Young's Modulus of Age-Hardend Aluminium Alloys1)

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Variations of the Young's modulus and of the internal friction during the aging of the supersaturated solid solution have been examined on polycrystalline specimens of aluminium alloys by the high frequency method at about 13 kilocycles per second; both the isothermal aging curve and the heating curve have been obtained.

The marked difference between the effects of the formation of the Guinier-Preston Zone and the precipitation of the new phase on the Young's modulus has been recognized in Al-Ag alloy, which has been discussed based on the point of view that the formation of the G-P zone is a discrete step in the aging process.

In all alloys, it has been confirmed that the Young's modulus increases accompanied by the precipitation of the new phase, but not by the formation of the G-P zone.

The effect of aging on the internal friction at room temperature has been found negligible in Al-Ag and Al-Cu alloys, but a certain increase has been recognized in Al-Zn alloy.

The internal friction versus temperature curves show steep increase at high temperature, which is identical with that in the case of pure aluminium specimen.

§ 1. Introduction

Precipitation is a decomposition of a supersaturated solid solution into a new phase of different composition and the initial solid solution with diminished solute concentration. In the cases of aluminium rich Al-Ag⁽¹⁾ (2), Al-Cu⁽³⁾, and Al-Zn⁽⁴⁾ systems, a new phase, AlAg₂, Al₂Cu, and Zn-phase respectively, will precipitate because of decreasing solid solubility with decreasing temperature, when the solid solution is cooled down from a temperature above the solvus (solid solubility curve) as shown by arrows in Fig. 1 (a), (b) and (c).

Although the phase diagram merely displays the initial and the final equilibrium states, it does not show the path by which a process

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of precipitation goes on. Changes of various properties, such as hardness, electrical resistivity, lattice parameter, and so on, during the aging of supersaturated (quenched) alloys have been investigated by many workers on various systems⁽⁵⁾⁻⁽⁹⁾.

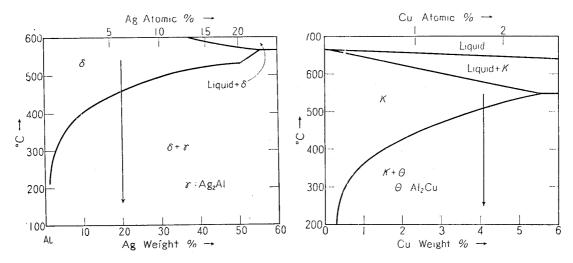


Fig. 1 (a): Solid Solubility Curve of Silver in Aluminium.

Fig. 1 (b): Solid Solubility Curve of Copper in Aluminium

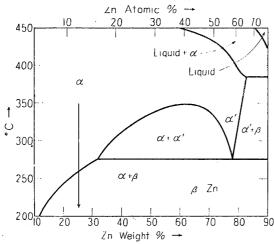


Fig. 1 (c): Solid Solubility Curve of Zinc in Aluminium.

Age-hardening is a most prominent phenomenon and it has been well investigated in many kinds of aluminum rich alloys. It has been shown that, at a certain aging temperature below the limiting temperature determined by the concentration of solute atoms in the matrix solid solution, the hardness increases in two steps. By the X-ray analysis it has been revealed that the first step, which corresponds to the initial rise of hardness, is attributed to the

formation of the solute atom segregation in the matrix named Guinier-Preston Zone (G-P zone) or "Complex". The G-P zone is known to be stable below the limiting temperature determined by the concentration of solute atoms in the matrix solid solution. When the aging proceeds further, a new Laue spot is set to be observed corresponding to the second rise of hardening, which is attributable to the precipitation of new phase. In this second step, it has also been confirmed in various alloys that a metastable precipitated phase, which is coherent to the matrix lattice is formed, preceding to the formation of the

equilibrium phase, which is incoherent to the matrix lattice.

