NRC Publications Archive Archives des publications du CNRC

HVOF coating case study for power plant process control ball valve application

Vernhes, Luc; Lee, David A.; Poirier, Dominique; Sapieha, Jolanta; Li, Duanjie

This publication could be one of several versions: author's original, accepted manuscript or the publisher's version. /
La version de cette publication peut être l'une des suivantes : la version prépublication de l'auteur, la version acceptée du manuscrit ou la version de l'éditeur.

Publisher's version / Version de l'éditeur:

Thermal Spray 2012 : Proceedings from the International Spray Conference, pp. 172-177, 2012-05-21

NRC Publications Record / Notice d'Archives des publications de CNRC:

<https://nrc-publications.canada.ca/eng/view/object/?id=70b6125e-0493-4d00-a14d-800e068ca8df>
<https://publications-cnrc.canada.ca/fra/voir/objet/?id=70b6125e-0493-4d00-a14d-800e068ca8df>

Access and use of this website and the material on it are subject to the Terms and Conditions set forth at

<https://nrc-publications.canada.ca/eng/copyright>

READ THESE TERMS AND CONDITIONS CAREFULLY BEFORE USING THIS WEBSITE.

L'accès à ce site Web et l'utilisation de son contenu sont assujettis aux conditions présentées dans le site

<https://publications-cnrc.canada.ca/fra/droits>

LISEZ CES CONDITIONS ATTENTIVEMENT AVANT D'UTILISER CE SITE WEB.

Questions? Contact the NRC Publications Archive team at

PublicationsArchive-ArchivesPublications@nrc-cnrc.gc.ca. If you wish to email the authors directly, please see the first page of the publication for their contact information.

Vous avez des questions? Nous pouvons vous aider. Pour communiquer directement avec un auteur, consultez la première page de la revue dans laquelle son article a été publié afin de trouver ses coordonnées. Si vous n'arrivez pas à les repérer, communiquez avec nous à PublicationsArchive-ArchivesPublications@nrc-cnrc.gc.ca.

National Research
Council Canada

Conseil national de
recherches Canada

Canada

310b753908
HVOF Coating Case Study for Power Plant
Process Control Ball Valve Application

Luc Vernhes*

Velan, Montreal, Quebec, Canada

*E-mail: luc.vernhes@velan.com

David A. Lee

Deloro Stellite Co. Inc., Goshen, Indiana, USA

Dominique Poirier

National Research Council of Canada (NRC), Boucherville, Quebec, Canada

Jolanta Sapieha, Duanjie Li

Department of Engineering Physics, Ecole Polytechnique, Montreal, Quebec, H3C 3A7, Canada

Abstract

This case study is the result of an investigation on HVOF Cr_3C_2 -NiCr coating failure of on-off metal seated ball valve (MSBV) used in supercritical steam line in a power plant and solution. HVOF Cr_3C_2 -NiCr coating is used to protect thousands of MSBVs without incident; however in this case the valves are challenged with exposure to rapid high pressure and temperature resulting in a unique situation where the coating experiences cracking and cohesive failure. A detailed investigation was undertaken by Velan, Deloro Stellite, the National Research Council of Canada and the Ecole Polytechnique to determine possible cause and develop solutions in which highlights are presented.

Introduction

A major US-based power company had new challenges with their supercritical steam drain and vent lines in one of their plants. MSBVs are being used on drain and vent lines to extract large quantity of condensate during plant start-up in order to get dry superheated steam rapidly. During normal operation, MSBVs must remain steam-tight to prevent energy loss and maximize plant efficiency. This particular fossil-fuel power station runs continuously from May to October due to high power demand to run air-conditioning systems. For the balance of the year, the plant only runs when the demand called for more power during peak usage times. Consequently the operation of the plant changed from base-load to peaking. Every time the plant shuts down or start-up, MSBVs are frequently cycled and exposed to supercritical steam, going up to 15 MPa (2,200 psi) and 675 °C (1,250 °F) ^[1].

MSBVs are made of a floating ball and a fixed seat, manufactured from forged Inconel® 718 PH and coated with a 200 µm (.008") thick HVOF 80% Cr_3C_2 + 20% NiCr coating. Coating is applied with a Diamond Jet® HVOF system using

propylene as fuel. The primary function of the HVOF coating is to increase the load carrying capacity of the Inconel 718 ball and seat by increasing galling threshold resistance (resistance to micro-welding) and by reducing wear rate.

