Study on the Recrystallization Temperature of Refined High Purity Aluminium (4 th Report) The Relation between the Purity of Aluminium and the Recrystallization Temperature.

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Synopsis

In our previous report, the effect of small amount of impurities had been examined on the recrystallization temperature of high purity aluminium (99.99 %) with the aid of X-ray analysis. It was found that a small addition of impurity to be dissolved in aluminium as Cu, Mg and Zn moderately raises the beginning temperature of recrystallization, and Fe and Si (over 0.05 %) lower it. Mn and Ti within their solubility limits also raise it, but the temperature range of recrystallization becomes narrow.

In the present investigation, it was ascertained that such aluminium as with the highest purity of 99.998 % recrystallizes at about 230°, but in the case of the aluminium of 99.99 % purity its recrystallization temperature should rise to the higher temperature (about 300°) as similarly as reported in our previous works. It was found that there exists a maximum point of the recrystallization temperature between 99.9 and 99.99 % purity.

I. Introduction

As mentioned in our previous reports, the recrystallization was found to occur at the higher temperature when the purity was higher so far as the pure aluminium (99.0~99.99 %) made in Japan at that time was concerned. According to Mr. Chossat⁽¹⁾, the aluminium with such a highest purity as 99.998 % recrystallizes at 165°, but those of 99.99 % shows the higher temperature of 325° similar to our experiment.

It is much interesting that the higher the purity of aluminium, the higher the recrystallization temperature will be so far as 99.0~99.99 % purity are concerned but in the aluminium whose purity is higher than 99.99 % the recrystallization occurs at a lower temperature when the purity is higher.

The authors, therefore, tried to investigate this interesting phenomenon in which a maximum can be observed in the recrystallization temperatures of aluminium between the purity of 99.5 % and 99.998 %.

II. Experiment

Table 1 Chemical Compositions of Various Aluminiums.

No.	Purity %	Chemical Analysis per cent							
		Cu	Fe	Si	Mn	Mg	Zn	Ti	Al(Rem)
1	99.998	tr.	0.002	tr.	0	0	0	0	99.998
2	99.996	0.002	0.002	tr.	0	0	0	0	99.996
3	99.994	0.001	0.005	tr.	0	0	0	0	99.994
4	99.992	0.003	0.004	0.001	tr.	tr.	tr.	0	99.992
5	99.92	0.027	0.012	0.032	tr.	tr.	tr.	0	99.929
6	99.5	tr.	0.221	0.140	0	0	tr.	0	99.589

aces, they were heated at 450° for 10 hours and hot-rolled to 8 mm in thickness and cold-rolled to 1 mm and then annealed at 400°. Finally, they were cold-rolled to 0.1 mm foil by a foil mill (90 % reduction).

(2) Method of Testing

The test specimens were cut off from these foils aged 1.5 month after rolling and

(1) Material and Rolling Procedure

The materials used in this experiment were 6 kinds of aluminium sheet ingot of 60 kgs in weight whose chemical compositions are shown in Table 1. After scalping their sur-

(1) H. Chossat, *Revue de Metallurgie*, 17 (1950), 167.

they were heated from 100° to 340° at the interval of 20° for one hour and cooled in air. Then their hardnesses were examined by Vicker's hardness tester (300 g/30 sec). The Laue photographs were also taken similarly as mentioned in the previous report.

(3) Results of Experiments

The relations between the hardness and the annealing temperature are graphically shown in Fig. 1. From these curves, it is found that the highest purity aluminium

Fig. 1 Annealing Curves of Various Aluminium Foils (99.5~99.998 % Al)

Fig. 2 The Relation between the Purity and the Temperature Range of Recrystallization of Aluminium Determined by X-Ray Analysis.

aluminium exists between 99.9 and 99.99 % purity, and the beginning temperature of

(99.998 %) recrystallizes at the lowest temperature about 230° and the commonly used high purity aluminium (99.9~99.99 %) recrystallizes at the temperature higher than that of the commercial aluminium (99.5 %).

