Optimization of 75% nickel – 25% graphite spray parameters to meet coating application design criteria

This paper outlines several application principals to enhance the user capability of nickel-graphite powders to meet engineered design coating criteria. For decades Oerlikon Metco nickel-graphite composite powders (Figure 1) have served the aerospace and IGT industries. Oerlikon Metco is well-known as the premium coatings solution provider for thermally sprayed clearance control solutions, having many years of experience and very extensive in-house expertise to manufacture, apply and test abradable powders and coatings.

By Brent Bartlett, Oerlikon Metco (Canada) Inc., Ft. Saskatchewan AB, Canada

1. General information

Over time, changes in equipment, personnel and engineering sourcing have led to the loss of many of the lessons learned in the coatings industry, and specifically by the users of these materials. The following technical information serves as a guide to enhance the application knowledge and overall usability of Durabrade 2223 and Metco 307NS-2 utilizing Oerlikon Metco coating application equipment.

Durabrade 2223 and Metco 307NS-2 are manufactured to the same specification and are used extensively in aerospace as gas path sealing systems in jet and IGT engines. The nickel-graphite powder is capable of producing coatings with properties that will satisfy many design criteria through alteration of the coating application parameters. Three separate Design of Experiments (DoE) were performed to better understand the coating application of nickel graphite. The experiments were devised to demonstrate Oerlikon Metco equipment and powder capability focused on gas parameters and the resulting coating properties attained. These DoE provide insight and guidance for optimization of parameters to meet the intended coating design criteria. A fourth set of tests were conducted using the most common equipment in the customer base, namely rotameters, to demonstrate parameter adjustments known to be used by the customer. This paper will demonstrate the lessons learned from this customer-focused testing.

2. Measuring properties

Measuring coating properties with confidence is the first step in achieving the desired coating with the right abradability to suit the design criteria. Hardness measured using the HR15Y scale is proven to be a reliable method of coating quality control when the method is controlled and repeatable. Keys to hardness measurement are coating thickness, surface preparation and statistical relevance of the data gathered. All hardness data reported in this document were generated on coated samples with a thickness of 2.05 ± 0.05 mm. All surface preparation was performed using fresh metallographic-quality 60 grit silicon carbide paper on flat specimens while moving the specimen over the grit surface with gentle force in a figure eight pattern. All hardness data points are the average of 15 impressions per specimen unless otherwise noted.

Coating cohesion testing following the practice defined in ASTM C633 is also a reliable method for characterizing the performance of the nickel-graphite coatings. Data shown in this paper was generated following the practice of ASTM C633. Other technical requirements for accurate data generation were the control of coating thickness and the use of glue wafers such as Cytec FM1000 applied under gravity load during curing to ensure the glue did not penetrate too far into the porous coating. Liquid glues should not be used as they have been proven to generate false cohesion test data.

When used in tandem, coating hardness and coating cohesion tests provide confidence for QA fit-for-use release. As shown in Figure 2, the two tests may be used to validate one another. The data in Figure 2 is from three separate DoE using two different lots sprayed with 9 different sets of gas

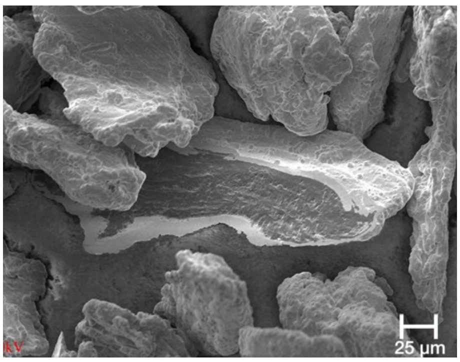


Figure 1. Durabrade 2223 Nickel-Graphite Powder

parameters and two different powder feed rates. The spraying and testing occurred over a period of one year and used both MultiCoat (mass flow control) and 3GF (flow tube) gas management systems. The physical property relationship remains strong even with negative hardness values reported.

The importance of consistent coating thickness for hardness measurement cannot be over emphasized. The curve shown in Figure 3 demonstrates that coating hardness increases as the coating thickness decreases, which indicates that the substrate hardness is influencing the data. Thicker coatings are required for accurate hardness measurement when soft coatings are produced. Each data point represents the average of three coupons with 15 impressions per coupon.

3. Coating application equipment capability

Paramount in any process is understanding the capability of the application equipment and the materials being applied. The experiments used to test these capabilities utilized current hardware presently in use by our customers and readily available to the industry.

All spray testing shown in this paper was performed using the equipment as defined in Table 1.

	MultiCoat System	3GF System
Controller	MultiCoat	3GF
Gas Control Type	Mass Flow	Flow Tube
Spray Gun	6P-II	6P-II
Siphon Plug	605 (4-jet)	605 (4-jet)
Nozzle	7C-D	7C-D
Air Cap	6P4	6P4
Air Jets	6P330 (parallel)	6P330 (parallel)
Powder Feeder	Twin 10-C	9MP

Table 1. Thermal spray application hardware used for DoE and 3GF parameter testing

A process gas capability map (Figure 4) was generated to demonstrate the Oerlikon Metco equipment capability using the defined hardware and input pressure settings as measured into the gas management systems. Changes in hardware, especially nozzles, will change the values represented in this map. It was demonstrated that the MultiCoat and the 3GF gas management systems produced very similar process maps (± 10% variation) and should be considered comparable when defining capability limits.