Based on the calorimetrical analysis⁽¹⁰⁾ and the electrical resistivity measurements⁽¹¹⁾ (11a), one of the authors (K.H.) has held that the formation of the so-called G-P zone is not a preliminary stage of the precipitation of the new phase, and considering the stability of the G-P zone as a function of the concentration of the solute atoms in the matrix solid solution, has proposed a mechanism of aging processes which can explain fairly well the observed results in various alloys. (11b)

It seems that the Young's modulus is suitable as a measure for the study of aging processes or precipitation phenomena in alloys because of its sensitivity to changes in the atomic configuration and concentration of solute atoms in the matrix solid solution.

Some experimental results have been reported on the composition dependence of the Young's modulus at equilibrium state in aluminium alloys⁽¹²⁾⁻⁽¹⁵⁾, but scarcely on its change accompanied by aging treatment.

The objects of the work described in the present paper were to examine the aging and the heating curves of Young's modulus for aluminium-rich Al-Ag, Al-Cu and Al-Zn alloys, and to test the proposed aging process.

§ 2. Specimens and Experimental Procedure

We have prepared six polycrystalline rods of aluminium-rich binary alloys, which are composed of super pure aluminium and one of the following metals: 20.18 wt. % assay silver, 1.06, 1.95, 3.11, and 4.04 wt. % of electrolytic copper, and 30.10 wt. % high purity zinc, respectively. The metals were melted in a graphite crucible, well stirred by a graphite rod, and cast into a chillmould. The specimens were annealed in a electrical furnace at about 500°~550°C for 100~200 hours after some cold working in order to disperse the casting structure. All specimens have the dimensions, 1.3 cm in diameter and 17 cm in length. The solution heat treatments were as follows; 2 hours at 540°C for the Al-Ag and the Al-Cu alloys and 2 hours at 300°C for the Al-Zn alloy. Supersaturated solid solutions were obtained by quenching the solution heat treated specimens into water (in the cases of Al-Cu and Al-Zn) and quenching oil (in the case of Al-Ag) of room temperature, about 19°C~22°C. Then they were aged at various temperatures in a electrical furnace. Measurements for the isothermal aging curves were made at about 20°C after quenching the specimen from the aging temperature down to the room temperature at every measuring procedure. Heating curves were obtained during heating the specimen at a rate of 0.5° ∼1°C per second. The temperature was measured by the Cu-Constantan thermocouple, which was set closely near the specimen.

The Young's modulus and the internal friction were determined from the resonance frequency curve of forced longitudinal vibration given to the specimen⁽¹⁶⁾⁻⁽²⁰⁾. The measurements were made by using the

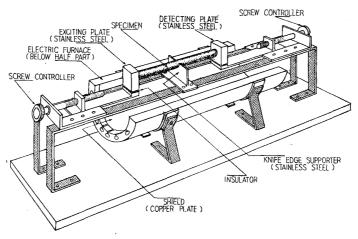


Fig. 2: Apparatus for Measurements of Young's Modulus and Internal Friction

apparatus illustrated in Fig. 2 and Fig. 3 (a). The center of the specimen, that is, a node of longitudinal vibration, is mounted on the knife edge supporter which has the earth potential. The specimen is

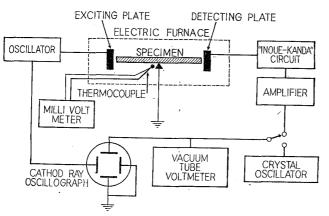


Fig. 3 (a): Block Diagram of the Measuring System.

