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## Splat morphology of WC-CO powder at different flow rate

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### Abstract

Conventional Hard Chromium plating is produced from chromic acid solutions, which contain catalytic anions. Hard Chromium plating has as a main properties high hardness and resistance against corrosion. It has also a good wear behavior (abrasive and friction wear). Hard Chromium is widely applied in the engineering industry (aerospace, automotive, naval. etc.). The problem with Hard Chrome is not with the plated metal, but with the environmental problems associated with the plating process. Hard Chromium plating uses chromic acid, which releases fumes into the air during the plating process. This mist contains chromium +6 ions that are carcinogenic and can cause other medical problems, such as performed nasal passages and skin rashes. Due to these reasons the industries has enhanced the research of an alternative process for hard chromium coating. In this paper we studied that Hard Chromium. Thermal Spray Coatings, e.g., WC-Co is a possible substitute of Hard Chromium plating.

**Keywords:** Hard chromium, WC-Co powder, thermal spraying, spat, X-ray diffraction, electroplating

### 1. Introduction

A coating is a covering that is applied to an object to protect it or change its appearing. They may be applied as liquids, solids or gases. The material on which the coating is deposited is usually known as substrate. "Coatings" may also be formed by other processes such as melt/solidification (e.g., laser glazing technique), by mechanical bonding of a surface layer (e.g., roll bonding), by mechanical deformation (e.g., shot peening), or other processes which change the properties without changing the composition.

List of Coating Techniques

(1) chemical Vapor Deposition

(2) physical Vapor Deposition

- arc-PVD
- Cathodic arc deposition
- EBPVD
- Pulsed laser deposition
- Sputter deposition

(3) Others

- Anodizing
- Electroplating
- Ion Implantation
- Plasma Spraying, etc.

#### 1.1 Advantages of Coating

- Resistance to corrosion
- Increases wear resistance
- Increases hardness
- High temperature resistance, Etc.

#### 1.2 Applications of Coating

The applications of coating in current technology may be classed into the following generic areas:

Optically Functional

- Laser Optics (reflective and transmitting),

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- Architectural glazing,
- Home mirrors,
- Automotive rear view mirrors,
- Reflective and anti-reflective coatings,
- Optically absorbing coatings,
- Selective solar absorbers.

#### Electrically Functional

- Electrical conductors,
- Electrical contacts,
- Active solid state device,
- Electrical insulators,
- Solar cells.

#### Mechanically Functional

- Lubrication films,
- Wear and erosion resistance coating,
- Diffusion barriers,
- Hard coating for cutting tools.

#### Chemically Functional

- Corrosion resistant coating,
- Catalytic coatings,
- Engine blades and vanes,
- Battery strips,
- Marine use equipment.

### 1.3 Thermal Spraying

Thermal Spray is a coating process that provides a functional surface to protect or improve the performance of an affordable substrate or component. Almost material can be thermally sprayed - which is why thermal spray has been used world wide to provide corrosion protection, protection from wear and abrasion, etc. All types of materials, metals, polymers and ceramics can be coated using thermal spraying. There are quite a few thermal spraying process available, e.g., flame spraying, high velocity oxy fuel spray, electric arc spraying, plasma spraying, etc. The present work is concerned with plasma spraying. The Plasma Spray Process is basically the spraying of molten or heat softened material onto a surface to provide a coating. Material in the form of powder is injected into a very high temperature plasma flame, where it is rapidly heated and accelerated to a high velocity. The molten powders impact on the substrate surface and rapidly cool forming splats. The consecutive layers of splats result in a coating.

Thermal Spray is a coating process that provides a functional surface to protect or improve the performance of an affordable substrate or component. Almost any kind and form of material can be thermally sprayed - which is why thermal spray has been used world wide to provide corrosion protection, protect from wear and abrasion, restore and repair components, and more.

