Strain Age Hardening of Aluminium Alloys

Strain Age Hardening of Aluminium Alloys*

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(Received Aug. 26, 1963)

Synopsis

The strain age hardening of Al-Cu, Al-Mg and Al-Si alloys was studied and the results obtained were as follows: (1) The characteristics of hardening are generally similar to those of carbon steels, copper alloys and austenitic steels, although its degree is appreciably low. (2) In Al-4.9 per cent Cu alloy solution-treated and aged, the strain age hardening and the inelastic effect are less marked in a fully age-hardened state than in an over-aged state, the difference being probably due to the circumstances that dislocations pass through GP zones, whereas the θ' and θ phases act as an effective barrier against dislocation movement, pile-ups of dislocation being formed in a large number. (3) The hardening is generally marked in such an alloy as exhibiting high degrees of work hardening rate and X-ray line broadening, these being relatively low in aluminium alloys. The effect of alloying on the strain age hardening seems to form strong barriers against the movement of dislocations and a high density of piled-up dislocations during cold working rather than to take short range ordering or to segregate into stacking faults during low temperature annealing.

I. Introduction

A strain ageing effect symbolized ordinarily by a rise of yield stress in tension test is considerably less marked in aluminium than in copper or in nickel[(1)(2)]. In addition, aluminium shows at room temperature behaviors different in many respects from those of other face-centered cubic metals. For example, changes in internal friction,[(3)] X-ray line broadening after deformation[(4)] and anisotropy of Young's modulus after cold rolling are all small,[(5)] and in addition, the stage II in work hardening is not observable.[(6)] Also the strain age hardening of aluminium alloys is considerably less marked than that of copper alloys.[(7)(8)]

In Al-Cu alloy, the deformation characteristics in the various stages of ageing after solution treatment are well established in connection to the age hardening.[(9)]

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* The 1098th report of the Research Institute for Iron, Steel and Other Metals.
(1) A.R.G. Westwood and T. Broom, Acta Met., **5** (1957), 77, 249.
(2) P. Haasen and A. Kelly, Acta Met., **5** (1957), 192.
(3) R.F. Hanstock, J. Inst. Metals, **83** (1954–55), 11.
(4) C.N.J. Wagner, Acta Met., **5** (1957), 477.
(5) M. Cook and T.L. Richard, J. Inst. Metals, **83** (1954–55), 41.
(6) T.S. Noggle and J.S. Köhler, J. Appl. Phys, **28** (1957), 53.
(7) I. Gokyu, J. Japan Inst. Metals, **7** (1943), 7.
(8) S. Yamada, J. Japan Inst. Metals, **6** (1942), 161.
(9) G. Greetham and R.W.K. Honeycombe, J. Inst. Metals, **89** (1960–61), 13.

As stated in the previous paper,(32) the strain age hardening is closely related with the work hardening, and with the distribution of second phase particles dispersed in the matrix. So, Al-Cu alloy was mainly studied in the present investigation in order to clarify these relations. Al-Mg and Al-Si alloys were supplementally examined.

II. Specimens and experimental methods

Al-Cu and Al-Mg alloys used were prepared by melting 99.9 per cent Al, 99.97 per cent Cu and 99.91 per cent Mg in graphite crucibles. Al-Si alloy containing 11.6 per cent Si was a commercial modified silumin alloy, and hot-rolled little by little to obtain a uniform structure.

The heat-treatments were as follows: Ageing treatments of Al–4.9 per cent Cu alloy were done by quenching at 540°C and reheating at desired temperatures for 1 hour followed by cooling at the rate of about 0.5°C per minute. Al–11.6 per cent Si alloy was heated at 550°C for 2 hours, and cooled stepwise by keeping at every 100° C below 500°C for about 15 hours. Al-Mg alloys were quenched after heating at 500°C for 2 hours, and reheated at 270°C for 10 hours and then cooled stepwise. Cold working was made at room temperature in the range of $-2 \sim 5$°C, and the subsequent low temperature annealing was performed by immersion for 5 minutes in oil bath at desired temperatures.

Thermoelectric force was measured by using a sensitive galvanometer with a couple formed by the drawn wire to be studied and a reference wire of the same alloy in unworked state. Experiments on Bauschinger effect were carried out by torsion. The specimens were in the form of thin walled hollow cylinder with the dimensions shown in Fig. 6. Other experiments were carried out in the same way as described in the previous paper(32).

III. Results

1. Strain age hardening

In Fig. 1 are shown the strain age hardening curves for various aluminium alloys rolled by 50 per cent. In Al–11.6 per cent Si alloy, the hardening occurs clearly in two stages, whereas in other alloys it occurs in one stage within the range of $50 \sim$ 150°C. The most outstanding feature of the hardening is that its degree is considerably small compared with those of carbon steels(10), copper alloys(11) and austenitic steels. It is also noticeable that pure aluminium shows little hardening (7)(8). Fig. 2 shows the relation between the degrees of cold rolling and the strain age hardening for Al–11.6 per cent Si and Al–4.9 per cent Mg alloys. The degree of the hardening is expressed by $(H_1\text{-}H_0)/H_0$ in per cent, where H_1 and H_0 are the maximum hardness in the strain age hardening curve and the hardness at as-worked

(10) K. Nishino and K. Takahashi, Trans. JIM, **3** (1962), 57, 63.
(11) T. Sato and K. Nishino, J. Japan Inst. Metals, **20** (1956), 115, 704; **22** (1958), 30; **23** (1959), 232, 236.

