# Research on the Structure and Low-Temperature Annealing Effect in Cold-Rolled $\alpha$-Brass. II
## Structures Cold-Rolled and Annealed at Low Temperatures*

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**Synopsis**

The structure changes by cold-rolling and low-temperature annealing of 70/30 brass were examined by a microscope and X-ray photography. The difference in ready-to-finish grain size affected the degree of development of the rolling structure. A coaise-grained material tended to delay the development, compared with a fine-grained one. The tendency seemed to relate to the hardness change by low-temperature annealing as previously reported. No remarkable change in structure was observable by annealing at 150°, while a kind of polygonization was seen at 250°. Though the nucleation by recrystallization developed preferentially at the regions strained severely, grain growth was observed along the slip lines at 350°. The difference in grain size affected the rate of recrystallization.

## I. Introduction

Many researches have been made on the so-called anneal-hardening phenomenon observable in the solid solution of copper alloys, but they are not yet ready for putting a decisive interpretation upon its mechanism(1). Especially, the fact that the anneal-hardening is hardly observable in single crystals(2) gives rise to some difficulties in interpreting the mechanism. In general, it is said that the grain size affects the amount of the anneal-hardening, and that the hardening in coarse-grained materials is smaller. The relation between them, however, is not so simple, as already shown in the previous report(3). The result of X-ray analysis by Shinoda and Amano concerning the anneal-hardening phenomenon in the vicinity of the grain boundary of $\alpha$ brass has recently been reported, and clarified that the vicinity of the grain boundary played an important role in the anneal-hardening(4). Furthermore, according to their report, the difference between the changes in hardness at the region of grain boundary and at that within the grain decreases in a highly reduced material.

In the present research, apart from discriminating the difference between the

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* The 932nd report of the Research Institute for Iron, Steel and Other Metals. Published in Japanese in the J. Japan Inst. Met., **21** (1957), 632.

(1) Kawasaki, Metals, **26** (1956), 755 : 875. [Published in Japan]

(2) Kato, Nishikawa and Suyama, J. Japan Inst. Met., **20** (1956), 234.

(3) Izumi, Sci. Rep. RITU, **A11** (1959), 120.

(4) Shinoda and Amano, J. Japan Inst. Met., **21** (1957), 59 ; 62.

influences in the grain boundary and within the grain, the development of the rolling structure in cold-rolled 70/30 brass sheets with different grain sizes was exanined, and the microscopic and X-ray observations of the structural change by annealing were also carried out.

## II. Experimental procedures

1. Specimen

The specimens were cold-rolled 70/30 brass sheets used in the previous study, and after measuring the hardness, microscopic and X-ray observations were carried out. The working procedure and the chemical composition of specimens were the same as those in the preceding case(3).

2. Experimental methods

(i) Microscopic observation

After the sufficient polishing with emery paper, the specimens were electrolytically polished and etched by using 50 per cent orthophosphoric acid solution, and then were chemically etched with ferric chloride solution, which showed a striking contrast of the etched figures. It is reported that the sensitivity to the detection of deformation structure varies with etching reagents(5), but no investigation into this was carried out this time. Therefore, if a different etching solution is used, a structure different from that shown in this report may appear.

The microscopic observation was made with the magnification of 50~1,046 folds, with regard to the rolling surface and the longitudinal and transverse section.

(ii) X-ray observation

The specimen was immersed in 50 per cent nitric acid solution and was made about 0.05~0.10 mm in thickness, and transmission, back-reflection photographs were taken with copper and tungsten targets. X-ray beams were made as thin as possible by using a slit, and the condition for photographing were as follows: the distance between the specimen and the film was 40 mm, the power 7 KV-30 mA, the exposure time of 3 hrs (in the case of copper-target), and the power 50 KV-10 mA, the exposure time of 6 hrs (in the case of tungsten).

## III. Experimental results and considerations

1. Working structure

In the range of low reduction of about 10 per cent, the slip-lines appeared locally on the rolling surface or near the grain boundary. The shapes of grains were almost the same as those in the annealed state, and the slip-lines within the grain were almost straight. The ready-to-finish grain sizes had no special influence on the structural change. The microstructure of the plate surface is shown in Photo. 1. At the reduction of 20 per cent, the development of the deformation structure was remarkable, especially in the fine-grained material, and the secondary

(5) L. E. Samuels, J. Inst. Metals, 83 (1954-55), 359.

slip and the bending of already made slip-lines were partly observed.

