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Microstructure, Physical and Mechanical Properties of Cu/WC-TiC-Co/GNP Nano-Composites by Powder Metallurgy Technique

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ABSTRACT

In the present study we have successfully bonded two dissimilar materials: copper metal and ceramic WC-TiC-Co/GNP. It was prepared by mixing 10 wt. In the case of WC-TiC-Co with different contents graphene, as reported in our previous studies [45], we prepared a kind volume fraction sample: (0, 0.25, 0.5, 0.75, and 1 wt. %) then had copper coating with electro-less precipitation method, generating five samples. Following this, Cu/WC-TiC-Co/GNP nano-composites were compressed at 900 MPa and sintered in a pure hydrogen atmosphere for 150 minutes at the temperature of 1000°C. The study examined many factors of these composites such as the relative density, microstructure, hardness and electrical/thermal properties. Thus, the density was found to decrease, showing an inverse relation with relative content of GNS in composites. Nevertheless, different from this situation the hardness, electrical and thermal conductivity increased as the graphene content reaches to 10 wt %. This result shows that the nano-composites prepared by introducing graphene into Cu/WC-TiC-Co matrix have high mechanical, electrical and thermal properties. The microstructural analysis revealed the distribution and interfacial bonding of graphene within the matrix, shedding light on the mechanisms underlying the observed property improvements. Through this detailed study, the design and enhancement of new nano-composite materials for different uses in electronics, aerospace, automotive, among others is well provided in the study.

KEYWORDS: Cu/WC-TiC-Co Composites; Copper graphene nano-composites; Electro-Less Deposition Process; Powder Metallurgy; Electrical Properties, Thermal Properties; Hardness.

1. INTRODUCTION

Efficiency in electronic appliances such as projectors, laptops, high-power chips, and computers depends a lot on effective heat management [1]. When current passes through these devices, they generate a significant amount of heat, necessitating aggressive thermal management to maintain their temperatures within optimal ranges [2,3]. Attaining and maintaining the peak operational temperature needed to attain maximum efficiency demands the use of strong cooling techniques that could be accommodated within the physically restricted dimensions of the electronic device [4.5]. Among the different cooling techniques proposed, heat sinks are one of the most common and low-cost hardware solutions with the view to controlling the thermal state of a microelectronic circuit [6]. The heat sinks conventionally comprise metallic structures that are in close interaction with hot surfaces of electronic components such as CPUs used in computing devices [7].

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The material selection for heat sink manufacturing is based on certain physical properties, such as high thermal conductivity and low CTE [8]. Copper and aluminum are mainly used owing to their favorable thermal properties. Both the metals have impressive thermal conductivity but suffer from considerable thermal expansion [9,10]. Thus, several efforts have been made by researchers in enhancing their mechanical properties by reducing CTE [11]. The main strategy involves the strengthening of copper and aluminum matrices with compatible materials, improving their mechanical strength with a reduction in thermal expansion at reduced loss in thermal conductivity [12]. The second class consists of new incoming materials that, from the beginning, have shown excellent thermal conductivity and low thermal expansion, such as graphene, diamond, and carbon nanotubes [13,14]. Graphene is an outstanding 2-D carbon nanofiller, which, because of its superior mechanical strength, ultra-high thermal conductivity of 5300 W/mK, low thermal expansion, high electrical conductivity, and very large specific surface area (SSA 500-1200 m^2/g), exhibits as a highly promising reinforcement candidate [15]. The research being conducted to combine graphene nanosheets with copper has resulted in some breakthrough findings. The obtained tensile strength for graphene-enhanced composite materials was very much higher-up to 500 times more than the value of the raw material [16,17]. Graphene can also enhance electrical and thermal conductivity.

A parallel study on graphene with respect to magnesium-based metal matrix nanocomposites derived conclusions relating to the attainment of homogeneous multilayer graphene dispersion throughout the matrix, strengthening mechanical properties [18]. Although graphene is promising, challenges remain, especially on how to establish strong interfacial bonding between copper and graphene due to its intrinsic lack of wetting and reactivity [19,20]. The compromised heat transfer interface leads to pore formation detrimental to the physical and mechanical properties of the composite [21]. To overcome these interfacial bonding deficiencies, the development of an electroless copper coating has emerged as the dominant strategy to improve composite integrity [22]. The current study is concerned with the impact of incorporated graphene on the microstructural, densitometric, hardness, electrical, and thermal conductivity properties of WCCo nanocomposites with copper for possible application as advanced heat sinks [23,24].

