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Erosion behavior of Supercritical Nano-plating Nickel on Copper-Zinc Substrate

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Abstract: Erosion behavior of Supercritical Nano-Plating (SNP) material was studied, especially focused on the dependence of the tilting angle between plate surface and blasting direction. Tilting assembly was installed in the commercially available blasting machine which is used for drawing graphical pattern on glass tableware by powder blasting. Damaged surface and cross-section were observed with optical microscope and scanning electron microscope. Normal and tangential force measurement unit was introduced for the measurement during erosion tests. Superiority of SNP was confirmed as thinner controllable plating of higher hardness with excellent integrity against powder-blasting erosion, compared with conventional method. Tangential force during erosion test was not measured successfully but measured normal force showed good agreement with theoretical expectation. It would be suggested that better understanding of the damage process during powder-mixed blasting needs the detailed research on powder impact behavior itself.

Keywords: supercritical nano-plating, erosion, nickel coating, silica grass powder

1. INTRODUCTION

Supercritical Nano-Plating (SNP) method seems to be one of breakthrough key technologies for sustainable earth environment.

SNP coatings are reported to have excellent characteristics[1]-[5], such as fine grain size, higher hardness and also defect-free, which have great potential to minimize the amount of raw materials and apply low-cost materials for high functional surface coatings. Further more, SNP method uses supercritical CO_2 very effectively so that the volume of waste fluid could be as little as possible by gasification returning from supercritical condition at higher temperature under severe pressure to the normal condition at room temperature under atmospheric pressure, and also higher dissolubility eliminates washing/rinsing process and cuts off the expense for such purpose[6]-[8].

Researches in chemical field for SNP processing are carried out energetically, but those for mechanical characteristics are not provided much.

In this study, erosion behavior of nickel SNP-coating on copper-zinc substrate was experimentally observed to discuss the effect of the tilting angle between plated surface and blasting direction, such as 15/30/45/60/75 degree (0° is normal to the blasting direction). Damage process of erosion and key factors controlling that process will be discussed.

2. SNP PROCESS USED FOR SPECIMEN

Supercritical electroplating was carried out for 15minites under the condition, such as a current density of 10A/dm^2, a temperature of 323K and a pressure of 10MPa, in a constantly agitated ternary system of supercritical CO_2 (15ml, minimum purity of 99.9%), the electroplating solution (30ml) and the surfactant (3wt% to the electroplating solution) as shown in Fig.1[9]. Pressure vessel of stainless steel was used for electroplating where magnetic agitator could work. Pressure vessel was placed in a heating chamber to control the temperature. CO_2 was introduced with the help of compressor.

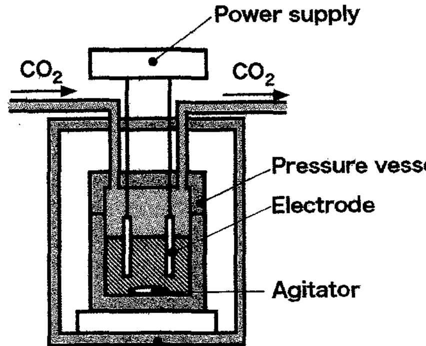

Fig.1 Schematic diagram of batch reactor for SNP procedure

The electroplating solution consisted of followings; nickel sulfate 372g/l, nickel chloride 88g/l, boric acid 95g/l, saccharin 3g/l and 1,4-

byutynediol 0.5g/l. As the surfactant, the non-ionic surfactantocta-(ethylene oxide)-dodecyl-ether, $H(CH_2CH_2O)_8O(CH_2)_{12}$, was selected. The anode was a pure nickel plate (purity 99.99%) of 15mm x 22mm. Conventional electroplating was also carried out for reference plating specimens, for 15minites under the similar condition, such as a current density of $10A/dm^2$, the room temperature and the atmospheric pressure, with just the electroplating solution of same compsitions for supercritical electroplating.

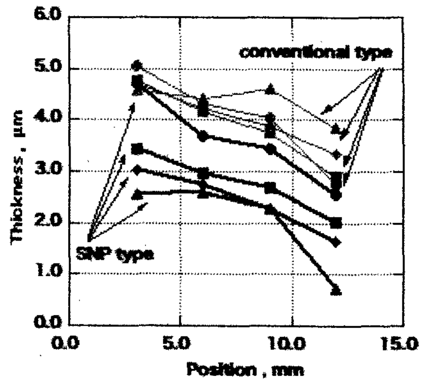

Fig.2 Thickness variation along center line

Typical thickness variation along the center line from the far end of the wired plate was shown in Fig.2. This results implies that thinner plated layer would obtained easily with SNP procedure.

