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Development of Graphene Nanoplatelets Reinforced HVOF sprayed Cr3C2-NiCr Coatings

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ABSTRACT

Thermal sprayed coatings are widely used for wear and corrosion resistant applications in various industrial applications as they have less porosity, higher hardness, and fracture toughness. The Properties of these coatings can be improved further by reinforcing them with nanomaterials. In recent years, nanomaterials have found their applications in diversified fields including composite materials, owing to their excellent mechanical properties. A wide range of nanomaterials such as nano-carbonaceous materials, nano-rare earth oxides, and nano-ceramics have been used as reinforcements in thermal sprayed coatings, leading to enhancement in the coating properties. However, homogeneous blending of the nano-reinforcement materials with coating powders is challenging as the nanomaterials have an affinity to form agglomerates due to van der Waals forces. Although various mixing methods are employed by the researchers such as mechanical ball milling and spray drying for mixing, they have some limitations. Here, an attempt has been made to develop a mixing method with which the nanomaterials can be uniformly mixed with the coating powder. In the present work, authors have prepared graphene nanoplatelets (GNP) reinforced cermet feedstock powder Cr_3C_2-NiCr for the HVOF spray process by three methods: 1. Dry ball milling, 2. 3-D tumbler mixing, and 3. a method involving the process of ultrasonication & 3-D tumbler mixing. Through FE-SEM with EDS analysis, it was observed that method-3 yielded homogeneously mixed composite powder. XRD and Raman spectroscopy were also carried out to identify the phases and to confirm GNP retention.

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Introduction

Thermal spray processes are widely used by industries and researchers as well for depositing coatings to provide resistance to wear, erosion, and corrosion. The most prominently used thermal spray processes are atmospheric plasma spray (APS), high velocity oxy fuel (HVOF) spray process, flame spray process, detonation gun (D-Gun) spray process and high velocity air fuel process (HVAF). They can be used to spray metals & alloys (Ni, Al, NiCrAlY), ceramics (Al2O3, ZrO2, TiO2), Carbides & Cermets (WC-Co-Cr, Cr3C2-NiCr), and Polymers (polyethylene, polyamide) [1]. HVOF sprayed Cr3C2-NiCr coatings are applied for protection against wear and corrosion to components subjected to harsh working conditions such as gas turbines, steam turbines, aero engines, automobile engines, and boilers etc.

The mechanical properties of thermal spray coatings can be further enhanced when they are reinforced with nanomaterials. Thermal spray processes can be used to deposit nanomaterials as the processes are simple, have high deposition efficiency, have a wide range of coating materials and matrix, and offer ease in the formation of composite coatings [2]. As reinforcements, nanomaterials, offer benefits such as grain refinement, toughening mechanisms, and change in physical and chemical properties. Graphene nanoplatelets (GNP) is a nano material that is being used by researchers as reinforcement for composite materials and coatings owing to its superior thermo-mechanical properties such as high elastic modulus (~1 TPa), high strength (~130 GPa) and high thermal conductivity (~5000 W/mK) [3]. It also offers lubricity leading to improved resistance against wear and friction. The flakes of GNPs can bridge the grain boundaries of the material so that the stress is uniformly transferred throughout the structure which provides a strengthening and toughening effect that prevents failures [4, 5]. GNP reinforced composites and coatings have shown occurrences of crack branching, crack deflection, and resistance to crack tip propagation [6]. GNP acts as a solid lubricant, forms and maintains a layer of tribo-film over the sliding surface in the sliding wear tests and gives excellent improvement in wear resistance.

However, the excellent characteristics of such nanomaterials can be incorporated into the coating if they are uniformly dispersed in the coating powder. Atif et al.[7] and Saboori et al. [8] reported that the formation of agglomerates implies their nonhomogeneous dispersion in the powder matrix, which eventually may not impart the desired improvement in the properties of the coatings. Clearly, the method employed for mixing nanomaterial with coating powder plays a very vital role in the performance of composite coating powders. Various methods have been used by researchers to carry out the mixing of nanomaterials with thermal spray coating powders such as various forms of mechanical milling and spray drying.

