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Quality characterization of HVOF thermal spray coating with NiCr matrix composite for protection application of coal fired **boiler tubes**

M Waldi¹, E A Basuki¹, B Prawara²

¹Metallurgical Engineering Department, Faculty of Mining and Petroleum Engineering – Bandung Institute of Technology, Ganesha 10 St., Bandung 40132, Indonesia

² Research Center for Electrical Power and Mechatronic – Indonesian Institute of Sciences (LIPI), LIPI Area, Cisitu 21/154D St., Bandung 40132, Indonesia

waldi05@gmail.com

Abstract. In line with the Indonesian government's policy on national electrification plans by developing a 35,000 MW power generating facility which has been started since 2014, the need arises for high temperature resistant materials for application in the program. Moreover, materials dedicated to engineering equipment such as boilers as a steam producer to drive the coal-fired power plant turbine is required to be compatible in extreme condition. The operational challenge of materials at high temperatures becomes serious, especially as the use of low quality coal as fuel in the combustion system creates degradation problems on tube surfaces such as erosion and corrosion thereby limiting boiler tube life. Thermal spray coating technologies for high temperature operations have been developed with the aim of protecting boiler tubes from aggressive environments, improving thermal efficiency, and reducing time losses from damage. High velocity oxy fuel (HVOF) technology is one of the recommended methods to be used in boiler tubes at coal-fired power plant (PLTU). The following report discusses the HVOF thermal spray coating deposition of metal-matrix composites with combination of feedstock NiCr as metal matrix which has -37µm of size particles with ceramic metal including chromium carbide and alumina as reinforcement on SUS 304 substrate.

1. Introduction

The economic growth rate of Indonesia and the rapid development of multipurpose technology to facilitate the needs of household and industry in the last decade has led to the increasing global demand for electricity from time to time. Based on the 2014 Indonesian Energy Outlook [1] exposition, Indonesia was predicted to experience a population increase from 255 million in 2015 to 335 million people by 2050. Furthermore in 2016, the electrification ratio was 91.35% [2]. Although the trend of installed capacity has been an increase of 4% per year since 2011, it has not been able to attain the ideal target of electrification ratio of 100%. Nevertheless, the construction of power generation facilities will still be upgraded for at least another decade.

The existing power plant facilities in Indonesia in 2015 have an installed capacity of 55,528.10 MW produced by several types of power plants, namely coal-fired power plants (abbreviated PLTU in Indonesian), gas-fired (PLTG), steam-powered and gas-fired (PLTGU), diesel powered (PLTD),

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hydroelectric (PLTA), geothermal (PLTP), wind-powered (PLT Bayu), solar-powered (PLTS), and coal gasification powered (PLTGB) plants, where the highest share of electricity supply is generated by PLN of 38,314.23 MW and the rest is supplied by private parties or also known as Independent Power Producer (IPP) of 17,213.87 MW [3]. The highest supply is produced by three large types of power plants, namely PLTU, PLTGU, and PLTD, in this particular order, followed by other power plants such as PLTA and PLTG. Thus, to pursue the electrification target of 100%, an additional supply of 35,000 MW is still needed.

With the priority for use of power plants to support electricity supply throughout the region based on feasibility studies [4, 5], the availability of coal, and the consideration of electricity production efficiency, the development of coal-fired power plants is still a solution to accommodate the 100% electrification. The significant constraint encountered in the operation of the coal-fired power plant is the degradation of the components of the boiler tubes at high temperature conditions combined with the destructive extreme environmental conditions, in the form of high temperature corrosion, erosion, wear and oxidation on the surface.

The frequent shutdown of operations due to component turnover decreases the generator's performance. Therefore, the selection criteria for boiler tube materials should be compatible with high operating temperature to support efficiency. In addition to protecting the substrate material against such extreme environments, a compatible metallic layer is required [6].

The application of thermal spray coating (TSC) is the solution to the need for high temperature resistant material that is also to corrosive, oxidative and erosive environments, as well as to creep resistance. TSC provide important properties such as: optimal thickness of up to 500µm, increased component life, good adhesive between coatings and substrates, relatively low production prices, thermal shock resistant, resistance and to corrosion and erosion. In conclusion, TSC acts as a structural jacket against combustion load [7].