### 1.4 The hard chrome plating

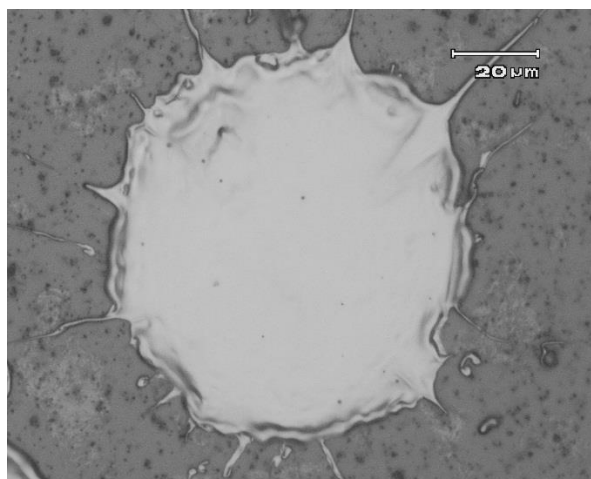
Hard Chrome is chromium plating which has been done by electrolytic process utilizing chromic acid based electrolyte for wear resistance. With its low co-efficient of friction, high hardness, moderate corrosion resistance, the process finds wide applications for prolonging the life of the components. A typical hard chrome plating process consists of a chromic acid based solution in plating bath. The D.C. current is required for the deposition of chromium which is supplied by an A.C. to D.C. rectifier. The component to be plated is connected to the

negative, whereas lead anodes are used to complete the circuit. Hard chromium plating is chrome plating that has been applied as a fairly heavy coating (usually measured in thousandths of an inch) for wear resistance, lubricity, oil retention, and other 'wear' purposes. Some examples would be hydraulic cylinder rods, rollers, piston rings, mold surfaces, thread guides, gun bores, etc. 'Hard chrome' is not really harder than other chrome plating, it is called hard chromium because it is thick enough that a hardness measurement can be performed on it and the chrome hardness can be measured, whereas thinner plating will break like an eggshell if a hardness test is conducted, so its hardness can't really be measured directly. During the process it produces chromic ions (hexavalent) in the environment which is highly carcinogen and causes medical problems to the people. The technique of thermal spraying has been proposed since several years ago, as an alternative to Cr electrode position, a process characterized by the need of post-deposition handling of a large amount of toxic slurry wastes. Chromium oxide and chromium carbide coatings, as well as Cr electrodeposits find applications, mainly, on the wear protection of metallic components participating in several tribosystems.

Influence of microstructure flaws on the tribological performance of Cr-based thermal-sprayed ceramic coatings was studied by P. Psyllaki *et al.* <sup>[1]</sup> In the present study, three different thermal spraying techniques were applied for the deposition of such ceramic coatings onto stainless steel substrates; namely, Flame Spraying (FS) and Atmospheric Plasma Spraying (APS) were employed for the deposition of chromium oxide coating, whilst High Velocity Oxygen Fuel (HVOF) technique for the elaboration of chromium carbide ones.

Comparative study of thermally sprayed coatings under different types of wear conditions for hard chromium replacement was done by S. Houdkova *et al.* <sup>[2]</sup> The tribological properties of part surfaces, namely their wear resistance and friction properties, are decisive in many cases for their proper function. To improve surface properties, it is possible to create hard, wear-resistant coatings by thermal spray technologies. The effect of deposition rate on microstructural evolution in WC-Co-Cr coatings deposited by high-velocity oxy-fuel thermal spray process was studied by K. Farokhzadeh *et al.* <sup>[3]</sup>. Tungsten carbide-reinforced cermet composites are widely used as wear- and erosion-resistant coatings due to their combination of hardness, strength, toughness and thermal properties. In this study, WC-Co-Cr coatings were deposited using high-velocity oxy-fuel thermal spray technology under different kinematic spray parameters. The microstructure of coatings was investigated using scanning electron microscopy and transmission electron microscopy techniques, and elemental/phase analysis was completed using x-ray diffraction, electron energy loss spectroscopy and x-ray energy-dispersive spectroscopy. A study aimed at developing a range of cermets coatings to replace hard chrome has been undertaken by Aw Poh Koon *et al.* <sup>[4]</sup> Typically, WC-Co coatings applied by HVOF are being used as a replacement for hard chrome. In this project, CrC-NiCr, WC-Co-Cr and WC-CrC-Ni powders will be used for coating development and evaluation. These materials are expected to have better corrosion resistance than the well-known WC-CO coatings. Various critical coating properties such as coating microstructure, phase changes due to thermal processing, adhesion strength to substrate material, corrosion, wear and fatigue tests were tested and compared against hard chrome.

