# Secular Change after Cold-Working and Anneal-Hardening of α Brass



| 著者 | IZUMI Osamu, YOSHIKI Tadatsugu |
|---|---|
| journal or publication title | Science reports of the Research Institutes, Tohoku University. Ser. A, Physics, chemistry and metallurgy |
| volume | 11 |
| page range | 201-209 |
| year | 1959 |
| URL | http://hdl.handle.net/10097/26916 |

# Secular Change after Cold-Working and Anneal-Hardening of $\alpha$ Brass*

Osamu IZUMI and Tadatsugu YOSHIKI

*The Research Institute for Iron, Steel and other Metals*

(Received March 6, 1959)

**Synopsis**

The secular change after cold-working and anneal-hardening of 70/30 brass was measured by the hardness and the spring test. An increase in hardness and a decrease in bending deflection were observed at room temperature after cold-working, especially in high reduction specimens. As in the previous work on 60/40 brass, the change after anneal-hardening of 70/30 brass was influenced by the length of time from final cold-rolling to heat-treatment, and by the heating time of anneal-hardening. In general, the longer the times, the less the change. Heat-treatment was carried out at 200°C for 15 min～3 hrs after the speciments had been allowed to remain at 40°C for various durations following the final cold-rolling.

## I. Introduction

The phenomenon of the so-called anneal-hardening is brought about when a brass plate was annealed at temperatures below the recrystallization temperature after cold-working (low-temperature annealing), and as well-known, this is utilized for improving its spring property. Brass is, however, accompanied by secular change phenomena, that is, its mechanical property changes with the time not only after cold-working, but also after the treatment of low-temperature annealing. Therefore, sometimes it is a grave defect according to the purpose of use. Accordingly, it is important from the practical point of use to establish preventive measures.

Secular change is, like anneal-hardening, a problem which has been studied as one of the anomalies observed in copper alloys, and although its cause and mechanism have considerably been investigated, the question still remains partly unsolved.(1),(2),(3) One of the present authors has researched into the phenomenon of secular change to improve spring materials of 60/40 brass series, and found that the length of time from final cold-rolling to heat-treatment (degrees of secular change after cold-working) gave influence to the secular change after anneal-hardening.(1) In the present case, the secular change after cold-working and anneal-hardening of cold-rolled 70/30 brass was examined by hardness and spring tests, that is, the ready-to-finish grain size, the working raduction, the

* The 937th report of the Research Institute for Iron, Steel and Other Metals. Reported in Japanese in the J. Japan Inst. Met., **22** (1958), 158.

(1) O. Izumi and M. Kawasaki, Sci. Rep. RITU, **A9** (1957), 32.
(2) M. Kawasaki, Metals, **26** (1956), 875. (in Japanese)
(3) G. Shinoda, J. Japan Inst. Met., **20** (1956), A-55.

length of time from final cold-rolling to heat-treatment, the time of annealing at low-temperatures were varied in many ways, and the change at the room temperature and the case in which the chaging velocity was accelerated by keeping the spicimens at 40°C were observed.

## II. Experimental procedure

1. Specimen

Every specimen was a sheet of 70/30 brass of commercial purity, chemical composition of which is shown in Table 1. The specimens (*H, W*), with which

Table 1. Chemical composition of specimens.

| Specimen | Zn | Fe | Pb | P | Cu |
|---|---|---|---|---|---|
| H | 31.36 | 0.003 | 0.007 | 0.0002 | R |
| W | 31.21 | 0.009 | 0.020 | 0.0005 | R |
| T | 30.40 | 0.002 | 0.004 | 0.0008 | R |

the hardness change after cold-rolling was marsured, were rolled to proper thickness with the grain size being controlled in the intermediate working process.(4) By ready-to-finish annealing at 500 and 750°C, they were made into fine-gained materials (the grain size was 0.03 mm) and coarse-grained materials (the grain size was 0.3 mm), and were finished up as plates of 0.5 mm in thickness by the working reductions of 10~90 per cent. The specimen (*T*), with which the change in spring property was measured, underwent ready-to-finish annealing at 600°C for 3 hrs (the grain size was 0.110 mm) and was finished up as a plate of 0.4 mm in thickness by the final reduction of 89 per cent. Furthermore, to minimize the fluctuation of the condtions of the specimens, each specimen of standard dimension ($0.5\times10\times150\,mm^3$) was cut off perpendicularly to the rolling direction. The rolling was straight rolling.

