



# The Role of Graphene on Microstructural, Mechanical, and Tribological Properties of Cu–AlCrFeCuNi Metal Matrix Composites Fabricated *via* Spark Plasma Sintering

HARUN YANAR, MÜSLİM ÇELEBİ, DURSUN MURAT SEKBAN, SERDAR ÖZKAYA, ABDULLAH HASAN KARABACAK, and ERTUĞRUL ÇELİK

In this study, Cu-based AlCrFeCuNi high-entropy alloy (HEA) composites reinforced with graphene were fabricated using a mechanical-alloying-assisted spark plasma sintering (SPS) technique. The HEA reinforcement content was kept constant at 20 wt pct for all samples, while the graphene content was varied at 0, 0.5, 1.0, and 2.0 wt pct to investigate its influence on the microstructure, hardness, and tribological performance. The results indicate that both the HEA particles and graphene exhibit an almost homogeneous distribution within the matrix; however, increasing graphene content led to noticeable graphene accumulation along grain boundaries. With higher graphene fractions, the relative density decreased from 97.51 to 94.92 pct, whereas the hardness increased up to the 1.0 wt pct graphene addition, reaching 134.16 HB, followed by a slight decrease to 129.25 HB at 2.0 wt pct. Furthermore, graphene reinforcement significantly improved the tribological behavior of the composites, providing a substantial reduction in wear. The addition of graphene significantly decreased the coefficient of friction from about ~ 0.70 in the Cu–HEA alloy to 0.08 at 2.0 wt pct graphene. Graphene also suppressed the pronounced frictional fluctuations observed in the base alloy, leading to a much more stable sliding behavior. As a result, wear loss was reduced by nearly 99 pct, primarily due to the formation of a continuous lubricating tribofilm on the contact surface.

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## 1. INTRODUCTION

COPPER, with its high electrical and thermal conductivity, is one of the most preferred metals in engineering applications. It has a wide range of uses in industry, from cables to cooling systems. Its high

ductility and formability make it suitable for the production of many different geometries.<sup>[1–5]</sup> However, pure copper has some mechanical and tribological limitations. While pure copper provides high conductivity, its low hardness and wear resistance make it inadequate for long-term operation under heavy loads. This leads to performance losses, particularly in friction and contact surfaces. Industrial requirements are increasing the need for materials that combine both high conductivity and advanced mechanical properties. Therefore, the use of copper in the form of metal matrix composites (MMC) offers a significant advantage. MMC production allows the addition of different reinforcement phases to the copper matrix. These reinforcements can increase hardness, wear resistance, fatigue strength, and thermal stability.<sup>[6–8]</sup> This transforms copper into an engineering material that provides both high performance and long life in specific applications.

The use of high-entropy alloys (HEAs) as reinforcement phases in MMC designs represents an innovative approach that offers versatile advantages compared to conventional ceramic or metallic reinforcements.<sup>[9,10]</sup> Owing to their complex structures composed of multiple

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HARUN YANAR is with the Mechanical Engineering, Karadeniz Technical University, 61080 Trabzon, Turkey and also with the Trabzon Teknokent, UTS Software and Engineering Services Industry Trade Limited Company, 61080, Trabzon, Turkey. MÜSLİM ÇELEBİ, SERDAR ÖZKAYA, and ABDULLAH HASAN KARABACAK are with the Metallurgical and Materials Engineering, Karadeniz Technical University, 61080 Trabzon, Turkey. Contact e-mail: [hasankarabacak@ktu.edu.tr](mailto:hasankarabacak@ktu.edu.tr) DURSUN MURAT SEKBAN is with the Department of Marine Engineering Operations, Karadeniz Technical University, 61080 Trabzon, Turkey and also with the Trabzon Teknokent, WMS Engineering Services Industry Trade Limited Company, 61080 Trabzon, Turkey. ERTUĞRUL ÇELİK is with the Department of Mechanical Engineering, Munzur University, 62000 Tunceli, Turkey.

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principal elements, HEAs exhibit high strength, superior wear resistance, thermal stability, and stable phase formation. Their ability to maintain mechanical properties over a wide temperature range, together with effective deformation hardening mechanisms and microstructural stability, extends the service life of MMCs.<sup>[9,11]</sup> In particular, the AlCrFeCuNi HEA provides both mechanical and tribological benefits due to its multiphase (FCC/BCC) nature, high hardness, and thermodynamic stability. This reinforcement enhances load-bearing capacity, suppresses surface wear mechanisms, and improves the composite's resistance to elevated temperature conditions. For example, in our previous study,<sup>[12]</sup> we reinforced the AA5083 alloy with 10 pct AlCrFeCuNi HEA and achieved increases of 60 pct in hardness, 35 pct in tensile strength, and 60 pct in wear resistance. In another study, Özkaya incorporated varying amounts of AlCrFeCuNi HEA into the ZA40 alloy and reported significant improvements in both mechanical and tribological properties.<sup>[13]</sup> The highest performance was observed in samples containing 20 pct HEA, where hardness increased by 95 pct, tensile strength by 64 pct, and wear resistance by a factor of 19. Based on these findings, AlCrFeCuNi HEA stands out as an effective reinforcement material that markedly enhances both functional and structural performance in MMCs.

Various ceramic and carbon-based reinforcement phases are used in copper-based MMCs. These reinforcements include materials such as SiC, Al<sub>2</sub>O<sub>3</sub>, B<sub>4</sub>C, TiC, CNTs, and graphene.<sup>[14–17]</sup> Graphene is a two-dimensional carbon nanomaterial that makes a significant contribution to tribological systems due to its high thermal conductivity, high elastic modulus, and excellent lubricity.<sup>[18–20]</sup> Graphene is expected to reduce the coefficient of friction and form a protective film on the surface. In case of using HEA and graphene together, it is expected that the HEA reinforcement will increase the structural strength in the composite to be formed, while the graphene will improve the friction performance and provide a more stable wear mechanism. Moreover, both HEAs and graphene offer advantages not only in structural strengthening but also in enhancing thermal stability and functional performance. HEAs exhibit remarkable resistance to thermal softening, while graphene contributes to heat dissipation due to its superior thermal conductivity. These characteristics make the HEA–graphene combination particularly attractive for applications where thermal management and tribological reliability are critical. Additionally, the simultaneous improvement of thermal stability, electrical conductivity, and wear resistance positions this reinforcement strategy as a promising approach for next-generation copper-based MMCs.

Many methods are used for the production of copper-based MMCs.<sup>[14,21–24]</sup> However, each of these methods faces certain limitations, particularly when combining copper with high-melting-point reinforcement phases. Powder metallurgy-based methods offer advantages in maintaining the structural integrity of the reinforcement phases because they require lower processing temperatures. Among these methods, spark

plasma sintering (SPS) stands out for its rapid sintering capabilities and reduced grain growth. SPS, thanks to its high heating rates and short processing times, enables composites to achieve a homogeneous and dense structure.<sup>[14,25,26]</sup> Passing electrical current directly through the powder mixture significantly accelerates sintering kinetics compared to traditional methods. It also allows for high density at low temperatures. This is a significant advantage, particularly in preserving the sensitive properties of copper, such as thermal conductivity. The SPS device promotes effective bonding between particles by applying pressure at high temperatures. This improves both mechanical properties and tribological behavior. There are many studies demonstrating successful results using SPS in various MMCs.<sup>[27–30]</sup>

There are numerous studies on copper matrix composites in the literature.<sup>[17,21,31–33]</sup> Some of these studies focus on ceramic reinforced composites, while others have investigated the tribological effects of carbon-based reinforcements.<sup>[34–36]</sup> Meanwhile, research on HEA-reinforced copper composites is increasing. These studies show that high-entropy alloys increase mechanical strength and improve wear behavior in the copper matrix.<sup>[37–39]</sup> Studies on graphene-reinforced copper composites reveal that graphene, due to its high surface area and two-dimensional structure, positively affects friction behavior.<sup>[36,40–42]</sup> However, there is no study in the literature in which HEA and graphene are produced in the same composite system within a copper matrix using the SPS method. Therefore, a holistic evaluation of the production and behavior of the hybrid reinforced Cu–HEA–graphene system is anticipated to fill an important scientific gap. In our study, Cu-based MMCs with constant HEA content and varying graphene amounts were produced using the SPS method, and the microstructure, density, hardness, and tribological behavior of the produced MMCs were investigated in detail.

## II. EXPERIMENTAL PROCEDURE

### A. Materials and Composite Fabrication Process

In this study, high-purity copper powder (99.99 pct,  $d_{50}$ : 21  $\mu\text{m}$ , Nanografi, Turkey) was used as the matrix material. HEA powder which consisting of the elements Fe–Ni–Al–Cu–Cr in equal-mass composition ( $d_{50}$ : 11.7  $\mu\text{m}$ ) and graphene nanoparticles ( $d_{50}$ : 100 nm, Nanografi, Turkey) were selected as reinforcement phases. The HEA reinforcement was kept constant in all samples and added at 20 wt pct. Graphene reinforcements were varied at 0.5, 1.0, and 2.0 wt pct to investigate their contribution to the mechanical and tribological properties of the composites. Sample codes and reinforcement contents are presented in Table I.

Milling was performed to ensure homogeneous mixing of all powders. This process was carried out in a Retsch PM100 planetary ball mill for 2 hours at a rotation speed of 400 rpm, with a ball-to-powder weight ratio of 10:1. 2 wt pct methanol was added as a process control agent to prevent powder adhesion and cold

**Table I. The Sample Codes and Milling Parameters**

| Sample Code  | Milling time (h) | Reinforcement Content |                   | Milling Speed (rpm) | Ball-to-Powder Weight Ratio |
|--------------|------------------|-----------------------|-------------------|---------------------|-----------------------------|
|              |                  | HEA (Wt Pct)          | Graphene (Wt Pct) |                     |                             |
| Cu-HEA       | 2                | 20                    | 0                 | 400                 | 10:1                        |
| Cu-HEA-0.5Gr | 2                | 20                    | 0.5               | 400                 | 10:1                        |
| Cu-HEA-1.0Gr | 2                | 20                    | 1.0               | 400                 | 10:1                        |
| Cu-HEA-2.0Gr | 2                | 20                    | 2.0               | 400                 | 10:1                        |

welding during the process. These selected parameters were found to be optimal based on preliminary trials, as they prevented insufficient dispersion at low energy levels and excessive cold welding at high energy levels.

After mechanical alloying, the hybrid powder mixtures were densified using a Çelmak SPS-160V SPS system. Sintering was carried out under a heating rate of 150 °C/min, a pulse on/off ratio of 12:2, a sintering temperature of 800 °C, a holding time of 4 minutes, and a uniaxial pressure of 35 MPa. The production atmosphere was vacuum and nitrogen controlled. These parameters were optimized as a result of preliminary studies to obtain high density, prevent excessive growth of grains, and limit undesirable interface reactions between copper and HEA/graphene phases.

### B. Characterization

After the SPS process was completed, the hybrid composite samples were metallographically prepared to be suitable for microstructure and phase analyses. Detailed microstructural characterization of these bulk materials was carried out using a Thermo Scientific Apreo 2S model field emission scanning electron microscope (FE-SEM) integrated with Thermo Scientific UltraDry energy dispersive spectrometer (EDS). The scars and surface deformations resulting from the wear tests were also examined by scanning electron microscope (SEM) in order to better understand the wear behavior and mechanisms. Quantitative grain size analysis was performed using ImageJ software. SEM images obtained at  $\times 500$  magnification were evaluated from three randomly selected representative areas for each sample to ensure statistical consistency. Grain dimensions were measured along orthogonal directions (horizontal and vertical), and the average of these measurements was taken as the representative grain size. The reported values correspond to the mean grain size calculated from all analyzed regions. Phase analyses were determined by X-ray diffraction (XRD) measurements performed with a PANalytical X'Pert3 Pro instrument equipped with a  $\text{CuK}\alpha$  ( $\lambda = 1.541874 \text{ \AA}$ ) X-ray source in the range of 30 to 90 deg  $2\theta$ , with 0.01 deg steps, using a scanning speed of one second at each step. Archimedes' Principle was used to determine the density values of the Cu-based composites samples. Hardness measurements were performed using the Brinell method (Innovatest-Nemesis 9000, 31.25 kg

load). Average values were obtained from six points for each sample.

The tribological response of the composites was evaluated at ambient temperature using a ball-on-disk type friction and wear tester (UTS Tribolog equipped with reciprocating module, Trabzon, Türkiye). In all tests, 100Cr6 bearing steel balls with a diameter of 6 mm and a hardness of  $\sim 60$  HRC were used as a counterface materials. In the literature, copper-based metal matrix composites are typically examined under light to moderate loads, and for Cu-h-BN-B<sub>4</sub>C-type hybrid systems, the commonly used load window is 2.5 to 20 N, where 1 N is considered the lower limit and 20 N the upper limit.<sup>[43,44]</sup> In preliminary trials of the present work, however, applied loads exceeding 10 N resulted in extensive surface damage, which hindered reliable assessment of wear mechanisms. For this reason, the applied normal loads were selected as 5N to obtain reproducible and meaningful results. The total sliding distance in the friction tests was fixed at 60 m (corresponds to 5000 reciprocating cycles) for all Cu-based composites. All tests were carried out following the standards of ASTM G133. During sliding, the stroke length and oscillation frequency were chosen as 12 mm and 3 Hz, respectively. Each condition was tested three times with a new steel ball to improve statistical reliability. After testing, volume loss and depth profile of wear scar were determined using an 3D surface profilometer (Nanofocus  $\mu$ scan, Germany).