2. EXPERIMENTAL WORK

2.1. Composite Material Preparation

Graphene nano-platelets used in this study have a thickness in the range of 2-10 nm, from the supplier ACS Material, LLC, whereas WC-TiC-Co powder of purity 99.97%, with a particle size of 0.5 - 1 μm is provided by Hart Metal Company. Cleaning has been done to ensure that graphene and WC-TiC-Co powder surfaces are clean [25]. Ionized water, sodium hydroxide, and acetone were provided from El-Nasr Chemical Company, Cairo, Egypt. A 10 wt.% sodium hydroxide solution was used to dissolve any organic impurities, followed by dipping the powders in acetone for another hour to remove non-organic impurities [26]. Finally, the powders were filtered using a Buchner funnel and Whitman No. 1 filter paper under vacuum. That can be considered a kind of vacuum filtration process that facilitates the separation of the liquid phase from the solid particles and allows for more efficient recovery of the powder [27]. After filtration, the collected powders were washed with deionized water to remove further impurities. Finally, filtered powders were kept in an electrical furnace for 2 hours at a temperature of 60°C to remove trace amounts of moisture [28].

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These powders were mixed with 10 wt.% WC-TiC-Co and different graphene percentages, 0, 0.25, 0.5, 0.75 and 1 wt.%, respectively, then coated with electroless copper deposition. Pre-treatment was necessary to assist the deposition of copper onto the mixed powders of graphene and WC-TiC-Co; this treatment was performed in the form of preliminary metalization, for which silver metal was used. Silver nitrate (purity 99.97%), ammonia (33%), and formaldehyde (concentration 38%) were obtained from El-Nasr Chemical Company. The metalization of the ceramic graphene and WC-TiC-Co powder surfaces was carried out with 2 wt.% silver deposition, done by dissolving silver nitrate in ionized water. Its pH was adjusted to 11 with ammonia solution, since this condition has already demonstrated the ability to optimize the interfacial bonding and dispersion of graphene in copper matrices, [29]. Then, the reaction was initiated with formaldehyde. The powders obtained were filtered and oven-dried in an electrical furnace at 60°C for 2 hours.

After the sensitization and metallization processes, electro-less copper deposition was performed. Copper (II) sulfate pentahydrate, potassium sodium tartrate, sodium hydroxide, and formaldehyde [30,31], all sourced from El-Nasr Chemical Company, Cairo, Egypt, were utilized for this coating process. The chemical composition of the electro-less copper coating bath adhered to previously established parameters, as detailed in Table 1.

Table 1: Chemical composition bath of copper electroless process at 60 °C.

Materials	weight
copper (II) sulphate, ($CuSO_4$)	70 g/l
potassium sodium tartrate, ($KNaC_4H_4O_6.5H_2O$)	170 g/l
sodium hydroxide, (NaOH)	50 g/l
formaldehyde	200 ml/l

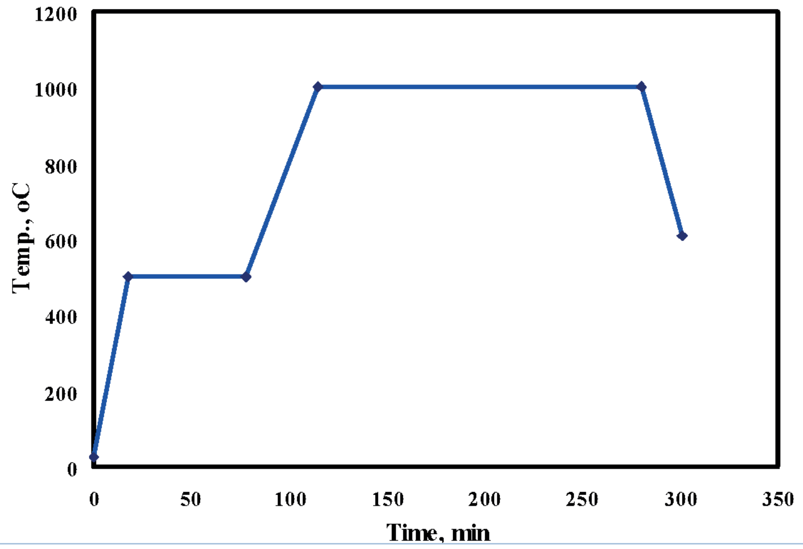

Fig. 1: Sintering cycle for Cu/WC-TiC-Co/graphene nano palettes composite.

These rigorous processes ensure that the coating of copper onto the graphene nanoplatelet and WC-TiC-Co powder is very well retained and effective for full analysis of properties and their possible applications [32]. The solution was then heated to 60°C to increase the rate of deposition of copper. At the completion of copper deposition, the graphene nanoplatelets and WC-TiC-Co powders were coated, filtered, and washed with distilled water.

