Fundamental Studies on the Copper and Nickel Electroplating Methods of Stress Analysis*
(Effects of the Ground Plating on the Proper Stress)

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Effects of the ground plating of alkaline copper on the proper stress in the acid plating of copper or nickel are studied for the purpose of further development of a direct plating method and its application to the foil strain gauge. By using the initial deposited surface and final deposited one of the plating foil as the cementing surface, the difference in occurrence of grown grains and flecks in the foil is inspected. The results are as follows: (1) In the copper plating method, the ground plating is unnecessary for both the direct plating and the foil. (2) In the ground plating method, the ground plating is necessary for the plating on steel specimens, but is unnecessary for the foil. (3) Flecks observed on the final deposited surface are slip bands produced in the grown grains. (4) If the final deposited surface of the nickel foil is utilized for the cementing surface, the proper stress of the foil gauge lowers and it is available for the practical use.

Key Words: Experimental Stress Analysis, Copper Electroplating Method, Nickel Electroplating Method, Direct Plating Method, Plating Foil Strain Gauge

1. Introduction

In the copper and nickel electroplating methods of stress analysis\(^{(D-w)}\), the plating which consists of two layers, namely the ground plating of alkaline copper and the upper one of acid copper or nickel, is usually used. When a specimen coated with the plating is subjected to cyclic loads, the elastic stress or strain on the surface of the specimen is measured accurately by observing flecks on the surface of plating, resulting from the grain growth in the acid plating caused by the applied cyclic shearing strains. It was made clear that the grain growth occurs at the boundary between the acid plating and the ground one, and that the flecks appear just above the grown grains developed on the surface of the acid plating with an increase in the number of strain cycles\(^{(D-w)}\). Besides, the appearance of flecks was presumed to be due to oxidation of the surface of the acid plating and many fine stripes in the direction of maximum shear were observed in the flecks\(^{(D)}\). However, effects of the ground plating on the occurrence of grown grains or flecks in the acid plating have not been clarified satisfactorily. Furthermore, the relation between the behaviour of grown grains owing to cyclic straining and the appearance of flecks must be investigated in detail.

In this paper, two kinds of the acid plating, namely the copper plating deposited in a sulfate bath and the nickel one deposited in a sulfamate bath, are tested for the inspection of a role of the ground plating deposited in a copper cyanide bath. It is investigated whether crystal grains in the ground plating are grown by cyclic straining or not. In regard to both the direct plating deposited on a specimen and the plating foil cemented on a specimen, effects of the ground plating on the proper stress of the acid one are examined by rotary bending tests. When the final deposited surface of the plating foil is adopted as the cementing surface, the occurrence of grain growth in the foil is inspected, and some examinations concerning the mechanism of structural change in the plating due to cyclic straining are made.

2. Experimental procedure

2.1 Testing machine and test specimen

An Omo-type fatigue testing machine (3600 rpm, 98 N·m (10 kgf·m) was used for rotary bending tests, and the copper plating and nickel plating were used at room temperature and 230°C, respectively. Using drawn rods of carbon steel SK-3 (φ19 mm) annealed after heating at 800°C for one hour, tapered calibration specimens were machined as shown in Fig.1.

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2.2 Plating condition

Table 1 shows the composition of plating solution and plating conditions of alkaline copper adopted for the ground plat-ing. Tables 2 and 3 show those for the acid copper and nickel plating, respectively. Applying the plating conditions tabulated, the thickness of ground plating is about 0.02 μm, that of acid copper 10 μm, and that of nickel 5 μm. After the tapered portion of the calibration specimen was polished with 800 emery paper and then treated with calcium carbonate and dilute sulfuric acid, it was plated with one layer or two layers. When a steel specimen is immersed in the plating bath shown in Table 2 in order to receive one layer plating of the acid copper, a substitutional reaction of ions occurs immediately and coarse copper precipitates on the surface of the specimen. Since the coarse copper layer precipitated prevents the acid copper from adhering to the specimen surface, the electroplated copper on this layer is insufficient to stick to the specimen, and so flecks due to cyclic straining appear irregularly. Hence, in the usual copper electroplating methods, the copper plating consisting of two layers with an alkaline ground plating shown in Table 1 has been utilized. In this experiment, however, the acid copper plating of one layer having a good sticking with the surface of the specimen could be obtained in the following way. Voltage in a circuit was applied previously and then the electroplating was started under the condition shown in Table 2 as soon as the specimen was immersed in the plating bath.