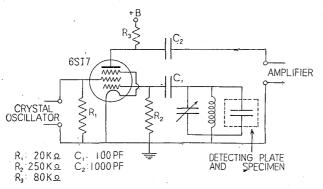


Fig. 3 (b): "Inoue-Kanda" Circuit.

free at its both ends, each of which is faced to a fixed plate, "excitor" or "detector". To keep the maximum amplitude of vibration to be constant at every measurement, the gaps between the end faces of the specimen and the exciting and the detecting plate respectively are controlled by the screws, as shown in Fig. 2. By alternating potential difference fed by an oscillator between the exciting plate and one of the endfaces of the specimen, longitudinal vibration is excited in the specimen. The oscillator is consisted of three parts: the master oscillator, one stage of the buffer, and one stage of the power amplifier.

master oscillator is the usual Hartley circuit and supplied by an electronically stabilized power source. The voltage at the final stage was about 200~300 volt. The oscillator was calibrated comparing with the standard crystal oscillator before each measurement. Between the other end face of the specimen and the detecting plate, a condencer is formed, whose capacity changes when the specimen is vibrated. This capacity change is translated into the current change in the "Inoue-Kanda" circuit (Fig. 3 (b)), which we used as the detecting circuit (21), and then the current is amplified by a broad band-pass amplifier and examined by a cathod ray oscillograph, or vacuum tube voltmeter, as shown in Fig. 3 (a).

The specimen can not be vibrated, if the applied frequency is not near its characteristic frequency.

At the maximum amplitude of the vibration in the specimen the resonance frequency (f) can be determined, from which the Young's modulus (E) is calculated. At the same time, from the half width of the resonance curve, the internal friction at that frequency is determined. The Young's modulus can be computed from experimentally obtained data by the equation⁽²²⁾,

$$E = \left(\frac{2}{n}\right)^{2} (\rho_{20}) (\mathbf{L}_{20}) \left(\frac{\mathbf{L}_{20}}{\mathbf{L}_{T}}\right) f^{2} \left(1 + \frac{1}{2} \mathbf{A} n^{2} \frac{\sum^{2}}{\mathbf{L}_{20}^{2}}\right)$$

in which ρ_{20} and \mathbf{L}_{20} are the density and length, respectively, of the specimen at 20°C, \mathbf{L}_T the length at the temperature of measurement, n the number of half-waves in the rod, Σ the average Poisson's ratio and \mathbf{A} its cross-sectional area. In the present case n=1 since the foundamental frequency wave was used.

For the study of the isothermal aging, it is necessary to know merely the relative values of the Young's modulus of aged specimen to that of as-quenched one. The change of length due to the aging is so small, as has been recognized by some workers⁽²³⁾ (24), that L_{20} may be regarded as constant throughout the aging process, and since all measurements were carried out at the same temperature, the relative value of square resonance frequency, f^2/f_0^2 , may be taken as a measure of the change in the Young's modulus, where f and f_0 are the resonance frequency of aged and that of as-quenched specimens respectively.

Whilst in the case of the heating measurements the effect of thermal expansion can not be neglected, but it does not affect the general feature of the Young's modulus versus temperature curves, and hence we have used the value of f^2 in place of the Young's modulus itself.

At the measurements of the internal friction we have no regard for the effect of air viscosity at the gaps between the end faces of the specimen and the exiting and the detecting plates, whence the plotted values must be somewhat larger than the true values.

The measured values of the Young's modulus and of the internal friction might have small deviation in each measurement by virtue of slight changes in the mounting situation. But this effect has been found negligible for the accuracy required in the present work.

§3. Experimental Results and Discussions

I. Aluminium-Silver Alloy

Fig. 4 represents the isothermal aging curves of the Young's modulus, which were obtained for Al-Ag (20.18 wt. % Ag) alloy.

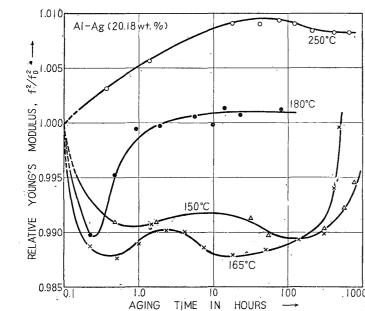


Fig. 4: Change in Young's Modulus of an Al-Ag (20.18 wt. %) Alloy during Aging.

