

# Microstructure Characteristics and Properties of NiGrFeS HVOF Coating for SAE 1008 Cold Rolled Steel

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## Abstract

This paper demonstrates the characterization of High Velocity Oxygen Fuel (HVOF) sprayed NiGrFeS coating on SAE 1008 cold rolled steel as a substrate to improve the surface properties for tribological applications. A combination of characterization techniques, such as XRD and SEM/EDAX procedures, have been used to evaluate the coatings as well as for mechanical and microstructural characteristics. Additionally, the porosity, microhardness, coating thicknesses, and coating density have been arrived at a view to obtain structure property co-relations. The HVOF overrides other thermal spray processes in terms of specific advantages of getting superior properties. Being a new development in the family of spray technology, the various issues such as coating quality, surface characteristics and other associated factors are being addressed and reported with the research work carried out. It is observed from the data the microhardness values have decreased across the depth of the coatings.

**Keywords:** HVOF coating, XRD Technique, Scanning electron microscopy (SEM)

## 1.0 Introduction

Early in the 1980s, surface engineering had a rapid development as a result of the realization that the great majority of engineering components could possibly decay or fatally fail in operation due to phenomena connected to the surface, such as wear, corrosion, and fatigue<sup>1</sup>. In surface engineering, components and materials are coated using both conventional and cutting-edge technologies. The goal is to create a new class of material with characteristics that are not possible using either the base material or the surface material. A component's operating environment may cause a material's

surface characteristics to vary noticeably. A coating can be described as a region close to the surface that has characteristics distinct from the bulk material as it proceeds on to deposition. One of the most powerful techniques for depositing small powder particles on a wide variety of surfaces has been the HVOF high velocity oxy fuel process, which is a relatively new technology of the thermal spray coating family. The HVOF method relies on combining heat and kinetic energy to melt and accelerate powder particles and deposit desired coatings. It is also possible to employ propane, acetylene, propane, kerosene (liquid), and hydrogen in the process. The combustion chamber is pressurized with ambient air rather than oxygen, which is used as an oxidizing agent. In order to tailor the desired coating, the coating

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components are deposited on the substrate in a molten or semi-molten state, thus HVOF is not a solid-state process. As part of the coating process, the particle size of the powders used as feedstock materials is crucial<sup>2,3</sup>. A supersonic jet is used in the HVOF, which provides superior coating characteristics over traditional flame spraying because particles are impacted much more quickly on the substrate. Utilizing a high-temperature, high-velocity gas stream, powder coating materials are heated to a molten condition and then accelerated toward a substrate. As the molten powder is pushed through the high-energy gas stream, a stream of droplets impacts the substrate item. Droplets deform or flatten on surfaces, resulting in splats. Splats are built up in layers to create the coating. The HVOF process has the advantage of requiring shorter dwell times, which results in less deterioration of the starting feedstock powder. Additionally, the molten powder stream's interaction with the surrounding atmosphere can be minimized or avoided due to the short processing time. An open-air thermally sprayed coating is made up of a mixture of sprayed material, oxide inclusions, and porosity<sup>4,5</sup>.

## 2.0 Materials and Method

In order to develop composite coatings with the High Velocity Oxygen Fuel (HVOF) technique, substrate material selected was SAE1008 cold rolled steel. The chemical composition of the substrate material is shown in Table 1. The cylindrical steel samples were cut and turned approximately  $\text{Ø}7.5 \times 25$  mm length sized specimens. The feedstock powder comprised with the combination of commercially available 308NS(NiGr) from Oerlikon Metco, Switzerland procured from Spraymet Surface Technologies Pvt. Ltd, Bengaluru-India; Ferrous Sulphide (FeS) supplied by Jainson Labs India, Meerut and Multi walled Carbon Nanotubes (MWCNT) received from Adnano Technologies, Shimoga, India. The blending of materials was made using a mechanical mixer to get the various combination prepared by varying the weight percentage of NiGr and FeS respectively. Prior to coating, substrate was cleaned with acetone to remove the inclusions and dirt while subjecting to grit blasting with a 40-grit size of  $\text{Al}_2\text{O}_3$  particles. The major advantage of the blasting process is to obtain a better bonding between the substrate and the coating materials deployed. The coating deposition was carried at Spraymet Surface Technologies Pvt. Ltd, Bengaluru-India using METCO Diamond Jet Gun - DJ 2600 attached with a nozzle system. The coating thickness was also thoroughly monitored. The process parameters for coating deposition is listed in Table 2.