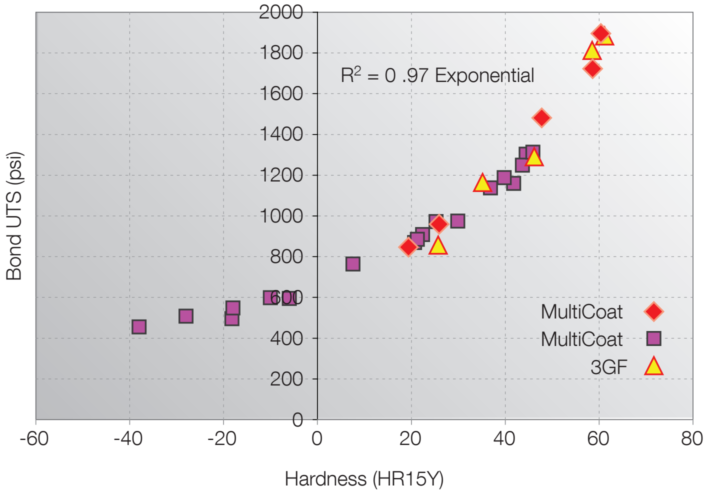


Figure 2: HR15Y Relationship to Cohesive Strength of Coating (Durabrade 2223 Compared in 3 DoE)

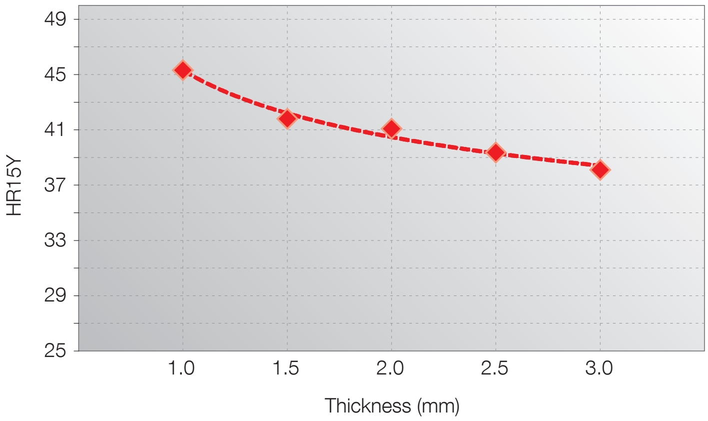
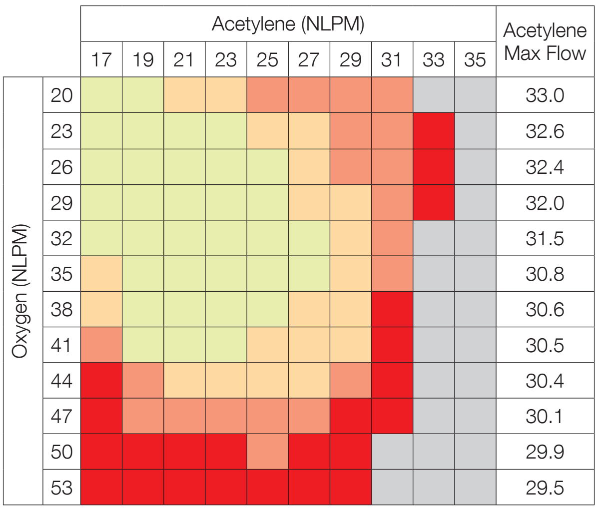


Figure 3: Hardness vs. Thickness (45 impressions per data point)
Note: Samples were milled to remove the influence of hand preparation on surface conditioning with grit paper.



Flow State	Spray Capability
Stable	Capable
Slightly unstable	Capable
More unstable	Marginal
Highly unstable	Unusable
Not possible	Gas flow not achievable

Constant Parameters		
C_2H_2	1.07 bar	15.5 psig
O_2	5 bar	72.5 psig
N_2	5 NLPM	10.6 SCFH
Air Cap	2.0 bar	29 psig

Figure 4: MultiCoat Process Map Capability

Besides other production advantages, the MultiCoat system offers the benefits of higher accuracy and precision of gas readings with no variation in interpretation of flow between operators. Values reported are for test purposes and some variation may exist in facilities due to hardware or other considerations.

The criteria for characterizing the gas jet are based on profile photos and observation. Figure 5 shows an indication of the criteria for characterizing the gas jet.

