

# On the Age-Hardening of Al-Cu-Mg Alloys.

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## Introduction

The present investigation has been carried out to ascertain the cause of the age-hardening of Duralumin and 24-S type-Duralumin. These alloys age-harden markedly at room temperature when they are quenched from such a temperature as 500°C, but the Al alloys containing Cu or Mg<sub>2</sub>Si only do not show at room temperature such a marked age-hardening as Duralumin. Hitherto the cause of the age-hardening of Duralumin is explained to be due to the decrease of the solid solubility of Cu or Mg<sub>2</sub>Si in Al with the fall of temperature similarly as is the case of those of the alloys containing only Cu or Mg<sub>2</sub>Si. However, this elucidation does not apply to the age-hardening of Duralumin at room temperature and to the necessity of tempering of the alloys containing Cu or Mg<sub>2</sub>Si. Hence the present study has been taken up to solve this contradiction.

## Age-Hardening and Equilibrium Diagram of the Al-Cu-Mg System.

The present writer has published his own investigation on the equilibrium diagram of Al-rich Al-Cu-Mg alloys. From his researches he found the existence of 2 ternary compound Al<sub>13</sub>Cu<sub>7</sub>Mg<sub>8</sub> and Al<sub>6</sub>CuMg<sub>4</sub> denoted S and T respectively to be in equilibrium with Al-solid solution.

The question arises here if this equilibrium enumerates the phenomena of ageing of Duralumin or 24-S type Super-duralumin, which are (as described above) different from those of the alloy containing Cu or Mg<sub>2</sub>Si only. Assuming now Duralumin to be an aluminium alloy containing Cu 4% and Mg 0.5%, we can see from the diagram that it consists of CuAl<sub>2</sub> and S-compound in the matrix of Al-solid solution, if it be cooled slowly. Considered similarly, 24-S type Super-Duralumin can be said to be an alloy on a pseudo-binary system between Al and S-compound.

If such alloys are quenched from higher temperature and age-hardened at room temperature, the hardening may be considered to arise principally in the course of precipitation of S-compound. This consideration, however, must be proved by experiments to be correct or not. Therefore we have made the present experiments as described later.

## Age-hardening of Chill-Cast Al-Cu-Mg Alloys.

### Method of Experiments.

Chill cast specimens 10mm wide, 5 mm thick and 30mm long, were polished and solution-treated

at 500°C for 16 hours. They were again heated at 500°C for 30 min. and quenched in water at 15°C. The change of hardness on ageing was measured with a small Brinell tester. The specimens employed were those alloys corresponding on the 8 sections A, B, C, D, E, F, G, H and the alloys containing Cu only, as shown in Fig. 1, their analyses are given in table 1.

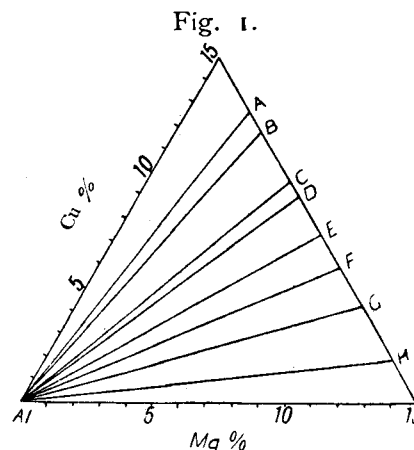


Fig. 1.

### Age-hardening of Al-Cu-Alloys.

Fig. 2 represents the phenomena of age-hardening of the alloys containing Cu only at room temperature. As is well-known, the hardening of these alloys is observed to occur less markedly than that of the alloys containing Cu and Mg together. In Fig. 3 the relation between the copper content and the degree of hardening due to ageing is shown with the diagram of Al-Cu at the Al-end. From this figure the degree of hardening due to natural ageing is seen to be at the most 15-20%, i.e., the alloys containing Cu do not age-harden so much at the room temperature.

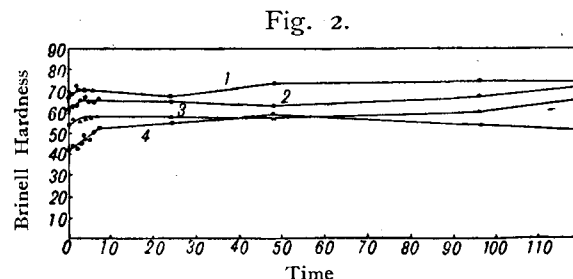


Fig. 2.

### Age-hardening of Al-Cu-Mg Alloys.

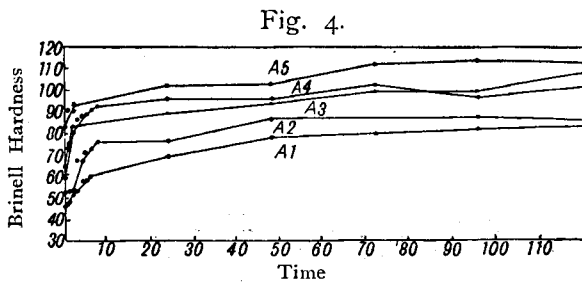
To examine the effect of Mg on the age-hardening of Al-Cu alloys, similar experiments were carried out on the alloys of the sections A, B, C, D, E, E, G, and H as shown in Table 1.

### Age-hardening of the Alloys of Section A.

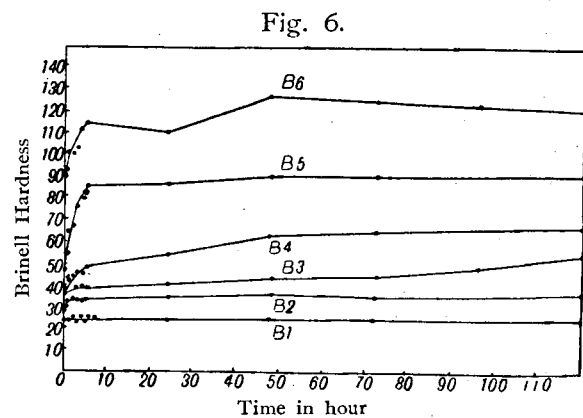
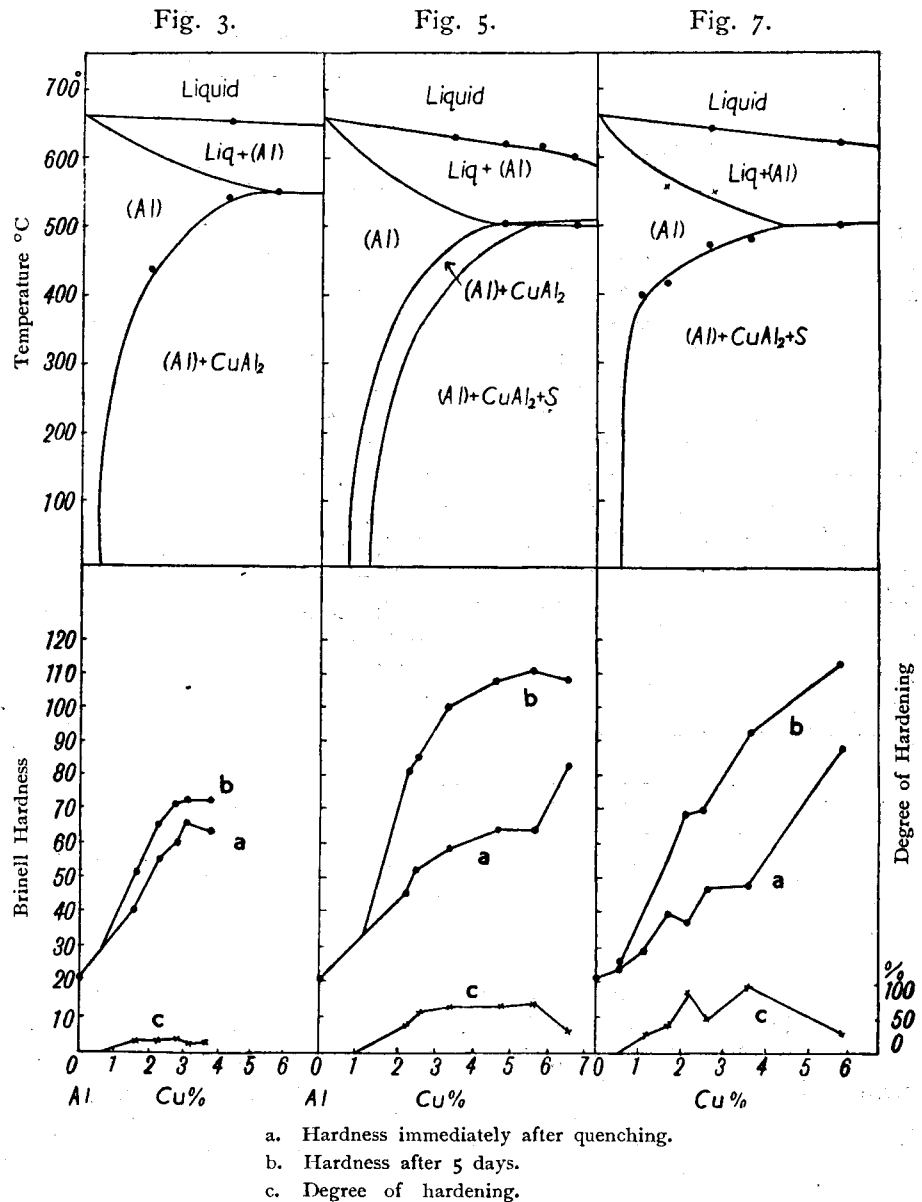
The ratio of Cu and Mg in this section was about 7:1. The change of hardness of the alloys

Table 1.

No.	Composition %		
	Cu	Mg	Al
1	2.29	—	Balance
2	2.79	—	"
3	3.15	—	"
4	3.74	—	"
A 1	0.34	0.10	"
A 2	2.72	0.21	"
A 3	3.51	0.40	"
A 4	4.74	0.94	"
A 5	5.62	1.07	"
A 6	6.73	1.25	"
B 1	0.34	0.10	"
B 2	1.10	0.26	"
B 3	1.51	0.12	"
B 4	2.20	0.49	"
B 5	3.72	1.05	"
B 6	6.01	1.72	"
C 1	1.63	0.94	"
C 2	1.94	1.17	"
C 3	2.81	1.74	"
C 4	4.01	2.40	"
C 5	4.90	2.90	"
D 1	0.83	0.81	"
D 2	1.12	0.82	"
D 3	1.59	1.33	"
D 4	1.89	1.43	"
D 5	2.37	1.87	"
D 6	3.13	2.34	"
D 7	4.40	3.22	"
E 1	0.43	0.41	"
E 2	0.98	1.10	"
E 3	1.83	1.75	"
E 4	2.02	1.93	"
F 1	0.78	0.93	"
F 2	1.23	1.64	"
F 3	1.42	1.98	"
F 4	1.57	2.35	"
F 5	4.02	6.07	"
G 1	1.06	2.34	"
G 2	1.74	4.61	"
G 3	2.73	6.87	"
G 4	3.20	8.08	"
H 1	0.80	5.46	"
H 2	0.83	7.13	"
H 3	1.12	8.50	"
H 4	1.32	10.24	"
H 5	1.68	11.20	"



quenched at 500°C and aged at the room temperature is shown in Fig. 4. Even if the hardening in 5 days is different according to the composition, it is, in general, marked and reaches 60–70% at the maximum. In Fig. 5 we see the relation between the hardening and the equilibrium section

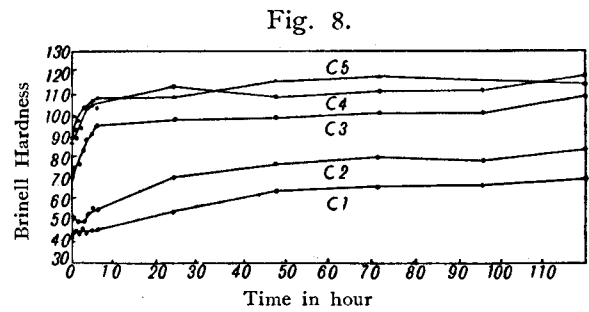


of these alloys. These alloys are cooled in equilibrium by the separation of  $\text{CuAl}_2$  and then  $\text{CuAl}_2$  and S-compound from Al-solid solution. If they are quenched at the temperature as 500°C, the solid solution is super-cooled for  $\text{CuAl}_2$  and S-compound but its super saturation is not kept and

the separation of  $\text{CuAl}_2$  or S-compound will be excited. In the course of this precipitation the hardening takes place spontaneously. As described later in detail, the separation of S-compound proceeds easily at the room temperature and  $\text{CuAl}_2$  are not separated so much. This is the explanation of the natural ageing of the Al alloys containing Cu and Mg, which is different in phenomena of ageing from the alloy containing Cu only.

