

# Effect of Sol–Gel Derived SiO<sub>2</sub> Nanoparticle Dispersion on the Mechanical Properties of PMMA Composites

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**Keywords:** Sol–gel process; PMMA composites; SiO<sub>2</sub> nanoparticles; Mechanical properties

## ABSTRACT

Titanium alloys are commonly used in artificial joint implants due to their excellent mechanical and physical properties; however, long-term implantation can lead to the release of Al and V ions, with vanadium posing toxicity concerns. Titanium alloys also exhibit poor tribological performance, including a high friction coefficient, low wear resistance, and susceptibility to corrosion in body fluids, which can accelerate surface degradation and ultimately contribute to implant failure.

In this study, silica (SiO<sub>2</sub>) particles were incorporated into poly(methyl methacrylate) (PMMA) to improve the mechanical properties of the polymer matrix. PMMA/SiO<sub>2</sub> composites were synthesized using the sol–gel method to minimize particle agglomeration and to clarify the effects of particle characteristics on material performance. Silica addition enhanced the elastic modulus and tensile strength, while strain behavior depended on particle size, dispersion, and particle density. Particle dispersion was quantified using the D0.2 parameter derived from Voronoi diagram analysis. Higher D0.2 values indicated improved dispersion and corresponded to enhanced mechanical performance.

## INTRODUCTION

In the field of artificial joint materials, surface modification technologies are widely employed to enhance the corrosion resistance, wear resistance, and antibacterial performance of implants (Budzynski et al.,

2006). Such techniques, including surface texturing, surface grafting, and surface coatings, are often applied to improve the biological, chemical, and mechanical properties of implant surfaces and to reduce friction and wear during articulation within the body (Ghosh & Abanteriba, 2016; Yeh et al., 2006).

Poly(methyl methacrylate) (PMMA), commonly known as acrylic, is a polymer synthesized from methyl methacrylate monomers. PMMA exhibits high optical transparency, excellent UV resistance, good weatherability, low density, and favorable thermomechanical properties. Its rigidity and thermal stability allow it to serve as a substitute for ceramic materials in various applications (Deka et al., 2022; Sansul et al., 2023; Yuan et al., 2022). Owing to its excellent biocompatibility, PMMA is widely used in biomedical fields and is one of the most common matrices in polymer composites. Its high strength, low water absorption, solvent resistance, and material compatibility also make it a promising candidate for medical coating applications (Burcea et al., 2024; Ramanathan et al., 2024).

PMMA-based coatings not only require sufficient corrosion resistance to prevent the release of harmful substances from artificial joints but must also function as an effective buffer layer between the implant and the surrounding bone (Chen et al., 2025; Ramanathan et al., 2024). Such a layer helps reduce stress concentration, thereby lowering the risk of corrosion and component loosening. Because artificial joints are subjected to long-term compressive, tensile, and torsional loading, the coating material must possess adequate mechanical strength. Simulating the mechanical conditions within the human body is therefore crucial for evaluating long-term performance (Pahlevanzadeh et al., 2018). Previous studies have used simplified muscle models and optimization approaches combined with gait analysis data to estimate hip joint contact forces.

To enhance the mechanical performance of PMMA, micro or nanoscale particles are commonly incorporated as fillers. These particles can improve the strength, hardness, and stiffness of the composite. At the nanoscale, the high surface-to-volume ratio

*Paper Received November, 2025. Revised January, 2026. Accepted January, 2026. Author for Correspondence: Shih-Chen Shi.*

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increases interfacial area and decreases defect density, further contributing to mechanical reinforcement (Ahmed & Salih, 2021; Ali Sabri et al., 2021). However, the degree of reinforcement does not increase indefinitely with higher filler loading; instead, an optimal loading exists. Excessive addition can lead to particle agglomeration due to their high surface energy, reducing effective interaction with the polymer matrix and ultimately degrading performance. These nanoparticle agglomerates have weak cohesive strength and often act as defects. Therefore, achieving effective dispersion is essential for maximizing the benefits of particle reinforcement. Well-dispersed particles increase the effective interaction volume with the matrix and subsequently result in superior mechanical enhancement (Wang et al., 2023).

The incorporation of particulate fillers is known to influence the mechanical properties of composite materials (García et al., 2025; Kuan et al., 2021; Siraj et al., 2022). In this study, we hypothesize that the addition of particles into the PMMA matrix can impede crack propagation and thereby enhance the overall mechanical performance of the composite (Fig. 1).