## III. RESULTS AND DISCUSSION

### A. Powder Characterization

Figure 1 presents the SEM micrographs and particle size distributions of the powders used in this study. The initial Cu powders exhibit a predominantly spherical morphology accompanied by a high degree of particle size uniformity [as shown in Figure 1(a)]. The particle size distribution demonstrates a very narrow range, reflecting the excellent homogeneity of the starting Cu powder system [Figure 1(a')]. In contrast, the SEM image of the HEA powders milled for 25 hours reveals a heterogeneous and irregular morphology, lacking any specific geometrical order [Figure 1(b)]. Alongside small spherical particles, the powder also contains large, irregularly shaped particles with sharp edges, indicating significant morphological diversity. The particle size distribution spans a broad range and displays a clear

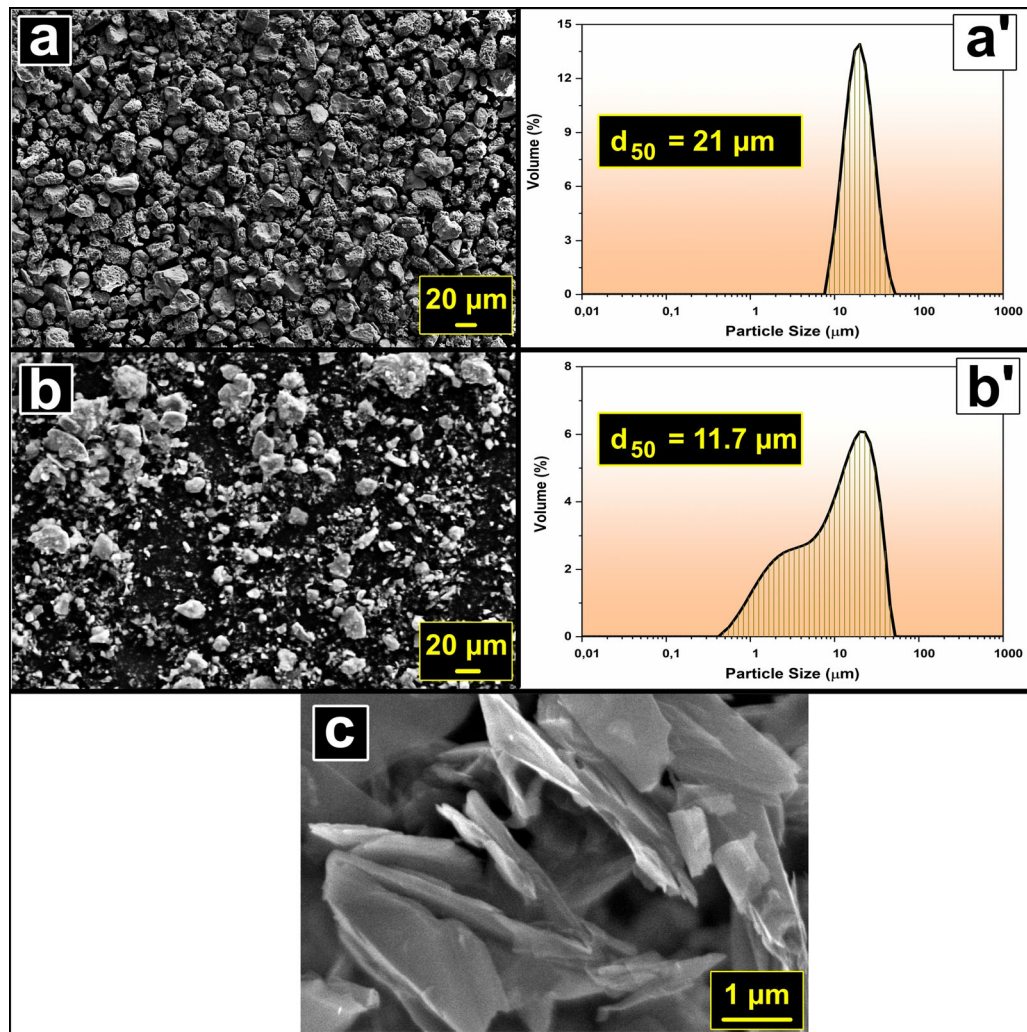


Fig. 1—(a) to (a') SEM image and particle size distribution of the as-received Cu powders, (b) to (b') SEM image and particle size distribution of 25 h milled AlCrFeCuNi HEA powders, and (c) SEM image of the as-received graphene powders.

bimodal character, with the coexistence of submicron particles and coarse particles reaching 70 to 80  $\mu\text{m}$  [Figure 1(b')]. Furthermore, the graphene reinforcement exhibits a nanosheet-like morphology, characterized by thin, layered structures typically associated with two-dimensional carbonaceous materials [Figure 1(c)].

Figure 2 presents the EDS mapping analysis of the AlCrFeCuNi HEA powder used in this study. In the figure, the red, pink, blue, green, and yellow colors correspond to the elements Cu, Fe, Al, Cr, and Ni, respectively. The elemental maps reveal that each element is distributed almost uniformly across the powder surface, demonstrating that the alloying process successfully produced a HEA with well intermixed constituent elements at the microscale. Despite this minor variation, the EDS analysis confirms a homogeneous distribution of all principal elements, consistent with the formation of a multi-principal element HEA structure.

The particle size and morphology of the powders used in the SPS process have a decisive influence on the densification behavior and the resulting properties of the

consolidated component. For SPS, powders typically within the range of 1 to 50  $\mu\text{m}$  exhibit a morphology close to spherical are preferred, as such features promote uniform packing, stable electrical contact, and efficient heat transfer during sintering.<sup>[45,46]</sup> Figure 3 presents the SEM images and particle size distribution curves of the powders subjected to 2 hours of milling. Since the initial powders already possessed relatively small particle sizes, extended milling would inevitably produce excessively fine or even submicron particles. Such particles may introduce agglomeration, disturb the uniform packing in the SPS die, and ultimately impair densification. In addition, prolonged milling of graphene-containing systems leads to the fragmentation of graphene nanosheets, thereby diminishing their structural integrity and functional effectiveness. For these reasons, the milling duration was limited to 2 hours, which was found to provide an optimal compromise between particle refinement and preservation of powder integrity. Figure 3(a) shows that the Cu/HEA powders maintain a nearly spherical morphology after 2 hours of milling. Despite the relatively high fraction of HEA (20 wt pct),

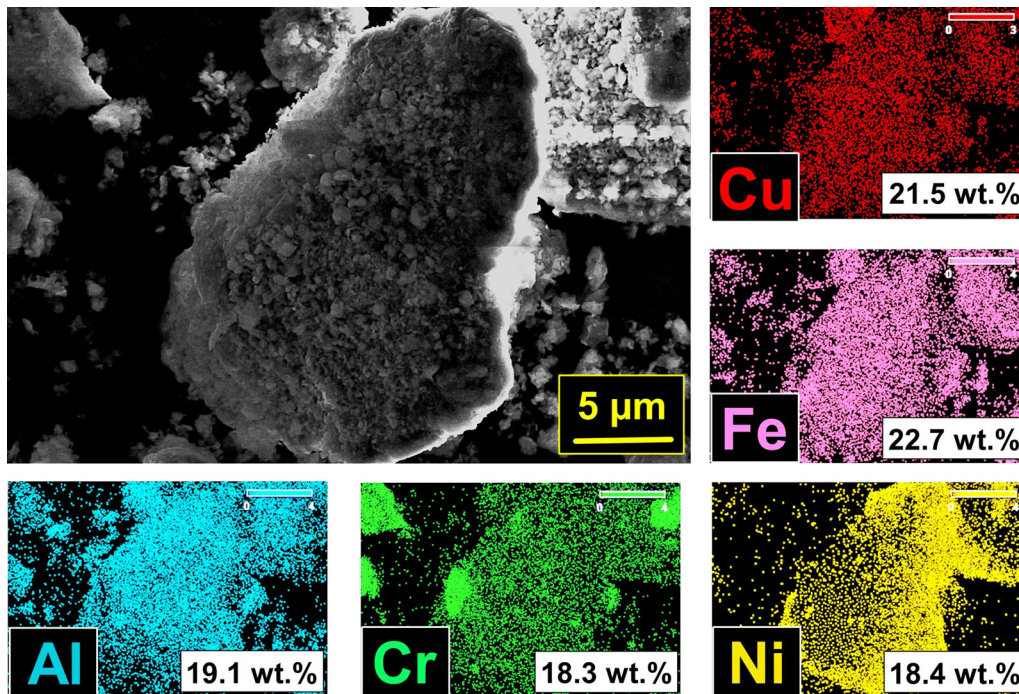


Fig. 2—SEM image and EDS mapping analysis of 25-h milled AlCrFeCuNi HEA powder (Color figure online).

the short milling duration preserves the overall spherical characteristics of the composite powders. However, the particle size distribution reveals the emergence of a secondary population of particles. This is attributed to the agglomeration of submicron HEA fragments, which combine to form clusters with typical sizes of  $\sim 6 \mu\text{m}$ . As a result, a bimodal distribution becomes evident [Figure 3(a')]. Considering that the as-received Cu powders possessed an average size of  $21 \mu\text{m}$ , the introduction of 20 wt pct HEA and 2 hours of milling effectively reduced the average particle size to  $14.91 \mu\text{m}$ . For the Cu-HEA-0.5Gr powder [Figure 3(b)], the spherical morphology is again largely preserved, but the presence of graphene leads to a more pronounced decrease in particle size. The average size decreases by approximately 15 pct compared with the Cu-HEA sample. The addition of 0.5 wt pct graphene also reduces the intensity of the bimodal character, gradually shifting the system toward a narrower unimodal distribution [Figure 3(b')]. Graphene acts as a solid lubricant at the particle-particle and particle-ball interfaces. By limiting cold welding events during milling, it prevents small fragments from bonding together into larger agglomerates, thus stabilizing the refinement process and providing a more controlled decrease in particle size.<sup>[47,48]</sup> When the graphene content is increased to 1.0 wt pct, the particle size continues to decrease, while the spherical morphological tendency remains preserved [Figure 3(c)]. The average size decreases from  $12.78$  to  $11.62 \mu\text{m}$ , corresponding to a reduction of approximately 9 pct. At this concentration, graphene further inhibits cold welding, allowing submicron fragments to remain dispersed; therefore, the presence of particles smaller than one micron is still detectable [Figure 3(c')].

Meanwhile, the transition toward a predominantly unimodal distribution becomes more evident. Upon increasing the graphene reinforcement to 2.0 wt pct, only a modest additional decrease in particle size is observed. The size decreases from  $11.62$  to  $10.43 \mu\text{m}$ , corresponding to approximately a 10 pct reduction. The spherical morphology becomes even more dominant at this stage, as nearly all powder particles adopt shapes approaching perfect sphericity [Figure 3(d)]. This effect is associated with the enhanced homogenization of the fracturing mechanism induced by higher graphene contents. Furthermore, the population of large particles decreases significantly, confirming that higher graphene levels effectively suppress agglomeration and promote a more refined and homogeneous powder structure [Figure 3(d')]. The results demonstrate that all powder compositions retain a morphology close to spherical despite variations in reinforcement content. This characteristic is particularly advantageous for SPS processing, as spherical and uniformly sized powders promote better packing behavior, enhanced current flow, and improved sinterability, ultimately contributing to superior densification and mechanical performance of the final consolidated materials.