Capability of the powder was demonstrated by spraying coatings and measuring hardness and cohesive strength characteristics. Knowing the gas parameter limits established from the gas parameter mapping in Figure 4, a DoE was conducted which examined three variables: oxygen mass flow, acetylene mass flow and powder feed rate. The parameters used are illustrated in Figure 6. The blue diagonal lines represent the oxygen / acetylene gas equilibrium while the green diagonal line divides a theoretical oxidizing reducing flame gas mixture. Resulting hardness values are depicted in red. The DoE was then repeated twice in a scaled-down version using both MultiCoat and 3GF. Powders sprayed in three separate DoE using these parameters showed hardness and bond capabilities as indicated in Figure 2. The powder capability hardness range using the DoE parameters is HR15Y (-38 to +62) while the range of coating cohesive strength is 420 psi to 1900 psi. Powder capability not tested in the DoE can be further expanded through parameter changes illustrated in Figures 16 and 17.

4. Main effects variable

Of the three variables examined in the DoE, Figure 7 graphically depicts that oxygen has, by far, the strongest influence on coating hardness. This is true whether using the MultiCoat or the 3GF as a gas management system. By increasing the oxygen to acetylene ratio in the gas mixture, the hardness increases dramatically as shown in Figure 6, where hardness values (shown in red) are indicated for the 45 g/min feed rate.

The main effects plot shows that acetylene does have an influence up to 25 NLPM in the DoE but then the positive effect on increasing hardness is slightly reversed after this point.

Accuraspray diagnostic equipment was used to measure particle temperature for the DoE. An experiment was conducted

Oxygen Flow	Acetylene Flow	Flame State	Gas Jet
20 NLPM	17 NLPM	Stable	
20 NLPM	21 NLPM	Slightly unstable	
20 NLPM	25 NLPM	More unstable	
28 NLPM	27 NLPM	Slightly unstable	
34 NLPM	24 NLPM	Stable	
40 NLPM	27 NLPM	Slightly unstable	

Figure 5: Sample Gas Flows and Gas Jet Photos for Characterization of Flame Stability