Average value of micro-Vickers hardness is around HV610 for SNP-coating, and 450 for conventional-coatings, where the hardness of Cu-Zn substrate is HV110. This results prove the excellent higher hardness of SNP-coating.

3. EROSION TEST

3.1 Erosion test machine and testing condition

Erosion test was carried out with a commercially available blasting machine. Specimen was set on the specimen stage in Fig.3 to measure the normal and tangential load during erosion test. Specimen was glued to the horizontal spring plate of phosphor bronze to bend smoothly for measurement of normal direction load. The horizontal spring plate and square-shape "load-transfer frame" were supported by the perpendicular spring plate to move horizontally and the tangential load cell was placed to press the horizontal moving unit to detect the tangential force acting on the specimen.

This specimen stage was assembled in the cradle to tilt the specimen against blasting. The cradle can be set at tilting angles of 0, 15, 30, 45, 60, 75 degree (0° is the condition that the specimen surface is normal to the blasting direction)

Powders mixed into the blasting air was glass of HV600, polygon shape and 0.1mm diameter.

Blasting intensity was predifined by the impact diameter by changing the compressor pressure valve as 0.2MPa. The protection plate was placed over the specimen stage to protect sensors and sliding joints from blasting powder interference, and then there was the window of 15mm x 15mm where powders could pass through. for this experiment is around 10μm for both coatings at the center point of specimen.

Silica glass powder was selected as blasted powder, whose characteristics are HV600, polygon shape, and 0.1mm diameter. Blasting condition is 0.2MPa at the compressor pressure gage, 40g of total blasting amount. When the stage was tilted as *q* degree in Fig.4, the amount of powder passing through the window becomes $cos\theta$ times, and the blasting period should be set longer as $1/cos\theta$ times.

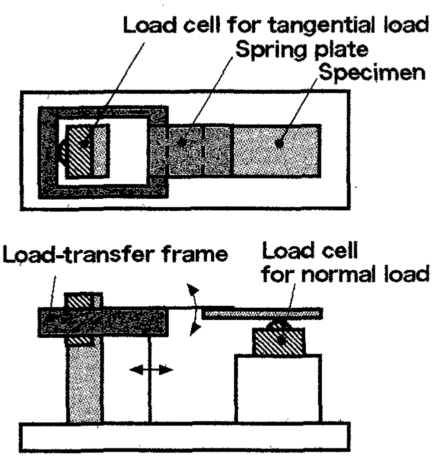

Fig.3 Schematic diagram of specimen stage.

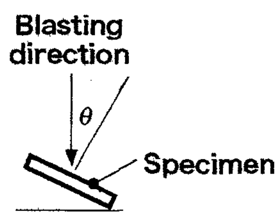

Fig.4 Definition of tilting angle θ

3.2 Surface observation and wear ratio evaluation

The blasted specimens were observed by optical microscope first, and cut and polished to observe the cross-section by scanning electron microscope. Using the photos for blasted area and unblasted area, plated area was measured to calculate "wear ratio" as the ratio of blasted area to unblasted area.

4. RESULTS AND DISCUSSIONS

4.1 Surface damage in macroscopic level

Observed images by optical microscope were summarized in Fig.5. It is clearly found that SNP type showed higher integrity against blasting than conventional type. One thing should be noted that monotonic change was not observed for conventional type then there was a possibility of low reproductivity in this result shown as Fig.5.

