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Effect of Physical Vapor Deposition Parameters on Coating Roughness and Adhesive Force for Lead-cooled Fast Reactor Structure

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1. Introduction

Small modular reactors (SMRs) are one of the most emerging topics as a new concept of nuclear power generation that can be used flexibly in a variety of environments and has economic feasibility and safety with a small and simple design. In order to satisfy this design, there is a movement to combine it with a lead-cooled fast reactor (LFR), which is one of the concepts of the 4th generation nuclear power plant. LFR is a fast reactor that uses lead or lead-bismuth eutectic (LBE) as a coolant and has many advantages in using lead as a coolant. Low neutron absorption cross-section and high boiling point of lead can increase efficiency. Also, LFR has is enhanced safety with near-atmospheric operating pressure and high gamma-ray shielding of lead [1].

However, high corrosiveness of lead causes several problems for structural materials which is even more severe for nuclear power plants that operate for a long time without replacing parts of structural materials.

To solve this problem, functionally graded composite (FGC) or coated structural materials are emerged as a new solution [1-2]. Coated materials have both higher mechanical properties of structural material and corrosion resistance from coated surface. As a coating method, physical vapor deposition (PVD) is widely used in industrial field since it is cheap, simple and have large field experiences [3].

SS316L, a main structural material in many nuclear reactors, was used as the substrate in this research. As a coated layer, SS304L was used instead of various austenite corrosion resistance alloys, but having similar major chemical composition. In this paper, the effect of substrate roughness and sputtering power during PVD on roughness of coated layer and adhesive force was investigated. Roughness of coated layer have close relationship with corrosion rate of it, and adhesive force is an important parameter which could evaluate the maintenance of the coating even in severe environments.

2. Experiments

2.1 Sample preparation

SS316L with a thickness of 1 mm was used as a substrate. The SS316L plate was cut to have size of about 15 mm in width and 20 mm in length, respectively. Surface roughness of specimens were controlled by polishing with SiC paper or diamond suspension. 3 groups of specimens were prepared. The first group was polished with 6 μm diamond suspension after grounding with 800 grit SiC paper. The second group was grounded with 600 grit SiC paper. And the third group was grounded with 60 grit SiC paper. Each substrate group was named 6 μm dialube, 600 grit, and 60 grit. The target used for deposition was SS304L and was cast in a size of 600×200×10 mm.

2.2 PVD process

The 3 groups of polished substrates were deposited with different sputtering energy of 0.5 kW, 1.0 kW, and 1.5 kW. After all process, 9 types of coated specimens were compared, and the specimen table is as follows.

Table I: Test matrix of PVD coated layer

		Surface polishing		
		60 grit	**600 grit**	**6 μm**
Sputtering energy	**0.5 kW**	0.5k60	0.5k600	0.5k6μ
	1 kW	1k60	1k600	1k6μ
	1.5 kW	1.5k60	1.5k600	1.5k6μ

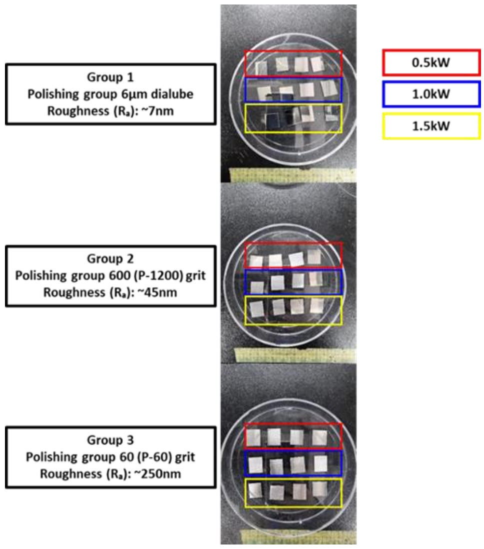

Fig. 1. Substrate specimens before deposition

Same deposition thickness was achieved by analyzing cross section since the deposition rate is different for each power. Deposition was carried out in the range of 100 mm×100 mm at the center of the substrate holder and specimens were arranged as shown in Figure 2. Before deposition process, ion etching was conducted with 30 W and 1.5 mTorr environment to remove foreign substances on the surface. And deposition was carried out at 2 mTorr for all cases.