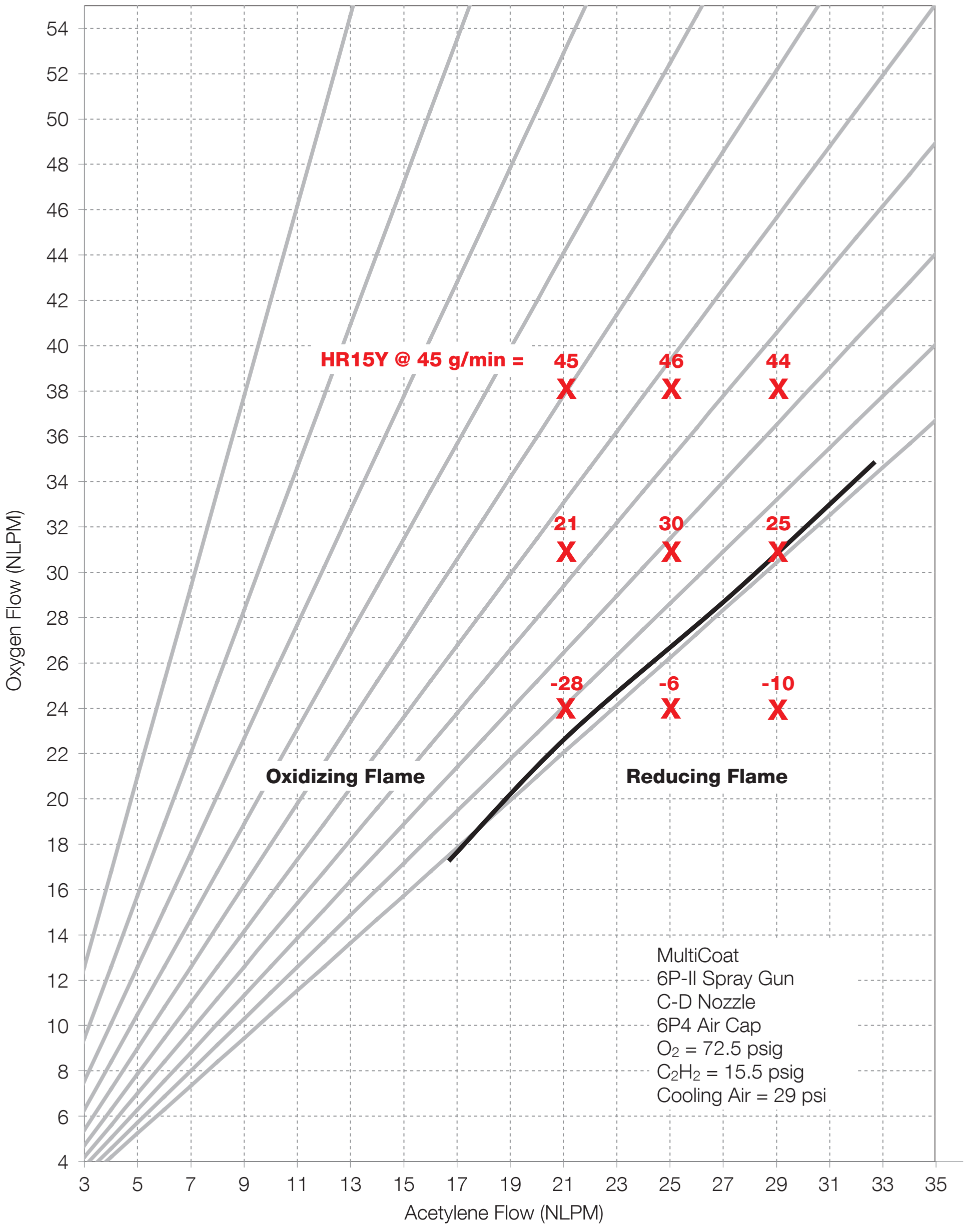


Figure 6: DoE MultiCoat Gas Parameter Map
[9 gas parameters (X) and 2 powder feed rates (45 g/min and 55 g/min) per parameter = 18 coatings]

where oxygen was held constant while increasing acetylene. The data in Figure 8 shows that particle temperature increases for parameters 1 and 2. Graphite is exothermic in the flame spray process and adds some degree of fuel, thus heat is added to the spray. Further investigation shows that in parameter 3, which has the highest acetylene flow, the increase in particle feed rate reduced the measured powder temperature. It may be concluded that:

- Oxygen is saturated by fuel
- The increased mass of the powder has decreased the enthalpy of the flame
- The closer we move to a reducing flame gas mixture the less efficient oxygen becomes at altering our coating hardness

Reduced powder feed rate consistently delivers harder coatings for nickel-graphite. The DoE showed that for every parameter there was an increase in hardness of the coating. Figure 9 graphically illustrates this consistency. Reduced feed rate will often yield somewhat higher deposit efficiency, but this is often offset by longer spray times to achieve the same coating thickness requirements.

5. Parameter effects on coating structure

The effect of this wide range of physical characteristics was examined at the chemical level within the coating. Oxygen, nickel and carbon were measured and the data correlated. Table 2 shows oxygen, nickel and carbon analysis for all nine sets of gas parameters which produced a hardness range of HR15Y -28 to +45.9. The analysis revealed the oxygen average range is 330 – 660 ppm. The range for nickel is 79.6 – 82.2 wt.% and the range for carbon (graphite) is 17.65 – 20.0 wt.%. Despite the wide range in hardness, the chemical changes are relatively minor. The data is validated in that carbon and nickel testing was performed on separate pieces of the same sample and the combined data is very near 100 wt.%. The hardest samples using the highest levels of oxygen in the spray parameters exhibit the least carbon in the coating indicating that some carbon is consumed in the process. The hardest samples also contain more nickel in the coating.

The photomicrographs in Figure 10 exhibit the nickel, graphite and porosity area levels associated with the gas parameters and hardness values as indicated above.

Visual comparison of microstructure is difficult to perform given the nature of the nickel

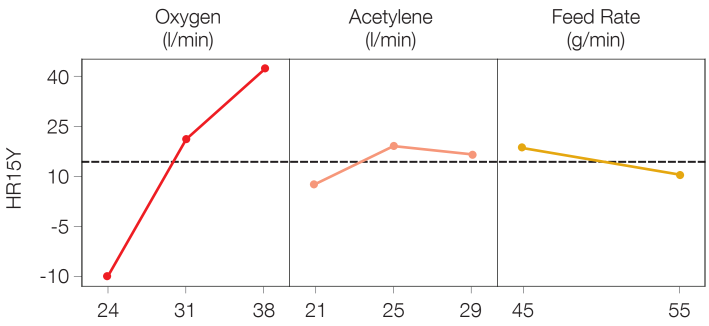


Figure 7: Main Effects Plot – Data Means for HR15Y

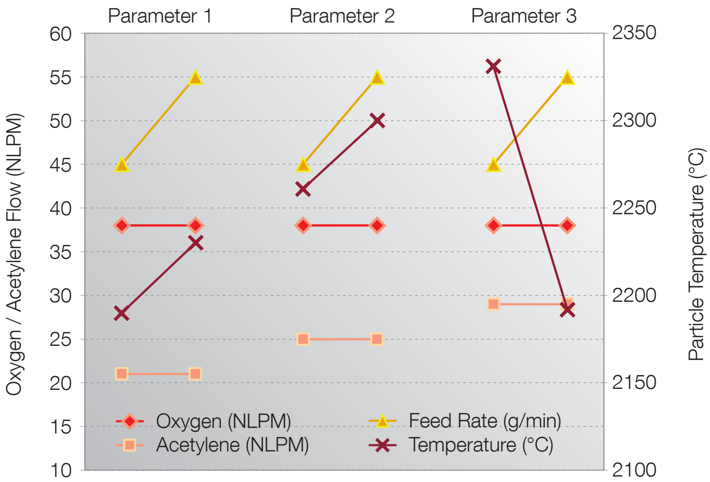


Figure 8: Graphite As a Fuel Source
Powder Feed Rate to Particle Temperature vs. O_2 / C_2H_2 Ratio