Thermal spraying is generally defined as a method of forming metallic or non-metallic coatings resulting from melted or semi-melted conditions caused by energy hereinafter using high velocity oxy fuel (HVOF). The energy is used to melt feedstock materials (derived from powder form) fed through a high speed spray gun and propelled towards the surface of the metal substrate by utilizing gas or atomization jets to form layers with lamellar structures. Referring to the term coating material, the material may be metal, oxide, or carbide. The thermal spray event occurs due to a combination of thermal energy, and the melt and semi-melt kinetic energy of the feedstock from the heat that formed by the interaction of fuel and oxygen, and by an electric source.

One of the most significant issues in boiler operation is erosion damage, the depletion of material due to the impingement of high velocity solid particles and impact on the material surface at the certain angles [8]. The coal used in Indonesia power stations has large amounts of ash (about 10-50%) which contain abrasive mineral species such as hard quartz (up to 15%) which increase the damage.

HVOF sprayed NiCr-Cr₃C₂ coatings are one of the most important candidates for protection of materials from high temperature erosion and have been successfully used to protect pulverized coal fired boiler tubes [9-11]. Stainless steel is used as substrate in the present research. It has higher strength at elevated temperatures so is often used for structural and pressure-containing applications at temperatures above about 500 °C and up to about 800 °C [12]. Austenitic stainless steel contains 16% of chromium and 6% nickel. NiCr powder is selected as bond coat and metal matrix for top coat deposition, chromium carbide and combination of chromium carbide and alumina are used as reinforcement particles in metal matrix composite (MMC). NiCr selection is based on its properties that could withstand in the elevated temperature condition, and hot corrosion issue. Further Martides *et al.* in 2017 has reported their works about the influence of finer size of NiCr matrix that gave improvement to coating properties [13].

Chromium Carbide (Cr_3C_2) was selected as a reliable protective coating to MMC materials that capable to resist oxidation in range 650 – 1200 °C [8]. This material has a good wear resistance which is chemically inert and stable at elevated temperatures. In addition, high purity alumina (white

alumina) has good electrical insulation and thermal conductivity, and applied in many applications that require resistance to abrasive and erosive loads.

The present work is aimed to develop a NiCr metal matrix composite (MMC) coating using HVOF. NiCr powder, and Cr_3C_2 with some addition of Al_2O_3 were used as metal matrix and reinforcement particles, respectively. Another purpose of this work is to give valuable information to established

2. Materials and methods

2.1. Materials

The substrate austenitic stainless steel SUS 304 is obtained from the inventory of the Research Centre for Electrical Power and Mechatronics, LIPI, Bandung - Indonesia. The material has been established to have the best performance for the working conditions of the ultra-supercritical pulverized coal (USC) power plant [14, 15]. Table 1 shows the verification result of hardness using Metkon Duroline M Microhardness Tester and chemical composition using Metavision 1008i spectrometer to check asreceived material meet the specification defined.

NiCr powder was obtained from Oerlikon Metco (Metco 43C-NS) and employed as bond coat material, and the selection process through sieve shaking process was performed to obtained finer powder distribution size to 400 mesh (-37 μ m) for metal matrix of top coat deposition. Reinforcement particles such as Cr₃C₂ were obtained from Oerlikon Metco (Woka 7102), and Al₂O₃ was obtained from Sulzer Metco (Metco 105NS).

Coating feedstock was made into two blending formula with each total weight 1 kg as follows: (1) $60\%\text{NiCr}_{[400\text{mesh}]} + 30\%\text{Cr}_3\text{C}_2 + 10\%\text{Al}_2\text{O}_3$, and (2) $60\%\text{NiCr}_{[400\text{mesh}]} + 40\%\text{Cr}_3\text{C}_2$. Hereinafter, formula 1 called as 3 components and formula 2 called as 2 components. After all material were prepared and weighed according to the formula, the material were blended respectively in V-Type blending machine within 64 rpm for as long 16 hrs. The appearance of blending result is shown in Figure 1 and Figure 2.

Table 1. Verification of tensile strength SUS 304, and its chemical composition analysis by.

Hardness	Elements (%-wt)							
(HVN)	С	Si	Mn	Р	S	Cr	Ni	
209.96	0.0208	0.4087	1.6706	0.0158	0.0055	19.1362	8.8657	



Figure 1. SEM micrographs of NiCr matrix composites powder with Cr_3C_2 and Al_2O_3 reinforcement; (a) feedstock; (b) EDS spectra of MMC 3 components feedstock.

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Figure 2. SEM micrographs of NiCr matrix composites powder with Cr_3C_2 reinforcement; (a) feedstock; (b) EDS spectra of MMC 2 components feedstock.

