

Advanced Optical Metrology

Composites







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Composites

THE CONCEPT

of combining two or more materials to produce an end material with improved and unique properties dates back to 3400 B.C. when Mesopotamians glued wood strips at different angles to create plywood for structural applications. Later, in the 12th century, the Mongols created the first composite bow made of a combination of wood, bamboo, bone, cattle tendons, horn, silk, and pine resin. The modern era of composites began with the development of plastics in the early 1900s ^[1]. Since then, manufacturers, engineers, and researchers have continued to develop composites made using a wide variety of materials for a broad spectrum of applications.

The two constituents of a composite are the matrix and reinforcement, whereby the matrix surrounds and binds the reinforcement. Composites combine the best properties of the constituent materials, which remain separate and distinct within the final composite structure as they do not blend or dissolve each other ^[2]. Composite materials are often preferred to conventional materials, such as metals and ceramics, due to their high specific strength, light weight, ease of fabrication, design flexibility, resistance to fatigue and corrosion, and low cost ^[3–7]. Today, composites are commonly applied in the aerospace, military, automotive, and construction industries, where they have largely replaced conventional materials.

As with all materials used to create objects, defects and damage can occur during manufacture or in service. In service, composites are subjected to static, fatigue, impact loads, and extreme conditions (e.g., high temperatures and moisture), affecting their performance ^[8]. Therefore, it is important to ensure composites' structural integrity, strength, and performance throughout their service life using robust and reliable inspection techniques, especially in safety-critical industries like aerospace. However, compared with conventional materials, which comprise only one type of uniform, isotropic material with known and predictable properties, composite materials are inhomogeneous and anisotropic with varying and less predictable properties ^[9]. Thus, several nondestructive testing techniques have been developed for composite inspection purposes. This introduction briefly describes some composite types discussed in the following digest articles and the most important techniques to inspect and monitor composite materials.

1. COMPOSITE TYPES

Particulate-reinforced metal matrix composites (PRMMCs): PRMMCs consist of a metal matrix reinforced by ceramic or organic particles and exhibit better mechanical properties (e.g., higher stiffness and strength) than the corresponding unreinforced matrix ^[10]. PRMMCs are most commonly manufactured by powder metallurgical and casting processes, with the latter being performed in the liquid or liquid-solid state [11,12]. Their mechanical properties depend mainly on the load-carrying capacity and morphology of the ceramic reinforcement particles. Aluminum has a low density, high ductility, and good corrosion resistance; therefore, it is widely used in PRMMCs. Particle-reinforced aluminum composites are popular because many inexpensive reinforcements and working processes to shape aluminum are available [13]. Moreover, aluminum-based composites are in great demand in the aerospace and automotive industry because of their high strength-to-weight ratio [14]. In the digest article "Wettability of Low Weight Borides by Commercial Aluminum Alloys—A Basis for Metal Matrix Composite Fabrication," the wetting behavior of different boron compounds by aluminum alloys was investigated to identify promising boride reinforcements for the fabrication of aluminum-based composites.

Piezoelectric composites: Piezoelectric materials are commonly used as actuator and sensor materials because of their ability to translate a mechanical impulse into an electric response ^{[15–}

^{17]}. Ceramic/polymer composites in which a ferromagnetic ceramic filler is embedded in a polymer matrix combine the piezoelectric properties of the filler with the flexibility of the matrix. The properties of a composite depend on the number of phases, the volumetric fraction of each phase, the properties of each phase, and the connectivity of the phases [18]. The connectivity describes the configuration; that is, how the phases are interconnected in the composite. Each phase can connect up to three directions, whereby the first configuration number indicates the connectivity of the dispersed filler and the second one that of the matrix. The connectivity of the filler controls the electric flow distribution in the composite ^[19]. Composites with 0-3 connectivity consist of homogeneously dispersed particles in the matrix and are most commonly produced because of their simple fabrication. However, composites with 1-3 connectivity exhibit higher piezoelectric activity because their particles align along the preferred direction in the composite [20, 21]. Piezoelectric composite-based sensors have various applications, especially in the automotive industry, where they are used to assess the acceleration and damping force in semi-active suspension [22, 23]. The digest article "Micro-Structuration of Piezoelectric Composites Using Dielectrophoresis: Toward Application in Condition Monitoring of Bearings" describes the fabrication of a piezoelectric composite-based sensor in 1-3 configuration, consisting of a lead zirconate titanate filler and polydimethylsiloxane matrix, for the condition monitoring of aircraft ball bearings.

Hybrid composites: Hybrid composites comprise two or more types of reinforcements in the same matrix. They are fabricated with the aim to synergize the properties of the reinforcements, leading to materials with superior properties to those of conventional and (non-hybrid) single composites. The overall properties of hybrid composites are considered a weighted sum of the individual constituents in which the inherent advantages and disadvantages are balanced [24]. Thus, the advantageous property of one reinforcement can counterbalance the disadvantageous property of another reinforcement. Accordingly, cost-effective hybrid composites with desired properties can be produced by proper material choice. The mechanical properties of a hybrid composite strongly depend on fiber orientation, fiber length, fiber content, fiber/ matrix adhesion, and failure strain of individual fibers ^[25]. A positive hybrid effect is obtained when the mechanical properties of the hybrid exceed those of the corresponding non-hybrid composites. The properties of a hybrid system consisting of two components can be determined by the rule of mixtures ^[24]: $P_{H} =$ $P_1V_1 + P_2V_2$, where P_H is the mechanical property of the hybrid, P_1 and P_2 the properties of the first and second system, respectively, and V_1 and V_2 the corresponding volume fractions of the systems. Hybrid composites are used in many engineering applications because of their light weight, high strength, and ease of fabrication. In particular, the automotive industry applies hybrid composites for various applications ^[26]. In the digest article "Mechanical Properties and Slurry Rrosion Resistance of a Hybrid Composite SiC Foam/SiC Particles/ EP," a novel hybrid composite is presented consisting of an E-51 epoxy resin (EP) as the matrix and SiC foam/SiC particles as reinforcements. The hybrid composite outperformed the non-hybrid composite SiC_{foam}/EP in terms of mechanical and anti-erosive properties.

2. COMPOSITE INSPECTION SOLUTIONS

Conventional ultrasound testing: Ultrasound testing is the most commonly used nondestructive inspection method for composite materials ^[27]. In ultrasound testing, a transducer generates high-frequency waves that propagate through the material before being received by the same or a second transducer. As the motion of any wave is affected by the medium (e.g., composites) through which it propagates, one or more of the following parameters asso-

ciated with high-frequency waves are changed: scattering, frequency, transit time, and attenuation^[27]. The changes in these parameters provide valuable information on the properties of materials, such as hardness, elastic modulus, density, and grain structure. Therefore, ultrasonic testing can evaluate composites and detect flaws, such as hidden cracks, voids, and porosity. However, conventional ultrasound testing is often unsuitable for composite inspection because of their inhomogeneous and anisotropic nature ^[28]. Ultrasound wave propagation in anisotropic composites is intricate and accompanied by random scattering and high attenuation of ultrasound waves, impeding defect detection ^[27, 29].

Phased array ultrasound testing: Phased array ultrasound testing can overcome the limitations of conventional ultrasound testing by steering ultrasound waves to create constructive interference of the wavefronts, focusing the energy ^[30]. In contrast to conventional ultrasound testing where a single-element transducer generates ultrasound waves, phased array ultrasound testing systems use multi-element transducers that can be separately pulsed in a programmed manner. Each transducer emits a spherical wave at a specific time so that the superimposed wavefront steers and shapes the final beam. In this way, a vast number of ultrasound beam profiles can be generated from a single probe, enabling electronic scanning. These capabilities make phased array ultrasound testing a powerful, nondestructive composite inspection solution that is rapidly becoming more popular in the composite industry.