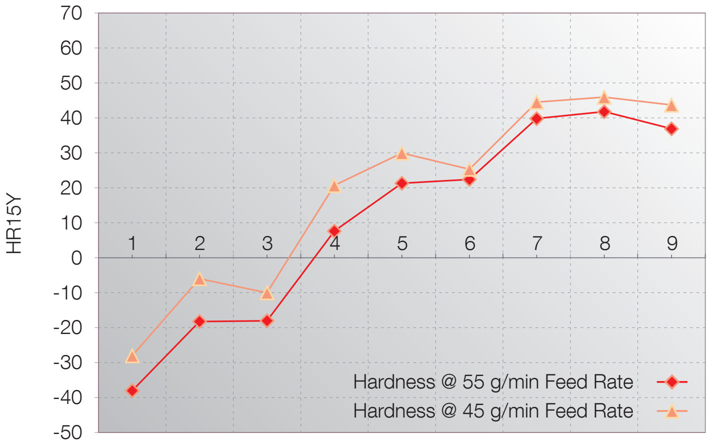


Figure 9: Effect of Powder Feed Rate on Hardness

graphite coating structure. Image analysis (IA) techniques were utilized to quantitatively compare structures based on hardness. It is understood that phase distribution analysis is not purely quantitative given the fact there are no standards of the same coating structure to calibrate the measurement device. The test method employed the use of an in-house reference standard as a baseline, as well as other quality controls on IA set-up, repeatability and sample polishing consistency.

Each data point shown represents an average of 20 fields of view transversing the entire cross-section of a 1 inch sample. The samples examined represent a hardness range of HR15Y 73.9.

Of the three constituents present in the coating, porosity had the strongest correlation to hardness. Reduced porosity showed an increase in coating hardness as seen in Figure 11. The maximum range of porosity content measured for the 9 coatings was 11.1 %.

The nickel phase has the second strongest influence on coating hardness. Figure 12 shows the maximum range area content for nickel to be only 7.3%.

Graphite area % to hardness is depicted in the in Figure 13. The maximum range of graphite area content is only 4% between the hardest and softest coatings. The IA correlation of graphite to hardness is not so strong in this analysis, but it can be concluded that area % and weight % ranges for graphite are relatively low given the wide range in hardness of the coatings. This indicates that the extreme changes in gas flow used in the DoE did not have a significant impact on carbon content.

6. MultiCoat and 3GF

The two gas management systems were compared in regard to spraying Durabrade 2223. While the MultiCoat employs mass flow control and 3GF employs a variable area - tube and float principle, the operator inputs the desired process gas flow for both systems as gas volume per unit of time. For the 3GF flow meter to be read in direct calibration, input pressures into the flow meter must be kept at 35 psig for oxygen and 15 psig for acetylene. The 3GF may be operated at other gas pressures but a flow conversion chart is required to factor the pressure variation as it relates to volume flow.

Oxygen (l/min)	Acetylene (l/min)	HR15Y	Carbon in Coating (wt. %)	Nickel in Coating (wt.%)	Oxygen in Coating (wt.%)
24	25	-6.0	19.65	80.35	0.038
24	21	-28.0	19.35	80.50	0.056
38	21	44.5	17.65	82.22	0.090
31	21	20.7	18.90	81.29	0.076
31	25	29.9	19.20	81.20	0.050
31	29	25.3	19.30	80.98	0.045
38	29	43.7	18.20	81.66	0.050
38	25	45.9	18.25	81.79	0.058
24	29	-10.0	20.00	79.62	0.032