2. Method of experiment

(i) Hardness

The micro-Vickers hardness tester (load 100 g) was used as in the previous case,(5) and the values of three directions, namely, parallel, diagonal and transverse to the rolling direction of the diagonal of Vickers' indentation, were read. The hardness was measured to see chiefly the change in the specimens kept at room temperature after cold-rolling.

(ii) Spring test

Furukawa's spring fatigue testing machitne(1) was used, and the static and the residual deflection under the constant load of maximum suaface stress ($\sigma_{max}=38.8\,kg/mm^2$) were read, the former being the deflection when the load was applied to the fixed end of cantilever, 50 mm in lengh, and the latter being that when

(4) Y. Toba and O. Izumi, Shindo Geppo (J. Japan Wrought Copper Alloys Assoc.), (1956), Nos. 3~6.
(5) O. Izumi, Sci. RITU, **A11** (1959), 120.

the load was removed. It took about half a day to finish up the specimens according with the standard size after final rolling, and after that the specimens were kept at 40°C in the isothermal heater box to accelerate the chnge.

(iii) Microscopic structure

The microscopic structure was deveoped electrolytically with orthophosphoric acid solution, and chemically with ferric chloride solution. The grain size was determined by the comparison method of ASTM.

## III. Experimental result and considerations

1. Hardness change after the final cold-rolling

The hardness change in the specimens of 10~90 per cent in the final reduction kept at room temperature fluctuated, especially in the case of low reduction, because perhaps of the micro-hardness, and further, in some cases even softening was observed. As the reduction increased, however, the tendency of hardening became striking, though at an early stage of ageing the hardness changed irregularly, and after a long ageing the hardness increased to a saturated value. This tendency almost coincides with the result obtained by Murakawa.[6] No special relation could be seen between this secular change and the condition of the specimen, like the grain size and impurities. The result of the measurement is shown in Figs. 1 and 2. Fig. 1 shows the secular change in hardness in the

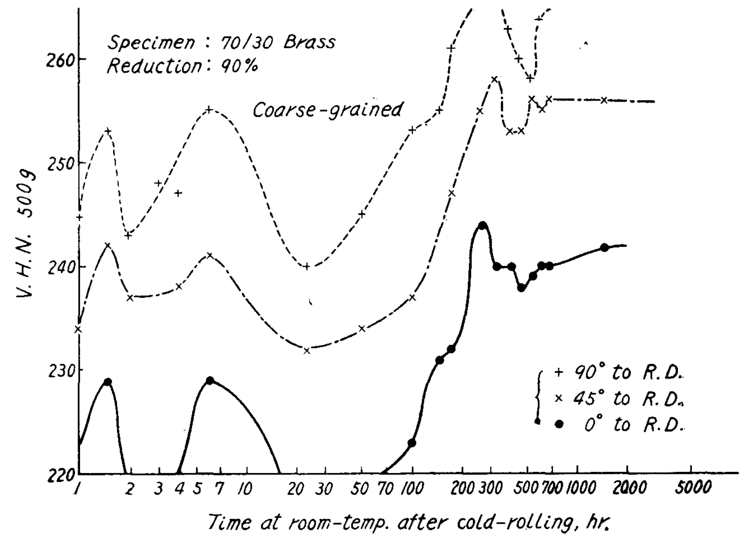


Fig. 1. Secular change in hardness of cold-rolld 70/30 brass sheets, Coarse-grained specimen, 90% reduction, kept at room-temp.

three directions of the specemen *W* cold-rolled by 90 per cent, while Fig. 2 shows the change in the diagonal direction of the specimen *H* cold-rolled by 10~90 per cent. Because perhaps of different materials, they differ in the minute details from each other, but the general tendency of the increase in hardening in highly reduced materials is the same. The change in the hardening after a long run never fails to be observed, irrespective of the different methods of rolling (straight,

(6) K. Murakawa, Rep. Inst. Sci. Tech. Univ. Tokyo, **7** (1953), 129.

reverse, cross), although they are omitted to state in details at present.