Mechanical milling is a traditional method for blending powders, in which the powders to be mixed are placed in a cylindrical jar and then rotated in the presence of a mixing medium such as balls [9]. Milling speed, ball to powder weight ratio, milling time, and process control agent are the main process parameters [10]. This method is used by

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some researchers to blend nano-carbonaceous materials such as graphene nanoplatelets (GNPs), graphene oxide (GO), and graphene nanosheets (GNs). Ward et al. [11] used a horizontal jar mixer to mix metallic powder Ni-Al with nanomaterial edge oxidized graphene oxide (EOGO) with steel balls as a mixing medium. The addition of EOGO improved hardness, adhesion strength, and wear resistance by 49%, 18% and 14% respectively. Planetary ball milling was used by Li et al.[12] for mixing ZrO2 with graphene nanosheets for the plasma spray process. The same was used by Ranjan et al.[13] and Kumari et al. [14] for mixing titanium powder with GNPs. They reported remarkable improvements in tribological and anti-corrosion properties of the composite coating. Some researchers have used the ball milling methods for mixing ultrasonicated GNP with coating powders. Forati et al. [15] ultrasonicated GNP and Cu in ethanol and then ball milled and dried to get the composite feedstock powder. Mukherjee et al. [16] mixed NiAl with ultrasonicated GNP in a planetary ball mill to develop a plasma sprayed coating that offered high tensile strength and good ductility. While the mechanical milling approach offers a viable option for the mass production of composite powders, it necessitates extended milling periods ranging from 3 to 48 hours. Furthermore, during mechanical milling utilizing balls as media, there's a risk of damaging the sensitive microstructures of the powders or reinforcements through grinding [17].

Wang et al. [18] and Qin et al. [19] prepared WC-Co/GO coating powders for the D-gun spray process with four methods: wet ball milling, wet mixing using a stirrer, sintering & crushing and spray drying. Spray dried powders were reported to give the best microstructure and wear resistance. The spray drying process is used by many researchers. In this process, the nano-sized powder particles are dispersed in an organic binder to form slurry and then the slurry is passed through an atomizing orifice into a hot chamber. The droplets coming out of the orifice dry and form micron size agglomerates of coating powder [20]. Amudha et.al.[21, 22] used spray drying process to mix 0.5, 1.0, 1.5 and 2.0% graphene oxide (GO) with Al2O3, deposited with plasma spray process, and reported increase in microhardness and fracture toughness. Al2O3 powder spray dried with graphene nanoplatelets (GNP) and carbon nanotubes (CNT), and then deposited with atmospheric plasma spray process showed remarkable improvement in toughness [20], electrical conductivity [23]and tribological properties [24]. Spray drying technique is a very effective method for preparation of nanocomposite coating powders. Nonetheless, the particle size of coating powders used by the researchers was ~ 0.3 µm. Fine size powders incur high costs, making the process economically unviable.

Another approach to deposit nanocomposite coatings is to employ suspension based thermal spray processes such as suspension plasma spray and suspension high velocity oxy fuel spray processes. Murray et al. [25] deposited Al2O3 coating with 1 wt.% GNP by suspension HVOF process and exhibited enhancement in wear resistance due to improved microstructure and fracture toughness. Mahade et al. [26] used suspension plasma spay process to deposit GNP reinforced Al2O3 coating with water based GNP + Al2O3 mixed suspension. They reported 36% lower friction coefficient and 69% lower specific wear rate. Suspension spray processes have the ability to apply a diverse array of coating materials [27-29]. Nevertheless, the drawbacks of these methods include their intricate nature and substantial initial investment costs [30].

It seems that there is a scope of development of a method to mix the nano reinforcements with the coating powders to be sprayed with the conventional thermal spray method. In this article, the authors have described the methods they used for mixing GNP with Cr3C2-NiCr to develop a nanocomposite feedstock powder for HVOF spray process. At first, the traditional method of mechanical milling was used for mixing. Then, 3D tumbler mixer was employed for attaining the uniform blending. Afterwards, a method was developed which includes ultrasonication and 3D tumbler mixing. In the present paper, the powder blending outcomes of three mixing processes: Ball milling; 3D tumbler mixing; and ultrasonication & 3D tumbler mixing are compared and characterized.

