preliminary experiments. With a composition difference reaching fivefold, the effect of Al2O3 on the material's performance was clearly observed.

2.2. Composite Preparation

The PA6 and Al_2O_3 powders were mixed in varying weight percentages of 0.3 wt.% and 1.5 wt.% Al_2O_3 using Retsch PM100 planetary ball milling apparatus running at 300 rpm. The ball-to-powder ratio (BPR) was maintained at 1:1 to ensure uniform mixing of the powders. After ball milling, the powder mixtures were processed into composite plates through compression molding [9, 10].

A square mold with dimensions of 11 cm \times 11 cm and a thickness of 1 mm was used to form the plates. Approximately 13 grams of the powder mixture was used for each composite. Various compression molding parameters were tested, including temperature, pressure, and compression time. The temperatures tested were 210°C, 215°C, and 220°C (the melting temperature of PA6), with a constant pressure of 7 MPa. Compression times of 5 minutes and 7 minutes were evaluated to determine the optimal conditions.

Before full compression, a preheating stage was implemented to minimize the presence of moisture in the powder mixture and improve material flow [14]. Here the temperature was gradually increased from 190°C to the target temperature of 215°C. This preheating process ensured proper fusion of the materials before full compression. After testing several samples, the optimal parameters were identified as 215°C for 7 minutes of compression. Following the main compression molding stage, a cooling down stage was employed where the plates were allowed to cool down from 215°C to 180°C under the same pressure of 7 MPa. The pressure was later released and the mold was taken out after allowing it to cool down to room temperature.

2.3. Characterization

The morphology of PA6 and AI_2O_3 powder was investigated using a field emission scanning electron microscope (FE-SEM) model JSM-7900 from Jeol Ltd., Japan. This same instrument was also utilized to examine the elemental mapping of the composite powder. Surface properties were observed using a microhardness tester, Wilson HV1102, with an applied pressure of HV 0.1 kgf for a 10-second indentation period. The tensile test was conducted in accordance with ASTM D638 standards using Type V specimens, with a specimen thickness of 1 mm. The tests were performed on an MTS Insight Electromechanical Standard Length (SL) machine, equipped with a 10 kN load capacity.

3. RESULT AND DISCUSSION

3.1. Material Characterization

As seen in Figure 1, the morphology of PA6 particles is characterized by irregular shapes with sharp, distinct edges rather than spherical forms [15]. Similarly, Al_2O_3 particles also exhibit irregular, non-uniform structures.

The morphology and elemental distribution of the composite powder after mixing were examined using SEM imaging, as presented in Figure **2**. The SEM image in Figure **2a** illustrates the composite powder containing 0.3 wt.% reinforcement at a magnification of 750x. PA6 was identified by the presence of oxygen (O) (Figure **2c**) and nitrogen (N) (Figure **2d**) elements, while Al_2O_3 was primarily composed of aluminum (Al) (Figure **2b**) and oxygen (O). As seen in Figure **2b**, the



Figure 1: SEM images of (a) PA6 and (b) Al₂O₃ as received.



Figure 2: SEM images of 0.3 wt.% of $Al_2O_3/PA6$ (a) 750 magnifications. EDS mapping of (b) aluminum distribution, (c) oxygen distribution, and (d) nitrogen distribution.

Al₂O₃ particles are well integrated into the Al₂O₃/PA6 powder blend, with aluminum being uniformly dispersed across the PA6 particles, indicating successful reinforcement mixing within the polymer matrix.

The SEM image in Figure 3a illustrates the composite powder containing 1.5 wt.%. Figure 3b shows the aluminum element, meanwhile, oxygen and nitrogen elements are shown in Figure 3c and 3d respectively. The SEM and EDS images demonstrate that at higher concentrations of Al_2O_3 , the particles tend to agglomerate. The biggest cluster of Al₂O₃ particles is highlighted by the blue arrow pointing to regions of concentrated aluminum in the SEM images. The agglomerated Al₂O₃ is further confirmed by the EDS analysis, where the green and red colors, representing the aluminum (AI) and oxygen (O) elements, exhibit a higher intensity in those specific areas. This suggests that the Al₂O₃ particles cluster in certain regions, leading to localized accumulations of the reinforcement within the PA6 matrix. A higher concentration of powder reinforcement often leads to increased agglomeration. This phenomenon occurs because as more particles are added to the polymer matrix, the likelihood of particle-to-particle contact rises, which can result in clustering and the formation of agglomerates [16].

3.2. Surface Properties

The microhardness values for pure PA6 and Al₂O₃/PA6 composites at various weight percentages are illustrated in Figure **4**. The composite materials demonstrated higher microhardness compared to pure PA6. The microhardness of pure PA6 was measured at 10.18 (\pm 0.3) HV, which increased substantially to 11.54 (\pm 0.6) HV with the incorporation of 0.3 wt.% Al₂O₃. However, as the Al₂O₃ content increased further, the hardness slightly decreased to 11.36 (\pm 0.9) HV. Despite this slight decrease, the similar standard deviations indicate that the microhardness of both composites remains statistically comparable. The addition of 0.3 wt.% and 1.5 wt.% of Al₂O₃ resulted in hardness improvements of 13.3% and 11.6%, respectively.