2.2. HVOF Thermal spraying process

The thermal spraying process is carried out using TECKNOTHERM HVOF 2007 thermal spray machine and spray gun Hipojet-2700 with constant parameters for each MMC formulas, Table 2 describes the HVOF process parameters employed during the. The thermal spraying process is applied on to the SUS 304 plate specimens with size 30 mm x 20 mm X 3mm to be prepared for general characterization including SEM, XRD, hardness, and roughness, and the other is solid cylinder Ø25.4 mm X 38.5mm for bond strength test purpose.

Grit blasting was performed to clean the specimen's surface from contamination and to activate surface condition in order to maximize coating adhesiveness. This latter occurs through the formation of rough textures on the substrate surface to facilitate mechanical interlocking of the splats. Mechanical interlocking plays an important role in the mechanism of cohesion when the splats fill and solidified on the rough surface. The process was employed white aluminium oxide (600 - 850 mesh) and the blasting process was conducted inside the blasting chamber.

Parameter	Unit	Value
Air pressure	bar	6.2
Pressure O ₂	bar	8
N_2	bar	5
Propane	bar	5.5
Flow rate O ₂	liter/min	271
N_2	liter/min	8
Propane	liter/min	62.4
Powder feeder rotation	rpm	5
Stand off distance	mm	200
Spray angle	0	90
Substrate preheating	°C	100

Table 2. Parameter process of HVOF thermal spray.

2.3 Characterization

The morphology of the powders and the coating microstructure were observed by means of a integrated scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS) system (JEOL JSM-IT300LA). Observations were carried out on a polished cross-section normal to the surface. The metallographic preparation was done by coarse to fine grinding followed by polishing

with 1 µm alumina suspension. The phase characterization of the coating deposit after HVOF thermal spray was conducted by X-ray diffraction (XRD) using Rigaku SmartLab X-ray Diffractometer.

The porosity within the coating microstructure was evaluated by analysis software ImageJ Ver. 1.50i (<u>http://imagej.nih.gov/ij</u>), the software identified the dark pores (cavities) by color mapping. The work by Sadeghimeresht *et al.* has shown that image analysis can reproducibly identify and measure pores, cracks, etc. within thermal spray coatings with a high level of confidence [16].

Surface roughness for each coatings were measured by stylus Kosaka Lab AS-1700 α stylus model profilometer equipped with Kosaka Lab SE1700 α recorder.

The microhardness was measured on a polished cross-section surface of the HVOF coating by using a Metkon Duroline M microhardness tester with a load selection 300 gf and dwelling time is 10 seconds in accordance with ASTM E92. Seven data point were acquired across the substrate to coating with three times indentation per each sampling points. Each data were collected and averaged for each hardness value.

Tensile bond strength was performed using Hung Ta tensile tester tipe HT-8503 capacity 2 ton and additional test fixture as recommended by ASTM C633. One untreated piece of cylindrical sample face was grit blasted by white alumina oxide and cleaned with compressed air then attached by adhesive Cytec FM1000 and assembled to another cylindrical sample piece that already coated. Five pairs of the specimens were held in V-BLOCK and remain compressed by threaded holder and tightened to 87 kg.cm, heated in the furnace to 180 °C held for 2 hr and cooled inside the furnace. Applied tensile load to each test specimen at a constant rate of cross-head travel between 0.013 mm/s to 0.021 mm/s until rupture occurs.

3. Results and discussion

Figure 1 reveals the blending of the 3 components feedstock performed on a V-Type blending machine for 16 hours at a speed of 64 rpm in which visible components of NiCr, Cr_3C_2 , and Al_2O_3 have been well distributed. NiCr component shown in Figure 1 appears to be incomplete, this is caused by collisions with reinforcing components during the blending process, in which the Al_2O_3 powder has a harder property than the NiCr matrix. Qualitatively the EDS spectra verifies the constituent elements of the MMC 3 components, in which the spectrum charts identified the existence of Ni, Cr, Al, and C.

Figure 2 in which the NiCr matrix is still relatively intact after blending process. The observation by using SEM shows that NiCr matrix and Cr_3C_2 particles have been well distributed and qualitatively EDS spectra identified the constituent elements in MMC 2 components, such as Ni, Cr, and C. The blending result of MMC 3 components and MMC 2 components found no contamination of foreign objects that could be reduced the quality of feedstock and the validity of TSC process results. The results obtained after the thermal spray coating process showed an evenly distributed layer, the physical difference between TSC results with MMC 3 components is darker than those produced by MMC 2 components (see Figure 3). This is due to the Al_2O_3 factor that gives the darker character to the exterior appearance of the coating. It is also noted from measurement that coating roughness (Ra) of MMC 3 components and MMC 2 components were 6.2284 µm and 7.3872 µm respectively.