Confocal laser scanning microscopy (CLSM): CLSM is an optical imaging technique that combines high-resolution optical imaging with depth selectivity, allowing the collection of two-dimensional images at different depths (optical slicing) and, consequently, the construction of three-dimensionally resolved images of the investigated sample. CLS microscopes comprise a confocal optical system with a spatial pinhole positioned in front of the detector that eliminates out-of-focus light, leading to micrographs of high optical resolution with excellent contrast. In detail, when a laser illuminates the pinhole, the light emitted from the pinhole passes through a beam splitter and is focused by an objective lens to a spot on the sample, which is placed at the focal plane [31]. Light reflected from this spot on the sample's surface travels back to the beam splitter, where it is converged on the pinhole and directed toward the detector. Light reflected from parts of the sample outside the focal plane is not detected.

Although the confocal microscope was invented in 1953 by Minky [32], his invention remained largely unused until the development of lasers, providing the system with a light source of sufficient brightness and stability. Since then, CLSM has been widely used to study and inspect composites. CLSM has some key advantages over conventional widefield microscopy in the field of material science ^[33]. For example, three-dimensional imaging by CLSM allows detailed analysis of the (micro-) structure of composites without the need for thin material sections. Furthermore, the horizontal and vertical resolution of CLSM is higher than that of conventional light microscopy. Thus, CLSM represents an excellent composite inspection solution. Three of the following digest articles demonstrate the application of CLSM to analyze the surface morphology of different composite materials.

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Wettability of Low Weight Borides by Commercial Aluminum Alloys — A Basis for Metal Matrix Composite Fabrication

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ABSTRACT

Boron compounds are promising candidates for reinforcing metals because of their high strength and stiffness at low specific weights. For composite fabrication, wetting behavior is important. Isothermal wetting of B_4C , AlB_2 , CaB_6 , MgB_2 , and TiB_2 by liquid pure aluminum (99.5%) and the alloy AlSi10MgMn was studied at 700 °C (1292 °F) for 15 minutes in a vacuum using the dispensed drop method. No wetting occurred for all substrates during testing. The contact angles remained constant for CaB_6 , MgB_2 , and TiB_2 , while those for AlB_2 and B_4C decreased continuously with time.

INTRODUCTION

Particulate-reinforced aluminum metal matrix composites (MMCs) combine the beneficial properties of aluminum (low density and high ductility) and ceramic reinforcement materials (high strength and modulus), making them attractive engineering materials for the aerospace and automotive industries [1, 2]. The most commonly used manufacturing processes for particulate-reinforced MMCs are powder metallurgical and casting processes ^[2,3]. Aluminum casting is typically performed at approximately 700 °C (1292 °F); thus, the particulate reinforcements should be stable under these conditions. To obtain stiff and lightweight MMC parts, the reinforcement particles should increase the mechanical properties and decrease the density of the composite. Depending on the loading geometry, the effect of density may be

more important than the mechanical properties, which is why low weight metal borides are attractive reinforcement materials.

Various metal borides are available; some of them are summarized in **Table 1**. In this study, we focused on stiffness, whereby particles with high Young's modulus and low density provide the greatest enhancement in composite stiffness. TiB₂ promises the most efficient enhancement in Young's modulus; however, taking the density into account, the highest specific stiffness can be achieved by adding CaB₆ and B₄C (**Figure 1**).

Many attempts have been made to disperse ceramic particles in liquid aluminum alloys, but the poor wettability of most ceramic particles with liquid aluminum promotes the agglomeration of particles in the melt and hinders homogenous particle distribution.

Properties	$A1B_2^{(a)}$	$\mathbf{B}_4 \mathbb{C}^{\mathbf{a}}$	$CaB_6^{a)}$	$\mathrm{MgB}_{2}^{\mathrm{a})}$	$TiB_2^{(b)}$
Purity [%] Theoretical density [kg m ⁻³] Young's modulus [CBa]	99.5 3.19 ^[23] 244. 269 ^[25,26]	99.5 2.52 ^[24] 441 472 ^[25–31]	99.5 2.45 ^[23] 451 ^[23]	99.5 2.57 ^[23] 278: 200 ^[25,32,33]	99.9 4.52 ^[23] 500 591 ^[23,25,34–36]
Bulk density [kgm ⁻³] Pycnometer density [kgm ⁻³]	2.43 2.68	2.51 2.52	1.78 2.46	1.90 2.57	3.74
Total porosity [%] Open porosity [%] Roughness Ra [µm]	23.82 9.03 1.84	0.40 0.63 0.30	27.35 27.50 2.98	26.07 25.50 1.31	17.26 12.67 1.99

Table 1: Properties of the ceramic substrates used for the wettability studies and calculation of the composite stiffness. a) produced by Metallic Flex GmbH (Habichtswald, Germany); b) produced by Mateck GmbH (Juelich, Germany).

There are different testing procedures to measure the contact angle and thus the wetting behavior of aluminum in contact with ceramic substrates. In the classical sessile drop procedure, the ceramic substrate and aluminum sample are jointly heated from room temperature (contact heating), whereas in the dispensed drop procedure, the aluminum sample is heated separately and squeezed onto the substrate from above (non-contact heating). In the dispensed drop procedure, the liquid aluminum drop is free of oxides; that is, the result is supposed to be free from artifacts, at least at the beginning of the test ^[4].

While there are studies on the wetting properties of TiB_2 and B_4C with pure aluminum, no data on the wetting properties of AlB_2 , CaB_6 , and MgB_2 with liquid aluminum and its alloys have been published so far.

Therefore, in this study, the wetting behavior of the aluminum alloys Al99.5 and AlSi10MgMn with the substrates AlB₂, B_4C , CaB_6 , MgB_2 , and TiB_2 was investigated at a moderate temperature (700 °C, 1292 °F) and time (15 minutes) using the dispensed drop method.

METHODS

The boride substrates for the wetting investigations were produced via hot pressing, and their densities were measured using a helium pycnometer and compared with the theoretical and bulk densities to quantify the amount of porosity. After grinding and polishing, the surface roughness of the substrates was measured using an Olympus[®] LEXT[™] OLS4000 laser scanning microscope. The properties of the ceramic substrates are listed in **Table 1**.

The wetting kinetic tests were performed at the Foundry Research Institute (Cracow, Poland).

A detailed description of the experimental procedure can be found in Eustathopoulos et al. ^[4] and Sobczak et al. ^[5]. The dispensed drop method was applied in all tests to produce oxide-free droplets ^[4]. For this purpose, the aluminum sample was placed in an alumina capillary located above the substrate inside a molybdenum furnace and then (non-contact) heated to 700 °C (1292 °F) within 60 minutes in vacuum (<10⁻⁵ mbar). When the testing temperature was reached, the melt was pushed and dropped onto the substrate. Images of drop/substrate couples at 700 °C were recorded for 15 minutes using a high-resolution CCD camera, and contact angles were calculated.

RESULTS AND DISCUSSION

Depending on the initial powder size and hot-pressing parameters of the ceramic substrates, the density and corresponding porosity varied over a wide range (**Table 1**). For



Figure 1: Stiffness index $E^{1/3}/\rho$ as a function of the reinforcement content.

