Thermal sprayed coatings are widely used for industrial applications. One of the main characteristics that have to be evaluated in a coating is its microstructure that finally determines the coating performance. Several techniques and processes are available for coatings deposition and new materials have been incorporated in the long list of the available ones. Therefore, since microstructure is a key factor to be evaluated, its preparation has to follow some rules in order to inhibit incorrect statements that can arise from wrong interpretation of an incorrect sample preparation. In this work, a series of distinctive materials are thermally sprayed onto low carbon steel substrates. The metallographic preparation of samples for the different coatings is presented and the effects of correct or wrong sample preparation are discussed and correlated to coating microstructure and process characteristics. The coatings were applied by Air Plasma Spray (APS) and Electric Arc Spray.

1 Introduction

Thermal sprayed coatings are used for several applications, including wear and corrosion protection, thermal barrier, thermal conductivity and diffusivity [1,2,3]. In this sense, to get such special characteristics, a wide range of distinctive materials is utilized like several metals and metallic alloys, polymers, ceramics and cermet, applied by distinct processes that include Air Plasma Spray (APS), Detonation Gun, Flame Spray (FSP), Arc Spray and High Velocity Oxygen-Fuel (HVOF). THE intensive use of such types of coatings in industries like aerospace, petrochemical, and automotive, to mention just some, makes the quality demand for coatings extremely high.

Microstructural analysis is used to characterize both the substrate and the coating, as well as adhesion and cohesion interactions. The main microstructural characteristic observed through microscopy they are morphology of the coating, thickness of the layers, porosity, unmelted particles, cracks, coating/substrate interface and oxides. A good contact between the coating and the substrate is a fundamental factor for good adhesion, integrity and coating performance. The interface should be homogeneous, completing all the irregularities. Pores, strange particles, like dirt, and oxides are harmful to the adhesion and should be kept minimum. Through Optical Microscopy and Scanning Electron Microscopy (SEM) the interface can be studied in a detailed way, provided the sample has been carefully prepared so that artifacts are not present. Therefore, the use of several methods of coatings evaluation is motivated and, among them, metallography is highly important as stated by several authors [4,5,6] and it is fundamental in the coating quality management and consequently in the effectiveness of its application [7,8]. A better understanding of coatings microstructure definitively helps a most scientific development of the sprayed coatings. This improves the process product development, quality and methods and processes management.

2 Experimental Procedure

For the development of this work, several coatings of different feedstock materials were applied on AISI 1020 carbon steel substrate. The spraying processes for metallic and ceramic coatings were Electric Arc and Air Plasma Spray (APS), respectively. Al2O3 coatings were applied by Air Plasma Spraying (METCO 7MB, Westbury, NY, USA). Copper, Aluminum, 420 Stainless Steel, Molybdenum and Babbit coatings were applied by Electric Arc (8830 - Tafa, Inc., Concord, NH, USA). The materials were applied in commercial conditions of use with optimized parameters, generating coatings in optimized conditions. The cut of samples was accomplished in a precision cutoff machine Isomet 2000 (Buehler, USA).

Abrasive disc was used for non-ferrous materials. Although there are some recommendations for the use of diamond disks in the cut of ceramic materials, an abrasive disc was chosen due to the ferrous nature of the substrate that would quickly wear away the expensive diamond disk. It should be pointed that the great chemical affinity between carbon and iron provokes excessive waste of diamond tools. To guarantee the flatness minimizing edge rounding of the polished surface, hot mounting was accomplished using a resin-containing fiberglass, which is harder than common Bakelite. This is a simple and very useful procedure.

Grinding and polishing were carried out in an automatic polish machine Motopol 2000 (Buehler, USA), allowing the control of applied load, wheel rotation speed, specimens holder and wheel rotation direction control, among other things. The control of these parameters allows better results compared to manual grinding and polishing. The parameters used in the metallographic preparation for the applied materials are presented in Table 1. Grinding papers of SiC and diamond pastes were used for polishing.

Some samples were chemically etched after polishing. These attacks will be specified later on. The coatings were analyzed by optical microscopy in an Optical Microscope Carl Zeiss, model Neophot 32. The porosity, amount of oxides and images were accomplished through an Image Analysis System Leica Q500.
Table 1. Grinding and polishing used parameters.

<table>
<thead>
<tr>
<th></th>
<th>Abrasive</th>
<th>Lubricant</th>
<th>Load (N)</th>
<th>Wheel rotation speed (rpm)</th>
<th>Time (min)</th>
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<tr>
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<td>Water</td>
<td>20</td>
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<td>20</td>
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<td>Alcohol</td>
<td>20</td>
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<td>6</td>
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<td></td>
<td>Paste .25 µm</td>
<td>Alcohol</td>
<td>20</td>
<td>150</td>
<td>7</td>
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</tbody>
</table>

3 Results and discussion

Figure 1 presents a representative image of a Copper coating. This sample was manually prepared and only polished, without chemical attack.

