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ON THE STRESS MEASUREMENT METHOD USING GRAIN GROWTH PROCESS OF ELECTRODEPOSITED COPPER FOIL

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Abstract

Using electrodeposited copper foil, the relation between the magnitude of cyclic stress and the number of cycles for the initiation of grain growth and the threshold stresses for grain nucleation and grain growth were examined. On the basis of the test results, the possibility of the stress measurement using grain growth process was investigated. The threshold stress of grain growth is remarkably lower than that of grain nucleation and the former is controlled by the amplitude of shearing stress as well as the latter. So, taking notice of the phenomenon of grain growth threshold, stress measurement can be done under lower strain amplitude compared with the electroplating method used until now.

1. Introduction

In the electroplating method of stress analysis[1], the elastic surface stress or strain of machine elements can be measured both easily and accurately by observing grown grains or flecks in the deposited layer, resulting from repeated loads acting on the elements. In this method, however, the range of measurable repeated strain is pretty narrow such as $0.7\times10^{-3}\sim1.1\times10^{-3}$ in normal strain ε and the strain sensitivity is not also quite sufficient.

In the electroplating method, the elastic local stress is conventionally measured on the basis of the calibration curve, which represents the relation between the magnitude of cyclic stress and the number of stress cycles at which the observable sized grown grains or flecks through an optical microscope of 100 magnifications begin to appear in the deposited layer. Accordingly, such sized grains must progress in grain growth process to a certain extent after completing grain nucleation process. That is, in the conventional method, cyclic strain energy must be provided indispensably for grain nucleation and furthermore for grain growth. Therefore, utilizing grain growth process alone, far more sensitive stress measurement could be done compared with the conventional method which requires both processes.

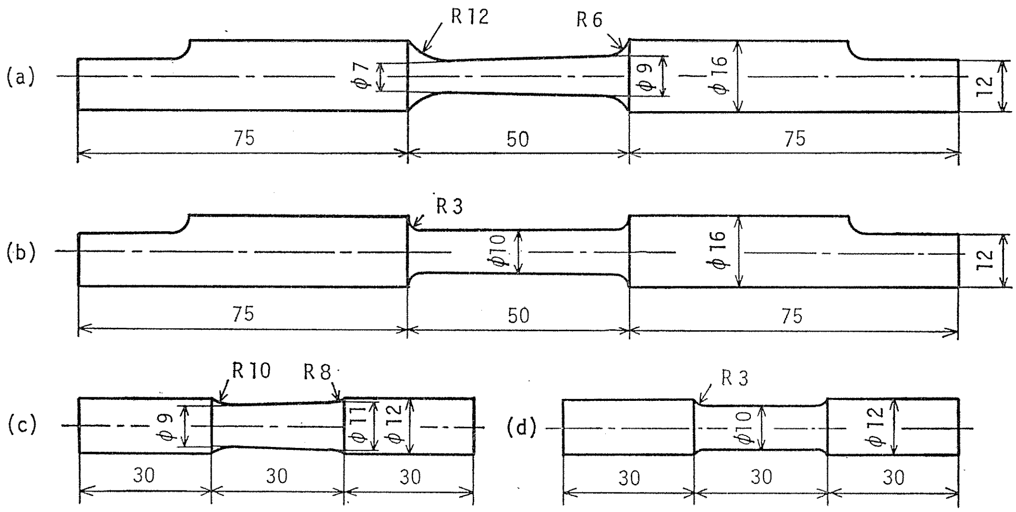
In this view point, using an electrodeposited copper foil, the relation between the magnitude of cyclic stress and the number of cycles for the completion of grain nucleation process, that is the initiation of grain growth process, was scrutinized. And the stress amplitude for maintaining grain growth was also examined.

On the basis of the results obtained, the probability of a brand-new stress measurement method utilizing grain growth process alone, which is quite different from the conventional electroplating method, was discussed to actualize far more sensitive stress measurement.