The recrystallization temperatures determined by X-ray analysis are shown in Fig. 2. Some of the Laue photographs taken are shown in Photo. 1~18. From these results, it is found that a maximum point of the recrystallization temperature in the

Photo. 1 Annealed at 40° for 1 hr.

Photo. 2 Annealed at 220° for 1 hr.

Photo. 3 Annealed at 240° for 1 hr.

Photo. 4 Annealed at 60° for 1 hr.

(Photo. 1~3; 99.998 % Al)

Photo. 5 Annealed at 220° for 1 hr.

Photo. 6 Annealed at 240° for 1 hr.

Photo. 7 Annealed at 160° for 1 hr.

Photo. 8 Annealed at 260° for 1 hr.

(Photo. 4~6; 99.96 % Al)

Photo. 9 Annealed at 280° for 1 hr.
(Photo. 7~9, 99.994 % Al)

Fig. 10 Annealed at 180° for 1 hr.

Photo. 11 Annealed at 280° for 1 hr.

Photo. 12 Annealed at 300° for 1 hr.
(Photo. 10~13; 99.992 % Al)

Photo. 13 Annealed at 220° for 1 hr.

Photo. 14 Annealed at 260° for 1 hr.

Photo. 15 Annealed at 280° for 1 hr.

Photo. 16 Annealed at 200° for 1 hr.

(Photo. 13~15; 99.92 % Al)

Photo. 17 Annealed at 240° for 1 hr.

Photo. 18 Annealed at 260° for 1 hr.

(Photo. 16~18; 99.5 % Al)

the recrystallization is the highest with the 99.92 % purity and its end with the 99.992 % purity. These phenomena coincide with the results obtained from the annealing curves, that is, when the aluminium is more than 99.99 % in purity, the recrystallization begins at a lower temperature, and but it occurs at the higher temperature in the aluminium with less than 99.99 % purity when the purity is higher. As was

reported that the recrystallization was found to occur at the higher temperature when the purity was higher so far as the aluminums of 99.0~99.99% in our previous works was concerned, these phenomena were again ascertained in the present investigation. The recrystallization temperature of the highest purity aluminium with 99.998% is about 230°, but it seems to begin to recrystallize at 40° when heated for one hour so far as the foil aged 1.5 month after final rolling is concerned. However, the recrystallization of the highest purity aluminium begins to occur even at the room temperature after ageing for several months, so it seems that its recrystallization temperature should depend upon the ageing time after rolling.

(4) Consideration

The amount of the impurities in 99.998% aluminium is considered to be in trace and, consequently, they seem to be dissolved in the aluminium, that is, it contains no Cu and Si but only 0.002% of Fe. And the impurities in 99.992% aluminium are 0.003% of Cu, 0.001% of Si and 0.004% of Fe. Then this dissolution of Cu and Si in aluminium effects the remarkable rise of the temperature of recrystallization in 99.992% aluminium in comparison with those of 99.998% aluminium. In the case of 99.92% aluminium, the iron content is distinctly over its solubility limit, but Cu and Si are considered to be in its limit, so that the recrystallization temperature will slightly be lowered by their combined effect as compared with those of 99.992% aluminium. However, it seems that the beginning temperature of the recrystallization will rise gradually in the aluminium from 99.998% to 99.9% purity by the combined effect of Cu and Si which are contained within the solubility limit in spite of the increasing of Fe content from the result of previous report. Further, in the case of 99.5% aluminium, owing to the further increase of Fe and Si the recrystallization temperature seems to fall slightly.

III. Conclusion

From the present investigation, the authors have obtained the following results.

(1) Studying the recrystallization temperature of 6 kinds of aluminium (99.5~99.998%), it was found that there exists a maximum point between them. And this phenomenon was explained by the characteristic effect of each constituent and its content on the recrystallization temperature from the results of previous report.

(2) It was also found that the beginning temperature of recrystallization was the highest with the 99.92% purity and its end temperature was the highest with the 99.992% purity.

(3) The recrystallization temperature of the highest purity aluminium with 99.998% is about 230°, but it seems to begin to recrystallize even at the room temperature after ageing for a few months.

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