2.3 Plating foil and its adhesion

A stainless steel plate (100x50 mm) was plated with one layer or two layers, and then a sheet of foil was made by tearing off the plating layer from the plate (5). Since the current density near the edges of the stainless steel plate became uneven throughout the plating operation, the edges of the foil were torn away to about 10 mm in width for the purpose of obtaining a foil of uniform quality, and the remaining portion was divided into a number of small pieces of a rectangular type (3x20 mm) for the tests.

An instant adhesive agent (Aron α) and a thermostetting type cement made of epoxy resin (XS-10) were used as adhesive agents for the copper and nickel foils, respectively. Here, the cementing condition of XS-10 is the same as that of the cement (XS-20) used in the previous papers (5-7). Four sheets of the rectangular type foil were used for one specimen, and they were attached to the tapered portion in the axial direction and at about regular intervals along the periphery.

2.4 Observation of flecks and grown grains

An optical microscope of 100 magnifications was used for observation of flecks and grown grains. Here, in order to observe the grown grains, the surfaces of copper and nickel plating were removed by about 1 μm in thickness by electrolytic polishing and then etched with Grard's reagent. Then, the electrolyte used was phosphoric acid for the copper plating and a mixture of perchloric acid and glacial acetic acid for the nickel plating.

In order to investigate the occurrence of grown grains more minutely, a cross section of the plated specimen or that of a specimen coated with the plating foil was observed by using an optical microscope of 1200 magnifications. The specimens cut off were embedded in a cold-setting epoxy resin, and the sections were finished by buffing and then etched with Grard's reagent for the copper plating and with an aqua regia and Grard's reagent for the nickel plating.

Table 1 Composition of plating solution and plating conditions (Alkaline copper plating)

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper cyanide CuCN</td>
<td>23 g</td>
</tr>
<tr>
<td>Sodium carbonate Na₂CO₃</td>
<td>10 g</td>
</tr>
<tr>
<td>Sodium cyanide NaCN</td>
<td>30 g</td>
</tr>
<tr>
<td>Distilled water H₂O</td>
<td>1 L</td>
</tr>
<tr>
<td>Bath temperature</td>
<td>30 °C</td>
</tr>
<tr>
<td>Plating duration</td>
<td>1 min</td>
</tr>
<tr>
<td>Current density</td>
<td>120 A/m²</td>
</tr>
<tr>
<td>Bath voltage</td>
<td>1.4-1.5 V</td>
</tr>
</tbody>
</table>

Table 2 Composition of plating solution and plating conditions (Acid copper plating)

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper sulfate CuSO₄·5H₂O</td>
<td>250 g</td>
</tr>
<tr>
<td>Sulfuric acid H₂SO₄</td>
<td>80 g</td>
</tr>
<tr>
<td>Distilled water H₂O</td>
<td>1 L</td>
</tr>
<tr>
<td>Bath temperature</td>
<td>23 °C</td>
</tr>
<tr>
<td>Plating duration</td>
<td>20 min</td>
</tr>
<tr>
<td>Current density</td>
<td>300 A/m²</td>
</tr>
<tr>
<td>Bath voltage</td>
<td>0.5 V</td>
</tr>
</tbody>
</table>

Table 3 Composition of plating solution and plating conditions (Nickel plating)