According to Mishima and co-workers,[(7),(8)] the secular change after cold-working of $\alpha$ brass is due to the process that the non-uniform distribution of

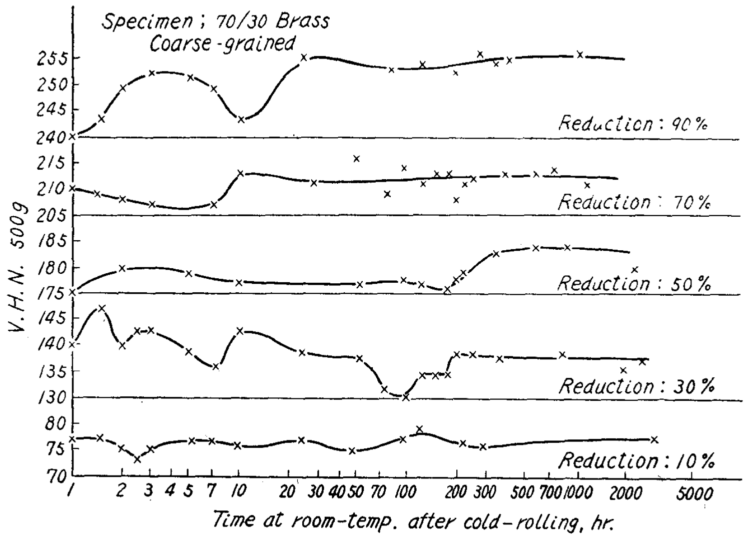


Fig. 2. Secular change in hardness of cold-rolled 70/30 brass sheets, Fine-grained specimens, 10~90 % reduction, kept at room-temp.

residual stress gradually becomes uniform, with which the hardness finally reaches somewhat lower value than that immediately after cold-working. Of course, it may naturally be acceptable that the residual stress tends to be uniform after cold-working even at room temperature. However, Mishima's insistence upon the hardness change is inconsistent with the present results. Therefore, from the relation between the change after cold-working and after low-temperature annealing, which will be mentioned later, it will be anticipated that the progress of complicated changes similar to that in anneal-hardening may take place even at room temperature after cold-working.

2. Secular changes after cold-working and after anneal-hardening

As it was difficult to see the details in the relation of these two kinds of secular change from the hardness measurement, the spring test was carried out. As already stated, the secular change after cold-working was more striking in the highly reduced materials, and so, the specimen ($T$) cold-rolled by 89 per cent was used in this case. The deflections, both static and residual, were measured under the constant maximum surface stress of 38.8 kg/mm$^2$. Since the secular change after heat-treatment in 60/40 brass, as previously reported,[(1)] seemes to be influenced by the length of time from final cold-working to heat-treatment, the deflection measurements were also carried out in the several cases of changing the length of time above-mentioned. Furthemore, the influence of the degree of heat-treatment was additionally examined by changing the length of heating time at 200°C from 15 min to 3 hrs. Therefore, the present experiment refers not only

(7) Y. Mishima, S. Morikawa and S. Yamanouchi, J. Japan Inst. Met., **18** (1954), 543.
(8) Y. Mishima, ibid., **19** (1955), 241.

to the influence of the degree of the secular change after cold-working, but also to that of the degree of anneal-hardening on the change after heat-treatment. In order to accelerate the changes, all specimens were kept at 40°C in an isothermal heater-box.

(i) Secular change after cold-working

The secular change in spring properties after final cold-rolling is summarized in Fig. 3. The amount of deflection tended to decrease gradually with time, though some irregularities could be seen in the initial state of ageing. The

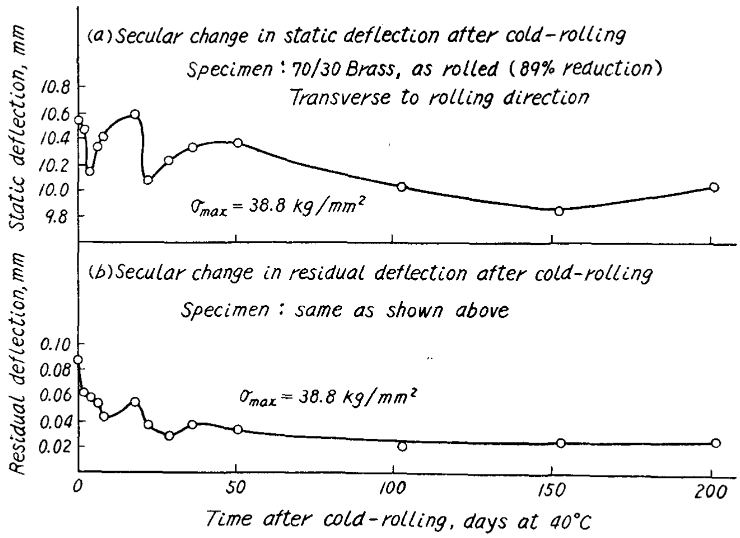


Fig. 3 (a), (b) Secular change in deflection after cold-rolling; Specimen : 70/30 brass, Reduction 89%, Aged at 40° after final cold-rolling.