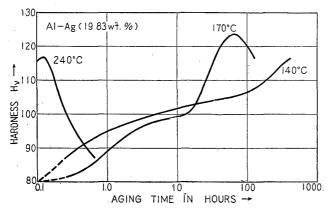


Fig. 5: Age-Hardening of an Al-Ag (19.83 wt. %) alloy (after Watanabe and Koda)

It will be noted that at the early stage of the aging at 150°, 165° and 180°C, the values decrease from their starting point, but at the later stage they turn to increase. On the other hand the value of 250°C-aging increase from the beginning without initial decrease.

Comparing the above results with the age-hardening curves, shown in Fig. 5 which have been obtained by Watanabe and Koda⁽²⁵⁾, it may be recognized that the initial decrease and the subsequent increase of the Young's modulus during the aging at lower temperatures than about 200°C respond respectively to the first and the second stage of hardening at the low temperature aging.

Based on the X-ray analysis⁽²⁶⁾⁻⁽²⁸⁾, on the calorimetric analysis^{(10) (28)}, and on the electrical resistivity

measurements⁽¹¹⁾, it has been shown that the first stage of the low temperature aging is the formation of the G-P zone and the second stage is the precipitation of a new phase. Therefore the intial decrease of the Young's modulus observed in the present case would be attributable to the formation of the G-P zone and the subsequent increase to the precipitation of coherent AlAg₂ phase (γ' phase). The appearance of an intermediate small maximum in the 150°C or 165°C-aging curve

may be interpreted as a result of superposition of the decrease due to the G-P zone and the increase due to the γ'-phase, as shown schematically in Fig. 6 (a), in correspondence to the case of the electrical resistance curve, Fig. 6 (b). Detailed examination of Fig. 6 (b) has been carried out by one of the authors (K. H.) previously¹¹⁾, by which the superposition of the two reactions has been confirmed.

On the other hand, it has been shown by many investigators⁽²⁶⁾⁻⁽²⁹⁾ that the G-P zone can not be formed during the aging at temperatures above 210°C, which is the limiting temperature of the formation of the G-P zone. The 250°C-aging in the present observation should be in this case, and in fact the

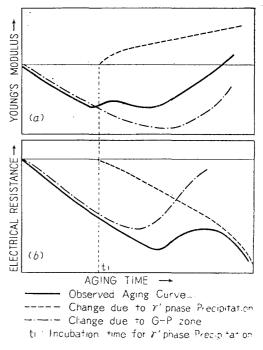


Fig. 6: Integration of Reactions contributing to Changes in the Young's Modulus and the Electrical Resistance.

Young's modulus increases monotonously from the start due to the γ' phase precipitation and any initial decreases can not be recognized from
this curve.

The slight decrease at the later stage of the 250°C-aging curve must be due to the transformation of the γ' -phase to the incoherent AlAg₂ phase, γ -phase, accompanied by the recrystallization⁽⁸⁾.

The change of the internal friction has not been observed during the above aging treatments. This seems to point out that the changes of the Young's modulus in Fig. 4 are not in correlation with the anelastic phenomena.

The fact that the change of the Young's modulus due to the formation of the G-P zone is in the opposite direction to that due to the precipitation of the new phase, may be regarded as a powerful evidence for the point of view that the former is not a preliminary stage of the latter.

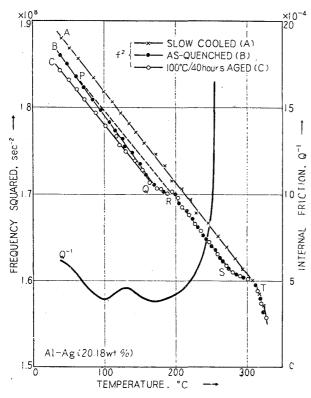


Fig. 7: Young's Modulus vs. Temperature Curves and Internal Friction vs. Temperature Curve of Al-Ag Alloy.