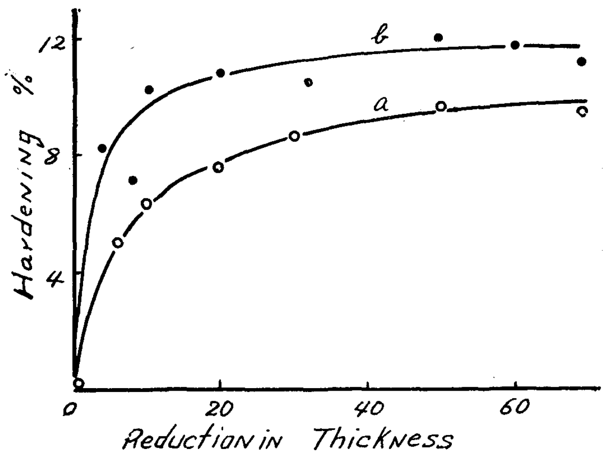
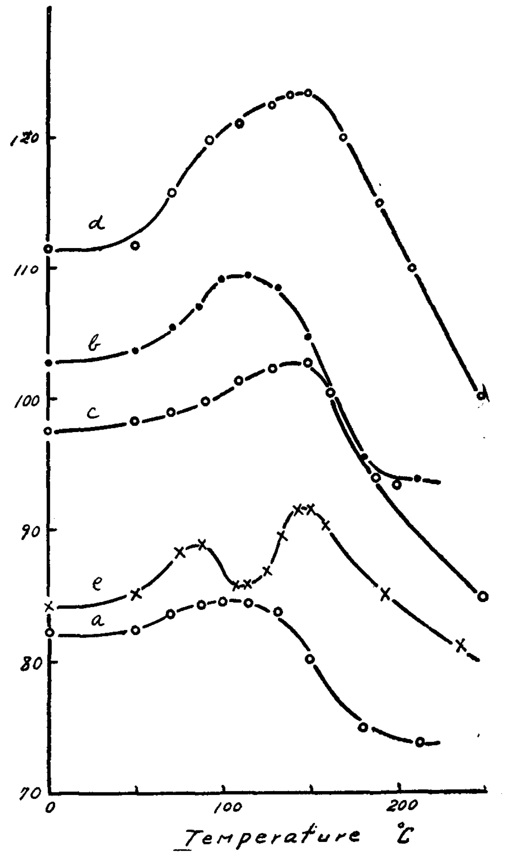


Fig. 2



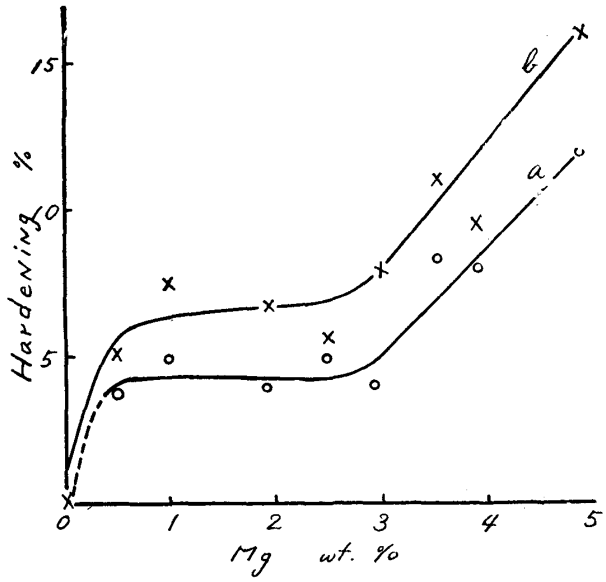


Fig. 1. Fig. 3.

Fig. 1. Strain age hardening curves for Al alloys 50% rolled.
a. Al–4.9% Cu, 430°C aged and 50% rolled.
b. Al–4.9% Cu, 270°C aged and 50% rolled.
c. Al–2.9% Mg, 50% rolled. d. Al–4.9% Mg, 50% rolled.
e. Al–11.6% Si, 50% rolled.

Fig. 2. Degrees of cold rolling and strain age hardening
a. Al–11.6% Si b. Al–4.9% Mg

Fig. 3. Mg content and degrees of hardenings in Al-Mg alloys
a. Strain age hardenig (50% rolled)
b. Secondary work hardening (50% rolled and bent)

state, respectively. The degree increases rapidly, in any case, in the first 10 per cent of cold rolling, clearly showing the feature of a two-phase alloy.

Curve (a) in Fig. 3 shows the relation of the magnesium content and the degree of strain age hardening in Al-Mg alloys rolled by 50 per cent. The degree is low and nearly constant in the range of α solid solution below 2 wt. per cent Mg. In the two-phase range, it increases with increasing quantity of the second phase in the way similar to those in austenitic steels containing martensite or in carbon steels.

2. Thermal dilatation and thermoelectric force

Curve (a) in Fig. 4 shows the change in thermoelectric force with low temperature annealing for Al–4.9 per cent Cu alloy aged at 360°C for 1 hour after solution treatment and then drawn to 55 per cent reduction in area. The thermoelectric force decreases clearly within the temperature range of strain age hardening. Curve (b) shows the change in thermal dilatation with the same specimen as the above, showing a contraction within the temperature range of strain age hardening. This trend is similar to those of copper alloys and austenitic steels although its amount is far less.