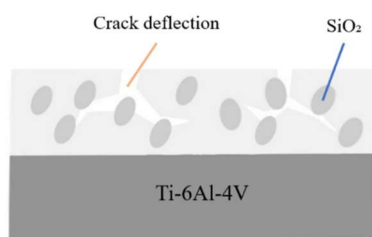


Fig. 1 Mechanism of crack propagation retardation by particle fillers.

## MATERIALS PREPARATION AND EXPERIMENTAL PROCEDURE

### Preparation of PMMA/SiO<sub>2</sub> Composites

This study used a PMMA/SiO<sub>2</sub> composite system consisting of organic and inorganic components (Jiang, 2025). The organic phase contained poly(methyl methacrylate) (PMMA, Sigma-Aldrich) as the matrix, tetrahydrofuran (THF, Reagents Duksan) as the solvent, benzoyl peroxide (BPO, Alfa Aesar) as the initiator, and 3-(trimethoxysilyl)propyl methacrylate (MPTS, Sigma-Aldrich) as the coupling agent to improve adhesion between the polymer and silica. The inorganic phase was derived from tetraethyl orthosilicate (TEOS, Thermo Scientific). Deionized water and ethanol (SHIN-SHIN CHEMICAL CO.) were used to support hydrolysis and condensation reactions, and aqueous ammonia (NH<sub>3</sub>, PanReac AppliChem) served as the catalyst. All chemicals were used without further purification to ensure reproducibility.

Silica nanoparticles were synthesized through a sol-gel process using TEOS as the precursor. Ethanol, deionized water, and aqueous ammonia acted as the solvent system, hydrolysis medium, and catalyst. A Taguchi experimental design was applied to improve nanoparticle dispersion. Four factors were selected, including TEOS concentration, ammonia concentration, reaction temperature, and reaction time. Each factor was tested at three levels, and an L<sub>9</sub> orthogonal array was used to establish nine experimental conditions. The design matrix is summarized in Table 1, and the experimental runs are listed in Table 2. This approach allowed identification of synthesis parameters that produce well-dispersed silica nanoparticles suitable for incorporation into the PMMA matrix (Azlina et al., 2016; Dixit et al., 2016; Kim et al., 2017).

Table 1. Control factors and levels

| Factor | 1          | 2                       | 3         | 4          |
|--------|------------|-------------------------|-----------|------------|
| Level  | TEOS (wt%) | NH <sub>3(aq)</sub> (M) | Temp (°C) | Time (min) |
| 1      | 5          | 0.01                    | 20        | 90         |
| 2      | 7          | 0.05                    | 40        | 180        |
| 3      | 10         | 0.1                     | 60        | 270        |

Table 2. L<sub>9</sub> Orthogonal Array

| Sample                      | Code Name   |
|-----------------------------|-------------|
| 5 wt%+0.01 M+20°C +90 min   | P5N012090   |
| 5 wt%+0.05 M+40°C +180 min  | P5N0540180  |
| 5 wt%+0.1 M+60°C +270 min   | P5N160270   |
| 7 wt%+0.01 M+40°C +270 min  | P7N0140270  |
| 7 wt%+0.05 M+60°C +90 min   | P7N056090   |
| 7 wt%+0.1 M+20°C +180 min   | P7N120180   |
| 10 wt%+0.01 M+60°C +180 min | P10N0160180 |
| 10 wt%+0.05 M+20°C +270 min | P10N0520270 |
| 10 wt%+0.1 M+40°C +90 min   | P10N14090   |

### Statistical Analysis of Particle Dispersion in Composite Materials

SEM observations were conducted using a field-emission scanning electron microscope (AURIGA, Carl Zeiss, Oberkochen, Germany). SEM is widely used to evaluate the dispersion of particulate fillers in composite materials, and recent advancements combining SEM imaging with computer-vision-based image analysis enable quantitative assessment of particle size, spatial distribution, and dispersion uniformity.

Among these methods, the Voronoi diagram has been extensively used to quantify the spatial dispersion of nanoscale fillers within polymer composites (Eckel et al., 2004; Luo & Koo, 2007). In this approach, each particle captured in the SEM micrograph is treated as a point on a plane. By connecting neighboring points and constructing the corresponding perpendicular bisectors, a series of

polygonal cells is generated, forming a complete Voronoi tessellation, as illustrated in

Fig. 2. This tessellation effectively partitions the composite microstructure into regions associated with each particle, enabling statistical analysis of the localized dispersion environment (Peng et al., 2014).

