Indentation fracture toughness of Aluminium-Graphite composites: influence of nano-particles

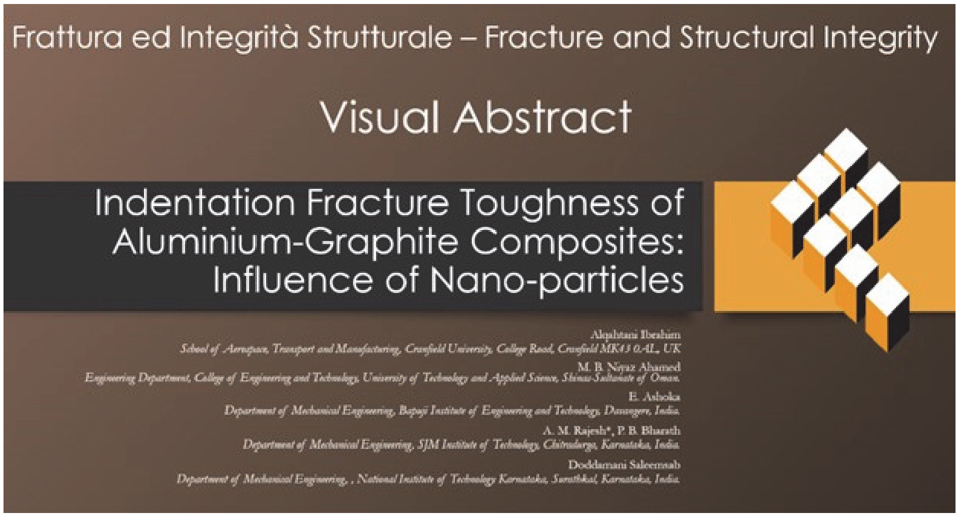
Alqahtani Ibrahim
School of Aerospace, Transport and Manufacturing, Cranfield University, College Road, Cranfield MK43 0AL, UK
zafer.268152@gmail.com

B. Niyaz Ahmed
Engineering Department, College of Engineering and Technology, University of Technology and Applied Science, Shinas-Sultanate of Oman.
Niyaz.budensab@utas.edu.om

E. Ashoka
Department of Mechanical Engineering, Bapuji Institute of Engineering and Technology, Davangere, India.
ashokamech06@gmail.com, http://orcid.org/0000-0002-3062-5883

A. M. Rajesh*, P. B. Bharath
Department of Mechanical Engineering, S.J.M. Institute of Technology, Chitradurga, Karnataka, India.
rajesh.am82@gmail.com; https://orcid.org/0000-0003-1843-239X
bharathauto999@gmail.com; https://orcid.org/ 0000-0001-5688-7323

Doddamani Saleemsab
Department of Mechanical Engineering, National Institute of Technology Karnataka, Surathkal, Karnataka, India.
saleemsabdoddamani@gmail.com; http://orcid.org/0000-0002-8498-1488



Frattura ed Integrità Strutturale – Fracture and Structural Integrity

Visual Abstract

Indentation Fracture Toughness of
Aluminium-Graphite Composites:
Influence of Nano-particles

Alqahtani Ibrahim
School of Aerospace, Transport and Manufacturing, Cranfield University, College Road, Cranfield MK43 0AL, UK
M. B. Niyaz Ahamed
Engineering Department, College of Engineering and Technology, University of Technology and Applied Science, Shinas-Sultanate of Oman.
E. Ashoka
Department of Mechanical Engineering, Bapuji Institute of Engineering and Technology, Davangere, India.
A. M. Rajesh*, P. B. Bharath
Department of Mechanical Engineering, SJM Institute of Technology, Chitradurga, Karnataka, India.
Doddamani Saleemsab
Department of Mechanical Engineering, , National Institute of Technology Karnataka, Surathkal, Karnataka, India.

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INTRODUCTION

Composite materials have emerged as key contenders in modern engineering and materials science, offering a unique combination of properties that can be tailored to meet specific performance requirements [1]. Among these, aluminum-graphite composites have garnered significant attention due to their potential to provide a remarkable balance between lightweight properties and mechanical strength [2]. This balance is particularly appealing to industries such as aerospace, automotive, and structural engineering, where the quest for materials that can enhance efficiency without compromising safety is perpetual [3].

