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EFFECT OF HIGH FREQUENCY HEAT TREATMENT ON THE MICROSTRUCTURE AND MACROSCOPIC PROPERTIES OF WC-50Ni+STELLITE 1 COATING LAYER FABRICATED BY HVOF SPRAY PROCESS

The microstructure and macroscopic properties of WC-50Ni+stellite 1(Co-Cr-W, ST1) coating layer fabricated by HVOF spray have been investigated. WC-50Ni powder and ST1 powders were mixed in the ratio of 1:0 and 5:5 wt.%, respectively. Argon heat treatment (Ar) and high-frequency heat treatment (H.F.) were conducted on the coating materials. WC was decomposed in the Ar heat treatment specimen, but decomposition of WC was not observed in the H.F. heat treatment specimen. Hardness was measured for as-sprayed WC-50Ni (821.5Hv) and as-sprayed WC-50Ni+ST1 (668.1 Hv). Hardness of Ar heat treatment specimen was reduced by about 14~18% than that of the as-sprayed coating layers. However, when the H.F. heat treatment was performed, the hardness inversely increased by about 6~10% than the as-sprayed coating layer. Based on these results, the method to improve the mechanical property of HVOF sprayed WC-50Ni+ST1 coating layer has also been also discussed.

Keywords: HVOF spray, WC-50Ni, Stellite 1, composite coating, high frequency heat treatment

1. Introduction

WC-50Ni self-fluxing alloy (SFA), typically formed by adding WC-12Co to Ni based SFA, is a material with enhanced wear resistance. Owing to such wear resistance characteristics, WC-50Ni is utilized in mold, boiler tube, and grate in power plants, mainly where excellent wear resistance property is required at room and high temperature. WC-50Ni SFA has been recently considered as a material for roller surface, with molten zinc, and aluminum plating pots. However, WC-50Ni SFA has low galvanic corrosion resistance in molten zinc and aluminum plating bath. Presently, bulk Co base alloy (Stellite, tribaloy) with excellent corrosion resistance is utilized in rollers for hit dip process [1], but bulk Co base alloy has low wear resistance property and is also expensive.

Thermal spray process makes use of thermal energy and kinetic energy to lay feedstock on the surface of substrate. Such laying imparts the intrinsic property of the feedstock to the surface of the substrate. Among many thermal spray processes, high velocity oxy-fuel spray (HVOF) is capable of making a coating layer with a relatively denser microstructure and higher adhesion strength between the matrix and coating layer, compared to other thermal spray coating processes [2,3]. However, microstructural defects such as partially molten particles and pores could still exist inside the coating layer. Argon (Ar) heat treatment is mainly employed as a defect control method for thermal spray coating layer [4]. However, Ar heat treatment takes a long time for heating rate and holding time. Furnace heat treatment also has an important downside that it is not operable if a substrate has a far lower melting point than the coating layer. Against this backdrop, the high frequency heat treatment has gained attention for its quick temperature rise and possibility of local heat treatment, say solely for a coating layer [5]. Unfortunately, enough studies have not been done to-date on the effect of high frequency heat treatment on the microstructure and mechanical properties of HVOF sprayed WC-50Ni coating layer.

In the current study, HVOF coating layers were made by mixing Stellite 1 alloy powder having excellent zinc and aluminum erosion resistance with WC-50Ni SFA powder, followed by high frequency heat treatment on the prepared coating materials. The as-sprayed or high frequency heat-treated WC-50Ni and WC-50Ni+Stellite 1 coating layers were then examined for their microstructure and mechanical properties. Additionally, the microstructure and mechanical properties of the conventional furnace heat-treated coating layers in Ar atmosphere were also conducted and compared.