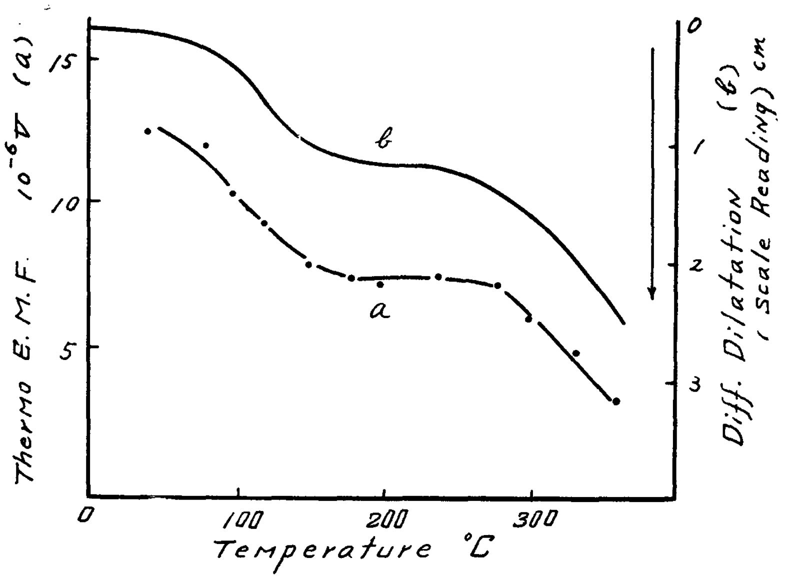


Fig. 4. Changes in dilatation and thermoelectric force (0 ~ 40°C) with annealing temperature of Al–4.9% Cu alloy aged at 360°C and then 55% drawn.

3. Inelastic effect

Curve (a) in Fig. 5 shows the changes of yield stress in the same direction as that of tensile working with annealing temperature for Al–4.9 per cent Cu alloy. In the range up to 110°C, the yield stress hardly rises but rather lowers, whereas the hardness does not change in an exactly corresponding way, but increases on the contrary (curve b). Such a behavior is analogous to those of the various alloys already reported, leading to the presumption that the hardening is also directional in aluminium alloys. To ascertain this point, experiments on Bauschinger effect were made. Fig. 6 shows the torsion stress strain diagrams of pure aluminium and Al–4.9 per cent Mg alloy. The former exhibits only a slight Bauschinger effect, but in the latter it is very marked as in the case of α brass(11) and high carbon steels.(10)

The yield stress in the reverse direction is raised by low temperature annealing.

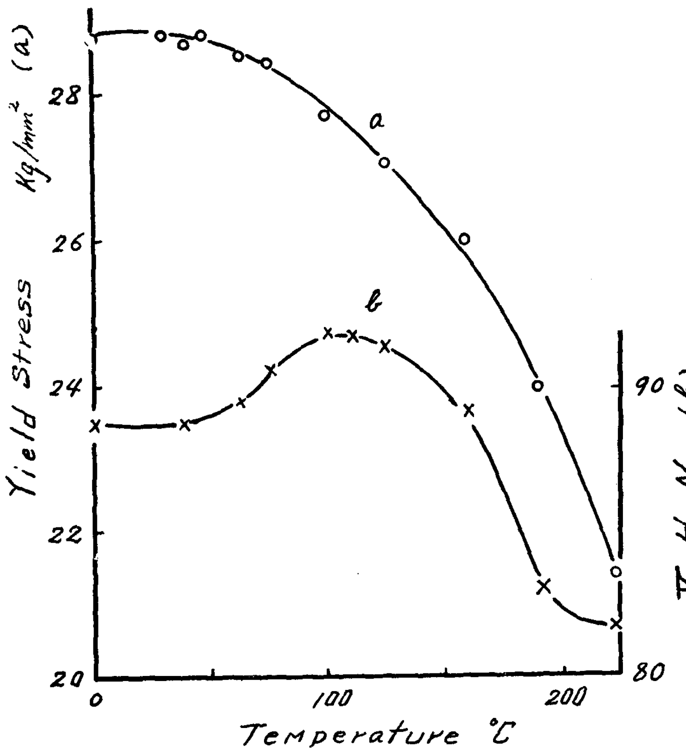


Fig. 5. Changes in yield stress and hardness with annealing temperature for Al–4.9% Cu alloy 300°C aged and 10% extended.

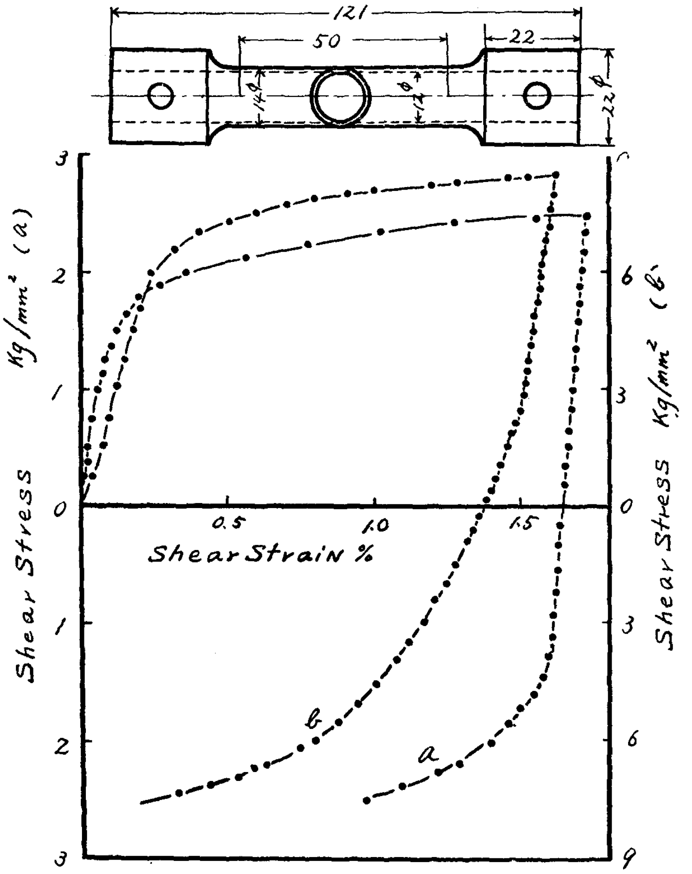


Fig. 6. Torsion stress-strain diagram of Al and Al–4.9% Mg alloy.
a. pure Al b. Al–4.9% Mg