2. Testing Machine and Test Specimen

A Schenck-type and an Ono-type testing machines (both operating at 3600 rpm) were used for cyclic torsion and rotary bending tests respectively.

Drawn rods of carbon steel SK-5 (ϕ 19 mm) were machined into several types of test specimens shown in Fig. 1. The parts of them where plating foils were stuck were polished with use of emery paper ♯ 600.



(a), (b) for cyclic torsion test (c), (d) for rotary bending test

Fig. 1. Test specimens.

3. Plating Foil and Its Adhesion

A stainless steel plate polished by buffing was coated with an acid copper plating. The condition for which was shown in a previous paper[2]. Then the deposited layer was stripped from the plate to obtain a sheet of copper foil and it was split into many pieces with a rectangular form of 20 mm×2 mm. Two or three of them were stuck with use of a strain gauge cement (CC-15A) on the specimen surface at the positions equally deviding the periphery of the cross section and in the axial direction of the specimen with the initial deposited side out[2]. The copper foil was about 11 μm in thickness and the adhesive layer about 2 μm respectively.

4. Grain Nucleation and Grain Growth

When grown grains are percieved their occurrence in the deposited layer to obtain the calibration values, the fact is that they have already grown to the observable size through an optical microscope after having completed grain nucleation process. Namely, the phase of the occurrence of the observable grains means neither the moment of the real initiation of grain growth nor the real completion of grain nucleation.

In this section, the relation between the magnitude of cyclic stress and the number of cycles for the initiation of grain growth was examined taking notice of grain growth rate.

4. 1. Experimental Procedure

At first, the calibration curve for cyclic torsion (τ_p-N curve) was obtained using a tapered calibration specimen shown in Fig. 1 (a). Next, on the basis of the curve, several kinds of combinations of the initial stress amplitude τ_1 and the number of the initial stress cycles N_1 were specified and grown grains were generated under each combination using a smooth specimen shown in Fig. 1 (b). Furthermore, the test was interruptedly maintained for $3.0\times10^6\sim4.0\times10^6$ in number of cycles under the secondary stress amplitude τ_2 same as the initial one, and taking several photographs of each grain with use of an optical microscope of 400 magnifications during each interval of the test, the change in grain size with the advance of stress cycles was examined.

4. 2. Experimental Results and Discussion

Figure 2 shows the calibration curve. Each grain grows larger with an increace in number of cycles keeping an elliptical form as shown in Fig. 3.

So, both the major and minor axes of the grain were measured. As an example, Fig. 4 shows the change in size of grains of various initial grain sizes a_i, which were generated under one of the specified combinations ($\tau_1=104$ MPa and $N_1=1.2\times10^6$), with the advance of the secondary stress cycles N_2.

It was found that increace in grain size has a linearity to the number of the secondary stress cycles up to about $N_2=4.0\times10^6$. So that, the relation between