#### Age-hardening of the Alloys of Section B.

The alloys of this section contain Cu and Mg nearly in the proportion of 5:1.5. The change of hardness due to ageing is shown in Fig. 6. The alloy B1 (Cu 0.34%, Mg 0.10%) does not show hardening, but the alloy B5 (Cu 3.7%, Mg 1.05%) hardens in 5 days about 90%. The relation between these hardenings and the equilibrium section is also observed in Fig. 7. This section corresponds to the alloys on the line connecting from Al to the ternary eutectic point liquid  $\rightleftharpoons (\text{Al}) + \text{CuAl}_2 + \text{S}$ . Therefore,  $\text{CuAl}_2$  and S-compound separate together from Al-solid solution on slow cooling. Being quenched, these alloys age harden more



marked than the alloys of the A section. This proves that the presence of Mg affects the natural ageing of Al-Cu-Mg Alloys. Here we can also say that the solubility of S-compound in Al has a great influence upon the ageing of Al-Cu-Mg alloys.

#### Age-hardening of the alloys of the section, C, D, and E.

As above described, we recognized distinctly the quench-hardening of the alloys whose compositions exist in the domain where  $\text{CuAl}_2$  and S-compound are the phases to be separated from the Al-solid solution. There remains however, the question how the hardening will occur in the

alloys whose compositions correspond to the sections where S-compound is the only phase to precipitate from Al-solid solution. For this purpose the alloys of the sections C, D and E were similarly treated and the change of hardness was measured. Fig. 8, 10 and 12 show the results of the experiments. Among the alloys of the section C, C1 and C2, whose Cu and Mg content are not so much, show a somewhat gradual hardening i.e., incubation, but C3, C4 and C5 did not show such a phenomenon of incubation. It is evident from the diagram shown in Fig. 9 that the age-hardening of these alloys takes place in the course of the precipitation of the ternary compound S.

Fig. 10 and 12 illustrate the phenomena of hardening of the alloys of the sections D and E due to ageing. The alloys of the section D show a similar change to those of the section C, but those of the

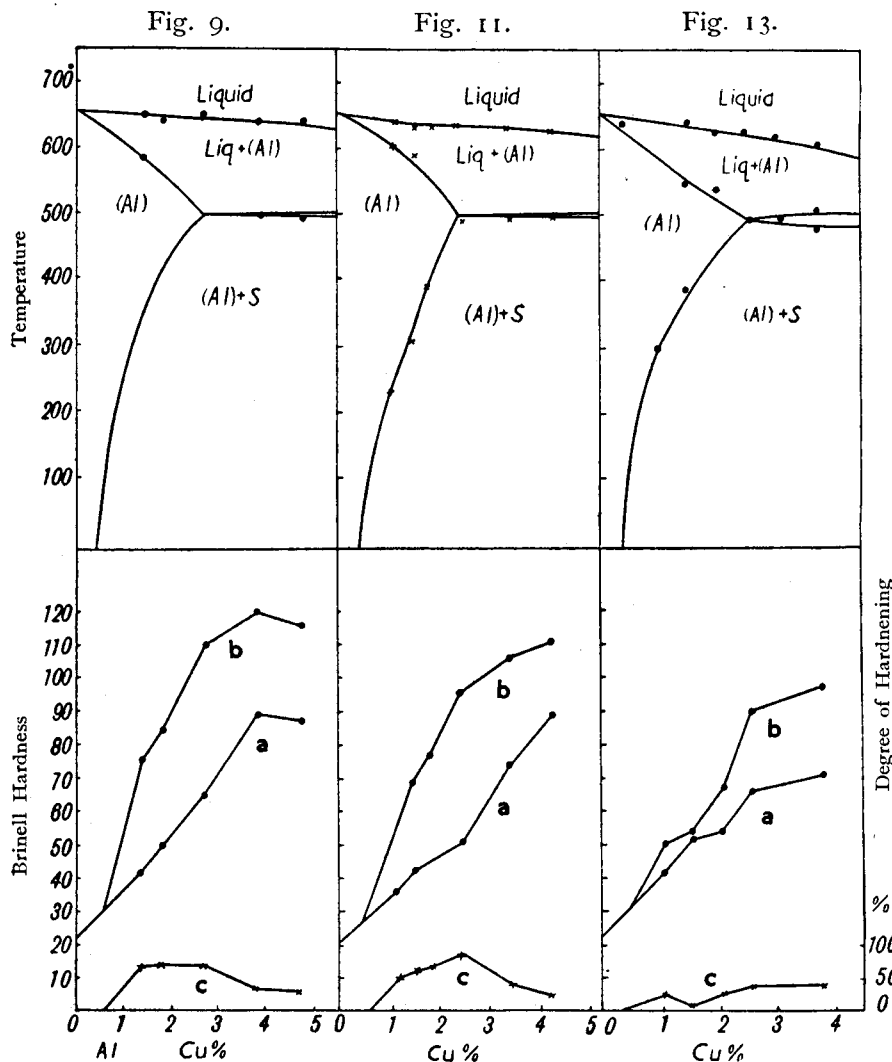


Fig. 10.

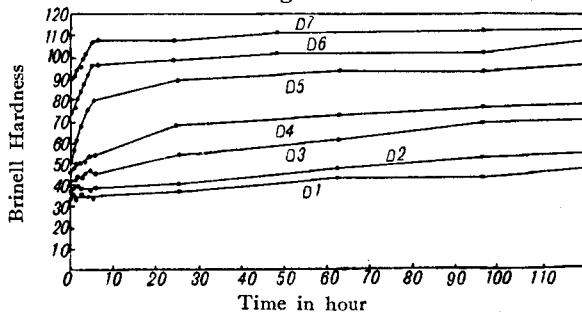


Fig. 12.

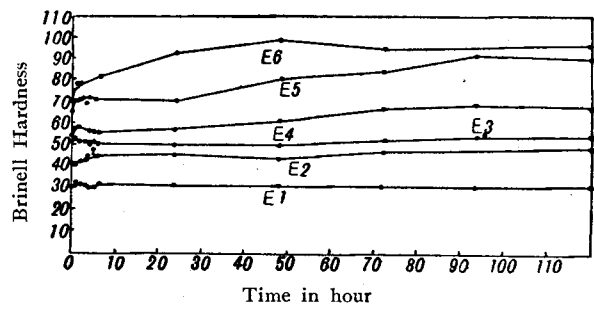


Fig. 14.

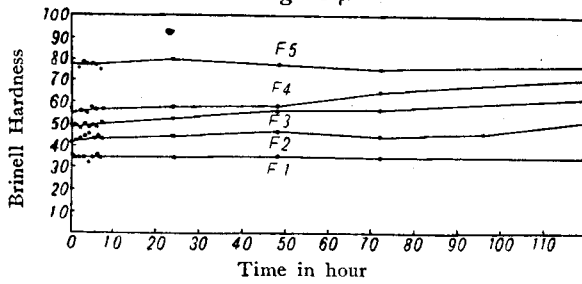


Fig. 16.

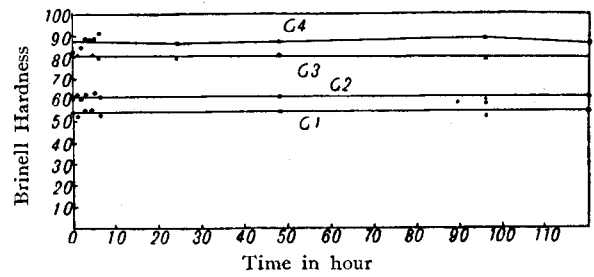


Fig. 15.

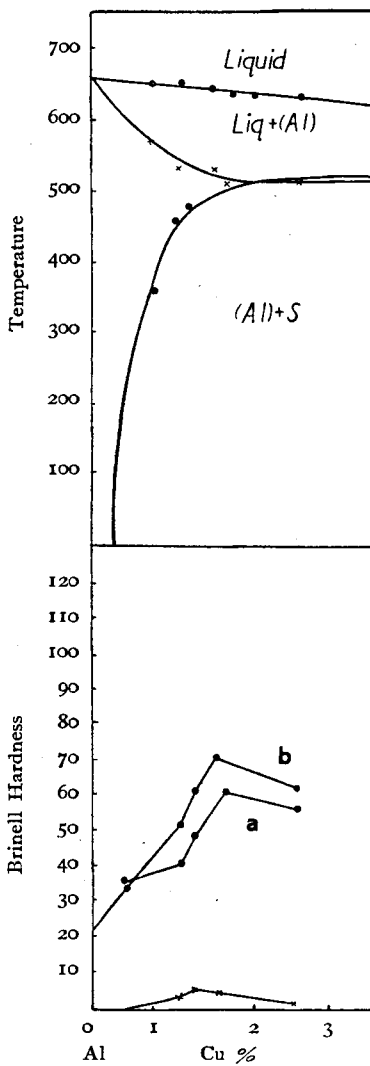


Fig. 17.

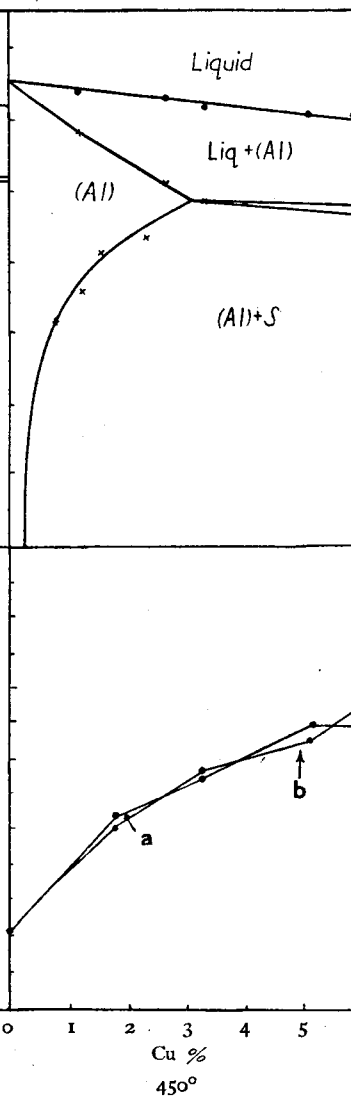


Fig. 19.

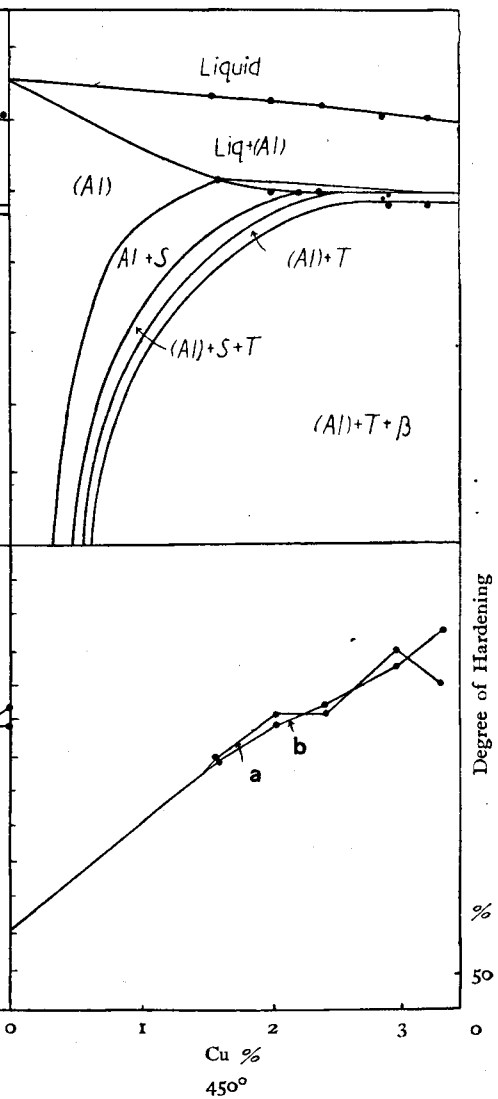
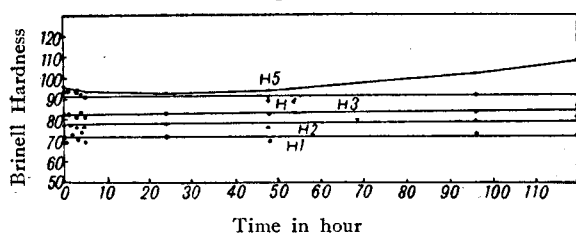


Fig. 18.



section E harden in less degree and the period of incubation is somewhat long. The hardening of the alloys of the section E is only 40% at the maximum as seen from Fig. 13.

