

Using scratch test to evaluate cohesive bond strength of Mo composite coating

Hathaipat Koiprasert, Sirinee Thaiwatthana, Panadda Sheppard

National Metal and Materials Technology Center, National Science and Technology Development Agency, Pathumthani

ABSTRACT

Bonding strength of a thermal sprayed coating is difficult to measure using a conventional pull-off test method. Scratch test is a potential alternative testing method. An adhesive and a cohesive bond strength of the coating can be measured by the pull-off test while the scratch test performed on the cross-section of the thermal sprayed coating can only demonstrate the cohesive bond strength of the coating. Nevertheless, it is still beneficial to perform the scratch testing on the cross-section of the coating for the sake of comparison thus providing an alternative to the pull-off test. The scratch test method can reduce testing time and cost in the long run due to a significant cost reduction in consumables and energy and time saving from the curing step of the glue used in the pull-off test. This research investigates the possibility of using the scratch test to measure the cohesive bond strength of Mo/NiCrBSi composite coating. The results from the pull-off test and the scratch test indicate that the cohesive bond strengths of the Mo composite coating show similar trend and that the cohesive bond strength are increased when increasing NiCrBSi content.

Keywords: *scratch test, thermal spray, MoNiCrBSi, bond strength*

1. INTRODUCTION

Mo/NiCrBSi coating is used to improve wear resistance of industrial wear components such as die in metal forming, rollers in paper industry and the wear components in automobile. Low friction and high hardness are important to selected wear resistant coating. Mo is a soft phase with low friction while NiCrBSi added Cr to improve hardness [1, 2], moderate strength and corrosion resistance at high temperature [3, 4]. Each portion of Mo/NiCrBSi coating results in level of bond strength and wear mechanisms. These properties are essential to consider for process quality control before coated products reach users. Conventional pull-off test is often use to measure the bond strength of thermal spray coatings following ASTM C633. However, an expensive high strength adhesive is required for the testing and a long curing time is unavoidable for a sample preparation step. Previous works [6, 7] propose an idea of drawing an indenter across the cross-section of the coating and measure the cone area, see Figure 1, as a representation for the bond strength of the coating. It is a comparative study for the bond strength that the coating with a larger cone area refers to the coating with a lower bond strength. This research studies the possibility of

replacing the pull-off test method with the scratch test method to measure cohesive bond strength of the mixed coatings.

2. EXPERIMENTAL PROCEDURES

Stainless steel coupons of 25.4 mm diameter x 5 mm thick were used as substrates. All substrate specimens were grit blasted using 740 μm Al_2O_3 in order to achieve the surface roughness of at least 7 μm Ra. The high surface roughness is essential to ensure adequate bonding of the coatings. The specimens were then ultrasonically cleaned and dried. The specimens were spray coated using Sulzer Metco 3MB II plasma sprayed gun to achieve coatings thickness of 350-400 μm of Mo/NiCrBSi blended in different proportions to create 5 powder blends which are 100/0, 75/25, 50/50, 25/75 and 0/100. The spraying parameters are shown in Table I. The specimens were divided into 2 groups as a group of specimens for scratch test and a group of specimens for pull-off test. The specimens for scratch test, the first group, were prepared from cross-sectioned specimens that mounted in an epoxy resin. The mounted specimens were ground using a 5 μm SiC papers until a surface roughness of 0.02 μm Ra was achieved. Then, the specimens were ready for scratch test. Scratch test was applied in a direction from substrate toward coating and resin, respectively. The test parameters show in Table II. After the test, optical microscope, I-solution DT program and Image Pro Plus program were used as an image analyser to study scratch tracks, projected cone area, see Figure 1, and percentage of coating porosity. Vickers micro hardness measurements with 300g load and 15s dwelling time were also tested on the cross-sectional coatings. The specimens for pull-off (tensile bond strength) test, the second group, were prepared by using Master Bond EP15ND-2 glue stick coated specimen surface on a stainless steel stub of 25.4 mm. diameter x 40 mm. length and left to cure at 190°C for 75 mins, see Figure 2. The tension tested following ASTM C-633 was carried out by a universal testing machine Instron 8801 at 10,000kg load, 1 mm./min speed rate.