Aluminum-graphite composites are characterized by the incorporation of graphite particles into an aluminum matrix. These graphite particles, traditionally in micron-scale dimensions, serve as reinforcing agents, imparting increased stiffness and strength to the composite material [4]. However, recent advancements in materials science have led to the exploration of nano-sized graphite particles as potential reinforcements in these composites [5]. Nano-sized particles, with dimensions typically in the nanometer range, offer distinct advantages over their larger counterparts, primarily due to their high surface area-to-volume ratio and unique mechanical properties [6].

Despite the extensive research into aluminum-graphite composites [2,7–11], there remains a critical research gap concerning the influence of nano-sized graphite particles on the fracture toughness of these materials. While previous investigations have examined the effects of larger (micro) graphite particles or different types of reinforcement materials, the specific mechanisms and impacts of nano-graphite particles on fracture toughness remain relatively unexplored. This knowledge gap is a pressing concern, given the potential of nano-sized graphite particles to revolutionize the design and application of aluminum-graphite nanocomposites.

Understanding the precise influence of nano-sized graphite particles on fracture toughness is vital for several reasons. Firstly, it can pave the way for the development of advanced composite materials that offer superior fracture resistance, making them well-suited for applications where structural integrity and safety are paramount. Secondly, such insights can lead to the optimization of manufacturing processes for these composites, ensuring consistent and predictable material behaviour. Lastly, by elucidating the fracture mechanisms at the nano-level, this research can contribute to the broader understanding of composite materials and their potential in addressing contemporary engineering challenges.

This study sets out to address this research gap by systematically investigating how the incorporation of nano-sized graphite particles affects the indentation fracture toughness of aluminum-graphite nanocomposites. The research encompasses a range of experimental and analytical techniques, including particle dispersion and characterization, hardness and indentation fracture toughness testing, and fractographic analysis. Through this comprehensive approach, it is aimed to shed light on the specific mechanisms through which nano-graphite particles enhance fracture toughness and contribute to the development of advanced composite materials with exceptional fracture toughness.

MATERIALS AND METHODS

Materials

The materials selected for this study encompass both the matrix and the reinforcing phase, with meticulous attention to their properties and preparation.

Matrix material: Al6061

Aluminum 6061 (Al6061) is a widely used aluminum alloy renowned for its excellent combination of strength, ductility, and corrosion resistance [12]. It is a popular choice for aerospace, automotive, and structural applications due to its favorable strength-to-weight ratio. Al6061 is primarily composed of aluminum, magnesium (0.81), and silicon (0.72), with trace amounts of other alloying elements. The density of Al6061 is approximately 2.70 g/cm³. Al6061 exhibits a typical yield strength of about 276 MPa. The ultimate tensile strength of Al6061 is around 310 MPa [13,14]. The fracture toughness of Al6061 is a critical parameter, and it can be influenced by factors such as grain size and microstructure.

Reinforcement material: graphite nano particles

The reinforcing phase in this study consists of nano-sized graphite particles with an average particle size of 100 nanometers (nm). These nano graphite particles were selected for their potential to enhance the mechanical properties of the composite material. The nano graphite particles have an average diameter of 100 nm, falling within the nanoscale range. Nano-sized particles exhibit a high surface area-to-volume ratio, which can promote strong interfacial bonding with the matrix material. These particles retain the graphitic structure inherent to graphite, which can contribute to mechanical reinforcement.

Ultrasonic-assisted stir casting process

The aluminum-graphite nanocomposites for testing have been created using a meticulously controlled ultrasonic-assisted stir casting process, shown in Fig. 1 [15]. In this technique, subsequent to thorough reinforcement mixing within the molten metal, at 720°C and 500rpm, the stirrer was substituted with an ultrasonic probe. A high-power ultrasonic vibration device, facilitated by the transducer, was deployed to induce ultrasonic treatment within the molten metal. Throughout this process, the operating temperature was meticulously controlled at 630°C. The application of ultrasonic vibration during casting serves to disrupt the dendritic structure and uniformly disperse the particles within the casting medium. The ultrasonic-assisted component of this procedure employs high-frequency vibrations to optimize the blending and even distribution of the nano graphite particles throughout the matrix. This advanced method ensures the uniform dispersion and integration of nano-sized graphite particles within the Al6061 matrix. Consequently, a homogenous composite material characterized by enhanced bonding between the particles and the matrix is achieved. The casting method has been iteratively replicated for varying weight percentages (1, 2, and 3wt%) of the reinforcement material.