*Age-hardening of the alloys of the sections F, G and H.*

The alloys of the sections F, G and H contain Cu and Mg in the proportion of 2.5:4, 2:5 and 1:7 respectively. As shown in Fig. 14, 16 and 18. These alloys do not show any marked hardening or do not harden at all at the room temperature according to the composition even if they are quenched, and aged. Fig. 15, 17 and 19 illustrate well these relation between the hardening and the equilibrium sections.

Summarizing the above described experiments, the natural ageing is not marked in the alloys containing Cu only, however, the alloys containing Cu and Mg together in the domain where the S-compound is to separated show a marked age-hardening at the room temperature. If the ratio Cu and Mg contained in the alloys surpasses 4:1.5, the degree of hardening decrease gradually with the increase of Mg content, and the phenomenon of incubation is recognized. From these phenomena we may conclude that the natural ageing of the Al alloys containing Cu and Mg is produced in

the course of the precipitation of S-compound, although we must consider the mechanism of hardening to be different from the cause of age-hardening.

**Effect of Tempering.**

As the compositions of the alloys to show age-hardening have been determined, the effect of tempering upon these alloys was studied by the measurement of hardness. Similar specimens to those used in the above experiments were quenched from 500°C in water at 15°C and then they were tempered at 50°C and 100°C in air bath for 5 hours. At each hour the hardness was measured. The reason for not tempering at 150–170°C as usual in the tempering of Al alloys was to observe whether they harden at a lower temperature than 100°C.

*Tempering of Al-Cu alloys.*

Results of the experiments are shown in Fig. 20. It is natural that these alloys do not harden by tempering at 50°C and 100°C, as they must be tempered at 150–170°C to obtain their maximum hardness.

*Tempering of the alloys of the section A.*

Fig. 21 shows results of the experiment upon the alloys of the section A. As seen in Fig. the effect of Mg is well recognized in the tempering of these alloys, i.e. they were hardened by this treatment, but the degree of hardening was somewhat larger when they were tempered at 50°C than at 100°C. The hardness of these alloys aged at the room temperature for 5 days was also higher than that of the tempered alloys. We can say that the ageing proceeds well at the room temperature in those alloys containing Cu and Mg. In other words, the content of Mg has a great in-

Fig. 20.

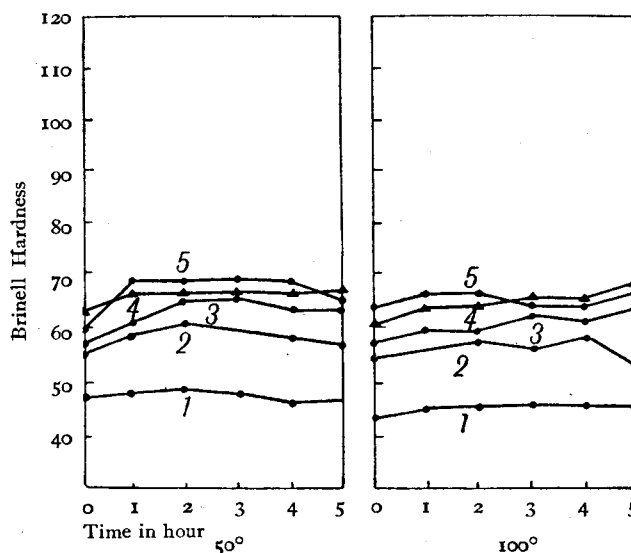


Fig. 21.

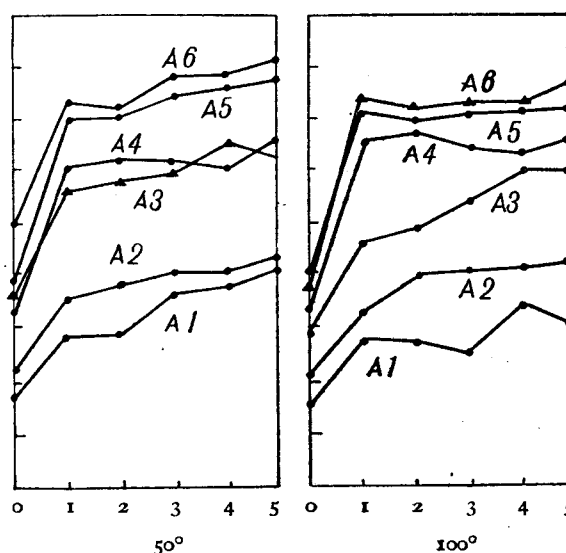


Fig. 22.

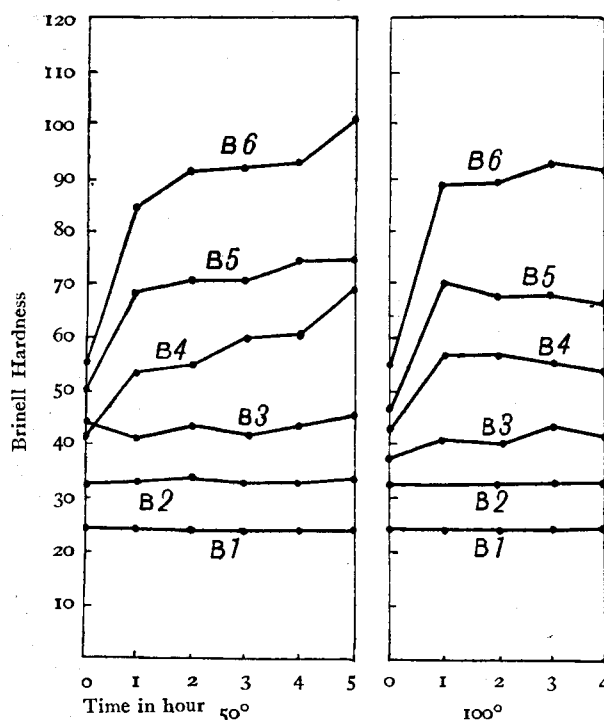
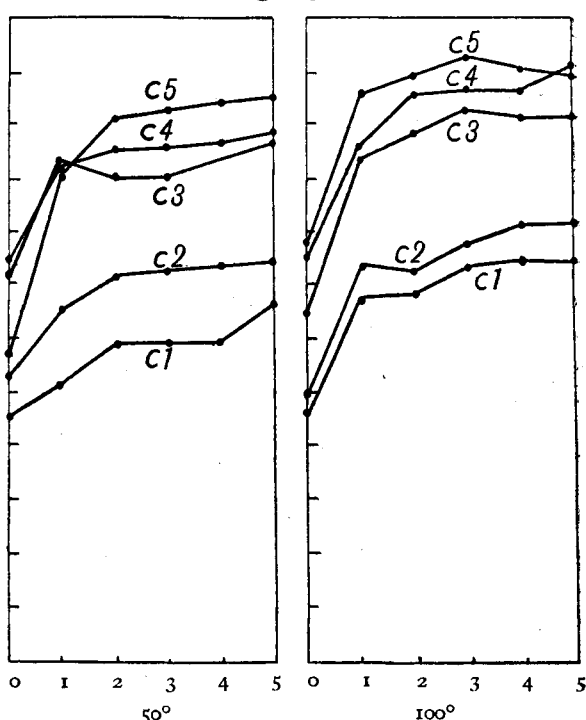


Fig. 23.



fluence upon the ageing of Al-Cu-Mg alloys, i.e. the age-hardening at the lower temperature is no doubt due to the separation of S-compound from the super-cooled Al-solid solution.

#### *Tempering of the alloys of the section B.*

From Fig. 22, we see distinctly that these alloys harden more by tempering at 50°C than at 100°C., and some alloys begin to soften by tempering for 4 hours.

#### *Tempering of the alloys of the sections C and D.*

The alloys of these sections show distinct age-hardening at the room temperature and it is evident from the equilibrium diagram already published that the age-hardening is due to the precipitation of S-compound and not  $\text{CuAl}_2$ . Fig. 23 and 24 show the hardness of the alloys of these sections tempered at 50°C and 100°C. Contrary to the case of the alloys of the sections A and B, the hardening was accelerated by tempering at 100°C. This elucidates the phenomenon that the hardening of the alloys is accelerated by the tempering at higher temperature if they contain Mg and Cu more than the proportion of 4:1.5.

#### *Tempering of the alloys of the sections E, F and G.*

These alloys contain Mg more than the alloys above described. Fig. 25-27 show the results of the similar experiments. The alloys of the section E and F show also the same tendency that the tempering at higher temperature is effective to make

them harden. However, the alloys of the section H are observed not to harden by the tempering at such temperature as 50°C and 100°C.

Summarizing the results of the experiments we conclude as follows. The Al-alloys containing Cu only do not show any age-hardening at the room temperature. If Mg is added to the alloy, age-hardening at the room temperature is observed to occur distinctly in the alloys containing Mg in the proportion of Cu to Mg more than 4:3. If it surpass 1:1, the phenomenon of incubation appears and it reaches the point of not showing age-hardening at all as in the alloys of the section G and H.

From these phenomena, we can say that the age-hardening occurs in a different manner with the section containing Cu and Mg in a 4:1.5 proportion as a boundary. Referring to the equilibrium diagram Al-Cu-Mg, the Al-rich alloys containing Cu and Mg less than the proportion 4:1.5 consist of (Al),  $\text{CuAl}_2$  and S-compound at the room temperature in equilibrium. If they are quenched from 500°C, it may be considered that the separation of S-compound is accelerated at the room temperature and it gives rise to the hardening.

The alloys containing Mg more than in this proportion, however, consist of at the room temperature (Al) and S-compound. Such alloys do not show so marked age-hardening, if Mg is contained more than in the proportion  $\text{Cu}:\text{Mg}=1:1$ . This will be explained by the fact that the Al-solid solution be easily super-cooled by quenching as in Al-Cu alloys or Al-Mg alloys.

Fig. 24.

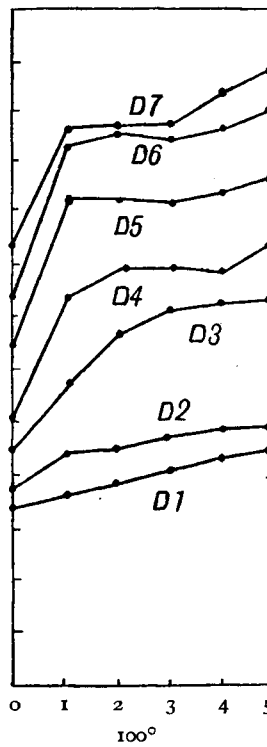
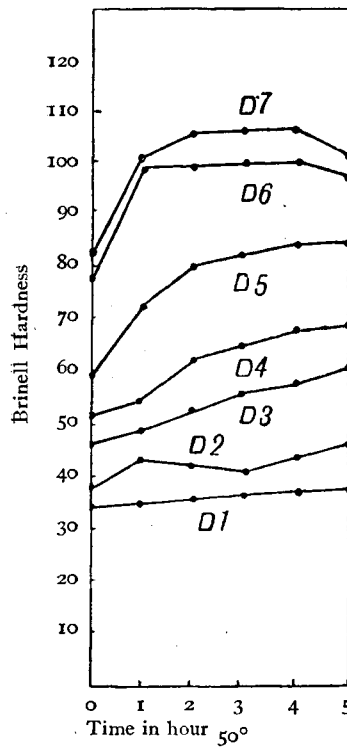


Fig. 25.

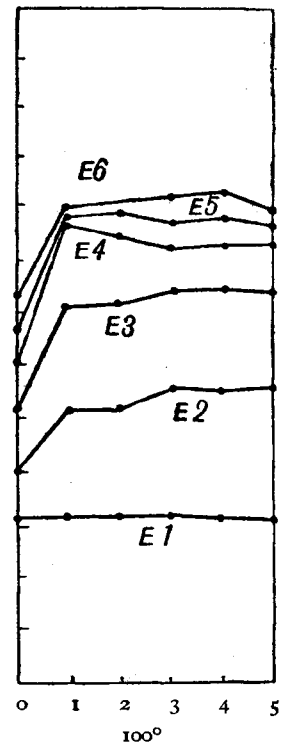
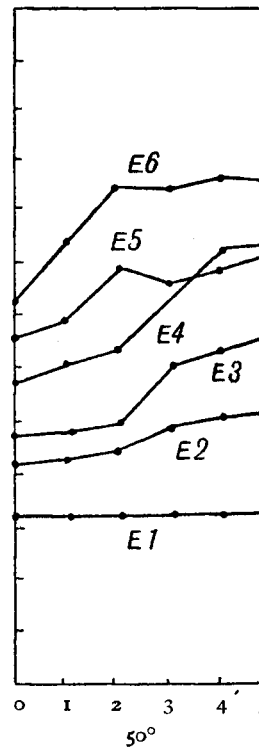


Fig. 26.

