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INFLUENCE OF GRAPHENE CONTENT ON MICROSTRUCTURAL REFINEMENT AND MECHANICAL PROPERTIES OF AA6063/SiC NANOCOMPOSITES

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ABSTRACT

This paper presents a mechanism-centred interpretation of stir-cast AA6063 hybrid nanocomposites containing 1.5 wt.% SiC and 0–1.0 wt.% graphene. Rather than reporting characterisation tests separately, the study links optical grain size, X-ray diffraction (XRD) crystallite size and dislocation density to microhardness, tensile strength and impact energy to identify a reinforcement threshold. Five material conditions were examined: base AA6063 (B), 1.5 wt.% SiC (C1), and SiC plus 0.2, 0.6 or 1.0 wt.% graphene (C2–C4). Relative to B, C3 (1.5 wt.% SiC+0.6 wt.% graphene) reduced the measured optical grain size from 52.4 to 24.8 μm (52.7%) and the XRD crystallite size from 62.962 to 55.846 nm (11.3%), while dislocation density increased from 2.52 to $3.21 \times 10^{-4} \text{ nm}^{-2}$. The same composition increased Vickers microhardness from approximately 65 to 90 HV (38.5%) and ultimate tensile strength from about 135 to 164.9 MPa ($\approx 22\%$). Increasing graphene to 1.0 wt.% reversed the refinement trend: grain size rose to 31.6 μm , crystallite size to 61.091 nm, dislocation density decreased to $2.68 \times 10^{-4} \text{ nm}^{-2}$, and both hardness and tensile response declined. A dimensionless microstructure–strength coupling index, based solely on measured normalised quantities and without fitted weighting factors, increased from 1.00 for B to 1.42 for C3 before falling to 1.21 for C4. SEM/EDS observations independently support the transition from improved dispersion at C3 to carbon-rich agglomeration and localized porosity at C4. The results establish 0.6 wt.% graphene as a reinforcement threshold for the present stir-cast system and explain its property maximum through simultaneous Hall–Petch refinement, interfacial load transfer and dislocation obstruction, balanced against the toughness penalty introduced by rigid reinforcement.

Keywords: AA6063; Graphene; SiC; Grain Refinement; XRD; Microhardness; Tensile Strength; Agglomeration; Hybrid Nanocomposite

1 INTRODUCTION

Hybrid aluminium matrix composites are often designed by pairing a hard ceramic with a nanoscale second-phase reinforcement so that one phase improves resistance to deformation while the other modifies interfaces, grain growth or tribological response [1-2]. For AA6063, SiC is a logical primary reinforcement because it is stiff, thermally stable and

capable of promoting heterogeneous nucleation [3]. Graphene offers an unusually high specific surface area and can obstruct dislocation motion or grain-boundary migration even at low additions [4-5]. However, the same high surface area creates a processing limitation: van der Waals attraction makes graphene increasingly difficult to disperse as its fraction rises [6-8]. The practical optimum therefore lies at a reinforcement threshold where the strengthening contribution of additional graphene is just balanced by agglomeration, porosity and weak local load transfer [9-10].

Many studies report hardness, tensile strength, XRD and microscopy in separate subsections. Such reporting establishes trends but does not directly demonstrate whether the property maximum and the microstructural optimum are the same phenomenon. A stronger mechanistic paper should test a causal sequence: reinforcement changes solidification and crystallographic length scales; those changes alter dislocation barriers and interfacial load transfer; and the resulting mechanical response rises until dispersion deteriorates. The present work analysis focuses on five compositions, ranging from unreinforced AA6063 to a fixed 1.5 wt.% SiC condition and three graphene levels.

1.1 Problem Statement, Research Gaps, Objectives and Contribution

1.1.1 Problem statement

Increasing nanoscale reinforcement does not guarantee greater strength. A composition can exhibit high local reinforcement content yet perform worse because particles cluster, grain refinement is lost, and defects become crack initiators. For the AA6063/SiC/graphene system, the engineering problem is to identify the graphene threshold and to connect it quantitatively to independently measured microstructural descriptors, rather than relying on a single mechanical response.