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The materials were then dried in an electric furnace at 60°C for 2 hours [33,34]. For the preparation of composite samples, five different variations were prepared in the chemical composition: 0%, 0.25%, 0.5%, 0.75%, and 1 wt.% graphene nanoplatelets with the addition of 10 wt.% WC-TiC-Co powders as reinforcement in a copper matrix. It should be noted that a certain amount of CuO was formed during the electroless deposition process. To reduce the oxide content in the deposited copper and further improve the bonding between graphene nanoplatelets, WC-TiC-Co powders, and copper, the prepared nano-composite powders were heat-treated at 500°C for 60 min under an atmosphere of hydrogen. In the next step, the powder composite samples were compressed at 900 MPa by using the universal hydraulic press with a round die of a diameter of 12 mm [35]. Then, all the composites were sintered in a hydrogenic pure atmosphere at 1000°C for 150 minutes with a heating cycle explained in Fig.1. The heating step at 500°C for one hour served to evacuate residual gases entrapped within pores and to remove oxide residues from the compacted samples [36]. Such careful processing maximizes the integrity and performance of the resulting composite materials and therefore provides a sound basis for the subsequent characterization and evaluation in a range of applications.

2.2. Characterization and Analysis

Archimedes' principle, explained in the standard ASTM B962-14, has been used as a basis of measurement for obtaining the density of the sintered composites within this research effort [37]. Using distilled water as the floating liquid, density measurements were carefully obtained at room temperature. With respect to measuring the relative density, an important parameter describing the densification of the sintered composites, it was done using the following well-known equation. This, still unexplained, formula stands for the principle behind the relative density determination; hence, it gives the quantitative measure of obtained density against the theoretical. Moreover, a detailed explanation of the experimental setup with sample preparation, measurement procedures, and methods of data analysis must be given to ensure strength and validity for the results obtained during the experiment itself. Moreover, full deliberation of the importance of relative density in explaining the structural integrity and performance characteristics of sintered composites is considered at the crossroads in materials science and engineering applications.

$$R_d = \frac{\rho_{Ar}}{\rho_t} \rho_{liquid} \quad [17] \tag{1}$$

where ρAr is the Archimedes density, ρt is the theoretical density, and ρliquid is the theoretical density. Accordingly, silicon carbide papers from 400 to 1200 grit were used in grinding the sintered PM composite samples to aid in preparation for analysis. Thereafter, the microstructure and morphologies of the sintered PM composites were carefully analyzed using a Scanning Electron Microscope (SEM) - Quanta FEG250. This provided enough information on the internal structure and surface characteristics of the composites. In addition to this, an X-ray Energy Dispersive Spectrometer was used, with a model D8 XPORT, to investigate the phase composition and crystal structure of the composites prepared. The test thus allowed for a comprehensive grasp of both the chemical composition and structural integrity of the composites. Meanwhile, the sintered composites were subjected to a test in order to find out their respective macro-hardness using a Vickers macro hardness tester. The parameters for testing included a loading force of 5 kg applied over a period of 15 seconds. Since this is a mechanical property being tested, the average of five readings was calculated to derive each hardness value as a way of developing a sound dataset.

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The electrical conductivity of the sintered composites was measured at room temperature using a precision measurement device from PHYWE SYSTEME GMBH, Gottingen, Germany. This test was very important for defining the electric behavior of composites for possible applications. To estimate the thermal conductivity values of composites, the so-called Wiedemann-Franz equation is used as it represents a good method of estimation of the thermal conductivity value by means of electrical conductivity. This supplementary analysis underlined the thermal transport properties of the composites in addition to both the mechanical and electrical characterizations of the latter, hence offering an overview of their properties in several aspects.

$$\frac{\lambda}{\sigma T} = \frac{\pi^2 k_B^2}{3e^2} = L = 2.443x\,10^{-8} w\,\Omega / k^2 \quad [17] \tag{2}$$

where, λ is thermal conductivity (W/mK), σ is electrical conductivity $\Omega.m^{-1}$, T is absolute temperature in degree Kelvin (293 k), KB is Boltzmann constant, and L is Lorentz number.

3. RESULTS AND DISCUSSION

3.1. Powder Characterization

Fig 2 presents high-resolution images depicting the particle morphology and size of WC-TiC-Co and graphene nano-sheets, along with their respective coatings. The composite examined comprises WC-TiC-Co with 1 wt.% graphene nano-platelets, surface-coated with copper. Examination of images (a) and (b) reveals the distinct characteristics of the constituents: WC-TiC-Co particles exhibit sub-rounded and irregular shapes and varying sizes, while graphene manifests as nano-layered sheets. Further analysis in images (c) and (d) unveils the deposition of copper nano-particles on both the WC-TiC-Co powders and graphene nano-sheets.