AlB₂, the bulk and pycnometer densities differed significantly from the theoretical density. This could be due to an incomplete reaction between the aluminum and boron powder during hot pressing, resulting in a mixture of boron, aluminum, and AlB₂ phases. The amount of total porosity (i.e., the difference between the theoretical and bulk density) can be divided into the closed and open porosity; the latter influences the surface roughness and wetting properties of the material. Although all substrates were polished in the same way, their surface roughness varied from 0.3 to 2.98 μ m due to differences in their open porosity. Rough surfaces can affect the spreading of the liquid drop, leading to apparent contact angles ^[4, 6]. Although MgB₂ exhibited a large amount of open porosity, its roughness was lower than that of TiB₂, AlB₂, and CaB₆ because of differences in the hot-pressing process and initial powder size. Moreover, polishing can clog the pores of the



Figure 2: Isothermal wetting kinetics of the dispensed drops at 700 °C with the changes in drop diameter and height for Al99.5 and AlSi10MgMn on (a) CaB₆, (b) MgB₂, (c) TiB₂, (d) B₄C, and (e) AlB₂.

surface, and, as a result, lower surface roughness is measured by laser scanning microscopy while still a large amount of open porosity can occur as the helium pycnometer measurement is not affected by surface clogging.

Figure 2 illustrates the time-dependent wetting behavior of the investigated ceramic substrates with Al99.5 and the AlSi10MgMn alloy at 700 °C (1292 °F) for 15 minutes. No wetting (contact angle $\theta > 90^\circ$) occurred for all examined couples. The contact angles for Al99.5 were higher than those for AlSi10MgMn, except for the MgB₂ substrate in which case the contact angle for Al99.5 was slightly lower (~4°). The reason for the lower contact angles for the AlSi10MgMn alloy is that most alloying elements, especially the combined addition of silicon and magnesium, reduce the surface tension ^[7].

Furthermore, different drop spreading characteristics were identified. A non-wetting behavior with no spreading was observed for CaB₆, MgB₂, and TiB₂. The contact angles remained almost constant for 15 minutes for both the pure aluminum and alloy. Consequently, the height and diameter of the liquid drop also remained constant (Figure 2). The initial contact angles measured immediately after drop dispensing were approximately 126°–127° for AlSi10MgMn on CaB₆, MgB₂, and TiB₂ and 134°–136° for Al99.5 on CaB₆ and TiB₂. After 15 minutes, the contact angle for Al99.5 on TiB₂ decreased by $3^{\circ}-4^{\circ}$, and the drop diameter increased slightly by 5%. However, these changes were too small to assume a reactive wetting behavior. Even the large amount of open porosity did not lead to the infiltration of liquid aluminum.

A different wetting behavior was observed for AIB_2 and B_4C for which drop spreading occurred with time. Liquid aluminum reportedly reacts with B_4C by forming intermetallic phases; however, the wettability is poor at temperatures below 900 °C (1292 °F) ^[8, 9]. The initial contact angles were 118° and 126° for AlSi10MgMn and Al99.5 on B_4C , respectively. A decrease in the contact angle by 11°–13° and slow spreading were observed along the B_4C surface as the drop diameter increased, and the drop height decreased during testing.

Both Al99.5 and AlSi10MgMn penetrated the AlB₂ substrate during testing, leading to decreased drop heights but constant drop diameters. The initial contact angles after drop deposition were 119° and 129° for AlSi10MgMn and Al99.5, respectively, which after 15 minutes decreased to 107° and 120°, respectively. The AlB₂ substrate was probably a mixture of boron, aluminum, and AlB₂ phases, significantly influencing its wetting behavior. In the absence of disturbing oxide layers, liquid metals wet metal surfaces. Therefore, aluminum impurities present in the substrate could exhibit strong adhesive interactions with the liquid aluminum drop, forcing the spreading of the drop. However, due to the open porosity and surface roughness of the substrate, the liquid aluminum drop preferred to infiltrate the AlB₂ substrate.

CONCLUSION

The wetting behavior of the liquid aluminum alloys Al99.5 and AlSi10MgMn on B_4C , AlB_2 , CaB_6 , MgB₂, and TiB₂ was investigated at 700 °C (1292 °F) for 15 minutes via the dispensed drop technique. No wetting occurred within 15 minutes for all substrates and alloys. The contact angles of both aluminum alloys remained almost constant on CaB₆, MgB₂, and TiB₂ during testing. However, for B₄C and AlB₂, changes in the drop shape were observed, indicating reactive wetting. In addition, alloying elements reduced the contact angles of AlSi10MgMn, which were lower than those of Al99.5 on most substrates.

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O2 Micro-Structuration of Piezoelectric Composites Using Dielectrophoresis: Toward Application in Condition Monitoring of Bearings

G. D'Ambrogio, O. Zahhaf, Y. Hebrard, et al.

ABSTRACT

A piezoelectric material based on inorganic/organic composites suitable for aerospace and aeronautical monitoring sensors was fabricated. The designed piezoelectric composite was made of a lead zirconate titanate (PZT) filler embedded in a polydimethylsiloxane (PDMS) matrix. To improve the piezoelectric properties of the film, we increased the connectivity of the ceramic filler via electric field-assisted structuration, leading to a columnar arrangement of the filler across the thickness. This structure (1-3)showed higher piezoelectricity than one with a randomly dispersed filler (0–3). Piezoelectric and dielectric activities of PZT/PDMS in 0–3 and 1–3 configurations were compared at different volume fractions. The 1–3 connectivity led to superior piezoelectric behavior. Thermal stability and high-temperature X-ray diffraction analyses indicated that the composites were stable and maintained a good piezoelectric response even at 200 °C (392 °F). Following process optimization, the piezoelectric behavior of this new class of composites approached that of fluorinated ferroelectric polymers, with the advantage that the stability of the piezoelectric properties was preserved at a higher temperature and lower poling electric field. In conclusion, there is potential for integrating the designed sensor in aircraft ball bearings for condition monitoring.

INTRODUCTION

In aeronautics and aerospace, there is a growing need for new sensor materials suitable for direct health condition monitoring of complex structures. The main requirements when designing such sensors are sensibility, frequency bandwidth, and temperature drift ^[1, 2]. For simple system integration, these sensors must also be light and non-intrusive for the system with high mechanical resistance and easy processability.

Due to their ability to translate a mechanical impulse into an electric response, piezoelectric materials are commonly used as sensors in vibration monitoring, impact detection, and ultrasonic receiving sensors ^[2–5]. Different classes of piezoelectric materials exist, such as ferroelectric ceramics, piezoelectric polymers, and piezoelectric composites made of a ferroelectric ceramic filler embedded in a polymer matrix. Piezoelectric composites are most suitable for the above applications as they combine the piezoelectric properties of the filler with the flexibility of the matrix ^[5]. Furthermore, they are characterized by a low poling electric field and high Curie temperature, making them more suitable than ferroelectric polymers ^[6]. Being well workable, piezoelectric composites can be simply integrated into complex designs ^[5].

Phase arrangement is a key factor influencing the piezoelectric properties of a composite. Composites with 0–3 connectivity are most commonly produced because of their simple fabrication. However, they exhibit low piezoelectric sensibility until they reach large volumetric filler contents, which in turn deteriorate the mechanical properties ^[7]. A solution to this problem is to align the fillers in columns within the matrix. In such anisotropic structures, called 1–3, the particles are closely aligned along the preferred direction, enhancing the piezoelectric properties of the whole material ^[4, 5].

In this study, dielectrophoresis was used to develop a piezoelectric sensor coating consisting of a polydimethylsiloxane (PDMS) matrix filled with lead zirconate titanate (PZT) particles. The effect of the particles' alignment on the piezoelectric and dielectric properties was investigated using 0–3 and 1–3 PZT/PDMS composites with particle volume contents ranging from 3.2% to 24%. The thermal stability and influence of the poling electric field amplitude on the piezoelectric properties of the composites were also investigated. Furthermore, the dielectrophoresis process was examined for its dependence on the electric field frequency. Finally, as piezoelectric composites are widely exploited in the health monitoring system of structural components ^[4, 5], a potential application is described, indicating that the proposed 1–3 material sensor is suitable for condition monitoring in aircraft bearings.