1.1.2 Research gaps

The literature identifies four recurring limitations: (i) many Al/graphene studies rely on powder metallurgy or friction-stir routes rather than economically scalable stir casting; (ii) SiC/graphene hybrid studies often use different aluminium grades, making the reinforcement threshold alloy-dependent; (iii) XRD-derived crystallite/dislocation parameters are rarely integrated with optical grain size and mechanical response into a single, compact mechanism map; and (iv) property improvements are often reported without explicitly quantifying the reversal caused by excess graphene [5-7-9-12].

1.1.3 Objectives

The study aims to: (1) quantify optical grain refinement across B–C4; (2) analyse the evolution of XRD crystallite size and dislocation density; (3) correlate these descriptors with Vickers microhardness and tensile strength; (4) evaluate the associated impact-energy penalty; (5) use SEM/EDS evidence to distinguish uniform dispersion from agglomeration; and (6) formulate a dimensionless microstructure–strength coupling index to test whether the same composition maximises both structural refinement and mechanical response.

1.1.4 Novelty and contribution

The paper reframes the material-development results as a threshold phenomenon rather than a sequence of independent tests. The central contribution is the quantitative demonstration that C3 simultaneously minimizes optical and XRD length scales, maximizes dislocation density, hardness and tensile strength, and yields the highest coupling index, whereas C4 reverses all of these trends. This multi-observable concurrence provides stronger evidence for an optimum than any single response.

2 CRITICAL LITERATURE REVIEW

Table 1 compares twelve studies on graphene- and ceramic-reinforced aluminium composites, focusing on processing routes, reinforcement levels, mechanical improvements, and reported limitations. The literature consistently indicates an optimum intermediate reinforcement level, while excessive graphene promotes clustering, degrading dispersion and mechanical performance.

Table 1: Comparative Literature on Graphene- and Ceramic-Reinforced Aluminium Composites with Identified Microstructure–Property Research Gaps

Study	Material/Processing	Key result	Limitation / gap
Hu et al. [1]	Stir-cast Al/graphene	0.2 wt.% graphene increased Vickers hardness by 42%; scalable stir casting retained graphene.	Pure Al system; no SiC hybrid or strength–microstructure threshold analysis.
Şenel et al. [2]	Al/SiC/GNP powder metallurgy	Hybrid Al-30SiC-0.5GNP reached 85 ± 2.6 HV; hybrid strengthening exceeded individual reinforcement effects.	Very high SiC fraction and powder-metallurgy route.
Chak & Chattopadhyay et al. [3]	Liquid-route Al/graphene	Up to 0.5 wt.% GNP produced 56.55% UTS and 53.39% microhardness gains.	No ceramic co-reinforcement; limited crystallographic coupling.
Venkatesan & Xavior et al. [4]	AA7050/graphene	0.3 wt.% graphene gave best tensile/dispersion response; higher content formed clusters.	Different age-hardenable matrix and squeeze-cast comparison.
Balasubramanian & Maheswaran et al. [5]	AA6063/SiC	Hardness increased with SiC; tensile response peaked then decreased at 15 wt.% as cleavage increased.	Micron SiC only; no graphene-assisted threshold.
Sarmah & Patowari et al. [6]	AA6063/SiC	Particle size and SiC fraction controlled hardness, tensile and wear response; intermediate condition was favorable.	Single reinforcement and no carbon dispersion transition.
Kumar et al. [7]	Al6063/Si ₃ N ₄	Increasing ceramic reinforcement improved mechanical and tribological properties over base alloy.	Different ceramic; no hybrid nanosheet interaction.
Wang et al. [8]	Al/graphene/SiC via FSP	Hybrid reinforcement and friction-stir processing improved microstructure and mechanical response.	Severe-plastic-deformation route, not as-cast microstructure.
Vishnoi et al. [9]	AA6063/graphene/SiC/fly ash	Hybrid stir-cast system showed large mechanical gains at an intermediate graphene level and deterioration at higher graphene.	Ternary reinforcement complicates isolation of graphene threshold.
El-Ghazaly et al. [10]	AA2124/graphene	Graphene addition changed strength and self-lubricating behavior; nanoscale dispersion controlled response.	Different matrix and powder processing.
Reddy et al. [11]	Al6063/TiC	Hardness/tensile strength rose with TiC; RSM linked reinforcement to wear.	Carbide-only response; no grain/crystallite coupling.
Prasad Reddy et al. [12]	Al6061/2SiC/xGr	Nano-clusters appeared as graphene increased; wear mechanism depended on carbon level.	Different matrix and ultrasonic casting; focus mainly tribology.