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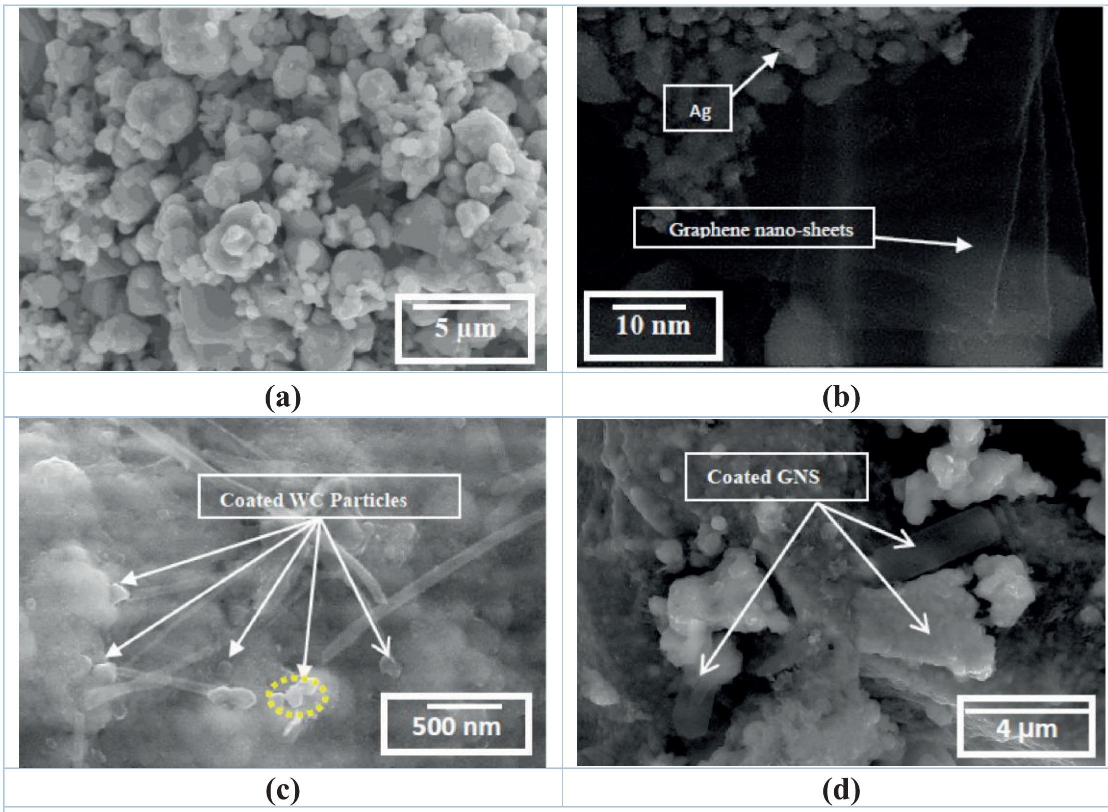

Fig. 2: SEM images of (a) WC-TiC-Co, (b) Graphene nano-sheets coated with silver, and (c, d) WC-TiC-Co / 1 wt.% GNP coated with copper.

This coating process enhances the interfacial bonding and dispersion uniformity within the composite, crucial for optimizing its mechanical and electrical properties. Additionally, the images provide valuable insights into the distribution and morphology of the constituents, facilitating a comprehensive understanding of the structure-property relationship in WC-TiC-Co/graphene composites. Such elucidation is crucial for fabrication process and fine-tuning the composite's performance for diverse applications, ranging from wear-resistant coatings to high-strength structural materials.

3.2. Density Measurement

The investigation into the impact of graphene nano platelets (GNPs) on the density of the Cu/WC-TiC-Co composite, as depicted in Fig. 3, provides significant insights into the composite's structural properties. Despite achieving high relative densities across all compositions, a notable trend emerges: the relative density of Cu/WC-TiC-Co composites declines with increasing weight percentage of GNP. This phenomenon can be elucidated by the lower density value of graphene compared to the other constituents of the composite; while graphene boasts a density of approximately 2.2 g/cm^3, WC-TiC-Co registers at 13.3 g/cm^3, and copper at 8.9 g/cm^3. Thus, the incorporation of a lighter material into a denser matrix inevitably lowers the overall density of the composite.

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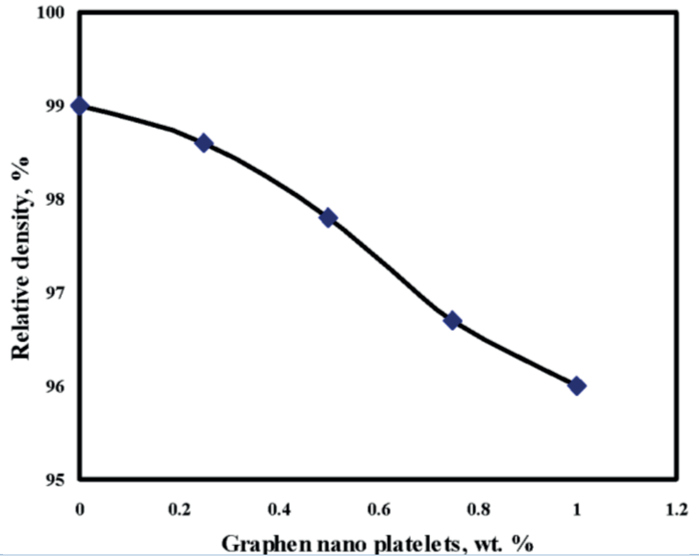
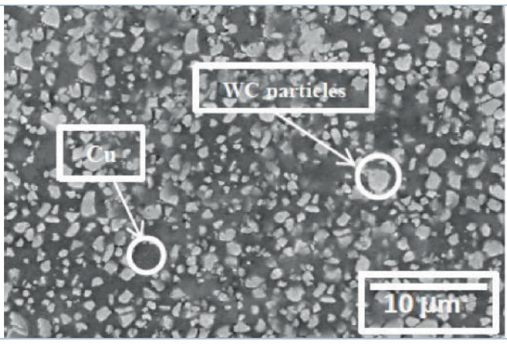