Temperature dependence of the ratio λ_1/λ_0 is shown in Fig. 7. Specimens were first twisted, followed by low temperature annealing at zero load, and then re-twisted in the direction reverse to the first working. λ_0 and λ_1 are the flow stress in the direction of primary working and the yield stress in the reverse direction, respectively. In general, the smaller the ratio λ_1/λ_0 is the more marked the Bauschinger effect. As shown in this figure, the ratio increases markedly within the temperature range of strain age hardening, and the shape of the curve is different widely from that of the yield stress in the worked direction.

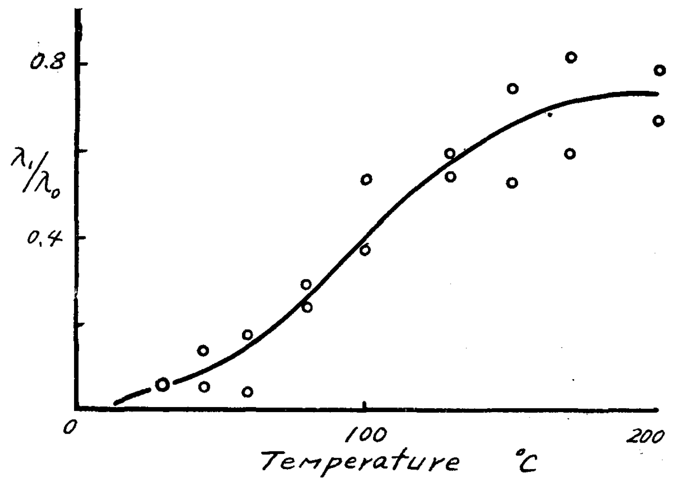


Fig. 7. Change in λ_1/λ_0 of Al–4.9% Mg alloy with annealing temperature. Prestrain, 1.5%

To clarify the relation between the inelastic behavior in the reverse direction and the age hardening, the inelastic effect was examined with Al–4.9 per cent Cu alloy aged after solution treatment. Inelastic deformation being considered to commence upon unloading after the first working at the point where the stress strain curve deviates from the straight line, the ratio λ/λ_0 was taken to represent the degree of inelastic effect. Strain rates on loading and unloading were about 4×10^{-4} sec^{-1} and 4×10^{-5}sec^{-1}, respectively. Curves (a) and (b) in Fig. 8 show the changes in λ/λ_0 and the tensile flow stress of Al–4.9 per cent Cu alloy aged at various temperatures and then extended by 8 per cent, respectively. On ageing at 200°C, that is, in a fully age-hardened state in which the GP zones precipitate, the flow stress is the highest, while the ratio λ/λ_0 is considerably small. As the ageing temperature further rises, the flow stress decreases, while the ratio shows a maximum at about 270°C at which θ' phase precipitates.

The relation between the inelastic effect and the magnesium content in Al-Mg alloys extended by 5 per cent is shown in Fig. 9. The ratio λ/λ_0 increases with increasing magnesium content in the way similar to the change in the degree of strain age hardening (curve a in Fig. 3), showing that the inelastic effect has a close relationship with the strain age hardening as in the cases of carbon steels,[10] copper alloys[11] and austenitie steels.

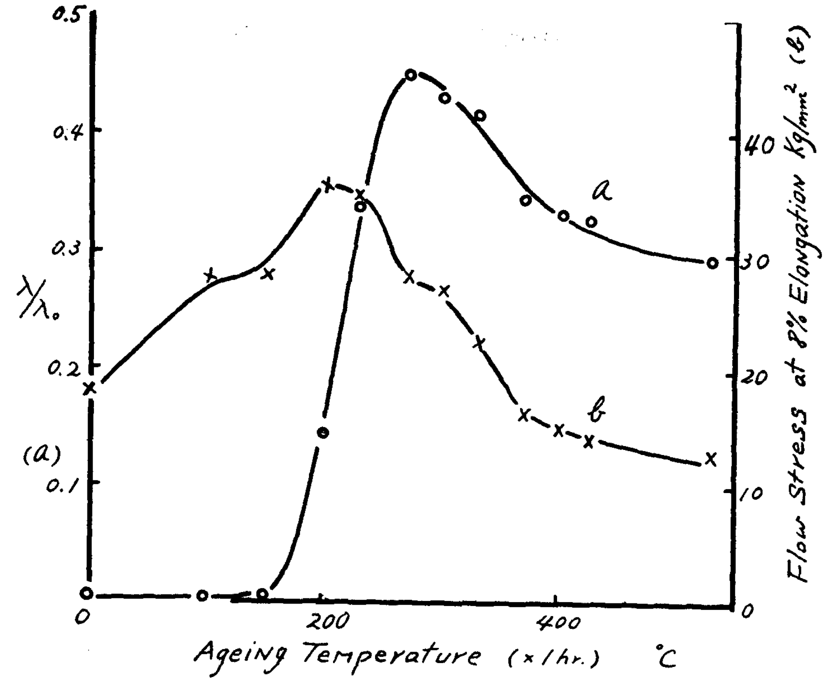


Fig. 8. Inelastic effect, flow stress and ageing temperature of Al–4.9% Cu alloy.