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2. Experiment

To fabricate a composite self-fluxing coating material, mixtures comprising WC-50Ni powder and Stellite 1 powder (Praxair Co.) in 1:0 and 5:5 wt.% ratio were used. Fig. 1 exhibits the SEM/EDS results of the used powder feedstocks in this study. First, the WC-50Ni powder was found to have the mixture of WC-Co and NiCrBSi powders. The WC-Co powder comprised spherical particles of average size 34.3 μm with a rough surface. The NiCrBSi powder consisted of spherical particles of average size 38.1 μm. In the WC-50Ni+Stellite 1 mixture, Stellite 1 powders were globular, the particles with average size of 26.9 μm. The optimal process conditions were identified by controlling several process parameters. Table 1 shows the HVOF spray process conditions applied to the fabrication of coating layers. The substrate selected was AISI 316L and JP-8000 HVOF spray equipment was used. The aforementioned powder and process conditions were employed to fabricate HVOF sprayed WC-50Ni and WC-50Ni+ST1 coating materials. Fig. 2 shows the images of the initial specimens of the fabricated coating materials. The coating area of the initial specimens was set to 50 mm × 50 mm. And then, high frequency heat treatment was performed to the manufactured coating layers. With respect to the high frequency heat treatment conditions, the traveling speed was 10 mm/sec. and the heating rate was 20°C/sec with 2 passes. The temperature of the specimens (950°C) was measured just after the heat treatment followed by air cooling. As the most common heat treatment process, the Ar atmosphere heat treatment was also conducted to the manufactured coating layer for comparison. The Ar heat treatment was performed at the heating rate of 3°C/sec., then kept at 950°C for 1 hour, followed by furnace cooling.

TABLE 1

HVOF spray process parameters used in this study

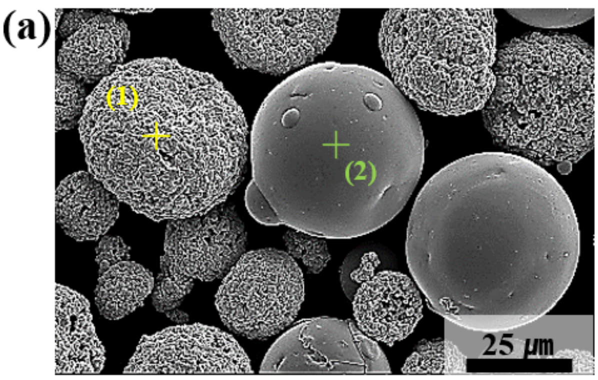
Parameter	Fuel flow [GPH]	O_2 flow [SCFH]	Distance [mm]	Feed rate [g/min]	Speed [mm/s]
HVOF	4.5	1400	300	65	10

To observe the microstructure, cross-section of the coating layer was first polished to #4000 using SiC paper, then mirror polished to 1 μm. For phase analysis and microstructure observation, X-ray fluorescence spectrometer (Rigaku, ZSX Primus II), X-ray diffractometer (XRD, Ultima IV, Cu Kα, step size: 0.05°, scan rate: 2°/min), and SEM/EDS (scanning electron microscope, Tescan, VEGA II LMU) were used.

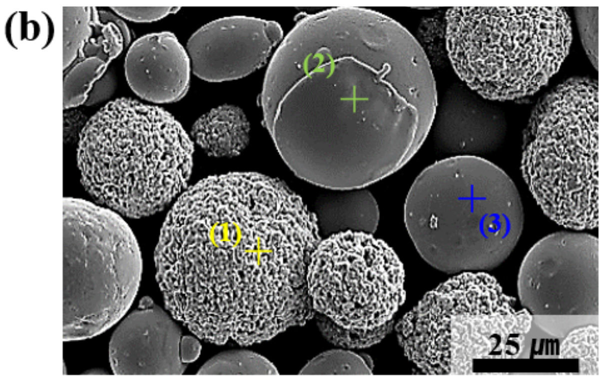
To assess the mechanical properties of the fabricated HVOF sprayed WC-50Ni and WC-50Ni+Stellite 1 coating layers, micro Vickers hardness tester (Akachi, AVK-C100) was used. Vickers hardness test condition was set as load: 300g and hold time: 10 sec. Hardness was measured 12 times and the average value was used except the maximum and minimum values.