<table>
<thead>
<tr>
<th>Component</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel sulfamate Ni(NH₄SO₄)·4H₂O</td>
<td>300 g</td>
</tr>
<tr>
<td>Nickel chloride NiCl₂·6H₂O</td>
<td>30 g</td>
</tr>
<tr>
<td>Boracic acid H₃BO₃</td>
<td>30 g</td>
</tr>
<tr>
<td>Distilled water H₂O</td>
<td>1 L</td>
</tr>
<tr>
<td>pH</td>
<td>4.8</td>
</tr>
<tr>
<td>Bath temperature</td>
<td>50 °C</td>
</tr>
<tr>
<td>Plating duration</td>
<td>10 min</td>
</tr>
<tr>
<td>Current density</td>
<td>250 A/m²</td>
</tr>
<tr>
<td>Bath voltage</td>
<td>1.4-1.6 V</td>
</tr>
</tbody>
</table>
Here, in order to facilitate the microscopic observation of the plating section, the copper plating and the nickel one were increased in thickness to about 20 \( \mu \)m and 15 \( \mu \)m, respectively, by extending each plating duration.

2.5 Measurement of the proper stress

The tapered specimen shown in Fig.1 is electroplated directly or coated partially with rectangular foils, and it is subjected to rotary bending under an assigned number of cycles \( N \). If the distance \( S \) from one end of the tapered portion to the location where flecks or grown grains begin to occur is measured under a bending moment \( M \) and then the diameter \( d \) of the location is determined, the proper stress \( \sigma_p \) in tension-compression of the plating is given by

\[
\sigma_p = \frac{32M}{\pi d^3}
\]  

(1)

The distance \( S \) was determined as an average of the values measured at four locations quartering the periphery of the cross section. The deviation of each measured value from the average one was within 0.5 \( \mu \)m and it was less than 2.0 MPa(0.2 kgf/mm\(^2\)) in terms of stress.

3. Effects of the ground plating on the proper stress

3.1 Measurement of the proper stress in the ground plating

It was inspected whether the proper stress on the basis of flecks could be obtained or not, when the ground plating of alkaline copper shown in Table 1 was subjected to cyclic strain at room temperature. Here, the rotary bending tests at \( N=3\times10^6 \) were carried out for the plating with various thicknesses deposited by changing the plating duration. First, steel rods (Φ8 mm) were plated under a constant current density \( D_k=120 \text{ A/m}^2 \) and by changing the plating duration \( t \), and the thicknesses \( \delta \) of the plating were measured by observing the cross section of the plated rod with an optical microscope of 400 magnifications. Figure 2 shows the results. In the figure, a solid line indicates the value calculated by Faraday’s law, and the difference between the calculated value and the measured one is based on hydrogen precipitated on the surface of a cathode specimen. From the results shown in the figure, the relation between \( \delta (\mu\text{m}) \) and \( t (\text{min}) \) is given by

\[
\delta = 0.2t
\]  

(2)

Figure 3 shows the relation between the proper stress \( \sigma_p \) and the thickness \( \delta \) of the ground plating. Figure 4 shows an example of the appearance of flecks in the ground plating. It is presumed from these results that the grain growth owing to cyclic strain occurs also in the alkaline copper plating tested, and so it is found that the proper stress of the ground plating can be obtained by the observation of flecks. The \( \sigma_p \)-value, however, is much higher than that in the usual copper plating method using a plating of two layers, as mentioned later. Besides, in the case of the plating of two layers, the thickness \( \delta \) of the ground plating is only 0.2 \( \mu \)m from Eq.(2) and the value of \( \sigma_p \) at \( \delta=0.2 \mu \)m becomes so high that it can not be obtained as shown in Fig.3. Consequently, it may be said that a structural change in the ground plating does not occur at the
stress acting upon the usual copper plating of two layers, when it is used at room temperature.