The following designation is allocated for the samples prepared with the HVOF technique and is as follows in Table 3.

**Table 1**

Element content	Per cent %	Specification of SAE 1008 in %
Iron	99.201	99.31-99.7
Carbon	0.087	0.1 Max
Silicon	0.234	Not specified
Manganese	0.421	0.3-0.5
Phosphorous	0.031	0.04 Max
Sulphur	0.026	0.05 Max

**Table 2**

Parameters	Value
Working Temperature	Around 2300°C
Nozzle gun velocity	700ms <sup>-1</sup>
Powder feed rate	55g/min
Total heat O/P	113kW
Oxygen flow pressure	10-12 bar
Barrel length	0.15m
Number of passes	25 $\mu$ /pass
Oxygen flow pressure	12 bar
Hydrogen pressure	9.7 bar

**Table 3**

Sample description	Sample identification
1. 100wt%	OF <sub>1</sub>
2. 50wt% + 50wt%	OF <sub>2</sub>
3. 50wt% + 49wt% + 1wt%	OF <sub>3</sub>
4. 75wt% + 24wt% + 1wt%	OF <sub>4</sub>
5. 25wt% + 74wt% + 1wt%	OF <sub>5</sub>

The aim of conducting density and porosity tests was to analyze the efficiency of the process deployed which is basically known for producing dense coatings and least minimum porosity. In order to calculate the volume of an object with an irregular shape, Archimedes' Principle offers a practical and precise method that helps determine density according to ASTM C135-96 (2022) standards. This approach is frequently employed. It is also referred to as hydrostatic weighing. Porosity measurements was carried out by ASTM E2109-01 test methods for determining area percentage porosity in thermal sprayed coatings. The recommended procedure was followed to prepare sample transverse sections for optical microscopy. Quantitative metallographic

analysis was carried using the commercial software attached to the microscope. A programme called an image analyzer software uses samples with images of the microstructure to determine physical properties. Buehler's VH 1102 Vickers microhardness tester is used in the present work for micro hardness measurements. Thin sections or case depth work are the main applications for the Vickers hardness test method, often known as a microhardness test method. Based on an optical measurement technique, the Vickers method is deployed. In order to create an indentation that can be measured and translated into a hardness value, a diamond indenter is used in the microhardness test procedure as per ASTM (E-384). Ten random impressions were made on the coated cross-section of each sample. Finally, coating thickness was assessed on the HVOF designated samples of (OF1 to OF5) samples with the standards based on IS:3203-1982.

### 3.0 Results and Discussion

Optical Emission Spectrometry according to the ASTM E415-21 standard procedure was used to confirm the composition of the substrate material and the elements present are as shown in Table 1. The material was validated as SAE 1008 steel with a supplement of cold rolled.

The physical and mechanical characteristics of the substrate and coating, as well as their microstructure, are both reflected in the microhardness and are, in turn, influenced by the materials and manufacturing procedures used to create them. Due to potential material heterogeneities, hardness is a quality that can be thought of as being variable in some areas of the coating. Accordingly, when measuring this attribute, it is important to take into account the surface preparation condition, the parts to be examined, as well as the total number of indentations made. Table displays the microhardness of the coating and substrate. An optical microscope, image analyzer and scanning electron microscope were used to study the microstructural characteristics of the coatings on mild steel substrate. In the HVOF system, the particle is first melted and ejected from the gun in the shape of a sphere; upon initial impact with the substrate, the shock wave is generated both inside the lamella and in the substrate. The particle takes on a pancake shape after impact<sup>6,7</sup>. In addition, spray distance as well as particle temperature and velocity have an impact on the coating microstructure. The microstructure of a coating is made up of a number of properties, and each one is crucial in determining how well coatings operate under various test conditions. Porosity, which varies according to the processing method used, is one of the characteristics. The HVOF deposition method is renowned for producing dense coatings with decreased porosity levels. Using image analyzer software, the maximum

