



Structural and sliding wear properties of Ag/Graphene/WC hybrid nanocomposites produced by electroless co-deposition



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ABSTRACT

The main objective of this work has been the deposition of hybrid silver/WC/Graphene nanocomposites and characterization of their tribological behaviors. Graphene as a conductive solid lubricant additive was introduced into Ag matrix from the electrolytes in which submicron WC particles and Graphene nanosheets were suspended. The main purpose for two different reinforcements is to improve both wear and friction properties. The friction and wear behaviors of Ag/WC/Graphene coatings on the metal substrates against M50 steel ball were tested under dry sliding wear conditions. Comprehensive characterizations were performed using Scanning Electron Microscopy, X-Ray Diffraction analysis, Raman spectroscopy and 3D profilometry facilities. Tribological test results have revealed that even small amounts of Graphene addition are able to drastically improve the antifriction and antiwear properties of hybrid nano Ag matrix composites. A possible explanation for these results is that the co-deposition of Graphene not only provides an enhanced effect for nanocomposites to produce better wear resistance, but also forms a local protective layer on the contact surfaces to reduce the friction. The investigation shown that hybrid reinforcements of sub-micron WC and Graphene hold great potential applications as effective load bearing and solid lubrication for Ag matrix composites and possibly for similar alloys.

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1. Introduction

Electrical contacts provide electrical connection and often perform other functions. The primary purpose of an electrical connection is to allow the uninterrupted passage of electrical current across the contact interface [1]. Electrical contact materials are widely used in different low voltage switch devices, such as relays, contactors, circuit breakers and switches, and their properties are of importance to the switching capacity, reliability, stability and service life of integral electrical systems. The materials used for electrical contacts in these applications have to be considered to keep the efficiency of the contact in term of electrical conductivity, thermal conductivity and high wear resistance [1–3]. Many kinds of contact materials have been used in severe condition. In most cases, the development of contact materials is accomplished by experimental selection of the composition of metal matrix composites that can resist to wear and have the required electrical

characteristics. The development of novel materials with excellent mechanical and electrical as well as corrosion resistance properties is of great importance for high efficiency and reliability of future electrical contacts [4].

Recently, among novel materials, silver and silver alloys are often chosen because of significant electric conductivity and oxidative stability. Silver based materials suggested in using electrical contacts must have a good combination of electrical conductivity, wearing qualities, and resistance to erosion and welding. Otherwise, the contacts will erode, causing poor contact and arcing [5–7]. Hence, it is a challenge for material researchers to develop a new material instead of Ag and Ag alloys contact materials. So far, many silver based composite contact materials, such as Ag/Graphite, Ag/WC and Ag/CNT have been developed. For example, Wang et al. [8] reported on an Ag/Graphite/CNT electrical contact composite material that was synthesized by powder metallurgical method. They suggested that Ag/Graphite/CNT electrical contact material, with the increase of graphite content, exhibits good self-lubricating property and wear-resistance but electrical conductivity, thermal conductivity declined. Grand in et al [9], investigated the production of silver-graphite composite materials for sliding electrical contact applications with the aim to optimize tribological

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and electrical properties. Such composites exhibited improved mechanical, physical and chemical properties such as lower density, good mechanical strength, oxidation resistance, increased high-temperature performance limits and improved wear-abrasion resistance, depending on the properties of the metallic matrix and those of the reinforcing phase as well [10].

The use of tungsten as a reinforcement material for silver matrix has been studied by several researchers [11–13]. Tungsten, being a refractory metal, provides some degree of wear and arcing resistance when used with silver as an electrical contact material. However, for electrical contacts and similar applications, materials having good electrical conductivity and wear resistance along with lower density are desirable. Due to its lower density, tungsten carbide in place of tungsten has been used as reinforcement for silver based composites for electrical contact applications [12,14]. There are a lot of advantages in using WC as the reinforcement in silver based composites. It has a lower density (15.63 g/cm^3) as compared to tungsten (19.3 g/cm^3) and WC retains its room temperature hardness up to 1400°C . Its wear resistance is better than that of wear-resisting tool steels. WC undergoes no phase changes during heating and cooling and retains its stability for very long service times at high temperatures [12,15].

Compared with carbon nanotubes, Graphene with a plate shape is easier to handle and disperse in solvents or all kinds of matrices [16]. Therefore, we are confident that Ag/WC/Graphene has a good potential to replace conventional Ag material as a candidate for the next-generation MMCs for contact materials, specifically for MEMs. Graphene has received considerable attention in recent years due to its superior properties, if compared to conventional materials. Graphene, as the perfect two-dimensional (2-D) lattice of sp^2 -bonded carbon atoms, has recently attracted tremendous attention because of its unique properties such as high Young's modulus, high fracture strength, and thermal conductivity, unique thermal, mechanical, and electrical properties and it is expected to be one of the emerging self-lubricating materials [16–18]. In comparison with polymers and ceramics, Graphene-based metal matrix composites have been little researched. Most of the existing reports focus on the deposition of nanoparticles of noble metals and oxides on the surface of Graphene to impart new functionalities, such as catalytic, energy storage, photocatalytic, sensory and optoelectronic [18]. However, to best of author's knowledge, there has been no report on the fabrication of Ag/WC/Graphene composite materials. In this paper, Ag/WC/Graphene composites were prepared for the first time by electroless silver coating on copper substrates. Structural, mechanical properties such as the wear properties and hardness of the Ag/WC/Graphene composites were tested for developing new generation contact materials for possible future MEMs/NEMs applications.