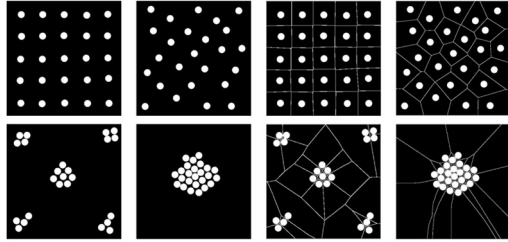


Fig. 2 Particle distribution maps and corresponding Voronoi diagrams at different dispersion levels are shown.

The dispersion parameter  $D_k$  is defined from the distribution of Voronoi cell areas (Tsuchikusa et al., 2023). A histogram of these areas is generated and fitted with a normal distribution. The dispersion level is quantified as the probability that the cell areas fall within  $\mu \pm k\%$ , where  $\mu$  is the mean area. A higher  $D_k$  value indicates that more particles are clustered near the mean, reflecting a more uniform dispersion. The calculation of  $D_k$  is given as follows:

$$D_k = \int_{\mu(1-k)}^{\mu(1+k)} f(a) da \quad (1)$$

### Hardness Test

The hardness of the PMMA/SiO<sub>2</sub> specimens was evaluated using a micro-Vickers hardness tester (SHIMADZU, Japan) equipped with a pyramidal indenter. A load of 48.9 mN was applied with a dwell time of 10 s for each measurement.

### Wear Test

The wear performance of the composite materials was evaluated using a rotary abrasion tester (FREEFORM P.M. Co., Ltd., Taiwan). The materials were fabricated into circular specimens with a diameter of 20 mm, and a zirconia counter ball with a diameter of 2.9 mm was used as the antagonist. Following the findings of the literature review, a simulated body fluid was prepared according to the formulation reported in (Smith et al., 2014), ensuring that the testing environment exhibited comparable mechanical characteristics. The applied normal load

(3.7 N) and total sliding distance (150 m) were selected based on the testing parameters reported in (Bergmann et al., 2001; Wang et al., 2017). Upon completion of the abrasion tests, the worn surfaces were examined using a 3D laser scanning microscope, and the wear volume was calculated from the measured wear track area.

### Tensile Test

The mechanical properties were evaluated using a nano-micro tensile testing system (Kammrath & Weiss GmbH, Germany). According to the operational constraints of the instrument, custom dog-bone-shaped specimens were designed, with a gauge length of 6.75 mm. Only specimens that fractured within the designated gauge section were considered valid, and a minimum of ten tests was conducted for each group. The tensile loading rate was set at 2  $\mu\text{m/s}$ ; a relatively slow rate was chosen to capture the deformation behavior and microstructural evolution during loading. Since the testing system records only force and displacement data, engineering stress-strain curves were obtained by recalculating the results based on the initial gauge length and cross-sectional area of each specimen. The dimensions of the tensile test specimens are listed in Table 3.

Table 3. Tensile specimen size chart

|               | Unit (mm) |
|---------------|-----------|
| Max thickness | 4         |
| Max width     | 10        |
| Max length    | 40        |
| min length    | 25        |

## RESULTS AND DISCUSSIONS

### Effect of SiO<sub>2</sub> Particle Dispersion on Hardness

Table 4 and Fig. 3(a) present the relationship between silica filler content, dispersion, and the hardness of the composites. As shown in Fig. 3(b), the composite hardness increases with silica content, while at a constant filler loading, higher dispersion also leads to improved hardness. In this study, silane coupling agents in combination with the sol–gel method were employed to enhance the uniform dispersion of silica within the polymer matrix, aiming to achieve optimal reinforcement. However, the results demonstrate that the effect of silica content on hardness is more direct and pronounced. This outcome can be attributed to the surface-related nature of hardness: when a greater number of silica particles are exposed at the surface, hardness correspondingly increases. Accordingly, the most significant reinforcement was obtained at 10 wt% filler loading. Nonetheless, dispersion still exerts a secondary influence; under identical SiO<sub>2</sub> contents, composites

with more uniform dispersion and smaller particle size exhibit superior hardness enhancement.