METHODS

For sample fabrication, linear vinyl terminated PDMS consisting of a base and a crosslinker was selected as the matrix and PZT as the piezoelectric phase. PZT powder and silicone base were mixed by ultrasonication, and the cross-linker was added at a ratio of 1:10 (curing agent/base). The 0-3 composites were prepared by casting the dried mixture as a film on a glass substrate. The casting film was cured for 30 min at 120 °C (248 °F). Finally, the fabricated films were cut into circular shapes, and gold electrodes were sputtered onto both sides of the composites. The 1–3 composites were prepared by pouring the dried mixture into a mold positioned between two aluminum plates. The aluminum plates acted as electrodes for the structuring field. The mold was first incubated for 15 minutes at 25 °C (77 °F) and then for 1 hour at 120 °C (248 °F). During incubation, an AC electric field (amplitude, 2 V µm⁻¹) was applied, leading to aligned particle chains along the poling direction. Samples in both 0-3 and 1-3 configurations were prepared with different PTZ volume contents (3.2%, 12%, and 24%).

The dielectric permittivity of the 0-3 and 1–3 PZT/PDMS composites was determined by dynamic dielectric spectroscopy. To assess the piezoelectric behavior of the composites as a function of particle volume fraction (ϕ), all samples were poled at 100 °C for 30 min under an applied DC electric field (amplitude, $20V \mu m^{-1}$). Composites with the same filler content (24 $\%_{vol}$) were poled under different electric fields (5, 10, 20, and $30\,V\,\mu m^{-1})$ to evaluate the dependence of the piezoelectric behavior on the poling electric field. In addition, the piezoelectric charge coefficient (d_{33}) of all samples was determined and evaluated as a function of the volume fraction and the poling electric field.

To investigate the thermal stability of the piezoelectric composites, we used samples with the same particle content $(24\%_{vol})$. After poling, the composites were annealed for 10 min-



Figure 1: Microscope images of (a) 0–3 PZT/PDMS with 3.2% vol, (b) 1–3 PZT/PDMS with 3.2% vol, and (c) 1–3 PZT/PDMS with 12% vol.

utes in a short circuit at various temperatures (25 °C, 50 °C, 100 °C, 150 °C, and 200 °C (77 °F, 122 °F, 212 °F, 302 °F, and 392 °F)). The samples were then cooled to 25 °C (77 °F), and the d_{33} response was recorded. The morphology and structure of the composites were investigated by X-ray diffraction (XRD) at 25 °C (77 °F), 150 °C (302 °F), and 200 °C (392 °F).

For the dielectrophoresis process, two parallel strips of conductive tape (electrodes) spaced 1 mm apart were placed on a glass microscope slide. A $3.2\%_{vol}$ PZT/PDMS solution was placed on the slide. After applying an electric field between the two stripes, the dependence of the particle's alignment on the electric field frequency was studied at 2 Hz and 2 kHz (constant amplitude, $2V\mu m^{-1}$). The microstructures of the particle chains were observed with an Olympus[®] BH microscope.

The full article of this digest includes a detailed description of 0–3 and 1–3 connectivity models.

RESULTS AND DISCUSSION

The dielectric permittivity of the structured sample (1–3) was significantly greater than that of the randomly dispersed sample (0–3). Compared with the 0–3 composites, the 1–3 composites filled with $12\%_{vol}$ and $24\%_{vol}$ PZT showed an approximate three- and two-fold increase in permittivity, respectively. In 1–3 composites, the dielectric particles are closely packed in the alignment direction and less shielded by the polymer matrix, causing a higher permittivity in the column direction. This is the first proof that dielectric poperties of composites. The dielectric permittivity

ity of the 1–3 composites was comparable to that of fluorinated ferroelectric polymers ^[8].

The 1–3 composites also exhibited markedly enhanced d_{33} values, regardless of their volume content. This can be explained by the more favorable distribution of the electric field in structured composites, allowing greater polarization. Plotting the d_{33} values of the 1–3 composite (24%_{vol} PZT) against increasing poling field amplitudes revealed a saturation of the poling level at 20 V µm⁻¹ above which all the dipoles in the particles were oriented. Higher electric fields did not further increase the piezoelectric response. Even at this low poling field, the d_{33} value of the 1–3 composite was similar to that of classical ferroelectric polymers, which usually require an electric field of >100 V µm⁻¹.

The piezoelectric activity of the composites was also analyzed at high temperatures to investigate their thermal stability. Both 0-3 and 1–3 PZT/PDMS (24%vol PZT) composites maintained their piezoelectric activity up to a temperature of 100°C (212 °F), above which d_{33} started to decrease gradually. Between 150 °C and 200 °C (302 °F and 392 °F), d₃₃ decreased by 20%-30% due to some temperature-induced structural changes in the crystalline cells. Beyond 350 °C (662 °F), which is the Curie temperature of PZT, the PZT particles exhibit a morphotropic phase boundary (MPB) perovskite structure, whereas, above the Curie temperature, the cell structure is cubic ^[9]. The MPB cell is formed by the coexistence of rhombohedral and tetragonal ferroelectric phases, whereas the cubic cell is symmetrical and paraelectric ^[9]. In the high-temperature XRD analysis, the rhombohedral phase peak of the 0-3 and 1-3 samples decreased with increasing temperature, leading to an increase in the tetragonal phase. At 200 °C





Volume fraction [%vol]	d₃₃ 0–3 [pC N ⁻¹]	d₃₃ 1–3 [pC N ⁻¹]
0	0	0
3.2%	0.07	2
12%	0.1	5.2
24%	5.4	12.45

Table 1: Piezoelectric charge coefficient, crystalline volume fraction, poling electric field, and FoM of the present composites compared with those of conventional piezo-electric composites and polymers.

(392 °F), the symmetry was only tetragonal. This was more noticeable in 0–3 than in 1–3 composites, indicating that the lattice stability of the structured composites was higher. These results indicate that the present composites, regardless of their structure (random or aligned), can withstand temperatures up to 200 °C (392 °F) without significantly changing their piezoelectric behavior. This makes them more suitable for high-temperature applications than classical ferroelectric polymers, which are more temperature-sensitive.

Electrically induced structures in 0–3 and 1–3 PZT/PDMS composites with volume fractions of $3.2\%_{vol}$ and $12\%_{vol}$ were visu-

ally observed. In the 0–3 sample $(3.2\%_{vol})$, the particles dispersed randomly (**Figure 1a**), whereas, in the 1–3 sample $(3.2\%_{vol})$, they exhibited a chain-like structure (**Figure 1b**). In the 1–3 PZT/PDMS composite with $12\%_{vol}$ PZT, the filler arranged in a columnar disposition with denser columns due to the increased volume fraction (**Figure 1c**).

The dependence of the dielectrophoresis process on electric field frequency was analyzed. **Figure 2** shows the isotropic structure of the 0–3 composite before the process and the aligned inorganic particles within the polymer matrix after applying an electric field at different frequencies (2 Hz and 2 kHz). Structuring was more effective at 2 Hz as the particle columns were more defined and continuous than those obtained at 2 kHz. The average thickness of the columns was 23.9 µm at 2 kHz and 48.6 µm at 2 Hz. Moreover, the d_{33} value of the 1–3 composite (24%_{vol}) processed at 2 Hz was 28% higher than that of the composite processed at 2 kHz.

The present composites were then compared with other piezoelectric composites and polymers in terms of d_{33} , poling electric field (E_{Pol}) , and volumetric fraction of the crystalline phase (φ_{Cr}) . On the basis of these parameters, a figure of merit [FoM = $d_{33}/(E_{Pol} \times \varphi_{Cr})$] was calculated, indicating the profitability of the materials (**Table 1**). According to this

FoM, the 1–3 PZT/PDMS composite is superior to other reported materials $^{[10-17]}$.