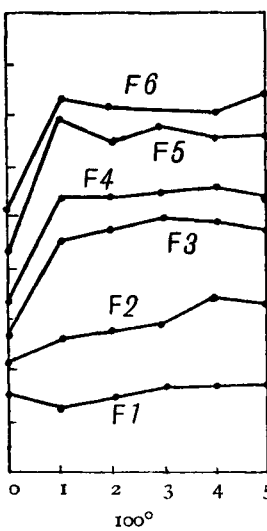
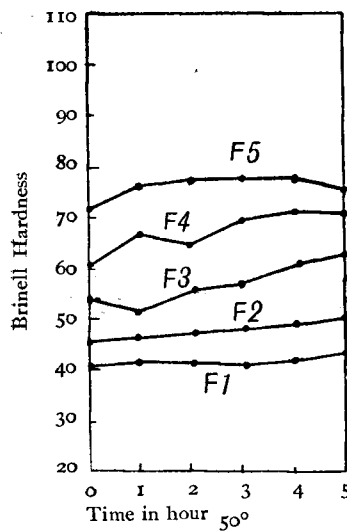
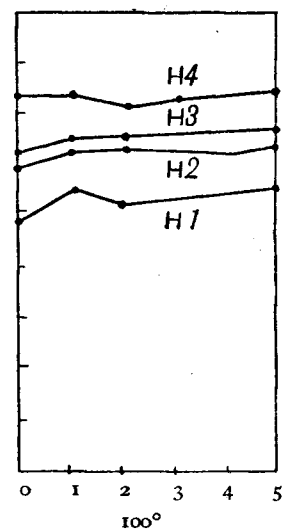
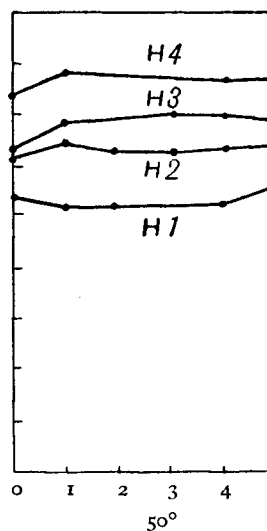


Fig. 27.



#### Age-hardening of Wrought Alloys.

The phenomena of the age-hardening of Al-Cu-Mg alloys have been examined thoroughly in chill-cast specimens, and age-hardening at the room temperature was observed to occur distinctly in the alloys containing Cu and Mg whose proportion was less than 1:1. It was most evident in the alloys containing Cu and Mg in the approximate proportion 4:1.0 to 4:1.5.

From these results we have undertaken to study the phenomena of ageing upon the wrought alloys which contain Cu and Mg in these proportions.

#### The Method of Experiments.

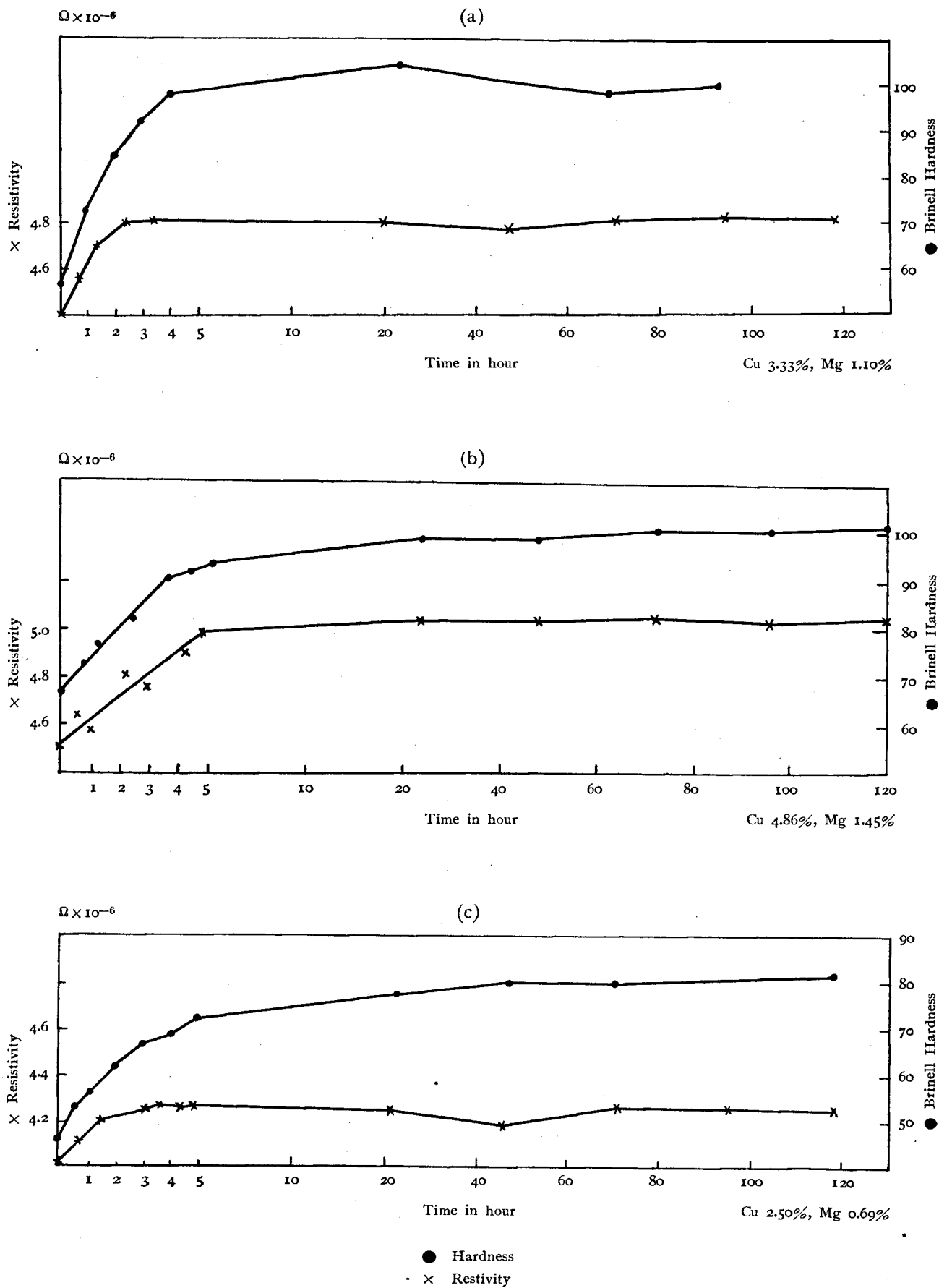
Table 2.

No.	Composition %		
	Cu	Mg	Al
K 1	3.33	1.10	Balance
K 2	3.86	1.45	"
K 3	2.50	0.69	"
K 4	2.35	1.52	"
× R 1	4.01	1.77	"
× R 2	4.09	1.12	"

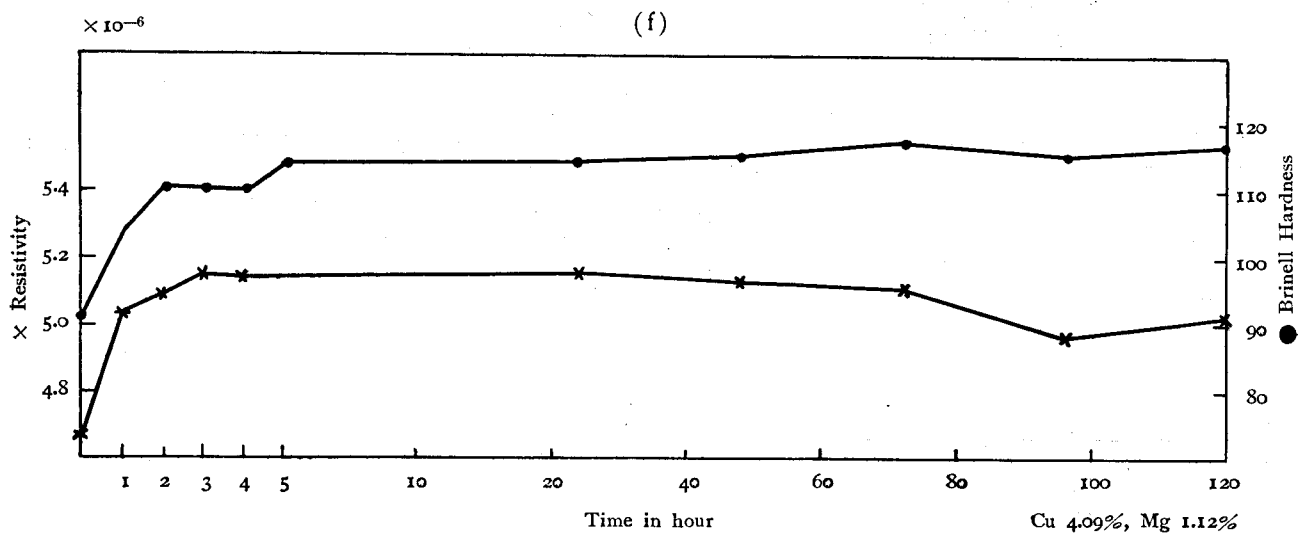
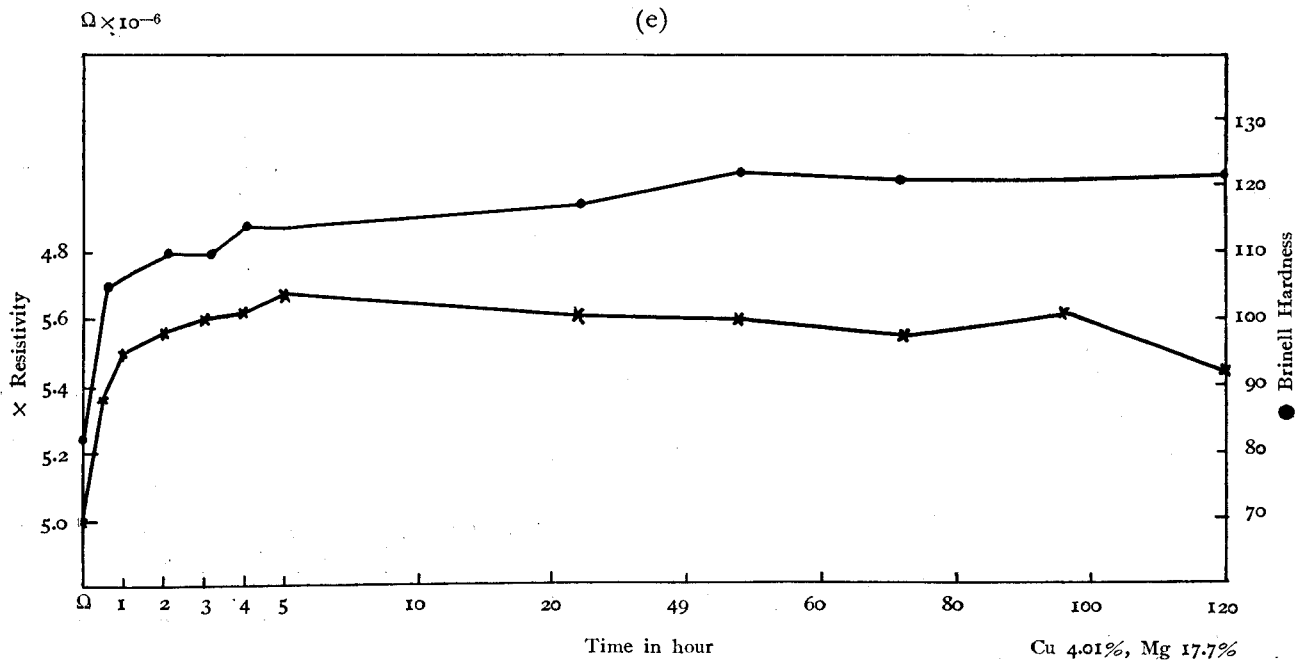
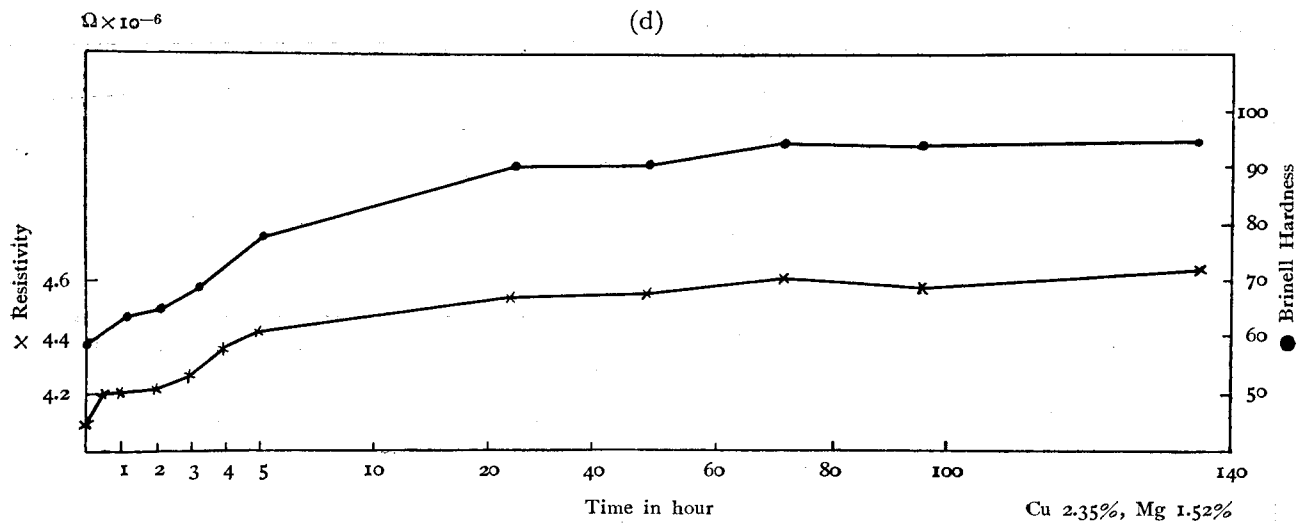
× Specially prepared with Al 99.992%.