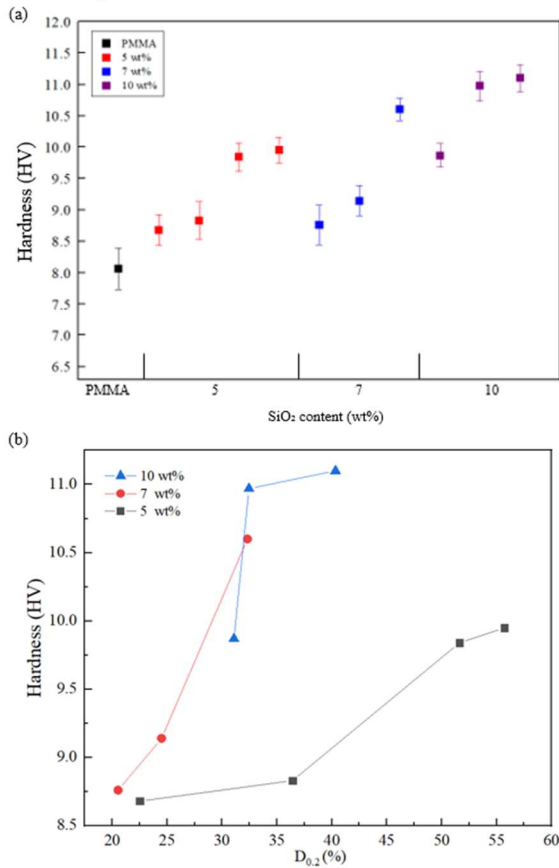


Fig. 3 (a) Variation of composite hardness with SiO<sub>2</sub> content; (b) Correlation between D<sub>0.2</sub> dispersion index and hardness at different SiO<sub>2</sub> loadings.

### Effect of SiO<sub>2</sub> Content on the Tribological Behavior of PMMA Composites

As shown in Table 4 and Fig. 4 (a)-(b), the relationships among D<sub>0.2</sub>, coefficient of friction, and wear rate indicate that wear behavior is primarily governed by filler content. Similar to hardness, wear is a surface-related property. With higher silica loadings in the composites, more particles are released during the wear process. These particles roll along the sliding interface and progressively adhere to the counterface, forming a transfer layer that reduces direct surface

contact and consequently enhances the tribological performance of the composites.

Further insights can be drawn from Fig. 5 (a)-(f), where all composites exhibited slight material removal during wear testing. In samples with poorer dispersion, block-like detachment was observed, representing typical adhesive wear behavior in polymers. This form of wear arises from the stretching and rupture of entangled polymer chains at the interface, leading to local adhesion, plastic deformation, and material detachment. The occurrence of adhesive wear increases surface roughness during sliding, thereby elevating the coefficient of friction and impairing wear resistance. At 5 wt% filler content, insufficient mechanical strength resulted in adhesive wear characterized by layered delamination. In contrast, at 7 wt% loading, the composites exhibited adequate structural integrity to withstand wear, showing substantially less damage compared with the 5 wt% condition. When the filler loading was further increased to 10 wt%, only minor wear scars were observed without significant material detachment. At this concentration, the release of silica particles contributed to interfacial lubrication, effectively lowering friction and markedly improving wear resistance.

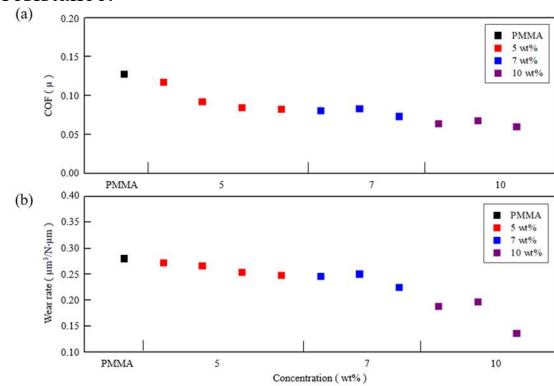


Fig. 4 (a) Relationship between SiO<sub>2</sub> content and coefficient of friction; (b) Relationship between SiO<sub>2</sub> content and wear rate.