## B. Microstructural Analysis

Figure 4 shows the microstructure images and grain size graphs of hybrid composites with a Cu matrix, fixed HEA reinforcement, and varying amounts of graphene reinforcement. In the SEM images, dark phases represent the HEA element, while light gray phases represent the Cu element in excess. As graphene reinforcement increases in the 20 wt pct HEA Cu matrix composites

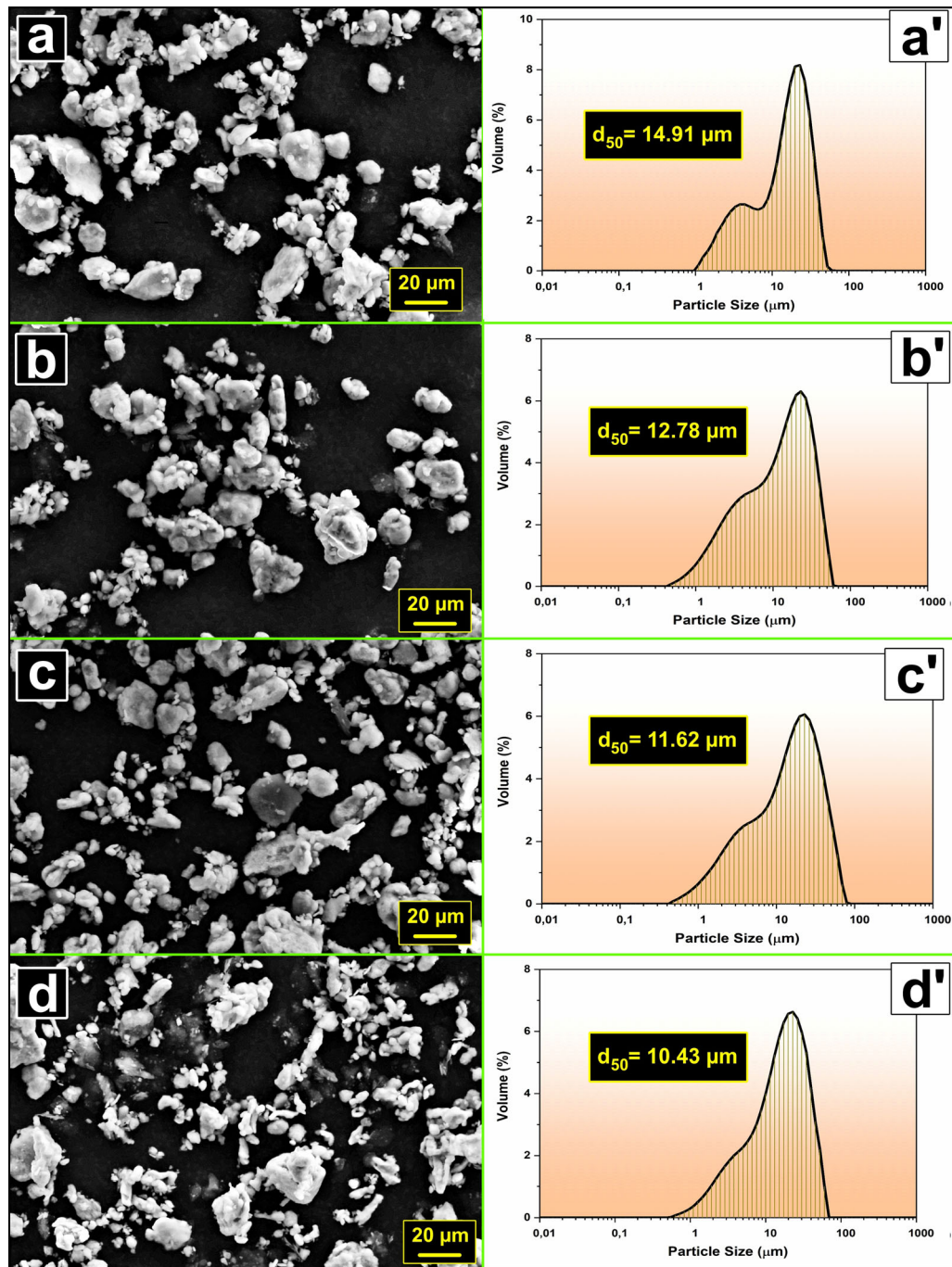


Fig. 3—SEM images and particle size distributions of 2 h milled powders: (a) to (a') Cu-HEA, (b) to (b') Cu-HEA-0.5Gr, (c) to (c') Cu-HEA-1.0Gr, and (d) to (d') Cu-HEA-2.0Gr.

produced by SPS, the grain size distribution curves indicate smaller and more uniform grains. The grain size distribution graphs for each sample show that the average grain size decreases with increasing graphene content. The measured grain sizes of Cu-HEA, Cu-HEA-0.5Gr, Cu-HEA-1.0Gr, and Cu-HEA-2.0Gr composite and hybrid composite samples are 13.705, 10.312, 7.823, and 7.279  $\mu\text{m}$ , respectively. It has also been reported in the literature that the addition of graphene provides significant grain refinement in metal

matrices. For example, the presence of graphene nanosheets in Cu-Graphene composites restricts grain growth during sintering, resulting in a smaller-grained microstructure.<sup>[17]</sup> As the graphene content increases from 0 to 2 pct by mass, this grain-reducing effect of graphene becomes clear: while the grains are largest in the sample without graphene, the finest grain sizes are achieved in the sample with 2 pct graphene. This further supports the fact that the presence of graphene reduces

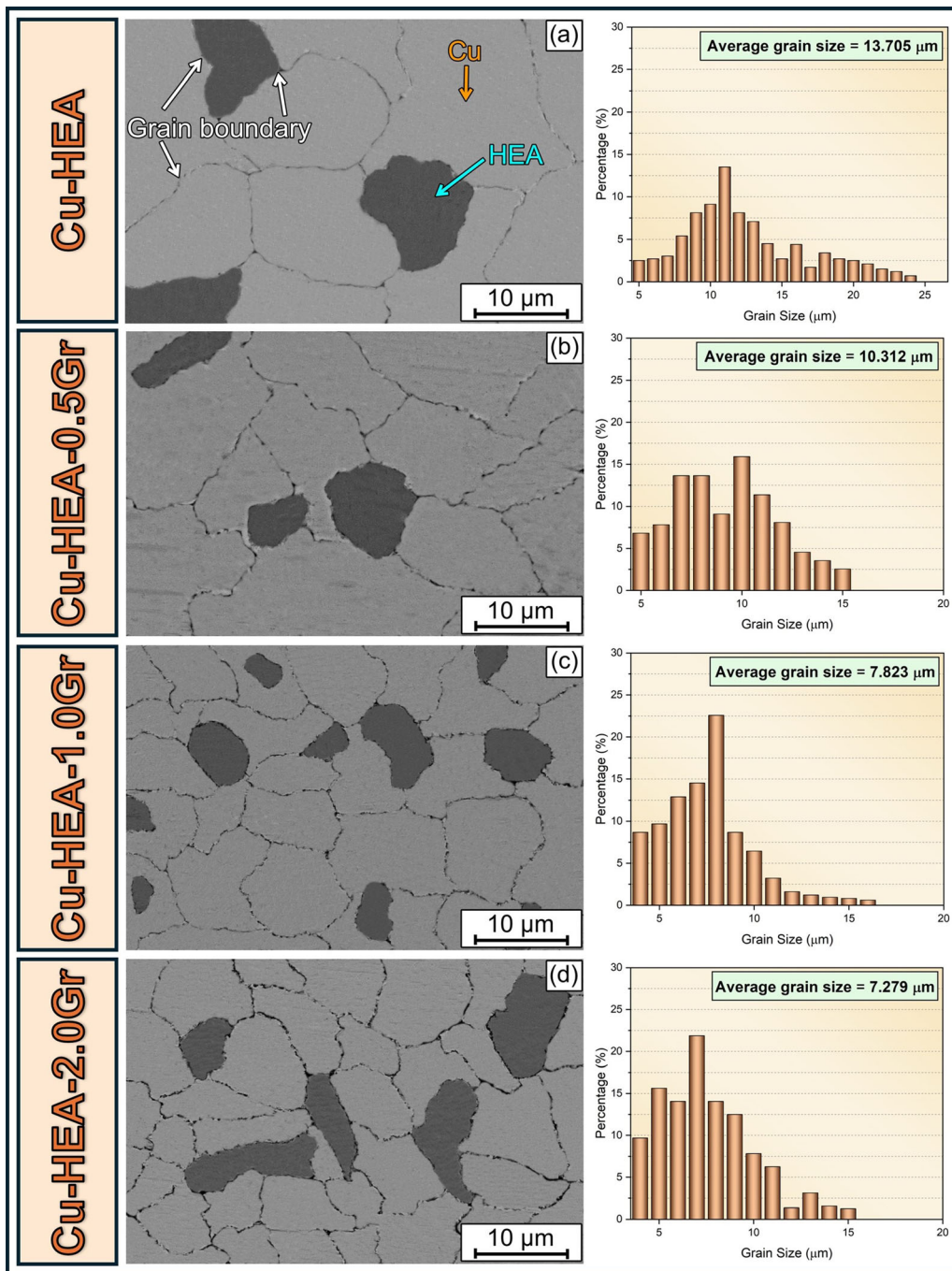


Fig. 4—Microstructure images and grain size graphs of Cu-HEA (a), Cu-HEA-0.5Gr (b), Cu-HEA-1.0Gr (c), Cu-HEA-2.0Gr (d).

grain size, which increases yield strength according to the Hall-Petch relationship.<sup>[17]</sup>

HEA phase, which is present at 20 pct in the composites, positively affects microstructural stability during and after sintering. HEAs are known for their “sluggish diffusion” effect, which slows grain kinetics at high temperatures. The literature emphasizes that slow atomic diffusion in HEAs stabilizes the microstructure by leading to high thermal stability, limited grain growth, and even the formation of nanoprecipitates. Therefore, the addition of HEA particles to the Cu matrix helps prevent excessive grain

growth during sintering; the HEA particles effectively “anchor” grain boundaries, providing dimensional stability. Indeed, one study reported that HEA particles in metal matrix composites increase dimensional stability and time-dependent deformation resistance by anchoring both grain boundaries and dislocation motion.<sup>[49]</sup>

Furthermore, the addition of HEA, as a metallic second phase, demonstrates good compatibility with the Cu matrix. Because HEAs possess high wettability and similar coefficients of thermal expansion to metal matrices, they do not create the interfacial tensions or reactions that ceramic reinforcements can cause. For

example, in a study in which HEA was added to AZ91 Mg alloy, no interphase reaction layer was observed between the HEA particles and the matrix, and good bonding was achieved.<sup>[50]</sup> Similarly, in Cu–HEA composites, because both HEA and Cu can have FCC structures or similar lattice structures, interfacial compatibility is high. This compatible interface ensures effective closure of voids during sintering and minimizes residual stresses due to the low thermal expansion mismatch.<sup>[37]</sup> Consequently, the HEA addition helps maintain microstructure stability without negatively affecting densification during sintering and contributes to the preservation of grain structure at high temperatures. This improves the long-term stability and mechanical performance of the composite.

Experimental results show that the 2.0 wt pct graphene-reinforced sample achieved the highest wear resistance. This superior performance is directly linked to the microstructural properties. Literature indicates that Cu composites containing approximately 1.0 wt pct graphene achieve the highest strength, while mechanical properties begin to decline at higher graphene contents due to interfacial adhesion problems. In this context, the selection of a 1.0 wt pct graphene content yielded the best hardness results, likely because it provided a near-optimal reinforcement amount. In terms of wear performance, the superiority of the 2.0 wt pct graphene composite stems from both the increased hardness and the solid-lubricant effect of graphene. Graphene forms a thin film between metal surfaces during friction, lowering the coefficient of friction, thus reducing adhesive wear between the two surfaces. Indeed, in Cu/graphene composites, the decrease in the coefficient of friction as graphene is added results in lower wear volumes under the same load. According to Archard's law of wear, higher hardness translates into lower wear. The 2.0 wt pct graphene composite exhibits the lowest wear rate due to the synergy of these two effects, both lowering the coefficient of friction (due to the lubricating effect of graphene) and higher surface hardness. For example, the literature reports wear volume reductions of up to 99 pct and a friction coefficient of 0.2 for Cu-based materials containing graphene, demonstrating graphene's tribological effectiveness.<sup>[51,52]</sup> The 2.0 wt pct graphene content in our composite minimizes surface damage by forming a protective carbon layer on the sample surface during wear.

The EDX maps in Figures 5 and 6 clearly show that copper is the dominant continuous matrix phase in both samples. The highest Cu content in Table I (similar ratios in both samples) confirms that copper forms the matrix. HEA elements such as Fe, Ni, Cr, Cu, and Al appear homogeneously distributed throughout all regions in both maps. This observation indicates that the HEA components are dissolved as stable, discrete phases within the copper matrix. According to Table II, no carbon detector signal was found in the Cu–HEA sample, while a C content of 1.95 pct was detected in the Cu–HEA–2.0Gr sample. This difference confirms the absence of carbon in Figure 5 and the clear presence of carbon in Figure 6. The carbon signal is particularly concentrated in the grain boundary regions, indicating

that the added graphene precipitates and accumulates primarily at the grain boundaries (Figure 6). This phenomenon has been reported similarly in literature: graphene nano cells have been observed to accumulate and stabilize grain boundaries in metal matrix composites.<sup>[53]</sup> Therefore, the distribution in Figure 6 clearly demonstrates that graphene particles accumulate and concentrate at the grain boundaries. As seen in Table II, the weight ratios of Cu, Fe, Ni, Cr, and Al are significantly similar in both samples. The only difference is the 1.95 pct carbon added to the Cu–HEA–2.0Gr sample. This indicates that graphene was successfully incorporated into the composite, while the unchanged proportions of other elements ensured that the copper matrix and HEA phase remained stable. The stable single-phase structures of high-entropy alloys are explained by the lower Gibbs free energy due to high entropy of mixing.