Table 1. Plasma spraying parameters

Powder	Plasma spraying parameters						
	A	Ar		H ₂		Powder feed rate (g/min)	Spray distance (mm.)
		Pressure (kPa)	Flow (LPM)	Pressure (kPa)	Flow (LPM)		
Mo	400	690	47.2	345	7.1	70	100
Mo-25%NiCrBSi	500	690	47.2	345	7.1	50	100
Mo-50%NiCrBSi	500	690	47.2	345	7.1	50	100
Mo-75%NiCrBSi	500	690	47.2	345	7.1	50	100
NiCrBSi	400	690	47.2	345	7.1	50	100

Table 2. Scratch parameters

Indenter type	Rockwell diamond radius 200 μm
Scratch load	16 N
Scratch speed	3.0 mm./min
Scratch length	1.5 mm.

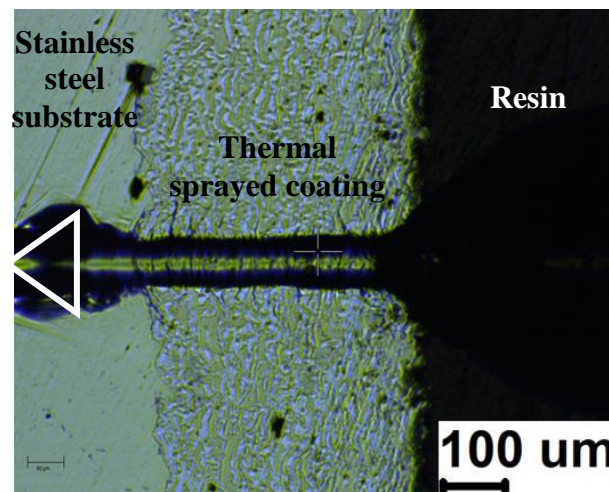


Figure 1. A cone area

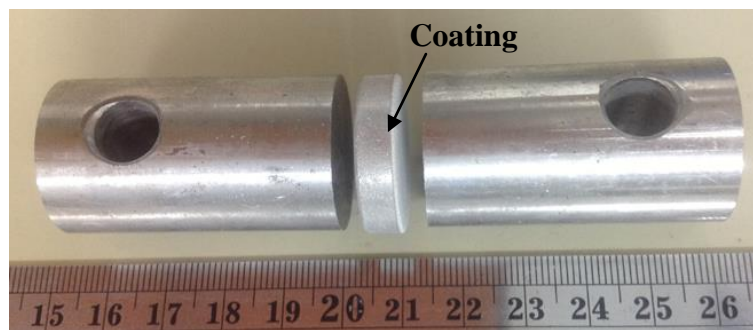


Figure 2. Tensile specimen.

3. Results

The cone area of each coating shows in Figure.3, A is Mo, B is Mo-25%NiCrBSi, C is Mo-50%NiCrBSi, D is Mo-75%NiCrBSi and E is NiCrBSi. Scratch indenter drawn across cross-section of the coating which started from stainless steel substrate through the coating into the epoxy resin. Small cracks, perpendicular to scratch direction, were observed around scratch tracks. A long crack was observed at an interface between coating and substrate of the NiCrBSi sample, Figure 3E. This might indicate the poorest adhesive bond strength than other coatings. Coating cohesive strength of specimens were investigated from cone area at the coating surfaces near the epoxy resin. The cone areas, according to Vencl A et. Al. [7], were estimated from Figure 3 and plotted in a graph in Figure 4. The larger the cone area, the lower scratch bond strength/weaker interlamellar cohesion bond strength/lower tensile bond strength [6, 7]. The cohesive bond strength of the coatings increased with increasing the percentage of NiCrBSi content.