3. Results and discussion

XRF analysis was performed to investigate the chemical composition of WC-50Ni and WC-50Ni+ST1 coating layers fabricated by HVOF spray process. The results are listed in Table 2. The HVOF sprayed WC-50Ni coating layers were found to have W 38.51 wt.%, Ni 39.98 wt.%, Cr 11.35wt.%, and Co 5.09wt.%. In the WC-50Ni+ST1 coating layers, compared to the WC-50Ni coating layers, the Co and Cr contents surged as Stellite 1 was added while W and Ni contents decreased.



El.(Wt.%)	(1)	(2)
Ni	-	**74.31**
Cr	-	**10.60**
W	**72.49**	-
Co	**12.79**	-
Si	1.48	**5.68**
C	**13.24**	9.41
Total	100.00	100.00



El.(wt.%)	(1)	(2)	(3)
Ni	-	**71.36**	-
Cr	-	**14.91**	**29.10**
W	**82.63**	-	**14.26**
Co	**6.89**	-	**47.91**
Si	2.34	**5.93**	1.44
C	**8.14**	7.80	7.29
Total	100.00	100.00	100.00

Fig. 1. Morphologies of initial powders used in this study; (a) WC-50Ni and (b) WC-50Ni+ST1

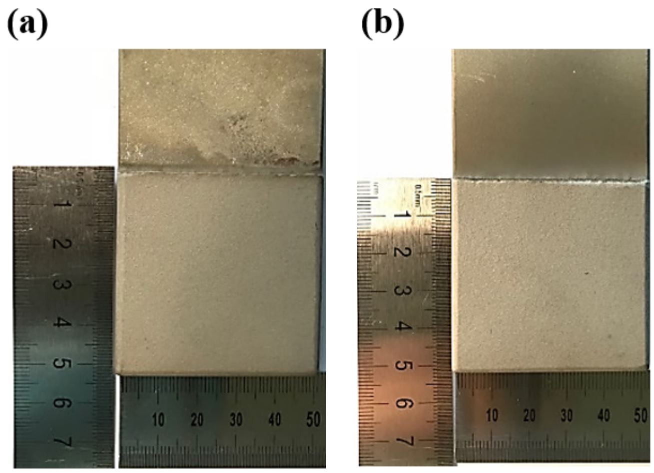


Fig. 2. Images of initial specimens; (a) of HVOF sprayed WC-50Ni and (b) of HVOF sprayed WC-50Ni+ST1

TABLE 2

Chemical compositions of HVOF sprayed WC-50Ni and WC-50Ni+ST1 coating layers

wt.%	W	Ni	Cr	Co	Fe	Si	Al	S
WC-50Ni	38.51	39.98	11.35	5.10	3.07	1.93	0.04	0.03
WC-50NI+ST1	24.80	18.88	23.35	30.00	2.22	1.39	0.02	0.03

Fig. 3 shows the macroscopic features of the cross-sections of the as-sprayed WC-50Ni and WC-50Ni+ST1 coating layers. The thickness of both types of coating layers was about 2 mm. Neither any macro crack was found inside the coating layers, nor any delamination from the substrate. The WC-50Ni coating layers were divided into bright region (Ni SFA area) and dark region (WC area). The fractions of each region, as measured using an image analyzer, were 49.82% the bright region and 50.18% the dark region. The Stellite 1-added WC-50Ni+ST1 coating layer was also divided into bright region and dark region. The fraction of bright region was 67.15% and that of the dark region was 32.85%.