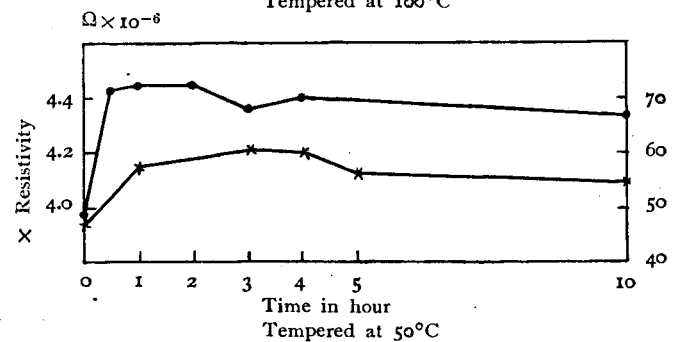
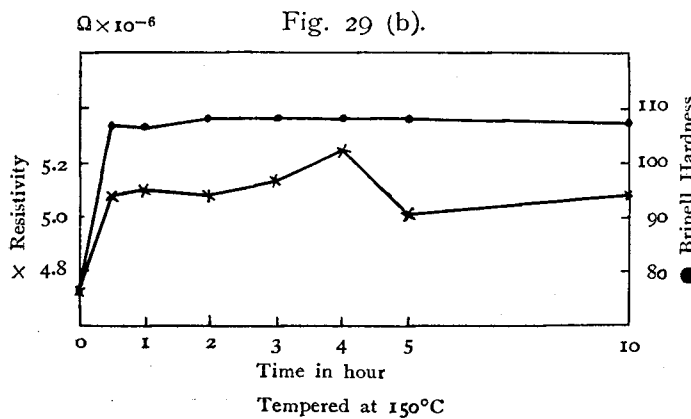
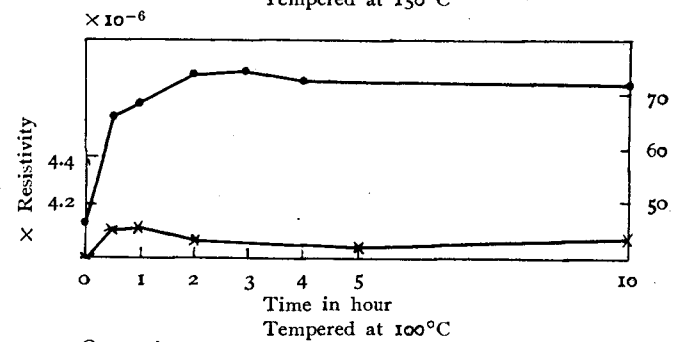
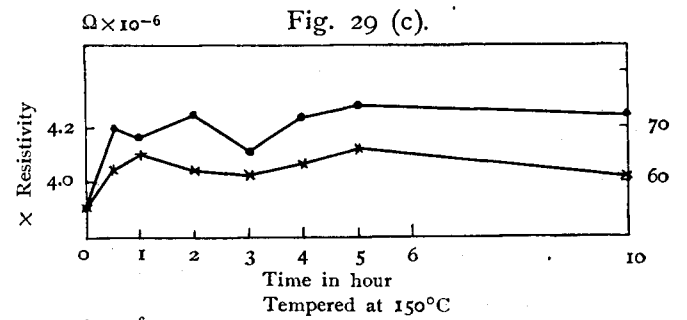
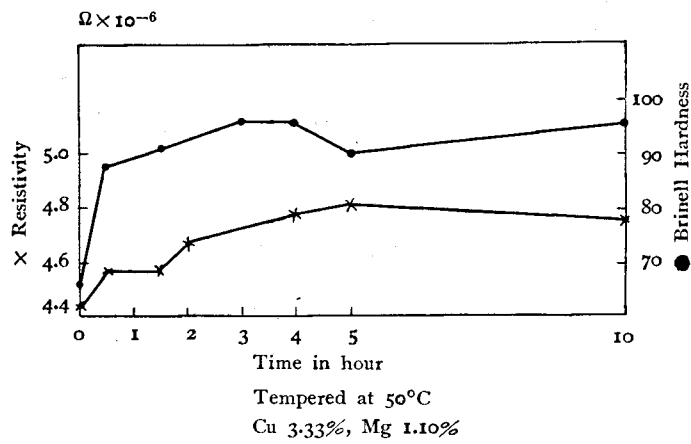
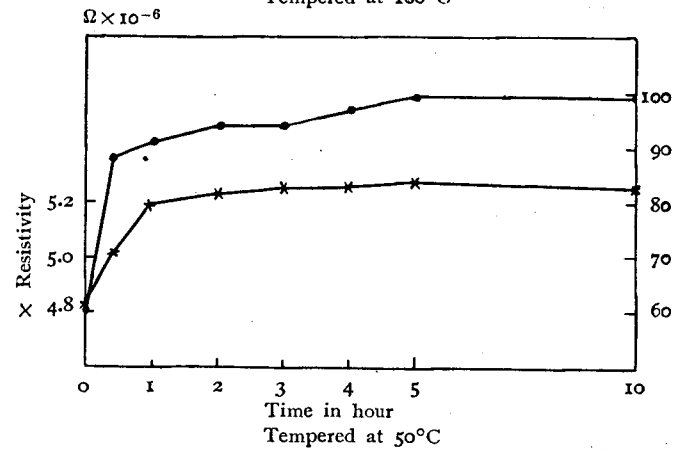
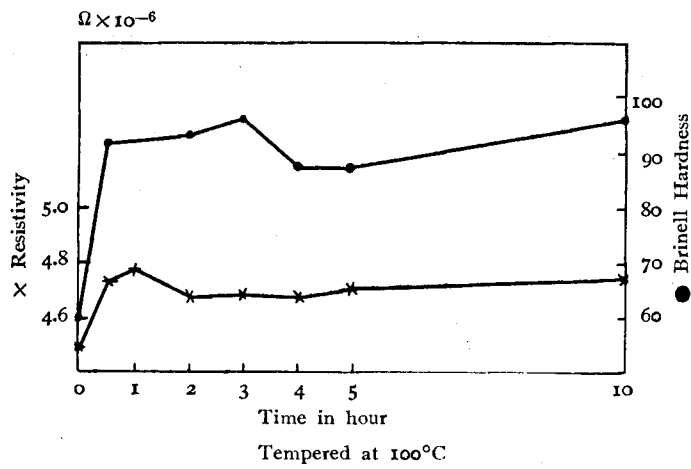
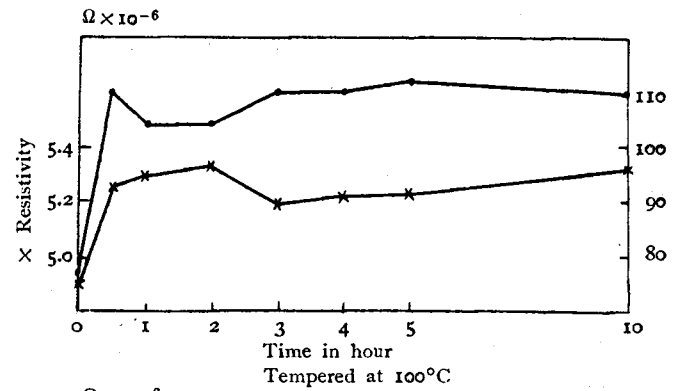
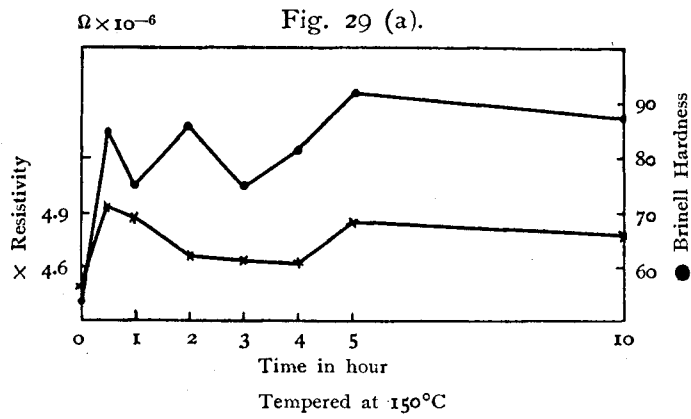
The alloys shown in Table 2, were prepared in the chill cast specimens as already described, were heat treated and rolled into sheet about 0.8 mm thick. They were heated at 500°C for 30 min and quenched in water. Their hardness was measured. Electric resistance was also