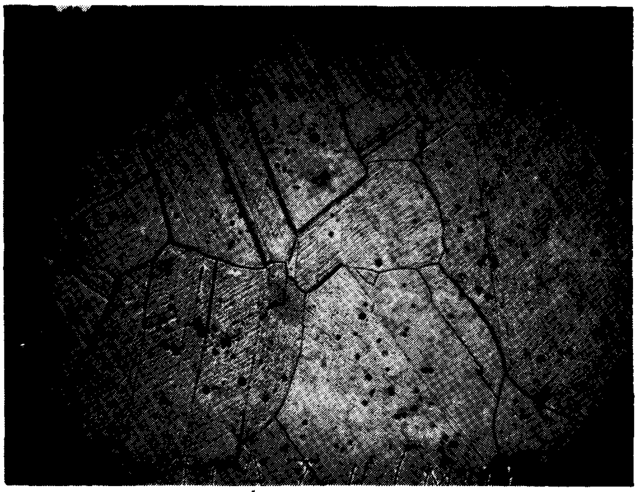


Photo. 1. Microstructure of plate surface of cold-rolled 70/30 brass, specimen : coarse-grained, reduction : 10%, magnification : ×100

At 30 per cent reduction, the development of secondary slips was striking. Further, when the slip-lines in the same grain became symmetrical with respect to the rolling direction, a herring-bone structure of a twin-like form divided by the straight borderline parallel to the rolling direction was formed as shown in Photo. 2. This was perhaps an accidental phenomenon relating to the crystal

Photo. 2. Microstructure of plate surface of cold rolled 70/30 brass, specimen : coarse-grained, reduction : 30 % magnification : ×100

orientation and the working direction. As compared with the coarse-grained specimen, the fine-grained one showed a more complicated development of slip-lines and a somewhat larger line-broadening of X-ray Debye-ring. The grain boundary has influence upon the development of slip-lines, which bend near the grain boundary owing to the boundary. In other words, the deformation mechanism in this stage progresses with the slips in individual grains, but the boundary still remains distinctly observable.

When the working reduction increased further to 50 per cent, the deformation mechanism by the slips in a grain reached almost the limit, and as seen in Photo. 3, not only the slip-lines but also the deformation of the grain boundary became

Photo. 3. Microstructure of plate surface of cold-rolled 70/30 brass, specimen : coarse-grained, reduction : 50%, magnification : ×100

remarkable, and finally, a new strain-marking became observable across the old slip-lines in the places full of strain concentrations as shown in Photo. 4

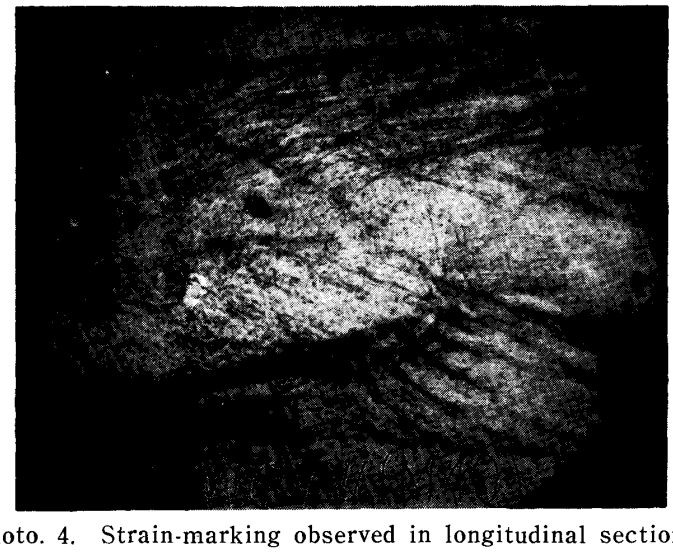
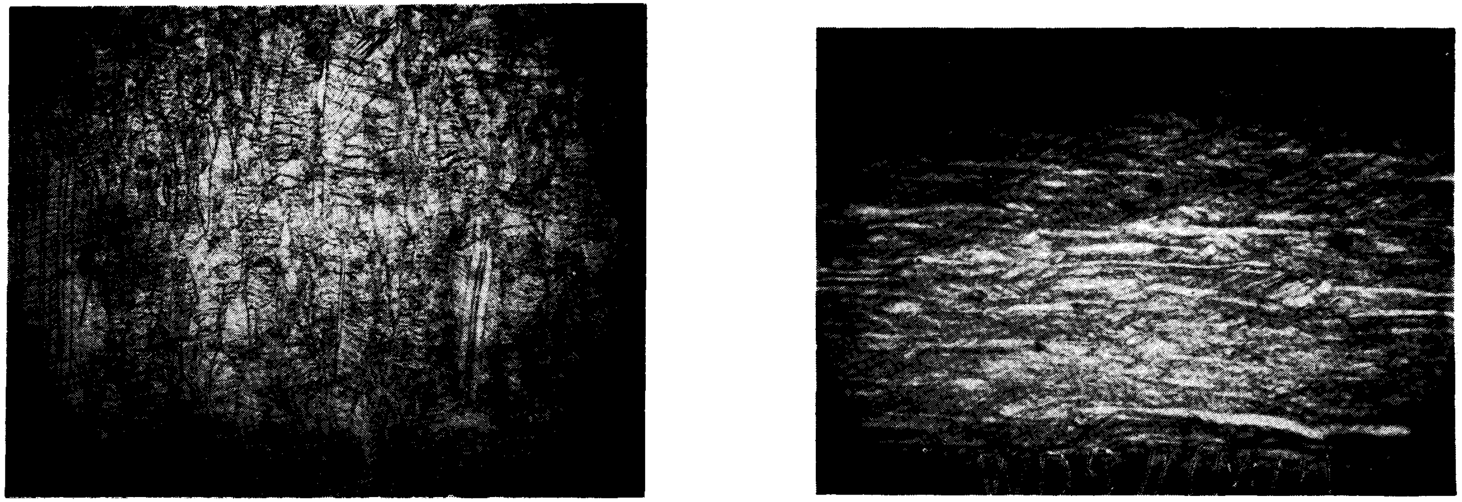