HVOF Cr_3C_2 -NiCr coating has failed after 1 year and less than 500 mechanical cycles in service on four balls exposed to supercritical steam, with deterioration extending to regions where there is no friction between ball and seats. Visual examination of damage components reveals minor frictional wear and typical stress/fatigue pattern (*Figure 1*).

Figure 1: HVOF Cr_3C_2 -NiCr coating exhibiting cohesive failure after 1 year in service.

The intent of this failure analysis is to provide valuable information to understand failure mechanism and to help eventually preventing this type of failure.

Failure Analysis

Experiment

The failure analysis was conducted by elimination. A list of potential failure mechanisms was established based on literature review and experience:

- 1) Coating quality :
 - High porosity level, allowing high pressure steam penetration
 - Lack of bonding between coating and substrate,
 - Lack of bonding between splats,
 - Embedded grit,
 - Residual stress (increase with thickness).
- 2) Design :
 - Mechanical stresses during operation (excessive load),
 - Thermal stresses due to thermal cycles or thermal shock.
- 3) Metallurgical changes :
 - Corrosion,
 - Reaction at interface,
 - Oxidation,
 - Carbide precipitation.

We then went on a series of experiments in order to find out the most dominant failure mechanism.

Coating quality: The first step was to compare a set of failed ball/seat with a set of new ball/seat through SEM observation. Coating thicknesses, porosity and oxide levels were measured as well as micro and nano-hardness. Coating microstructures were observed using field emission SEM (S4700, Hitachi Instruments Inc., Tokyo, Japan) while porosity levels were evaluated from image analysis on ten images obtained with the backscattered mode of an SEM equipment (JSM-6100, JEOL, Tokyo, Japan). Coating hardness values were measured using a Vickers microhardness tester (Micromet II, Buehler, Lake Bluff, IL, USA) operating under a 300 gf load. Nanohardness of coatings were evaluated using the Nano G200 from MTS/Nano Instruments (Oak Ridge, USA) with a Berkovitch tip under a load of 0.5 gf. The average micro and nano hardness values were obtained from ten and twenty indentations, respectively, performed on the cross section of the coatings. Results were then compared to the OEM purchasing specification (See Table 1)

Table 1: Comparison of failed versus new HVOF 80% Cr_3C_2 + 20% [80Ni-20Cr] coating and OEM specification.

	Failed component	New component	OEM specification
Thickness (μm)	215	155 ± 35	200
Porosity	2%	2%	<3%
Oxide	<2%	<2%	N/A
Bond (MPa)	No failure	>70	>70
Microhardness (Vickers tip, 300 gf)	1092 ± 44	897 ± 69	>700
Nanoindentation (Berkovitch tip, 0.5 gf)	18 ± 7 GPa	15 ± 5 GPa	N/A

Figure 2: HVOF 80% Cr_3C_2 + 20% [80Ni-20Cr] coating, sprayed with C_3H_6 after 1 year in the field

The interface between coating/substrate was qualitatively inspected. Residual grit was regularly observed at the bond. Cracks formed in the coatings did not appear to originate at the coating/substrate interface nor due to excess porosity or grit. SEM observations unveil that cracks were propagating across the NiCr binder. SEM also shows the presence of oxides at the coating surface, at the surface of cracks, and as veins propagating in the coating (appears in dark gray on backscattered electron images – See Figure 2).

Figure 3: HVOF 80% Cr_3C_2 + 20% [80Ni-20Cr] coating, sprayed with C_3H_6 after 1 year in the field

However, it's difficult to determine if cracks propagate along oxides or if the crack surfaces oxidize afterward. Cracks form and propagate across the coating matrix or at the carbide/matrix interface, delaminating layer by layer the coating (See Figure 3).

Design: The second step was to perform two validation tests to confirm that design and materials were able to sustain mechanical and thermal stresses

For the mechanical stress resistance, one prototype valve was assembled with brand new coated components and connected to a 100 KW electrical boiler supplying superheated steam at 426°C (800 °F) and 12.5 MPa (1,821 psig), the maximum in-house boiler capacity. The prototype valve was mechanically cycled using an automated pneumatic actuator, while monitoring seat leakage (in closed position). Valve was cycled every 2 min (open-to-close and close-to-open) under the steam atmosphere. The prototype valve was able to perform over 2,000 mechanical cycles with 0 seat leakages. After dismantlement and visual inspection, no catastrophic failure was observed.