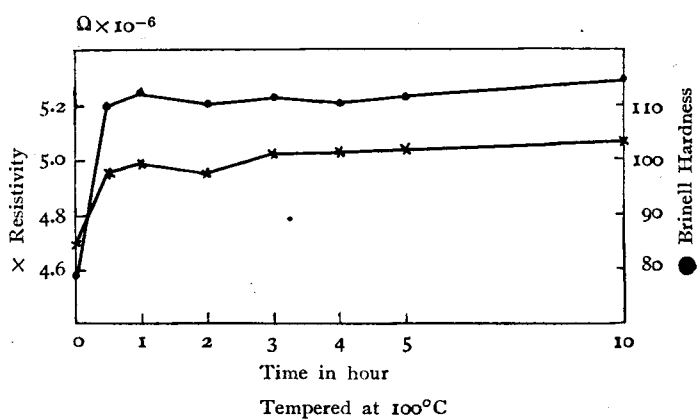
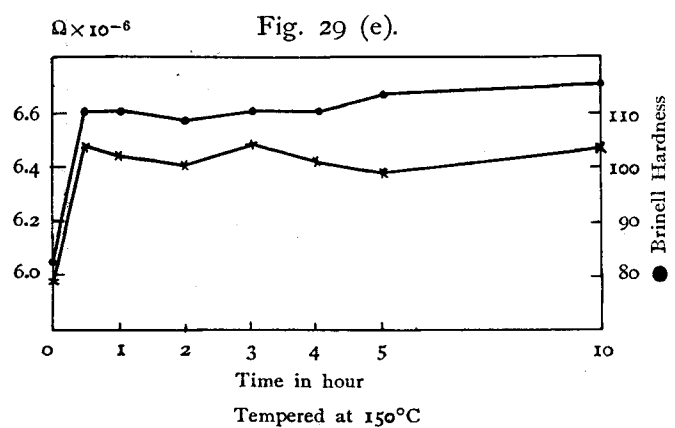
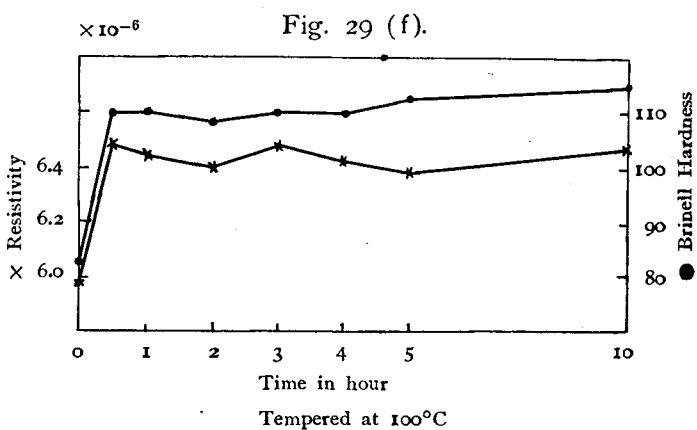
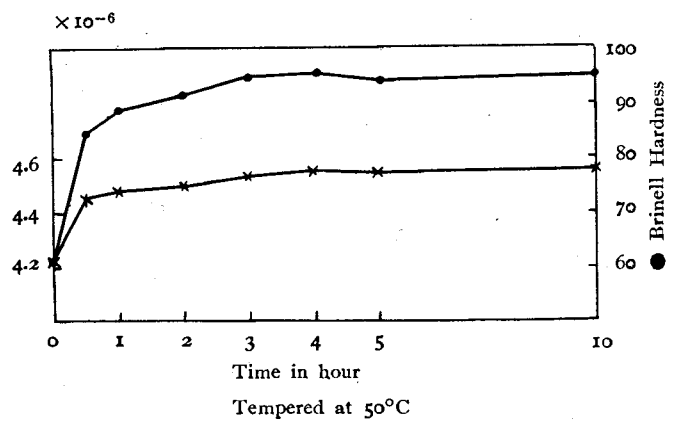
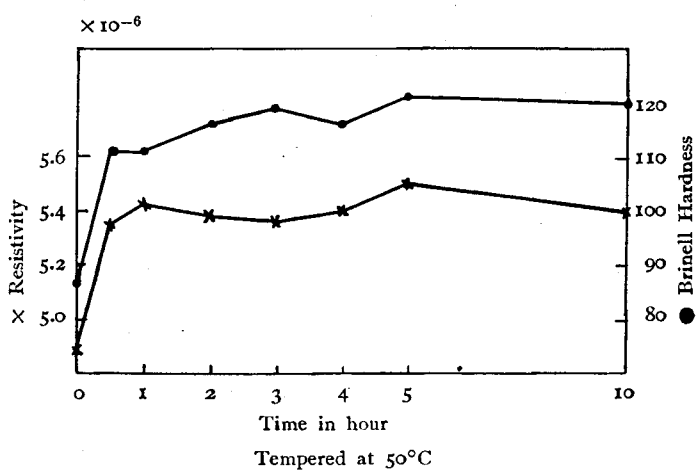
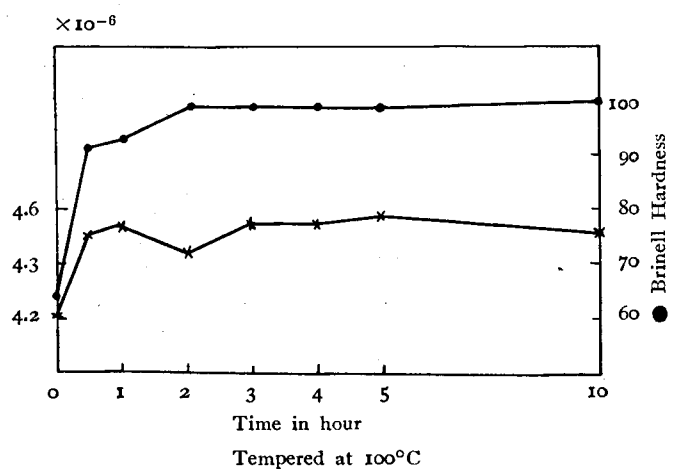
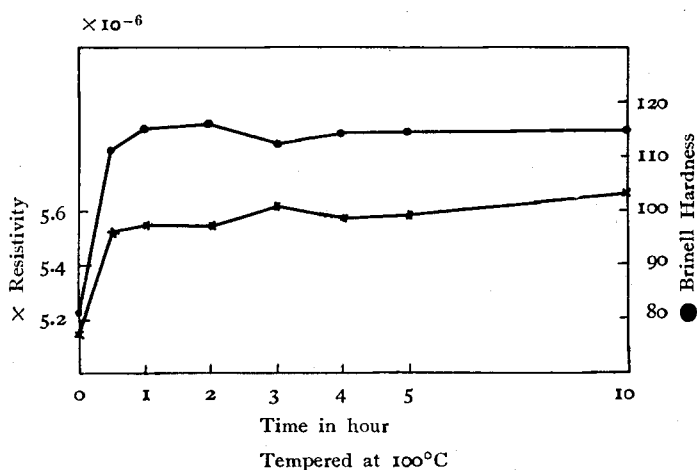
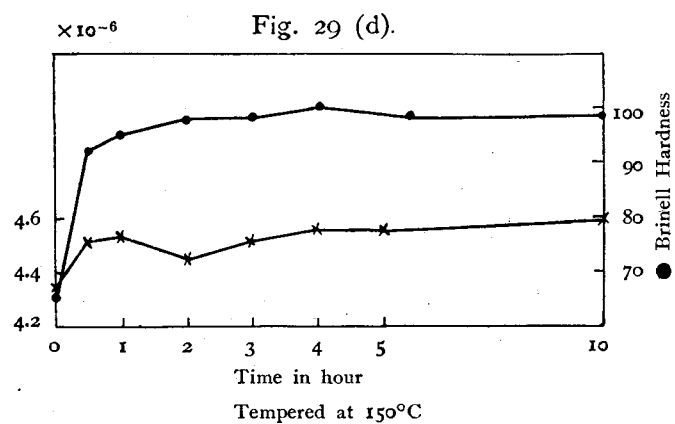
Fig. 28.

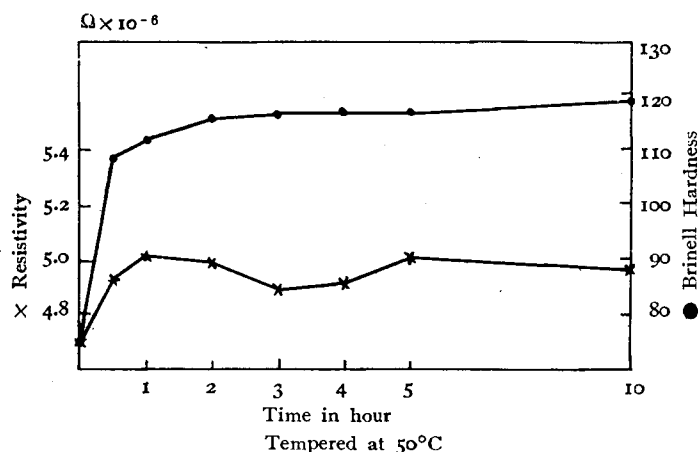












measured at 30°C by keeping the specimen in a thermostat to observe how the ageing proceeds.

### Result of the Experiments.

Fig. 28 illustrate the change of hardness due to ageing at the room temperature. They show also as marked hardening as the chill cast alloys. The electric resistance changes with the hardness, but it reaches its max. value sooner than the hardness does.

These specimens were tempered at 50°C, 100°C and 150°C for 10 hours. The hardness at each hours heating till 5 hours and 10 hours after was measured. The results of measurement are shown in Fig. 29. From the Figs. we see that the hardness of these alloys become rather less by tempering at a higher temperature. This phenomenon is more distinct in the wrought alloys than in the chill cast alloys.

### Measurement of the Dilatation of Quenched Specimens.

As often described, the alloy containing Cu only does not show any marked age-hardening at the room temperature, but the alloy containing Cu and Mg as Duralumin or 24-S type Super-Duralumin shows age-hardening considerably.

This difference has been explained from the equilibrium diagram Al-Cu-Mg given by the present writer, by the fact that a ternary compound denoted S is apt to separate easily from the Al-solid solution super-cooled by quenching, but that CuAl<sub>2</sub> is not so easy to be separated at the room temperature.

To give to this explanation a more exact proof, the specimens whose composition are given in Table 3, were quenched from 500°C after heating for 16 hours. Immediately they were set in a differential dilatometer, and the change of dilatation was measured on heating. The heating was

made very slowly as its rate was 1°C per min or less.

The results of measurement are shown in Fig. 30. From the Fig. we see that the alloy containing Cu

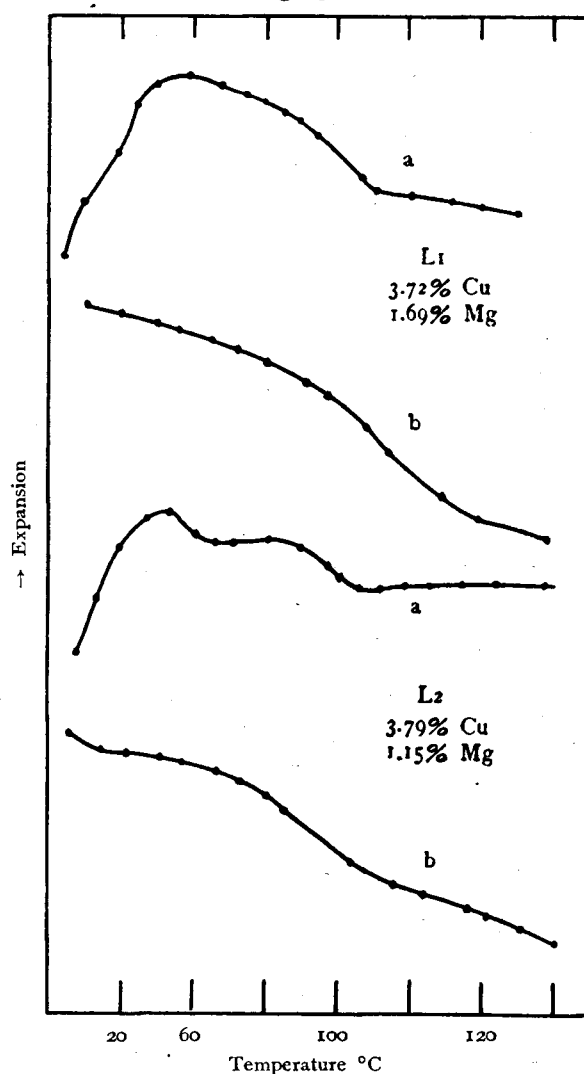
4% does not show marked irregular change of dilatation till 200°C but from about 220°C it begins to expand markedly and after reaching its maximum it commences to contract from about 280°C as this irregular change of dilatation is already reported by Kokubo. This irregular change of dilatation is evidently due to the separation of CuAl<sub>2</sub> from Al-solid solution. But the alloy containing Cu 4.24% and Mg 0.56%

Table 3.