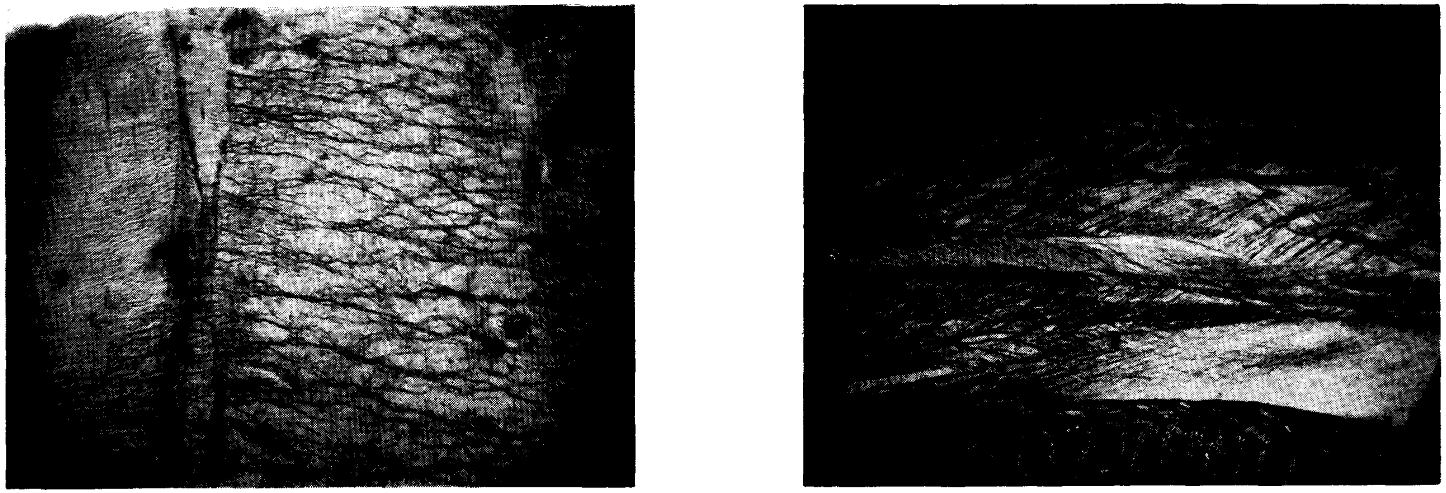
Photo. 4. Strain-marking observed in longitudinal section of cold-rolled 70/30 brass plate, specimen : coarse-grained, reduction : 50%, magnification : ×364

At 70 per cent reduction the development of this strain-marking became conspicuous, and on the rolling surface it developed nearly perpendicularly to the rolling direction in the longitudinal section at an angle of about 30~40° to the rolling surface. The way in which the strain-marking appears varied with the ready-to-finish grain sizes, and in the fine-grained material it was fine, short and dense as shown in Photos. 5(a) and (b). In the coarse-grained specimen it was



(a) (b)