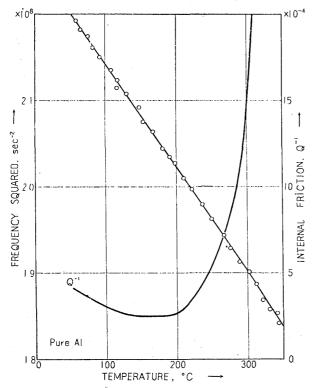


Fig. 8: Young's Modulus vs. Temperature Curve and Internal Friction vs. Temperature Curve of Pure Aluminium.

Fig. 7 represents the results of the heating measurements with the same specimen as in the case of Fig. 4.

The curve marked with A was obtained for a slow cooled alloy, and shows that the Young's modulus decreases monotonously with temperature up to about 300°C, where it begins to deviate downward slightly.

The curve marked with B was obtained for a as-quenched specimen, where two inflexion points (R and T) were observed. The downward deviation (PO) in the region between 50°C and 180°C may be attributed to the formation of the G-P zone during the measurement, the phenomena relating to which have been observed also in the electrical resistance versus temperature curves(11) and in the specific heat versus temperature curves(10). Subsequent rise (Q. R) between 170°C and 200°C may be attributed to the dispersion (re-solution) of the G-P zone. From the fact that the point R on the curve B falls on the dotted line (BS). which represents the value for the quenched state, it will be deduced that the alloy returns to the quenched state by the dispersion of the G-P After the upward zone. deviation (ST) between 250°C and 310°C, the curve B coincides with the curve A completely. The upward deviation (ST) is obviously due to the precipitation of the γ' -phase during the measurement.

The curve marked with C was obtained for the specimen that is aged at 100°C for 40 hours (low temperature aging). In this case the Young's modulus is already decreased before the measurement due to the G-P zone, which was formed during the aging. Between 170°C and 200°C, the curve C deviates upward owing to the dispersion of the G-P zone and at higher temperatures it completely coincides with the curve B.

The temperature limit of the existence of the stable G-P zone determined from Fig. 7 which is about 200°C, is in good agreement with those determined from the results of the calorimetric and of the electrical resistance measurements.

The internal fcirtion versus temperature curve is also shown in

Fig. 7, where we can not detect any difference among as-quenched, low temperature aged, and slow cooled alloys. However, recently Damask and Nowick⁽³⁰⁾ have repoted that they could detect the effect of the aging treatment on the low frequency internal friction.

Steep increase of the internal friction was observed above 250°C, but peak can not be observed in the temperature range of the present experiment. The same steep increase was observed also with the

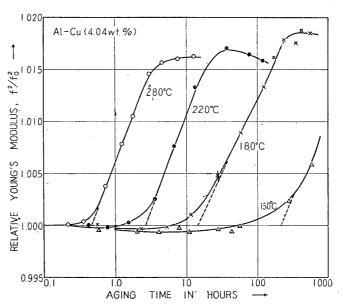


Fig. 9: Change in Young's Modulus of an Al-Cu (4.04 wt. %) Alloy during Aging.

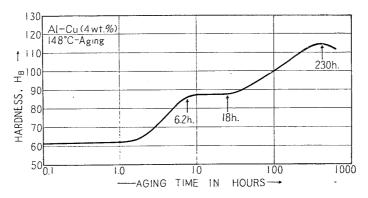


Fig. 10: Age-Hardening Curve of an Al-Cu (4 wt. %) Alloy at 148°C. (after Gayler and Parkhouse).

polycrystalline specimen of pure aluminium, as shown in Fig. 8. Ké has reported in his paper⁽³¹⁾ on the low frequency internal friction in pure aluminium, that the internal friction of polycrystal increases and shows a peak at 280°C, but that it does not occur in single crystal. He concluded that the origin of this phenomena should be associated with the viscous behaviour of the grain boundary. Comparing our results with those of Ké, it seems that the remarkable increase of the internal friction above 250°C is also due to the grain boundary viscosity, although the temperature where the increase occurs is much higher than that of Ké.

Origins of the initial decrease up to 100°C and of the slight maximum at 130°C in Fig. 7 are not yet obvious, although it was confirmed that they are independent of the aging process.