3.2 Measurement of the proper stress in the acid copper plating

Figure 5 shows the $\sigma_p-N$ curves of the acid copper plating (thickness: about 10 $\mu$m) shown in Table 2. In the figure, a solid line and a broken line indicate the results of the direct plating and the copper foil, respectively. It is found from the figure that in both the direct plating and the plating foil, the $\sigma_p-N$ curve of one layer almost coincides with that of two layers. However, the $\sigma_p$-value of the copper foil is slightly higher than that of the direct plating owing to existence of an adhesive layer. Figure 6 shows appearances of the flecks initiated in both the direct plating (a), (b) and the copper foil (c), (d), consisting of one layer and two layers. Here, these flecks are produced at a higher cyclic stress than the proper stress. It is found from the figures that the appearances of flecks in each case are almost identical regardless of the existence of the ground plating. From the results obtained above, it may be said that the acid plating of one layer alone is satisfactory for both the direct plating and the plating foil in the copper electroplating method and the ground plating of alkaline copper is not always necessary.

3.3 Measurement of the proper stress in the nickel plating

3.3.1 Proper stress in the direct plating

Effects of the ground plating on the proper stress of the direct plating of nickel (thickness: about 5 $\mu$m) were inspected at the test temperature $T=230^\circ$C. The proper stress was obtained by observing flecks on the nickel plating. Here, the thickness $\delta$ of the ground plating was varied in the range of 0-0.2 $\mu$m on the basis of Eq.(2). The results are shown in Fig.7. It is found from the figure that the $\sigma_p$-value at each $N$ is almost constant in the range of $\delta=0.03-0.2$ $\mu$m, but it increases sharply with a decrease in $\delta$ within the range of $\delta<0.03$ $\mu$m. It is considered that this is mainly due to the coherence of nickel deposits on the surface of the steel specimen. Namely, in the case of the nickel plating of one layer ($\delta=0$ $\mu$m), crystal lattices in the initial deposited nickel are warped and strain-hardened owing to the differences in both the crystal structure and lattice constant between the basic iron and the plated nickel, and so the coherence between them is not good. On the other hand, nickel plating sticks fast on the surface of copper because copper and nickel have the same crystal structure (f.c.c.) and both lattice constants are almost identical. Furthermore, since copper is a more ductile and less strain hardened material than nickel, a copper plating sticks fast on the surface of iron. Accordingly, when a ground copper plating is deposited in thicknesses $\delta=0.03-0.2$ $\mu$m, the coherence between the basic iron and the nickel layer is improved remarkably and cyclic strains on the surface of the steel specimen are reliably transmitted to the nickel plating. But when $\delta<0.03$ $\mu$m, the $\sigma_p$-value rises sharply and the flecks come to appear irregularly and thickly at local parts. This may be attributable to the facts that the ground plating is extremely thin and so the surface of the
specimen is not coated uniformly with the ground plating. From the results obtained above, when the direct plating of nickel is utilized for a steel specimen to measure the proper stress, it is effective to adopt the plating of two layers with the ground one of alkaline copper. The suitable thickness of the ground plating is in the range of 0.1–0.2 μm for the convenience of plating operation.