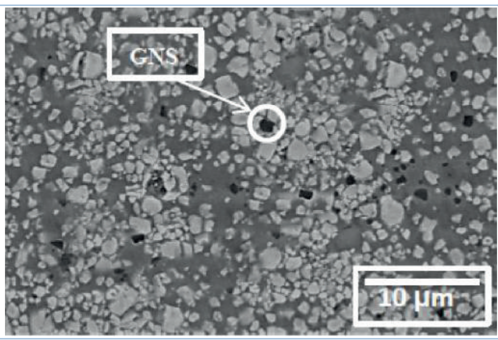
Fig. 3: The relative density of Cu/WC-TiC-Co/ GNP nano composites versus the graphene percent.

Further analysis reveals that the density reduction is intricately linked to the contact area between the diverse constituents of the composite. This factor assumes paramount importance, particularly as copper serves as the reinforced matrix accommodating two distinct materials. The microstructural examination unveils a lack of pores or voids within the composite, indicative of robust interfacial bonding. This phenomenon can be attributed to the coating of WC-TiC-Co/GNs mixture with copper, which enhances adhesion between all constituents of the composite. By mitigating surface energy disparities, the copper coating fosters strong interfacial interactions, thereby fortifying the structural integrity of the composite.

While the density increases with rising GNP percentages, material composition and microstructural features are very much interrelated with processing parameters. Upcoming research should discuss in more detail optimization of the distribution and orientation of GNPs in the composite matrix to improve further mechanical and thermal properties. Moreover, the densification process may also be improved in view of the adoption of new coating techniques and/or interfacial engineering, which opens a new way toward the preparation of advanced composite materials with engineered properties for a wide range of applications.



(a)



(b)

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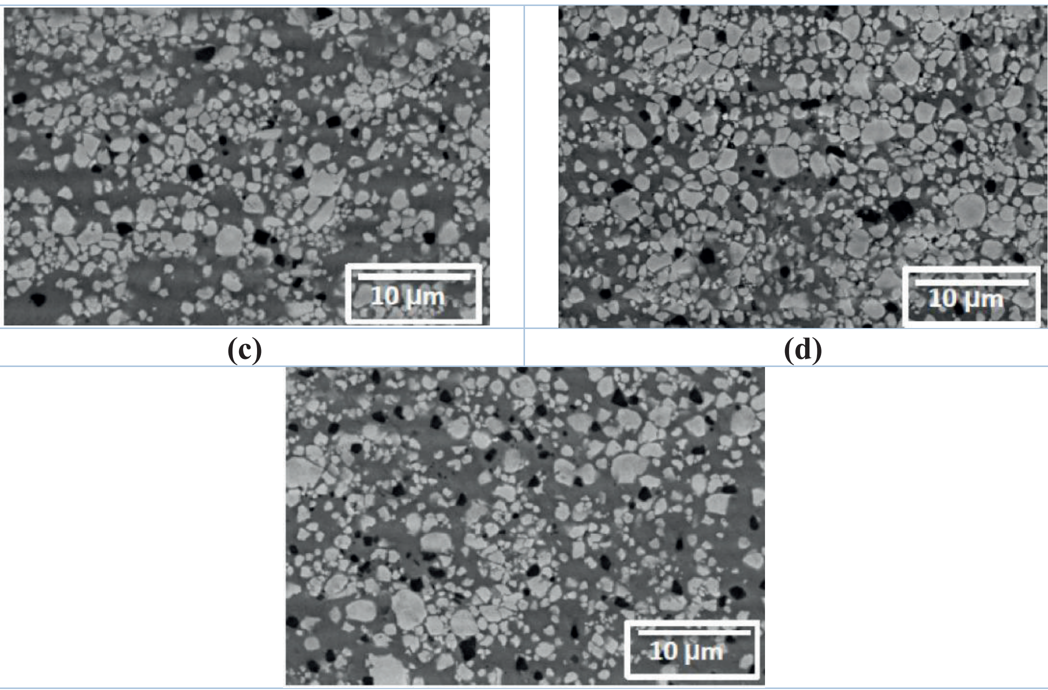

(c) (d)

(e)

Fig. 4: SEM for (a) 0 wt. % graphene (b) 0.25 wt. % graphene (c) 0.5 wt. % graphene (d) 0.75 wt. % graphene (e) 1 wt. % graphene sintered nano-composite.