 Al_2O_3 is a ceramic material with a much higher hardness compared to PA6. When these hard particles are embedded in the surface layer of the PA6 matrix, they increase the material's resistance to localized deformation. This leads to a higher microhardness



Figure 3: SEM images of 1.5 wt.% of $Al_2O_3/PA6$ (a) 750 magnifications. EDS mapping of (b) aluminum distribution, (c) oxygen distribution, and (d) nitrogen distribution.



Figure 4: Microhardness Vickers value for pristine PA6 and the Al₂O₃/PA6 composite.

value because the harder Al_2O_3 particles reduce the indentation depth during testing. The slight reduction in hardness at higher levels of Al_2O_3 reinforcement can be attributed to the uneven distribution of Al_2O_3 particles within the matrix. This uneven dispersion likely leads to localized agglomeration, which weakens the overall structural integrity [15, 16]. The increased

variability in the hardness measurements, as evidenced by the larger standard deviation of ± 0.9 compared to ± 0.6 at lower reinforcement levels, further supports the observation that poor particle distribution contributes to inconsistent mechanical performance across the composite.



Figure 5: Ultimate tensile stress value for pristine PA6 and the Al₂O₃/PA6 composite.

3.3. Mechanical Properties

The ultimate tensile strength, as presented in Figure 5, was evaluated using eight tensile specimens. The base material exhibited a strength of 52.91 ± 3.48 MPa. Upon adding a small quantity of reinforcement, the strength increased to 56.77 ± 2.91 MPa. However, with the introduction of 1.5 wt.% reinforcement into the matrix, a slight decrease was observed, with the tensile strength reducing to 53.98 ± 1.04 MPa. This behavior is consistent with the hardness measurements, where the composite with a low level of Al₂O₃ exhibited the highest performance, and overall, the composite outperformed the pure material. The strength improvement was 7.3% and 2%, adding 0.3 wt.% and 1.5 wt.% of Al₂O₃, respectively. Nevertheless, agglomeration phenomena also impact the tensile test, like the microhardness. These results further prove that 0.3 wt.% is the optimal or the limit value for the Al₂O₃.

The strength of a composite is greatly affected by the bonding strength at the interface between the fillers and the matrix materials [19]. Tensile test results show that Al_2O_3 particles are distributed evenly within the matrix when proper mixing procedures are applied before compression molding. The combination of heat and compression promotes strong interfacial bonding between the components. The uniform distribution of fine Al_2O_3 particles within the matrix allows the composite to better withstand stress by dispersing the stress more evenly and minimizing areas of highstress concentration.

4. CONCLUSION

In this study, the composite material of $AI_2O_3/PA6$ was successfully produced using compression

molding. The composite containing 0.3 wt.% $AI_2O_3/PA6$ showed a significant improvement in microhardness, increasing by 13.3% from 10.18 HV to 11.54 HV. In terms of tensile strength, the maximum enhancement of 7.3% was achieved by incorporating 0.3 wt.% of reinforcement, increasing the strength from 52.91 MPa to 56.77 MPa. With a higher amount of reinforcement, the improvements in hardness and strength were not optimized due to the agglomeration of AI_2O_3 . In summary, the addition of AI_2O_3 particles significantly improved the PA6 performance.

DECLARATION OF INTEREST STATEMENT

The authors declare that there are no conflicts of interest regarding this study.

ACKNOWLEDGEMENT

The authors would like to express their gratitude to the National Science and Technology Council of Taiwan for providing financial support through the International Internship Pilot Program (IIPPP) in Taiwan.

REFERENCES

- [1] Lan P, Nunez EE, Polycarpou AA. Advanced Polymeric Coatings and Their Applications: Green Tribology. Encyclopedia of Renewable and Sustainable Materials, Elsevier 2020; pp. 345-58. <u>https://doi.org/10.1016/B978-0-12-803581-8.11466-3</u>
- [2] Patil A, Patel A, Purohit R. An overview of Polymeric Materials for Automotive Applications. Materials Today: Proceedings 2017; 4: 3807-15. <u>https://doi.org/10.1016/j.matpr.2017.02.278</u>
- [3] Pradeepa KG, Shashidhara GM. Comparative Study on Experimental and Kerner Model Predictions of Viscoelastic Properties of Polyamide 6/ Polyvinyl Alcohol Blends. J Res Updates Polym Sci 2018; 7: 14-20. <u>https://doi.org/10.6000/1929-5995.2018.07.01.3</u>