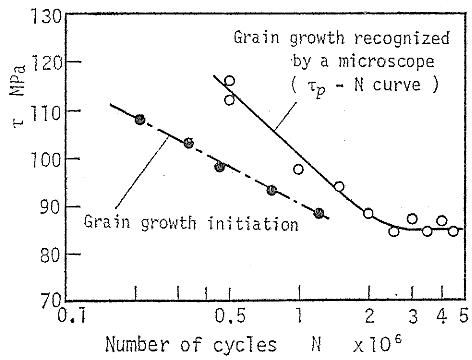
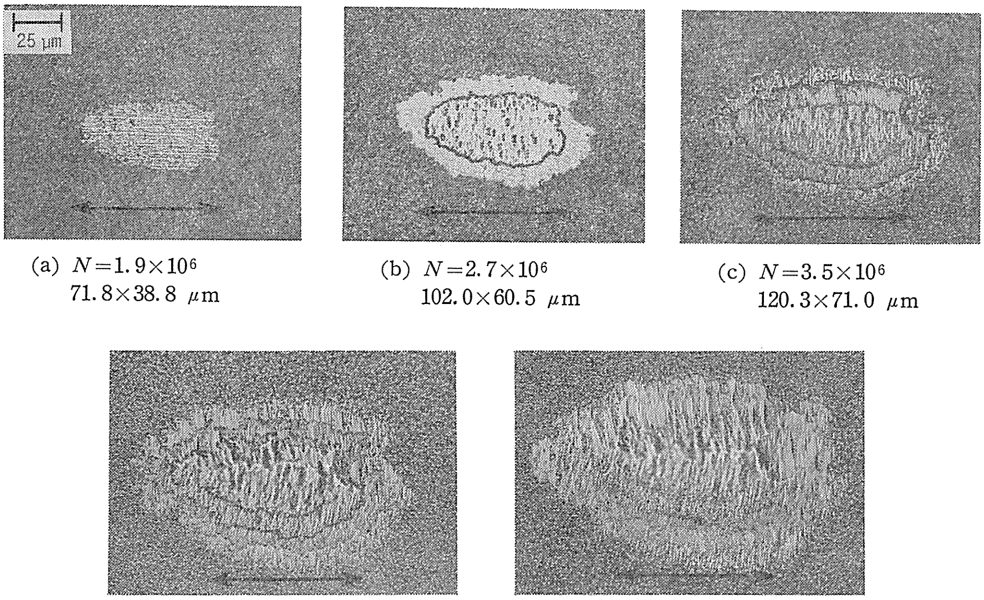


Fig. 2. Calibration curve of copper foil.



(a) $N=1.9\times10^6$ 71.8×38.8 μm
(b) $N=2.7\times10^6$ 102.0×60.5 μm
(c) $N=3.5\times10^6$ 120.3×71.0 μm
(d) $N=4.3\times10^6$ 145.5×94.0 μm
(e) $N=5.1\times10^6$ 162.3×102.5 μm

Fig. 3. State of grain growth (τ=93MPa).

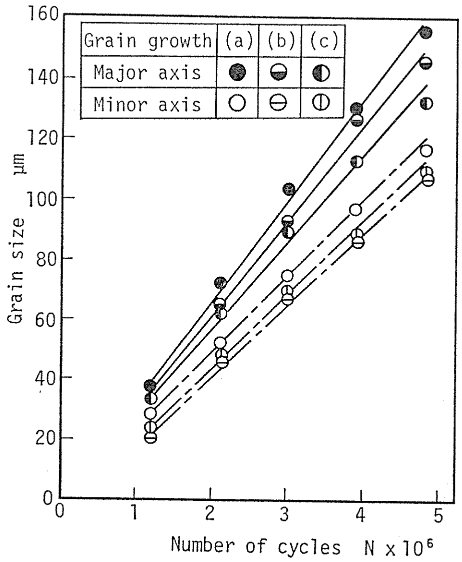


Fig. 4. Increace in grain size with the advance of stress cycles (τ=104MPa).

the initial grain size a_i and the grain size a at the number of the secondary stress cycles N_2 can be expressed by the following formula for each grain within the limits of $N_2 \leqq 4.0\times10^6$

$$a=a_i+kN_2 \tag{1}$$

where a constant k represents grain growth rate and it tends to take larger value for the grain of larger a_i.

On the basis of the above formula (1), the number of cycles at which the length of the minor axis became 0 was obtained for each grain at each stress amplitude. And the number of cycles for the initiation of grain growth was determined as an average of these values at each stress amplitude and was denoted by N_i for each stress amplitude.

Table 1 shows the values of N_i at which grain growth initiates, N_0 at which the occurrence of grains is perceived through a microscope in the calibration test, N_0-N_i, mean grain growth rate $\bar{k}$ and recognizable minimum grain size a_0 given by the following formula

Table 1. Grain growth rate and the calcurated value of the observable grain size at each stress amplitude.