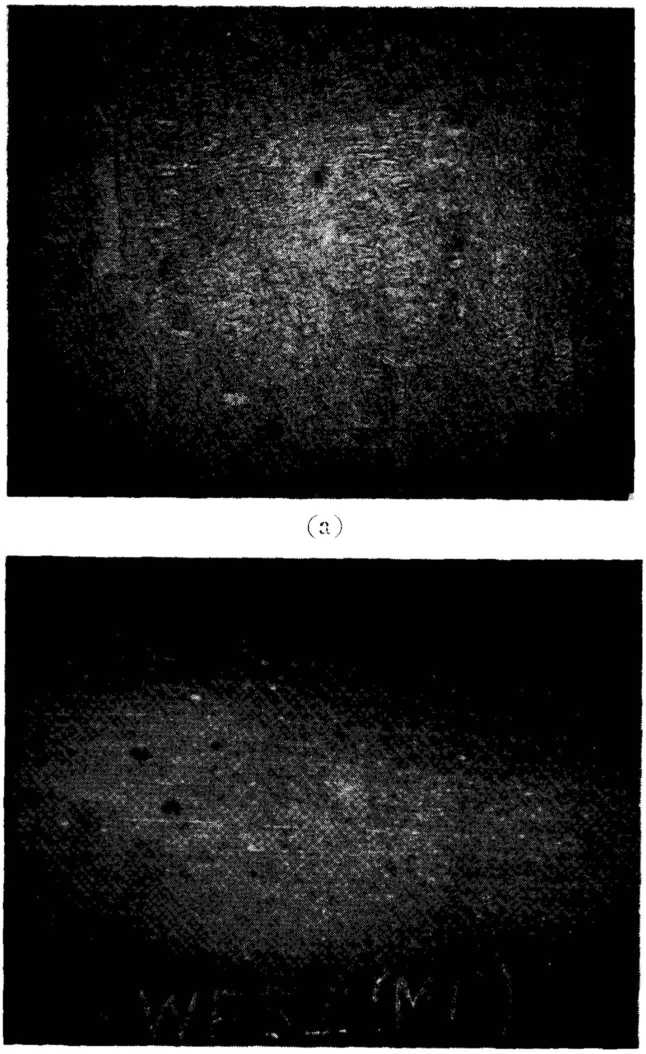
Photo. 5. Develoment of strain-marking at 70% reduction in fine-grained 70/30 brass plate, (a) plate surface, (b) longitudinal section, magnification : ×100



(a) (b)

Photo. 6. Development of strain-making at 70% reduction in coarse-grained 70/30 brass plate, (a) plate surface, (b) longitudinal section, magnification : ×100 thick, long and sparse as shown in photos. 6(a) and (b). In the longitudinal section, the strain-markings were observed to cross each other, forming a rhombic shape. The coarse-grained specimen was more delayed in the development of the strain-marking. It was also hard to see the development in the grain, in which the slip-lines formed a herring-bone structure.

At 90 per cent reduction the strain-marking became minute, and in the fine-grained specimens the development of the structure change almost reached the limit, and the longitudinal section was studded with a few rhombic shapes as shown in Photos. 7(a) and (b). In coarse-grained specimens this development