Experimental

Materials

The coating powder used was commercially available Cr3C2-NiCr powder (CrC-410, C&M Technologies, Germany) having average particle size -45+15 µm. Figure-1 (a) shows Cr3C2-NiCr powder. It is seen that the morphology of Cr3C2 particle is spherical in shape, has uneven surface and has pores on it. This morphology is expected to help in getting reinforcement material settled on the surface or get into the pores of the carbide particles. The reinforcement material, graphene nanoplatelets (GNP) procured from Sigma Aldrich is shown in Figure-1 (b). It has average particle diameter of 5 µm and surface area of 120 – 150 m2/g. The GNP particles are thin and flaky in morphology. The feedstock powder composition is Cr3C2-NiCr – 1 wt.% GNP (henceforth referred as CG). The prepared composite powder was sprayed on SS304 substrate.

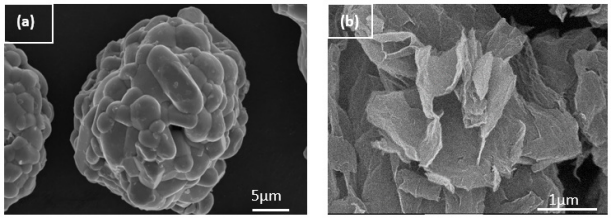

Figure 1 : FE-SEM micrographs of (a) Cr3C2-NiCr powder, (b) Graphene Nanoplatelets

Mixing methods

Three different methods were used to prepare GNP reinforced Cr_3C_2-NiCr powder and the results were evaluated.

Figure 2 : Dry ball milling (Jar milling) setup

Method-1 Dry ball milling: Cr_3C_2-NiCr powder was mixed with 1 wt.% GNP in a ceramic jar. Steel balls were used as

grinding media, with ball to powder ratio as of 1:10. The jar rotated at 100 rpm for 6 hours. The set up was made a lathe machine as shown in Figure-2.

Method-2 3D tumbler mixing: A 3D tumbler mixer, Alphie3 (Figure-3) was used for mixing. It has a unique 3D tumbling motion that does not generate centrifugal force, so there is no possibility of local heating. It does not have a blade or agitator shaft, so there is no involvement of shear force while mixing. The coating powder and nano-reinforcement materials were mixed using this mixer in two ways: (1) Dry mixing: powders mixed in a dry state, in the absence of any medium, and (2) Wet mixing: powders were mixed in the presence of acetone. For both the mixing, the mixer was operated at 50 rpm for 1 hour.

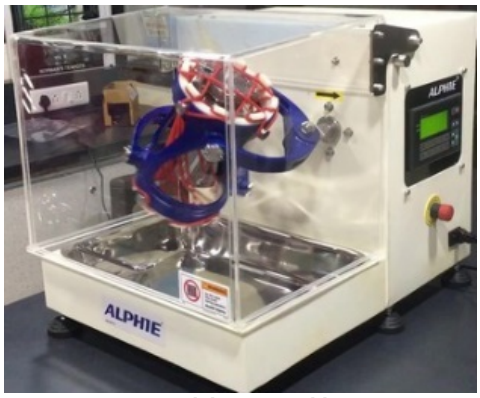

Figure 3 : Alphie 3D tumble mixer
(source: https://www.alphiemixer.com/)

Method-3: Ultrasonication followed by drying and 3D mixing: Figure-4 below shows the schematic representation of the mixing methods used. Ultrasonic probe sonicator (VCX500, Sonics, USA) was used to first sonicate GNP along with surfactant sodium dodecyl sulfate (SDS) in deionized water (Figure-4(a)). GNP was sonicated for 30 minutes. This sonication process gave nano-suspension of GNP (Figure-4(b)). Coating powder Cr3C2-NiCr was then added and the mix was sonicated for 30 minutes further (Figure-4(c)). The mixture slurry obtained thereafter was kept overnight in a laboratory heating oven at 60°C (Figure-4(d)) for drying. The dried powder was collected in a jar and was then blended using a 3D tumbler mixer, Alphie 3D, for 1 hour at 50 rpm (Figure-4(d)). The powder was then sieved to obtain the final feedstock powder.