No.	Composition %		
	Cu	Mg	Al
P 1	4	—	Balance
P 2	4.24	0.56	"
P 3	4.12	1.15	"
P 4	3.75	1.15	"
L 1	3.72	1.69	"
L 2	3.79	1.15	"

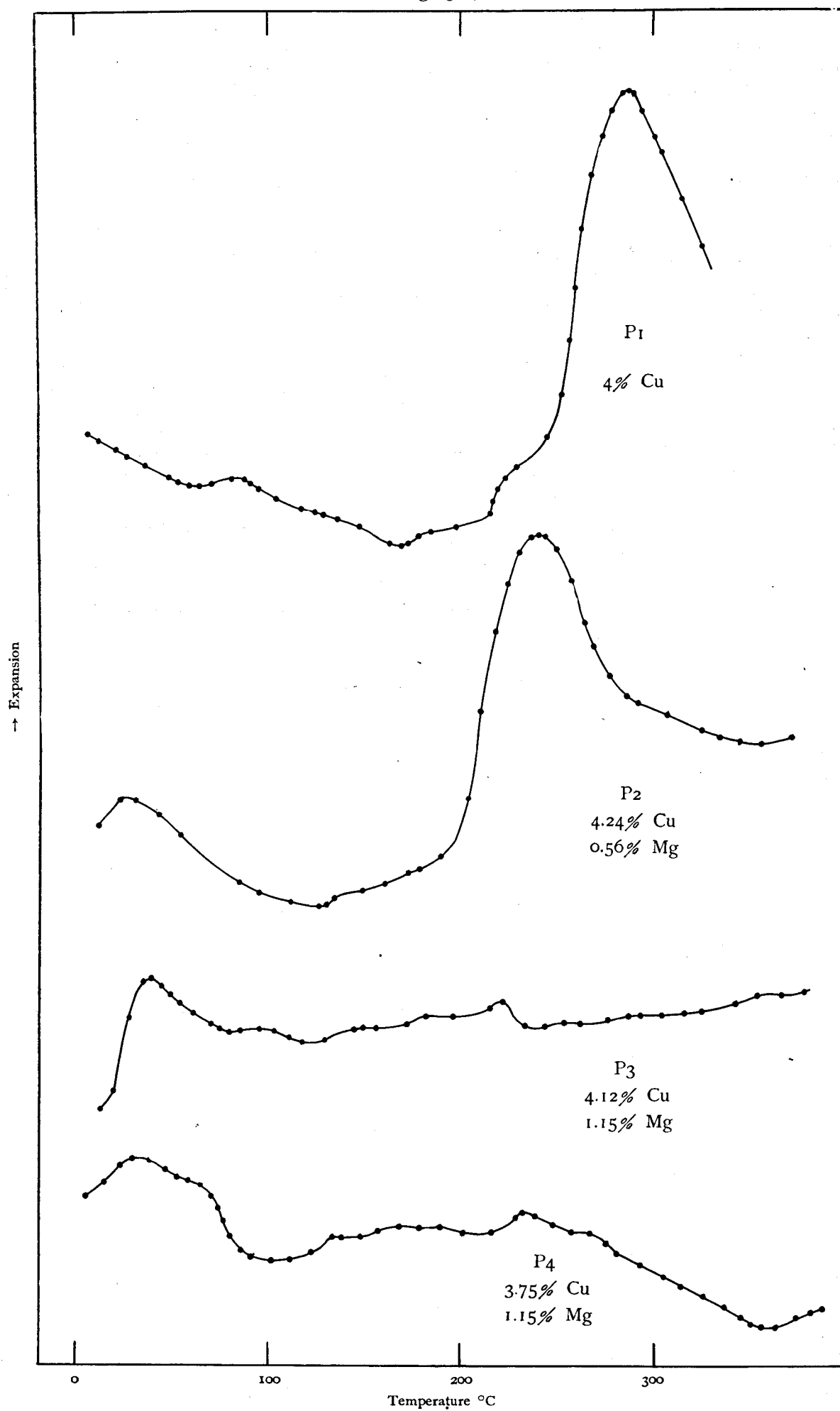
× Specially prepared with Al 99.992%.

Fig. 31.



a. Immediately after quenching at 500°C  
b. 1 week after quenching at 500°C

Fig. 30.



begins to expand slightly from the room temperature and the contract again from the temperature. It also shows the irregular change of dilatation due to the separation of  $\text{CuAl}_2$  at about  $230^\circ\text{C}$ . However, the alloy containing Cu 4.12% and Mg 1.13% shows more markedly an expansion and contraction below  $100^\circ\text{C}$ , but the marked irregular change of dilatation due to the separation of  $\text{CuAl}_2$  is not observed to occur as in the alloys above described.

We observed a similar change of dilatation in the alloys containing Cu 3.75% and Mg 1.15%. This expansion and contraction below  $100^\circ\text{C}$  may be considered to be a phenomenon to occur in the course of the separation of S-compound because this phenomenon is marked with the increase of Mg content in the alloys.

We measured the change of dilatation of the specimen aged for 1 week after being quenched. The results are shown in Fig. 31. This experiment showed that the expansion and contraction below  $100^\circ\text{C}$  is observed to occur, slightly or not to occur at all. Therefore this expansion and contraction below  $100^\circ\text{C}$  is evidently a phenomenon to occur with the age-hardening.

From these experiments our conclusion is that

the age-hardening of Duralumin or 24-S type Super-duralumin occurs in the course of the separation of S-compound and not that of  $\text{CuAl}_2$  or  $\text{Mg}_2\text{Si}$ .

### Conclusion.

Summarizing the present experiments, we conclude as follows.

1) Age-hardening of Al-rich Al-Cu-Mg alloys was examined and the domain of the alloys to show age-hardening at the room temperature was determined. It was found that the alloys containing Cu and Mg in the proportion of 4:1 to 4:1.5 show marked age-hardening.

2) The separation of S-compound found in Al-Cu-Mg system is considered to be the cause of the natural ageing of Duralumin or 24-S type Super-Duralumin. This was proved by the change of dilatation of the specimens on heating immediately after quenching.

### Acknowledgement.

In conclusion, we express our sincere thanks to Hattori-Hokokwai a subsidy from which enabled the present research to be undertaken.

# On the Equilibrium Diagram of Nickel-Aluminium Alloys.

By Hideo Nishimura and Satoshi Watanabe.

## Introduction.

The equilibrium of Nickel and Aluminium alloys has been investigated by many authors, but the diagram given by Gwyer in 1908 was the only one which is shown on the whole system. The nickel end of this system was later studied by I. Iitaka. These two diagrams of the Nickel end given by Gwyer and Iitaka are shown in Fig. 1, for purposes of comparison.

From this Fig. 1 we see a great difference between these diagrams namely that the max. solubility of the  $\alpha$  solid solution is about 14.5% Al by Gwyer, but it is only 8.3% Al by Iitaka and there exist  $\beta$  and  $\gamma$  between 8.3%–14.5% Al. The liquidus curves are also very different from each other.

Thus the present investigation was carried out to ascertain the reason for this lack of coincidence.

## Materials Employed.

Mond Nickel and Aluminium of 99.8% purity were employed.

## Method of Investigation.

Nickel and Nickel-Aluminium hardener were melted in a Tammann tube heated in a Tammann furnace under the flux of borax and cast in a

tube under borax cover was cooled in the furnace and the time required to cool in a constant fall of temperature was recorded with a chronograph; the temperature being read with a Pt-PtRh-couple.

## Results of Experiments.

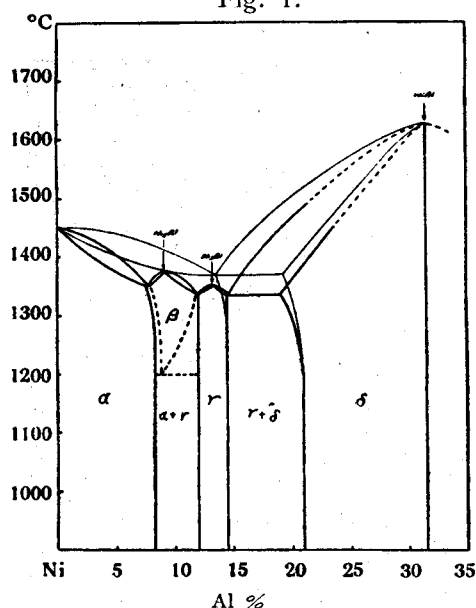
The results of thermal analysis are shown in Table I. and these results are plotted with the

Table I.

No.	Composition %		Arrest points °C		
	Ni	Al			
1	96.4	3.5	1,445		960
2	94.7	5.3	1,360		1,015
3	93.5	6.5	1,360		1,060
4	92.2	7.8	1,335		1,075
5	91.2	8.8	1,300		1,020
6	90.2	9.8	1,255		960
7	89.5	10.5	1,325		1,100
8	88.9	11.1	1,375		1,085
9	88.7	11.3	1,365		1,065
10	87.7	12.3	1,320		1,075
11	87.6	12.4	1,315		1,085
12	86.5	13.5	1,290		1,060
13	85.5	14.5	1,330		1,075
14	84.1	15.9	1,330	1,300	1,045
15	82.4	17.6	1,350	1,300	1,100

results microscopical study in the equilibrium diagram shown in Fig. 1.

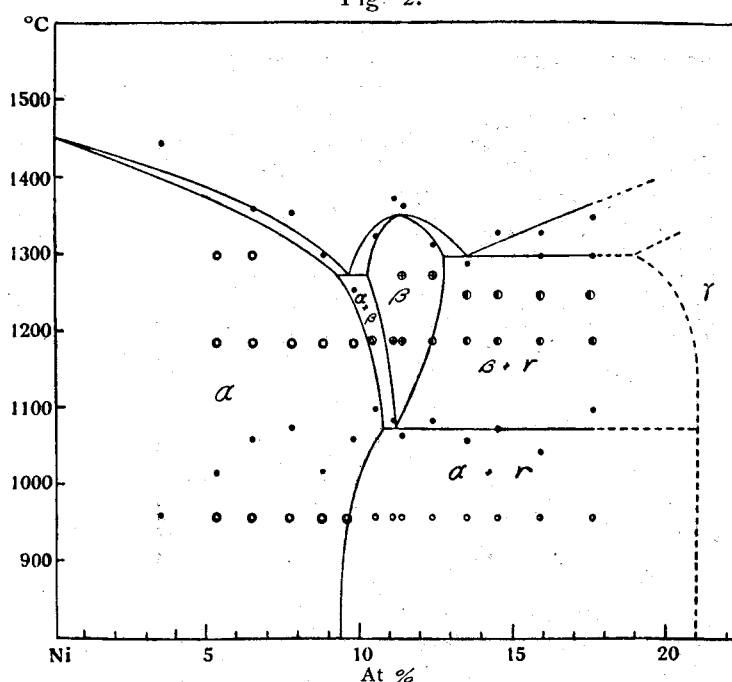
Fig. 1.



cylindrical chill mould. The specimens thus prepared were cut in 2 pieces and one of them was employed for thermal analyses and the other for microscopic study.

Thermal analysis was carried out by inverse method, i.e., the specimen melted in a Tammann

Fig. 2.

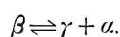


As seen from this diagram, we found the existence of  $\beta$ -phase and its eutectoid decomposition as reported by Iitaka even if the position and the temperature are somewhat different.

From the results of the thermal analysis of the alloys containing Al 3.5–9.8% which are not to show the thermal change due to the eutectoid transformation of  $\beta$  in equilibrium condition, we observed the heat evolution at about 1050°C.

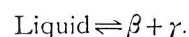
These phenomena are considered to be due to the fact that they were cooled so rapidly from liquid that the equilibrium condition was not held and the reaction liquid  $\rightarrow \alpha + \beta$  occurred. And the so formed  $\beta$  was also super-cooled and the reaction  $\beta \rightarrow \alpha + \gamma$  occurred at about 960°C. When the microstructures of these alloys were observed after the thermal analysis, we observed that they consist of the dendritic crystals of  $\alpha$ -solid solution and eutectic mixture. But they were transformed into uniform solid solution after long annealing at higher temperature.

In the range of the alloys containing Al 9.8–13.5%, the liquidus showed a maximum at about 11% of Al, and the evolution of heat was observed distinctly at about 1060°C. It was evident from the microscopical examination that this was due to the eutectoid transformation



The eutectoid point was also considered to exist at about 11% of Al as the heat evolution was maximum at this alloy.

In the range of the alloys containing Al 13.5–17.6%, a heat evolution subsequent to the primary separation was found to occur at about 1300°C. This was determined to be a eutectic reaction



and the eutectic point was considered to be 13.5% of Al. From our investigation we did not find the existence of a phase denoted  $\gamma$  by Iitaka which is said to exist between 12–12.5% Al.

### Microscopical Examination.

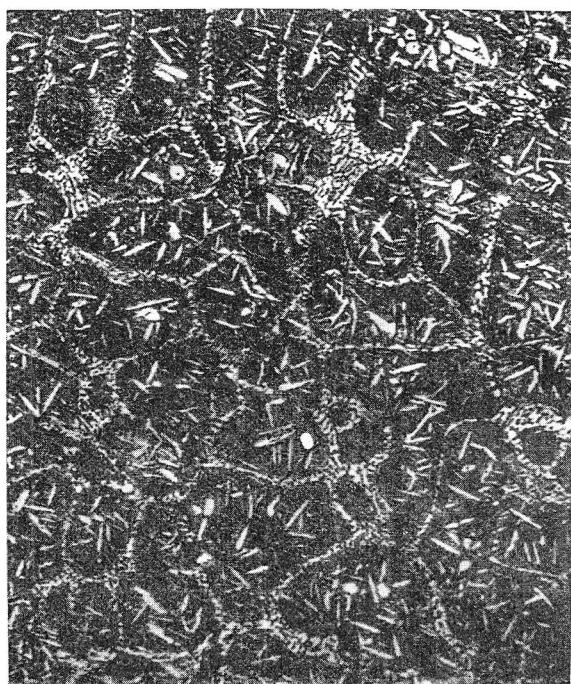
Specimens were heated for long time in an electric furnace automatically regulated at the desired temperature and quenched in water. Their microstructures were examined. Etching was carried out with a hot mixture of nitric acid and acetic acid.