### C. XRD Analysis

In three different XRD patterns analyzed in this study, the HEA pattern data belong to the powder sample ground by mechanical milling for 25 hours, while the Cu–HEA and Cu–HEA–2.0Gr patterns belong to the compact composite samples obtained after SPS processing (Figure 7). The matrix phase in all samples is Cu, with HEA and graphene as reinforcement phases. In the XRD pattern of the HEA powder, characteristic diffraction peaks belonging to the BCC phase at 44.5, 64.6, and 82.3 deg and to the FCC phase at 51.7 deg were observed, revealing that HEA has a dual-phase structure. The stable formation of both FCC and BCC phases in HEA as a result of prolonging mechanical milling is also supported in the literature. Indeed, Abbas *et al.* (2024) reported that long-term mechanical milling promotes dual-phase formation rather than phase separation in HEAs with similar compositions.<sup>[54]</sup> In the XRD pattern of the Cu–HEA composite sample after SPS, three main peaks (JCPDS card no: #04-0836) corresponding to the 43.4 deg (111), 50.5 deg (200), and 74.2 deg (220) planes of the Cu element were observed quite intensely; this shows that the crystal structure of the Cu matrix phase is preserved with SPS. In addition, the BCC (110) peak (44.5 deg) of HEA appears quite weak, suggesting that the HEA phase can be partially dissolved with the matrix during the sintering process. In the hybrid Cu–HEA–2.0Gr sample, the main Cu peaks are still dominant, while the BCC (110) reflection remains weak and becomes even less intense. This behavior should not be interpreted as a direct loss of phase stability solely based on intensity; rather, it can be related to XRD-intensity-dependent factors such as preferred orientation, grain refinement, and increased microstrain introduced by graphene, which typically broaden and attenuate secondary-phase reflections. Consistent with the literature, graphene does not introduce new diffraction peaks in Cu-matrix composites; instead, it mainly affects the microstructure through interfacial phenomena and refinement effects.<sup>[55]</sup> Therefore, the XRD results confirm the dominance of the Cu matrix after SPS and show that the BCC phase of the

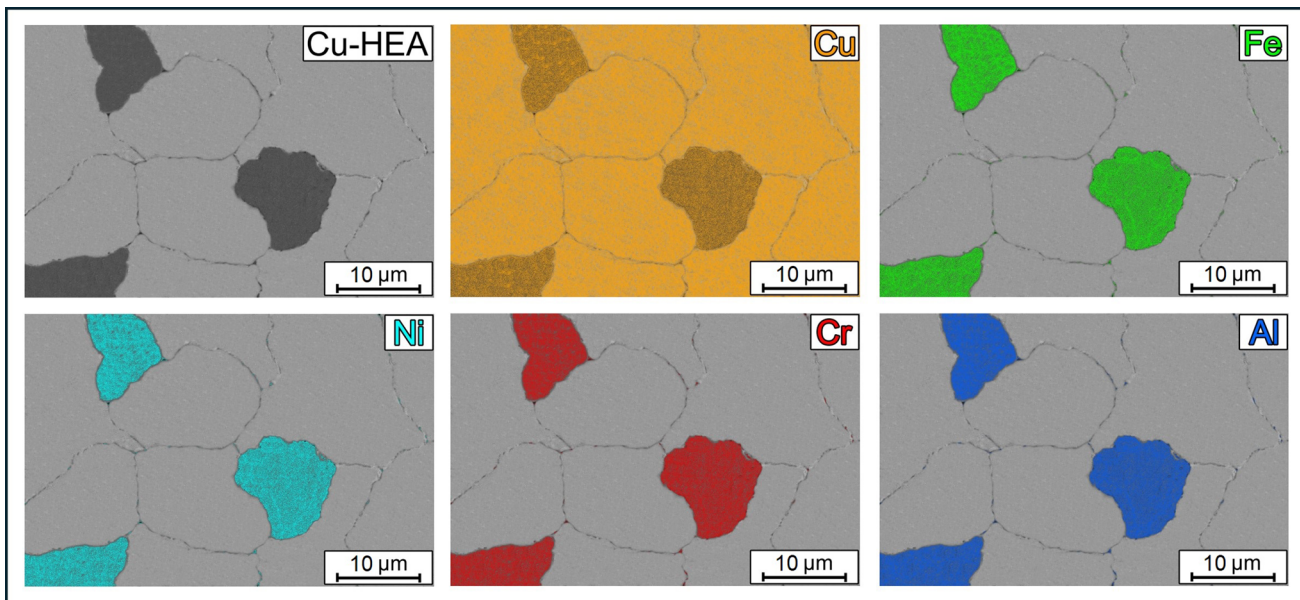


Fig. 5—EDX mapping analysis of Cu-HEA composite sample.

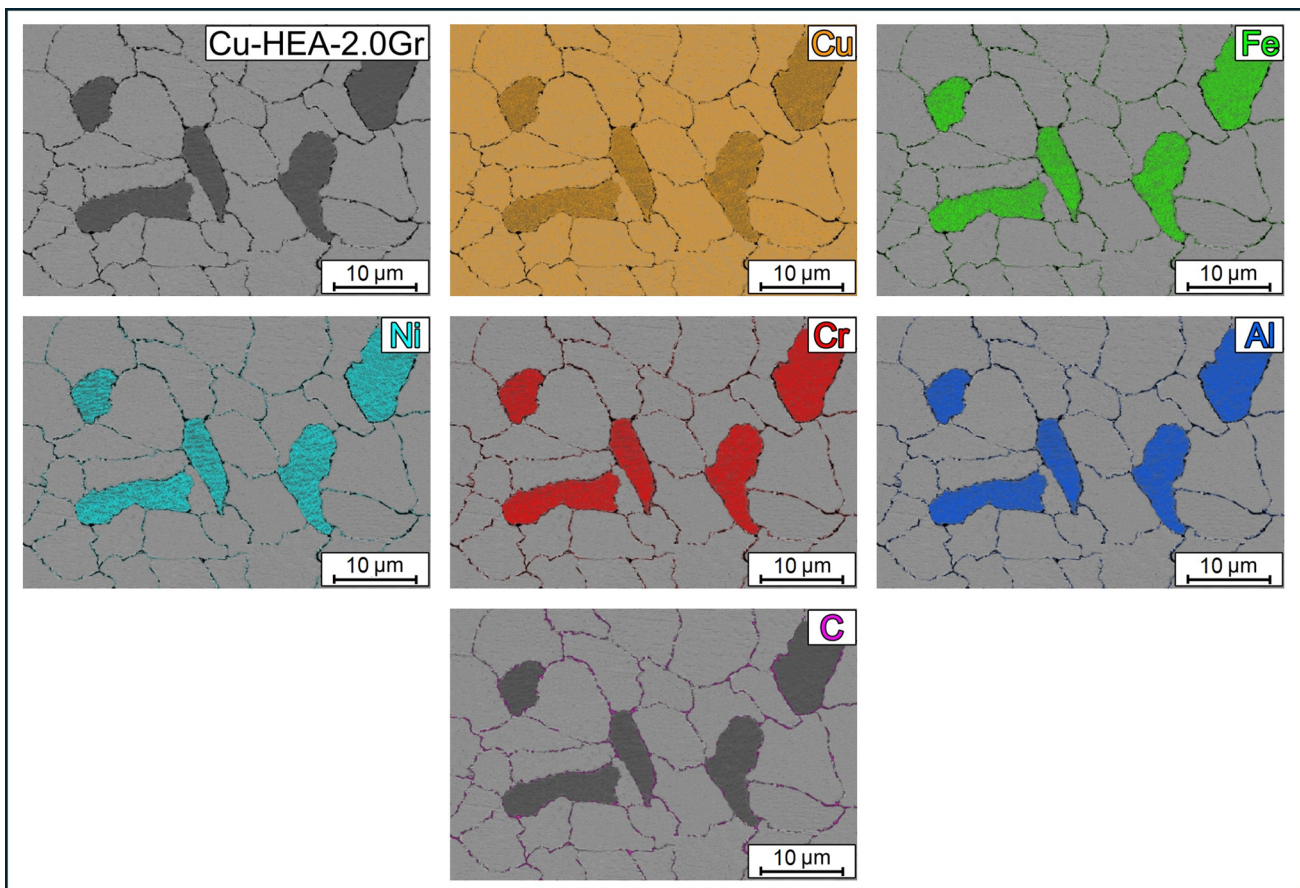


Fig. 6—EDX mapping analysis of Cu-HEA-2.0Gr hybrid composite.

HEA remains present, with a reduced peak intensity in the graphene-containing composite. This change reflects microstructural modifications induced by graphene

addition rather than a loss of phase stability, and it is expected to influence the final microstructure and mechanical behavior of the composites.

**Table II. Weight Fractions (Wt Pct) of the Elements Identified by EDX Analyses Performed on the SEM Micrographs of the Produced Samples (Fig. 4)**

| Sample Code  | Cu    | Fe   | Ni   | Cr   | Al   | C    |
|--------------|-------|------|------|------|------|------|
| Cu-HEA       | 83.95 | 4.01 | 4.02 | 3.98 | 4.04 | 0    |
| Cu-HEA-0.5Gr | 83.51 | 4.25 | 3.89 | 3.79 | 4.10 | 0.46 |
| Cu-HEA-1Gr   | 83.22 | 4.12 | 3.92 | 3.83 | 3.98 | 0.93 |
| Cu-HEA-2.0Gr | 82.02 | 4.02 | 3.99 | 3.99 | 4.03 | 1.95 |

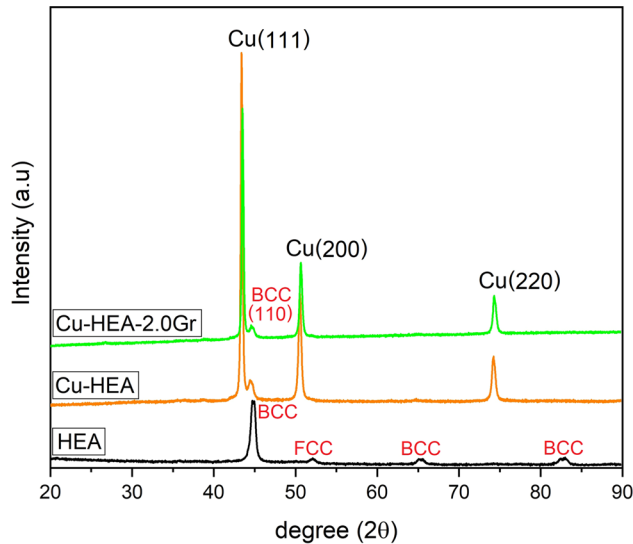


Fig. 7—XRD analyses of HEA, Cu-HEA, and Cu-HEA-2.0Gr samples.

#### D. Relative Density

Figure 8 illustrates the variation in the relative density values of the specimens fabricated *via* SPS. Examination of these values clearly shows that the relative density decreases gradually with increasing graphene content. The Cu-HEA specimen exhibits the highest density of 97.51 pct. Despite containing as much as 20 pct HEA, achieving such a high density is primarily attributed to the highly effective sintering capability of the SPS process combined with the presence of Cu within the HEA composition. Since Cu exists both in the matrix and in the HEA particles, a more compatible interfacial condition is formed during sintering, which promotes the development of sintering necks and facilitates densification. Upon the addition of graphene, the density decreases to 96.84 pct in the Cu-HEA-0.5Gr sample, to 96.13 pct in the Cu-HEA-1.0Gr sample, and to 94.92 pct in the Cu-HEA-2.0Gr sample. It is noteworthy that, despite the relatively high amount of graphene reinforcement, the reduction in density remains moderate, which confirms the overall effectiveness of the SPS process. The observed decrease with increasing graphene content is attributed to the inherently high surface area of graphene, which acts as a physical barrier between particles, reduces direct metal-metal contact, and restricts the formation and growth of sintering necks.<sup>[56,57]</sup> As shown in Figure 6,

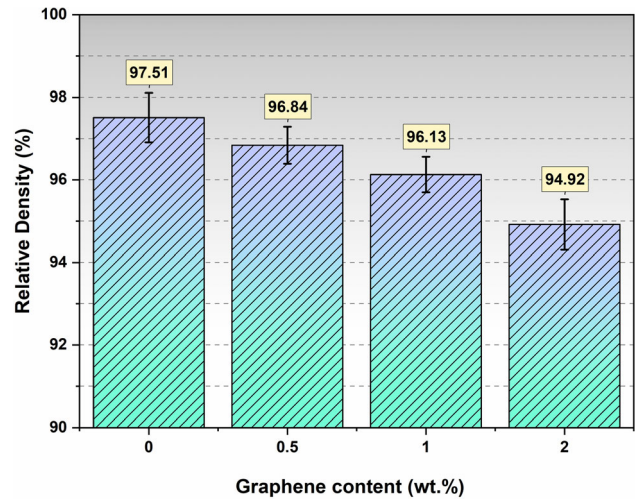


Fig. 8—Relative density values of fabricated samples.

the amount of graphene accumulated along grain boundaries increases with reinforcement content, which further impedes densification. In addition, the lamellar structure of graphene provides a solid-lubricant effect, thereby reducing the degree of microscale plastic deformation that must occur under the applied pressure during SPS. This effect limits the collapse of pores and hinders full consolidation.<sup>[58,59]</sup> With increasing graphene content, cold welding between particles becomes further suppressed, which diminishes particle bonding and increases the persistence of fine, poorly bonded regions. Graphene sheets may also form discontinuous three-dimensional networks, promoting open-pore continuity and further decreasing the overall density. Moreover, the Joule heating mechanism intrinsic to SPS becomes less uniform due to the anisotropic and heterogeneous electrical conductivity introduced by graphene, leading to localized regions with insufficient thermal energy for proper neck growth.<sup>[60]</sup> Consequently, increasing graphene content results in noticeable reductions in relative density, with the most pronounced decrease observed in the specimen containing the highest reinforcement level of 2.0 wt pct graphene.