Characterizations

Scanning electron microscopy was carried out using a field emission scanning electron microscope (FE-SEM) JEOL 7900F (JEOL, USA) equipped with an energy-dispersive x-ray (EDX) detector. FE-SEM was carried out to examine the microstructure of the mixed powders and as sprayed coatings. For phase identification, the powder samples were analyzed by X-ray diffraction (XRD) using a Bruker AXS D-8 Advance diffractometer with Cu Kα radiation. Raman spectroscopy was carried out using Renishaw InVia Raman Microscope, Model: Qontor with a wavelength of 532 nm at 0.3 mW power.

Coating Deposition

Coating deposition was done on SS-304 substrate material using MEC HIPOJET 2700 HVOF equipment at Keepsake Engineering Consultancy Pvt. Ltd., Ahmedabad, Gujarat. The specimens were grit blasted prior to coating deposition. The spray parameters used are listed in Table-1.

Table 1: HVOF spray parameters

HVOF spray parameters	
Fuel (LPG) flow rate	55 lpm
Fuel supply pressure	6.3 bar
Oxygen flow rate	280 lpm
Oxygen supply pressure	10 bar
Air flow rate	600 lpm
Air pressure	5.88 bar
Powder feed rate	38 g/min
Spray distance	165 mm
Coating thickness	250 – 300 µm

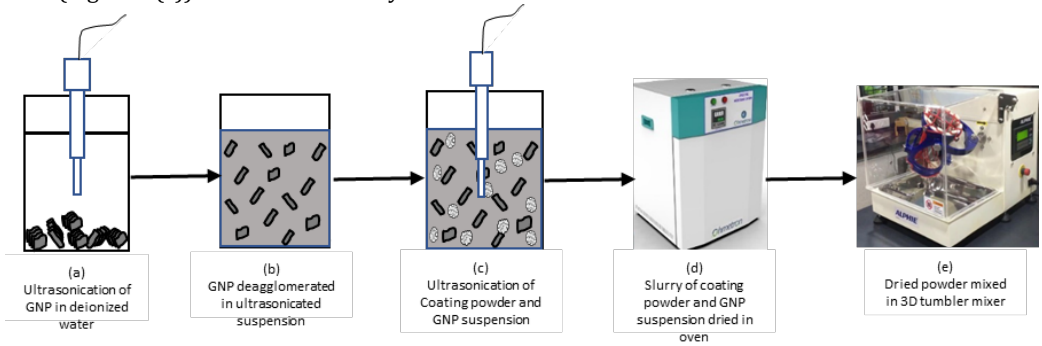

Figure 4: Mixing process involving ultrasonication & 3D tumbler mixing

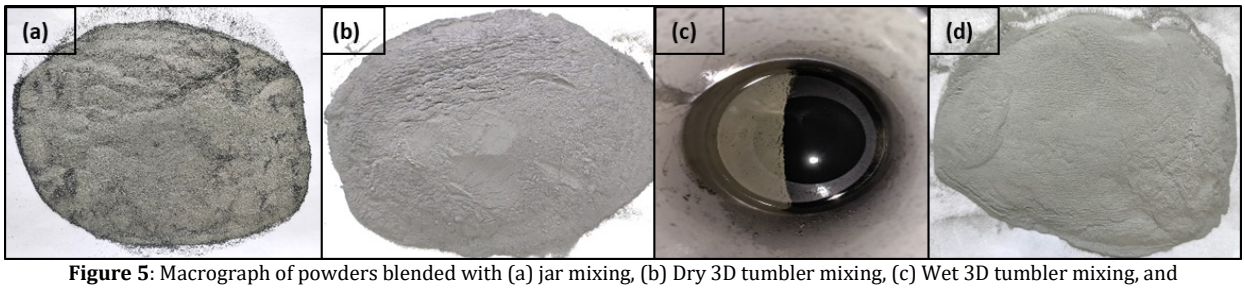

Figure 5: Macrograph of powders blended with (a) jar mixing, (b) Dry 3D tumbler mixing, (c) Wet 3D tumbler mixing, and (d) Ultrasonication & 3D tumbler mixing