The results of examination are plotted in Fig. 2. In the figure  $\ominus$  and  $\oplus$  show  $\alpha$  and  $\beta$  respectively, and  $\odot$ ,  $\circ$ , and  $\bullet$  show the fields  $\alpha + \beta$ ,  $\alpha + \gamma$  and  $\beta + \gamma$  respectively.

Some of the photographs of the specimens heat-treated are shown in Fig. 3. (a) is a microphotograph of the alloy containing 8.8% Al cooled in furnace in which black  $\alpha$ -crystals are surrounded by the eutectic mixture  $\alpha + \beta$ .

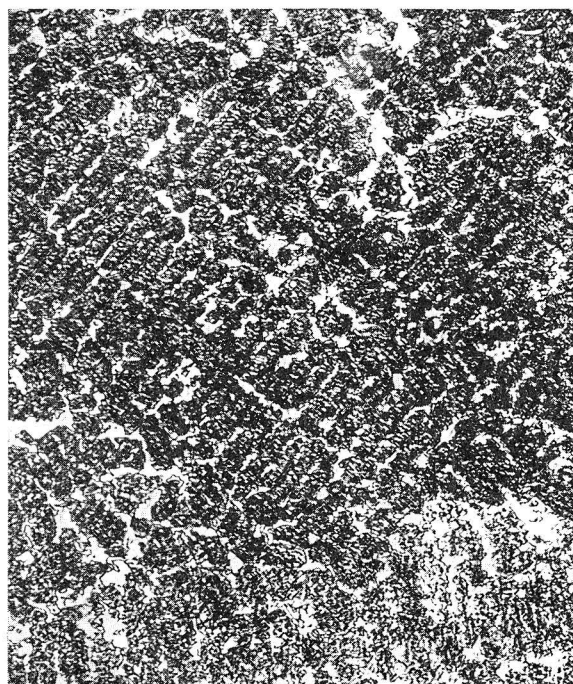
Chill cast specimen of the same alloy was annealed for 5 hours at 800°C, but the chill cast structure was not destroyed by this treatment, as shown in Fig. 3 (b). Therefore it was ascertained to require longer time and higher temperature for the complete annealing of these alloys. For example the same alloy was heated for 17 hours at 1170°C, for 10 hours at 1050°C and then for 15 hours at 950°C. Then it was quenched in water.

Fig. 3 (a).



8.8% Al  
Furnace cooled.

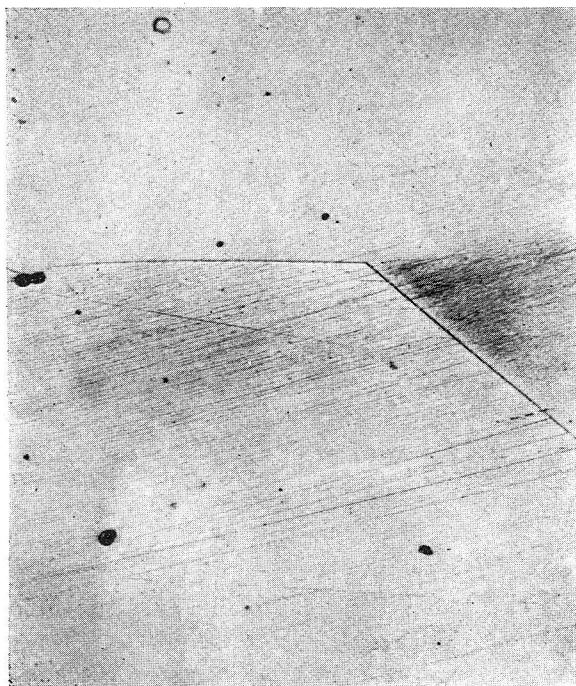
Fig. 3 (b).



8.8% Al  
Heated at 800°C for 15 hours and quenched.



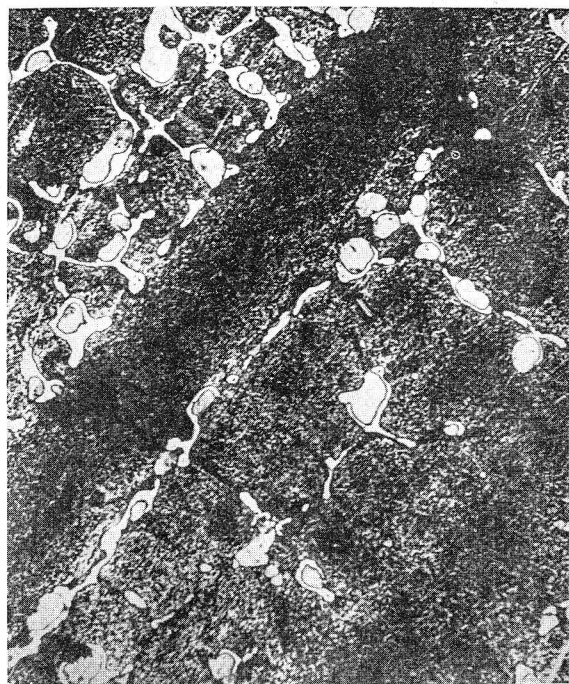
Fig. 3 (c).



8.8% Al

Heated at 1170°C for 17 hours, at 1050°C for 10 hours, at 950°C for 15 hours and then quenched.

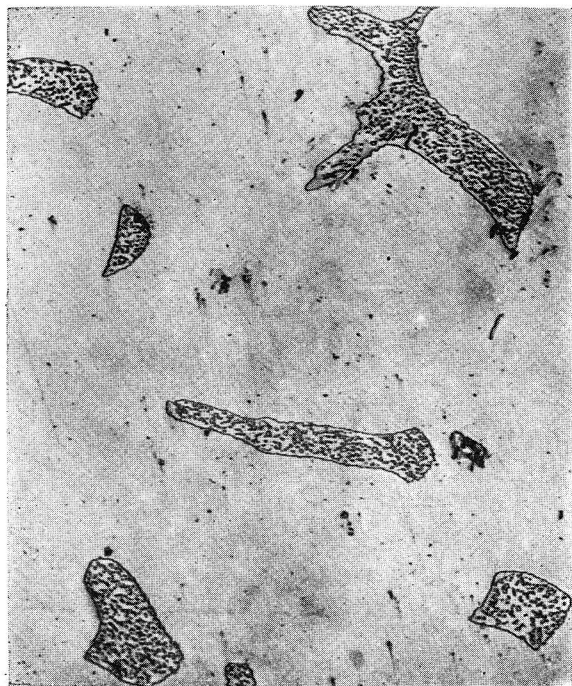
Fig. 3 (d).



9.8% Al

Heated at 1000°C for 15 hours and quenched.

Fig. 3 (e).



10.5% Al

Heated at 1190°C for 17 hours and quenched.

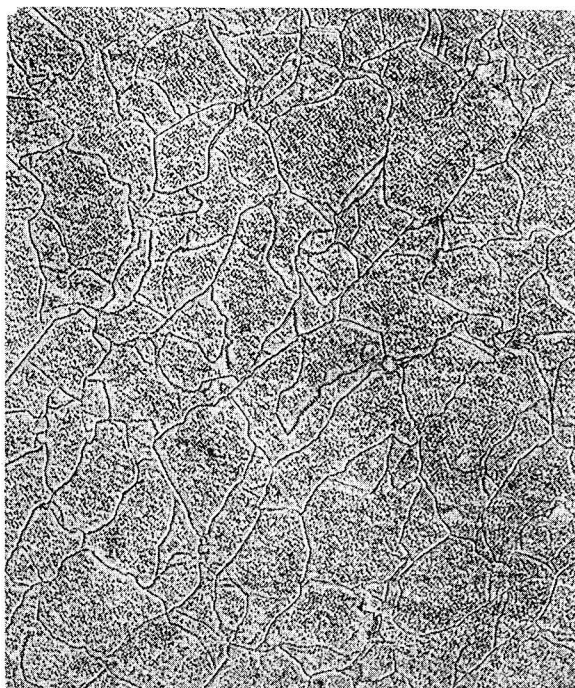
Fig. 3 (f).



11.0% Al

Heated at 1190°C for 17 hours and quenched.

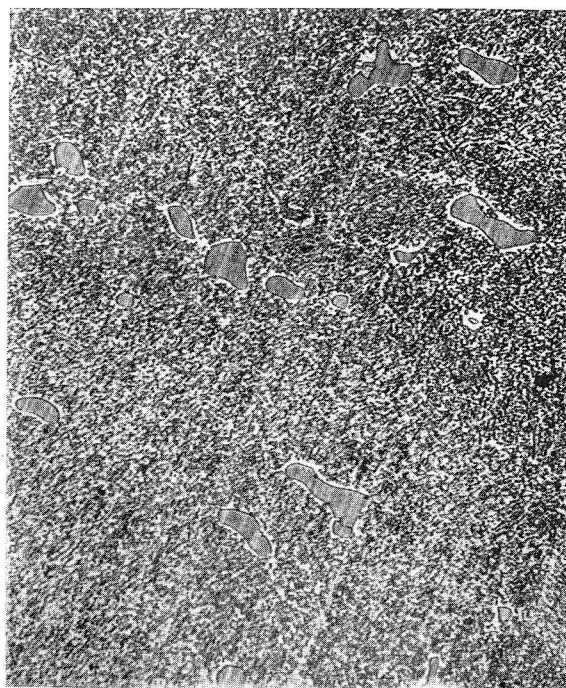
Fig. 3 (g).



11.1% Al

Heated at 1170°C for 17 hours, at 1050°C for 10 hours, at 960°C for 15 hours and quenched.

Fig. 3 (h).



11.5% Al

Heat-treated as (g).

Fig. 3 (i).



14.2% Al

Heated at 1200°C for 24 hours and quenched.

Fig. 3 (j).



17.6% Al

Heated at 1190°C for 18 hours and quenched.

The microstructure is shown in Fig. 3 (c). This evidently consists of  $\alpha$ -solid solution only.

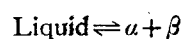
As shown in Fig. 3 (d) chill cast specimen No. 6 (Al 9.8%) was not transformed into uniform structure even if it was heated for 15 hours at 1000°C, but it began, after longer annealing, to show structure similar to that of Fig. 3 (c). Fig. 3 (e) illustrates the microstructure of the alloy containing 10.5% Al which was annealed at 1190°C for 17 hours. It consists of  $\alpha$  and decomposed  $\beta$ .

The alloys containing 11.2–11.4% of Al crystallize in  $\beta$  phase from Liquid, and that  $\beta$  phase decomposes into  $\alpha$  and  $\gamma$ . It is illustrated in Fig. 3 (f) and (g) i.e. the former is the alloy 11.1% Al quenched at 1190°C which contains  $\beta$  phase. But Fig. 3 (g) show an eutectic structure of the same alloy quenched at 960°C. Fig. 3 (h) explains the similar structure of the alloy containing 11.5% of Al. The alloy containing 11.5% of Al contains  $\beta$  and  $\gamma$ . Fig. 3 (i) and (j) are the micro-structure of the alloy containing 14.2% and 17.6% of Al respectively which were quenched at 1200°C and 1190°C. They are observed to contains of 2

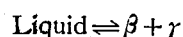
phases  $\beta$  and  $\gamma$ . These microstructures coincide well with the equilibrium diagram.

### Conclusion.

The constitutions of the nickel-Al alloys containing Al up to 82% were investigated with thermal analysis and microscopical study. From the results of the experiments the equilibrium diagram at the nickel side was determined. In this diagram liquidus shows a maximum at about 10% of Al corresponding to the melting point of  $\beta$  ( $\text{Ni}_4\text{Al}$ ). Hence 2 eutectic reactions



and



were observed to exist.

The  $\beta$  phase decomposes into  $\alpha$  and  $\gamma$  at above 1060°C.

### Acknowledgement.

In conclusion, we express our sincere thanks to Mr. James A. Rabbitt of Japan Nickel Information Bureau who has given great help to the present investigation.