Rolling-element bearings are an important part of jet engines, and condition monitoring can help detect bearing faults and predict bearing life. However, the development of smart bearing coatings is complex because of the extreme bearing environment (e.g., high shaft rotational speed, vibration, and temperature). Piezoelectric coatings are promising candidates for developing smart bearings as they are thermally stable and easy to process. **Figure 3** shows the implementation of the present piezoelectric layer on a bearing.

The sensing response of the piezoelectric coating was compared with that of a classical accelerometer. Regarding the temporal response, the signals of both sensors had a similar appearance of vibrations; that is, the amplitude was relatively small and steady before rotation but varied and increased significantly during rotation (Figure 4a). This observation indicates that piezoelectric materials can provide information on the vibration. To further analyze these responses, we performed a fast Fourier transform (FFT)-based frequency method. Figure 4b shows the frequency content of the sensing signal of the piezoelectric coating and the classical accelerometer, which is a simple way to locate frequencies characteristic of faults [18]. The frequency spectra of both sensors differed as they represented different physical quantities. The piezoelectric coating reflected bearing deformations, the frequency harmonics of which (multiples of 12 Hz) were

lower than those produced by the accelerometer (50 Hz), which, in contrast, were related to bearing rotation. Based on the frequency-time analysis shown in **Figure 4b**, variations in rotational speed could be identified.

CONCLUSION

This work highlights the effects of dielectrophoresis on piezoelectric composites made of a silicone matrix doped with PZT particles. The columnar arrangement of particles along the field direction led to significantly improved dielectric and piezoelectric performances. The d_{33} values of 1–3 composites were markedly higher than those of 0-3 composites, regardless of their PZT concentration. The achieved properties make 1-3 composites an excellent alternative to classical ferroelectric polymers as they exhibit similar piezoelectric properties but at a lower poling electric field. The piezoelectric activities of the 0-3 and 1-3 composites were thermally stable up to 200 °C (392 °F). XRD analysis confirmed the structural stability of the composites at different temperatures. These results indicate that the present composites are more suitable for high-temperature environments than ferroelectric polymers. Furthermore, the 1–3 composite was superior to other piezoelectric composites and organic ferroelectric polymers in terms of piezoelectric activity, filler volume content, and poling field. Therefore, the 1-3 piezoelectric material is an excellent candidate for various applications in multifunctional sensing devices, particularly for condition



Electronic interface

Figure 3: Illustration of the piezoelectric coating for bearing condition monitoring.



Figure 4: Responses of a piezoelectric coating mounted on a bearing (red curve) and a classical accelerometer (blue curve). (a) Temporal response and (b) frequency response obtained via FFT and frequency-time analysis (blue background).

monitoring of ball bearings in the aeronautic area. By depositing the piezoelectric element on a bearing ring, we could detect local vibrations indicative of imminent bearing failure. Thus, the piezoelectric material represents a cheap alternative to common accelerometers.

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03 In Situ Integration of SERS Sensors for On-Chip Catalytic Reactions

B. Han, Y.-Y. Gao, L. Zhu, et al.

ABSTRACT

The rapid progress of microfluidics has contributed to modern analytical methodology. However, in situ integration of chemical/biological sensors for on-chip reaction monitoring remains a big challenge. Here, a graphenebased catalytic chip with surface-enhanced Raman scattering (SERS) sensors was fabricated by laser scribing of a graphene oxide (GO)-silver nanoparticle (AgNP) composite (AgNPs@GO). The surface plasmonic effect of AgNPs promoted the photoreduction of GO (RGO), inducing a highly porous nanostructure. By combining the AgNPs@RGO composite patterns with polydimethylsiloxane microfluidic channels, a bifunctional microfluidic chip was fabricated capable of on-chip catalysis and SERS detection. As a proof-of-concept demonstration, onchip AgNP-catalyzed 4-nitrophenol reduction was performed while the reaction process was monitored by the in situ SERS sensor. The integration of SERS sensors with functional microfluidic devices holds great promise for developing advanced Lab-on-a-Chip systems.

INTRODUCTION

Lab-on-a-Chip (LoC) systems are highly integrated devices allowing multiple experimental procedures, including sample injection, reaction handling, product separation, and component analysis, on a single chip ^[1]. The ultralow consumption of fluids makes LoC favorable in medical diagnostics, environmental monitoring, and chemical analysis ^[2–4]. To further extend the functionalities of LoC devices, various functional components have been successfully integrated with microfluidic chips, which significantly facilitated automatic analysis ^[5]. Nevertheless, most of these proof-of-concept devices cannot be applied in real production as the fabrication of these multifunctional LoCs generally involves complex manufacturing procedures ^[6]. The channel networks need to be specially designed, and the on-chip fabrication techniques are usually highly precise 3D nanotechnologies that are not yet fully developed for market-oriented commercialization. Therefore, realizing multi-functionalization on a single chip remains a big challenge.

Laser scribing technology that converts near-infrared light (NIR, \approx 780 nm) inside a digital versatile disc (DVD) drive into localized heat is a cost-effective and simple tool for flexible patterning of graphene and many composite materials ^[7]. Laser-scribed graphene oxide (GO) has been widely used to produce electronic devices because of its good mechanical properties, high electrical conductivity, and high surface area ^[8]. Thus, laser scribing of GO is promising for fabricating graphene-based chemical sensors, actuators, and various electronics, which may contribute to the development of microfluidics. However, laser scribing of GO has not been fully applied to LoC functionalization.

In this study, a surface-enhanced Raman scattering (SERS)-active catalytic chip was fabricated by laser scribing of GO/silver nanoparticle composites (AgNPs@GO). The surface plasmonic effect of AgNPs promoted the photoreduction of GO, leading to a highly porous nanostructure. The well-exfoliated graphene served as an excellent catalytic scaffold for AgNPs. The dense distribution of AgNPs on the laser-scribed graphene led to a plasmonic structure, making the microfluidic chip SERS-active. Combining a catalyst bed with the SERS substrate enabled on-chip catalysis and in situ SERS monitoring of 4-nitrophenol (4-NP) reduction. Thus, integrating SERS sensors with functional microfluidic devices holds great promise for developing advanced LoC systems in the future.

METHODS

The composites AgNPs@GO-1–3 were prepared by UV photoreduction. Briefly, GO (10 mL, 4 mg mL⁻¹) was mixed with different amounts (5, 10, and 15 mL) of Ag(NH₃)₂OH, and the resulting solution was irradiated under UV-light (500 W) for 10 minutes. The final composites were obtained after washing with deionized water.

Each AgNPs@GO composite (20 mL) was cast onto a LightScribe-enabled disc to obtain a homogenous film. The disc was then inserted into a laser-scribe-enabled DVD drive equipped with an NIR laser (780 nm), and predesigned patterns of channel networks were directly "written" onto the composite film. Each AgNPs@GO composite was reduced to AgNPs@ RGO (RGO, reduced GO) during laser scribing. Laser-scribed AgNPs@RGO was selectively transferred to a polydimethylsiloxane (PDMS) substrate by simple stamping. A PDMS microfluidic layer with conformal channel networks was then combined with the SERS-active PDMS substrate, and the two PDMS layers were sealed together to create an SERS-active catalytic chip.

For SERS detection, the laser power on the samples was adjusted to \approx 30 µW, and the average spot size was 1 µm in diameter using a long-working distance 50 × objective. SERS spectra were recorded on an instrument equipped with a liquid-nitrogen-cooled argon-ion laser at 532 nm using an exposure time of 5 seconds and three accumulations.

The composites were characterized by scanning electron microscopy (SEM), high-resolution transmission electron microscopy (HRTEM), and X-ray photoelectron spectroscopy (XPS). Confocal laser scanning microscopy (CLSM) images were obtained with an Olympus[®] LEXT^M OLS4100 3D measuring laser microscope.

