Study on the Recrystallization Temperature of Refined High Purity Aluminium (7th Report) On the Abnormal Hardening on the Annealing Curve.*

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Synopsis

This paper deals with the abnormal hardening phenomena on the annealing curves of pure aluminium (99.5~99.998 %) which have not yet been brought clearly to light. An abnormal hardening at lower temperature was clearly observed in the aluminium of 99.92 % purity which had undergone a lighter reduction less than 50 %, but it could not be recognized in the case of such a heavier reduction as 90 %. And the second abnormal hardening or incubation period was observed in all pure aluminiums. By X-ray analysis, it was ascertained that the beginning temperature of recrystallization exists in the incubation period or in the neighbourhood of second abnormal hardening.

I. Introduction

It may generally be said that the abnormal hardening phenomena of cold-rolled pure copper and aluminium are not clearly ascertained on their annealing curves. However, Cooks and Richard[1] observed that a long incubation period exist

* The content of this paper was read before the Kobe meeting of the Japan Institute of Metals held on 11th, October 1951.

(1) M. Cook and T. Richards J. I. M., **73** (1947).

before the softening in a long annealing experiment, and also a slight abnormal hardening at lower temperature is observed in many experiments[2)~5)] of pure copper and aluminium. Recently, Chossat[6)] observed the abnormal hardening phenomena on the annealing curves of high purity aluminium and its alloys.

The authors have experienced that the abnormal hardenings on the annealing curves of pure aluminiums are often observed, so that tried to investgate these abnormal hardening phenomena appearing at the lower temperature and the neighbourhood of the beginning for recrystallization.

II. Effect of Rolling Reducntion

1. Material and Testing Method.

Annealed sheet 1 mm in thickness whose purity is 99.93% was rolled down to

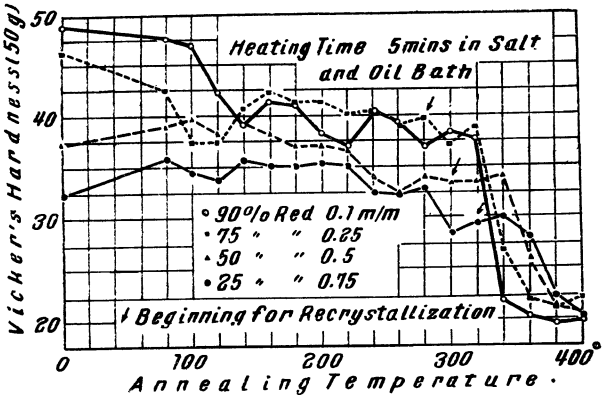


Fig. 1 Influence of the Cold Rolling Reduction on the Annealing Curves of 99.9 % Aluminium.

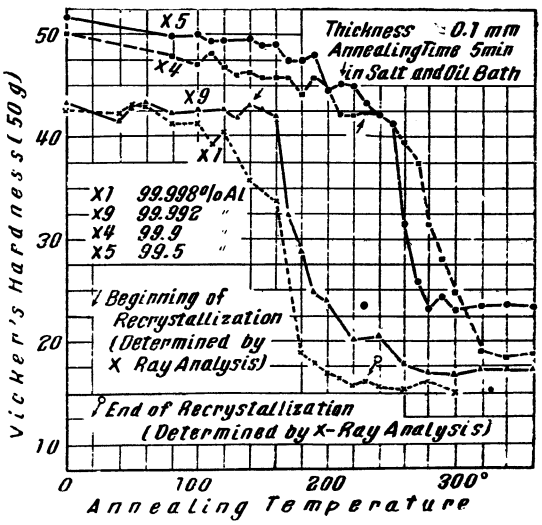


Fig. 2 Annealing Curves of Various Aluminium Foils.

0.75, 0.50, 0.25, 0. 1mm (25, 50, 75, 90% Red.) and the specimens were cut off from these sheets. Then they were annealed for 5 minutes in an oil or sal t bath at 100~400°in an interval of 20°and cooled in air. And Vicker's hardness was measured by a micro-hardness tester.

2. Result of Hardness Test

The annealing curves obtained are shown in **Fig.1**. In the case of heavier reduction (75~90 %), the decrease of hardness by the release of internal stress was evident even at a lower temperature and consequently the abnormal hardening did not appear. However, in the case of a lighter reduction (25~50 %), it was clearly recognized at the temperature about 100°. On the other hand, the second abnormal hardening was observed in all curves, that is, the incubation period (horizontal part) or irregular slight hardening was seen before a sudden softening. It seems to be inversely proportional to the degree of reduction corresponding to the change of recrystallization.