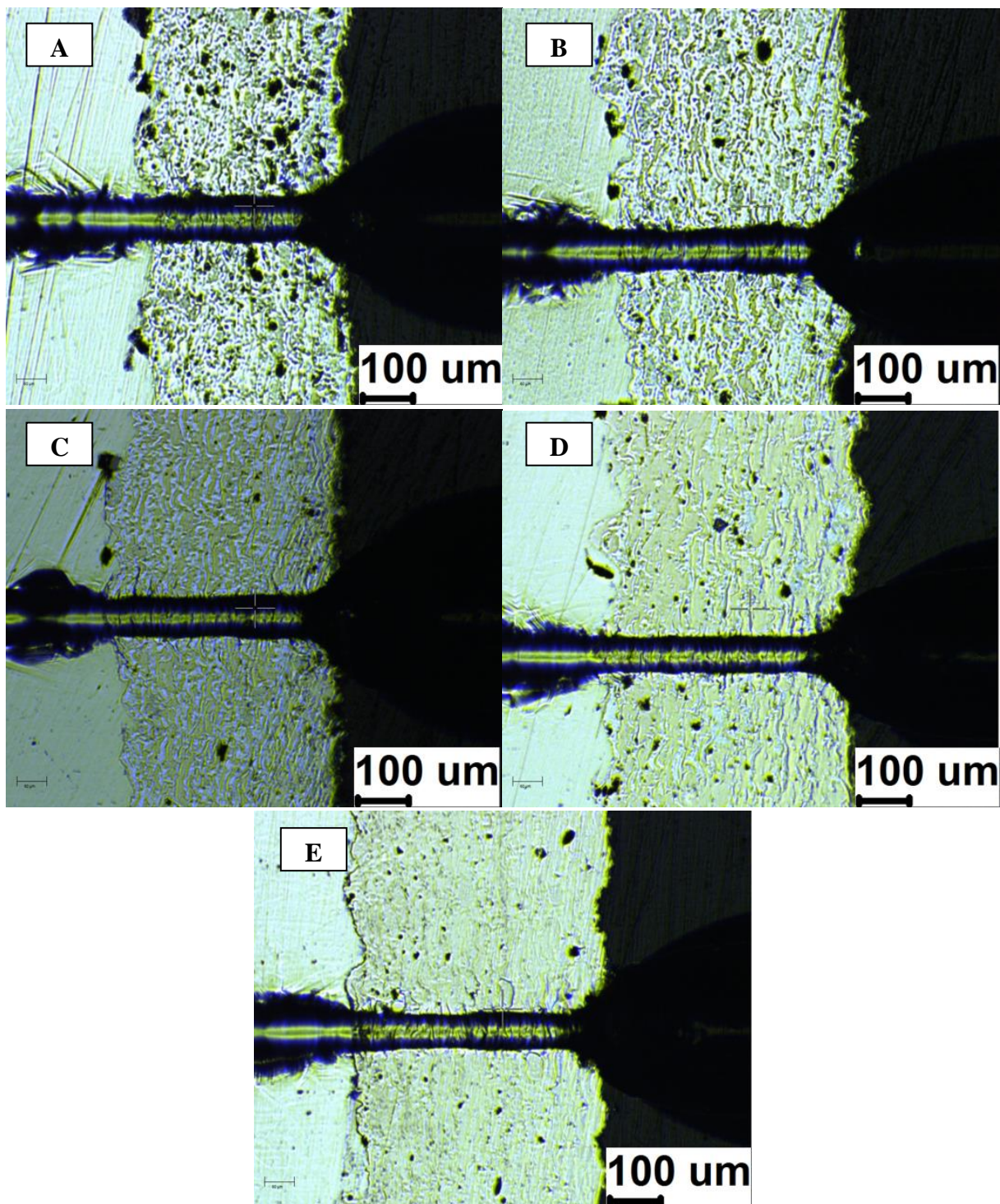


Figure 3. Optical micrographs of scratched specimens where A = Mo, B = Mo-25%NiCrBSi, C = Mo-50%NiCrBSi, D = Mo-75%NiCrBSi and E = NiCrBSi tested at 16N load, 3.0 mm/min scratching speed.