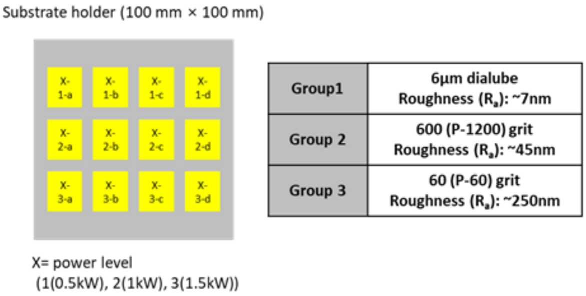

Fig. 2. Specimen layout on substrate holder

2.3 Analysis method

The roughness of the polished substrate before deposition was analyzed. Roughness of substrate before and after PVD were measured by non-contact 3D shape measuring equipment that uses white light scanning interferometry. Each surface was three-dimensionally scanned at 50x magnification and the roughness was analyzed from it. For all specimens, the roughness of the substrate and the roughness of the surface were measured at least three times.

The adhesion strength of the coated layer was analyzed by scratch test. Progressive loading was used, which is a method of increasing the load at a certain rate while proceeding the scratch. The starting load was 0 N and the final load was 30 N. The total scratch length was 1 mm. The tip was scratched while moving at 0.1 mm/s, and a constant force was applied at 3 N/s.

3. Results and Discussion

3.1 Relation between coating roughness and PVD process parameters

Surface roughness measurement results of each case are summarized in figure 3, 4 and 5. The figures show the average and maximum/minimum values of the substrate roughness before coating and roughness of coated layer by power in a specific roughness group. Roughness of coated specimen has similar value with that of substrate in most cases regardless of power. However, in the 1.5kW sputtering power environment, the coating roughness of 600 grit and 60 grit tends to be lower than the substrate roughness before coating. The temperature of the substrate and coated layer might be increased more than that of other specimens due to higher power. When deposition proceeds on 600 grit and 60 grit group, which are relatively high substrate roughness, formation of defects or porosity are expected due to shading effect [4]. However, it is thought that the relatively high substrate roughness at a higher temperature shows a tendency to decrease slightly compared to the substrate roughness before deposition because even if porosity occurs during deposition, diffusion occurs, and the porosity tends to decrease [5].