3.3. Microstructure Investigation

Figure 4 presents the microstructural analysis along with the morphological characteristics of the sintered composites, which gives important information about the structural integrity and distribution of constituent phases within the composites. Apparently, different regions are distinguishable, having various shades like gray, dark gray, and black spots corresponding to a particular constituent within the composite. The gray area stands for the WC-TiC-Co particles, while the dark gray shows the copper matrix in which those particles are evenly dispersed. The black dots in this figure correspond to the graphene sheets that were purposely introduced into the composite structure.

A critical review reveals that the process of pre-coating of WC-TiC-Co particles with graphene before copper coating, in fact, leads to good dispersion and distribution of graphene in the copper/WC-TiC-Co matrix. Gradual addition of graphene from 0.25 to 1 wt.% exhibits a remarkable enhancement in graphene distribution throughout the composite. This uniform dispersion is reflected again in the SEM images, which indicate a completely integrated constituent with no visible large pores that would suggest high densification across all composite formulations. Besides, copper coating promotes good interfacial adhesion between the constituents for intimate contact and structural integrity. It is this improved adhesion that has helped not only in avoiding porosity but also in improving the mechanical properties and overall performance of the composites. The good constructive collaboration of graphene, WC-TiC-Co, and a copper matrix is showing great potential for such composites in the case of industrial applications, from cutting tools to wear-resistant coatings, because of enhanced microstructural features and excellent densification. The obtained results illuminate the way for further optimization and exploitation of graphene-reinforced copper/WC-TiC-Co composites in advanced engineering applications.

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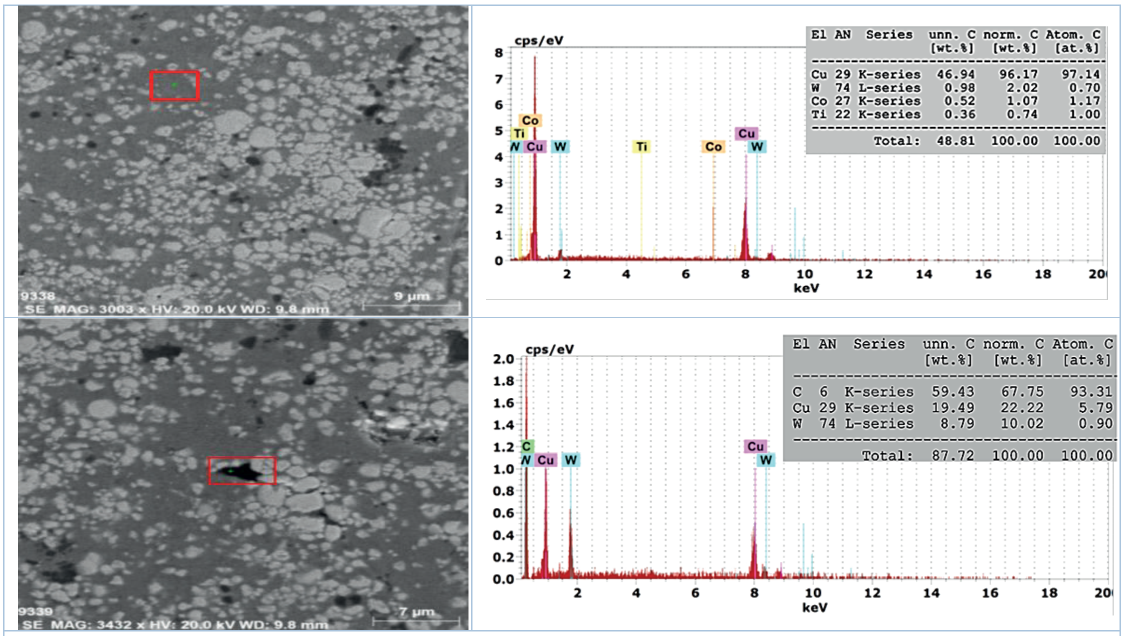

Fig. 5: EDAX analysis for coated Cu-WC-TiC-Co/1 wt. % GNs nano composite.

Fig. 5 presents the EDAX analysis for the sintered Cu-WC-TiC-Co/1 wt. % GNs nano-composite conducted in two distinct regions. The first region consists of Cu-WC-TiC-Co, while the second region includes a graphene layer. The analysis reveals that the sintered sample exhibits peaks corresponding to titanium, cobalt, copper, and tungsten, along with a pronounced peak for carbon in the graphene region. The absence of oxygen peaks indicates that no oxides were formed, likely due to the sintering process being conducted in a hydrogen atmosphere. This environment is particularly suitable for the stability and integrity of Cu-WC-TiC-Co/GNs nano-composites, preventing oxidation and ensuring the desired material properties are maintained.