Stress τ MPa	Number of cycles for grain growth initiation $N_i \times 10^5$	Number of cycles for observational occurrence of grown grain $N_0 \times 10^5$	N_0-N_i $\times 10^5$	Mean grain growth rate $\bar{k} \times 10^{-4} \mu m/cycle$	Observable grain size a_0 μm
108	2.2	6.5	4.4	0.28	12
103	3.2	8.0	4.8	0.25	12
98	4.8	10.2	5.4	0.18	10
93	7.2	14.0	6.8	0.20	13
88	12.0	20.0	8.0	0.16	13

1 MPa=0.102 kgf/mm²

$$a_0=\bar{k}(N_0-N_i) \tag{2}$$

for each stress amplitude.

The value of N_i increases successively with a decreace in stress amplitude, namely, to complete grain nucleation process, a larger number of stress cycles is required at a lower stress amplitude, which is restricted above the threshold of the calibration curve. The value of N_0-N_i shows stress amplitude dependence as well as N_0. In correspondence with this, the value of $\bar{k}$ decreases with stress amplitude. And the calculated values of a_0 by the formula (2), taking 10~13 μm in length, are in good agreement with the grain size usually adopted in the calibration test (10 μm). And this result supports the propriety of the calculated values of N_i and k.

5. Stress Measurement Taking Notice of the Grain Growth Rate

As mentioned in the above section, grain growth rate was dominated by the magnitude of cyclic stress and grain growth was still maintained under the stress near the threshold of the calibration curve. So in this section, the relations among stress amplitude, grain growth rate and the initial grain size were investigated. And the probability of the stress measurement method utilizing grain growth rate of individual grain was examined.

5. 1. *Experimental Procedure*

Copper foils in which grown grains were appropriately generated under the initial stress of the specified conditions were subjected to various cyclic stresses lower than the threshold stress of the calibration curve as the secondary stresses. And the state of grain growth was observed for each grain.

5. 2. *Experimental Results and Discussion*

Figure 5 shows the relation between the initial grain size a_i and grain growth rate k, plotted for the secondary stress amplitude τ_2 as parameter. Grain growth is maintained under τ_2=59 MPa and 69 MPa, which are lower than the threshold

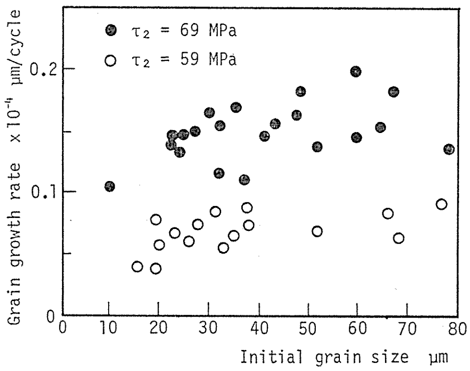


Fig. 5. The relation between the initial grain size and grain growth rate.

stress of the calibration curve. Namely, the cyclic stress amplitude required to maintain the growth of grains once appeared in the deposited layer is less than the minimum stress amplitude required to generate grain nucleation.

In spite that growth rate shows stress amplitude dependence as well as the case shown in Table 1, it doesn't take unique value for the same a_i and τ_2. Namely, growth rate is not dominated so strongly by the initial grain size as by stress amplitude. So, it is difficult to carry out an accurate stress measurement utilizing growth rate of individual grain, namely, referring the measured value of growth rate of a grain with an optional grain size to the calibration values for various initial grain sizes.

As shown in the figure, however, growth rate shows obvious stress amplitude dependence. So, taking notice of the mean growth rate of plural grains, the stress might be estimated rather accurately.

Furthermore, the dependence of growth rate on the initial grain size and its dispersion tend to reduce with a decreace in the secondary stress amplitude. And they might become negligible at the threshold of grain growth. Namely, the threshold stresses of growth of grains with various initial grain sizes would coincide with one another. So, the probability of the stress measurement taking notice of the threshold of grain growth was examined in the next section.