#### E. Hardness

Figure 9 presents the hardness values of the fabricated specimens. The Cu-HEA sample exhibits an initial hardness of 127 HV, which can be attributed to the multi-principal-element nature of the HEA particles, whose inherently high hardness provides a significant strengthening contribution to the Cu matrix. During the SPS process, the rapid thermal cycle and enhanced diffusion promote the formation of strong metallurgical bonds between Cu and the HEA particles, which further amplifies the strengthening effect of the HEA phase. With the addition of graphene, a moderate increase in hardness is observed. The hardness increases to 132 HV for the Cu-HEA-0.5Gr sample and reaches 134 HV for the Cu-HEA-1.0Gr sample. Although the increase

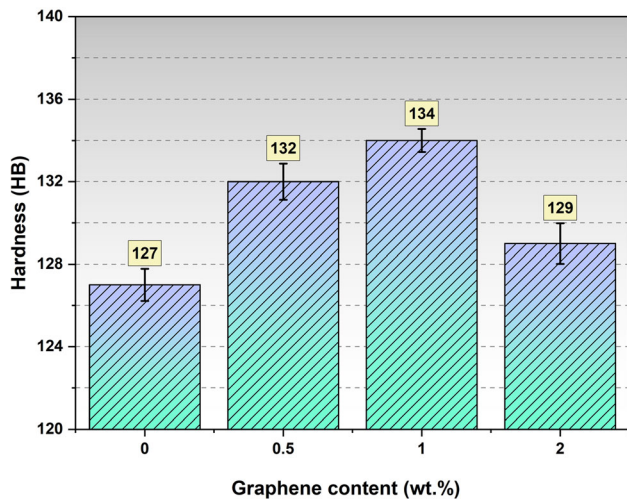


Fig. 9—Hardness values of fabricated samples.

between 0.5 and 1.0 wt pct graphene is relatively small, the overall improvement compared to the graphene-free composite suggests a beneficial reinforcement effect. This enhancement can be associated with the high elastic modulus of graphene and its ability to contribute to load transfer within the composite structure.<sup>[61]</sup> When uniformly dispersed, graphene nanosheets may act as obstacles to dislocation motion, contributing to a strengthening mechanism analogous to the Orowan bypass process. Additionally, the lamellar morphology of graphene may support interfacial strengthening at grain boundaries and particle–matrix interfaces.<sup>[47]</sup> However, when the graphene content is increased to 2.0 wt pct, the hardness decreases to 129 HV. This reduction is attributed to the tendency of graphene sheets to agglomerate at higher concentrations, resulting in non-uniform distribution and the formation of weak local regions. Such agglomerations diminish effective load transfer, limit interfacial bonding between the matrix and reinforcement phases, and consequently suppress the overall hardening response. Furthermore, an excessive amount of graphene reduces the extent of metal–metal contact between particles, leading to sub-optimal densification during SPS, which also contributes to the decline in hardness relative to the 1.0 wt pct graphene-reinforced sample.

#### F. Wear Properties

Figure 10 illustrates the variation of the coefficient of friction (CoF) for the Cu–HEA and Cu–HEA/graphene composites as a function of the repeated number of reciprocating cycles. The addition of graphene into the Cu–HEA matrix significantly changes the frictional behavior of the Cu–HEA system and leads to a remarkable reduction in the coefficient of friction. The unreinforced Cu–HEA alloy exhibits an average CoF in the range of 0.65 to 0.75, characterized by noticeable fluctuations but maintaining overall stability during the entire test period (black line). This elevated friction coefficient is characteristic of an adhesion-dominated

tribological regime, which is commonly observed in ductile metallic alloys under dry sliding conditions.<sup>[62]</sup> The increased CoF arises from intense plastic deformation caused by the deeper penetration of the counter ball, which enlarges the real contact area during sliding and consequently increases the coefficient of friction.<sup>[44,63,64]</sup> Moreover, the pronounced fluctuations observed in the Cu–HEA composites may also arise from the dynamic behavior of HEA particles during sliding. These particles may detach or become dislodged from the matrix and roll between the contacting surfaces, acting as a third body within the interface. This third-body effect disrupts stable sliding and contributes to the observed fluctuations.<sup>[65,66]</sup> The addition of 0.5 wt pct graphene (red line) causes a marked decrease in the CoF of the Cu–HEA alloy; however, the behavior of the CoF is quite unstable, with the CoF exhibiting significant fluctuations after stabilizing within the range of 0.45 to 0.55.<sup>[67]</sup> This unstable frictional response confirms that the graphene concentration is below the critical level for sufficient lubrication between the contacting surfaces. This behavior observed in the friction coefficient can be attributed to a poorly formed tribofilm, which undergoes intermittent rupture during sliding and consequently gives rise to localized high-frictional contacts. When the graphene content in the matrix reaches 1.0 wt pct (blue line), the CoF notably declines to the 0.10 to 0.15 level and exhibits a smooth, stable profile throughout the sliding process. This behavior indicates that a continuous and protective graphene film forms on the surface and effectively maintains self-lubrication during sliding.<sup>[67–69]</sup> Following this trend, the composite containing 2.0 wt pct graphene (green line) exhibits even superior performance, achieving an ultra-low CoF in the range of 0.05 to 0.08. These results show that once a sufficient amount of graphene is present, the excellent lubricating film can continuously renew itself, reducing interfacial shear stress, preventing adhesion between surfaces, and lowering energy loss throughout the sliding process.

The wear-track topographies and corresponding volumetric wear-loss data presented in Figures 11 and 12 evidently demonstrate a strong correlation between the graphene content and the improved wear resistance of the Cu–HEA/graphene composites. The unreinforced Cu–HEA alloy displays the most severe wear damage, with a maximum wear-track depth of approximately  $155 \pm 8 \mu\text{m}$  and a track width around  $1775 \pm 22 \mu\text{m}$ . This deep and irregular wear morphology [seen in Figures 11(a) and (e)] is indicative of a wear mechanism governed by severe adhesion, abrasion, and plastic deformation, consistent with the high and unstable coefficient of friction (0.65 to 0.75) observed in Figure 10. In contrast, the addition of graphene into the matrix drastically changes the surface morphology and reduces material removal. With 0.5 wt pct graphene, the maximum wear depth decreases to  $35 \pm 5 \mu\text{m}$ , corresponding to a 77.4 pct reduction relative to the Cu–HEA reference, while the wear-track width narrows to  $980 \pm 13 \mu\text{m}$  [Figures 11(b) and (e)]. A more pronounced improvement is achieved for 1.0 wt pct graphene, as the wear depth is reduced to only

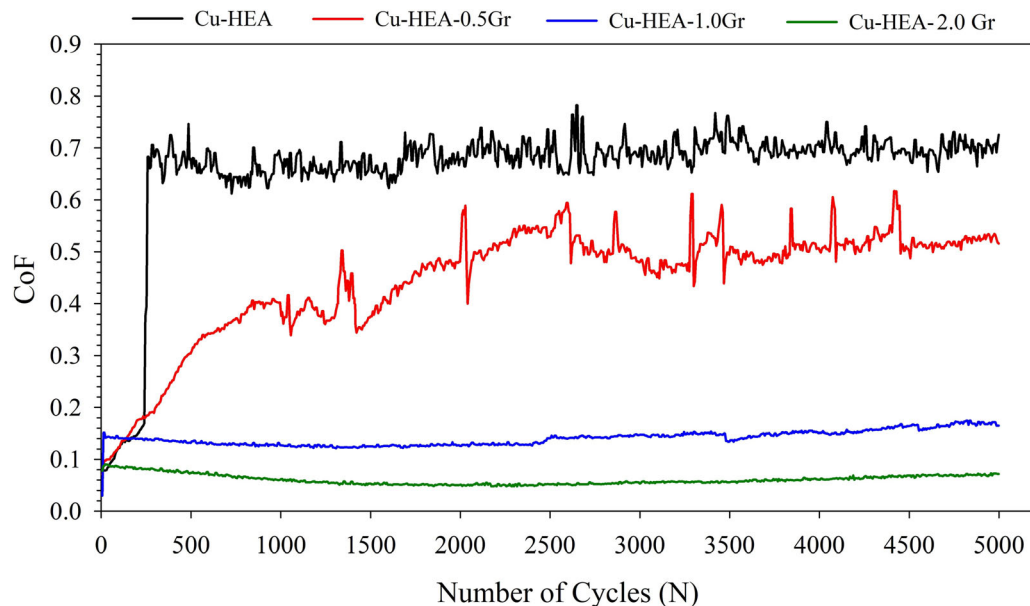


Fig. 10—Variation in the coefficient of friction (CoF) as a function of the number of cycles (N) for the Cu–HEA and Cu–HEA/graphene composites (Color figure online).

$2.0 \pm 0.05 \mu\text{m}$  ( $\sim 98.7$  pct reduction) accompanied by a substantial narrowing of the wear track to  $291 \pm 16 \mu\text{m}$ , indicating a stable and uniform contact interface [Figures 11(c) and (e)]. The 2.0 wt pct graphene addition further advances this performance, with an ultra-shallow wear depth of  $1.2 \pm 0.02 \mu\text{m}$  ( $\sim 99$  pct reduction) and an extremely narrow track width of  $113 \pm 11 \mu\text{m}$  [Figures 11(d) and (e)]. The volumetric wear-loss results obtained from the 3D surface topography (given in Figure 12) exhibit a consistent trend with the wear-depth profiles. The Cu–HEA sample exhibits the highest volume loss of  $1775 \times 10^{-3} \text{ mm}^3$ , while the 0.5 wt pct graphene composite shows a steep reduction to  $280 \times 10^{-3} \text{ mm}^3$  ( $\sim 84$  pct decrease). The reduction becomes more pronounced with higher graphene contents, with the 1.0 and 2.0 wt pct composites exhibiting wear losses of only  $6.4 \times 10^{-3} \text{ mm}^3$  ( $\sim 99.6$  pct reduction) and  $2.3 \times 10^{-3} \text{ mm}^3$  ( $\sim 99.9$  pct reduction), respectively. Similar findings have also been reported in previous studies, where graphene-based solid lubricants were shown to form stable lubricating tribofilms that effectively decrease the interfacial adhesion and abrasion.<sup>[51,70]</sup> The remarkable coherence among the declining friction coefficient, the reduced wear depths, and the substantially lower volume losses provides clear evidence that a stable, lubricating graphene film dominates the interfacial dynamics at higher graphene contents, thereby transforming the wear mechanism from a severe adhesive and abrasive regimes in Cu–HEA to a graphene film-controlled mild wear regime. Among all tested compositions, the Cu–HEA–2.0Gr composite exhibits the excellent tribological performance (most stable and lowest CoF, negligible wear), owing to the formation of a continuous, self-replenishing graphene film that effectively suppresses metal-to-metal adhesion and provides a sustained low-friction interface essential for long-term reliability in industrial applications.

To clarify the dominant wear mechanisms and their correlation with the tribological performance, detailed SEM examinations were carried out on the worn surfaces of the Cu–HEA composites with different contents of graphene, and the obtained SEM images are given in Figures 13 and 14. The morphology of the Cu–HEA worn surface, marked by significant roughness and obvious plastic deformation, suggests the prevalence of an intense adhesion-driven wear regime. There are also many wear debris and flakes on the worn surface [Figures 13(a) through (a3)]. The lack of a protective graphene-derived tribofilm increases the surface damage, as repeated sliding of the hard 100Cr6 ball on the soft, unreinforced sample causes severe plastic deformation and the formation of both deep and shallow grooves. This formation is also confirmed by the high friction coefficient value measured for the Cu–HEA composite sample, as well as by the fluctuations observed in its instantaneous variations (Figure 10). During the wear test, the soft Cu–HEA alloy tends to adhere to the ball surface and subsequently retransfer onto the sample surface. This repeated material transfer results in a progressively rougher and more heterogeneous surface morphology on the specimen. On the other hand, deep cracks also exist on the deformed and smeared layers as clearly seen in Figure 13(a1). These cracks progress under repetitive stresses during the sliding, causing the large lamellar debris and a significant rise in wear loss for the Cu–HEA specimens (Figure 12). These findings are also confirmed by the worn debris, which consists heavily of deformed and flake-like particles collected from the wear surfaces [Figure 13(a3)]. Incorporating 0.5 wt pct graphene into the matrix partially modifies the surface morphology. Localized tribofilm patches [Figure 13(b1)] are observed, which provide a limited reduction in both the coefficient of friction and wear loss; however, their discontinuous