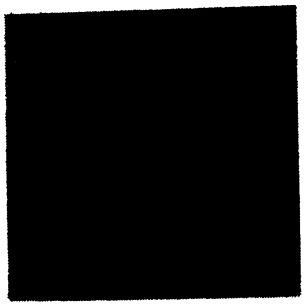
III. Effect of Purity

1. Meterial and Testing Mathod

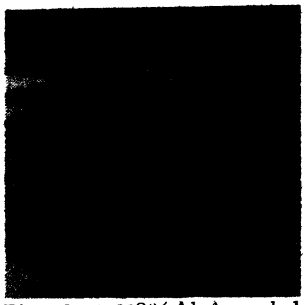
Specimens were cut off from the foils of four kinds of pure aluminium (99.5, 99.92, 99.992, 99.998 %) rolled down to 0.1mm in thickness (90 % Reduction). And they were heat-treated at 40~360° similarly as before. Then Vicker's hardness was measured and X-ray analysis was carried out to determine the relation between the abnormal hardening and the pattern of Laue photograph.

2. Result of Hardness Test

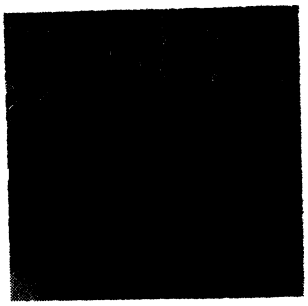
(2) G. S. Farnham and H. O'Neill, J. I. M., **55** (1934-II), 201.
(3) A. H. Cottrell, J. I. M., **68** (1942).
(4) P. C. Varley, J. I. M., **75** (1948), 185.
(5) C. Grussard, Rev. de Mét., **16** (1944), 111.
(6) H. Chossat, Rev. de Mét., **47** (1950), 167.



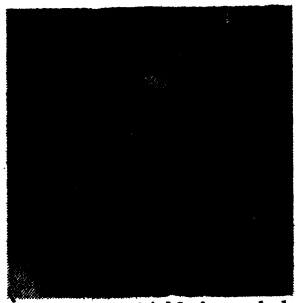
Phot. 1 99.998% Al Annealed for 5 mts. at 40°.



Phot. 2 99.998% Al Annealed for 5 mts. at 80°.

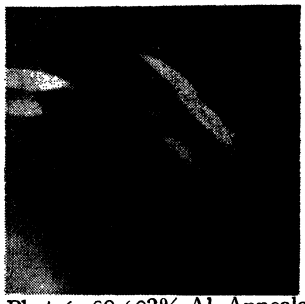


Phot. 3 99.998% Al Annealed for 5 mts. at 100°.

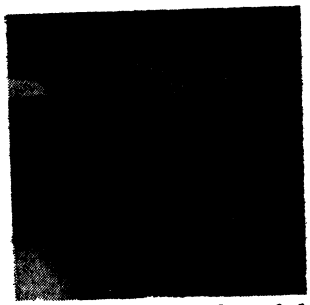


Phot. 4 99.998% Al Annealed 5 mts. at 110°.

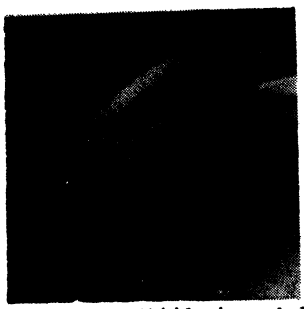
Phot. 5 99.998% Al Annealed for 5 mts. at 230° Showing End of Recrystallization.



Phot. 6 99.992% Al Annealed for 5 mts. at 140° Showing Beginning of Recrystaillization.



Phot. 7. 99.92% Al Annealed for 5 mts. at 2 0° Showing Beginning of Recrystallization



Phot. 8 99.5% Al Annealed for 5 mts. at 210° Showing Beginning of Recrystallization

The annealing curves obtained are shown in **Fig. 2**. It is seen that there did not appear any abnormal change in the recovery course of 99.998% Al as compared with other alminiums and its recrystallization seems to begin at about 100°. The abnormal hardening at a lower temperature could hardly be observed in the annealing curves of 99.998 and 99.992% Al which had undergone such a heavier reduction as 90%. However, the second abnormal hardening or the incubation period was observed in each curves. The incubation period in which the recrystallization shall begin, appeared at 140, 210, 200° in 99.992, 99.92, 99.5% Al respectively.