Figure 1: Ultrasonic-assisted stir casting setup

Heat treatment

The specimens were treated with heat before testing to study the impact of age hardening on fracture toughness of Al6061-graphite nanocomposites. The specimens were heated in a muffle furnace at a temperature of 460°C to promote precipitation hardening, a commonly used technique to enhance the strength and hardness of aluminium alloys [16,17]. The specimens were held at this temperature for 2 hours to allow complete precipitation.

Specimen preparation

Circular specimens were precisely machined with a diameter measuring 10mm and a thickness of 3mm as shown in Fig. 1. The wire cut EDM has been used with wire diameter 0.4 mm [18]. The prepared samples were further surface grinded to obtain the precision surface.

Experimentation

The Tab. 1 outlines the design of experiments (DOE) for the process parameters and their respective levels investigated in the study. Three parameters are considered: Composition (weight percentage of graphite), load applied during Vickers' indentation, and holding time (indicating the duration for which the indenter is kept in position during the indentation process) [19].

S.N	Parameters	Level 1	Level 2	Level 3
1	Composition (wt% of Gr)	1	2	3
2	Load (kg)	10	20	30
3	Holding Time (s)	5	10	15

Table 1: DOE of process parameters and their levels.

For Composition, the levels correspond to 1%, 2%, and 3% weight percentage of graphite. Load levels are represented by 10kg, 20kg, and 30kg, while Holding Time levels are set at 5 seconds, 10 seconds, and 15 seconds. This DOE matrix allows for a systematic investigation of the effects of these parameters on the properties of the aluminum-graphite composites [20]. As per the Taguchi's DOE the Vickers' indentation experiment has been carried out. The Vickers hardness test is a straightforward method that eliminates the need for indenters of specific dimensions [21]. It assesses a material's resistance to plastic deformation under a standardized load. Hardness is determined by the load divided by the indentation's length, making it independent of the indenter's area [22]. This method is widely applicable across various materials, regardless of their hardness [23]. The unit of hardness provided by the test is known as the Vickers hardness number (VHN) . The VHN number is determined by the ratio F/A, where F represents the force applied to the diamond in kilograms-force, and A denotes the surface area of the resulting indentation in square millimeters [19]. This can be approximated by evaluating the sine term to give, Eqn.1 [24,25]:

$$A \approx \frac{a^2}{1.8544} \tag{1}$$

where a is the average length of the diagonal left by the indenter in millimeters. Hence, Eqn.2 [26]:

$$VHN = \frac{1.8544F}{a^2} \tag{2}$$

where F is in kgf and a is in millimeters, VHN is in GPa.

The hardness of the specimens was determined using the Vickers' method, employing loads ranging from 10 to 30 kg and holding times varying between 5 and 15 seconds. This method utilizes a Vickers' indenter characterized by a pyramid shape with an included angle of 136° between its opposite faces [27]. During the testing process, the indenter is pressed into the surface of the specimen under the specified load and for the designated holding time. Subsequently, the indentation impressions left by the indenter are measured, typically the lengths of the diagonals of the indentation, using a microscope or optical measurement system. The Vickers' hardness value is then calculated based on the ratio of the applied load to the surface area of the indentation, providing an accurate measure of the material's resistance to plastic deformation.

The fracture toughness of a material can be determined from the length of indentation cracks (shown in Fig. 2) using Anstis' equation. Initially, the length of the indentation cracks on the material's surface is measured, typically using microscopy or similar techniques. Anstis' equation relates fracture toughness (KIc) to parameters such as the applied load during indentation (F), the Young's modulus (E), Vickers' hardness (VHN) of the material and the crack length (C).

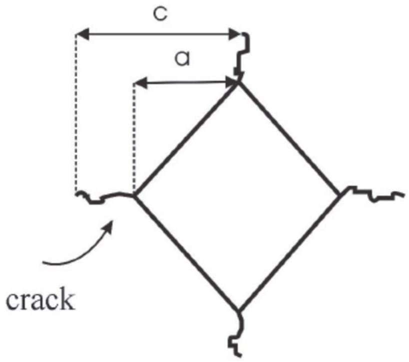


Figure 2: Vickers indentation mark.