3.4. Hardness Measurement

The investigation into the impact of graphene nano-sheets addition on the hardness of Cu/WC-TiC-Co composite, as depicted in Fig. 6, elucidates a compelling relationship between graphene content and mechanical properties. Notably, an augmentation in graphene nano-sheets content correlates positively with an increase in the hardness of the Cu/WC-TiC-Co composite. Despite graphene's low density of 2.2 g/cm³, its exceptional mechanical attributes, including a high Young's modulus (~ 1.0 TPa), render it a promising reinforcement agent for soft metals like copper [38].

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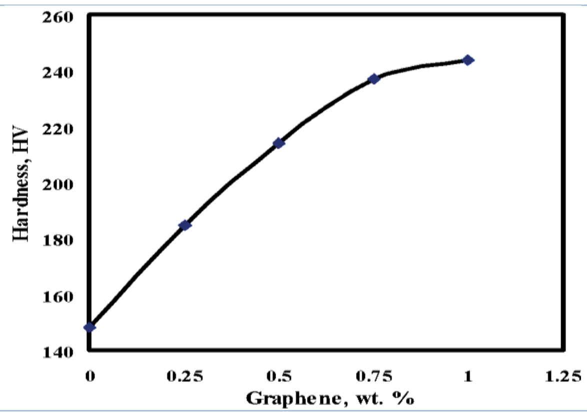

Fig. 6: Micro hardness of Cu/WC-TiC-Co/ GNP nano composites as a function of graphene percent.

The quality in the distribution and adhesion amongst constituent elements has remained a key factor in determining mechanical performance in composites. Fig. 4 represents microstructural analysis highlighting exemplary homogeneous dispersion of graphene within copper metal matrix composites. Noticeable is the orientation of the graphene layers in the matrix, mostly horizontal, enhancing the tensile resistance in this direction while at the same time reducing the penetration of the indenter of the micro-hardness tester in the vertical plane.

Fanyan Chen *et al.* [10] investigated in detail the effects of graphene content on the microstructure and properties of copper matrix composites, showing that hardness increased significantly with the increase in graphene content up to 0.8 v. % when compared to pure Cu. In the study of C. Ayyappadas *et al.* [11], higher values of hardness for the Cu-Gn composites were reported due to better mechanical properties of graphene. Given that the reinforcements are of hard nano-graphene of high strength, with hard ceramic WC-TiC-Co as reinforcement for the nano-copper matrix, this accounts for a great improvement in hardness. Because the reinforcements of these hard ceramic materials are dispersed homogeneously in the Cu matrix without agglomeration, effective transfer of stiffness can be ensured, and hence, uniform reinforcement leads to the increase in hardness of the composite material. It would mean that these insights hint at an extreme influence of graphene nano-sheets on mechanical performance improvement in Cu/WC-TiC-Co composites, promising further advances in material engineering and application.

3.5. Electrical Resistivity Measurement

Fig. 7 illustrates the profound impact of graphene nano-platelets (GNP) addition on the electrical conductivity of Cu/WC-TiC-Co composite. The data reveals a noteworthy trend wherein electrical conductivity experiences a remarkable enhancement with the incorporation of GNP, peaking at 1 wt. %. Notably, the introduction of 10 wt. % WC-TiC-Co to copper results in a significant reduction in electrical conductivity to 2.7 x 10^7 Ohm m^{-1}. Contrastingly, the integration of 1 wt. % GNP leads to a substantial increase in the electrical conductivity of the Cu/WC-TiC-Co/GNP nanocomposite to 4.9 x 10^7 Ohm m^{-1}. This augmentation in electrical conductivity is ascribed to several contributing factors. Firstly, the homogeneous dispersion of GNP within the copper matrix and the consequent increase in interfacial area between copper and graphene layers due to effective sintering processes play a pivotal role in enhancing electrical conductivity.

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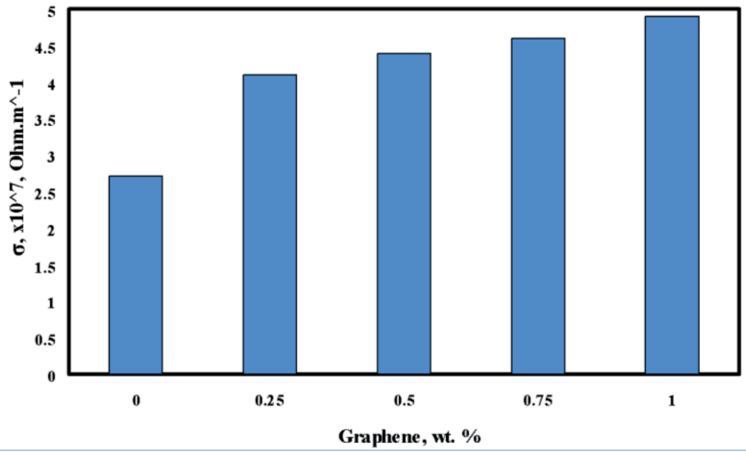

Fig. 7: Electrical conductivity of Cu/WC-TiC-Co/ GNP nano composites as a function of graphene percent.