For the thermal stress resistance, new coated balls were brought to 675°C (1247°F) during 30 min using a conventional electrical furnace. Balls were then quenched into a bucket of room temperature water (20°C – 68°F). After 30 consecutive similar thermal shocks going from 675°C to ambient temperatures, HVOF Cr₃C₂-NiCr exhibits a crack network relatively similar to the one observed on the failed valves (See Figure 4).

Figure 4: HVOF 80% Cr₃C₂ + 20% [80Ni-20Cr] coating, sprayed with C₃H₆, after 30 thermal shocks going from ambient temperatures to 675°C.

Figure 5: XRD spectra of HVOF 80% Cr₃C₂ + 20% [80Ni-20Cr] powder and coating, sprayed with C₃H₆.

Metallurgical change: One of the observations of the failed coating was the significant increase in microhardness values from nominal 900 HV (.3 kg) to well over 1100 (.3 kg) up to 1400 HV (.3 kg). This lead to evaluation of the phase changes that were thought responsible for the heat treatment affect of the coating in service [2].

In this third step, XRD measurements were performed on powder and “as sprayed” coating in order to estimated the change in crystalline and other phase composition, amorphous content and to determine the relative Crystallinity index (Ic) [3]. Ic provides relative proportions of crystalline and amorphous material. XRD measurements were performed between 30° deg and 55° deg range because peaks and amorphous hump are found within those values for Cr₃C₂ and WC based coatings. Crystallinity index is estimated with the following formula:

$$I_c = \frac{\sum \text{Peak Areas}}{\sum \text{Peak Areas} + \text{Amorphous Hump Area}}$$

Measurements were performed using a D8 Advance Bruker AXS diffractometer (CuKα, 40mA and 40 kV). Spectrum areas were measured with Eva software. Table 2 summarizes results and Figure 5 shows both spectrums.

Table 2: Crystallinity Indices of powder and “as sprayed” coating of the original HVOF coating

Chemical composition		Crystallinity index	
Powder composition	Fuel	Powder	Coating
80%Cr ₃ C ₂ -20%[80Ni20Cr]	C ₃ H ₆	90%	56%

The crystallinity index reduces by more than 35% after coating deposition, indicating significant crystalline phase dissolution during spraying.

Discussion

The main difference observed between failed versus brand new “as sprayed” coating is a 25% hardness increase, leading to measurements well over 1,200 Hv. It was proposed that either carbide precipitation and/or oxidation could theoretically increase the hardness of the coatings. Therefore, the following 2 steps failure mechanism is suspected:

1) Carbon dissolution into 80% Ni - 20% Cr matrix during spraying :

During spraying, carbon is dissolved into the matrix. Carbon could come from two sources: I- chrome carbides are getting partially dissolved, creating a range of Cr composition in matrix from Carbon-rich to the original Ni alloy (The higher the spraying temperature, the more dissolution occurring); II- carbon comes also from the carbon-rich propylene fuel. The high cooling rate “freezes” the carbon-rich matrix in a supersaturated state, with amorphous or nanocrystalline zones – a metastable system that can change if heated ^[4].

2.a) Chrome carbide precipitation:

In high temperature service, matrix recrystallisation can occur. Fine Cr₂C₃ could precipitate in high dissolution NiCr regions leading to hardness increase. Hardness increase could be observed with values exceeding the coating initial hardness due to carbide precipitation and to a “sintering” effect (decrease in porosity and improved adhesion between splats). The hardness increase could keep increasing due to the formation of a fine carbide network ^[2].

2.b) Chrome Oxidation:

When sufficient Cr₃C₂ dissolution occurs during spraying to get at least 27.5% Cr in the matrix (either due to spraying conditions or the type of powder used), only Cr₂O₃ is formed in a continuous scale ^[5]. Cr₃O₂ can be adherent or eventually buckled if carbides are large. If there is not enough Cr in solution in the matrix, a NiO scale forms below which a continuous Cr₂O₃ scale develops. NiO can further be consumed to form NiCr₂O₄ ^[6].