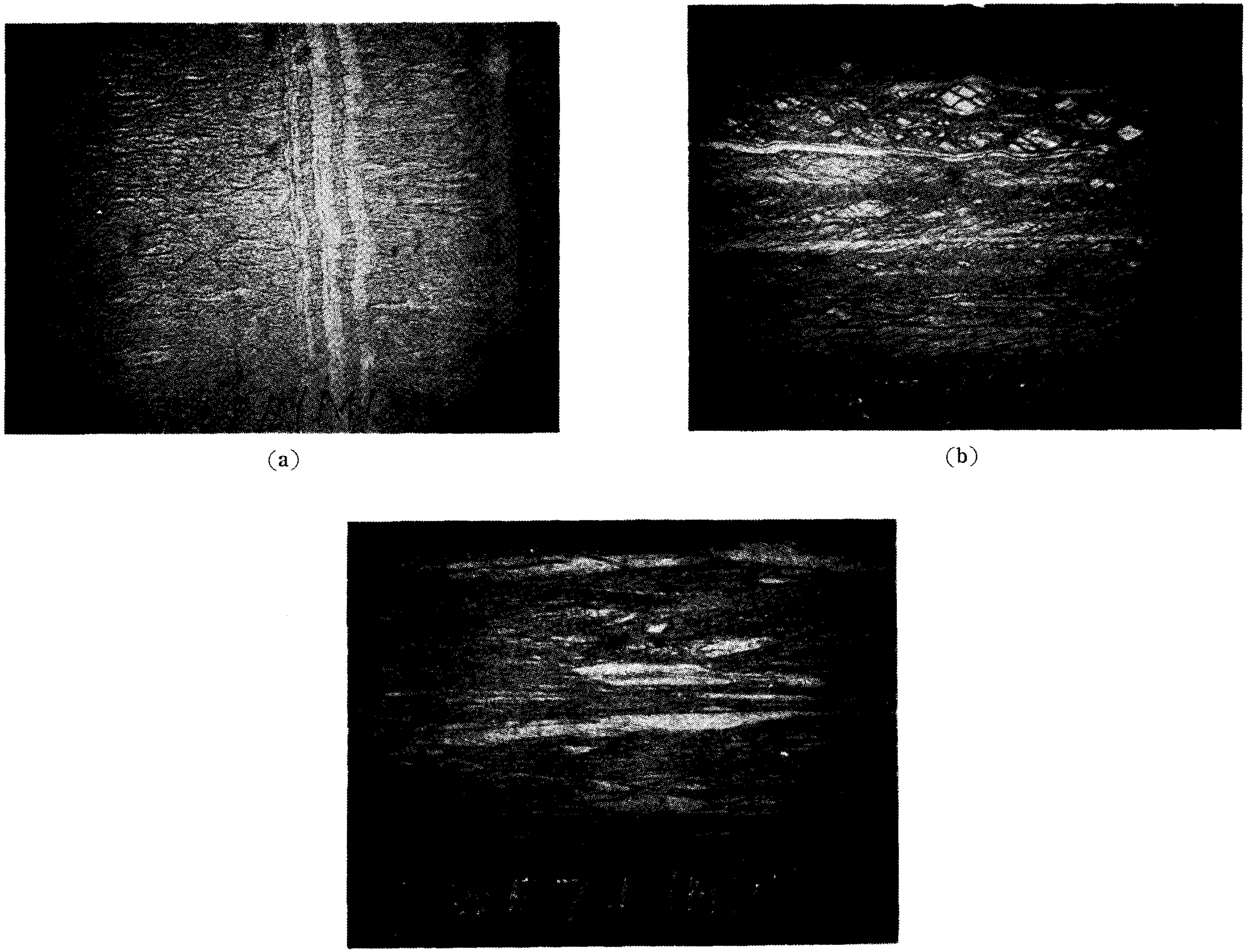


(a)

(b)

Photo. 7. Development of strain-marking at 90% reduction in fine-grained 70/30 brass plate, (a) plate surface, (b) longitudinal section, magnification : ×100

delayed as shown in Photos. 8(a), (b) and (c), and the rhombic shapes in the longitudinal section were large, while in the transverse section there existed the layers without strain-marking corresponding to a rhombic shape in the longitudinal section. In this stage the grain boundary was hardly observable.



(a) (b) (c)

Photo. 8. Development of strain-marking at 90% reduction in coarse-grained 70/30 brass plate, (a) plate surface, (b) longitudinal section, (c) transverse section, magnification : ×100

The above-mentioned is the structural change in the case of straight-rolling. Different methods of rolling will naturally alter the aspect of the structure*. As stated above, the development of the rolling structure goes through a different process approximately at 50 per cent reduction, which forms a turning-point. The development degree varies with ready-to-finish grain sizes. It was already stated that the development degree related to the effect of low-temperature annealing. Now, what influence has the low-temperature annealing on the working structure? The result of the present observations will be stated in the following.

2. Low-temperature annealing structure

(i) 150°-annealing structure

As stated in the previous report, at 150°-annealing the hardness showed a marked change, but according to the present microscopic and X-ray observation there was litile or no difference between the structure after annealing and that as rolled. Only the line-broadening of Debye-ring (Target: copper, back-reflection) decreased after a long annealing. Even in the transmission photograph with tungsten-target the diffusion of asterism near the centre became somewhat less and the ring caused by L-spectrum of tungsten-beam came in sight as shown in Photo. 9.

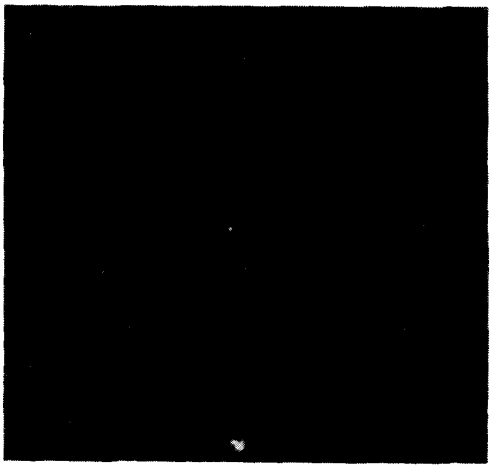


Photo. 9. X-ray photograph of 70/30 brass plate, cold-rolled 70% and annealed at 150° for 24 hrs, traget: W, transmission

(ii) 250°-annealing structure

In the annealing at 250°, as stated in the preceding report, the highly reduced material showed anneal-hardening at an initial stage of annealing, and then the recrystallization took place. In this case the structure was somewhat different from that in the annealing at 150°. For instance, the specimen of 70 per cent reduction annealed for 20 min. showed the greatest anneal-hardening before the recrystallization. In the transmission X-ray photographs with copper target (Photos. 10 (a) and (b)), the Debye-ring was scarcely different from that of the specimen as rolled, but the ring taken by back-reflection showed the tendency to be finely

* The structural change due to the rolling methods will be reported on another occasion.