Some porosity can be observed (6.87 % according to image analysis measurement), which is typical of detachment of material from incorrect procedures in metallographic preparation. Figure 2 exhibits the same coating prepared in an automatic machine. Now, only the normal coating porosity (1.41 %) resulting from the spray process can be observed.

Figure 3 exhibits a representative image of a coating of 1080 carbon steel that was chemically etched after polishing with Nital 2%. Chemical etching in such material facilitates the identification of existent phases in the carbon steel structure, as well as porosity.

Comparing the images of Fig. 3 and 4, it can be noticed that in Fig. 3 present phases are much clearer facilitating its distinction and even quantification. In Fig. 4 from the sample without attack is more evident and easy to verify the amount of present oxides. The oxidation is relative to the use of compressed air in the Electric Arc Spray process as carrier gas. The oxidation degree can be minimized using an inert gas instead of compressed air, even making the process a little bit more expensive.
Figures 5 and 6 show representative images of a 420 stainless steel coating. In Fig. 5, it can be noticed the debonding of the coating from the substrate. This debonding could lead to the erroneous conclusion of process application problems and consequent low adhesion of the coating to the substrate. In fact, it is a debonding that happened due to problems in the metallographic preparation, specifically in the specimen cutting process.

Fig. 5. Microstructure of a 420 stainless steel coating debonded from the substrate

Even the high porosity level in the coating observed in Fig. 5 can be credited to inadequate polishing, since metallographic preparation of this sample was done with less steps of grinding, generating an aggressive specimen preparation. The sample showed in Fig. 6 was correctly prepared and its cutting was carried out in precision cutting machine, evidencing the good adhesion of the coating.

Fig. 6. Microstructure of a 420 stainless steel coating with good adhesion to the substrate

The sample prepared without etching, as seen in Fig. 7, facilitates porosity observation, mainly macroporosity that can be in the range of 1 to 10 µm [9] or be considered varying from 10 to 30 µm [10]. Chemical etching helps in grains structure and layers boundary observation.

Fig. 7. Polished Molybdenum coating

Fig. 8. Polished and etched Molybdenum coating

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Figure 9 shows an Aluminum coating with central coating particle detachment caused by excessive pressure in the grinding process. Aluminum is very sensitive to grinding and polishing work and it is important to keep this in mind while preparing specimens for observation at the microscope.

Fig. 9. Aluminum coating with central coating particle detachment

Figure 10 exhibits a coating of Zinc. Metallographic preparation of this kind of material type is quite difficult, since soft materials like Zinc, Aluminum and Babbit scratch out easily. To minimize the problem of risks carefully cleaning of the samples and polish cloths is fundamental. Another problem that can occur is smearing. The use of worn-out SiC papers favors smearing, therefore new SiC papers have to be used for such materials.

Fig. 10. Zinc coating
Fig. 9 Microstructure of an aluminum coating with particles detachment

Fig. 10 Coating of Zinc only polished

Figure 11 shows an \textit{Al}_2\textit{O}_3 coating applied with correct standoff distance (100 mm). In that illustration it can be seen that there is good contact between coating and substrate.

Fig. 11 \textit{Al}_2\textit{O}_3 coating applied with correct standoff distance exhibiting good adhesion.

On the other hand, Figure 12 shows a coating of the same material, deposited with the same power but with a longer standoff distance (200 mm). In this case, the contact between coating and substrate is not so good, presenting some flaws in the interface. This happens since a longer standoff distance means that particles tend to be colder when reaching the substrate, presenting, therefore, lower plasticity and more difficulty to fill the irregularities. In these two cases, the problem is a real process problem since metallographic was correct, not influencing in final result. In this metallographic preparation the sample was only polished, without any chemical attack.

Fig. 12 \textit{Al}_2\textit{O}_3 coating applied with 200 mm standoff presenting adhesive flaws.

Conclusions

The obtained results and the topics discussed in this work allow the following conclusions:

- Microstructure of coatings defines, in ultimately, coatings performance when in service for a wide variety of applications.

- Metallographic preparation is a fundamental aspect for correct interpretation of coatings microstructure evaluation for the several spraying processes used.

- Despite of long time discussion of the subject, new materials, new processes and their consequent peculiarities turn even more important the discussion on cares that have to be considered in the evaluation of tests and all quality rehearsals.

- This work has presented some practical aspects on the metallographic preparation and interpretation showing different materials and problems considerations related both the spraying process and the metallographic procedure.

4 Literature