6. Stress Measurement Taking Notice of the Threshold of Grain Growth.

6. 1. Experimental Procedure

Cyclic torsion tests and rotary bending tests were carried out to the copper foils in which grown grains were generated in the foregoing way. And the threshold stress of grain growth was sought by varying the secondary stress amplitude stepwise with the specified number of cycles $N=3.0\times10^6$.

In each grain, when an increace over 2.5 μm in length of either axis was perceived, the grain was regarded as "grown". And at each stress amplitude, when more than a half of the observed grains had grown, it was defined "grown" at the stress and "not grown" in the case of less than a half. And the threshold stress of grain growth was determined as an average of the stresses corresponding to "grown" and "not grown".

6. 2. Experimental Results and Discussion

The threshold stress of grain growth for cyclic torsion is shown in Table 2 and Table 3 for rotary bending respectively. The fraction in the column of "State

Table 2. The threshold stress of grain growth (cyclic torsion).

Primary stress τ_1 MPa	Secondary stress τ_2 MPa	State of grain growth	Threshold stress τ_{th} MPa
88	52	18/20	49
	50	10/20	
	47	8/20	

Table 3. The threshold stress of grain growth (rotary bending).

Primary stress σ_1 MPa	Secondary stress σ_2 MPa	State of grain growth	Threshold stress σ_{th} MPa
176	107	17/23	101
	103	24/29	
	99	6/27	

of grain growth" in each table indicates the ratio of the number of "grown" grains to the total number of grains. It is obvious from the results that the threshold stress of grain growth (τ_{th}=49 MPa) is remarkably lower than that of grain nucleation(τ_p=88 MPa). And it was also found that this stress little depends on a_i.

The maximum shearing stress amplitude at the threshold of grain growth equals to τ_{th} in cyclic torsion and 1/2 σ_{th} in rotary bending respectively and as is seen in the table, they are in good agreement. Namely, the threshold stress of grain growth as well as grain nucleation is dominated by the maximum shearing stress amplitude acting on the foil.

Accordingly, taking notice of the threshold of grain growth and adopting the method mentioned above, it is possible to carry out far more sensitive stress measurement than the conventional method utilizing the calibration curve.

7. Conclusions

The possibility of the stress measurement utilizing grain growth process was discussed in this paper. Using an electrodeposited copper foil, the relation between the magnitude of cyclic stress and the number of cycles for the initiation of grain growth, and the dependence of grain growth rate on stress amplitude were investigated. On the basis of the test results, a stress measurement method taking notice of the threshold of grain growth was developed.

The following conclusions are drawn from the results obtained.

(1) The number of cycles for the initiation of grain growth increases with a decreace in cyclic stress amplitude and the threshold stress of the calibration curve is the minimum value of cyclic stress to generate grain nucleation.

(2) The relation between the initial grain size a_i and the grain size a at the number of cycles N_2 is expressed by the following formula for each grain within the limits of a certain N_2

$$a = a_i + kN_2$$

where k is grain growth rate and dominated by both cyclic stress amplitude and the initial grain size of the grain.

(3) The values of k don't necessarily agree among the grains of the same a_i.

(4) Accordingly, it is difficult to carry out an accurate stress measurement utilizing growth rate of individual grain.

(5) More than a certain value of shearing stress amplitude peculiar to the foil is required to maintain grain growth. Namely, grain growth has its threshold stress.

(6) The threshold stress of grain growth is remarkably lower than that of the calibration curve, which is the threshold of grain nucleation, and it is independent of a_i.

(7) Accordingly, taking notice of the threshold of grain growth, it is possible to carry out a stress measurement under much lower stress amplitude compared with the conventional method on the basis of the observation of the generation of grown grains.

8. References

1) Ohkubo, H., The Copper Electroplating Method of Stress Analysis (in Japanese), (1965), Asakura Shoten.
2) Seika, M., et al., Bull. JSME, Vol. 25, No. 209 (1982-11), p. 1653.