(a)

Photo. 10 (a) X-ray photograph of 70/30 brass plate cold-rolled (70%) and annealed at 250° for 20 min, target : Cu, (a) transmission

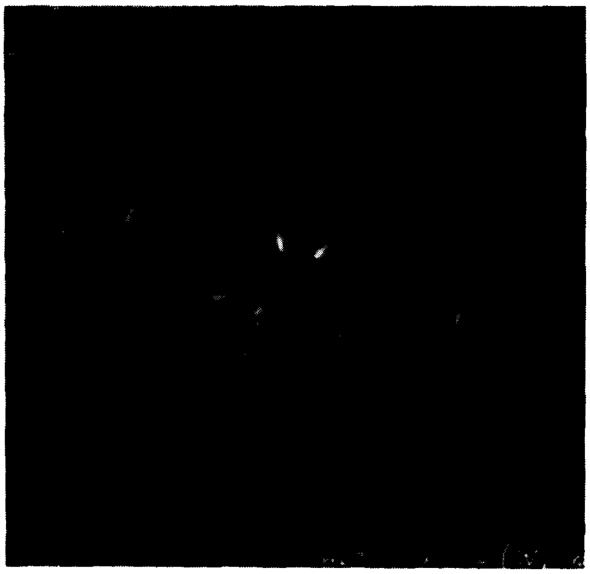

(b)

Photo. 10 (b) X-ray photograph of 70/30 brass plate cold-rolled (70%) and annealed at 250° for 20 min, target : Cu, (b) back-reflection

dotted and spotty. From these observations, together with the microscopic photographs, which will be mentioned later, a kind of polygonization may be inferred[6]. The specimens, after the beginning of recrystallization, make it difficult to analyse the structure by X-ray photographs on account of a new grain. Further, in transmission X-ray photographs with tungsten target, the asterism was considerably

(6) C. Crussard et al., Prog. Met. Phys., **3** (1950), 193.

different from that of the above-mentioned material annealed at 150° as shown in Photos. 11 (a) and (b). The photograph (a) refers to the 70 per cent reduced material annealed for 20 min. showing the greatest anneal-hardening, where as (b)



(a)

Photo 11 (a) X-ray photograph of 70/30 brass plate, cold-rolled and annealed at 250°, target : W, transmission, (a) 70% reduction, 250°×20 min

(b)

Photo 11 (b) X-ray photograph of 70/30 brass plate, cold-rolled and annealed at 250°, target : W, transmission, (b) 50% reduction, 250°×3 hrs

to the specimen reduced at 50 per cent and brought about partial recrystallization after annealing for 3 hrs. In these cases the asterism was conspicuous, as compared with that of the material annealed at 150°. The asterism became sharpened and steppedly prolonged (the latter might be due to the absorption effects of Ag and Br of film), and the Debye-ring by tungsten-L spectrum became distinct. From this it will be inferred that by annealing, the internal stress is recovered with the result of diminishing the diffusion of diffracted beams, and that at the same time the structural changes like polygonization or recrystallization ***in situ*** occur.

On the other hand, the microstructure showed phenomena which may testify to the structure change observed by X-ray, that is, the places where the slip-lines are bent are locally divided into small blocks, in each of which slip-lines have a tendency to become straight. This tendency became striking when the annealing time was prolonged. An example is shown in Photo. 12. Considering this together

Photo. 12. Microstructure of cold-rolled 70/30 brass plate, annealed at 250° for 3hrs, reduction : 50%, magnification : ×1,046

with the above-mentioned X-ray photographs, it may be concluded that in the annealing at 250° a kind of polygonization is progressing. It is not yet ascertained, however, how this relates to the hardness change stated in the previous report.

3. Recrystallization structure

Corresponding to the working reduction, the recrystallization began at 250°

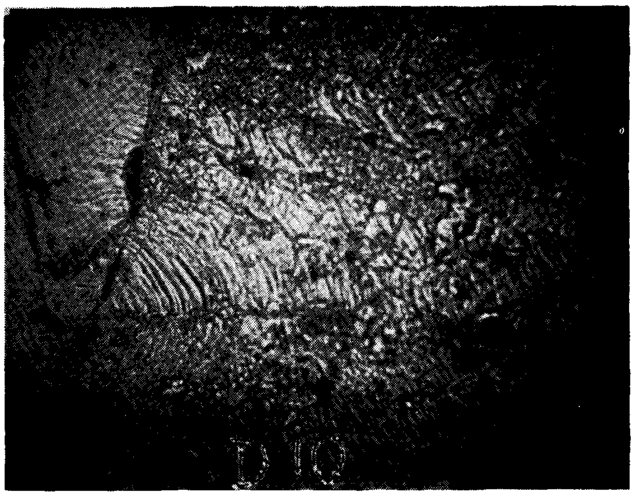


Photo. 13. Recrystallization in strain-marking region of cold-rolled 70/30 brass plate, reduction : 70%, annealed at 250° for 5 hrs, magnification : ×1,046