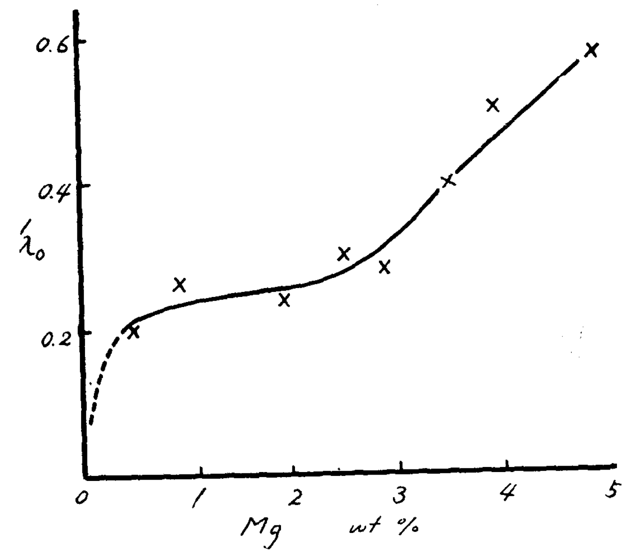


Fig. 9. Mg content and degree of inelastic effect in Al-Mg alloys (Pretension; 5%)

4. Strain age hardening and dispersion of second phase

The effect of the dispersion of second phase on the strain age hardening was studied with Al–4.9 per cent Cu and Al–11.6 per cent Si alloys. Straight lines (a) and (b) in Fig. 10 give the relations of the total surface area of the precipitates per unit volume of specimens to the degrees of strain age hardening and the inelastic effect in Al–4.9 per cent Cu alloy, respectively. The size of precipitates was controlled by ageing at various temperatures above 270°C after solution treatment,

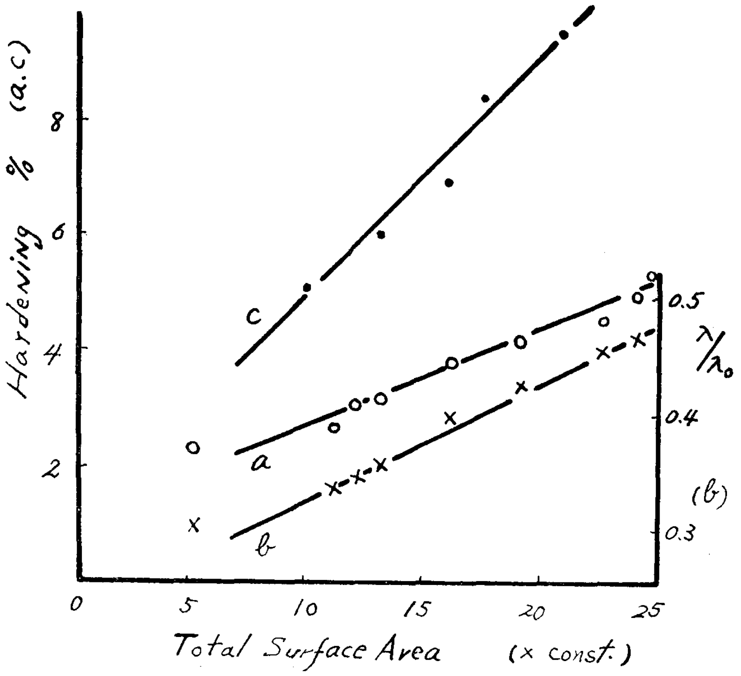


Fig. 10. Particle size of second phase and degrees of strain age hardening and inelastic effect.
a. Al–4.9% Cu, 50% rolled b. Al–4.9% Cu, 8% extended c. Al–11.6% Si, 50% rolled

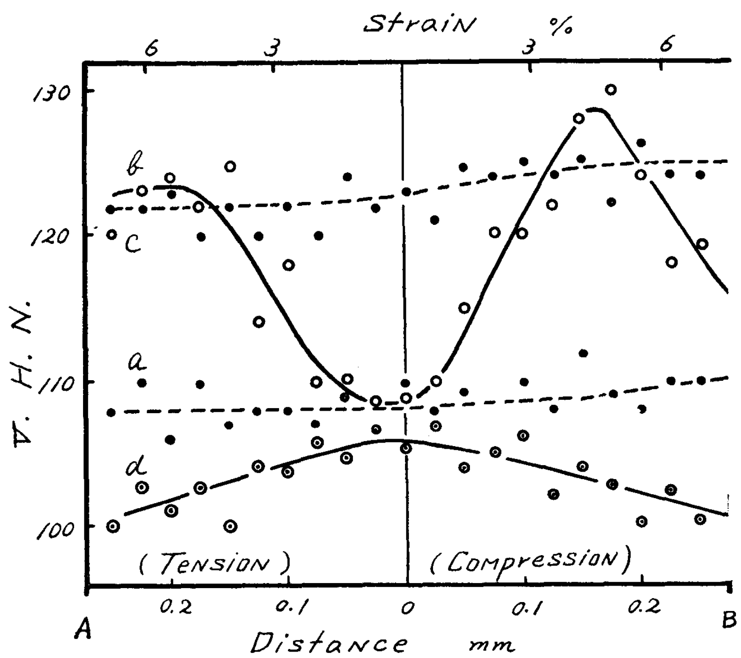


Fig. 11. Hardness changes by bending of Al–4.9% Mg and Al–4.9% Cu alloys.
a. Al–4.9% Mg, as rolled (50%) b. Al–4.9% Mg, rolled and bent
c. Al–4.9% Mg, rolled, bent and then 140°C annealed.
d. Al–4.9% Cu, 50% rolled and bent.