## 1.5 Splat



**Fig 1:** Optical micrograph of WC-Co splat.

In thermal spraying molten and soft particles deform and spread on impact with the substrate. These deformed particles are known as splats. These are the unit of thermally sprayed coatings. The quality of the coating strongly depends on splat geometry, adhesion between the first layer of splat and the substrate and inter-splat cohesion. Hence, a study of splat is very important the building block of the coating. Investigations on splat/substrate interactions during the plasma spray process have been carried out using two high speed pyrometers. This measurement system allows study of particle flattening and splat cooling as a function of particle parameters (size, velocity, and temperature) at impact. This report is confined to the study of the effect of impact particle velocity on spreading and cooling processes on a smooth substrate kept at a temperature below 100 °C [6]. Impacts of plasma-sprayed molybdenum particles were monitored by detecting thermal radiation emitted by the hot particles when they flatten on the substrate surface. Evolution of the light intensity collected at two different wavelengths was used to obtain information about flattening time, flattening degree, and cooling time of the impinging particles. Variations of these parameters with substrate surface roughness were investigated on glass and molybdenum substrates. The substrate roughness significantly influenced the flattening degree and flattening time of the particles: the smoother the substrate, the larger the surface of the splats and the longer the flattening time. The cooling time, as determined from the decay time of the light signals after impact, was shorter on smooth substrates. In this case, the temperature of the splats was not radially uniform, with a lower cooling rate at the periphery [7]. The influence of the substrate surface temperature and oxidation state on the flattening of alumina and stainless steel particles and the morphology of resulting splats were studied by A.A. Syed *et al.* [8]. Particles were sprayed by a D.C. plasma gun on polished plain carbon steel and low alloy steel substrates preheated by plasma jet at different temperatures in air or in an oxidation limiting nitrogen shroud system added around substrates. Many properties (thermal, electrical, mechanical) of thermal sprayed coatings are strongly linked to the real contacts between the “piled-up” splats. The quality of this contact depends on droplet parameters at impact (size, temperature, velocity) and substrate parameters (temperature, topography). Two different

techniques have been developed in order to study the plasma sprayed particle behavior at impact. The first one allows direct studying under direct current (dc) plasma spray conditions, while the latter one, based on the millimeter-sized free-falling drop, enables the visualization of flattening phenomena, but at larger (about three orders of magnitude) time and size scales [9].

## 2. Experimental Details

The powders used for this study along with the spray parameters are shown in Table 2.1. The as received powders have been characterized using a Phillips x-ray diffractometer and a JEOL JSM 8000 scanning electron microscope (SEM). Low carbon steel test coupons have served as substrates. All substrates have been grit blasted using a suction type grit blasting cabinet. For grit blasting purpose alumina grits of grit size 60 is used at a pressure of 7 bars and a standoff of 100 mm. The roughness of the blasted surface is around 5 μm. Plasma spraying has been done using a Sulzer Metco 3 MB plasmatron. For splat collection purpose samples are held in a tongue and quickly passed through the plasma plume. Then the splats are observed under a DP 12 Olympus optical microscope. On the other hand the coated samples have been sliced and the cross sections are polished metallographically. These cross sections are observed using scanning electron microscope (SEM) and the phases are studied using a Phillips x-ray diffractometer.

For each specimen around 10 readings have been taken and the average is reported. The tribological characterization for both friction and wear has been done in a DUCOM pin on disc tribometer.