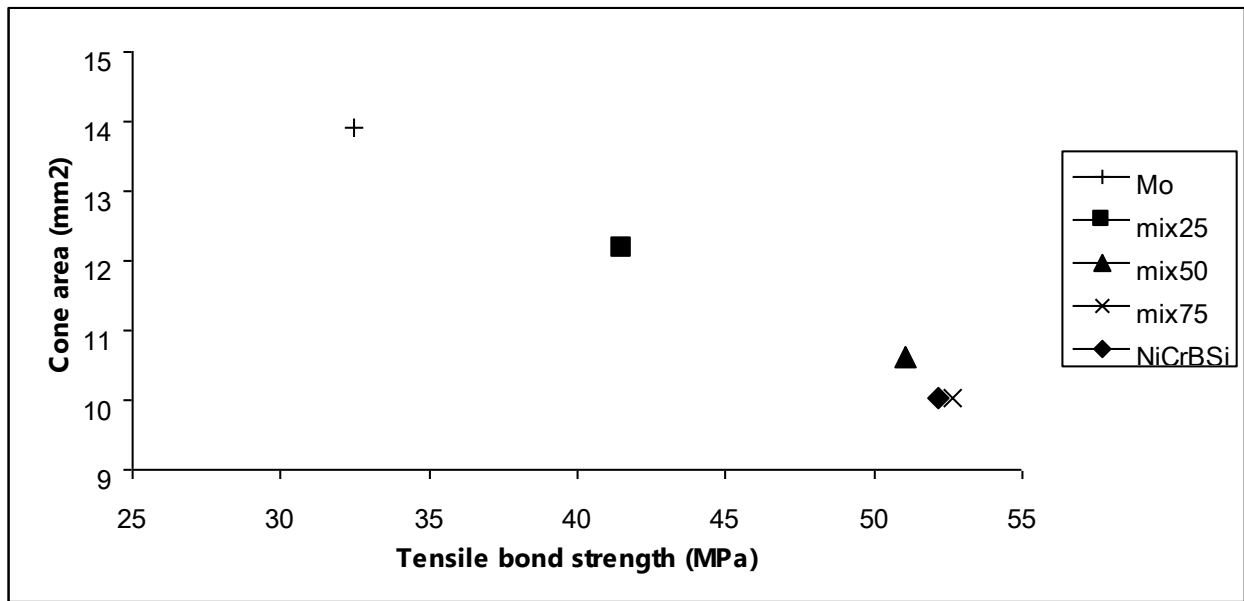
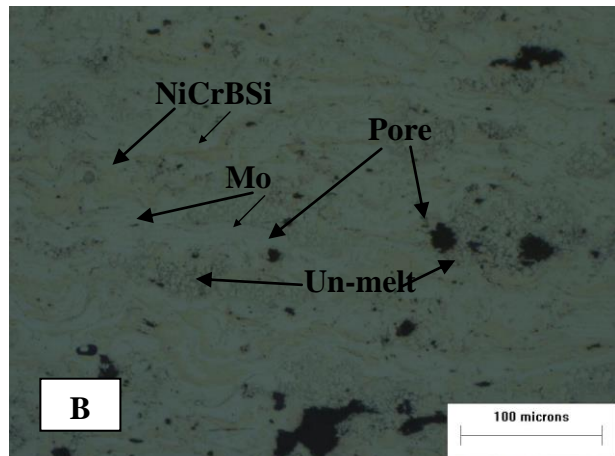
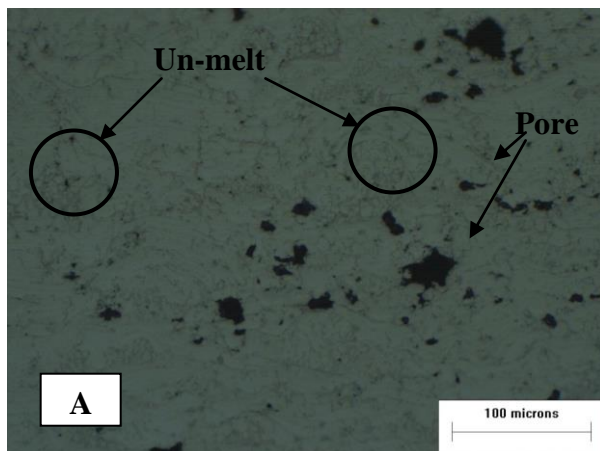


Figure 4. Correlation of the cone area from scratch test and tensile bond strength of Mo and Mo mixed with NiCrBSi plasma spray coatings where Mo = Mo, mix25 = Mo-25%NiCrBSi, mix50 = Mo-50%NiCrBSi, mix75 = Mo-75%NiCrBSi and NiCrBSi = NiCrBSi.

Cross sectional microstructure of the coatings prior to scratch test are shown in Figure 5. It can be seen that voids or porosity reduced when NiCrBSi content were increased. Calculated porosity percentage can be seen in Table III. Grinding process may pull out the un-molten particles especially Mo. Mo has a melting point of approximately 2,610°C, which probably too high for all Mo particles to completely melted in a plasma flame for short time. Mo agglomerated un-molten particles also appeared in the coatings. These areas may cause weaker cohesive bond strength than other splats.

Tensile bond strength of the coatings from pull-off test are shown in Table III. Tensile bond strength from pull-off test plotted against cone area achieved from scratch test is illustrated in Figure 4. A linear relationship between tensile bond strength and cone area was established. The higher tensile bond strength, the lower cone area. This suggested that the results of both tests were in agreement and shared similar trend that increasing NiCrBSi contents increased bond strength. Fracture specimens after pull-off test demonstrated in Figure 6.