Table 2: Coating Chemical Analysis and Coating Hardness for Multiple Parameters

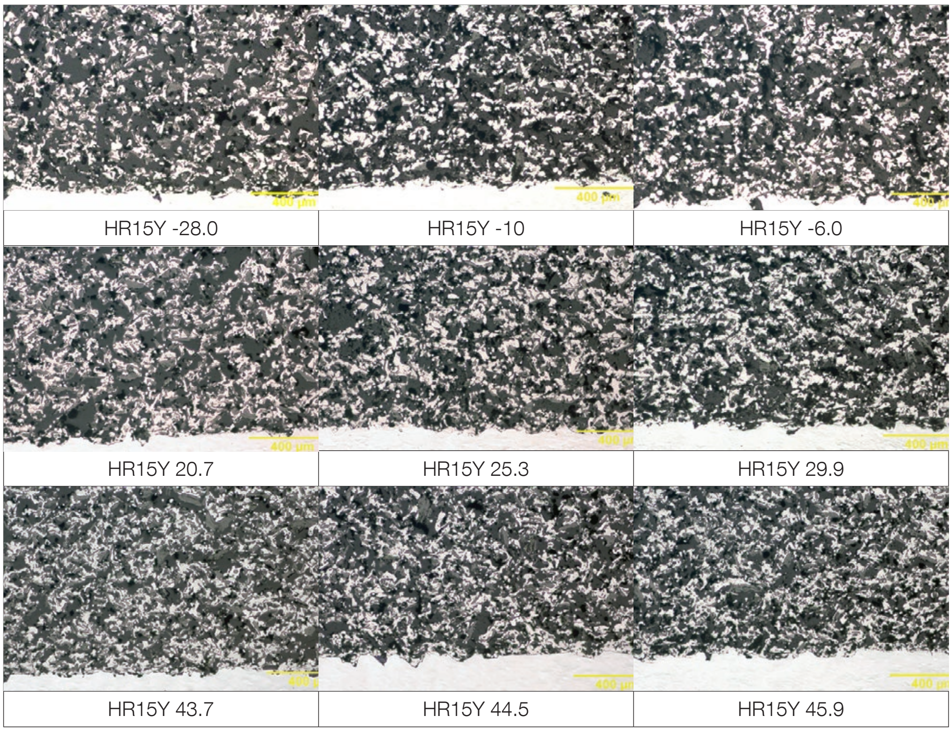


Figure 10: Coating Photomicrographs for Multiple Parameters

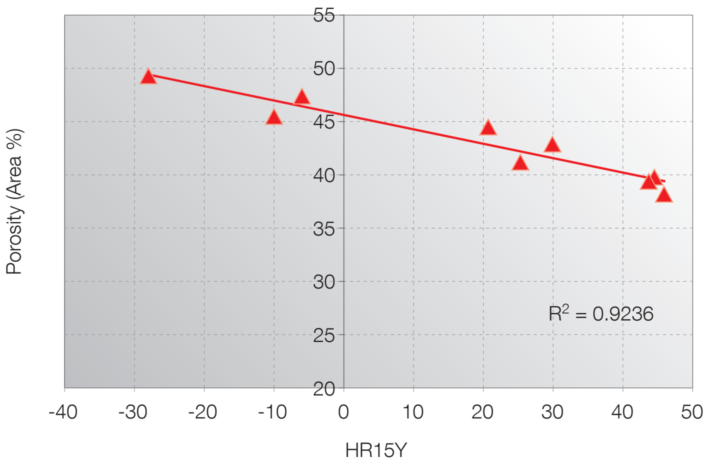


Figure 11: Porosity IA to Coating Hardness DoE D2223
(Each IA porosity data point is the average of 20 fields of view; Standard Deviation between 1.6 – 2.4)

Figure 14 shows the results of a comparison of one lot of powder sprayed ten months apart at two different locations using the MultiCoat and the 3GF systems. The comparison of the two systems is quite reasonable with some variation evident at the low gas flows. The variation can be easily managed with slight gas parameter changes.

The comparison indicates the influence of gas parameter changes on coating physical characteristics is nearly the same. Oxygen as expected has a major influence on coating hardness.

7. Parameter Testing

The following section will demonstrate how to adjust coating hardness using various parameter changes. This knowledge may be used as a road map to manage coating properties to achieve coatings that meet specification requirements. The principals demonstrated in the graphs shown represent an example of cause and effect to parameter conditions and should not be considered as always achievable quantitative values. Spray application hardware aging, process configuration, laboratory practices and powder lot variables will all contribute to results variation.

3GF flow meters were used with the hardware defined in Table 1 unless otherwise noted. Powder feed utilized nitrogen carrier gas at a flow rate of 5 l/min (10.6 SCFH) for all spray work. The standoff distance was 195 mm (7.68 in) unless otherwise noted.

Figure 15 shows a baseline gas parameter set that offers the user the capability to adjust coating hardness with great latitude. The parameter uses oxygen as the principal driver to increase or decrease hardness. The deposit efficiency variation is minimal considering the range in hardness demonstrated.

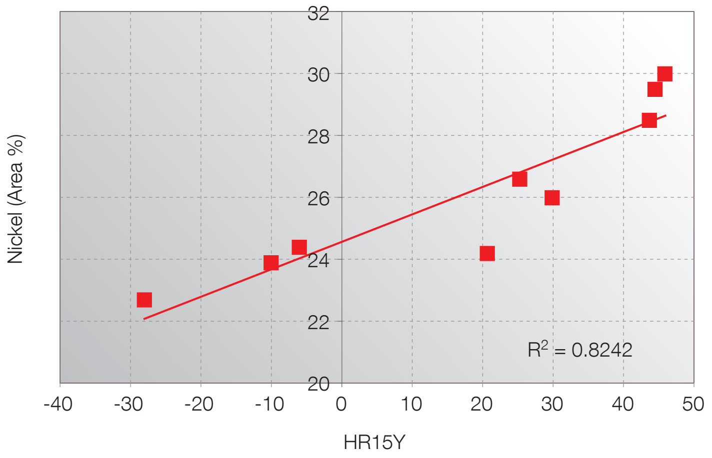


Figure 12: Nickel IA to Coating Hardness DoE D2223
(Each IA porosity data point is the average of 20 fields of view; Standard Deviation between 1.0 – 1.4)