**Table 2.1:** The parameters selected for the experiment are stated below:-

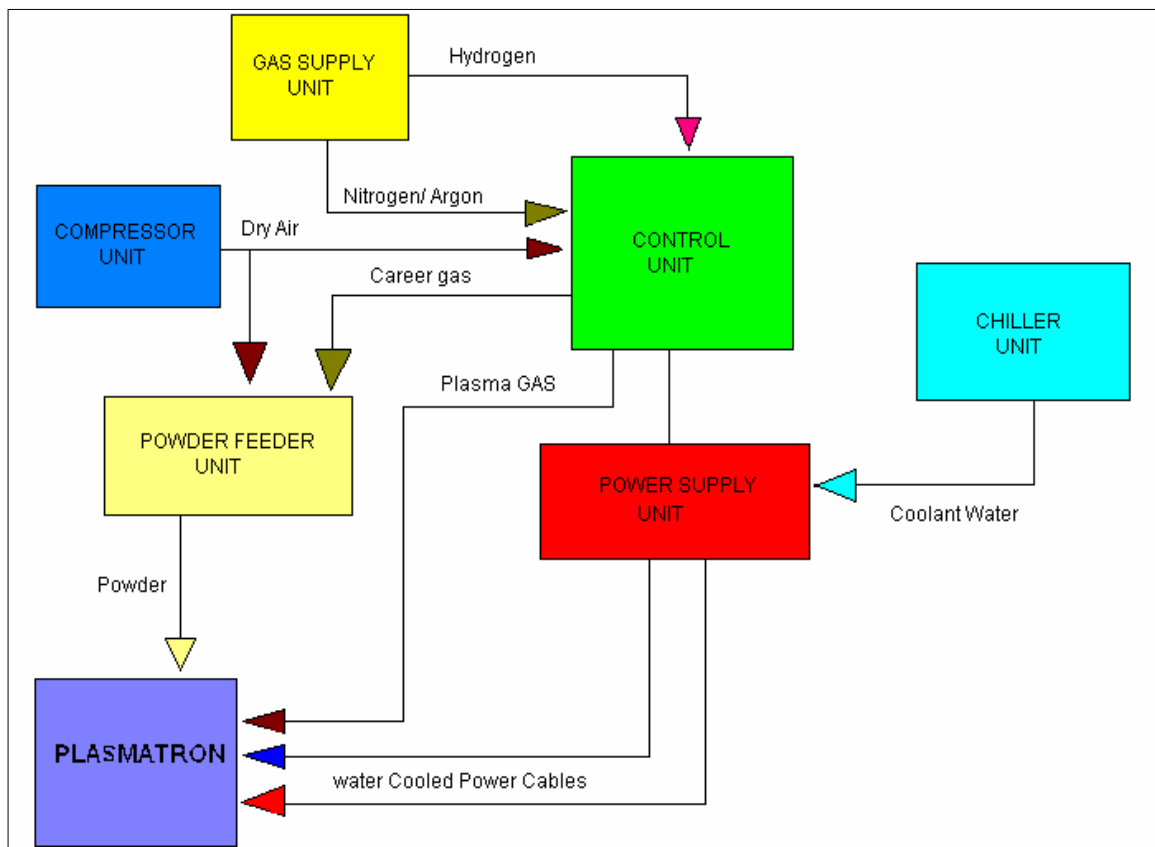
1	Powder(Metco 72F-NS)	WC- 12 wt% Co
2	Powder size(72F-NS), μm	-45+15
3	Stand of distance (SOD), inch	6 & 4
4	Career gas(nitrogen) flow rate, slpm	37.5
5	Nozzle diameter(ND), mm	7 & 5
6	Arc current, A	500
7	Arc voltage,V	65
8	Powder injection angle	90°
10	Secondary gas (hydrogen) flow rate, slpm	10 & 20
11	Primary gas (nitrogen) flow rate, slpm	80
12	Powder flow rate, kg/hr	1.5
13	Powder heating temperature, °C	80
14	Substrate material	C20 mild steel
15	Preheating of the substrate, °C	At room temp., 100 & 200

### 2.1 Experimental set up

The whole plasma spraying set up is consists of following equipments:

1. The plasma gun
2. Power supply unit
3. Chilled water supply unit
4. Powder feeder unit
5. Control unit
6. Gases, hoses and regulators etc.

The experimental set up used for the deposition of alumina based coating tool set up is schematically shown in Figure 2.1.