RESULTS AND DISCUSSION

Figure 1a shows the fabrication procedure of the catalytic chip and in situ integration of the SERS sensors. Due to the photothermal effect of GO and the plasmonic effect of AgNPs, GO was effectively reduced (AgNPs@RGO) by laser irradiation. Various complex channels were patterned on the composite (Figure 1b). After laser irradiation, the compact AgNPs@GO film was exfoliated into a highly porous structure so that the resulting patterned AgNPs@ RGO could be easily transferred onto a PDMS substrate by simple stamping. The PDMS substrate was then combined with another conformal channel layer, and a flexible catalytic chip with in situ SERS sensors was fabricated (Figure 1c). The chip was integrated with a catalyst bed capable of SERS monitoring.

The morphology of the AgNPs@GO film was investigated before and after laser scribing (**Figure 2a**). The original AgNPs@GO film was compact and had a smooth surface, whereas laser-scribed AgNPs@RGO was expanded and exfoliated, providing many active sites for the subsequent catalytic reaction and SERS detection. SEM of the AgNPs@RGO material indicated that AgNPs were uniformly distributed on the wrinkled RGO sheet (**Figure 2b**). The average size of AgNPs was ≈10 nm, and the gap between particles was ≈2 nm. AgNPs with



Figure 1: *In situ* integration of SERS sensors on a catalytic chip. (a) Fabrication scheme of a catalytic chip by laser scribing. (b) Various computer-designed patterns written on an AgNPs@GO-coated disc. (c) Photograph of the catalytic chip.



Figure 2: Surface morphologies of AgNPs@RGO films. (a) SEM image of a laser-scribed AgNPs@GO film. (b) High-resolution SEM image of an AgNPs@RGO layer. (c,d) SEM and CLSM images of AgNPs@RGO (c) before and (d) after PDMS stamping. Scale bar: 20 µm. (e) The cross-section height of the AgNPs@RGO film before and after PDMS stamping.

diameters smaller than 20 nm are considered attractive catalysts because of their negative redox potential and high surface-to-volume ratio ^[9, 10]. Agglomeration hampers their practical application, but in this study, the exfoliated RGO acted as a scaffold, preventing AgNP aggregation and thus allowing long-term usage of the catalytic chip and SERS sensors.

SEM and CLSM confirmed the expanded structure of AgNPs@RGO (**Figure 2c**). As the lasertreated region could be easily stamped onto the PDMS substrate, any designed pattern was selectively integrated into the microfluidic channels. **Figure 2d** shows the hierarchical structure of the transferred AgNPs@RGO substrate, which retained a loose and exfoliated morphology. A large amount of AgNPs was exposed to the environment, facilitating catalytic reactions and SERS detection. The surface morphology of AgNPs@RGO before and after PDMs stamping was comparable, with an expanded height of ≈10 µm (**Figure 2e**).

To investigate the influence of AgNPs during laser reduction, we varied the AgNPs/GO ratio in the AgNPs@GO composite (AgNPs@GO-1-3). TEM images showed that AgNPs dispersed uniformly on the GO sheets, whereby the distribution density of AgNPs correlated positively with the silver ion concentration. No aggregation was found as the GO scaffold impeded the merging of AgNPs.

Laser-scribing of GO is a photothermal reduction process ^[11], and the addition of AgNPs greatly facilitates GO reduction because of the synergistic effect of the high photothermal conversion efficiency of GO and the remarkable plasmonic effect of AgNPs ^[12]. Pure GO and AgNPs@GO-1-3 were scribed under the same conditions and characterized by SEM. The laser paths were recognizable due to the expanded cracking edge of the graphene layer. The degree of expansion increased with increasing AgNPs/GO ratio, thus, exposing more active sites. AgNPs remained highly dispersed on the RGO scaffold in laser-scribed AgNPs@ RGO-1 and -2. In AgNPs@GO-3, however, the excessive amount of AgNPs, which packed closely on the GO sheet, led to laser-induced coalescence and poor uniformity of AgNPs.

Laser scribing induces not only hierarchical structures but also removes oxygen-containing groups (OCGs) on the GO layer. XPS spectra of C 1s peaks were collected to compare the amount of OCGs before and after laser-scribing. The spectra resolved into three peaks corresponding to C–C, C–O, and C=O. The C–O and C=O peaks of the composites decreased dramatically after laser scribing compared with those of pure GO. The C–O and C=O peaks also decreased with increasing AgNP content in the composites, indicating that AgNPs play a role in the photoreduction of GO. The laser intensity could also control the morphology of the laser-scribed composites. Thus, simple laser scribing can regulate the OCG content and achieve a porous structure of AgNPs@RGO, which are crucial for performing catalytic reactions and SERS detection.

A functional microfluidic chip was formed (Figure 1a), and the closely packed AgNPs endowed the chip with catalytic capability. RGO and AgNPs@RGO-1-3 were transferred into different channels of the chip (Figure 3a). The reduction of 4-NP by sodium borohydride (NaBH₄) was chosen as a classic reaction to evaluate the catalytic activity of AgNPs@ RGO ^[13]. 4-NP (6 mM) and NaBH₄ (20 M) were injected into different inlets of the Y-shaped channels. RGO-based materials can trap 4-NP molecules via noncovalent π - π stacking interaction ^[14]. Reduction of 4-NP occurred only at the AgNPs@RGO region, whereby the fluid changed its color from light yellow to colorless. UV-vis spectroscopy was used to assess the reduction efficiency of the chip as 4-NP and its reduction product 4-aminophenol (4-AP) produce characteristic absorption peaks at 400 and 295 nm, respectively. The conversion of 4-NP to 4-AP depended on the fluid velocity on the chip (Figure 3b,c). In the channel containing AgNPs@RGO-1, 4-NP was completely reduced at a liquid flow velocity of <1 mL h⁻¹. This critical flow rate was defined as the threshold velocity (v_{th}). In the channel containing AgNPs@RGO-2, complete conversion of 4-NP occurred at a much higher v_{th} (5 mL h^{-1}). The difference in catalytic activity between AgNPs@RGO-1 and -2 was due to the different contents of AgNPs and exposed active sites.

We used a pseudo-first-order kinetic model to investigate the catalytic performance on different substrates quantitatively ^[15]:

$\ln(C/C0) = \ln(A/A_0) = -k_{\rm app}t$

where C and C_0 are the real-time and initial concentration of 4-NP, respectively, A and A_0 the corresponding absorbance intensities, and k_{app} the apparent first-order reaction rate constant. Accordingly, k_{app} was determined to be 0.0054, 0.0207, and 0.0109 s⁻¹ for AgNPs@RGO-1, -2, and -3, respectively, indi-

cating that AgNPs@RGO-2 exhibited the highest catalytic activity (**Figure 3d**). This result implies that laser scribing can improve the catalytic capability of AgNPs@RGO. However, the excessive amount of AgNPs in AgNPs@ RGO-3 led to the coalescence of AgNPs during laser scribing and thus to a dramatically reduced reaction rate. Therefore, microfluidic channels containing AgNPs@RGO-2 were selected to produce practical chips.

The closely packed AgNPs on the RGO scaffold can also be used as efficient SERS sensors ^[16], enabling on-chip, in situ SERS detection to directly monitor the catalytic reaction. We compared the SERS spectra of 4-NP on different substrates. As the SERS signals are attributed to "hot spots" at the nanogaps between neighboring nanoparticles, the electric field and Raman signals were significantly enhanced on the AgNPs@RGO-1 and -2 substrates. The signal from the AgNPs@RGO-2 substrate exhibited the greatest enhancement owing to the plasmonic effect of AgNPs and the effective interaction between 4-NP and RGO.