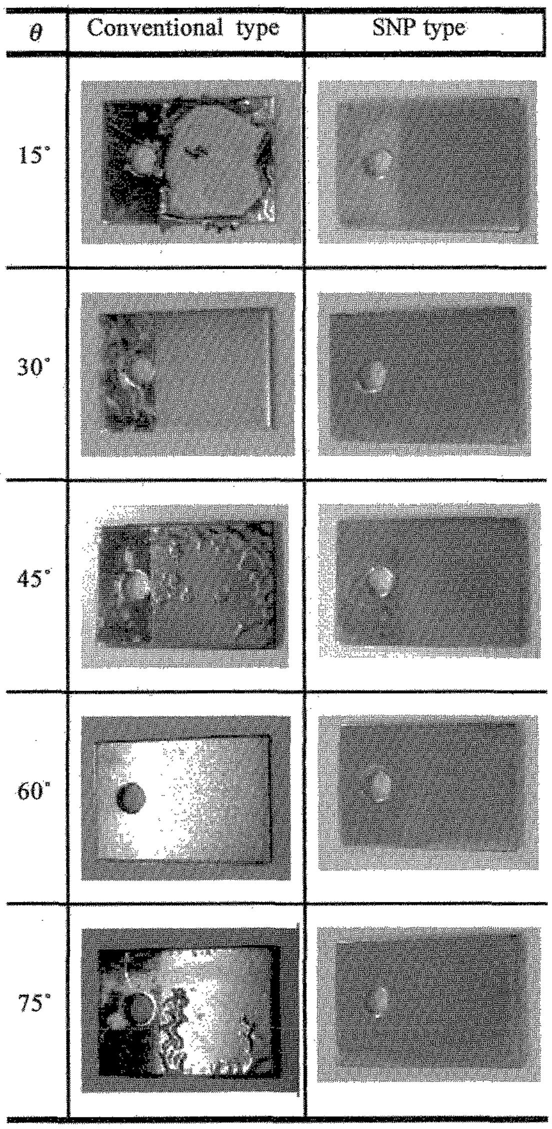

Fig. 5 Surface damages after erosion test (specimen size is 15mm x 22mm)

4.2 Measured normal force and wear ratio

Measured normal force is shown in Fig.6 with a theoretical calculation curve.

The assumption for the curve is as follows; 1)measured normal load is acting on specimen of given area, 2)normal component of blasting force is $F\cos\theta$, where F is total load acting over specimen when blasting is normal to surface, 3)by tilting of protection window in front of specimen, of area S, the effective blasting window size become $S\cos\theta$ and the blasting period becomes $1/\cos\theta$ times long to keep the total blasted amount as same.

From 2) and 3), theoretical normal component is given as $F\cos^2\theta$, and then the theoretical curve is drawn passing at the data point of θ=0 degree.

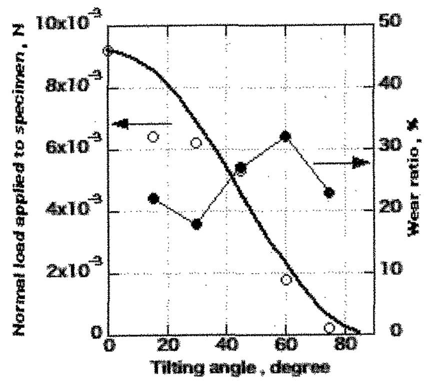

Fig.6 Tilting angle θ dependence of normal load and wear ratio

Theoretical curve seems to have fairly good agreement with experimental results, but here is a question; by what tangential component $F\cos\theta\sin\theta$ is carried?

If shear force generated by viscosity behaves as an intensity of that value as $F\cos\theta\sin\theta$, it would be inconsistent with the simple dependence of geometry, not including viscosity.

With the same analogy as the dynamic theory of gas [10], powders mixed in blasting air could be the candidate, but that should be equal to the friction force and similar inconsistency happens.

One of the helpful information should be the measured tangential force, but the measurement has not be succeeded at this moment. This problem is under consideration to be solved.

From here, friction coefficient treatment will be applied whether the results can be explained.

Assuming constant friction coefficient, tangential force acting over the surface is rational to the calculated normal load $F\cos^2\theta$. As mentioned former, blasting period is $1/\cos\theta$ times long and the amount of wear should be rational to $F\cos\theta$. Comparing that result with wear ratio in Fig.6 obtained by the cross section observation for both blasted area and unblasted area, such simple treatment would be insufficient to explain the wear behavior.

Near normal blasting, powders indent the plated layer into the substrate and bulging would be the main mechanism of damage. Near parallel blasting, powders scratch the plated layer surface and both scratching and shearing would accelerate the damage. It would be needed to consider the impact behavior to understand these.

5. CONCLUSIONS

Erosion behavior of Supercritical Nano-Plating, which provides fine grain size and defect free plating, is studied by changing the tilting angle in the commercially available blasting machine.

Conclusions are as follows;

1)thinner plating layer is obtained under the same current density and the same processing period,

2)micro-Vickers hardness is highly improved,
3)higher integrity is obtained against blasting.
Further tasks are also found that,
1)impact process should be analyzed,
2)tangential measurement device should be modified.

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