amount of porosity along the cross-sectional area of all the coatings was determined to be on an average of 1.5%. The higher kinetic energy of powder particles and the melting behaviour displayed by the particles may be related to the lower value of porosity produced for the HVOF sprayed coatings.

When the partially melted material reaches the substrate at a high rate of speed, it forms a nearly porous-free covering.

Morphology of powders procured is as shown in the Figure 1 and the particle size was confirmed from both analysis and the specifications sent from the powder supplying agencies. The typical morphology of spray powders that have been screened into various sizes is shown in Figure. It has been discovered that every powder has a predetermined form. Particle size was measured utilizing SEM pictures, and the obtained data sheet was consistent with the results that were obtained. The range of the NiGr and FeS particles was evaluated to be from 45 to 60  $\mu\text{m}$ . Since their

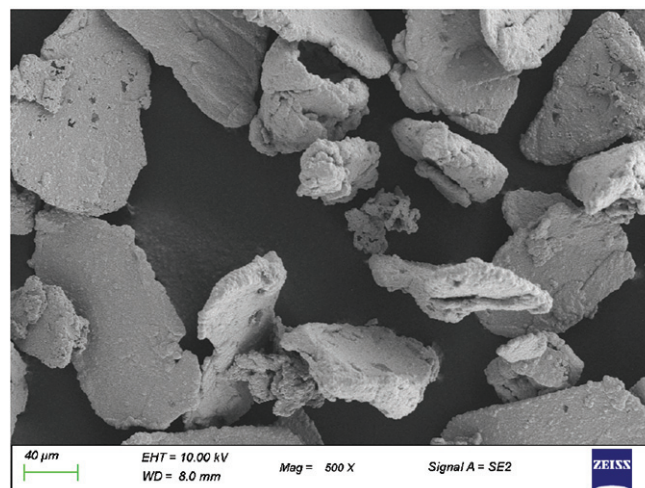


Figure 1: SEM Morphology of 308NS (NiGr) powder

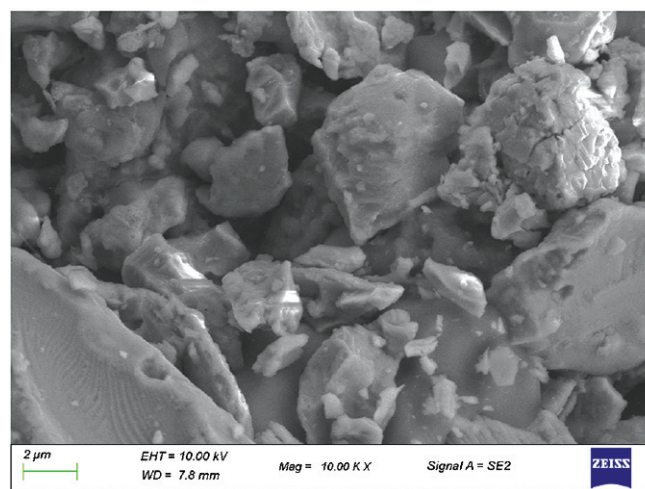


Figure 2: SEM Morphology of FeS powder

morphology is heterogenous, there are numerous procedures that can be used to create powders viable for thermal spraying. The best grains are those that resemble ball shaped as it boosts the looseness and makes it easier for the entry to the spray stream convincingly. Additionally, the whole grain can be heated evenly while residing in the spray stream due to its spherical shape.

SEM measurements of powder particles are made using the energy-dispersive X-ray analysis (EDAX) technique. A modern SEM typically comes with an EDS X-ray spectrophotometer, which is used in this approach to evaluate the particles by activation. After maintaining the appropriate instrumental conditions, this approach provides accurate findings for element detection in addition to determining their concentration. Figures 3 and 4 respectively exhibit NiGr and FeS powders utilized in the HVOF process that have had their SEMs combined with EDAX.