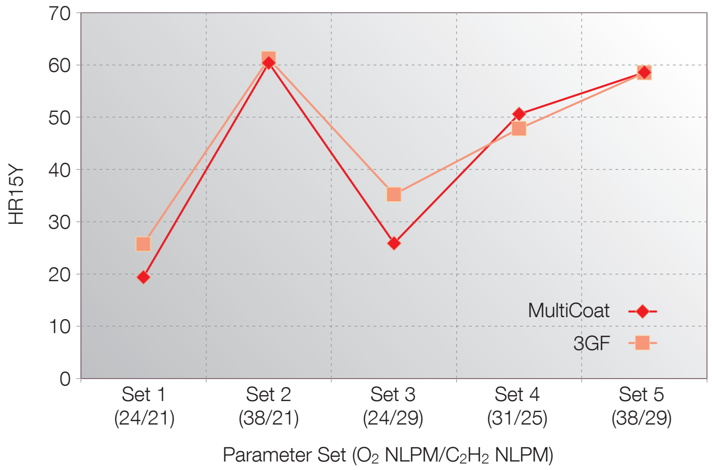


Figure 14: MultiCoat and 3GF Spray Comparison
(Same Powder Lot; Different Spray Sites)

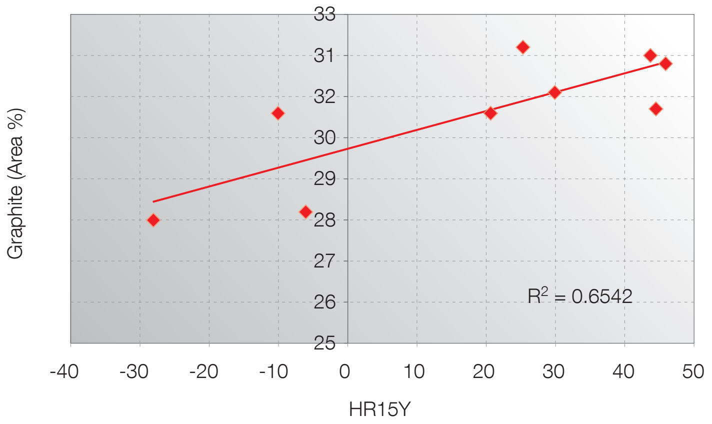


Figure 13: Graphite IA to Coating Hardness DoE D2223
(Each IA porosity data point is the average of 20 fields of view; Standard Deviation between 0.9 – 1.4)

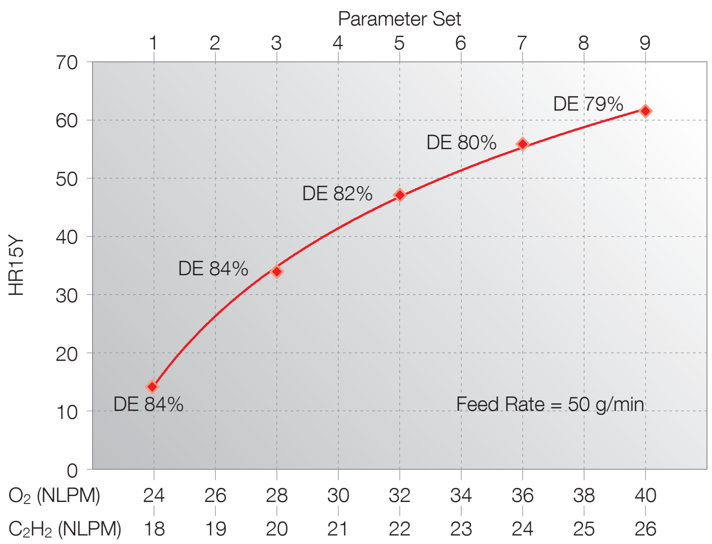


Figure 15: Gas Parameter Guide - Hardness to 3GF Parameter Set

Air caps can play a principal roll in spray coating properties. Oerlikon Metco has several types of air caps for 6P-II applications. Figure 16 demonstrates that replacing the 6P4 air cap with the 6P3 air cap with the (C) cool facing the target, the hardness can be increased considerably. The spot size of the powder striking the target is slightly reduced using the 6P3 as the flame is more condensed and focused. Changing of air caps has only a minor impact on DE in these experiments. The effectiveness of the air caps in altering hardness is diminished with higher hardness.

Another common tool used to effect coating hardness is standoff distance. The principals behind standoff distance having an influence on coating properties is based on particle dwell time and particle velocity. Figure 17 provides a demonstration of how standoff distance affects coating hardness using spray distances commonly in practice for Durabrade 2223 and Metco 307NS-2 with the 7-CD nozzle. The influence of standoff distance is diminished with higher hardness coatings.

Maintenance of the substrate temperature below 177 °C (350 °F) is critical during the spraying of nickel-graphite coatings. When the temperature exceeds recommended limits coating cohesive strength is reduced while measured hardness values may appear normal or slightly elevated. Bond fractures appear linear rather than a normal topographical fracture, which tends to follow concentrations of porosity and graphite. Do not allow the flame to dwell on any part of the coating or substrate.