Across these studies, a consistent pattern emerges: a small graphene fraction can refine grains, increase dislocation barriers and improve load transfer, but higher additions tend to cluster. The threshold is not universal, as it depends on

alloy chemistry, primary ceramic fraction, particle size, wetting and processing route. The present AA6063 system is therefore analysed using its own measured microstructural and mechanical evidence rather than importing an optimum from another alloy.

3 MATERIALS AND METHOD

3.1 Matrix and reinforcements

Commercial AA6063 was used as the matrix. Its measured composition (wt.%) was approximately Al 97.91, Mg 0.4596, Si 0.335, Fe 0.36, Mn 0.705, Cu 0.0925, Cr 0.10, Ti 0.0208, and Zn 0.0136. The primary SiC reinforcement was nanoscale (about 50 nm, approximately 99% assay) and held at 1.5 wt.% in C1–C4. Graphene powder was about 10 nm, with reported purity above 99%, and was varied at 0.2, 0.6, and 1.0 wt.% in C2, C3, and C4, respectively. Table 2 defines the five investigated material conditions used to evaluate reinforcement effects. The AA6063 base alloy serves as the reference, while C1 isolates the SiC contribution. C2–C4 progressively increase graphene content, enabling direct assessment of low, intermediate, and high graphene reinforcement levels.

Table 2: Composition and Functional Classification of AA6063/SiC/Graphene Composite Specimens

ID	Matrix	SiC (wt.%)	Graphene (wt.%)	Role in comparison
B	AA6063	0	0	Reference matrix
C1	AA6063	1.5	0	Ceramic-reinforced control
C2	AA6063	1.5	0.2	Low graphene
C3	AA6063	1.5	0.6	Intermediate graphene
C4	AA6063	1.5	1.0	High graphene

3.2 Composite fabrication

The composites were produced by liquid-metallurgy stir casting. The AA6063 charge was preheated and melted in a crucible furnace at approximately 800 °C. Magnesium chips and degassing additions were used to improve wettability and reduce gas-related defects. SiC and graphene powders were preheated at about 400 °C for 1 h before being added to the melt. A ceramic-coated mechanical stirrer operated at approximately 350 rpm, and mixing continued for 8–10 min before the slurry was poured into the mould. All reinforced compositions followed the same sequence, making reinforcement content the primary controlled variable.

3.3 Microstructural and mechanical characterization

Optical microscopy was used to quantify grain size after standard metallographic preparation. The measured mean grain sizes were 52.4, 38.1, 27.2, 24.8 and 31.6 µm for B through C4. XRD patterns showed FCC aluminium reflections near 38°, 45° and 65°, SiC-associated reflections near 34–35.8°, and a weak carbon-related feature near 26° for graphene-containing composites. Crystallite size D was estimated from the Scherrer relation, and dislocation density δ from the inverse-square dependence on D . SEM with EDS and elemental mapping was used to assess particle distribution and Al–Si–C co-localization. Vickers microhardness was measured under low-load conditions consistent with ASTM E384; the experimental campaign used a 100 g load and 10 s dwell. Tensile specimens were prepared according to ASTM E8 and tested using an Instron universal testing machine at 2 mm/min. Charpy impact specimens followed ASTM E23. Multiple specimens were tested in the experimental campaign; the present paper focuses on the reported mean trends and their cross-property correlation.

3.4 Coupling index

To combine independent indicators without assigning arbitrary regression weights, a dimensionless microstructure–strength coupling index (CI) was defined as $CI = [(H/H_0)(UTS/UTS_0)(d_0/d)(D_0/D)]^{1/4}$, where H is microhardness, UTS is ultimate tensile strength, d is optical grain size, D is XRD crystallite size, and subscript 0 denotes the base alloy. The fourth root keeps the index on a scale comparable to individual normalized ratios. By definition, $CI=1$ for AA6063. A higher value indicates that strength increases concurrently with reductions in both microstructural length scales. Impact energy is intentionally excluded so that the separate strength–toughness trade-off remains visible rather than being hidden in the index.