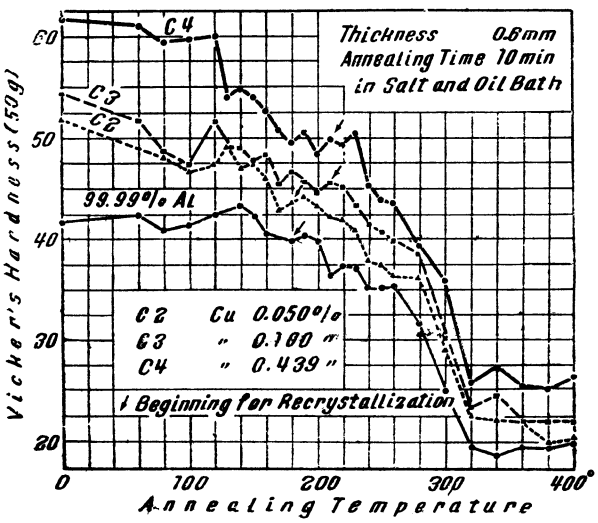


Fig. 3 Influence of Cu on the Annealing Curve of 99.99% Al.

3. Result of X-ray Test

Some of the Laue photographs are shown in **Photos. 1~8**. It is noticeable that the recrystallization of 99.998% Al aged for 6 months after rolling began at room temperature inspite of the

result derived from annealing curve in which it seems to beabout 100°, and it was also noted that the recovery of initial stress had already occurred at room temperature. We could not find the relation between the abnormal hardening and pattern of Laue photograph, but the beginning temperature of recrystallization determined by X-ray analysis coincided with the incubation period of anneling curve.

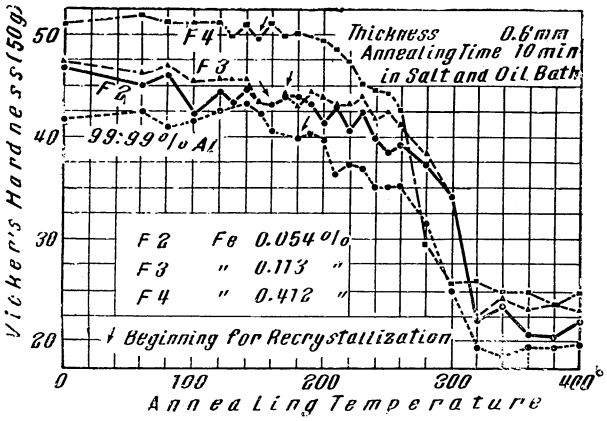


Fig. 4 Influence of Fe on the Annealing Curve of 99.99%Al.

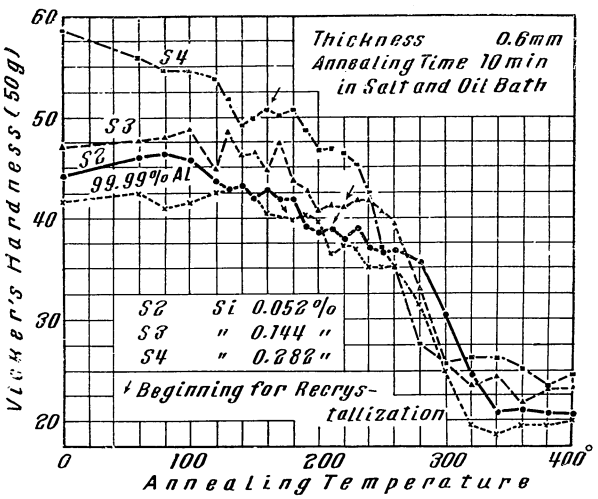


Fig. 5 Influence of Si on the Annealing Curve of 99.99 % Al.

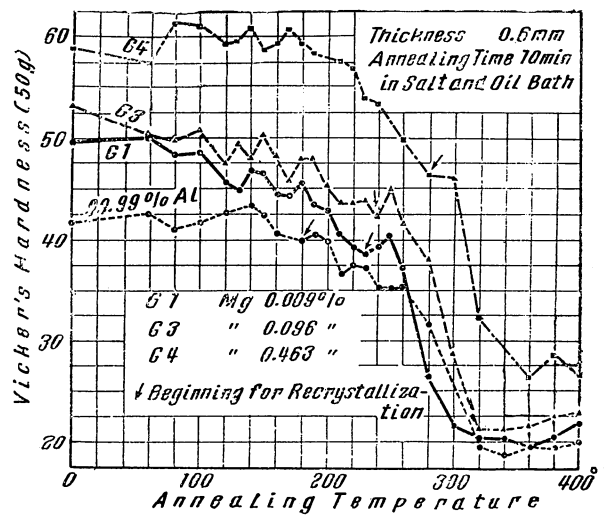


Fig. 6 Influence of Mg on the Annealing Curve of 99.99 % Al.

IV. Effect of Impurity

1. Material and Testing Method

The high purity alminium of 99.992 % purity and its alloys to which Cu, Fe, Si, Mg, Zn, Mn and Ti were added in such contents as 0.05, 0.10 and 0.5 % were cast, and rolled to 0.6 mm in thickness. (40 % cold-reduction) and they were annealed for 10 minuts at 60~400° and then Vicker's hardness was measured similarly as before.