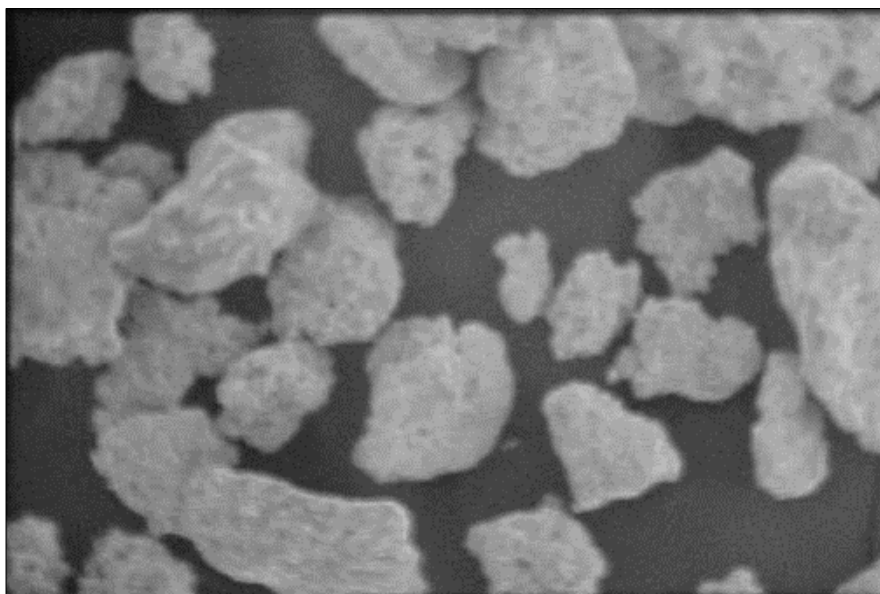
**Fig 2.1:** Schematic diagram of plasma spraying set up

The plasma spraying system mainly consists of a chiller unit, power supply unit, compressor unit, powder feeder, control panel and plasma gun. As the chiller unit gets on, it starts cooling the water which moves through a centrifugal pump into the gun for the extraction of heat. For generating plasma, first nitrogen gas passes through the control panel to the gun. Then with the help of power unit, spark is generated between the cathode (copper nozzle) and anode (tungsten). This spark

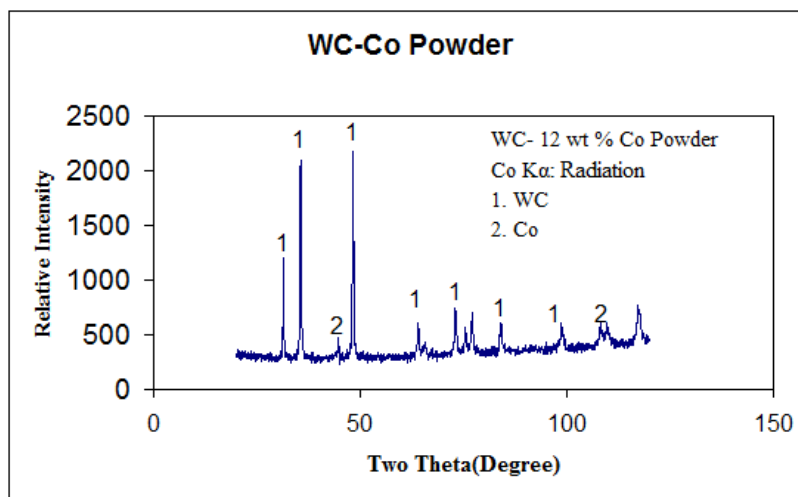
ionizes the nitrogen gas and produce tremendous amount of heat which move through the nozzle in a flame shape. The flame get intensify with the use of secondary gas ( $H_2$ ). Through the powder feeder unit, powder starts supplying. The powder started melting in the flame and gets strike on the substrate surface. The major role of control unit is to control the current, primary and secondary gas.