- [4] Hussein AH, Dong Z, Lynch-Branzoi J, Kear BH, Shan JW, Pelegri AA, et al. Graphene-reinforced polymer matrix composites fabricated by in situ shear exfoliation of graphite in polymer solution: processing, rheology, microstructure, and properties. Nanotechnology 2021; 32: 175703. <u>https://doi.org/10.1088/1361-6528/abd359</u>
- [5] Anand T, Senthilvelan T. Investigation of the Mechanical Properties of Polyamide 6 Hybrid Nanocomposites with MWCNT and Copper Nanoparticles. In: Vijayan S, Subramanian N, Sankaranarayanasamy K, editors. Trends in Manufacturing and Engineering Management, Singapore: Springer Singapore; 2021, p. 261-72. <u>https://doi.org/10.1007/978-981-15-4745-4_24</u>
- [6] Bragaglia M, Lamastra FR, Russo P, Vitiello L, Rinaldi M, Fabbrocino F, et al. A comparison of thermally conductive polyamide 6-boron nitride composites produced via additive layer manufacturing and compression molding. Polymer Composites 2021; 42: 2751-65. https://doi.org/10.1002/pc.26010
- [7] Tanoto YY, Huang S-J. The Effect of AZ61 Content on Mechanical Strength and Surface Hardness of PA6-AZ61 Magnesium Alloy. J Res Updates Polym Sci 2023; 12: 180-5. <u>https://doi.org/10.6000/1929-5995.2023.12.15</u>

Nobben A. Tehe M. Chazely NM Filler lead

- [8] Nabhan A, Taha M, Ghazaly NM. Filler loading effect of Al₂O₃/TiO₂ nanoparticles on physical and mechanical characteristics of dental base composite (PMMA). Polymer Testing 2023; 117: 107848. <u>https://doi.org/10.1016/j.polymertesting.2022.107848</u>
- [9] Ain QU, Wani MF, Sehgal R, Singh MK. Role of Al₂O₃ reinforcements in polymer-based nanocomposites for enhanced nanomechanical properties: Time-dependent modeling of creep and stress relaxation. Ceramics International 2024; 50: 33817-38. <u>https://doi.org/10.1016/j.ceramint.2024.06.201</u>
- [10] Çoban O, Bora MÖ, Sinmazçelik T. Effect of mixed size particles reinforcing on the thermal and dynamic mechanical properties of Al₂O₃/PPS composites. Polymer Composites 2016; 37: 3219-27. https://doi.org/10.1002/pc.23520
- [11] Rawat MK, Kukreja N, Gupta SK. Effect of reinforcing micro sized aluminium oxide particles on mechanical properties of

Received on 12-09-2024

https://doi.org/10.6000/1929-5995.2024.13.22

© 2024 Jeng-Huang et al.

This is an open-access article licensed under the terms of the Creative Commons Attribution License (<u>http://creativecommons.org/licenses/by/4.0/</u>), which permits unrestricted use, distribution, and reproduction in any medium, provided the work is properly cited.

polymer based composite. Materials Today: Proceedings 2020; 26: 1306-9. https://doi.org/10.1016/i.matpr.2020.02.260

- [12] Emaldi I, Liauw CM, Potgieter H. Stabilization of Polypropylene for Rotational Molding Applications. J Res Updates Polym Sci 2016; 4: 179-87. https://doi.org/10.6000/1929-5995.2015.04.04.2
- [13] He H, Li K. Study on Flexural Strength and Flexural Failure Modes of Carbon Fiber/Epoxy Resin Composites. J Res Updates Polym Sci 2014; 3: 10-5. <u>https://doi.org/10.6000/1929-5995.2014.03.01.2</u>
- [14] Ying Q, Jia Z, Wang X, Liu L, Li J, Rong D. Effect of molding process parameters on the mechanical properties of CGFRPP products. J Mech Sci Technol 2024; 38: 2949-59. <u>https://doi.org/10.1007/s12206-024-0515-0</u>
- [15] Verbelen L, Dadbakhsh S, Van Den Eynde M, Kruth J-P, Goderis B, Van Puyvelde P. Characterization of polyamide powders for determination of laser sintering processability. European Polymer Journal 2016; 75: 163-74. https://doi.org/10.1016/j.eurpolymj.2015.12.014
- [16] Huang S-J, Adityawardhana Y, Kannaiyan S. Enhancement strength of AZ91 magnesium alloy composites reinforced with graphene by T6 heat treatment and equal channel angular pressing. Arch Civ Mech Eng 2024; 24: 235. https://doi.org/10.1007/s43452-024-01048-8
- [17] Chen J, Yu Y, Chen J, Li H, Ji J, Liu D. Chemical modification of palygorskite with maleic anhydride modified polypropylene: Mechanical properties, morphology, and crystal structure of palygorskite/polypropylene nanocomposites. Applied Clay Science 2015; 115: 230-7. https://doi.org/10.1016/j.clay.2015.07.012
- [18] Khan A, Shamsi MH, Choi T-S. Correlating dynamical mechanical properties with temperature and clay composition of polymer-clay nanocomposites. Computational Materials Science 2009; 45: 257-65. <u>https://doi.org/10.1016/j.commatsci.2008.09.027</u>
- [19] Wang K, Wu J, Ye L, Zeng H. Mechanical properties and toughening mechanisms of polypropylene/barium sulfate composites. Composites Part A: Applied Science and Manufacturing 2003; 34: 1199-205. <u>https://doi.org/10.1016/j.compositesa.2003.07.004</u>

Accepted on 04-10-2024

Published on 28-10-2024