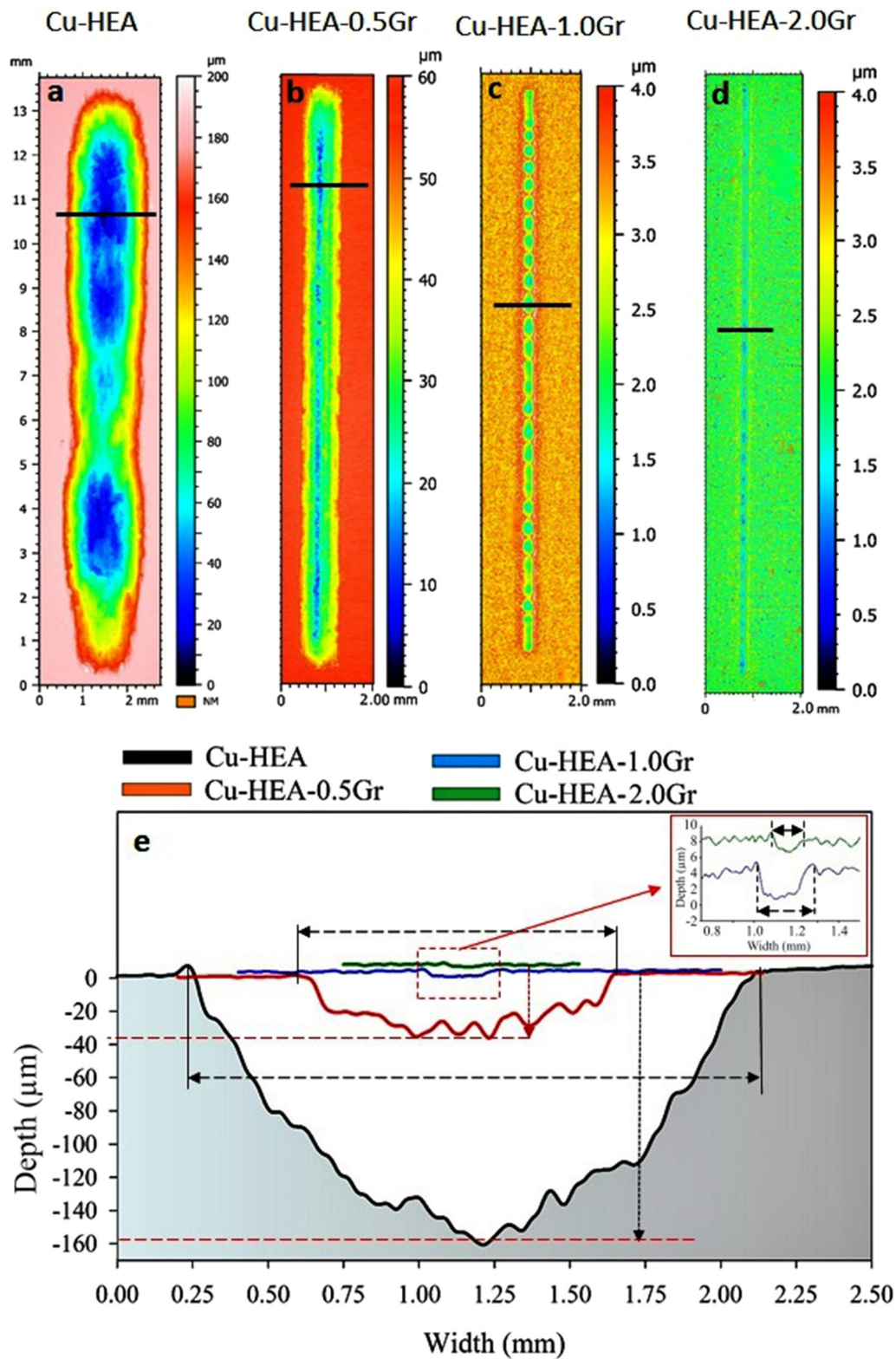


Fig. 11—3D surface topography and the cross section of the wear tracks (e) for Cu-HEA (a), Cu-HEA-0.5Gr (b), Cu-HEA-1.0Gr (c), Cu-HEA-2.0Gr (d).

nature results in intermittent and unstable lubrication between the contacting pairs. The sudden peaks and drops observed at specific periods in the instantaneous

friction coefficient (Figure 10) support the formation of an unstable-localized film and intermittent film break-downs during the sliding. Furthermore, the addition of a

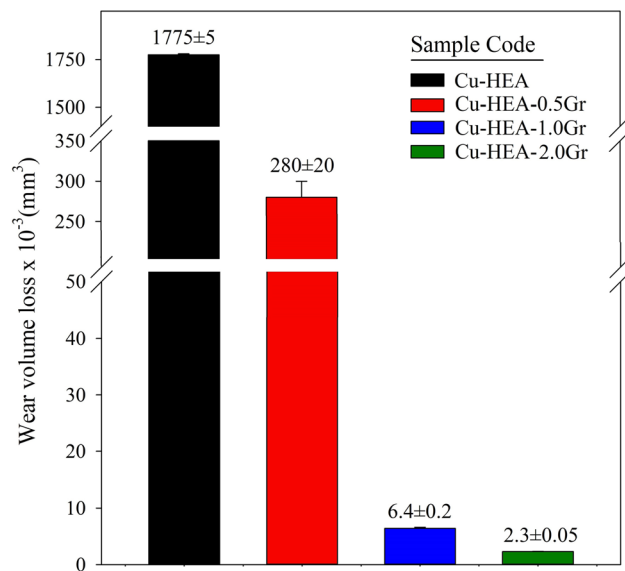


Fig. 12—Variations of volume loss of Cu–HEA composites with different graphene content under the same test conditions.

small amount of graphene promotes fragmentation of the wear debris, narrows the wear scar, and gradually smoothens the worn surface [Figures 13(b) through (b3)]. Despite the observed partial surface modifications, the presence of plastic deformation on the worn surface [Figures 13(b2) and (b3)] and the relatively high average friction coefficient value ( $\mu_a \sim 0.50$  seen in Figure 10) of the Cu–HEA–0.5Gr sample indicate that the wear process is still largely driven by adhesion. Increasing the graphene content in the Cu–HEA matrix to 1.0 pct facilitated the formation of a more consistent lubricating film, while also significantly altering the nature of the surface interactions [Figures 14(a) through (a2)]. The wear surface exhibits a markedly smooth and homogeneous appearance, with virtually no evidence of material transfer and abrasive effects associated with plastic deformation. It is well known that graphene exhibits excellent self-lubricating properties.<sup>[17]</sup> Once the graphene content exceeds a critical threshold, a more stable and continuous tribofilm forms on the worn surface, leading to a significant reduction in wear losses compared to the Cu–HEA and Cu–HEA–0.5Gr specimens (Figures 12, 13). The reduction in the average friction coefficient to the level of 0.15, along with the noticeable decrease in CoF fluctuations compared to the Cu–HEA and Cu–HEA–0.5Gr samples, confirms the formation of a stable and continuous tribofilm on the surface and a significantly homogeneous contact between the surfaces [Figures 10, 14(a) through (a2)]. Increasing the graphene content in the matrix to the 2.0 wt pct level optimized the film formation [Figures 14(b) through (b2)] resulting in peak tribological performance. Specifically, the friction coefficient was minimized to 0.08 level, and the wear loss reached  $2.3 \times 10^{-3} \text{ mm}^3$ . This wear loss represents an exceptional 99 pct reduction compared to the Cu–HEA reference sample. This improvement is attributed to graphene's ability to redirect crack initiation and propagation, which

stabilizes the lubricating film, lowers friction, and enhances wear resistance. Moreover, higher graphene content refines the copper matrix grains, increasing hardness and further improving wear behavior (Figures 4, 9, 12).<sup>[71,72]</sup> Graphene mainly locates at grain boundaries, where it acts as a barrier to dislocation motion and crack propagation, while also helping a load transfer within the matrix.<sup>[73]</sup>

To further support the identified wear mechanisms, SEM and EDS analyses were also carried out on the surfaces of the counter balls, and the obtained images and findings are presented in Figure 15. The counter-ball surfaces have different appearances that clearly reveal the differences in wear mechanisms observed in the Cu–HEA and Cu–HEA/graphene composites. For the ball tested against the Cu–HEA reference composite, a heavily smeared transfer layer is evident, indicating intense plastic deformation and dominant adhesive interactions at the sliding interface [Figures 15(a) and (a1)]. The high coefficient of friction measured for this sample may be directly attributed to this smeared layer, which is repeatedly formed and fractured during sliding. Moreover, under combined compressive and shear stresses, the smeared tribolayer repeatedly adheres and fractures. This recurring instability may be seen as the primary reason for the marked fluctuations in friction of the Cu–HEA samples. In contrast, for the ball tested against the Cu–HEA composite containing 0.5 wt pct graphene, both the real contact area and the smeared regions are noticeably reduced [Figures 15(b) and (b1)]. This reduction is partially attributed to the limited lubricating effect provided by the low content of graphene within the matrix. However, the presence of adhered and plastically deformed material on the ball surface indicates that adhesion-driven wear is still active for Cu–HEA–0.5Gr composite. On the other hand, increasing the graphene content within the composite matrix to the level of 1.0 wt pct and subsequently to 2.0 wt pct drastically alters the tribological interactions occurring at the counter-ball surface. For the ball tested against the 1.0 wt pct graphene-reinforced composite, only a very limited real contact area is observed, and the smeared layer is extremely thin and localized [Figures 15(c) and (c1)]. In the case of the 2.0 wt pct graphene composite, the ball surface displays only minimal and weakly detectable smearing marks [Figures 15(d) and (d1)]. The counter ball surface, much like the composite's wear track [Figures 14(b) through (b2)], appears remarkably smooth, clearly demonstrating the effective lubrication offered by 2.0 wt pct graphene content. Overall, these observations evidently demonstrate that the addition of graphene directly influences the wear mechanisms at the sliding interface. The reduction of plastic deformation observed with increasing graphene content is a clear indication of graphene's effective lubrication behavior. The substantial reduction in the coefficient of friction with higher graphene content further supports this conclusion.

To evaluate the obtained results in a broader context, the findings were compared with studies on some Cu–graphene composites in the literature.<sup>[74]</sup> As summarized in this review, the addition of graphene to

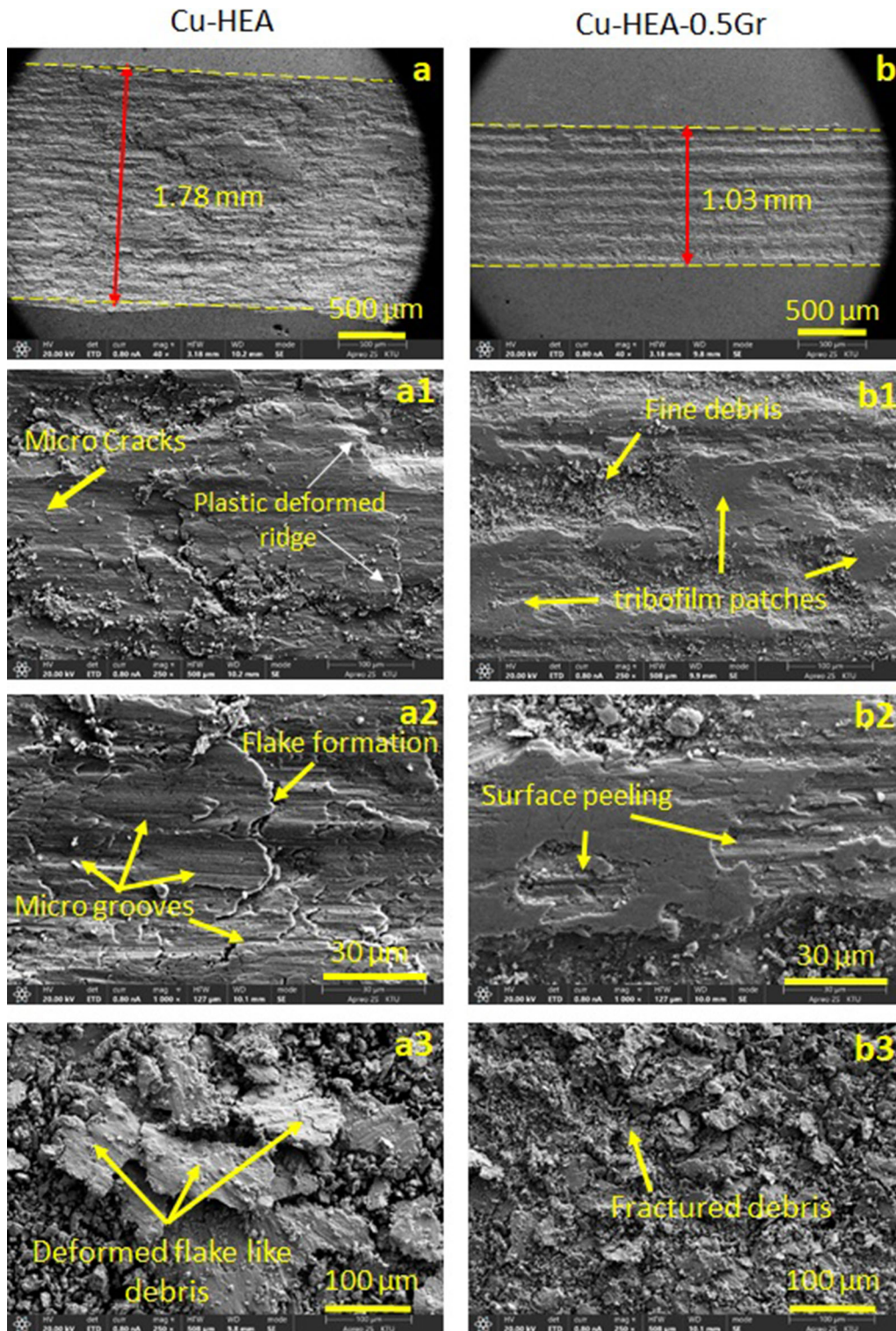


Fig. 13—SEM images of worn surfaces of Cu-HEA (a) and Cu-HEA-0.5Gr (b) composites.