4 RESULTS AND DISCUSSION

4.1 Grain refinement and crystallographic scale

Figure 2 shows two refinement scales. Adding 1.5 wt.% SiC reduced the optical grain size from 52.4 μm for B to 38.1 μm for C1, consistent with heterogeneous nucleation and particle pinning during solidification. Adding 0.2 wt.% graphene further reduced the size to 27.2 μm , and C3 reached a minimum of 24.8 μm . This represents a 52.7% reduction relative to the base alloy. C4 reversed the trend: at 1.0 wt.% graphene, the grain size increased to 31.6 μm , despite remaining finer than B. This rebound is important because it indicates that simply increasing graphene no longer increases the number of effective grain-growth barriers. XRD provides an independent nanoscale descriptor. Crystallite size decreased from 62.962 nm (B) to 60.761 nm (C1), 59.374 nm (C2), and 55.846 nm (C3), corresponding to an 11.3% reduction at C3. At C4, it returned to 61.091 nm. The matching minima of optical grain size and XRD crystallite size at C3 strengthen the interpretation of a true refinement optimum rather than image-to-image variability. The two techniques probe different length scales, yet both show the same composition threshold.

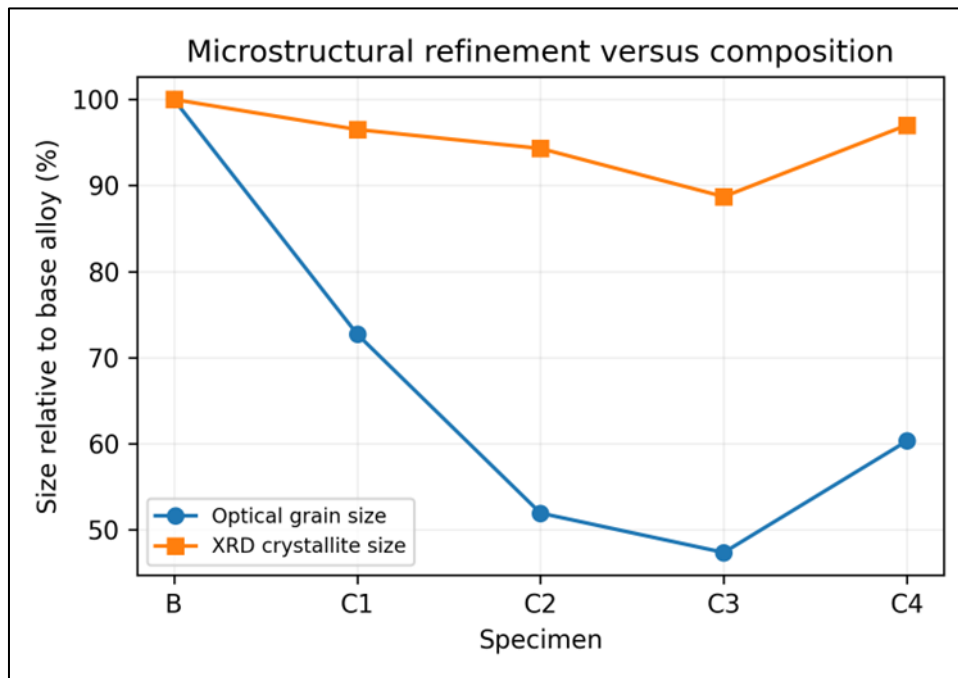


Figure 2: Optical grain size and XRD crystallite size normalized to the AA6063 base alloy.

4.2 Dislocation density and strengthening response

The inverse relation between crystallite size and the dislocation-density estimate yielded δ values of 2.52, 2.71, 2.84, 3.21 and $2.68 \times 10^{-4} \text{ nm}^{-2}$ for B through C4. C3 therefore exhibited approximately 27.4% higher dislocation density than B as discussed in Table 3. This increase is consistent with greater resistance to plastic flow, as dislocations encounter both grain boundaries and strain fields around the reinforcement/matrix interface. Thermal-expansion mismatch between aluminium and ceramic/carbon phases can generate geometrically necessary dislocations during cooling, while uniformly dispersed particles shorten the free path for dislocation glide. Mechanical data mirror this crystallographic progression. Microhardness increased from approximately 65 HV for B to 75 HV for C1 and 82 HV for C2, reaching approximately 90 HV for C3. This represents a 38.5% increase over the base alloy. At C4, hardness declined to about 78 HV. Ultimate tensile strength followed the same non-monotonic behaviour: the base alloy was approximately 135 MPa, C1 and C2 increased to about 153 and 159 MPa, respectively, C3 reached 164.9 MPa, and C4 declined to roughly 142 MPa as shown in Figure 3. Thus, the high-graphene specimen retains some hardness benefit but loses a large fraction of the tensile strengthening achieved at C3, which is typical when local clusters act as stress concentrators.