There are three key elements to accurate coating hardness measurement: Sufficient and consistent coating thickness, consistent surface preparation and a statistically valid number of hardness impressions based on the confidence required.

9. Conclusion

Oerlikon Metco nickel-graphite continues to be successfully applied for aerospace and IGT applications. Material reliability and process versatility give the product a long history of success. Durabrade 2223 and Metco 307NS-2 can be sprayed with Oerlikon Metco application hardware to meet engineered design coating criteria using either mass flow control or volumetric flow meters. By changing parameters, it has been demonstrated that coatings can be altered with great latitude while maintaining efficiency.

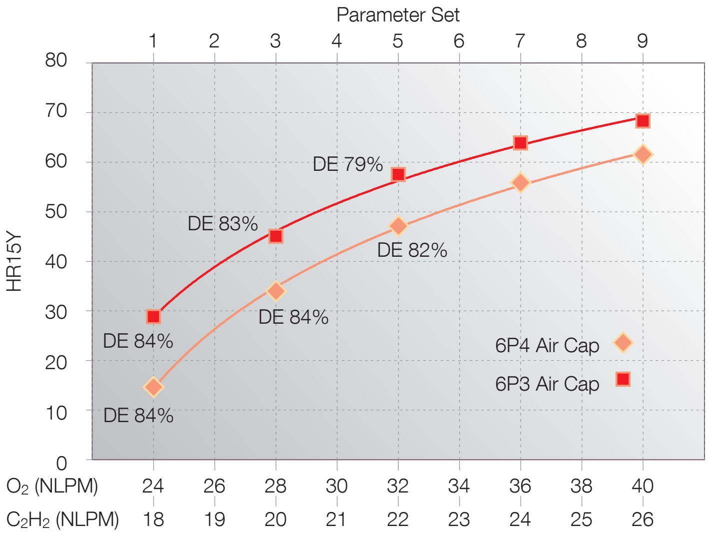


Figure 16: Effect of Air Cap on Coating Hardness

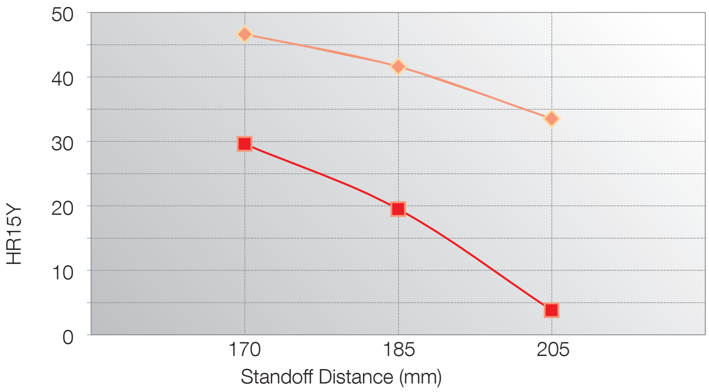


Figure 17: Effect of Standoff Distance on Coating Hardness (2 Parameter Sets)

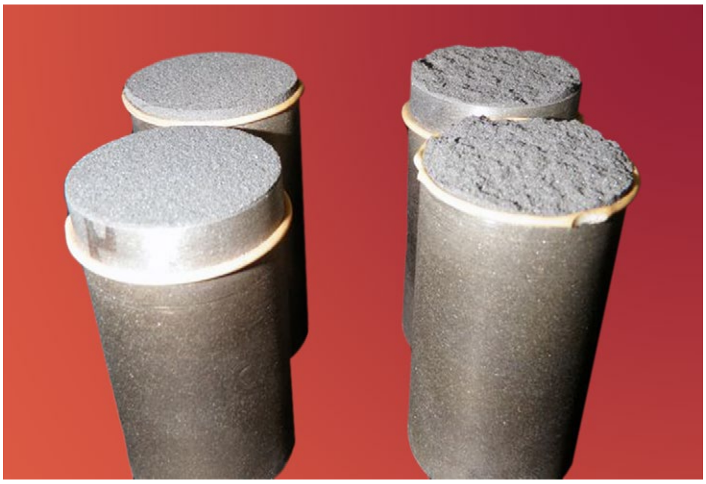


Figure 18: Left: linear coating fracture resulting from overheating during spray processing. Right: A properly temperature controlled coating results a in a topographical fracture.

About Oerlikon Metco

Oerlikon Metco enhances surfaces that bring benefits to customers through a uniquely broad range of surface technologies, equipment, materials, services, specialized machining services and components. The surface technologies such as Thermal Spray, Thin Film, Plasma Heat Treatment and Laser Cladding improve the performance and increase efficiency and reliability. Oerlikon Metco serves industries such as aviation, power generation, automotive, oil & gas, industrial and other specialized markets and operates a dynamically growing network of more than 50 sites in EMEA, Americas and Asia Pacific. Oerlikon Metco, together with Oerlikon Balzers, belongs to the Surface Solutions Segment of the Switzerland-based Oerlikon Group.

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www.oerlikon.com/metco
info.metco@oerlikon.com