Fig. 4 shows the microstructure characteristics of fabricated coating materials according to the heat treatments (Ar and H.F.) and Stellite 1 powder mixture. The porosity of the As sprayed WC-50Ni material was found to be 1.02%. Adding Stellite 1 greatly increased the porosity to 3.97%. To note, both the types of the material had pores concentrated at particle boundary, which were in a very sharp form. The porosities of Ar heat-treated WC-50Ni and WC-50Ni+ST1 coating layers were 0.64% and 0.11%, respectively. As the heat treatment was implemented, the pore size reduced. The H. F. heat-treated WC-50Ni and WC-50Ni+ST1 showed porosity of 0.51% and 0.07%, respectively. These values are 1.25 and 1.57 times lower than those of the Ar heat-treated materials. They indicate that defect control was more effective with the implementation of high frequency heat treatment process. The H. F. heat treated coating layers also had some pores at the particle boundary, but their shapes changed to a round one. Defect at the particle boundary could undermine the mechanical properties of the coating layers. Sharp pores, in particular, work as a stress concentration site to reduce the toughness of coating layer and the bonding strength between particles.

XRD phase analyses of WC-50Ni and WC-50Ni+ST1 coating layers are shown in Fig. 5. The as-sprayed WC-50Ni and WC-50Ni+ST1 materials consisted of WC, Co and Ni phases, and fine W_2C peaks were also detected. As the Ar heat treatment was performed, small increases in the WC, Co, Ni and W_2C peak intensities were observed for WC-50Ni and WC-50Ni+ST1. In the H. F. heat treated WC-50Ni and WC-50Ni+ST1 coating layers, similar to the as-sprayed materials, WC, Co and Ni peaks were mainly observed and W_2C peaks were slightly detected.

Fig. 6 exhibits the SEM/EDS analysis results of HVOF sprayed WC-50Ni and WC-50Ni+ST1. The as-sprayed WC-50Ni material consisted of 2~5 μm angular shaped WC particles and metal matrix. In the Stellite 1-added WC-50Ni+ST1 material, WC particles were about 2~5 μm in size, and a little change in the form was observed. The base element of Stellite 1, Co, has excellent wetting angle with WC that can be used as a metal matrix element to produce WC cermet material, but

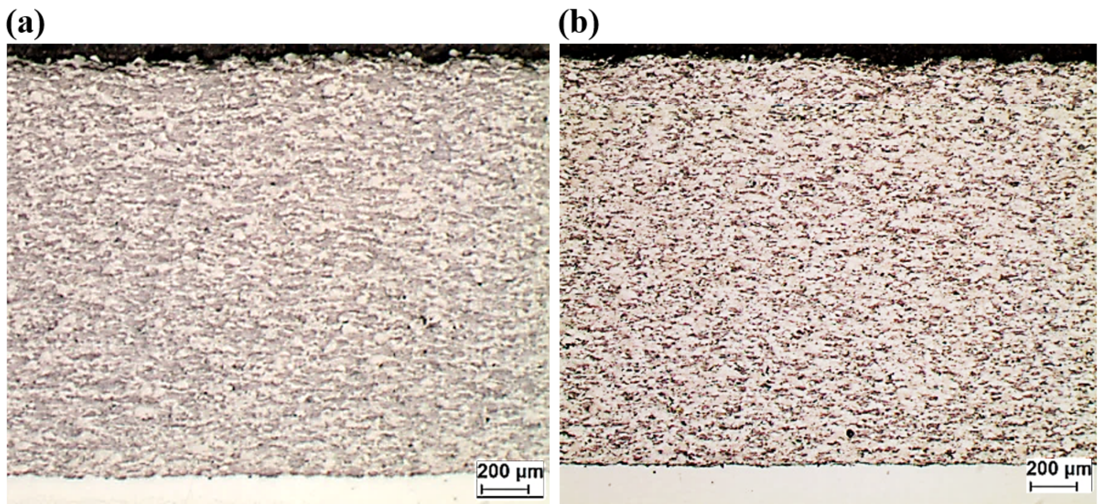


Fig. 3. Macroscopic images of as sprayed coating layers; (a) WC-50Ni and (b) WC-50Ni+ST1