II. Aluminium-Copper Alloy

Fig. 9 shows the isothermal aging curves of the Young's modulus obtained with Al-Cu (4.04 wt. %) alloy. The Young's modulus begins to increase after the incubation period, which is fixed by the aging temperature, and then attaines to the maximum values, after which it decreases slightly.

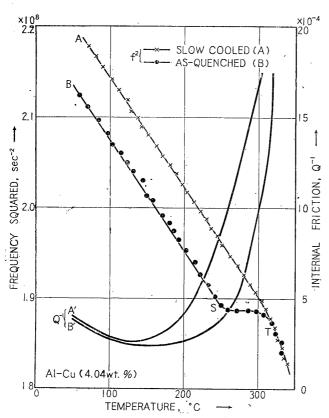


Fig. 11: Young's Modulus vs. Temperature Curves and Internal Friction vs. Temperature Curves of Al-Cu Alloy.

The incubation period for the increasing of the Young's modulus was determined by extrapolating the curve down to the base line, which represents the as-quenched value, as shown by dotted lines in Fig. 9. From the relation of the incubation period to the aging temperature, the activation energy for this process was estimated to be about 21,000 cal/mol. value is comparable to 24,000 cal/mol, which is estimated age-hardening the curves⁽³²⁾, so it seems that the origin of the increasing Young's modulus in Fig. 9 is much the same as that of the second rise in the hardeness as shown

Fig. $10^{(32)(33)}$, which has been, by the X-ray measurements⁽³⁴⁾, attributed to the formation of coherent CuAl₂ precipitate (θ' -phase).

The slight decrease after the maximum would be attributed to the precipitation of incoherent $CuAl_2$ phase (θ -phase).

On the other hand we could not detect clearly the change of the Young's modulus during the aging period that corresponds to the first step of the hardening, that is the stage of the formation of the G-P zone. The negligible effect of the G-P zone on the Young's modulus in Al-Cu alloy may be imputed to the low concentration of the solute atom in the matrix solid solution. The same things should also occur in the case of Al-Ag alloys of low silver concentration. In fact, we could not detect clearly the change of the Young's modulus due to the G-P zone in Al-Ag alloy containing about 10 wt. % of silver, the solute atom concentration of which has the value 2.7 measured in atomic percent which is comparable to that of the present Al-Cu alloy, 1.7 atomic percent.

In Fig. 11, the curves marked with A and B represent the Young's modulus versus temperature curves that were obtained with the same specimen as in the case of Fig. 9, after quenched and slow cooled.

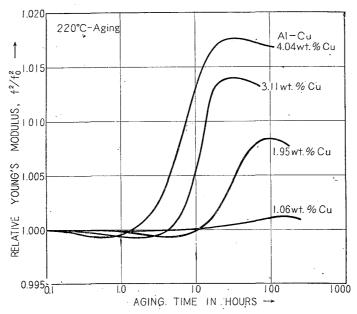


Fig. 12: Change in Young's Modulus of Al-Cu during 220°C Aging.

Besides the linear decrease, the curve B shows considerable upward deviation between 240°C and 310°C as indicate by ST, which is attributed to the precipitation of the θ' -phase during the measurement. This corresponds to the second upward deviation, ST in curve B of Fig. 7, obtained for Al-Ag alloy.

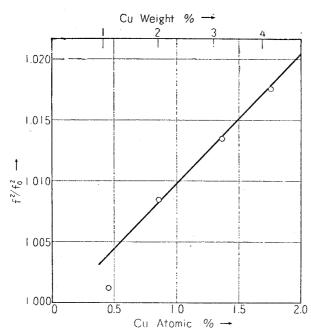


Fig. 13: Maximum Value of Young's Modulus

Temperature dependence of the internal friction is also shown in Fig. 11. The steep increase at high temperature was likewise observed which corresponds to those in curves for Al-Ag alloy and pure aluminium, shown in Fig. 7 Fig. 8. It will be found that the value for slow cooled alloy (curve A') is always higher than that for as-quenched alloy (curve B'). This probably originates in the change in viscousity of the grain boundary due to the precipitation.