Table 4. Summary of SiO<sub>2</sub> dispersion, average particle size, hardness, friction coefficient, wear rate, and surface roughness of PMMA composites.

| Code name   | D <sub>0.2</sub> (%) | Average particle size (nm) | Hardness (HV) | Coefficient of friction (μ) | Wear rate (μm <sup>3</sup> /N·μm) | Surface roughness, Ra (μm) |
|-------------|----------------------|----------------------------|---------------|-----------------------------|-----------------------------------|----------------------------|
| PMMA        |                      |                            | 8.1           | 0.128                       | 0.291                             | 5.584                      |
| P5N012090   | 22.52                | 118                        | 8.68          | 0.117                       | 0.272                             | 5.371                      |
| P5N0540180  | 36.42                | 156                        | 8.83          | 0.092                       | 0.266                             | 4.631                      |
| P5N160270   | 51.63                | 204                        | 9.84          | 0.085                       | 0.183                             | 2.037                      |
| P7N0140270  | 20.53                | 260                        | 8.76          | 0.081                       | 0.262                             | 4.361                      |
| P7N056090   | 24.5                 | 200                        | 9.14          | 0.083                       | 0.247                             | 3.388                      |
| P7N120180   | 32.32                | 238                        | 10.6          | 0.073                       | 0.135                             | 1.885                      |
| P10N0160180 | 31.09                | 339                        | 9.87          | 0.064                       | 0.088                             | 0.311                      |
| P10N0520270 | 32.45                | 435                        | 10.97         | 0.068                       | 0.097                             | 0.755                      |
| P10N14090   | 40.33                | 332                        | 11.1          | 0.060                       | 0.036                             | 0.144                      |
| P5N160180V  | 55.73                | 148                        | 9.95          | 0.082                       | 0.258                             | 3.875                      |

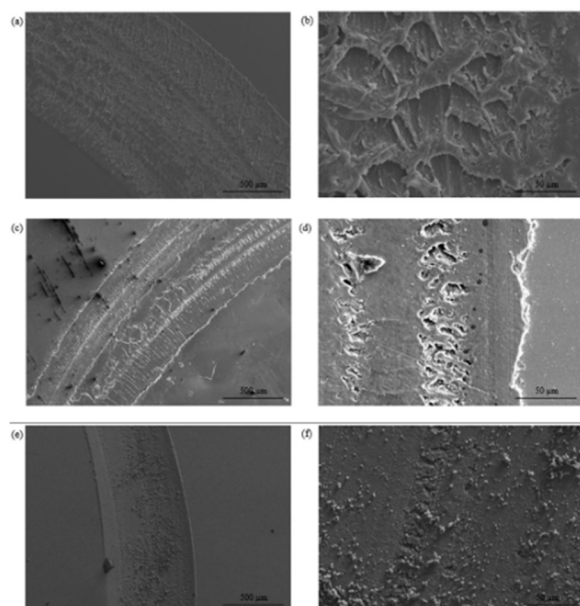


Fig. 5 Worn surfaces of PMMA/SiO<sub>2</sub> composites under different filler contents: (a)–(b) 5 wt%, (c)–(d) 7 wt%, and (e)–(f) 10 wt%

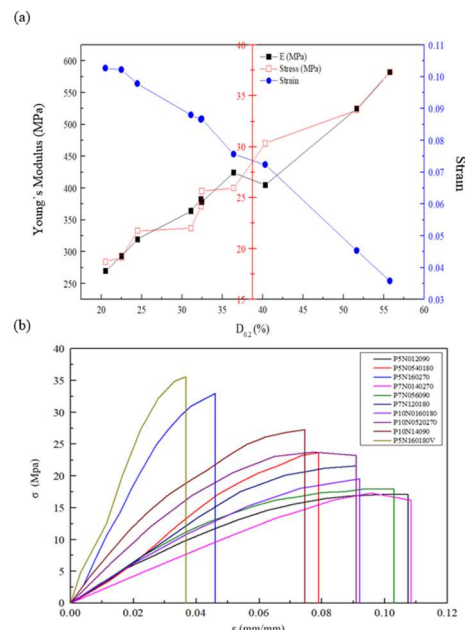


Fig. 6 Results of tensile tests of PMMA/SiO<sub>2</sub> composites: (a) correlation with  $D_{0.2}$ ; (b) representative stress–strain curves.