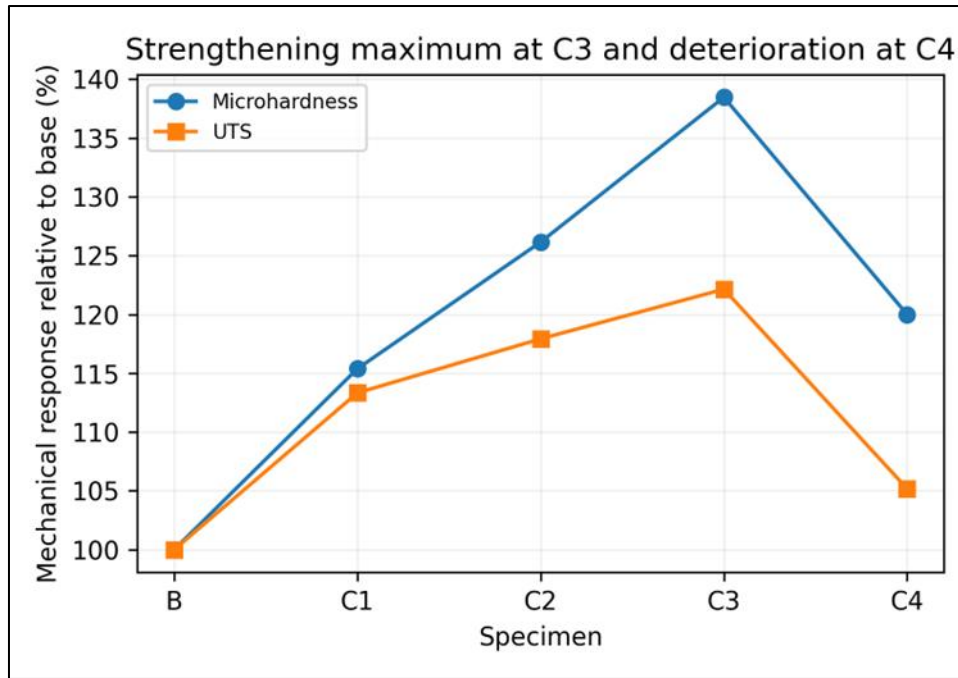


Figure 3: Normalized microhardness and ultimate tensile strength across the five material conditions.

Table 3: Microstructural, Mechanical, Impact, and Coupling-Index Response of AA6063/SiC/Graphene Composite Specimens

ID	Grain (μm)	Crystallite (nm)	δ ($\times 10^{-4} \text{ nm}^{-2}$)	HV	UTS (MPa)	Impact (J)	CI
B	52.4	62.962	2.52	65	135.0	9.95	1.000
C1	38.1	60.761	2.71	75	≈ 153	7.80	1.168
C2	27.2	59.374	2.84	82	≈ 159.2	7.95	1.320
C3	24.8	55.846	3.21	90	164.9	6.40	1.417
C4	31.6	61.091	2.68	78	≈ 142	5.60	1.212

4.3 Microstructure–strength coupling index

Figure 4 combines the independent trends without altering their physical direction. CI rises from 1.000 for B to 1.168 for C1, 1.320 for C2, and 1.417 for C3, before falling to 1.212 for C4. The maximum therefore coincides exactly with the highest dislocation-density ratio. The index is useful because it penalizes a composition if high hardness is achieved without refinement, or if refinement is achieved without a corresponding increase in tensile strength. C3 is the only condition that simultaneously maximizes hardness, UTS, and dislocation density while minimizing both grain and crystallite sizes.