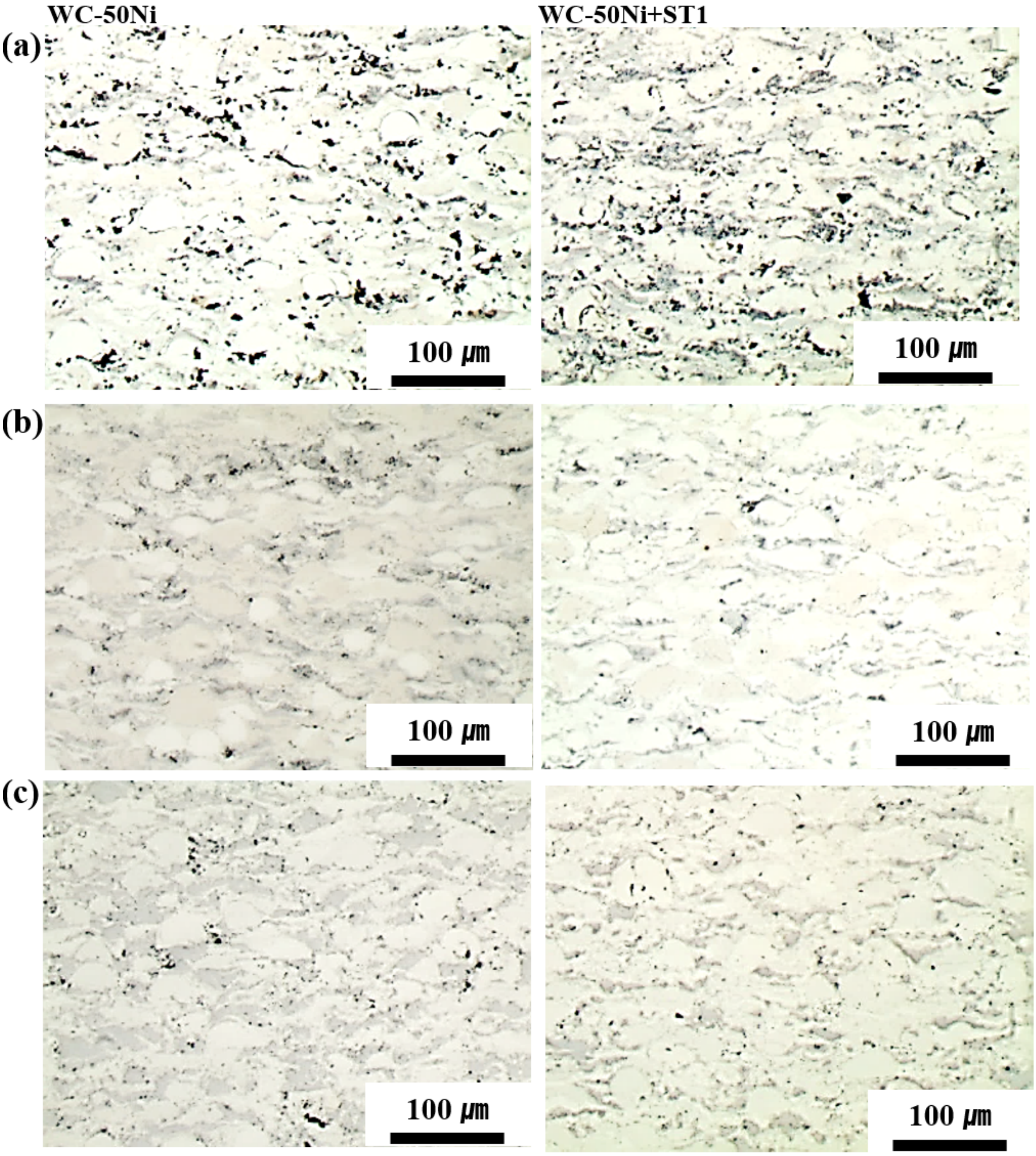


Fig. 4. Microstructures of WC-50Ni and WC-50Ni+ST1 coating layers; (a) as sprayed, (b) Ar heat treated and (c) H.F. heat treated