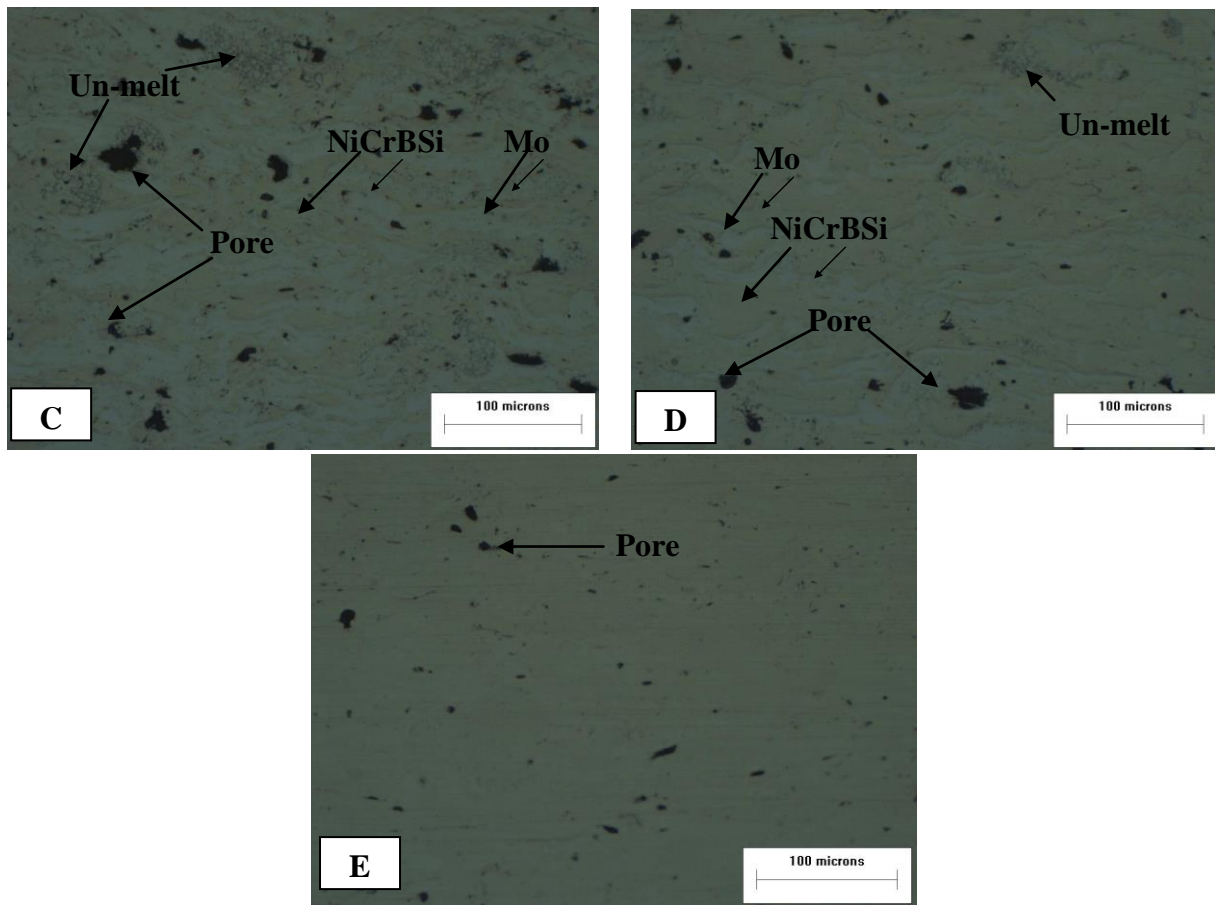


Figure 5. Optical microscope illustration cross-sectional view of the coatings at magnification 200x, A = Mo, B = Mo-25%NiCrBSi, C = Mo-50%NiCrBSi, D = Mo-75%NiCrBSi and E = NiCrBSi

Table 3. Porosity and Vickers micro hardness of the coatings.