The thickness of the coating has been measured through the SEM instrument and desired thickness composition of 50-75 μ m for the bond coat layer and 125-150 μ m for the top coat layer. Measurements were performed at least three times at different locations and average of thickness measurement of bond coat and total thickness for each specimens are: 67 μ m and 262 μ m (MMC 3 components); 72 μ m and 368.4 μ m (MMC 2 components).

Figure 4 reveals SEM micrograph TSC deposition of MMC 3 components with back scattering feature which capable to distinguished splats, un-melted particles, porosity, and oxide. Bond coat formation is evenly distributed on the substrate with varying thickness across the section that caused by the repeated and accumulation of spray process. Every single bond coat that built and solidified on the substrate can be observed as similar as grain boundaries. Un-melted particles identified in MMC 3 components are quite significant and distributed evenly within the coatings. The presence of un-melted

particles is strongly influenced by the parameters during the thermal spray process such as low combustion temperatures and lack of heat input given to the substrate, so it did not have enough time to melt some powder feedstock at the time it propelled out toward the substrate with a high velocity. The non-constant particle rate also greatly affects the melting of the feedstock powder when it travelled from feeder towards the substrate surface, so that the powder was solidified earlier before the substrate strikes the surface. A small part of the oxide also appears to form on the coating structure.



Figure 3. The as-coated result of MMC 3 components and 2 components using TECKNOTHERM HVOF 2007 thermal spray machine and spray gun Hipojet-2700.

Another factor that affects the formation of un-melted particles is the accumulation of spraying conditions to meet the desired thickness targets. The previous coating deposit layer is subject to remelt from the molten coating powder that propelled by the spray gun, but the heat received by the deposit does not melt entirely and solidified faster than the new coating layer. The coatings have a dense structure with the porosity 1.43% randomly distributed in the coating. Cross-sectional micrograph shows the coating microstructure consists of NiCr matrix (bright area) and chromium carbide (dark area).



Figure 4. SEM micrograph of HVOF spray MMC 3 components, constituent element mapping, and point analysis.

SEM micrograph of MMC 2 components coating that shown in Figure 5 reveals the different distribution of *splats*, *un-melted particles*, porosities, and oxides with MMC 3 components coating. It

is noted that the population of un-melted is lower than the MMC 3 components. The molten droplets that were sprayed continuously were generally spherical and become splats when impacting the surface; thus the shape spreads and fills space in a flat shape, rapidly solidified, and then evolved with the final lamellar morphology.



Figure 5. SEM micrograph of HVOF spray MMC 2 components, constituent element mapping, and point analysis.

Splats, signified with lamellar morphology are characteristic of thermal spray coating process. However, only a limited number of un-melted particles can be observed in the microstructures. The coating also possesses some voids and oxide inclusions that are typical characteristics of the HVOF sprayed coating. It is noted that the porosity is 1.29% and is randomly distributed in the coating.

Both in the Figure 4 and Figure 5 have been found that rich of Ni and Cr, and contain small amount of Al within the MMC 3 components coating. It is also shown that Fe has been found to be restricted to the substrate only, which shows that inter-diffusion of the substrate constituents to the coating has not been taken place. This statement is also confirmed by the point analysis performed with the EDS feature to seven points sampling across substrate to coating section.

For both samples, XRD analyses were carried out to identify the different phases present in the coatings. Figure 6 shows the X-ray diffraction pattern of the as coated MMC 3 components, it is seen in the peak that Ni is dominant phase in NiCr based coatings, Cr_3C_2 and Al_2O_3 were also detected and indicated in the microstructure as carbide and oxide. Another phase is NiCr₂O₄ that probably formed due to reaction of NiO and Cr₂O₃ during the thermal spray process and had influence to reduce the coating hardness, see point analysis of hardness in Figure 8.

The X-ray diffraction pattern of the specimen MMC 2 components coating is displayed in Figure 7. A significant peak characteristic identify substantial amount of Ni element. The Cr_7C_3 major intensities of peaks as the decarburization result of Cr_3C_2 in the coating, this one similar to the works that has been reported by Hong, *et al.* [17] and Guilemany, *et al.* [18], even though it was also present in the initial powder. It is understood from XRD characterization that existence of Cr₃C₂ phase provides an increase in hardness of MMC 2 components coating.