In the XRD analysis of the as-received powder, the high-intensity peaks of nickel, graphite (in form of carbon), and iron, sulphur phase can be seen, as shown in Figures 5 and 6 respectively.

Micrographs of coatings on the SAE1008 cold rolled steel substrate is shown in Figure 7. This gives a clear indication on the surface inter action and the coating details with viable characteristic features involved.

The measurements taken at the coating substrate contact are known as microhardness values. Figure 8 illustrates the micro hardness profile of each coating as a function of proximity to the coating substrate contact. It is discovered that the microhardness of the coatings varies along the cross-section, and a further significant rise in micro-hardness values were measured on the substrate region closer to the coating substrate-interface<sup>8</sup>. The hardness values and the thickness of coating measurements are tabulated in Table 4.

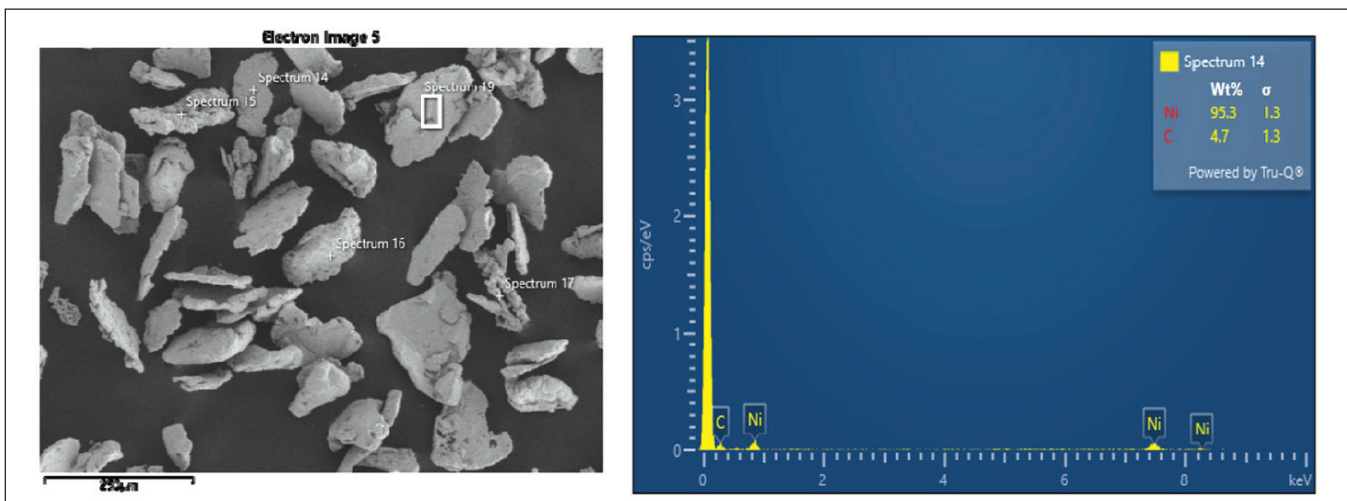


Figure 3 SEM with EDAX of 308NS (NiGr) powder

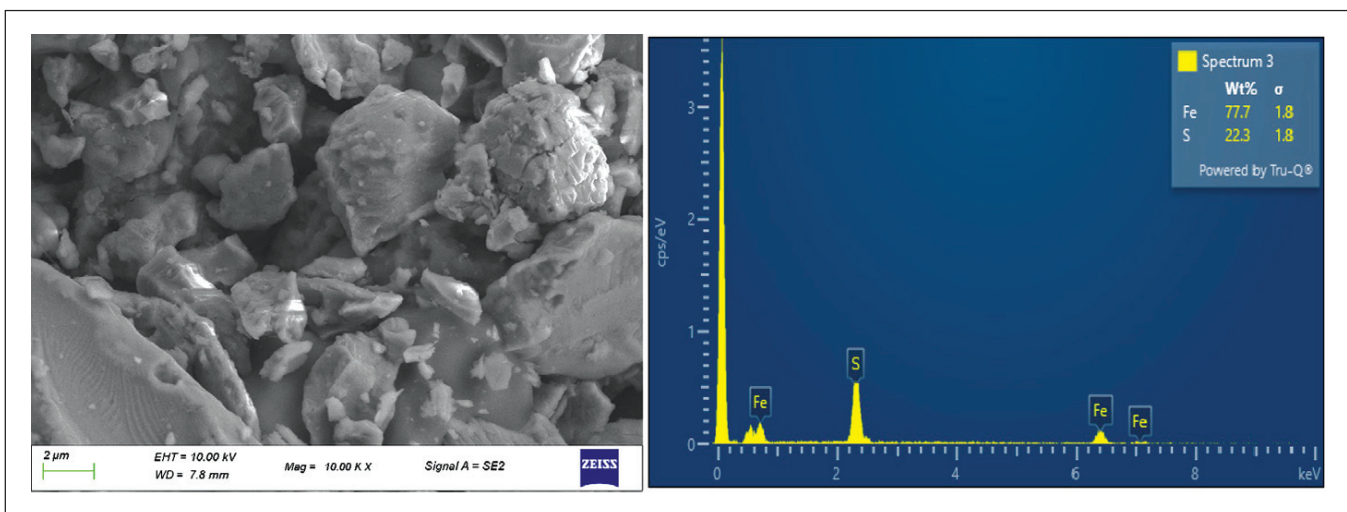


Figure 4 SEM with EDAX of FeS powder

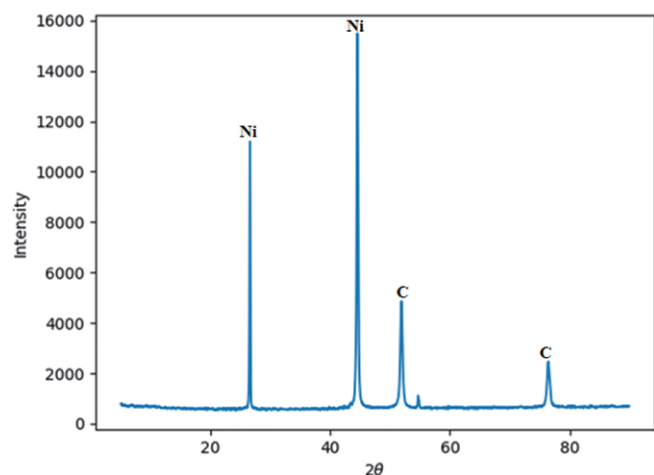


Figure 5: XRD of 308NS (NiGr) powder

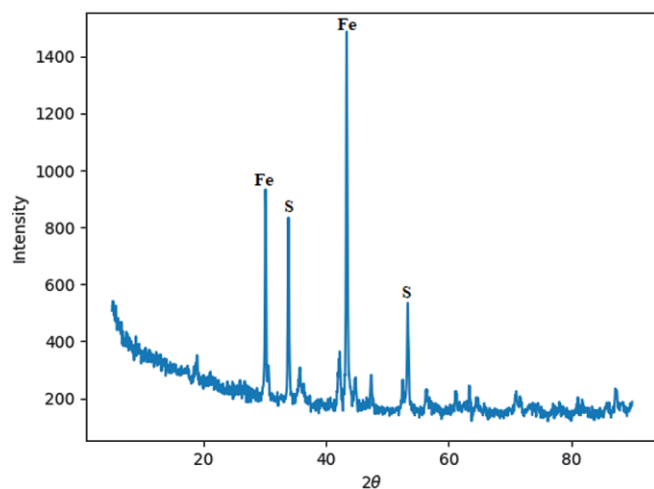


Figure 6: XRD of FeS powder

Table 4

Sl No.	Sample Identification	Hardness Value (HV <sub>1</sub> )	Coating Thickness(μm)
1.	OF <sub>1</sub>	282	275
2.	OF <sub>2</sub>	272	260
3.	OF <sub>3</sub>	275	275
4.	OF <sub>4</sub>	270	255
5.	OF <sub>5</sub>	263	250