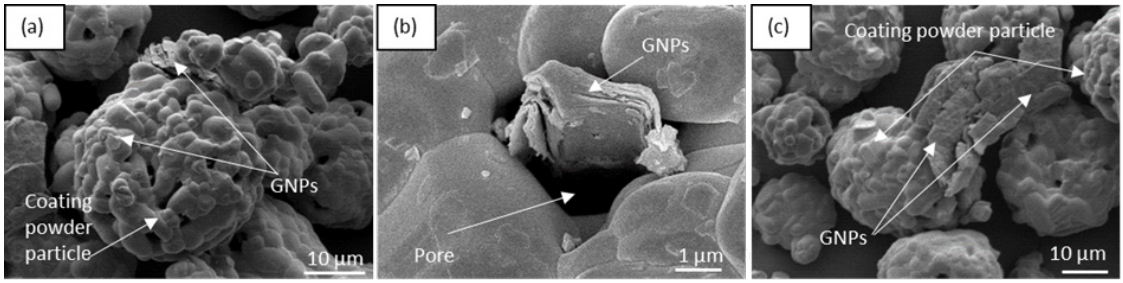

Figure 6 : FESEM micrographs CG powder mixed with dry 3D tumbler mixing

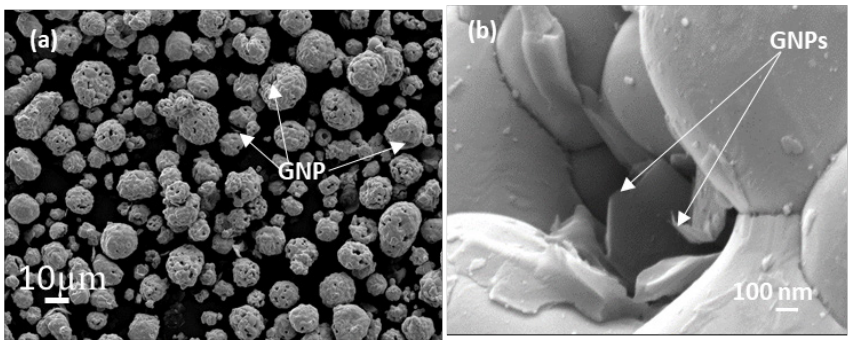

Figure 7: FESEM micrographs of powder blended with ultrasonication and 3D tumbler mixing

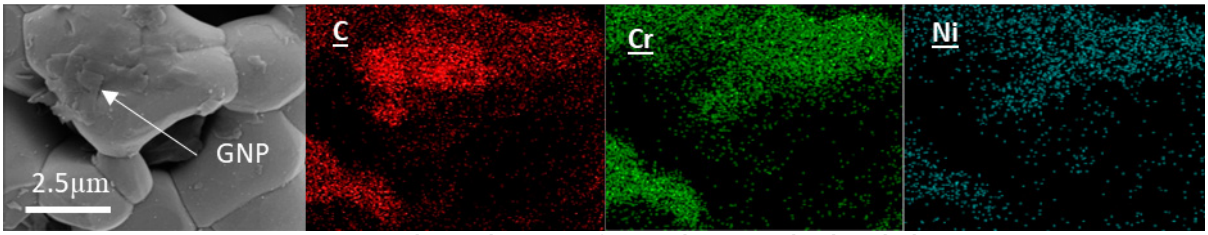

Figure 8*:* EDS elemental mapping of CG powder prepared with method-3

Results and Discussion

The macrographs of output powders of all the methods used in this study are shown in Figure-5. In Figure-5(a), the powder mixed with method-1 dry ball milling, shows GNP agglomerates as small dark-coloured particles lying on the light grey-coloured coating powder. It is evident that the uniform mixing has not taken place due to GNP agglomeration. The result of dry mixing in the Alphie 3D tumbler mixer is as shown in Figure-5(b). In visual inspection, GNP agglomerates were not sighted. Looking at the uniform colour and texture of the mixed powder it was assumed that uniform mixing had taken place. Figure-5(c) shows the result of mixing in the presence of acetone. It clearly shows the coating powder in light grey colour and GNP in black colour, as two separate layers. It was clear that mixing did not happen during this process. After drying in oven, this powder also showed agglomerated GNP particles. Figure-5(d) shows feedstock powder obtained from the mixing method-3 involving ultrasonication, drying and 3D tumbler mixing. In visual inspection, the powder shows uniform colour and texture throughout, without any visible agglomerates of the GNP. As the powder prepared with the dry ball milling (Fig.5-(a)) and wet 3D tumbler mixing (Fig.-5(c)) did not exhibit uniform mixing upon visual inspection, they were not considered for advanced characterizations.