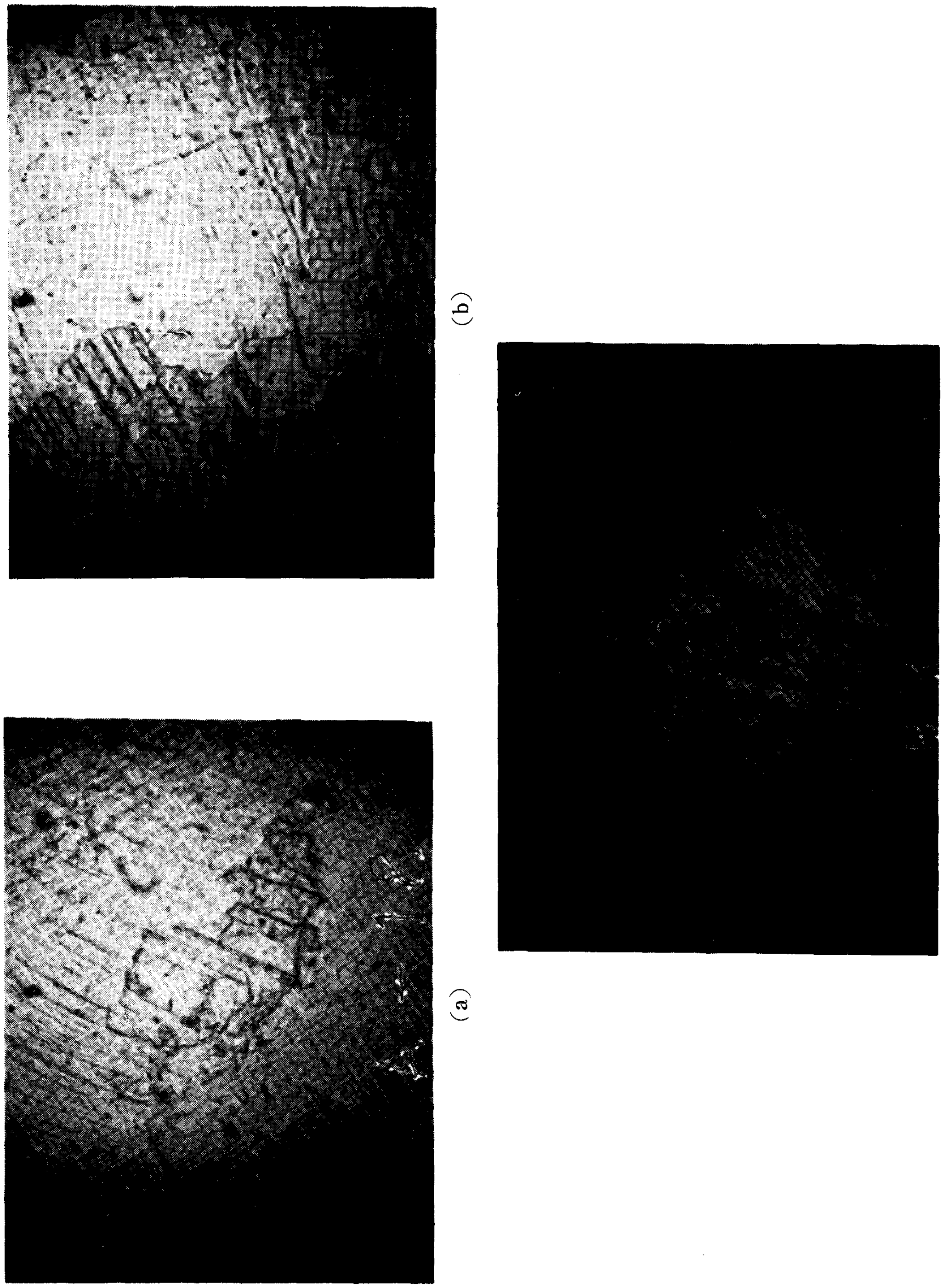



Photo. 14. Nucleation and grain growth observed in bent slip line region of cold-rolled 70/30 brass, (a), (b) reduction 70%, annealed at 350° for 30 min ($\times$1,046), (c) reduction 70%, annealed at 350° for 10hrs ($\times$364)