In practice, complete conversion of the reactant is desired; therefore, we acquired the real-time SERS signals at the end of the channel to determine whether 4-NP has been completely reduced. At a flow rate lower than v_{th} , 4-NP was almost completely converted to 4-AP. However, at a velocity higher than v_{th} , the product was a mixture of 4-NP, 4-AP, and NaBH₄. These results indicate that catalytic chips with *in situ* SERS sensors can effectively avoid the intricate collection of the products and subsequent UV-vis measurements.

To evaluate the reproducibility of the SERS device, we collected SERS spectra of rhodamine 6G (R6G, 10⁻⁷ M) at nine randomly selected positions on the SERS sensor. Consistent signal intensities were obtained, indicating excellent reproducibility. Moreover, the SERS intensities at 1509 cm⁻¹ were compared, and the relative standard deviation (RSD) was ≈2.3%. To test the error of concentration detection, we measured the SERS signals of R6G at different concentrations $(10^{-5}-10^{-10} \text{ M})$; the SERS intensity increased evidently with concentration. The SERS chip is also reusable. After the first SERS detection of R6G, the SERS device was regenerated with water and reused for three further detection and regeneration cycles. The signal intensity at 773 cm⁻¹ was measured after each cycle, and the RSD was ≈6.5%, demonstrating good reusability. Furthermore, the SERS signals of devices stored for 15, 30, 45, and 60



Figure 3: (a) Schematic illustration of the catalytic chip. (b,c) UV-vis absorption spectra of products obtained at various flow rates from (b) AgNPs@RGO-1 and (c) -2. (d) Real-time changes in $\ln(C/C_0)$ for the reduction reaction on different substrates.

days were measured and compared to study the shelf life of the sensor. The SERS intensity of the device stored for 60 days decreased only by 10% compared with that of a fresh device, indicating a shelf life of at least 60 days.

CONCLUSION

In summary, a catalytic chip with in situ SERS sensors was prepared by simple laser scribing. By adjusting the AgNPs/GO ratio, AgNPs@ RGO substrates with designable structures and optimized performance were obtained. The hierarchical structures of the composites exposed numerous active sites to the environment, which contributed to the reduction of 4-NP. Due to the excellent adsorption properties of RGO and the remarkable plasmonic effect of AqNPs, the microfluidic substrates exhibit SERS sensor capability. Thus, the catalytic products could be monitored by in situ SERS detection. This cost-effective strategy for fabricating catalytic optofluidic chips is favorable for future practical applications.

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04 Mechanical Properties and Slurry Erosion Resistance of a Hybrid Composite SiC Foam/SiC Particles/EP

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ABSTRACT

The novel hybrid composite SiC foam/SiC particles/EP (SiCfoam/SiC_p/EP) was prepared, consisting of an E-51 epoxy resin (EP) as the matrix and SiC foam/SiC particles as the reinforcements. The slurry erosion properties of the SiCfoam/SiC_p/EP and SiC_{foam}/EP (SiC_{foam}/epoxy resin) composites were investigated using a rotating disc rig. SiC_{foam}/SiC_p/EP exhibited better mechanical and anti-erosive performances than SiC_{foam}/EP. The SiC particle size had a great impact on the mechanical and anti-erosive properties of the hybrid composites. Smaller SiC particles improved the mechanical strength but tended to decrease the anti-erosive performance of the composite. The hybrid composite SiC_{foam}/ SiC_p/EP can be applied under solid particle erosion conditions because of its excellent anti-erosive properties.

INTRODUCTION

Solid particle erosion, a typical dynamic wear mode, results from the repeated impact of small, solid particles ^[1]. It occurs in various engineering fields, such as automotive, aerospace, marine, and energetics. Polymers and polymer composites are widely used in engineering fields because of their high specific strength and stiffness ^[2, 3]. The erosive resistance of various polymer composites has extensively been investigated, including fiber-reinforced or particle-reinforced composites and hybrid polymer composites. Fiber/matrix adhesion and fiber orientation strongly affect the erosive resistance of fiber-reinforced composites, which show a semi-ductile erosion behavior ^[4, 5]. In particle-reinforced composites, the particle fillers have a significant effect on the erosive performance of the composite ^[6, 7]. The incorporation of particle fillers into fiber-reinforced polymer composites improves the material's erosive resistance. However, the erosive performance of ceramic foam-reinforced polymer composites is poorly studied even though ceramic foams have attracted much interest in the past decade. Ceramic foams, with their unique 3D network architecture and outstanding mechanical and anti-erosive properties, are excellent reinforcements in composite materials [8-11]. The interpenetrating 3D structure of the ceramic foam reinforcement greatly enhances the mechanical and physical performance of composites. For example, the incorporation of SiC foam into SiC particle-reinforced aluminum composites markedly decreased the composite's thermal expansion because of the interpenetrating structure of the SiC foam [8]. Furthermore, co-continuous ceramic foam/aluminum composites were up to twice as wear-resistant as metal-matrix composites made of ceramic powder/fibers, indicating that ceramic foams are more efficient than ceramic powder/fibers as reinforcements in aluminum alloys^[9]. We previously showed that a SiC foam/epoxy co-continuous phase composite (SiC_{foam}/EP) exhibits better slurry erosion resistance than the epoxy matrix alone and the widely used pipeline steel, indicating that SiC_{foam}/EP composites can be applied under solid particle erosion conditions ^[11]. The reinforcing effect of SiC foam on the composite's slurry erosion resistance is greater than that of dispersed SiC particles. However, erosion of the epoxy matrix to a certain degree is inevitable, and increasing the volume fraction of SiC foams to enhance the erosive resistance is an inefficient approach because the preparation of SiC foams at high volume fractions is difficult. Therefore, a hybrid composite in which the reinforcing effects of two reinforcements synergize each other is a good choice for improving the erosive resistance.

This article proposes a hybrid SiC foam/SiC particles/epoxy (SiC_{foam}/SiC_p/EP) composite that can be applied under solid particle erosion conditions. SiC_{foam}/SiC_p/EP exhibited not only improved mechanical properties but also desirable slurry erosion resistance. The mechanical properties and slurry erosion resistance of the SiC_{foam}/EP composite and hybrid composites containing SiC particles of different sizes were systematically compared. The slurry erosion mechanism of the SiC_{foam}/SiC_p/EP composite different sizes was also investigated.

METHODS

The SiC_{foam}/SiC_p/EP composite was prepared by a modified procedure previously reported ^[11]. The SiC foam volume fraction was 60%. The SiC foam had a cell size of ~1.5 mm, and the SiC particles had grain sizes of 0.74, 0.41, and 0.1 mm in SiC_p I, SiC_p II, and SiC_p III, respectively. SiC foam, consisting of α -SiC, β -SiC, and Si, was produced by a solid-state sintering process. SiC particles were treated with the silane-coupling agent KH-550 to modify the adhesion between the epoxy matrix and SiC particles. The SiC particles were then dispersed into SiC foam cells to obtain SiC_{foam}/SiC_p. The SiC_p volume fraction in the three hybrid composites was ~25%. The epoxy resin was then squeezed into SiC_{foam}/SiC_p by vacuum infiltration and compression molding. The uncured sample was kept in a vulcanization machine at 100 °C (212 °F) and 10 MPa for 4 hours to obtain a completely cured epoxy matrix.

For the compression and three-point bend experiments, the samples were tested at a constant displacement rate of 0.5 mm/min with a load capacity of 100 kN. The impact tests were conducted in a pendulum impact-testing machine with a load capacity of 0.5 J.

Slurry erosion experiments were performed in a rotating disc rig. The slurry consisted of water and erodent quartz sand with a grain size of 70–150 mesh. The sand content was 10 kg/m³, and the flow velocity was kept at 10 m/s. The sample surfaces were ground with a 1000-grit abrasive paper and then cleaned in deionized water and dried before the tests. The slurry erosion of each sample was characterized by the erosion rate (*E*): $E = (m_0 - m)/A$ /*t*, where m_0 , *m*, *A*, and *t* represent the initial mass, the mass after the test, the surface area of the sample, and test duration, respectively.