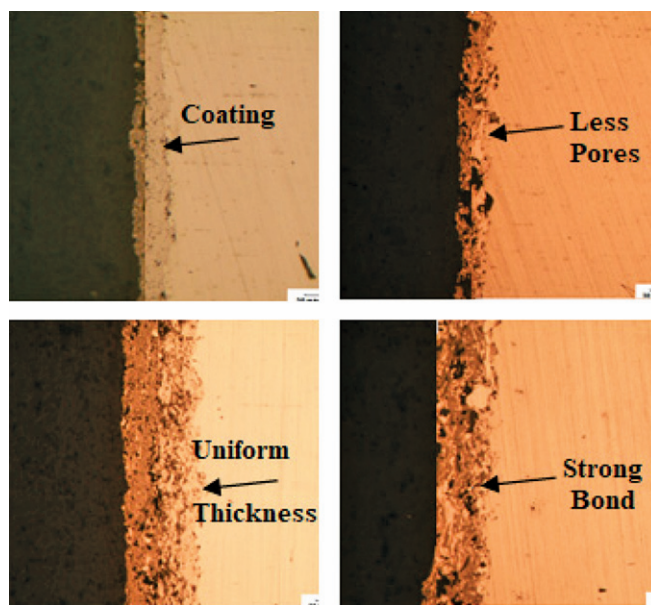


Figure 7: Coating on the substrate

## 4.0 Conclusions

The most popular feedstocks for thermal spraying are powders, and the powder shape has a significant impact on the effectiveness of feeding and injection systems as well as deposition and coating build-up mechanisms. Spherical particles avoid interruptions in feeding and blockage of injections due to their flowability. Additionally, the behaviour of spherical particles is predictable and under control because of their uniform morphology. This study aims to provide the findings of the ongoing research into the thermal spray coating on SAE1008 steel substrate material utilizing a high velocity-oxy fuel (HVOF) thermal spray system.

- By manually mixing and agglomerating a feedstock made up of nickel-graphite/ferrous sulphide/MWCNT, the requirements for the HVOF spraying process were effectively met.
- Excellent homogeneity and uniformity could be seen in the SEM micrograph of the coated cross-sections.
- The coating had excellent contact with the substrate, little porosity, and least to no oxide content. The layer is roughly 250 to 275 microns thick when measured according to the ASTM standards. These coatings did not contain any non-melted or semi-melted particles.

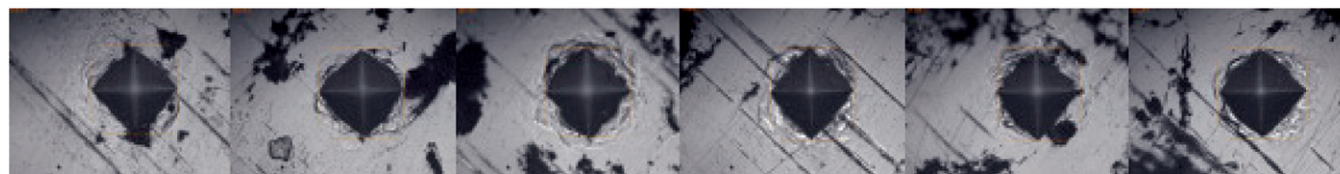


Figure 8: Coating on the substrate

- Coating and substrate microhardness values were evaluated and found to be H<sup>272</sup> HV and 98 HV, respectively. This accounts to coating are thrice the hardness of substrate material.
- The coating has a microstructure that is homogeneous, consistent, dense, and free of the least number of pores.
- It was determined that the coating had a porosity value of less than 1.5%.

As a result, it can be enunciated that HVOF composite spray coatings could easily serve as a strong replacement to chrome plated coatings wherein the manufacture process could be stopped due to its carcinogenetic nature.

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