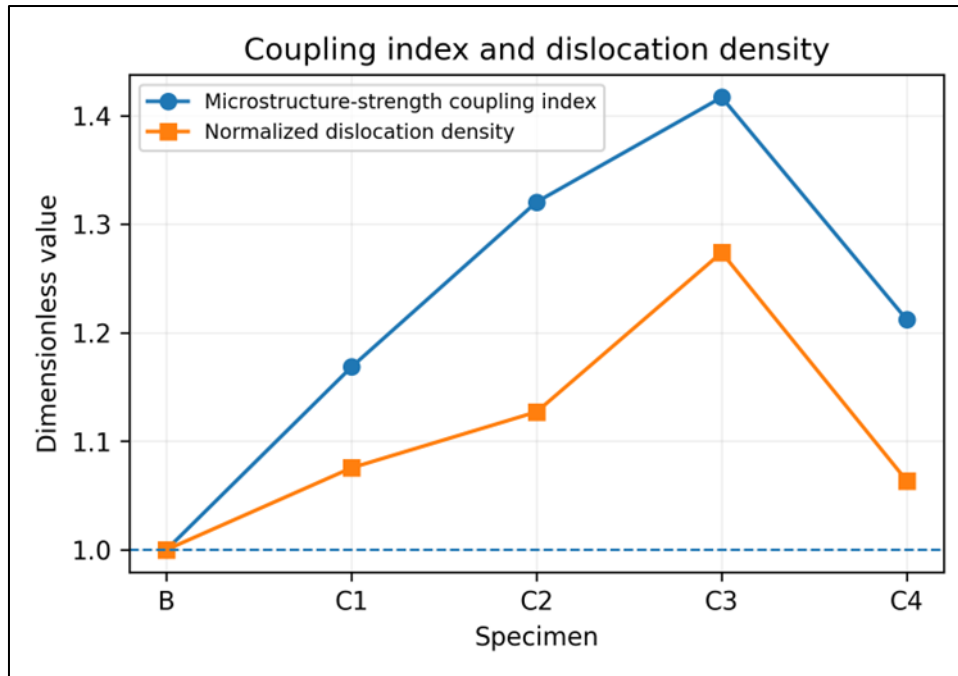


Figure 4: Microstructure–strength coupling index and normalized dislocation density.

4.4 SEM evidence for the threshold mechanism

SEM provides the morphological explanation for the quantitative reversal. The base alloy shows a comparatively uniform aluminium-rich matrix. C1 introduces dispersed angular SiC particles but still exhibits local clustering. At 0.2 wt.% graphene, the distribution becomes more homogeneous, and carbon begins to overlap with Si-rich reinforcement regions. C3 exhibits the most continuous Al–Si–C elemental overlap, relatively uniform reinforcement spacing and minimal large clusters. Such a morphology supports efficient load transfer, grain-boundary pinning and dislocation obstruction. In contrast, C4 contains localized carbon-rich regions and increased porosity near reinforcement clusters. These defects reduce the effective interfacial area and create preferred sites for crack initiation. The mechanism can therefore be divided into two regimes. From C1 to C3, adding graphene increases the number and effectiveness of interfacial barriers, reduces characteristic structural length scales and improves stress transfer. From C3 to C4, marginal graphene is increasingly consumed in clusters rather than at uniformly distributed interfaces. The effective reinforcement density falls even though the nominal reinforcement content rises. This explains why both refinement measures and mechanical properties deteriorate together at 1.0 wt.% graphene.

4.5 Strength–toughness trade-off

The reinforcement threshold should not be interpreted as a universal improvement across all properties. Charpy absorbed energy decreases from 9.95 J for B to 7.8 J for C1, 7.95 J for C2, 6.4 J for C3 and 5.6 J for C4. Figure 5 therefore shows a clear trend towards higher strength but lower impact energy. For C3, the $\approx 22\%$ increase in tensile strength is accompanied by an $\approx 35.7\%$ reduction in absorbed energy relative to the base alloy. This trade-off is physically consistent with reduced plasticity, as hard particles, refined grains and high dislocation density restrict deformation. C4 is undesirable because it sacrifices even more impact energy while losing much of the tensile advantage; it is therefore dominated by C3 from a strength–integrity perspective.