The structures of the SiC_{foam}/SiC_p/EP composites were analyzed using an Olympus[®] SZX16 stereoscopic microscope. The fracture morphologies and microstructures of the composites after the mechanical and slurry erosion tests, respectively, were observed with a scanning electron microscope (SEM) and an Olympus LEXT[™] OLS4000 confocal laser scanning microscope (CLSM).

RESULTS AND DISCUSSION

The cured SiC_{foam}/SiC_p/EP hybrid composites showed no voids and piles, and the epoxy matrix/SiC particles and SiC foam interpenetrated mutually. The SiC particles and epoxy matrix were trapped within the SiC foam cells of the interpenetrating composite structures.

The SiC_{foam}/SiC_p/EP composites exhibited better mechanical properties (i.e., compressive strength, flexural strength, and impact tough-



Figure 1: Flexural fracture morphologies of the four composites. (a) SiC_{foam}/EP, (b) SiC_{foam}/SiC_p I/EP, (c) SiC_{foam}/SiC_p II/EP, (d) SiC_{foam}/SiC_p II/EP).

ness) than the ${\rm SiC}_{\rm foam}/{\rm EP}$ composite. Among the SiC_{foam}/SiC_p/EP composites, SiC_{foam}/SiC_p III/ EP performed best as the mechanical properties of the hybrid composites increased with decreasing SiC particle size. Therefore, the grain size of the SiC particles plays a pronounced role in the mechanical properties of the SiC_{foam}/SiC_p/EP composites. Reinforcing the epoxy resin by SiC particles improves its mechanical properties and poor resistance to crack growth ^[12, 13]. Therefore, the SiC_{foam}/ SiC_p/EP composites exhibited better mechanical properties than the SiC_{foam}/EP composite. Figure 1 shows the flexural fracture morphologies of the four composites. The failure mechanism of the hybrid composites included the fracture and detachment of SiC particles. Cracks propagated more tortuously in the hybrid composites than in the ${\rm SiC}_{\rm foam}\!/{\rm EP}$ composite, and the grain size of the SiC particles affected the morphologies markedly. CLSM images of the flexural fractures in SiC foam cells show that the fracture surface of the SiC_{foam}/EP composite was smoother than that of the SiC_{foam}/SiC_p/EP composites (Figure 2). These observations indicate

that more fracture energy is absorbed during $SiC_{foam}/SiC_p/EP$ fracture than during SiC_{foam}/EP fracture. The roughness of the fracture surface and fracture energy increased gradually with decreasing SiC particle size. Smaller particles have a larger total surface, and the strength of particle-reinforced composites increases with the particle surface area ^[14]. Therefore, the mechanical properties of the hybrid composites increased with decreasing grain size.

The SiC_{foam}/SiC/EP composites showed lower erosion rates than the SiC_{foam}/EP composite. The slurry erosion resistance of SiC_{foam}/ SiC_p I/EP, SiC_{foam}/SiC_p II/EP, and SiC_{foam}/SiC_p III/ EP increased by 190%, 94%, and 2%, respectively, compared with that of SiC_{foam}/EP. SEM images of the eroded surfaces of all composites were taken to investigate the erosion mechanism. Severe breaking of SiC_p II particles was observed in SiC_{foam}/SiC_p II/EP, and plenty of SiC_p III particles were detached from the epoxy matrix in SiC_{foam}/SiC_p III/EP (**Figure 3**). Nevertheless, the SiC particles in the hybrid composites were less seriously dam-



Figure 2: CLSM images of the flexural fractures in the SiC foam cells of the four composites. (a) SiC_{foam}/EP, (b) SiC_{foam}/SiC_p I/EP, (c) SiC_{foam}/SiC_p II/EP, (d) SiC_{foam}/SiC_p III/EP).



Figure 3: SEM images of eroded composites. (a) SiC_{foam}/EP , (b) SiC_{foam}/SiC_p I/EP, (c) SiC_{foam}/SiC_p II/EP, (d) SiC_{foam}/SiC_p III/EP).

aged than those in SiC_p/EP, which can be attributed to the shadowing effect of the SiC foams. The shadowing effect decreases the impact energy of quartz sand on SiC particles. Quartz sand with small grain sizes selectively scoops out the epoxy matrix in particle-reinforced, epoxy-based composites ^[15]. However, no scooping of the epoxy resin was observed in the hybrid composites because of the synergistic effect of the SiC foam and SiC particles. Therefore, the hybrid composites eroded to a lesser extent than SiC_{foam}/EP. The SiC foam and SiC particles greatly improved the anti-erosive properties of the epoxy matrix, indicating that SiC_{foam}/SiC_p/EP composites behave completely different from traditional particle-/fiber-reinforced polymer composites under solid particle erosion conditions.

Figure 4 shows sketches of the slurry erosion mechanism for the four composites. During slurry erosion, local removal of EP from the impacted surface of the sample results in the exposure and extrusion of the SiC foam and SiC particles. After 24 hours, the angle (α) between the damaged and initial surface of the SiC_{foam}/EP composite approximately reflects the impact angle of quartz sand (Figure 4a). Less EP is eroded and complex surface morphologies are formed during the slurry erosion of hybrid composites because of the synergistic effect of the SiC foam and SiC particles. The severe breaking of SiC_p II and detachment of SiC_p III occur with successive impact of quartz sand, which inevitably leads to further erosion of the EP after the removal of SiC particles (Figure 4b). The erosion of a material depends on the energy exchange between the erodent particle and the impacted material surface. When the particles striking against the target are softer than the target material, fractures (due to individual impacts) and plastic indentation may not occur ^[9]. Since quartz sand is softer than SiC, the plastic deformation of SiC is negligible during the collision of quartz sand with the sample surface. This means that most of the kinetic energy is transformed into rebounding energy while the rest dissipates through stress wave propagation.

Because of its unique structure, SiC foam dissipates the impact energy of moving quartz sand, whereas SiC particles absorb and dissipate the kinetic energy to the surrounding epoxy resin. Therefore, quartz sand with enough impact energy will inevitably break SiC particles (**Figure 3c**). The contact area and consequently adhesion between SiC particles and EP increase with SiC particle size.





Therefore, SiC_{foam}/SiC_p I/EP exhibited the best anti-erosive properties, without detachment of SiC_p I particles. In contrast, because of the weak adhesion in SiC_{foam}/SiC_p III/EP, the impact energy of moving quartz sand was high enough to detach SiC_p III particles from the composite. Consequently, SiC_{foam}/SiC_p III/EP showed weaker erosion resistance than the other two hybrid composites.

CONCLUSION

A novel hybrid composite, SiC_{foam}/SiC_p/EP, was successfully prepared. The four composites SiC_{foam}/EP, SiC_{foam}/SiC_p I/EP, SiC_{foam}/SiC_p I/EP, and

SiC_{foam}/SiC_p III/EP exhibited better mechanical properties than the SiC_{foam}/EP composite because of the reinforcement of the epoxy resin with SiC particles. The grain size of SiC particles had a marked effect on the mechanical properties of the SiC_{foam}/SiC_p/EP composites, which increased with decreasing particle size. The SiC_{foam}/SiC_p/EP composites also had better anti-erosive properties than the SiC_{foam}/EP composite. Among the tested hybrid composites, SiC_{foam}/SiC_p III/EP exhibited the highest erosion rate, leading to the detachment of SiC_p III particles from the epoxy resin. The SiC_{foam}/SiC_p/EP hybrid composite can be applied under solid particle erosion conditions because of its excellent mechanical and anti-erosive properties.

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