after a certain time and at 350° and 450° at an initial stage of annealing. The nucleation was predominantly observable in the regions of more complicated deformation structure, that is, in the regions of slip-lines concentrated or bent, and of the grain boundary or the strain-markings appearing in a highly reduced specimen. For instance, Photo. 13 shows the structure of the specimen of 70 per cent reduction annealed at 250° for 5 hrs. The nucleus of a new grain grows mostly along the strain-marking. In a region with comparatively simple slip-lines, for example, a herring-bone structure, the recrystallization is more delayed than in other regions. It has already been stated in the previous report[3] that the development degree of the working structure affects the change in hardness due to annealing effect. Also the velocity of nucleation of recrystallization varies with the ready-to-finish grain sizes, that is, as the development of the working structure is slow in the coarse-grained material, even under the same condition of reduction and heat-treatment, the recrystallization process is also delayed. In the annealing at 350°, the direction of the growth of nuclei was often the same with that of the slip-lines. Photo. 14 shows an example. It is not clear whether this is because the sub-grain formed in the region where the slip-lines are bent has grown as a nucleus, or it is an accidental result. As the nucleation velocity changes with the location because of unhomogeneous strain distribution, the grain sizes are not uniform on the way of recrystallization, but when the recrystallization progresses enough, they gradually become uniform. As for the discontinuous process of hardening at 350°, which has been stated in the previous report, its structural causes could not be ascertained by either microscopic or X-ray observation. Though the recrystallization process at 450° has no special characteristic, the transmission photograph with tungsten target is shown, for comparism, in Photo. 15, in which the asterism near the centre such as can be seen in Photos. 9 and 11 vanishes and is replaced by fine dots owing to new grains.

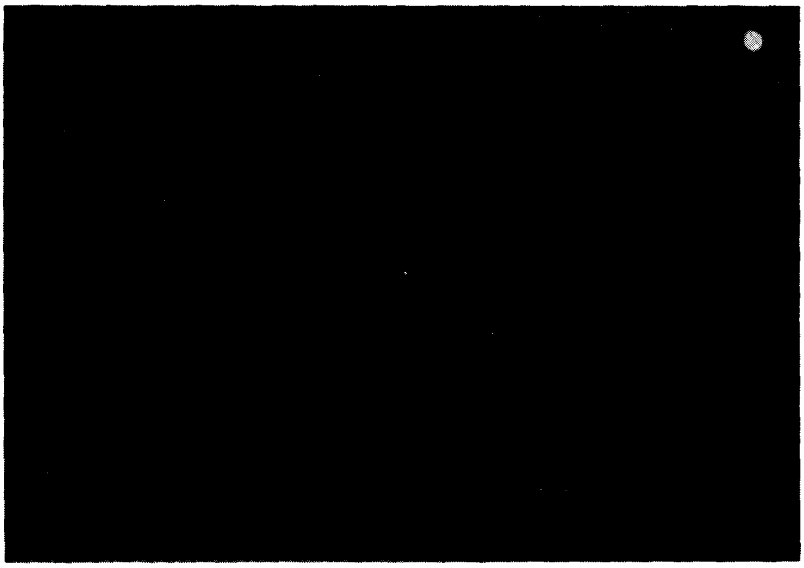


Photo. 15. X-ray photograph of 70/30 brass plate, cold-rolled 50% and annealed at 450° for 30 min, target: W, transmission

## Summary

The rolling structure of 70/30 brass cold-rolled 10～90 per cent, and the structure annealed at 150～450° for various intervals of time were observed with microscope and X-ray. The results may be summarined as follows:

(1) The rolling structure begins with 50 per cent of reduction as turning-point to shift from the development of slip-lines in the grain to the striking strain-marking. In coarse-grained material the structural change is slow.

(2) In the annealing at 150° no remarkable structure change is observable except the decrease of line-broadening of X-ray Debye-ring after a long time annealing.

(3) In the annealing at 250° the bent slip-lines are divided into small groups, in which the slip-lines become comparatively straight. In the hardening stage before recrystallization, the Debye-ring taken by transmission shows no remarkable difference from that of the specimen as rolled, but the ring taken by back-reflection shows the tendency to be dotty. From these a kind of polygonization is inferred.

(4) According to the transmission photograph with tungsten target, the asterism is more remarkable in the material annealed at 250° than in that annealed at 150°, while in the recrystallized material annealed at 450° it vanishes and is replaced by fine spots irregularly produced owing to new grains.

(5) The nucleation of recrystallization is predominantly observable in the regions where the working structure has made a remarkable progress. The coincidence of the direction of slip-lines and that of nucleus growth is often perceived in the case of the annealing at 350°.


In conclusion, the writer wishes to express his heartfelt thanks to the late Professor Toba and Assistant Professor Mizuno, of the Research Institute for Iron, Steel and Other Metals, Tôhoku University, for their guidance and advices on this subject, and to Mr. Takayoshi Monma and Mr. Shin-ichi Ishida for their hearty cooperation in the experiments.