residual deflection (Fig. 3(b)) finally diminished to almost a quarter in amount after aged at 40°C for more than 100 days, compared with the value of initial state. The tendency of the decrease in deflection after cold-rolling corresponds to the above-mentioned increase in hardness after cold-rolling.

(ii) The case of annealing immediately after cold-working ($T_0$ series)

The changes in deflection at 40°C, when the specimens were heat-treated at 200°C for 15 min~3 hrs immediately after cold-rolling, are shown in Fig. 4. As seen in the figure, the longer the time of annealing, the less the absolute value of initial deflection. Furthermore, the longer the time of annealing, the less also the secular change. This tendency was remarkable especially in the case of static deflecton. However, it seems that, when the annealing was carried out immediately after cold-rolling, the secular change showed the tendency similar to the above-mentioned one as rolled.

(iii) The case of annealing after ageing at 40°C for 20 days since the final cold-rolling ($T_{20}$ series)

The specimens were heat-treated at 200°C for 5 min~3 hrs after they were allowed to remain at 40°C for 20 days since their final cold-rolling, and then the secular change in spring properties was measured. The result is shown in Fig. 5.

In this case, it was characteristic that the change became considerably slight, especially in the case of the residual deflection, as compared with the result shown in Fig. 4. When the time of annealing at 200°C was short, the tendency

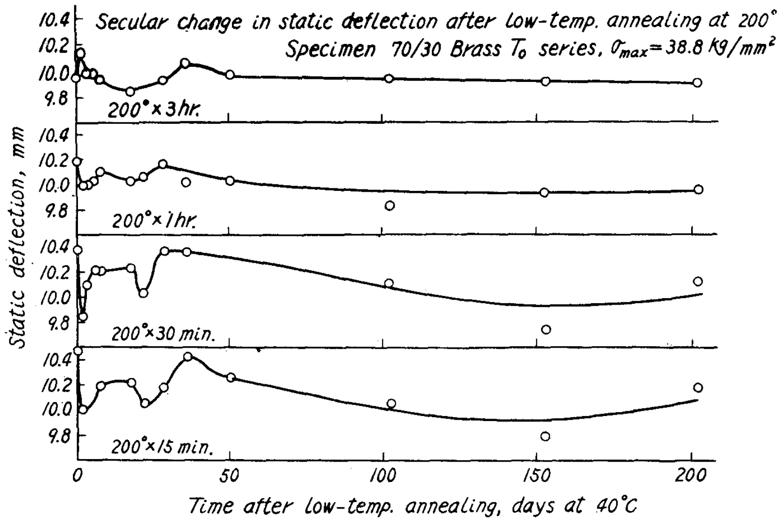


Fig. 4 (a)

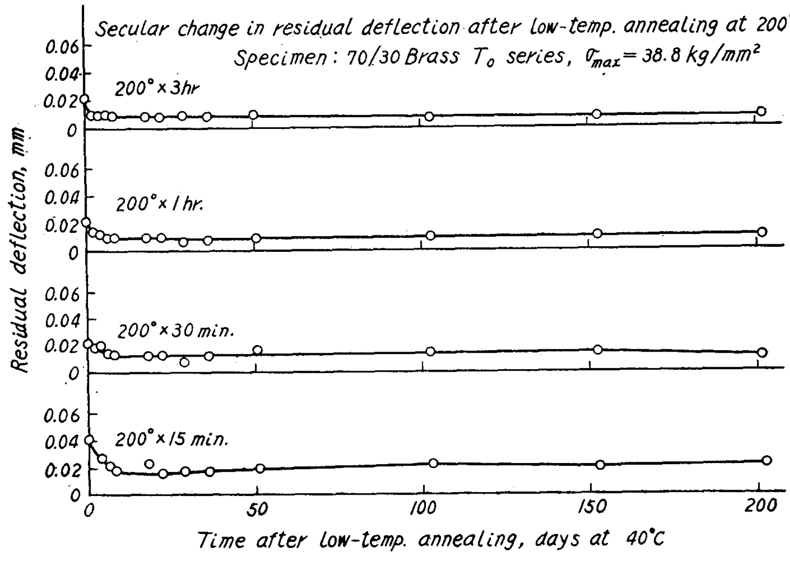


Fig. 4 (b)