Characterizations of coating powders

FE-SEM micrographs of the 3D tumbler mixed powder in a dry state are shown in Figure-6. The GNP flakes can be seen settled over the irregular surface of the coating powder particle (Fig. 6(a)), getting inside the pores on the coating powder particle (Fig. 6(b)), and lying between the coating powder particles (Fig. 6(c)). The GNP flakes seemed to be well distributed throughout the powder, close observation of the micrograph, however, revealed that the GNP flakes were piled up over each other and had agglomerated which implied that homogeneous dispersion of GNP was not obtained with this method. On account of the presence of agglomerated GNP flakes, the powder mixed with method-2, dry 3D tumbler mixing, was excluded from subsequent characterizations and coating deposition.

Figure-7 shows the FE-SEM micrographs of the powders mixed by method-3 i.e. ultrasonication followed by drying and 3D tumbler mixing. Figure-7(a) shows the micrograph of the composite powder CG in which GNP flakes were seen settled on the irregular surface of the Cr3C2 particles. The flakes didn't look agglomerated and were placed over the irregular surface of the powder particle. Figure-7(b) shows the GNP flakes that got inside of the pore present on the powder particle surface. This was similar to the coating powder developed by Tian et al [31] by spray drying. To verify the presence of GNP, the CG coating powder was subjected to EDS elemental mapping as shown in Figure-8. The presence of accumulated carbon element on the powder particle in the mapping suggests that the flaky substance settled on the coating powder particle is GNP. The FE-SEM analysis showed that the GNPs were deagglomerated and homogeneously distributed in the coating powder mixed with method-3.

The XRD diffraction patterns of powders Cr_3C_2-NiCr and CG are shown in Fig.9. From XRD analysis of unreinforced Cr_3C_2-NiCr powder two phases were identified: carbide phase Cr_3C_2 and matrix phase NiCr. XRD analysis of the CG powder showed that the presence of Cr_3C_2 and NiCr remained intact. It is evident that after the blending process, there are no undue phase changes in powders.

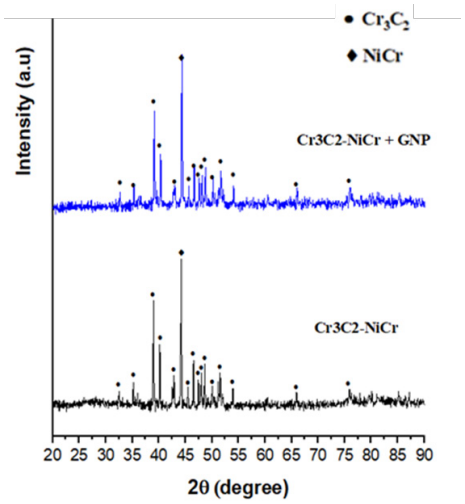

Figure 9: XRD diffraction pattern of Cr3C2-NiCr and CG powders

Raman spectroscopy is an analytical tool to prove the presence of carbonaceous materials. Raman spectroscopy of CG coating powder is shown in Figure-10. The GNP powder shows the typical graphene peaks at 1350 cm^{-1}, 1580 cm^{-1}, and 2700 cm^{-1} corresponding to D-band, G-band, and 2D-band respectively[32]. The CG powder shows identical peaks as GNP. This indicates that GNP was retained in CG powder without much structural damage.