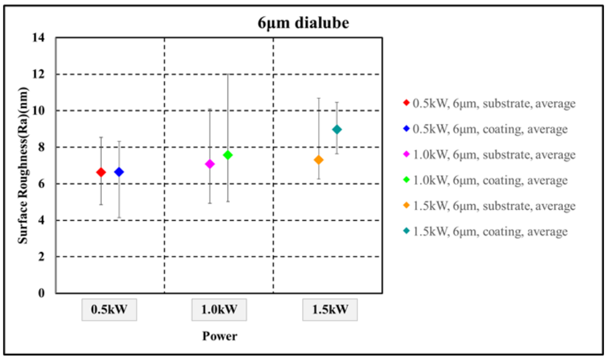

Fig. 3. Substrate and coating roughness of 6 μm dialube group

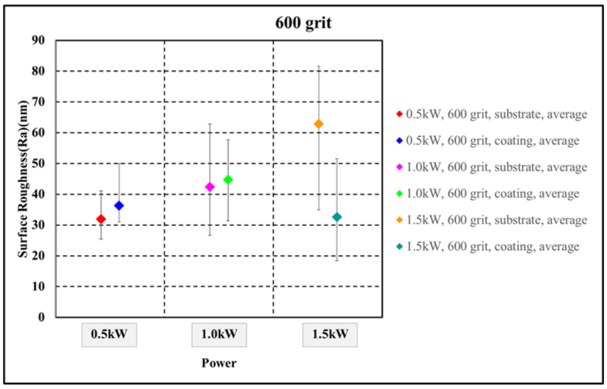

Fig. 4. Substrate and coating roughness of 600 grit group

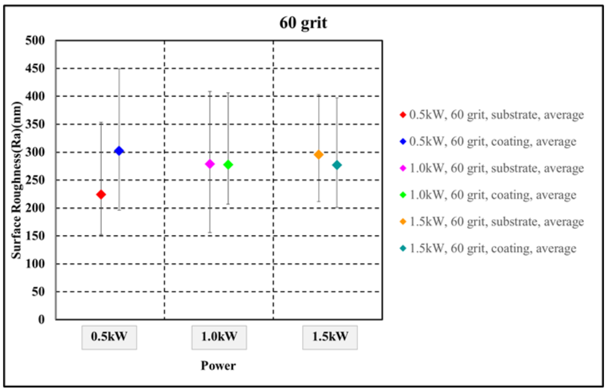

Fig. 5. Substrate and coating roughness of 60 grit group

3.2 Relation between adhesive force and PVD process parameters

Critical forces, L_{c1}, L_{c2} and L_{c3} for changes in substrate roughness and sputtering power are presented in table II and figure 6. A value of 30 N in the figure 6 indicates more than 30 N. As shown in figure 6, the 1.0 kW deposition specimen had a higher L_C value than other coated specimens, which means that the adhesive strength is strong. In particular, there were two specimens that did not show a critical load even at the final load of 30 N in the scratch test. The reason is that as the power increases, the sputtered element applies a strong compressive stress to the substrate, but if energy is applied over a certain power, undue compressive stress is applied to the coating and coating failure can occur [6]. Up to a power of 1.0 kW had a positive effect on in terms of compressive stress and adhesive properties, but it can be expected that as the power went up to 1.5 kW, the compressive stress became excessive and negatively affected the adhesive properties. For this reason, the adhesive force of 1.5 kW power was lower than that of 1.0 kW. As regards substrate roughness, there is a slight influence between substrate roughness and adhesive force, but it does not seem to show a large trend.

Table II: Critical forces, L_{c1}, L_{c2} and L_{c3} of coated SS304L on 0.5, 1, 1.5 kW sputtering energy with different surface roughness SS316L by using PVD method

Power	Surface polishing, Roughness	L_{c1}	L_{c2}	L_{c3}
0.5 kW	6μm dialube Ra: ~ 7 nm	3.6 N	6.9 N	7.7 N
	600 grit Ra: ~ 45 nm	10.5 N	13.1 N	13.6 N
	60 grit Ra: ~ 250 nm	10.3 N	> 30 N	> 30 N
1.0 kW	6μm dialube Ra: 4 nm ~ 8 nm	> 30 N	> 30 N	> 30 N
	600 grit Ra: 25 nm ~ 60 nm	21.1 N	> 30 N	> 30 N
	60 grit Ra: 200 nm ~ 300 nm	> 30 N	> 30 N	> 30 N
1.5 kW	6μm dialube Ra: 4 nm ~ 8 nm	10.4 N	16.9 N	> 30 N
	600 grit Ra: 25 nm ~ 60 nm	11.9 N	24.9 N	28.1 N
	60 grit Ra: 200 nm ~ 300 nm	10.4 N	19.0 N	> 30 N

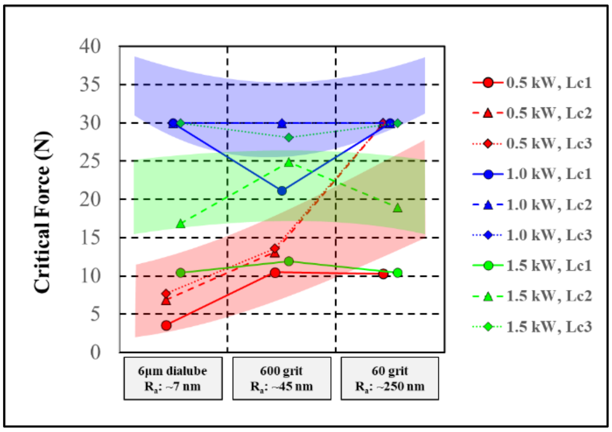

Fig. 6. Critical forces, L_{c1}, L_{c2} and L_{c3} for changes in substrate roughness and sputtering power with its schematic range

4. Conclusion

In order to prevent severe corrosion of components like steam generator by lead/LBE coolant in LFR, it is important to develop coating technology of corrosion resistance material on the structural materials. PVD method, which is powerful tool to apply coating, is cheap, simple and have large field experiences. Appling alloys as corrosion resistance materials for coating, however, have been not studied in PVD research field. Therefore, this research conducted the alloy PVD process with coated SS304L on SS316L substrate and evaluated mechanical properties like roughness and adhesive force of coated material by controlling sputtering power and roughness of the SS316L substrate.

1. The roughness of coated layer is closely related to the substrate roughness than sputtering power.
2. Adhesive force is greatly affected by sputtering power, and it seems to have a tendency of increase as the power increases, but to decrease when it exceeds a certain power. The relationship between adhesive force and substrate roughness did not show a noticeable relationship.

Table III: Relevance of PVD process parameters and coating characteristics

		Coating characteristic	
		Coating roughness	Adhesive force
PVD process parameter	Substrate roughness	**Closely related**	Little relevance
	Sputtering power	Little relevance	**Closely related**

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