3.3.2 Effects of the plating thickness on the proper stress

When the direct plating of nickel with a ground copper plating of 0.2 μm in thickness was utilized, effects of the thickness of the nickel plating on the proper stress was examined at T=230 °C. Here, the thickness h of the nickel plating was in the range of 2.5–10 μm and was determined by controlling the plating duration on the basis of Faraday’s law under a constant current density D_p=250 A/m². The results are shown in Fig.8. Here, solid lines and broken lines indicate the Q_p-values obtained by observing flecks and grown grains, respectively. Figure 9 (a), (b), (c) show appearances of flecks and grown grains at h=3×10⁶. Here, the flecks and grown grains shown in these figures are produced at higher cyclic stresses than the proper stresses shown in Fig.8. As shown in Fig. 8, the Q_p-values obtained by grown grains are uniformly lower than those obtained by flecks when h is constant. This is explained by the following reasons: When the plating is subjected to cyclic strain of a certain amplitude, crystal grains of the nickel deposits contacting with the ground plating begin to grow while joining adjacent grains, and they develop with an increase in the number of cycles. Finally, when the grown grains develop on the plating surface, flecks begin to appear. It is found from the figure that the Q_p-value becomes minimum in the range of h=3–7 μm when h<7 μm, the Q_p-value becomes larger and the contour of flecks becomes indistinct as shown in Fig.9. This may be attributable to the fact that it becomes difficult for grown grains to develop on the surface with an increase in h. When h>4 μm, clear outline flecks appear, resulting from a marked rising of the Q_p-value. It is considered that this depends upon the growing process of grains. Namely, the grains grow in the lateral direction first while joining the adjacent grains and then gradually develop in the longitudinal direction with an increase in the number of strain cycles, and finally thus grown-up grains attain the surface. After three kinds of platings with different initial thicknesses, namely h=2.5, 5, 10 μm, were subjected to the cyclic stresses of nearly equal amplitude by N=3×10⁶, respectively, the thicknesses of them were reduced to the final thickness h=0.5 μm by electrolytic polishing in order to compare mutually the sizes of grown grains. Figure 10 shows the appearances of grown grains obtained in this way. It is found from the figures that the grown grains in the case of h=2.5 μm are restrained from the development in the lateral direction unlike those in the other cases. Therefore, it may be considered that the grown grains in the case of a thin plating (h=4 μm) are restrained from the development in the lateral direction at an initial stage of growing and the growth up to the plating surface is suppressed, so a higher stress amplitude is needed for the occurrence of flecks. From the above results, it can be said that an effective thickness of the nickel plating is within the range of 5–7 μm, because the proper stress holds the lowest value and moreover the flecks occur invariably.

3.3.3 Proper stress in the plating foil

Effects of the ground plating on the proper stress in the nickel foil were inspected at T=230 °C. Here, thickness of the ground plating and the nickel plating were 0.2 μm and 5 μm, respectively, and the adhesive layer was about 1 μm in thickness. Figure 11 shows the Q_p-curves obtained. In the figure, a broken line indicates the result of the direct plating (two layers), and the Q_p-value in the plating foil is
higher than that in the direct plating by about 20% owing to the existence of an adhesive layer. Though the $\sigma_p$-value in the plating foil of one layer is somewhat higher than that in the plating foil of two layers, the difference between them is a little as shown in the figure. Figure 12 shows the appearances of the flecks produced in both foils. As shown in the figure, a notable difference is not found between them. Thus, it may be said that when the nickel plating is used as the plating foil, the ground plating is not always necessary. When $N<1\times10^8$, however, cracks are apt to occur in the foil of one layer, since the proper stress becomes higher.

Fig. 11 $\sigma_p-N$ curves of nickel foil
ly, it is considered that the internal strain in the platting owing to deposition is a main factor in the occurrence of grown grains.

As a difference in the appearance of the grown grains was found between the outside and the inside of foils as mentioned above, effects of the cementing surface on the proper stress were inspected, using a foil of 10 μm in thickness. The results are shown in Fig. 14. As shown in the figure, the $\sigma_p$-value in the outside of foil almost coincides with that in the inside of foil. But the phenomenon appearing on the observing surface differs between them, namely, flecks appear in the outside of foil as shown in Fig. 6 (d), while on the other hand slip bands appear in the inside of foil as shown in Fig. 13. So, in order to investigate the mutual relation between the flecks and the slip bands, the platting foil in which structural change had been produced

4. Mechanism of the initiation of flecks

Using a copper foil and a nickel one both consisting of one layer, the initial deposited surface and the final deposited one of both foils were adopted as the cementing surface for the specimen, respectively. From the test results, a mechanism of the initiation of flecks was studied and effects of the cementing surfaces adopted on the proper stress were examined. Here the foil, the initial deposited surface of which is utilized as the cementing surface, is named "the outside of foil" and the one, the final deposited surface of which is utilized as the cementing surface, is named "the inside of foil".