2. Experimental

2.1. Synthesis of graphene

Natural graphite flakes were exfoliated according to the procedure given in the paper by Çetinkaya et al. [16], in which the details were described. In brief, Graphite oxide (GO) was obtained from graphite flakes (Alfa Aesar, 100 mesh in size) using the method described by Hummers. The graphite particles were pretreated to activate their surface facilitating the exfoliation of the van der Waals bonds between the Graphene layers. The graphite flakes were dispersed in a 3:1 nitric acid (HNO_3): sulfuric acid (H_2SO_4) 50 ml solution for 2 h with magnetic stirring. After the acid treatment, the graphite particles were heated to 800°C for 120 s in an open air atmosphere. After the pretreatment process, the Hummers method was used to synthesize graphite oxide. To prepare the

Graphene oxide, 30 mg of the graphite oxide particles was added to 100 ml distilled water, and the Graphene oxide sheets were separated from the graphite oxide structure using an Ultrasonic Processor (UP400S) at 20 kHz, with a power of 60 W for 2 h. Finally, the reducing agent hydrazine hydrate (1 ml) was added and the solution was heated on water bath to 85°C for 24 h. Graphene was gradually precipitated out as a black solid, which was filtered and washed repeatedly with distilled water.

2.2. Electroless deposition process

The Ag, Ag/WC, Ag/Graphene and Ag/WC/Graphene composite coatings were deposited on $5 \text{ cm} \times 5 \text{ cm} \times 0.5 \text{ cm}$ copper plates by using electroless coating technique. The composition and deposition parameters of the bath are listed in Table 1. All the chemicals used in the pretreatment and electroless plating procedures are of analytical purity ($>99\%$). The copper plates were mechanically polished with different abrasive papers in order to obtain a smooth, bright and uniform surface and then the copper substrates were activated in 25% H_2SO_4 solution for 2 min. After preparation of the solution, specimens were put into the electroless plating bath. The specimens were immersed in the electroless plating bath, which contained silver salts solution (A), glucose-based reducing agent (B), and deionized water; at room temperature for 30 min. The two solutions A and B were mixed in a 1:1 ratio just before the bath was applied for electroless coating. AgNO_3 (99.8%) and ammonia were used for preparation of Ag solution. Transparent Ag solution was first prepared by dissolving the AgNO_3 in the water with addition of the aqueous ammonia, then NaOH was added to the solution, finally several drops of aqueous ammonia were added until the $\text{Ag}(\text{NH}_3)^+$ solution became transparent again. After preparation of the solution, copper plates were immersed into the electroless plating bath. Plating time kept constant at 2 h for each electroless coating run. The Ag/WC, Ag/Graphene and Ag/WC/Graphene composite coatings were obtained from the bath solution containing the WC particles (10 g L^{-1}) and Graphene (100 mg L^{-1}), respectively. The range of WC particle size used in the experiment was $0.1\text{--}1 \mu\text{m}$. The amount of surfactant, CTAB (cetyltrimethylammonium bromide) was fixed at 300 mg L^{-1} . Prior to composite coating the bath solution was stirred using a magnetic stirrer at 600 rpm for about 12 h, and subsequently ultrasonic agitation was conducted using an Ultrasonic Processor (UP400S) at 20 kHz, with a power of 60 W for 30 min in order to prevent agglomeration of WC particles and Graphene and also providing suspension of reinforcements in the electrolyte.

2.3. Characterizations

The morphology and microstructures of the composite coating were characterized by a scanning electron microscope (SEM, model JEOL – JSM 6060 LV). XRD analysis was performed with X-ray

Table 1

Composition and deposition parameters of the plating bath for Ag, Ag/WC, Ag/Graphene and Ag/WC/Graphene composite coating.

Silver solution (A)		Reduction solution (B)	
AgNO_3	5 (g/100 ml)	Glucose	3 g
$\text{NH}_3 \cdot \text{H}_2\text{O}$	3.5 ml/100 ml	$\text{C}_2\text{H}_5\text{OH}$	10 ml
NaOH	4 (g/100 ml)	H_2O	100 ml
pH	11–12		
Plating time (h)	2		
Temperature ($^\circ\text{C}$)	25–30		
WC	10 g L^{-1}		
Graphene	100 mg L^{-1}		

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