copper matrices generally leads to grain refinement, improved load transfer, and a significant reduction in friction and wear due to the formation of a lubricating tribofilm. These trends are in good agreement with the results obtained in the present study. In particular, the dramatic reduction in the coefficient of friction and wear

volume observed with increasing graphene content can be attributed to the same mechanisms discussed in the review, namely graphene-induced solid lubrication and grain boundary stabilization. However, while the review primarily focused on conventional Cu-graphene systems, the present study expands on these findings by

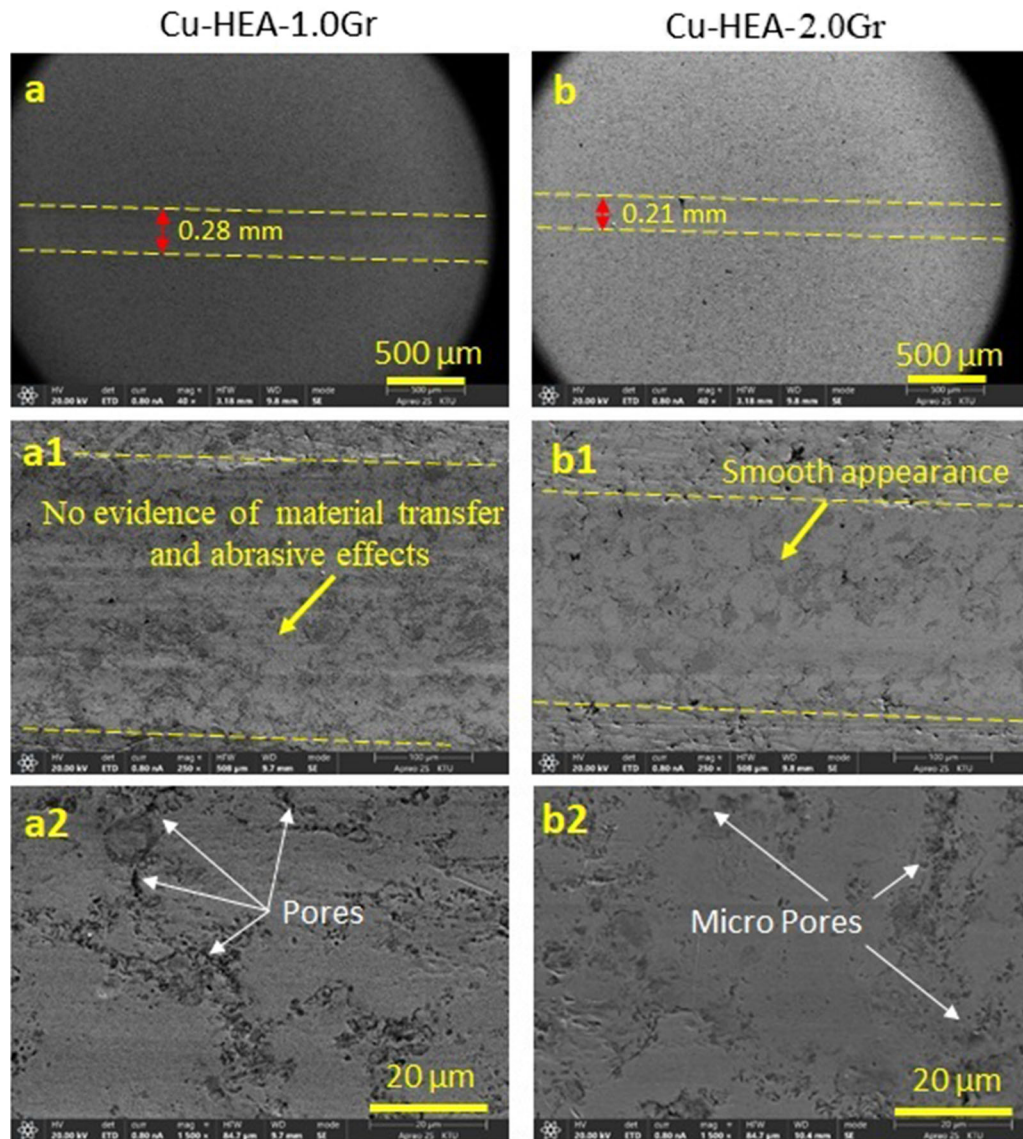


Fig. 14—SEM images of worn surfaces of Cu-HEA-1.0Gr (a) and Cu-HEA-2.0Gr (b) composites.

demonstrating that the addition of graphene to a Cu matrix already reinforced with AlCrFeCuNi high-entropy alloy particles provides a synergistic effect. The HEA phase enhances load-carrying capacity and microstructural stability, while graphene dominates the tribological response by forming a continuous and self-renewing tribofilm. Therefore, the present results are fully consistent with the established Cu-graphene literature and also provide new insights into the role of graphene in hybrid Cu-HEA composite systems processed via spark plasma sintering.

#### IV. CONCLUSION

In this study, Cu/AlCrFeCuNi/Graphene hybrid metal matrix composites were successfully fabricated *via* the spark plasma sintering (SPS) method, and the role of varying graphene content on the microstructural,

mechanical, electrical, and tribological properties were systematically investigated. The key findings are as follows:

- (1) With increasing graphene content, the relative density decreased from 97.51 to 94.92 pct, whereas the hardness increased up to the 1.0 wt pct graphene addition, reaching 134.16 HB, followed by a slight decrease to 129.25 HB at 2.0 wt pct.
- (2) The wear volume decreases dramatically from  $1775 \times 10^{-3} \text{ mm}^3$  in the unreinforced Cu-HEA alloy to only  $2.3 \times 10^{-3} \text{ mm}^3$  (~99 pct reduction), and the coefficient of friction drops from 0.65 to 0.75 to 0.05 to 0.08 for the composite containing 2.0 wt pct graphene, and this improvement arises from the formation of a stable graphene-based lubricating film that

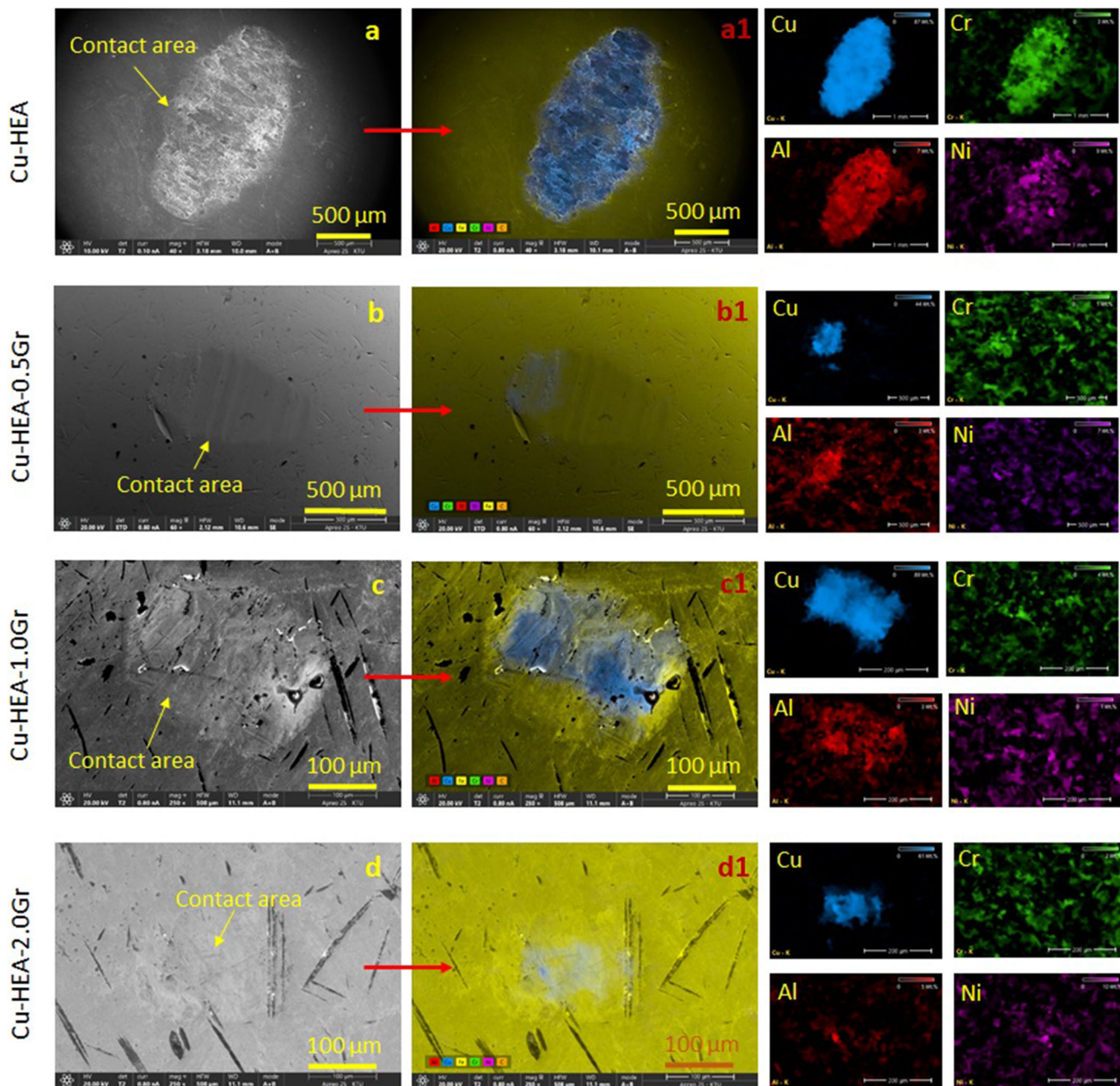


Fig. 15—SEM images and EDX analysis taken from the contact surfaces of the counter balls for the Cu-HEA (a), Cu-HEA-0.5Gr (b), Cu-HEA-1.0Gr (c), Cu-HEA-2.0Gr (d).

minimizes interfacial shear and suppresses metal-to-metal adhesion.

- (3) The Cu-HEA/graphene composites exhibit progressively smoother and more stable friction coefficients with increasing graphene content, in contrast to the pronounced fluctuations observed in the unreinforced Cu-HEA alloy, and this improvement is directly associated with the excellent friction film provided by graphene's self-lubricating capability.
- (4) The progressive transition from severe adhesion-dominated wear in the unreinforced Cu-HEA alloy to a graphene-controlled mild wear regime demonstrates that increasing

graphene content fundamentally alters the wear mechanism by enabling the formation of a continuous, self-lubricating tribofilm that suppresses plastic deformation, prevents material transfer, and redirects crack propagation, thereby achieving exceptionally low friction and nearly complete elimination of wear.

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## AUTHOR CONTRIBUTIONS

Harun Yanar contributed toward writing—review & original draft, supervision, methodology, and validation. Müslim Çelebi contributed toward writing—review & editing, writing—original draft, and validation. Dursun Murat Sekban contributed toward writing & editing formal analysis and data curation. Serdar Özkaya contributed toward conceptualization, visualization, methodology, and writing & editing. Abdullah Hasan Karabacak contributed toward supervision, writing—review & editing, writing—original draft, and validation. Ertuğrul Çelik contributed toward methodology, conceptualization, validation, and writing & editing.

## DATA AVAILABILITY

The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding author.