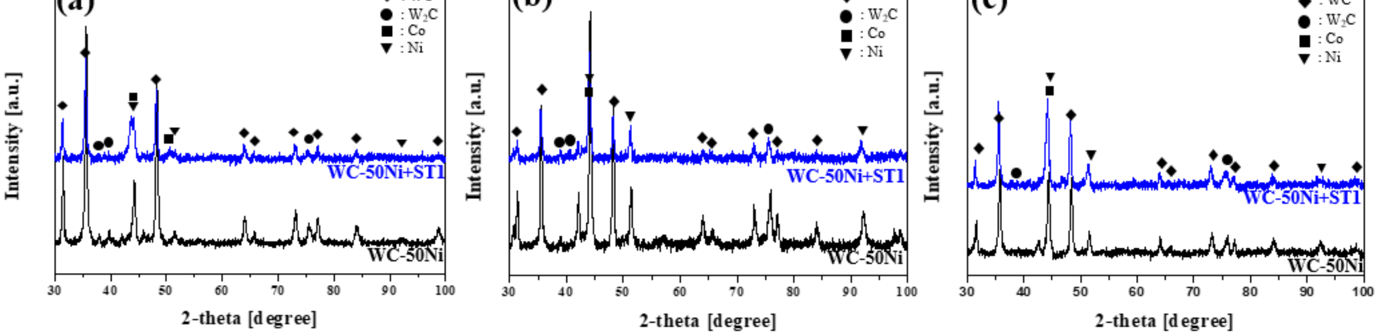
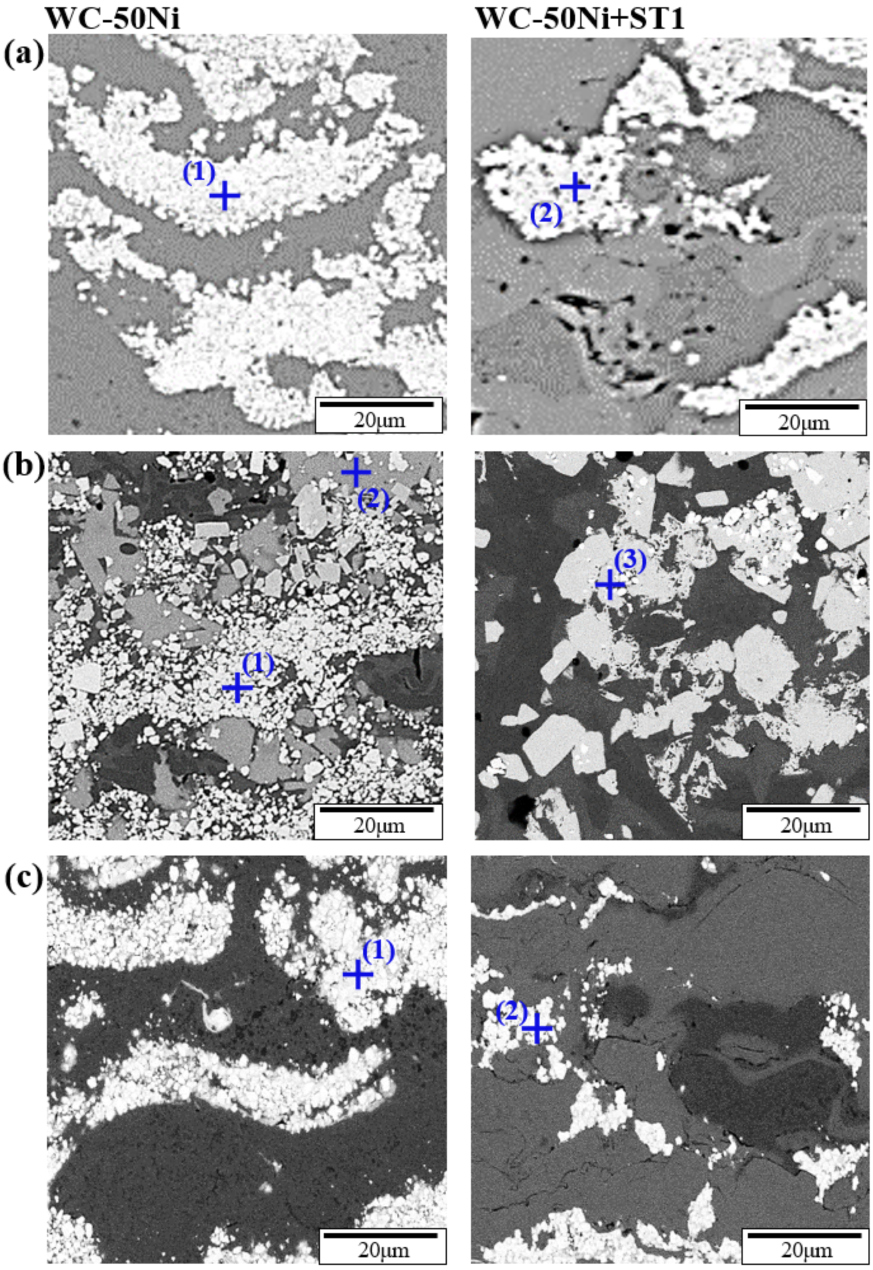