4.1 Examinations of the copper foil

Figure 13 (a), (b) show an example of the sectional view of grown grains at the initial stage of growth in both the outside and the inside of foils. Here, they were obtained at $N=3\times10^6$ and the thickness of the platting was increased to about 20 μm in order to facilitate the sectional view of grown grains. As shown in the figures, an initial grown grain in the outside of foil occurs at the cementing surface and that in the inside of foil occurs at the observing surface. Namely, it may be said that grown grains occur at the initial deposited layer in which the internal strain becomes larger than that in the other portion. According-
by cyclic straining was torn off from the specimen and the same position on both sides of it was observed. Figure 16 (a), (b) show an example of the appearances of slip bands and flecks at the same position of both sides of the inside of foil. As shown in the figure, both the contour and the size of slip bands on the observing surface correspond well to those of flecks on the cementing surface. Namely, it may be said that the slip band and the fleck are the phenomena on the initial deposited surface and on the final deposited one, respectively, occurring in one developed grown grain. Besides, when the final deposited surface of the foil was finished by electrolytic polishing, slip bands similar to those in Fig.15 could also be found on the observing surface of the outside of foil. Therefore, it is considered that a bundle of slip lines initiated in a grown grain may be seen like a fleck because of fine asperities existing on the final deposited surface.

4.2 Examinations of the nickel foil

Figure 17 (a), (b) show the sectional view of grown grains in both the outside and the inside of foils tested at \( T = 230\, ^\circ\text{C} \) and \( N = 3 \times 10^6 \), the thickness of foil being about 15 \( \mu\text{m} \). In both the outside and the inside of foils, the grown grains occur at the initial deposited layer where the internal strain is heavy, which is similar to the case of the copper foil. Figure 18 shows the \( \sigma_p - N \) curves of the outside and the inside of foils (thickness: 5 \( \mu\text{m} \)).
are produced on the surface of it. But the proper stress is much higher than that in the acid copper plating. Therefore, in the copper electroplating method the ground plating is not always necessary, but the acid copper plating of one layer (thickness \(\approx 10 \mu m\)) is satisfactory for both the direct plating and the plate coating.

(2) When the direct plating of nickel is utilized for a steel specimen, the ground plating is necessary for the fast cohesion between the nickel plating and the surface of the specimen. The suitable thickness of the nickel plating is in the range of 5 - 7 \(\mu m\) and that of the ground plating is 0.1 - 0.2 \(\mu m\).

(3) On the other hand, when nickel is used as the plating foil, the ground plating is not always necessary.

(4) In both the acid copper plating and the nickel one, grown grains occur at the initial deposited layer where the internal stress is heavy and they develop to the final deposited surface with an increase in the number of strain cycles. Slip-deformation occurs in these grown grains, and slip bands are observed directly on the initial deposited surface, while on the other hand, they look like flecks on the final deposited surface because of fine asperities existing on the surface.

(5) In the copper foil, the proper stress has almost the same value when both the initial deposited surface and the final deposited one are utilized as the cementing surface. On the other hand, in the nickel foil the proper stress is reduced by adopting the final deposited surface as the cementing one and it is very effective for the practical use.

5. Conclusions

In the electroplating method of stress analysis, a role of the ground copper plating in the structural changes in the acid copper plating and the nickel one is investigated, and effects of thickness of the nickel plating on the proper stress are examined. Furthermore, adopting the initial deposited surface or the final deposited one as the cementing surface of the foils of copper and nickel, effects of the cementing surfaces on the proper stress are examined and a mechanism of the initiation of flecks is investigated. The following conclusions are drawn from the results obtained.

(1) When a ground plating of alkaline copper is subjected to cyclic strain, flecks

References