Water vapor containing atmospheres cause more rapid oxidation of chromia-forming alloys than do dry oxygen or air, according to the equation shown below ^[7]:

In summary, two reactions involving Cr are competing: Cr can form carbides or can oxidize. At the temperatures of interest in this study, i.e. 675°C or below, Cr and C diffusion is slow and it is most probable that the Cr in contact with the oxidizing atmosphere will preferentially form oxides ^[5]. However, the Cr present deeper in the coating and not in contact with O₂ would form carbides.

In this specific case, oxides region displaying low hardness have been observed, possibly due to presence of pores and

cracks, and perhaps the formation of non-stable oxides. Furthermore, no significant trend in hardness from coating top to bottom, where a gradient in oxide concentration is expected, was measured. Cr₂O₃ hardness being around 1,200 Hv and Cr₃C₂ hardness around 2,000 Hv, the presence of Cr₃C₂ in the matrix seems reasonable.

No fine chrome carbides have been detected under SEM into the NiCr binder most probably because those fine carbides would be too small to be seen.

Solution

After identifying the most dominant failure mechanism, it was time to test and qualify potential coating solutions, better suited to carry high load at elevated temperature and to withstand thermal shock.

List of potential solutions

Based on our analysis, the dominant failure mechanism is coating embrittlement due to chrome carbide precipitation in-service across coating binder. 3 potential coatings were selected to overcome this issue. On the top of it, to minimize carbon dissolution during spraying, hydrogen was used instead of propylene (carbon-free fuel) and Jet-Kote® HVOF system was used instead of Diamond Jet® (lower in-flight temperature). Table 3 lists the selected potential solutions.

Table 3: List of potential solutions

#	Chemical composition		Process	Remark
	Powder	Fuel		
0	80% Cr ₃ C ₂ 20%[80Ni20Cr]	C ₃ H ₆	HVOF	Original (Failed)
1	75% Cr ₃ C ₂ 25%[80Ni20Cr]	H ₂		25% matrix to increase ductility
2	65-70% WC 22-25% Cr ₃ C ₂ 7% [Ni/NiCr]	H ₂		Almost chrome-free binder to minimize Cr ₃ C ₂ and Cr ₃ O ₂ formation
3	55% Ni, 17% W, 15% Cr, 4% Si 3.5% Fe 3% B <1% C	H ₂	S&F	55% Ni to provide ductility. Fusing at 1,000 °C to sinter, stress relieve and precipitate carbon.

Solution qualification

Brand new samples were produced with those three new coatings. A battery of tests was performed to assess their behaviors for this specific application.

Design: Mechanical resistance of the three coatings was evaluated using the cycle test previously described. Coating #1 and #3 exhibited similar mechanical resistance than the original HVOF coating. Coating #2 demonstrated an exceptional mechanical resistance by performing 5 times more mechanical cycles than the original coating without any leakage.

Thermal stress resistance was evaluated using the thermal shock test previously described. Coating #1 and #3 passed the test with flying color (No crack indication after 60 thermal cycles – See *Figure 6*). Coating #2 exhibited chipping and delamination after exposure to 675°C during 30 min (Most probably due to high tungsten content ^[8]).

Metallurgical change: The third test was to perform XRD measurements on powder and “as sprayed” for the HVOF coatings in order to estimate crystalline phases dissolution during deposition. The fusing process allowing stress relieving and carbon precipitation, the Ic of coating #3 was not evaluated. The following *Table 4* summarizes results.

Table 4: Crystallinity Indices of powder and “as sprayed” HVOF coating

Coating ID	Crystallinity index	
	Powder	Coating
0	90 %	56 %
1	99 %	55 %
2	95 %	80 %

While coating #1 displays similar degradation level than the original coating, coating #2 crystallinity index remains much higher, indicating less amorphisation (See *Figure 7*). However, for coating #2, a partial phase transformation ($WC \rightarrow W_2C$) was observed on the XRD spectra after deposition. Therefore, amorphisation is not the only degradation mechanism for this composition material.

Figure 7: XRD spectrums of HVOF material #1 and #2; powder and “as sprayed” coating.

Coating Selection

Based on those data, coating #1 and #3 seem well suited to withstand high bearing load and thermal shock. However, the low Ic of the “as sprayed” coating #1 indicates a high content of metastable phase, which could evolve in-service. The coating #3 seems the less sensitive to aging at high temperatures and therefore more durable under these very specific conditions.