From the length of the indentation cracks fracture toughness was calculated by applying the Anstis´ equation, Eqn.3 [26–28]:

$$K_{Ic} = \left(\frac{E}{VHN} \right)^{0.5} \frac{F}{C^{1.5}} \tag{3}$$

where, η – Shape factor (0.016±0.04), E – Young´s modulus (GPa), VHN – Hardness (GPa), F – Indentation load (N), C – Crack length (μm).

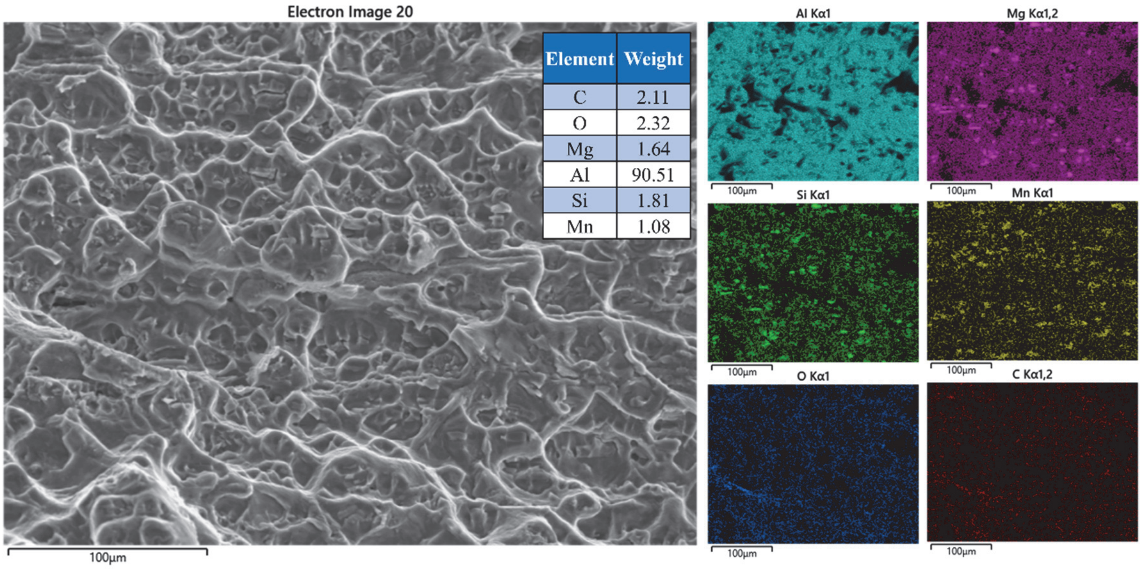
RESULTS AND DISCUSSIONS

Microstructure

Fig. 3 displays the energy dispersive x-ray spectroscopy (EDS) composition and mapping of the Al6061-Graphite nanocomposite. EDS is a technique used to analyze the elemental composition of materials [29]. In this figure, different elements present in the nanocomposite are identified and mapped across the surface of the sample. The composition analysis provides insights into the distribution and concentration of elements within the composite material. By visually representing the elemental mapping [3], the figure allows for a comprehensive understanding of the spatial distribution of aluminum and graphite constituents within the nanocomposite.

Carbon content, indicative of graphite, was measured at 2.11 atomic percent, highlighting the presence of graphite as a reinforcement material contributing to the composite's mechanical properties, such as strength and stiffness. Oxygen content was detected at 2.32 atomic percent, likely originating from surface oxidation or contamination during sample preparation and handling. Additionally, magnesium, an alloying element in Al6061 known for enhancing strength and corrosion resistance, was present at 1.64 atomic percent. Silicon, another common alloying element in aluminum alloys, was measured at 1.81 atomic percent, contributing to improved mechanical properties and corrosion resistance. Furthermore, manganese, detected at 1.08 atomic percent, is often added to aluminum alloys as a deoxidizer and grain refiner, enhancing mechanical properties and casting characteristics.

Fig. 4 presents scanning electron microscope (SEM) images of the Al6061-Gr nano-composites at different weight percentages of graphite reinforcement. These images offer detailed views of the microstructure of the nano-composites, revealing the distribution and morphology of the graphite particles within the aluminum matrix. Notably, the micrographs depict a ductile surface morphology across all compositions of the Al-Gr nano-composites. This ductile surface indicates the ability of the material to undergo plastic deformation and accommodate energy dissipation, which are favorable characteristics in applications where mechanical resilience and toughness are desired. The presence of graphite reinforcement contributes to the enhancement of stiffness and strength in the composite material, as evidenced by the observed microstructure.