### 3. Results and discussions

#### 3.1 Characterization of powder



**Fig 3.1:** Secondary electron image of the WC-Co powder

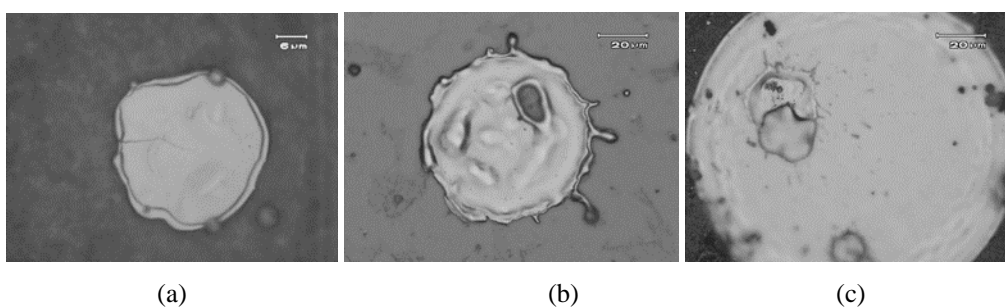


**Fig 3.2:** X-ray diffraction pattern of WC-Co powder.

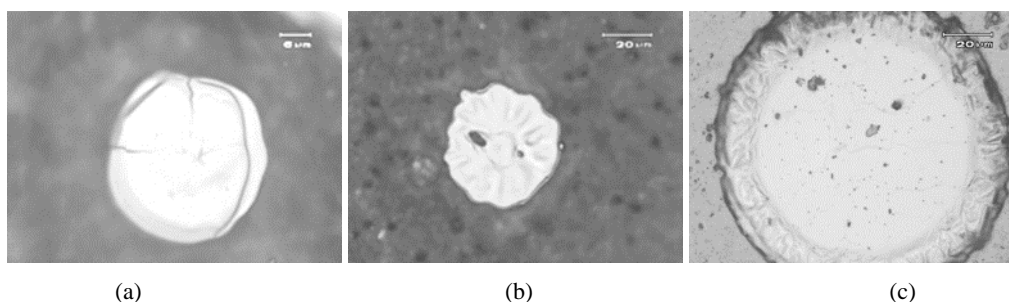
The morphology of WC–12%wt Co powders is shown in the Figure 3.1. It can be seen that the feedstock is irregular shaped with a size in the range of  $-45+15\ \mu\text{m}$ . An X-ray diffraction spectrum of the starting powder is shown in Figure 3.2. WC and cobalt phases are detected in the starting powders.

### 3.2 Splat Morphology

In thermal spraying the individual molten particles collide with the substrate and assume the shape of a pancake after deformation. These are known as splat; the building block of the coating.



**Fig 3.3:** Optical Micrographs of WC – Co splats obtained using (a) No Preheating, (b) preheating at  $100^{\circ}\text{C}$  and (c) preheating at  $200^{\circ}\text{C}$  using Nozzle diameter- 7mm and secondary gas flow rate- 10slpm.



**Fig 3.4:** Optical Micrographs of WC – Co splats obtained using (a) No Preheating, (b) preheating at  $100^{\circ}\text{C}$  and (c) preheating at  $200^{\circ}\text{C}$  using Nozzle diameter- 7mm and secondary gas flow rate- 20slpm.

In this present work a thorough study of splats is made on splat morphology for air plasma sprayed (APS) WC-Co powder using different hydrogen flow rates and different preheating temperatures on polished low carbon steel samples.

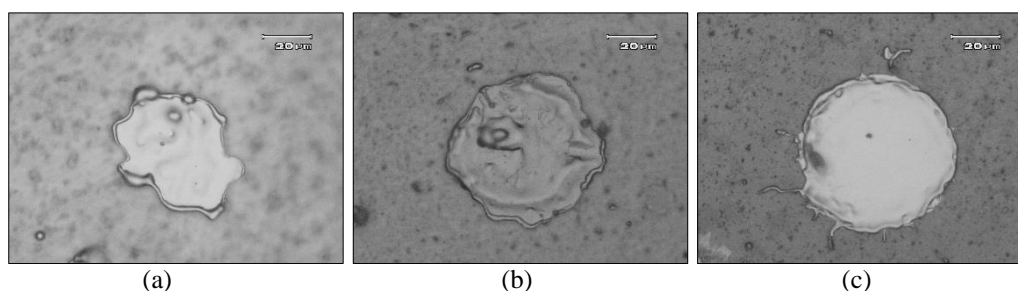
Figure 3.3 is the optical micrographs of the splats taken on the polished low carbon steel surfaces using a nozzle diameter of 7mm, secondary gas flow rate of 10 slpm and at different other parameters shown in Table 2.1. Figure 3.3(a) shows a splat, while the substrate surface has not been preheated. Under the circumstances the splat is deformed but not well spread. It has failed to wet the substrate surface. As the preheating temperature is raised to  $100^{\circ}\text{C}$  (Figure 3.3 (b)), a

partial change in the scenario is observed. In this case a partial spreading of the liquid droplet has been found to occur. As the preheating temperature is raised to  $200^{\circ}\text{C}$ , a round well formed splat is obtained. This is shown in Figure 3.3(c). Even after ultrasonic cleaning, a thin layer of absorbates and condensates remain attached to the substrate surface. This layer of absorbates and condensates prevents proper contact and heat transfer between the splat and substrate. On preheating of the substrate the layer of absorbates and condensates goes away and the substrate surface is well wetted with the liquid splat. In this case when no preheating has been used, no wetting of the substrate surface has been observed by the splat. On raising the preheating temperature