Table 5. Summary of tensile strength, Young's modulus, and strain of PMMA/SiO<sub>2</sub> composites with different dispersion degrees.

| Code name   | $D_{0.2}$ (%) | Tensile strength (MPa) | Young's Modulus (MPa) | Strain            |
|-------------|---------------|------------------------|-----------------------|-------------------|
| P5N012090   | 22.52         | $18.88 \pm 1.22$       | $289.4 \pm 8.1$       | $0.102 \pm 0.005$ |
| P5N0540180  | 36.42         | $25.53 \pm 1.56$       | $391.44 \pm 5.0$      | $0.076 \pm 0.003$ |
| P5N160270   | 51.63         | $34.48 \pm 1.12$       | $516.95 \pm 4.2$      | $0.045 \pm 0.003$ |
| P7N0140270  | 20.53         | $17.95 \pm 1.65$       | $272.32 \pm 1.7$      | $0.103 \pm 0.006$ |
| P7N056090   | 24.5          | $19.82 \pm 1.05$       | $310.04 \pm 3.8$      | $0.098 \pm 0.007$ |
| P7N120180   | 32.32         | $23.50 \pm 1.74$       | $383.12 \pm 7.5$      | $0.087 \pm 0.008$ |
| P10N0160180 | 31.09         | $21.38 \pm 1.16$       | $347.83 \pm 6.3$      | $0.088 \pm 0.005$ |
| P10N0520270 | 32.45         | $25.36 \pm 1.01$       | $391.13 \pm 9.1$      | $0.087 \pm 0.002$ |
| P10N14090   | 40.33         | $29.32 \pm 1.38$       | $432.02 \pm 8.7$      | $0.072 \pm 0.002$ |
| P5N160180V  | 55.73         | $36.89 \pm 1.28$       | $550.78 \pm 2.6$      | $0.036 \pm 0.004$ |

### Correlation Between SiO<sub>2</sub> Dispersion and Tensile Properties of PMMA Composites

As presented in Table 5, the elastic modulus of the composites increased progressively with improved SiO<sub>2</sub> dispersion, exhibiting an approximately linear growth trend. However, a deviation from linearity was observed in the 10 wt% group. This anomaly is attributed to the relatively larger particle size under this condition, where only a limited number of particles could be detected at the same magnification, resulting in an apparently elevated dispersion index. In practice, larger particles are more prone to agglomeration, leading to a weaker reinforcing effect compared with smaller and more uniformly dispersed particles.

The trend in tensile strength was consistent with that of the elastic modulus, as shown in Table 5 and Fig. 6 (a), both demonstrating a linear correlation with dispersion. Table 5 further summarizes the fracture strain, where the sample with a  $D_{0.2}$  value of 51.63% exhibited the best performance, with a fracture strain of only 0.045. As illustrated in Fig. 6 (b), strain also maintained a stable linear relationship with dispersion.

Overall, these findings confirm that particle size, distribution, and quantity significantly affect the mechanical properties of the composites, including elastic modulus, tensile strength, and fracture strain. The results suggest that appropriate particle spacing and dispersion are crucial for delaying crack propagation and achieving effective toughening. Although the 10 wt% exhibited a higher dispersion index than the 5 wt% and 7 wt% groups, the excessive particle size reduced the specific surface area and weakened the interfacial strengthening effect between the particles and the matrix. Consequently, the overall reinforcing effect did not continue to increase with higher filler content.

### CONCLUSIONS

The hardness and wear tests revealed that both properties are governed by the surface characteristics of the composites. At the same filler content, particle size and dispersion significantly influenced the Young's modulus and tensile strength. Although smaller particle sizes generally provide superior reinforcement, the degree of dispersion is equally

critical, as both modulus and strength increase with improved uniformity of dispersion.

In contrast, fracture strain is affected by multiple factors, with particle dispersion and particle number per unit area playing dominant roles. To achieve effective toughening, particles of different sizes must maintain appropriate spacing and dispersion, thereby enhancing crack resistance and overall ductility.

## ACKNOWLEDGMENT

This work was supported by the National Science and Technology Council (NSTC), Taiwan, under Grant Nos. 112-2221-E-006-173, 113-2221-E-006-087-MY2, 113-2221-E-006-112-MY2, and 113-2221-E-006-116. The authors gratefully acknowledge the Core Facility Center at National Cheng Kung University, Taiwan, for providing access to the EM000800 and SQUID000200 instruments, which were funded by the NSTC project 114-2740-M-006-001. Additional support from the Higher Education Sprout Project of the Ministry of Education, Taiwan, through the Headquarters of University Advancement at National Cheng Kung University (NCKU), is also gratefully acknowledged.

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