Element	Weight
C	2.11
O	2.32
Mg	1.64
Al	90.51
Si	1.81
Mn	1.08

Figure 3: EDS composition and mapping of the Al6061-3%wtGr nanocomposite

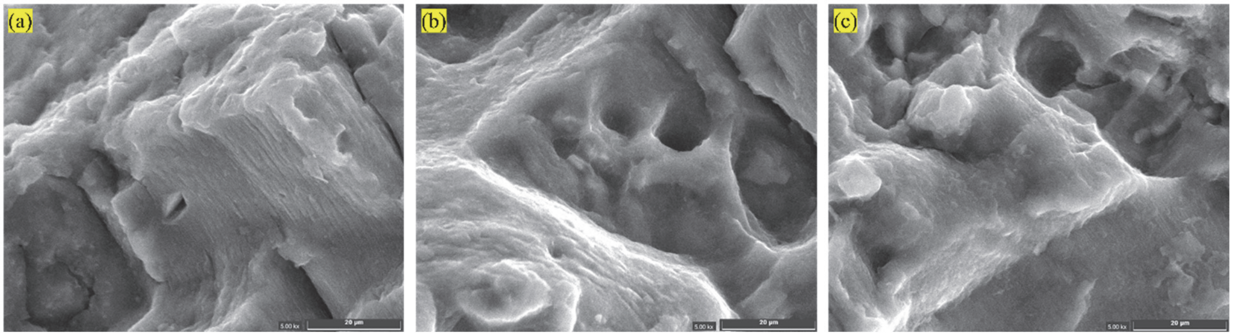


Figure 4: SEM images of Al-Gr nano-composites (a) 1wt%Gr, (b) 2wt%Gr, (c) 3wt%Gr

Vickers' hardness and indentation fracture toughness

The Vickers' hardness experimentation was conducted with the objective of determining the indentation fracture toughness of the specimens. Following indentation, the length of the indentation diagonal was determined using an optical microscope, and the crack length at the end of the diagonal edges was subsequently measured. Utilizing the obtained hardness values and crack length data, the indentation fracture toughness was calculated. Tab. 2 presents the results of the Vickers' hardness and fracture toughness for the various volume fractions of the Al6061-Gr particulate nano-composite. These values were calculated using Eqns. 2 and 3, respectively.

Composition (wt% of Gr)	Load (kg)	Holding Time (sec)	Half Diagonal length (a) mm	VHN (GPa)	Half Crack Length (c) mm	Young's modulus (E) GPa	Fracture toughness (MPa√m)
1	10	5	204	4.46	302.55	67.2	14.48
1	20	10	214.1	8.09	360.75	67.2	16.50
1	30	15	241.9	9.51	442.7	67.2	16.80
2	10	10	215.4	4.00	286.05	67.1	16.62
2	20	15	255.6	5.68	384.6	67.1	17.89
2	30	5	245.4	9.24	407.9	67.1	19.26
3	10	15	238.3	3.27	314.3	66.9	15.94
3	20	5	214.1	8.09	321.6	66.9	19.56
3	30	10	237.3	9.88	386.65	66.9	20.15

Table 2: Experimental Fracture Toughness.

Taguchi's analysis was conducted using an L9 orthogonal array to determine the indentation fracture toughness of the Al6061-Gr nano-composites. The mean effect plot for indentation fracture toughness, obtained from this analysis, is illustrated in Fig. 5. This plot shows the influence of three key parameters: composition, load, and holding time on the indentation fracture toughness.

The plot indicates that increasing the graphite content from 1% to 3% results in a significant increase in fracture toughness. This suggests that higher graphite content, with in the range, contributes positively to the toughness of the composite material. The presence of graphite particles within the aluminum matrix can impede crack propagation by acting as bridges across the crack faces. The graphite particles can deflect cracks, causing them to follow a more tortuous path, which increases the energy required for crack propagation.