2. Result of Hardness Test

The annealing curves of each specimens are shown in Figs. 3~9. The abnormal hardening at lower temperature was clearly observed in 99.99 % Al, but it gradually disappeared with the increase of impurities except the case of addition of Si and Ti. However, the second abnormal hardening or incubation period corresponding to the beginning of recrystallization was clearly observed in each specimen.

V. Consideration

It was found that the abnormal hardening at lower temperature seems to appear in the higher purity aluminium than 99.99 % even though its reduction is higher, but that in the case of lower reduction it appears clearly even in 99.92 % Al. It may be attributed to the precipitation of slight traces of impurity or to the change of mosaic structure, but its reason is not yet evident.

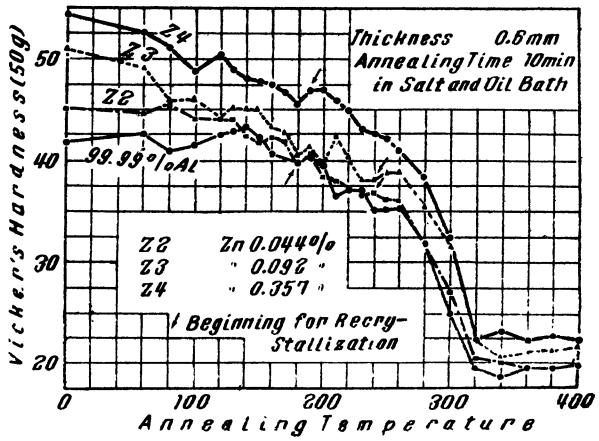


Fig. 7 Influence of Zn on the Annealing Curve of 99.99% Al.

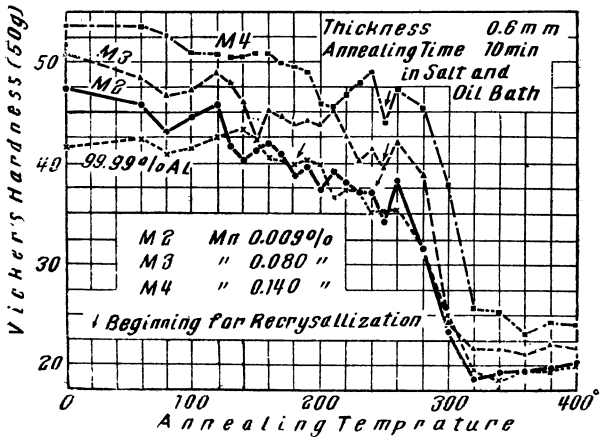


Fig. 8 Influence of Mn on the Annealing Curve of 99.99% Al.

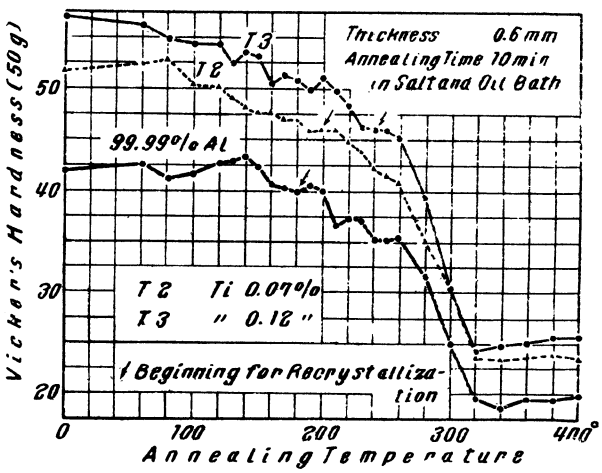


Fig. 9 Influence of Ti on the Annealing Curve of 99.99% Al.

therefore, the authors would investigate this problem in future. The second abnormal hardening or incubation period seems to be attributed to the disintegration of the crystals before the beginning of recrystallization. By X-ray analysis, it was ascertained that the beginning of recrystallization exists in the incubation period.

VI. Cunclusion

In this investigation, we conclude as follows :

(1) The abnormal hardening at lower temperature is clearly observed in the aluminium of 99.92% purity whose reduction is less than 50% and it seems to appear slightly in the higher purity aluminium than 99.99% even though its reduction is heavier.

(2) The second abnormal hardening or incubation period in the neighbourhood of beginning of recrystallization is observed in all pure aluminium independently of the degree of reduction.

(3) By addition of small amounts of impurities the second abnormal hardening before the beginning of recrystallization seems to become evident.

(4) It was ascertained that the beginning of recrystallization exists in the incubation period or at the neighbourhood of second abnormal hardening.

The authors are indebted to Dr. H. Nishimura of Kyoto Universlty for his kind advices.