Secular change in deflection after low-temp. annealing; Specimens were immediately annealed at 200°C after final cold-rolling, and aged at 40°C

of the change in static deflection was similar to that of $T_0$ series mentioned above. However, according as the time of annealing was prolonged, it showed a markedly different tendency.

(iv) The case of annealing after ageing at 40°C for 40 days since the final cold-rolling ($T_{40}$ series)

The results of measurement, when the specimens were heat-treated at 200°C for 5 min～2 hrs after they were allowed to remain at 40°C for 40 days since their final cold-rolling, are shown in Fig. 6. The tendency of the change in this case seems to be similar to that of $T_{20}$ series, and is quite different from that of $T_0$ series.

In surveying the above results, it can clearly be seen that the secular change

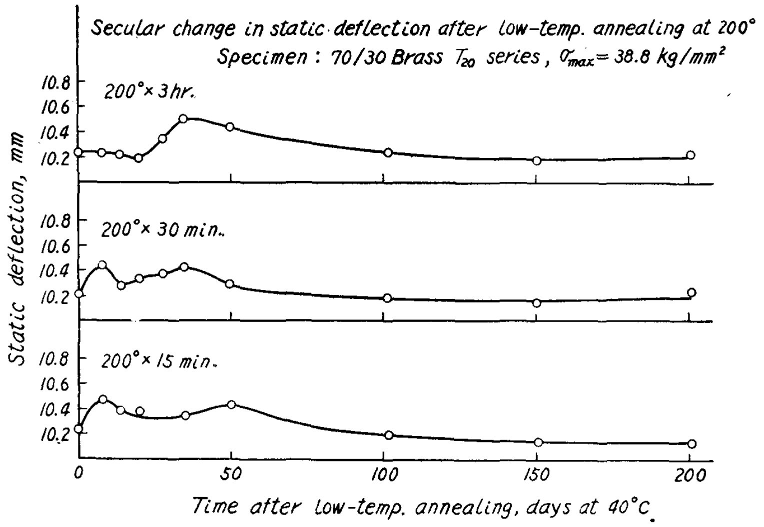


Fig. 5 (a)

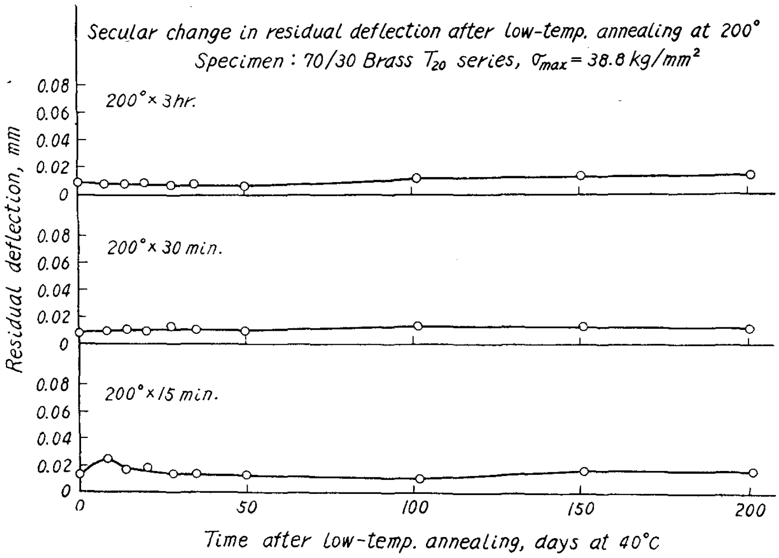


Fig. 5 (b)

Secular change in deflection after low-temp. annealing ; Specimens were allowed to remain at 40°C for 20 days after final cold-rolling, then annealed at 200°C.