The plot demonstrates a marked increase in fracture toughness with increasing load. The fracture toughness improves as the load increases from 10 kg to 30 kg, indicating that higher loads enhance the material's resistance to crack propagation during indentation. When subjected to higher loads, the material's response includes plastic deformation around the indentation area. This plastic deformation absorbs energy, contributing to higher fracture toughness. The ability of the material to deform plastically before fracturing indicates a higher resistance to crack initiation and propagation

The plot shows a different trend for holding time. Initially, the fracture toughness remains relatively constant when the holding time increases from 5 to 10 seconds. However, there is a noticeable decrease in fracture toughness when the holding time is extended to 15 seconds. This suggests that prolonged holding times might negatively affect the fracture toughness of the composite material.

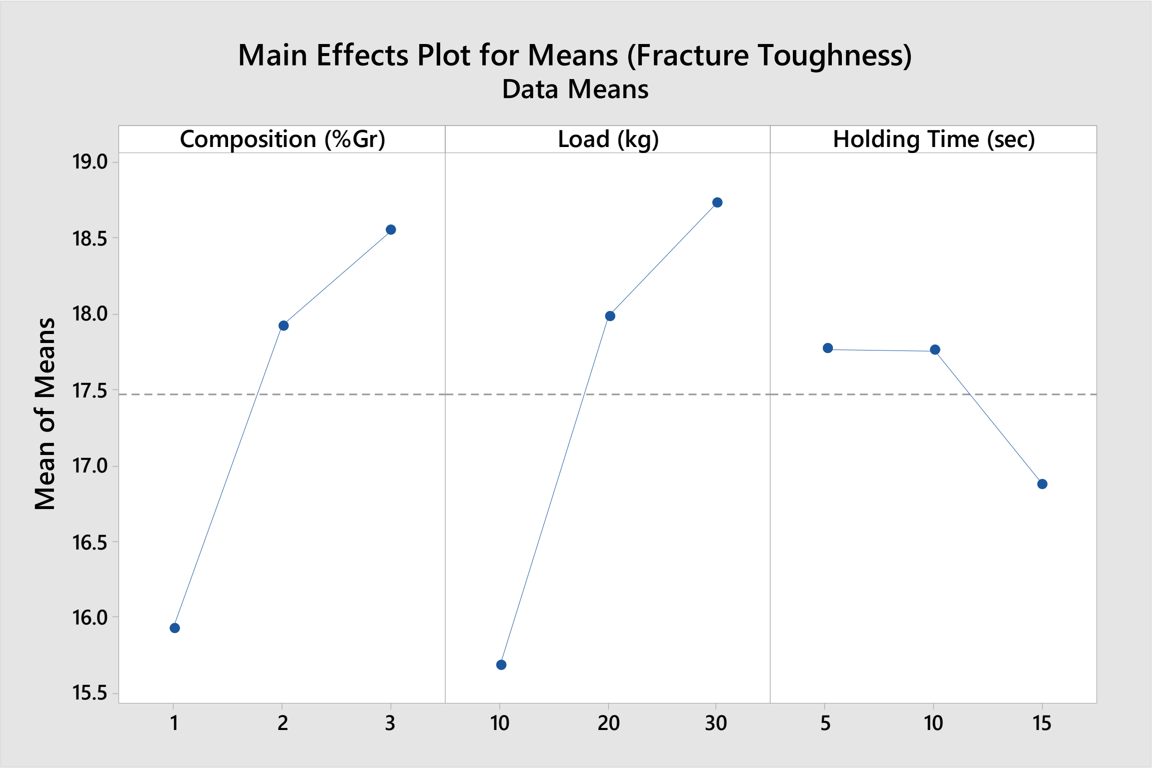


Figure 5: Mean effect plot for indentation fracture toughness.

Tab. 3 presents the analysis of variance (ANOVA) for the indentation fracture toughness of Al6061-Graphite nano-composites. The ANOVA table is used to determine the significance and contribution of each process parameter—composition (weight percentage of graphite), load, and holding time—on the fracture toughness.

Source	DF	Adj SS	Adj MS	F-Value	P-Value	% Contribution
Composition (%Gr)	2	11.2374	5.6187	39.99	0.024	39.7
Load (kg)	2	15.2295	7.6148	54.19	0.018	53.8
Holding Time (sec)	2	01.5714	0.7857	05.59	0.152	05.5
Error	2	00.2810	0.1405			01.0
Total	8	28.3193				100.0

Table 3: Analysis of variance for indentation fracture Toughness.