## CONFLICT OF INTEREST

Author Harun YANAR was employed by the company UTS Software and Engineering Services Industry Trade Limited Company. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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## REFERENCES

1. S.G. Jia, P. Liu, F.Z. Ren, B.H. Tian, M.S. Zheng, and G.S. Zhou: *Wear*, 2007, vol. 262, pp. 772–77. <https://doi.org/10.1016/j.wear.2006.08.020>.
2. X.C. Ma, G.Q. He, D.H. He, C.S. Chen, and Z.F. Hu: *Wear*, 2008, vol. 265, pp. 1087–92. <https://doi.org/10.1016/j.wear.2008.02.015>.
3. W.-J. Zhang, L. Huang, X.-J. Mi, H.-F. Xie, X. Feng, and J.H. Ahn: *CMT*, 2024, vol. 1, p. e13. <https://doi.org/10.1002/cmt2.13>.
4. H. Yang, Z. Ma, C. Lei, L. Meng, Y. Fang, J. Liu, and H. Wang: *Sci. China Technol. Sci.*, 2020, vol. 63, pp. 2505–17. <https://doi.org/10.1007/s11431-020-1633-8>.
5. R.P. Minneci, E.A. Lass, J.R. Bunn, H. Choo, and C.J. Rawn: *Int. Mater. Rev.*, 2021, vol. 66, pp. 394–425. <https://doi.org/10.1080/09506608.2020.1821485>.
6. K. Ganesan: *Int. J. Fatigue*, 2026, vol. 203, p. 109282. <https://doi.org/10.1016/j.ijfatigue.2025.109282>.
7. G. Li, N. Peng, D. Sun, and S. Sun: *J. Tribol.*, 2014, <https://doi.org/10.1115/1.4028486>.
8. A. Fathy and O. El-Kady: *Mater. Des.*, 2013, vol. 46, pp. 355–59. <https://doi.org/10.1016/j.matdes.2012.10.042>.
9. D. Kumar, R. Seetharam, and K. Ponappa: *J. Alloys Compd.*, 2024, vol. 972, p. 172732. <https://doi.org/10.1016/j.jallcom.2023.172732>.
10. Y. Wang, Y. Chen, J. Xie, J. Ni, T. Zhang, S. Wang, and L. Yin: *J. Alloys Compd.*, 2023, vol. 960, p. 170882. <https://doi.org/10.1016/j.jallcom.2023.170882>.
11. V. Pandey, R. Seetharam, and H. Chelladurai: *J. Alloys Compd.*, 2024, vol. 1002, p. 175095. <https://doi.org/10.1016/j.jallcom.2024.175095>.
12. Y. Liu, J. Chen, Z. Li, X. Wang, P. Zhang, and J. Liu: *Vacuum*, 2021, vol. 184, p. 109882. <https://doi.org/10.1016/j.vacuum.2020.109882>.
13. S. Özkaya: *JOM*, 2025, vol. 77, pp. 5510–25. <https://doi.org/10.1007/s11837-025-07401-3>.
14. Y.-F. Yan, S.-Q. Kou, H.-Y. Yang, S.-L. Shu, F. Qiu, Q.-C. Jiang, and L.-C. Zhang: *Int. J. Extrem. Manuf.*, 2023, vol. 5, p. 32006. <https://doi.org/10.1088/2631-7990/acdb0b>.
15. A.K.B. Hanumantharaya, C. Prasanna, C.C. Ragavendra, C.S. Beekam, L.S. Bolivar, M. Nagaral, and S. Rangappa: *J. Mech. Behav. Mater.*, 2021, vol. 30, pp. 199–206. <https://doi.org/10.1515/jmbm-2021-0020>.
16. D. Janas and B. Liszka: *Mater. Chem. Front.*, 2018, vol. 2, pp. 22–35. <https://doi.org/10.1039/C7QM00316A>.
17. P. Hidalgo-Manrique, X. Lei, R. Xu, M. Zhou, I.A. Kinloch, and R.J. Young: *J. Mater. Sci.*, 2019, vol. 54, pp. 12236–89. <https://doi.org/10.1007/s10853-019-03703-5>.
18. J. Sun and S. Du: *RSC Adv.*, 2019, vol. 9, pp. 40642–61. <https://doi.org/10.1039/C9RA05679C>.
19. H. Fu, G. Yan, M. Li, H. Wang, Y. Chen, C. Yan, C.-T. Lin, N. Jiang, and J. Yu: *RSC Adv.*, 2019, vol. 9, pp. 42481–88. <https://doi.org/10.1039/C9RA09201C>.
20. F. Zhang, K. Yang, G. Liu, Y. Chen, M. Wang, S. Li, and R. Li: *Compos. Part A*, 2022, vol. 160, p. 107051. <https://doi.org/10.1016/j.compositesa.2022.107051>.
21. V. Kumar, A. Singh, and G. Gautam: *Interactions*, 2024, <https://doi.org/10.1007/s10751-024-02200-9>.
22. V.K. Parikh, V. Patel, D.P. Pandya, and J. Andersson: *Heliyon*, 2023, vol. 9, p. e13558. <https://doi.org/10.1016/j.heliyon.2023.e13558>.
23. A. Jamwal, P. Mittal, R. Agrawal, S. Gupta, D. Kumar, K.K. Sadasivuni, and P. Gupta: *J. Compos. Mater.*, 2020, vol. 54, pp. 2635–49. <https://doi.org/10.1177/0021998319900655>.

24. A. Anand and S.K. Tiwari: *IOP Conf. Ser. Mater. Sci. Eng.*, 2022, vol. 1248, p. 12087. <https://doi.org/10.1088/1757-899X/1248/1/012087>.
25. Y. Qin, Y. Tian, Y. Peng, L. Luo, X. Zan, Q. Xu, and Y. Wu: *J. Alloys Compd.*, 2020, vol. 848, p. 156475. <https://doi.org/10.1016/j.jallcom.2020.156475>.
26. N. Saheb, Z. Iqbal, A. Khalil, A.S. Hakeem, N. Al Aqeeli, T. Laoui, A. Al-Qutub, and R. Kirchner: *J. Nanomater.*, 2012, vol. 2012, p. 983470.
27. M. Pellizzari and G. Cipolloni: *Materials (Basel)*, 2020, vol. 13, p. 2602.
28. M. Chmielewski, K. Pietrzak, A. Strojny-Nędza, K. Kaszyca, R. Zybala, P. Bazarnik, M. Lewandowska, and S. Nosewicz: *Sci. Sinter.*, 2017, vol. 49, pp. 11–22.
29. M.R. Akbarpour and S. Alipour: *Ceram. Int.*, 2017, vol. 43, pp. 13364–70.
30. Z. Yang, Y. Ge, X. Zhang, B. Shangguan, Y. Zhang, and Y. Wang: *Materials (Basel)*, 2019, vol. 12, p. 2825.
31. X. Zhu, Y. Jiang, G. Shen, and L.I. Zhou: *Trans. Nonferrous Met. Soc. China*, 2024, vol. 34, pp. 3789–3821.
32. D.V. Dudina and V.G. Tokarev: *Trans. Indian Inst. Met.*, 2025, vol. 78, p. 113.
33. S. Ali, F. Ahmad, P.S.M.M. Yusoff, N. Muhamad, E. Oñate, M.R. Raza, and K. Malik: *Compos. Part A*, 2021, vol. 144, p. 106357.
34. S.C. Tjong and K.C. Lau: *Mater. Lett.*, 2000, vol. 43, pp. 274–80.
35. J.P. Tu, Y.Z. Yang, L.Y. Wang, X.C. Ma, and X.B. Zhang: *Tribol. Lett.*, 2001, vol. 10, pp. 225–28.
36. X. Gao, H. Yue, E. Guo, S. Zhang, L. Yao, X. Lin, B. Wang, and E. Guan: *J. Mater. Sci. Technol.*, 2018, vol. 34, pp. 1925–31.
37. P.J. Mane, M. Shantharaja, B. Manne, B.S. Raju, and JOM.: *J. Miner. Met. Mater. Soc.*, 2023, vol. 75, pp. 4421–34.
38. R. Zhu, Y. Li, Y. Sun, J. Feng, and W. Gong: *J. Alloys Compd.*, 2023, vol. 940, p. 168906.
39. S. Seenivasan, K. SooryaPrakash, S. Nandhakumar, and P.M. Gopal: *Proc. Inst. Mech. Eng. Part C*, 2021, vol. 235, pp. 5555–66.
40. M. Wu, Z. Chen, C. Huang, K. Huang, K. Jiang, and J. Liu: *RSC Adv.*, 2019, vol. 9, pp. 39883–92.
41. J.F. Li, L. Zhang, J.K. Xiao, and K.C. Zhou: *Trans. Nonferrous Met. Soc. China*, 2015, vol. 25, pp. 3354–62.
42. L. Zeng, Y. Liang, and P. Chen: *Front. Mater. Sci.*, 2024, vol. 18, p. 240704.
43. W. Zhu, W. Liu, H. Tan, and J. Yang: *Vacuum*, 2025, vol. 242, p. 114772.
44. G. Purcek, H. Yanar, O. Saray, I. Karaman, and H.J. Maier: *Wear*, 2014, vol. 311, pp. 149–58. <https://doi.org/10.1016/j.wear.2014.01.007>.
45. T. Talapaneni, V. Chaturvedi, Consolidation of Mechanically Alloyed Powders, in: *Mech. Alloy. Nov. Mater. Process. Appl. Prop.*, Springer, 2024, pp. 111–69.
46. A. Cuccu, S. Montinaro, R. Orrù, G. Cao, D. Bellucci, A. Sola, and V. Cannillo: *Ceram. Int.*, 2015, vol. 41, pp. 725–36.
47. M. Çelebi, A. Çanakçı, and S. Özkaya: *Adv. Powder Technol.*, 2025, vol. 36, p. 104835.
48. M. Çelebi, A. Çanakçı, and S. Özkaya: *Powder Technol.*, 2025, vol. 449, p. 120439.
49. D. Górniewicz, K. Karczewski, Z. Bojar, and S. Jóźwiak: *Materials (Basel)*, 2023, vol. 16, p. 7361.
50. C. Chiu and H.-H. Chang: *Materials (Basel)*, 2021, vol. 14, p. 6520.
51. D. Almonti, D. Salvi, N. Ucciardello, and S. Vesco: *Materials (Basel)*, 2024, vol. 17, p. 6017.
52. H.S. Maharana, P.K. Rai, and A. Basu: *J. Mater. Sci.*, 2017, vol. 52, pp. 1089–1105.
53. Z. Yang, F. Deng, Z. Tao, S. Yan, H. Ma, M. Qian, W. He, Z. Zhang, Y. Liu, and L. Wang: *Materials (Basel)*, 2023, vol. 16, p. 5534.
54. M.A. Abbas, W.M. Sadek, and S.A. Ibrahim: *Trans. Indian Inst. Met.*, 2024, vol. 77, pp. 1661–69.
55. D.D. Phuong, P. Van Trinh, P.N. Minh, A.A. Shtertser, and V.Y. Ulianitsky: *Crystals*, 2024, vol. 14, p. 1000.
56. L. Zhang, Q. Ye, X. Zeng, S. Liu, H. Chen, Y. Tao, X. Yu, and X. Wang: *Electronics*, 2023, vol. 13, p. 53.
57. M. Ghatge and M. Shrivastava: *IEEE Trans. Electron Devices*, 2015, vol. 62, pp. 4139–47.
58. D. Berman, A. Erdemir, and A.V. Sumant: *Mater. Today*, 2014, vol. 17, pp. 31–42.
59. S. Rengifo, A comparison between graphene and WS<sub>2</sub> as solid lubricant additives to aluminum for automobile applications, 2015.
60. X. Ding, Z. He, J. Li, X. Xu, and Z. Li: *Nanoscale*, 2024, vol. 16, pp. 12309–28.
61. K. Chu, F. Wang, Y. Li, X. Wang, D. Huang, and H. Zhang: *Carbon N. Y.*, 2018, vol. 133, pp. 127–39.
62. H. Yanar and A. Coskun: *Wear*, 2025, vol. 574, p. 206100.
63. W. Wang, R. Cui, S. Zhang, Y. Sun, and C. Wan: *Surf. Coatings Technol.*, 2024, vol. 479, p. 130486.
64. G. Purcek, H. Yanar, D.V. Shangina, M. Demirtas, N.R. Bochar, and S.V. Dobatkin: *J. Alloys Compd.*, 2018, vol. 742, pp. 325–33. <https://doi.org/10.1016/j.jallcom.2018.01.303>.
65. P.K. Verma and A. Singh: *Tribol. Int.*, 2024, vol. 191, p. 109055. <https://doi.org/10.1016/j.triboint.2023.109055>.
66. Y. Zhang, G. Lei, K. Luo, P. Chen, C. Kong, and H. Yu: *Tribol. Int.*, 2022, vol. 175, p. 107868. <https://doi.org/10.1016/j.triboint.2022.107868>.
67. S. Shu, Q. Yuan, W. Dai, M. Wu, D. Dai, K. Yang, B. Wang, C.-T. Lin, T. Wuebben, and J. Degenhardt: *Mater. Des.*, 2021, vol. 203, p. 109586.
68. A.D. Moghadam, E. Omrani, P.L. Menezes, and P.K. Rohatgi: *Compos. Part B*, 2015, vol. 77, pp. 402–20.
69. C. Ayyappadas, A. Muthuchamy, A. Raja Annamalai, and D.K. Agrawal: *Adv. Powder Technol.*, 2017, vol. 28, pp. 1760–68. <http://doi.org/10.1016/j.appt.2017.04.013>.
70. A.I. Khair and A. Ibrahim: *Int. J. Miner. Metall. Mater.*, 2022, vol. 29, pp. 161–67.
71. X. Chen, Z. Han, X. Li, and K. Lu: *Sci. Adv.*, 2016, vol. 2, p. e1601942.
72. Y. Kim, J. Lee, M.S. Yeom, J.W. Shin, H. Kim, Y. Cui, J.W. Kysar, J. Hone, Y. Jung, and S. Jeon: *Nat. Commun.*, 2013, vol. 4, p. 2114.
73. K. Chu, F. Wang, X. Wang, and D. Huang: *Mater. Sci. Eng. A*, 2018, vol. 713, pp. 269–77.
74. P. Hidalgo-Manrique, X. Lei, R. Xu, M. Zhou, I.A. Kinloch, and R.J. Young: *J. Mater. Sci.*, 2019, vol. 54, pp. 12236–89.

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