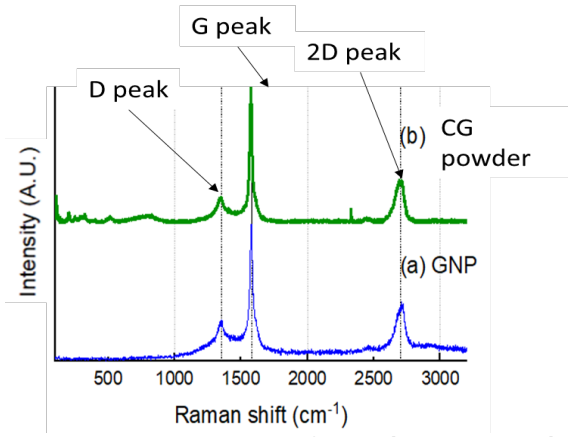

Figure 10: Raman spectroscopy of GNP and CG coating powder

Characterization of the as sprayed coating

The CG powder obtained by method-3 was deposited with the HVOF spray process on the SS304 substrate. Figure-11 shows the FE-SEM analysis of as sprayed CG coating. Figure-11(a) shows that molten and partially molten carbides deposited and solidified uniformly. Further magnification shows thin GNP flakes present on the coating surface, as shown in Figure-11(b)&(c). The presence of GNP on the coating surface proved that GNP survived the high temperature and velocity during the coating deposition. GNP retention can be attributed to the fact that the GNP flakes were uniformly distributed on the surface and were in the pores of the coating powder particle, which might have protected them from high temperature and velocity environment during the coating process. The EDS elemental mapping of the surface of as sprayed coating is shown in Figure-12 which confirms that the thin flake-like object seen on the coating surface is GNP.

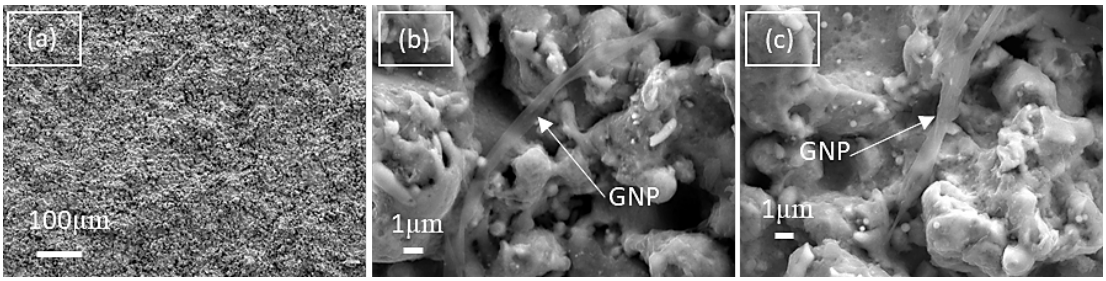

Figure 11: FE-SEM micrographs of as sprayed Cr3C2-NiCr+1% GNP coating

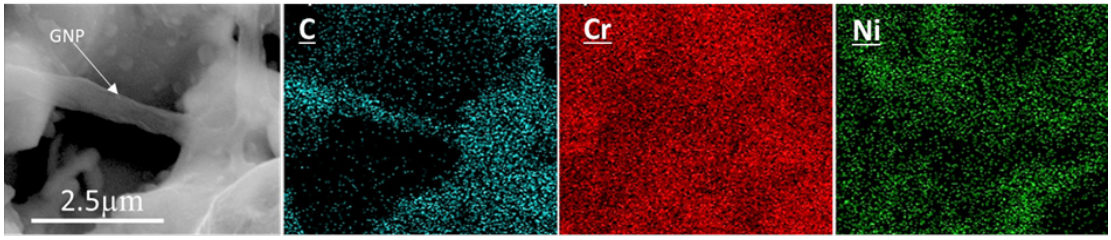

Figure 12: EDS elemental mapping of as sprayed CG coating

Conclusions

This study narrates the authors' journey of finding an appropriate method for blending cermet coating powder, Cr3C2-NiCr with nano-carbonaceous material, GNP. The mixing method involving ultrasonication followed by drying and 3D tumbler mixing, not only deagglomerated GNP flakes but also dispersed them in the coating powder homogeneously. The FE-SEM/EDS analysis, Raman spectroscopy, and XRD analysis of the coating powders proved the homogeneous distribution of GNP into the coating powder without any undue changes in the phase. The retention of GNP in the as-sprayed coating, which was established by carrying out FE-SEM/EDS analysis, could be attributed to the homogeneous distribution of GNP in the coating powder.

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