Coating	Porosity (%)	Cross-sectional micro hardness (Hv0.3)	Scratch test cone area(mm ²)	Pull-off tensile strength (MPa)
Mo	5.0±1.5	499.4±61	13.9	32.4
Mo-25%NiCrBSi	3.7±0.6	490.3±120	12.1	41.5
Mo-50%NiCrBSi	4.0±0.6	535.9±132	10.6	51.0
Mo-75%NiCrBSi	1.9±0.2	601.3±127	10.02	52.7
NiCrBSi	1.2±0.2	612.6±28	10.01	52.2

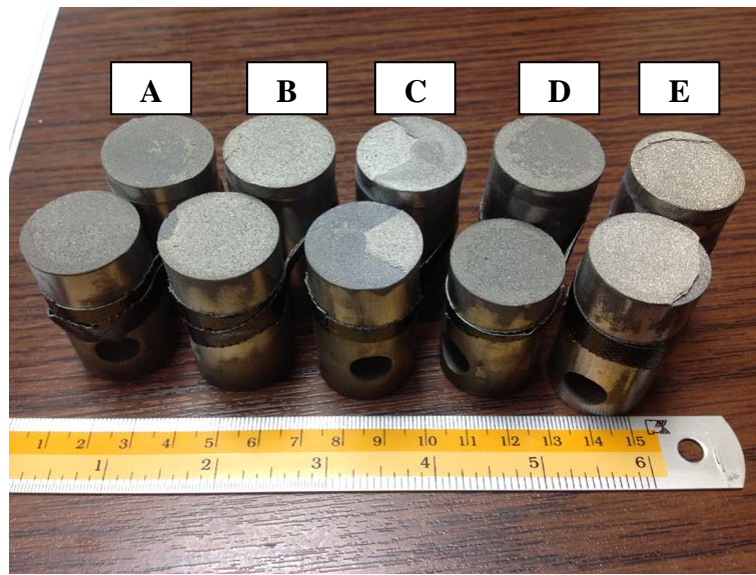


Figure 6. Failure of tensile bond strength specimens when A = Mo, B = Mo-25%NiCrBSi, C = Mo-50%NiCrBSi, D = Mo-75%NiCrBSi and E = NiCrBSi

4. CONCLUSION

Using scratch test to evaluate cohesive bond strength of Mo composite coating replacing pull-off test is possible to compare the bond strength in qualitative test. The largest cone area shows the weakest cohesive bond strength that is the lowest scratch bond strength. The scratch bond strength is correlation with the tensile bond strength. For increasing confidential to use scratch test to evaluate cohesive bond strength as a replacement testing method for the pull-off test, comparative testing on several more kinds of coatings are required.

REFERENCES

- [1] Grigorescu, C, Di Rauso, C, Drira-Halouani, R, Lavelle, B, Di Giampaolo, R, Lira, J. 1995. Phase characterization in Ni alloy-hard carbide composites for fused coatings. *Surface and Coatings Technology*. 76-77. pp494-498.
- [2] Tobar, M.J., Alvarez, C, Amado, J.M., Rodríguez, G., Yanez, A. 2006. Morphology and characterization of laser clad composite NiCrBSi-WC coatings on stainless steel. *Surface and Coatings Technology*. 200. 22-23. pp6313-6317.
- [3] Nicoll, A.R. 1982. Self-fluxing coatings for stationary gas turbines. *Thin Solid Film*. 95. pp285-295
- [4] Rodríguez, J, Martín, A, Fernández, R, Fernández, J.E. 2003. An experimental study of the wear performance of NiCrBSi thermal spray coatings. *Wear*. 255. pp950-955.
- [5] Niranatlumpong, P, Koiprasert, H. 2010. The effect of Mo content in plasma-sprayed Mo-NiCrBSi coating on the tribological behaviour. *Surface and Coatings Technology*. 205. pp 483-489.
- [6] Nohava, J, Bonferroni, B, Bolelli, G, Lusvardi, L. 2010. Interesting aspects of indentation and scratch methods for characterization of thermally-sprayed coatings. *Surface and Coatings Technology*. 205. pp1127-1131.

- [7] Vencl, A, Arostegui, S, Favaro, G, Zivic, F, Mrdak, M, Mitrović, Popovic, V. 2011. Evaluation of adhesion/cohesion bond strength of the thick plasma spray coatings by scratch testing on coatings cross-sections. *Tribology International*. 44. pp1281-1288.
- [8] Koiprasert, H, Thaiwatthana, S, Sheppard, P. 2013. Feasibility study on the use of scratch test to evaluate bond strength of thermal sprayed coating and optimization of test parameters. *International Symposium on the Fusion Technologies between Korea and Thailand 2013(ISFT2013)*. 1st-3rd August. Thailand.