and the total surface area was calculated from the number of the precipitates of θ' or θ phase detected under electron and optical microscopes. On the assumption that the precipitates are globular and their volume fraction (f) is invariable, it follows that the mean size of particle $c_1 f^{1/3} n^{-1}$ is inversely proportional to the total surface area of particles per unit volume of specimen $c_2 f^{2/3} n$, where n is the number of particles per unit length of specimen, and c_2 and c_1 are constants. The fact that the degrees of strain age hardening and inelastic effect are approximately proportional to the total surface area as shown in Fig. 9, may lead to the presumption that they are also proportional to the back stress(12) originated in piled-up dislocations around the particles as in the cases of carbon steels(10) and austenitic steels.

Straight line (c) in Fig. 10 shows the result on the strain age hardening of Al–11.6 per cent Si alloy. The size of the second phase was controlled by annealing at $250 \sim 550$°C for $1 \sim 350$ hours after heavy working. The relationship is also linear. The slope of the straight line (c) is, however, different from that in Al–4.9 per cent Cu alloy (a); this is probably due to the differences in the volume fraction of the particles and in the mechanical properties of the particle or the surrounding matrix.

5. Secondary work hardening

Secondary work hardening was also studied with aluminium alloys by the same method as in the case of austenitic steels. Fig. 11 shows the results when the primary working was made by rolling and the secondary by bending. The shapes of the three curves for Al–4.9 per cent Mg alloy, first rolled to 50 per cent reduction (curve a), secondly bent (curve b) and finally low temperature-annealed (curve c), are quite similar to those for carbon steels(10) and austenitic steels. The relation between the degree of secondary work hardening and the magnesium content in Al-Mg alloys is shown in curve (b) in Fig. 3, the shape of which is in good similarity to that of strain age hardening (curve a in Fig. 3). Here, the degree of secondary work-hardening is expressed by $(H_1\text{-}H_0)/H_0$, where H_1 and H_0 are the maximum and the minimum hardness in curve (b) in Fig. 11, respectively.

Curve (d) in Fig. 11 shows the hardness distribution after secondary bending in Al–4.9 per cent Cu alloy aged at 300°C for 1 hour after solution treatment and then rolled by 50 per cent. There is no rise in hardness corresponding to the secondary work-hardening, but rather lowering. When a low temperature annealing is made after different type-secondary workings, the degree of strain age hardening becomes large by that of the softening. Such behaviors are quite different from those of various alloys already examined.

IV. Discussion

The strain age hardening of aluminium alloys shows a general similarity in the characteristics to those of carbon steels, copper alloys and austenitic steels. Hence, it may be acceptable that the strain age hardening is due to the same cause as described already with reference to austenitic steels. In aluminium alloys, however, the degree of hardening is considerably less. Such a pecularity may be noteworthy for a further elucidation of the nature of strain age hardening, and

(12) J.C. Fisher, E.W. Hart and R.H. Pry, Acta Met., **1** (1953), 336.

the relationship between the work hardening and the strain age hardening will next be discussed.

1. In pure aluminium at room temperature, the strain ageing effect hardly appears[1][2] and the stage II of work hardening is absent.[4] Consequently, room temperature may not necessarily be a low temperature for aluminium.

On ageing at room temperature after deformation at the temperature of liquid air, not only aluminium but also other pure metals undergo easily a marked recovery in electrical resistance,[14] and a marked annealing-out of lattice defects.[4] One possible cause, by which pure metals and aluminium alloys show only a slight strain age hardening, may be attributed to such an instability. In copper alloys, lattice defects are more stable and more remain even after ageing at room temperature.[14]

2. Strain age hardening is generally marked in alloys showing a marked X-ray line broadening after cold working and a high rate of work hardening. These are also easily guessed from the theory proposed by Fisher et al.[12] with reference to dislocations piled up around the second phase particles in a two-phase alloy. In carbon steels, the higher the carbon content is the higher the rate of work hardening,[15] and the more marked the strain age hardening.[10] Also, the trend of the increase of line broadening with the progress of cold working[16] is in good correspondence to those of strain age hardening and inelastice effect.[10] In Al-Cu alloy in a fully age-hardened state, in which the GP zones precipitate, the rate of work hardening and the line broadening are small, although the yield stress is appreciably high, compared with those in an over-aged state in which θ' and θ phases precipitate.[9] It appears highly possible that in the former dislocations pass through GP zones, whereas in the latter the precipitated particles act as an effective barrier against the movement of dislocations and are encircled by many piled-up dislocations during deformation. Such behaviors at various stages of ageing generally correspond to those of inelastic effect and strain age hardening. It is considered that the principal effect of the dispersion of particles on strain age hardening is to strain matrix, by which they are encirlced rather than to be related with chemical change such as dissolution into matrix or further precipitation from matrix. This may be supported by the remarkable strain age hardening of two-phase alloys, in which thermally stable alumina particles are finely dispersed.[10] In general, the lower the deformation temperature is the more marked the line broadening,[17] and the larger the rise in yield stress on ageing.[1] With regard to pure aluminium, however, the line broadening is not marked even when deformed at the temperature of liquid air.[13]