In order to study the conduring Aging at 220°C. centration dependence of the change in the Young's modulus during the aging, we have carried out the measurement with alloys of various copper concentration aged at 220°C, the results are shown in Fig. 12.

Fig. 12 shows that the higher the copper concentration is, the shorter the incubation period becomes. Furthermore, plotting the maximum value of the Young's modulus against the concentration of copper, the linearity between them is recognized, as shown in Fig. 13.

Aluminium-Zinc Allov III.

The results for Al-Zn (30.10 wt. %) alloy, the isothermal aging and the heating curves, are shown in Fig. 14 and Fig. 15 respectively. The curves in Fig. 14 indicate a pronounced rise of the Young's modulus during the aging and a subsequent slight decrease at the later stage; their general feature resembles to the curve obtained for Al-Cu alloy shown in Fig. 9. It seems likely that the increase of the Young's modulus during the aging is due to the precipitation of zinc phase $(\beta$ -phase), and that the subsequent slight decrease at the later stage is due to the reduction of coherency between the precipitate and the matrix. These situations are qualitatively the same as those in Al-Ag alloy(35).

No explicit change due to the G-P zone could be distinguished in this case, although the formation of the G-P zone has been suggested by the previous investigators (36) (37).

In spite of the fact that no change of the internal friction during the aging could be observed in the accuracy of our measurements in

Al-Ag and Al-Cu alloys, a certain increase can be recognized in Al-Zn alloy as shown in Fig. 14 by dotted curve, which was obtained by 110°C-aging.

Fig. 15 represents the heating curves of the Young's modulus

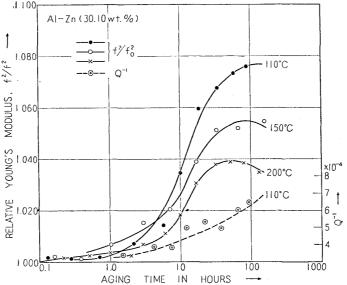


Fig. 14: Change in Young's Modulus and Internal Friction of an Al-Zn (30.10 wt. %)
Alloy during Aging.

(curves marked with A and B) and of the internal friction (curves marked with A' and B'). The curves A and A' show the value for slow cooled alloy, while the curves B and B' show the value for asquenched alloy. Upward deviation of the curve B at the point S should be attributed to the precipitation of the β phase during the heating. After the point T, the curve B coincides with the curve A. This shows the completion of the precipitation during the heating.

Comparing the curve A' with the curve B', it will be found that the internal friction of the slow cooled alloy is somewhat higher than that

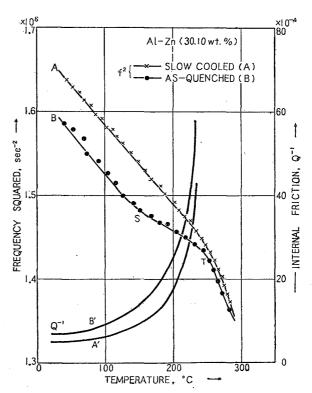


Fig. 15: Young's Modulus vs. Temperature Curves and Internal Friction vs. Temperature Curves of Al-Zn Alloy.

of as-quenched alloy, corresponding to the increase during the aging, and further that the temperature of steep increase of the internal friction for the former is somewhat lower than for the latter. These facts are probably the result of change in the viscous behaviour of the grain boundary due to the precipitation in it or the result of the formation of internal interfaces by the discontinuous precipitation, which causes the coupled relaxation effect suggested by Nowick⁽³⁸⁾.

§ 4. Acknowledgments

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NOTE ADDED IN PROOF

We are now carrying out the same investigations as described in the present paper on some age-hardening ternary aluminium alloys such as Al-Mg-Zn, Al-Cu-Mg and Al-Mg-Si, as well as some copper base alloys, Cu-Be, Cu-Mn-Ni etc. The results will be published soon.