Fig. 5. XRD analysis results of WC-50Ni and WC-50Ni+ST1 coating layers; (a) as sprayed, (b) Ar heat treated and (c) H. F. heat treated



El.(wt.%)	(1)	(2)
W	**83.22**	**82.15**
Co	**8.11**	**8.44**
Si	0.24	0.74
C	**8.43**	**8.67**
Total	100.00	100.00

El.(wt.%)	(1)	(2)	(3)
W	**85.21**	**97.55**	**97.09**
Co	**7.09**	**0.32**	**0.97**
Si	0.10	0.21	0.14
C	**7.60**	**2.03**	**1.80**
Total	100.00	100.00	100.00

El.(wt.%)	(1)	(2)
W	**82.72**	**81.86**
Co	**9.14**	**10.06**
Si	0.21	0.18
C	**7.93**	**7.90**
Total	100.00	100.00

Fig. 6. SEM/EDS results of WC-50Ni and WC-50Ni+ST1 coating layers; (a) as sprayed, (b) Ar heat treated and (c) H. F. heat treated

could facilitate WC decomposition. Although Stellite 1 was added in the as spray condition, the WC form and size did not change greatly. That is, it seemed that adding Stellite 1 through powder mixing does not cause WC decomposition under the as-sprayed condition. With the Ar heat treatment, the WC-50Ni and WC-50Ni+ST1 materials showed coarse phase which was not detected in the as-sprayed material. Generally, W based cermet material (WC-Co) could show W_2C and Co_6W_6C phases because of WC decomposition with heat treatment [6]. The coarse phase generated in the Ar heat treatment was found to have W base, about 2 wt.% C and 1 wt.% or lower Co, forming the W_2C phase. On the other hand, similar to the as-sprayed material, the H. F. heat-treated WC-50Ni and WC-50Ni+ST1 coating layers consisted of 2~5 μm WC particles and metal matrix. Their microstructure was slightly denser than that of the as-sprayed material. The WC particles of the H. F. heat-treated WC-50Ni and WC-50Ni+ST1 coating layers were observed to have similar sizes and forms as that of the WC in the as-sprayed material. EDS analysis found that the WC particles in the two layers also had similar compositions. In other words, it seems that even though H. F. heat treatment was performed, WC particle decomposition did not occur. Such a finding is noteworthy given that the temperature of the material surface immediately after the H. F. heat treatment was 950°C, as an identical temperature in the Ar heat treatment condition.