The effect is also enhanced by the intrinsically high electrical conductivity of GNP. Besides, during the coating process with copper, strong interfacial bonding, as demonstrated by a high contact angle between GNP and WC-TiC-Co, is favorable for minimal pore formation. It allows unhindered electron motion, boosting conductivity accordingly. Besides, good sinterability, enabled by appropriate sintering temperature and time, led to conditions that were most favorable for constituent interaction and diffusion, as confirmed from microstructural analysis. Such close interaction among the constituents favors pathways for electron transfer, hence enhancing conductivity in the composite material. These findings put into evidence the complex interaction of material composition, processing parameters, and microstructural characteristics controlling electrical properties in Cu/WC-TiC-Co/GNP nanocomposites and hence provide useful insights for the advancement of those materials in various technological applications.

3.6. Thermal Conductivity

Fig. 8 illustrates the influence of the addition of graphene nano-platelets on the thermal conductivity of the Cu/WC-TiC-Co nanocomposites. Literature reports in this regard indicate a dramatic rise in the thermal conductivity of the Cu/WC-TiC-Co composite when graphene nano-platelets are added, especially up to 1 wt.%. This again demonstrates the critical contribution of reinforcing agents in enhancing the thermal properties of composite materials. The formation of the conductive network in the composite structure is basically a critical factor for high thermal conductivity values. Therefore, it requires strong cohesion among the various components of composites. Surface coating with copper enhances the particle-particle contact and adhesion between graphene nano-platelets and hence accelerates the formation of a well-defined conductive network across the matrix of the composite. This is the principal phenomenon that enhances thermal conductivity because of the possibility for effective heat transfer paths to be developed. This is further assisted by the synergistic effect of good sinterability, together with effective coating, which ensures that there are no pores within the composite structure and thus presents no limit to the conductive parameters. The result obtained is a tremendous enhancement in the thermal conductivity of the Cu/WC-TiC-Co nanocomposite, as represented in previous studies [39,40]. From this point of view, it would be intriguing to study the morphology, dispersion, and orientation effects of graphene nano-platelets in the composite matrix on the enhancement in thermal conductivity.

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Also, further studies may focus on optimization of loading percentage of graphene and seek other ways to enhance interfacial adhesion for maximization in thermal conductivity improvement in Cu/WC-TiC-Co nanocomposites. A deep understanding of the operative mechanisms for such enhancement in graphene-reinforced composites would be needed to ensure the future development of practical materials for a wide range of thermal management and heat dissipation applications.

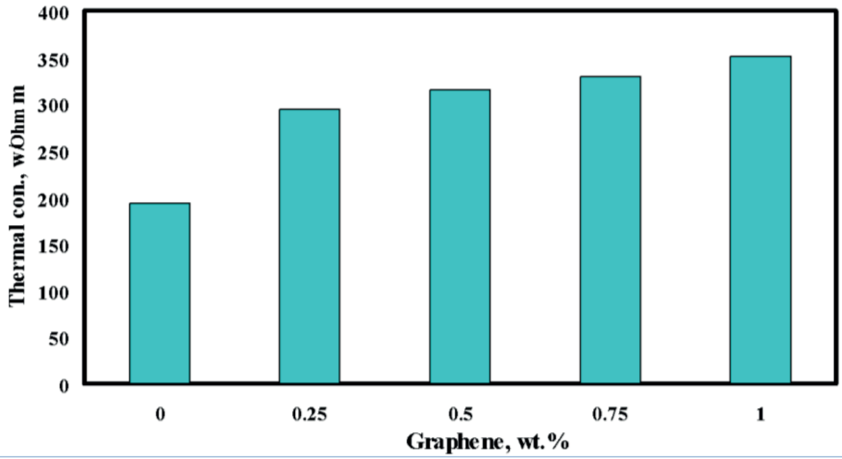

Fig. 8: Thermal conductivity of Cu/WC-TiC-Co/ GNP nano composites as a function of GNP percent.

4. CONCLUSION

1. The composite of Copper, WC, TiC, Co, and GNP was fabricated by the powder metallurgy technique; it is thus demonstrated that the method can indeed work in composite fabrication.
2. The compatibility of the composite powder WC-TiC-Co/GNP was tested with electro-less copper deposition, and accordingly good coating has been easily achieved, increasing the possibility in application to industries.
3. The addition of GNP was found to improve the microstructure of copper-WC-TiC-Co composite considerably, and therefore, better structural integrity with the possibility to optimize for improved mechanical properties is presumed.
4. Our experimental results show that with the presence of GNP, dramatic enhancements in both electrical and thermal conductivity can extend the applicability of composite material to electrical systems and thermal management applications.
5. The addition of hard GNP-WC-TiC-Co components has caused an improvement in hardness properties; hence, materials shall have improved wear and abrasion resistance which may be widely employed in different engineering applications.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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