The probability (P) value indicating the significance level. A lower P-value (< 0.05) suggests that the parameter has a statistically significant effect on the fracture toughness. In this table, composition and load have P-values of 0.024 and 0.018, respectively, indicating they are significant, while holding time has a P-value of 0.152, indicating it is not significant. This means that the holding time does not have a statistically significant effect on the fracture toughness of the Al6061-Graphite nano-composites within the range of values tested.

The percentage of the total variation in fracture toughness explained by each parameter. It shows the relative importance of each parameter in influencing the fracture toughness. Composition of graphite contributes 39.7% to the variation in fracture toughness, with a significant P-value of 0.024, indicating a strong influence. Load has the highest contribution of 53.8%, with a significant a P-value of 0.018, making it the most influential parameter in determining fracture toughness. Holding Time contributes only 5.5% to the variation, with a P-value of 0.152, indicating it has a lesser and statistically insignificant effect on fracture toughness. Error accounts for 1.0% of the total variation, suggesting that the experimental setup and measurements were well-controlled with minimal unexplained variation.

Regression analysis has been performed to quantify the relationship between the indentation fracture toughness (K_{Ic}) and the process parameters: composition (%Gr), load (kg), and holding time (sec). The resulting regression Eqn. (4) provides a mathematical model that predicts the fracture toughness based on the values of these parameters.

$$K_{Ic} = 12.68 + 1.311*\text{Composition (\%Gr)} + 0.1528*\text{Load (kg)} - 0.0892*\text{Holding Time (sec)} \quad (4)$$

Both the graphite composition and the applied load positively influence the fracture toughness. Increasing the graphite content and the load results in higher toughness, suggesting that these parameters are critical in enhancing the composite's performance. The holding time negatively affects the fracture toughness, indicating that longer durations under load can degrade the material's properties, likely due to stress relaxation and other microstructural changes.

Fractographic studies

The fractographic SEM images in Fig. 6 represent the fracture surfaces of Al-Gr nano-composites with varying weight percentages of graphite reinforcement as mentioned. The presence of deep dimples suggests ductile fracture behavior. Dimples observed were characteristic of microvoid coalescence, where voids grow and merge under stress, leading to material failure. The large and deep dimples indicate significant plastic deformation before fracture, which is typical in materials with higher toughness. The appearance of cracks suggests the initiation of failure points within the composite.

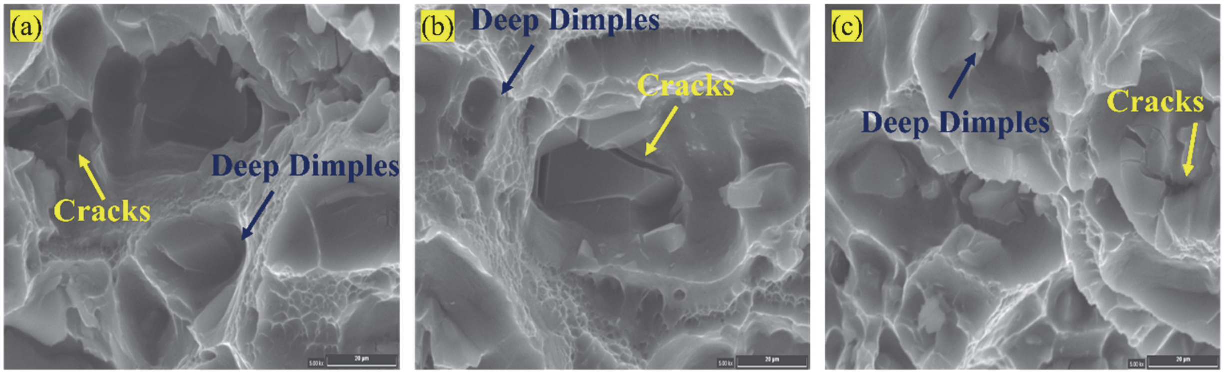


Figure 6: Fractographic images of Al-Gr nano-composites (a) 1wt%Gr, (b) 2wt%Gr, (c) 3wt%Gr

As the graphite content increases from 1 wt% to 3 wt%, the fractographic images show a trend from more ductile to more brittle fracture behavior. The presence of deep dimples across all compositions indicates that ductile mechanisms are present, but the increasing number and prominence of cracks suggest a growing influence of brittle fracture. This is likely due to the role of graphite particles, which, while enhancing certain properties like wear resistance, can also reduce the material's ability to undergo plastic deformation, thus leading to earlier crack initiation and propagation.