in spring properties either after cold-working or after low-temperature annealing is a decreasing tendency in the amount of deflection, and that the change after anneal-hardening is influenced by the length of time from final cold-working to heat-treatment; that is, the longer the time, the less the change. Considering this together with the results on 60/40 brass previously reported,(1) the factors affecting the secular change can be deduced to be quite similar to each other. Those results are incompatible with the opinion that the secular change after cold-working has no relation to that after anneal-hardening.(7),(8) Furthermore, it is obvious from the present results that the time of annealing itself, that is, the development degree of the phenomenon of anneal-hardening has also relation to the secular change there-after. Though the mechanism of these phenomena

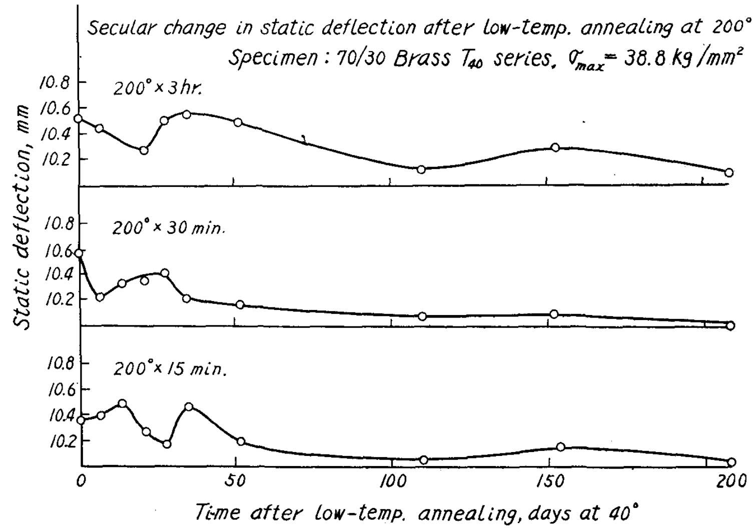


Fig. 6 (a)

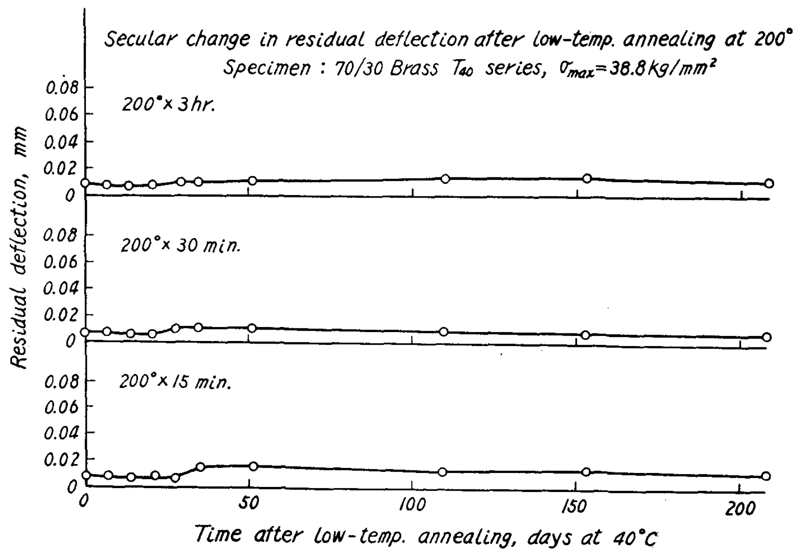


Fig. 6 (b)

Secular change in deflection after low-temp. annealing ; Specimens were allowed to remain at 40°C for 40 days after final cold-rolling, then anealed at 200°C.

is yet uncertain, the preventive measures against the secular change in brasses will be obtained, if these phenomena are synthetically investigated.

## Summary

The secular changes in 70/30 brass after cold-rolling and anneal-hardening were investigated by hardness and spring tests. The results may be summarized as follows:

(1) The change after cold-rolling shows a tendency of hardening, especially in

highly reduced materials. The amounts of both static and residual deflections show decreasing tendency.

( 2 ) The secular change after anneal-hardening, in the same way as the case of 60/40 brass, is influenced by the length of time from the final cold-rolling to heat-treatment (namely, degrees of the secular change after cold-working), and further, by the time of annealing (namely, degrees of anneal-hardening). On the whole, the longer the times of the above are, the less becomes the secular change in amount.

( 3 ) To prevent the material from the secular change after anneal-hardening, it should be necessary to consider the relation between them.