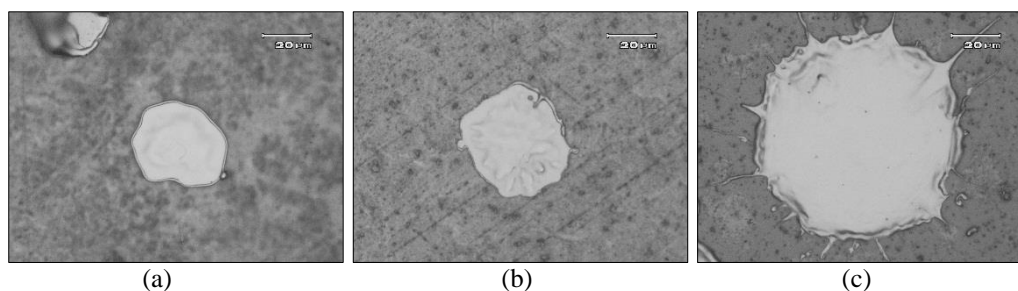
to 100 °C, only a partial removal of the absorbates and condensates layer is found to occur, and that is why a partial wetting of the substrate is observed. On further raising the preheating temperature to 200 °C, a round and well formed splat is obtained. Figure 3.4 shows the splat geometries obtained using a secondary gas flow rate of 20 slpm instead of 10 slpm. All other conditions similar to the splats recorded in Figure 3.3. The splat geometries in Figure 3.4 are very similar to those in Figure 3.3. This indicates that a rise in secondary gas flow rate does not cause a change in splat geometry.

Figure 3.5 and 3.6 show similar micrographs of splats taken under different conditions as stated in Table 2.1. In this case a nozzle with 5 mm diameter is used replacing the previous

nozzle of diameter 7 mm. The scenario which has been described above for Figure 3.3 is valid for these conditions as well. In Figure 3.5(c) and 3.6(c) some splashing is observed. Sometimes depending on the substrate and splat contact, a thin layer of the splat undergoes freezing immediately on contact, while the remnant of the liquid in the splat is still in motion. In such cases, splashing occurs around the periphery of the splat. A reduction in nozzle diameter results in higher plasma enthalpy and temperature and this in turn results in higher particle temperature. The viscosity of the liquid decreases at a higher temperature and consequently splashing occurs.



**Fig 3.5:** Optical Micrographs of WC – Co splats obtained using (a) No Preheating, (b) preheating at 100°C and (c) preheating at 200°C using Nozzle diameter- 5mm and secondary gas flow rate- 10slpm.



**Fig 3.6:** Optical Micrographs of WC – Co splats obtained using (a) No Preheating, (b) preheating at 100°C and (c) preheating at 200°C using Nozzle diameter- 5mm and secondary gas flow rate- 20slpm.

Degree of splashing in splat formation can vary considerably. One can obtain either extensively splashed formed or splat with finger like projections along the periphery. In these cases only limited splashing is observed while a nozzle diameter of 5 mm is used.

It seems from the above discussion that it is possible to produce dense and hard WC – Co coatings using APS. Such coatings can have a greater wear resistance than electroplated hard chrome and hence can be used as a hard chrome replacement solution. While using this solution one must be cautious about the parametric combinations so as not to sacrifice density and hardness. In addition, decomposition of WC to form  $W_2C$  should be prevented.

#### 4. Conclusions

The conclusions of the present work are the following: Preheating controls the geometry of splats. Upon preheating the substrate to 200 °C adherent layer of contaminant is vaporized from the substrate and good contact between substrate and coating takes place. As a result round well formed splats are obtained. In absence of preheating the substrate is not wetted at all. Preheating to an intermediate temperature, i.e., 100 °C, results in partial removal of absorbates and condensates and this in turn results in partial wetting of the substrate by splat. A rise in secondary gas flow

rate to 20 slpm does not cause any major change in splat geometry.

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