Fig. 7 shows the Vickers hardness results of HVOF sprayed coating layers. The hardness of the as-sprayed WC-50Ni coating material was 821.5Hv which dropped to 668.1 Hv, i.e. 18% on addition of Stellite 1. Generally, WC-50Ni coating layers are known to have hardness of approximately 700~800 Hv,

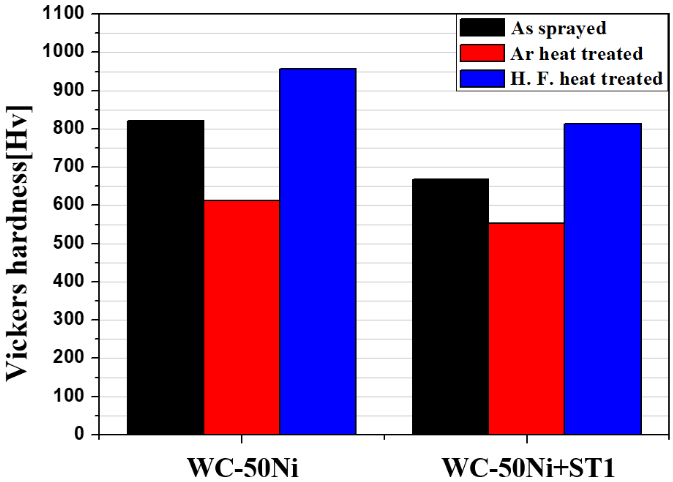


Fig. 7. Vickers hardness results of WC-50Ni and WC-50Ni+ST1 coating layers

while the hardness of Stellite 1 is 550~720 Hv (Praxair Co. data sheet). It seems that the hardness reduced with Stellite 1 addition because Stellite 1 has a relatively lower hardness and increased porosity. The hardness of the Ar heat-treated WC-50Ni and WC-50Ni+ST1 coating layers were 612.4 Hv and 552.3 Hv, respectively. The Ar heat treatment effectively controlled porosity, compared with the as sprayed material; but the hardness was found to decrease by 25% (WC-50Ni) and 17% (WC-50Ni+ST1) from those of the as-sprayed materials. The WC-added self-fluxing alloy can have higher hardness and better wear resistance. However, the Ar heat treatment seems to have caused the WC decomposition and W_2C generation which is stable at high temperature to reduce the hardness of coating layer. The hardness of H. F. heat-treated WC-50Ni and WC-50Ni+ST1 coating layers were 956.1 Hv and 812.9 Hv. The above results are 1.1 (WC-50Ni) and 1.2 (WC-50Ni+ST1) times higher than those of the as-sprayed materials, and 1.5 (WC-50Ni) and 1.4 (WC-50Ni+ST1) times improved than those of the Ar heat treated materials. The excellent hardness properties of H. F. heat treatment was mainly because of the quick temperature increase and short heat treatment time that caused no WC decomposition and reduction in the pore size.

4. Conclusions

The HVOF sprayed WC-50Ni and WC-50Ni+ST1 coating layers with 2 mm thickness were fabricated. No detachment between the substrate and the coating layers was observed. The porosity of the as-sprayed WC-50Ni material was 1.02% which increased to 3.97% after addition of Stellite 1. The porosity was effectively controlled via Ar heat treatment and H. F. heat treatment. However, the H. F. heat treated material had a lower porosity than the Ar heat treated material.

The WC particles inside the as-sprayed WC-50Ni and WC-50Ni+ST1 coating layers were 2~5 μm in size having square shape. On the other hand, the WC particles inside the Ar heat-treated coating layers were partially decomposed. By contrast, the H. F. heat treated coating layers did not undergo any WC particle decomposition while maintaining the similar size and shape as those of the particles of the as-sprayed coating layers.

The as-sprayed WC-50Ni coating material was found to have 821.5Hv hardness, and the hardness of the WC-50Ni+ST1 coating layer was 668.1Hv. The hardness of the Ar heat treated coating layers was 612.4 Hv for WC-50Ni, and 552.3Hv for WC-50Ni+ST1, which were lower than the hardness values of the as-sprayed coating layers. The hardness of H. F. heat treated coating layers were 956.1 Hv for WC-50Ni and 812.9 Hv for WC-50Ni+ST1. The excellent mechanical properties of H. F. heat treatment was probably because of the quick temperature increase and short heat treatment time.

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