3. Strain age hardening is generally marked in alloys having a high density of pile up dislocations after deformation. The formation of cell structure is con-

(13) J.E. Wilson and L. Thomassen, Trans. ASM, **22** (1934), 769.
(14) W.R. Hibbard, Acta Met., **7** (1959), 565.
(15) E. Schmidtman and U. Kalla, Arch. Eis., **31** (1960), 299.
(16) F.H. Andrew and H. Lee, J. Iron and Steel Inst., **165** (1950), 369.
(17) M.S. Paterson and E. Orowan, Nature, **162** (1948), 991.

sidered to be a recovery process, resulting in substantial decrease in the density of piled-up dislocations. In aluminium, piled-up dislocations are easy to induce leakage during deformation,[18] the cell wall is thin and sharp,[19] and the dislocation density is appreciably low.[20] At another extreme from it is cold-worked α brass, in which the density is very high,[20] and the cell structure is hard to form [19][31]. Considering from the observations on electrical resistivity,[8] thermoelectric force and density, dislocation density in α brass and α Cu-Al alloys[11] is dependent on solute concentration, and varies widely with the type of cold working.

Aluminium field at the temperature of liquid nitrogen and maintained at −160°C shows no trace of peak displacements or peak asymmetry,[4] and is apparently an extreme example having no stacking fault. The absence of fault in aluminium may be due to high twinning energy compared with other face-centered cubic metals. At another extreme from aluminium are cold-worked α Cu-Zn[12] and α Cu-Al[22] alloys and austenitic Ni-Cr steels[23]. The probability of forming deformation fault in α Cu-Al alloys[22] is small in a low range of aluminium content, but it increases markedly above 3 wt. per cent Al, reaching a high value at 8 wt. per cent Al. Such a concentration dependence is quite similar to those of the cell formation[31], of the magnitude of changes in electrical resistivity,[8] thermoelectric force and density with cold working, and it is also apparent in the degrees of inelastic effect and strain age hardening.[11] The probability of forming stacking faults is small in Al-Cu alloy, while it is large in Al-Mg alloy.[30] This is in good correspondence to the results in the present investigation, and thus may support the presumption above stated.

4. In two-phase alloys, the degrees of strain age hardening and inelastic effect increase rapidly about in the first 10 per cent of deformation. On the other hand, in one-phase alloys, they begin to increase abruptly above a particular degree of cold working, and this degree is dependent on the deformation temperature. Figs. 12 and 13 are the results on α brass. The inelastic effect λ/λ_0 was measured at various temperatures by the same method as that concerned with Fig. 8. The degree of cold working, above which the degrees of inelastic effect and strain age hardening increase abruptly, are the larger the lower the temperature of deformation. Above about 60°C, they behave similarly to the case of two-phase alloy.

Such a temperature dependence is analogous to that of strain extent of easy glide in single crystals[24]. The extent generally becomes wide with the increase of solute content in solid solution,[25] and with the lowering of deformation tem-

(18) A. Kelly, Acta. Cryst., **7** (1954), 554.
(19) A. Howie, Direct Observations of Imperfections in Crystals, Metal Soc. AIME, (1962), 283.
(20) S. Seeger, J. Diehl, S. Mader and H. Rebstock, Phil. Mag., **2** (1957), 323.
(21) B.E. Warren and E.P. Warekois, Acta Met., **3** (1955), 473.
(22) K. Nakajima, Sci. Rep. RITU, **A12** (1960), 209.
(23) M.J. Whelan and P.B. Hirsch, Proc. Roy. Sec., **A240** (1957), 524.
(24) E.N. Andrade and D.H. Aboav, Proc. Roy. Soc., **A240** (1957), 304.