Comparison of micro and nanocomposites

The comparison between micro (Tab. 4) and nanocomposites highlights how the reduction in reinforcement particle size to the nanoscale significantly enhances the material's mechanical properties.

Composite	Indentation load (kg)	Holding time (sec)	Fracture toughness (MPa√m)
Al6061+3% Gr	30	10	09.40
Al6061+6% Gr	30	10	09.51
Al6061+9% Gr	30	10	10.15

Table 4: Indentation fracture toughness of Al6061-graphite micro-particulate composites [19].

The comparison (Tab. 2 and 4) reveals a significant enhancement in fracture toughness when nano-sized graphite particles are used as reinforcement. For micro-composites, the fracture toughness values range from 9.40 MPa√m to 10.15 MPa√m, showing a relatively modest improvement as the graphite content increases. In contrast, the nano-composites exhibit much higher fracture toughness values, ranging from 16.50 MPa√m to 20.15 MPa√m, with the highest value observed at 3% nano-Gr. This substantial increase in toughness highlights the effectiveness of nano-sized reinforcement. Interestingly, despite the micro-composites containing a higher graphite content (3%, 6%, and 9%) compared to the nano-composites (1%, 2%, and 3%), the nano-composites demonstrate significantly greater toughness. The consistent indentation load and holding time across both sets of composites ensure that these differences in toughness are primarily attributable to the size and content of the graphite reinforcement.

Composite	Indentation load (kg)	Holding time (sec)	Fracture toughness (MPa√m)
Al6061+3% Gr Micro-composite	30	10	09.40
Al6061+3% Gr Nano-composite	30	10	20.15

Table 5: Comparison of fracture toughness between micro- and nano-composites with 3% graphite reinforcement

Comparison of fracture toughness in micro- and nano-composites with 3% graphite reinforcement has been given in Tab. 5. The nano-composite with 3% nano-graphite shows a significantly higher fracture toughness (20.15 MPa√m) compared to the micro-composite with the same graphite percentage (9.40 MPa√m). Both composites were tested under the same conditions, with an indentation load of 30 kg and a holding time of 10 seconds, yet the nano-composite outperforms the micro-composite in terms of fracture toughness. This indicates that the size of the graphite particles plays a crucial role in enhancing the mechanical properties of the composite.

CONCLUSIONS

Based on the results obtained, the following conclusions were drawn:

- The addition of nano-sized graphite particles significantly enhances the fracture toughness of Al6061 composites. The results demonstrated a clear correlation between the increasing weight percentage of graphite and the improvement in fracture toughness. Higher graphite content, within the range of 1% to 3%, contributes positively to the composite material's ability to resist crack propagation by promoting crack deflection and increased plastic deformation.
- The applied load during indentation testing was found to be a critical factor in determining the fracture toughness. The fracture toughness increased with higher loads, indicating that greater loads enhance the material’s resistance to crack initiation and propagation. The plastic deformation around the indentation area under higher loads absorbed more energy, leading to improved fracture toughness.
- While the holding time showed an initial increase in fracture toughness from 5 to 10 seconds, a further increase to 15 seconds resulted in a decrease in toughness. This suggests that prolonged holding times may negatively affect the composite material's fracture toughness, potentially due to stress relaxation or other time-dependent phenomena.
- The analysis of variance (ANOVA) identified graphite composition and applied load as significant factors affecting fracture toughness, with contributions of 39.7% and 53.8% to the variation, respectively. Holding time had a minimal and statistically insignificant effect, contributing just 5.5%. A regression model confirmed that both graphite composition and applied load positively impacted fracture toughness, while holding time had a minor negative effect.

The findings of this study have important implications for the development of advanced aluminum-graphite nanocomposites with enhanced mechanical properties. The significant improvement in fracture toughness with the addition of nano-sized graphite particles makes these composites highly suitable for applications in aerospace, automotive, and structural engineering, where lightweight materials with high fracture resistance are crucial.

Further research is recommended to explore the long-term durability and environmental stability of these composites, as well as the effects of varying the size and type of reinforcement particles on other mechanical properties such as fatigue and wear resistance.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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