Hardness is the significant mechanical property of the coatings. Microhardness distribution data has been acquired to characterize the hardness change along the cross section. The graphic of

microhardness shown in Figure 8 and Figure 9 were formed "S" profile. As shown on both profile the microhardness of point 1 exhibited nearly uniform and the value even quite similar to non-sprayed substrate.

At point 2, nearby substrate and coating interface shows little higher value of microhardness, it was both attributed to the work hardening during grit blasting prior to coating [19], and high velocity of molten droplets [20]. At point 3 from both figures shown the increment trend of microhardness, this was associated with bond coat buildup of NiCr powder on the substrate, then at point 4, the increased hardness value at the interface was attributed to the formation of chromium carbide at this region [19]. As indicated in Figure 8 at point 5 the microhardness falls from 397.8 HVN to 315.95 HVN and stay steady to the top region of the coating. In the contrary, Figure 9 from point 4 to 7 the microhardness distribution was relatively stable, only slightly falling to 481.57 at point 6 and rising up again to 538.24 HVN at point 7.



Figure 6. X-ray diffraction pattern of MMC 3 components coating.



Figure 8. Microhardness profile of MMC 3 components coating.



Figure 7. X-ray diffraction pattern of MMC 2 components coating.



Figure 9. Microhardness profile of MMC 2 components coating.

As shown in Table 3 along with the summary of characteristics of coating quality, the tensile bond strength of MMC 3 components, and MMC 2 components were 5,778 psi and 6,570 psi where both

results were greater than acceptance threshold for TSC application on boiler tubes material (> 5,000 psi), MMC 2 components coating was much stronger than the MMC 3 components coating that signified better adhesion property. One thing that has been agreed that adhesion was strongly dependent on the roughness of the substrate surface [21]. The results indicated that adhesion is much stronger between metal/similar metal carbide (NiCr/MMC 2 components) than between metal/cermet (NiCr/MMC 3 components), because of the easy diffusion between similar metal atoms [22]. From the appearance given in the Figure 10 the fracture mode of MMC 3 components shown adhesive fracture since the fracture has separated the whole bond coat off the substrate, and in the other hand as seen in the Figure 11 coating failure of MMC 2 components after the test shown cohesive fracture where the delamination of some top coat section from the bond coat is observed.

Table 5. Summary of characteristics data of fivor mermal spray coating quant	Table 3. S	Summary of	characteristics	data of HVOF	thermal spray	v coating quality	ty.
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	Coating thickness		Hardness value			Dorositu	Doughpass	Dond
MMC	Bond	Tot.	Substrate	*BC - BC	Average	lovel	indox	strongth
Coating	coat	thickness	(HVN)	/ TC	top coat	(%)	(um)	(Psi)
	(µm)	(µm)		(HVN)	(HVN)	(70)	(µIII)	(1 31)
3 components	67	262	218.92 -	346.58 -	313 68	1 / 29	6 2284	5 778
5 components	07	202	252.93	397.38	515.00	1.427	0.2284	5,778
2 components	72	368 /	210.91 -	305.78 -	507.00	1 280	7 3872	6 570
2 components	12	508.4	235.08	498.17	307.99	1.207	1.3012	0,570

* BC: bod coat; BC / TC: interface between bond coat and top coat.



Figure 10. Coating fracture mode of MMC 3 components is adhesive failure, bond coat were detached more than 80% from substrate.



Figure 11. Coating fracture mode of MMC 2 components is cohesive failure, top coat deleamination from its bond coat.

4. Conclusion

This works has produced the following findings about quality characterization of HVOF thermal spray coating with NiCr matrix composite for protection application of coal fired boiler tubes.

- 1. MMC 3 components and MMC 2 components coating were successfully deposited by HVOF spray technique. The properties reveal uniform and adherent coating, and free from surface and cross sectional cracks with porosity less than 2%.
- 2. The presence of high carbide in the coating may be the reason for the high microhardness values in coatings as compared with the substrate. Except in MMC 3 components coating the presence of $NiCr_2O_4$ may be the reason for low microhardness.
- 3. Tensile bond strength test of both MMC 3 components and MMC 2 components were greater than the acceptable threshold. The results indicated that adhesion is much stronger between

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metal/similar metal carbide (NiCr/MMC 2 components) than between metal/cermet (NiCr/MMC 3 components), because of the easy diffusion between similar metal atoms.

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