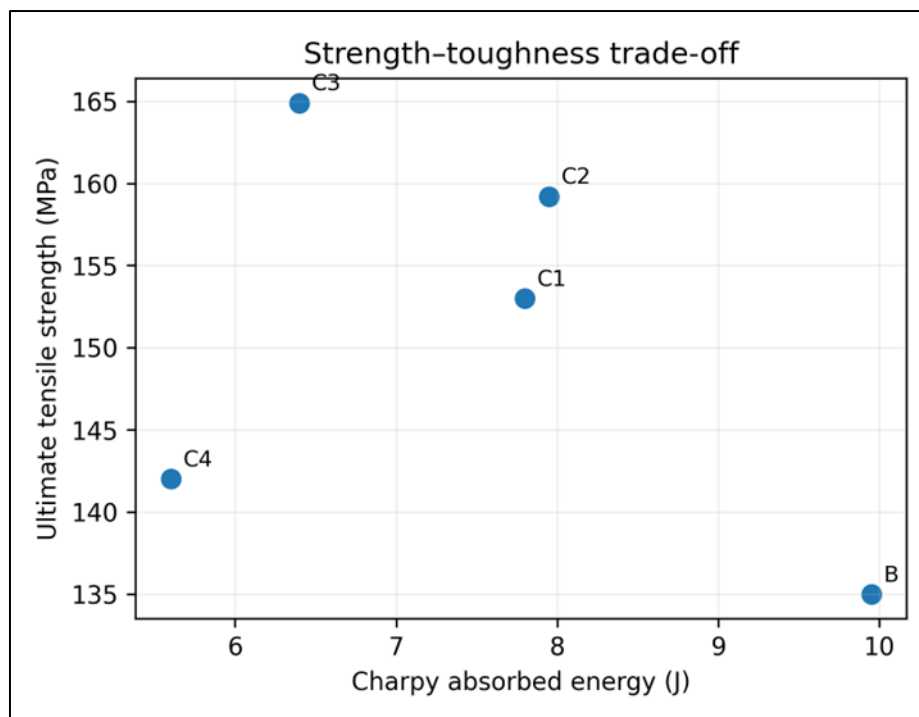


Figure 5: Strength-toughness map showing the mechanical consequence of progressive hybrid reinforcement.

4.6 Comparison with published work

The present threshold aligns with the qualitative behaviour reported for other graphene–aluminium systems, though its exact location differs. Hu et al. found an optimum as low as 0.2 wt.% graphene in stir-cast aluminium [1], and Venkatesan and Xavier observed clustering above 0.3 wt.% in AA7050 [4]. Chak and Chattopadhyay reported substantial improvements up to 0.5 wt.% GNP [3]. Differences are expected because the current material already contains 1.5 wt.% SiC, which modifies nucleation, interfacial area and melt rheology. The SiC can help establish a distributed rigid skeleton, allowing graphene to remain beneficial at a somewhat higher fraction before clustering dominates. The observed 0.6 wt.% threshold should therefore be regarded as specific to the current particle sizes, SiC fraction and stir-casting procedure. The data also reinforce an important methodological point. A peak in hardness alone could arise from local particle-rich indentation sites, while a peak in tensile strength alone could be affected by specimen defects. Here, five independent indicators—optical grain size, XRD crystallite size, dislocation density, microhardness and UTS—change direction at the same composition, with SEM/EDS showing the expected dispersion-to-agglomeration transition. That convergence is the strongest evidence for the proposed threshold mechanism.

5 CONCLUSION

A mechanism-focused re-analysis of AA6063/1.5 wt.% SiC/xGraphene hybrid nanocomposites identifies a clear reinforcement threshold near 0.6 wt.% graphene. C3 reduced the optical grain size from 52.4 to 24.8 μm and the XRD crystallite size from 62.962 to 55.846 nm, while increasing the estimated dislocation density from 2.52 to $3.21 \times 10^{-4} \text{ nm}^{-2}$. The same composition increased microhardness from approximately 65 to 90 HV and UTS from about 135 to 164.9 MPa. Increasing graphene to 1.0 wt.% caused a grain-size rebound (31.6 μm), crystallite coarsening (61.091 nm), a lower dislocation density ($2.68 \times 10^{-4} \text{ nm}^{-2}$), and reduced hardness and tensile strength. SEM/EDS observations explain the reversal through carbon-rich agglomeration, localized porosity, and a reduced effective load-transfer area. The proposed dimensionless coupling index rose from 1.00 for AA6063 to 1.42 for C3 and then fell to 1.21 for C4, independently confirming that the microstructural and mechanical optima coincide. The principal limitation is the strength–toughness trade-off: C3 gains tensile strength but loses approximately 35.7% of the base-alloy Charpy energy. Future work should refine the 0.4–0.8 wt.% graphene interval, quantify particle-cluster size distributions and porosity statistically, and test whether heat treatment or ultrasonic assistance can preserve a C3-like dispersion while recovering ductility.

STATEMENTS ON COMPLIANCE WITH ETHICAL STANDARDS

Disclosure of conflict of interest

The authors declare no conflicts of interest regarding this manuscript.

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