Coating #2 is not suitable for temperature over 540 °C (1,000 °F) however it has demonstrated the best overhaul performance at lower temperature.

Figure 6: Coating #1 and #3 were repeatedly subjected to thermal shocks going from ambient temperatures to 675 °C. Then they were put through liquid penetrant tests to check for any cracking: they passed with flying colors.

Conclusion

In this study, the failure of a HVOF $\text{Cr}_3\text{C}_2\text{-NiCr}$ coating applied on an on-off metal seated ball valve (MSBV) used in supercritical steam line was investigated. While oxidation definitely contributes to coating degradation, it is believed that carbide precipitation is the major factor causing embrittlement of the coating. Once the coating toughness and ductility is reduced, thermal, mechanical and residual stresses can initiate and propagate cracks more easily, causing coating failure.

Once the cause was known, it was possible to qualify a spray-and-fuse NiWCrBSi coating more suitable to support high bearing load when facing thermal shock. Coating is less sensitive to aging at high temperatures and therefore more durable under these very specific conditions.

An enhanced HVOF coating, produced from a mixed $(\text{W,Cr})\text{C}$ and WC carbides in a nickel matrix, also emerged of those testings. While it showed similar ductility compared to the original HVOF $\text{Cr}_3\text{C}_2\text{-NiCr}$ coating, it consistently provides longer in-service life on service under 540°C ($1,000^\circ\text{F}$).

Armed with these new coating technologies, new valves were installed in the plant: They've worked flawlessly for the past year. As a result, Velan's MSBVs line for the power industry now features two coatings: One designed for regular service – HVOF $(\text{W,Cr})\text{C-Ni}$ and one specially designed for applications involving severe thermal shocks – S&F NiWCrBSi .

Acknowledgements

The authors wish to thank Mr. Subhash Saini (Velan), Michel Thibodeau (NRC) and Jimmy Sykes (NRC) for their technical support as well as Dr. Rogerio Lima (NRC) and Professor Ludvik Martinu (Ecole Polytechnique) for useful discussion. Finally, we greatly appreciate Velan's interest and support of this research.

References

1. D. Xu, J. Bao, and Y. Jin, Mechanical jamming analysis of the reheat-stop-valve mechanism in the supercritical steam turbine, *Jixie Gongcheng Xuebao/Journal of Mechanical Engineering*, 2010, **46**(1), p. 121-127
2. S. Matthews, M. Hyland, and B. James, Microhardness variation in relation to carbide development in heat treated $\text{Cr}_3\text{C}_2\text{-NiCr}$ thermal spray coatings, *Acta Mater.*, 2003, **51**(14), p. 4267-4277
3. C. Verdon, A. Karimi, and J.L. Martin, A study of high velocity oxy-fuel thermally sprayed tungsten carbide based coatings. Part 1. Microstructures, *Mater. Sci. Eng. A, Struct. Mater., Prop. Microstruct. Process.*, 1998, **A246**(1-2), p. 11-24
4. S. Matthews, M. Hyland, and B. James, Long-term carbide development in high-velocity oxygen fuel/high-velocity air fuel $\text{Cr}_3\text{C}_2\text{-NiCr}$ coatings heat treated at 900°C , *J. Therm. Spray Technol.*, 2004, **13**(4), p. 526-536
5. S. Matthews, B. James, and M. Hyland, The role of microstructure in the high temperature oxidation mechanism of $\text{Cr}_3\text{C}_2\text{-NiCr}$ composite coatings, *Corros. Sci.*, 2009, **51**(5), p. 1172-1180
6. S. Matthews, B. James, and M. Hyland, Erosion of oxide scales formed on $\text{Cr}_3\text{C}_2\text{-NiCr}$ thermal spray coatings, *Corros. Sci.*, 2008, **50**(11), p. 3087-3094
7. N.K. Othman, J. Zhang, and D.J. Young, Effect of water vapour on cyclic oxidation of Fe-Cr alloys, *Materials and Corrosion*, 2011, **62**(6), p. 496-503
8. L.M. Berger, S. Saaro, T. Naumann, M. Kasparova, and F. Zahalka, Microstructure and Properties of HVOF-Sprayed $\text{WC-(W,Cr)}_2\text{C-Ni}$ Coatings, *J. Therm. Spray Technol.*, 2008, **17**(3), p. 395-403