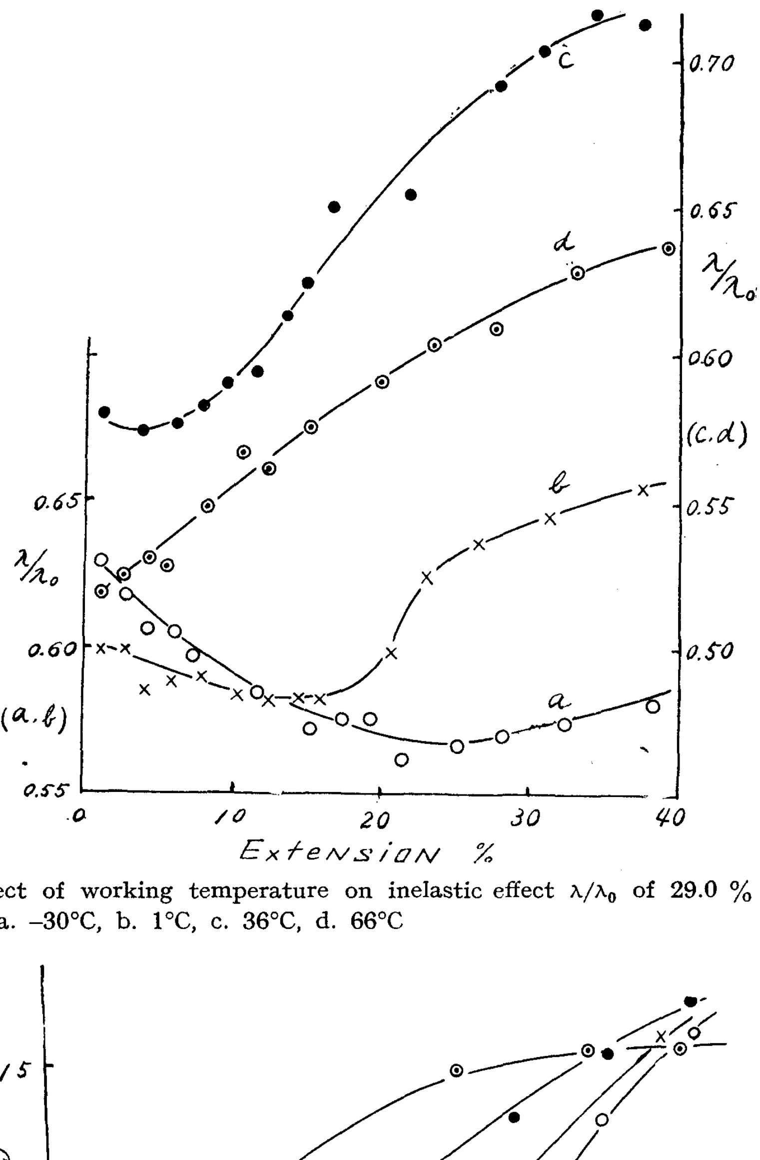


Fig. 12. Effect of working temperature on inelastic effect λ/λ_0 of 29.0 % Zn-Cu alloy.
a. -30°C, b. 1°C, c. 36°C, d. 66°C

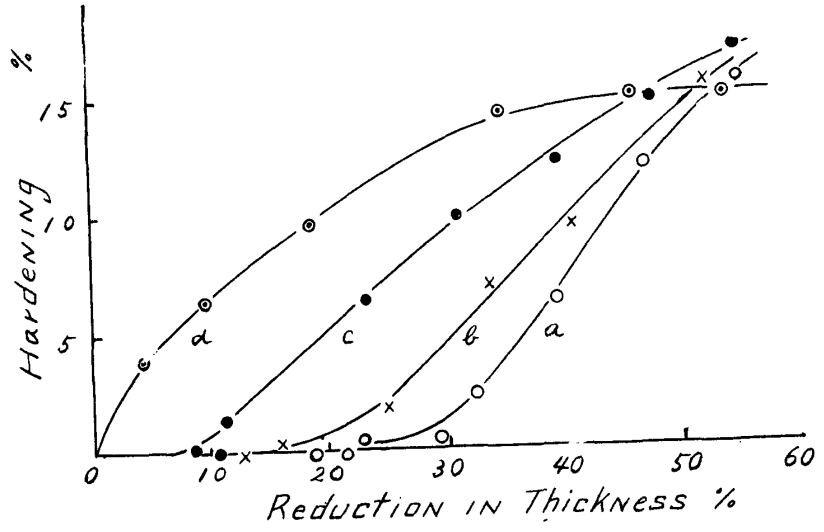


Fig. 13. Effect of rolling temperature on strain age hardening of 29.0% Zn-Cu alloy.
a. -30°C, b. -10°C,. c. 19°C, d. 95°C

perature.[24] The width of the extent has been interpreted in terms of the difficulty in the formation of sessile dislocation of Cottrell-Lohmer type[20]. This sessile

(25) J. Garstone and R.W.K. Honeycombe, Acta Met., 4 (1956), 485.

dislocation is a strong barrier and it is the stronger the wider the stacking faults, and thus the higher the solute content. It may be unacceptable to connect the extent of easy glide in single crystal with the abrupt increase in strain age hardening or inelastic effect above stated, as grain boundary exists. The abrupt increase in one-phase alloy above stated, however, may be explained from the viewpoint that above the working degree where the abrupt increase begins, obstalces and piled-up dislocations are formed in a large number. On the presumption above stated, it may be said that the principal effect of alloying on the strain age hardening is to form strong barriers against the movement of dislocations and to produce a high density of piled-up dislocations with an appreciable resistance to their leakage during deformation, rather than to take short range ordering or to segregate into stacking faults during low temperature annealing. This may also be understood from the observation that the strain age hardening is common to one-phase alloys including pure metals and two phase alloys.

Strain age hardening is closely related with inelastic effect as stated previously. Inelastic effect is related with work hardening,(26) especially with work hardening rate;(27) This effect is not so much affected by Heyn's stress.(28) These may also be recognized from the present investigation. Hence, this effect is considered to be due chiefly to the back stress under which dislocations in piled-up groups easily move back. Further, it is likely that elastic after-effect is an ageing phenomenon due to the same cuase, that is, α brass undergoes a hardening on ageing at room temperature after cold working simultaneously with a contraction in the direction of extension, drawing or rolling,(11) and also with a decrease in internal friction.(29)

Summary

Strain age hardening of aluminium alloys was studied and in addition, the relation of the strain age hardening and the work hardening was discussed. The conclusions obtained may be summarized as follows:

1. The strain age hardening of aluminium alloys is generally analogous in its characteristics to austenitic Ni-Cr steels previously reported, and thus it may be induced by the same cause.

2. In Al-Cu alloy, the inelastic effect and the strain age hardening are more marked in an over-aged state than in a fully age-hardened state, the cause being probably that in the latter dislocations pass through GP zones, while in the former the precipitated particles act as an effective barrier against the movement of dislocations, inducing a large number of piled-up dislocations.

3. Strain age hardening is generally marked in such alloys as containing a high density of pile-up dislocations after cold working. It is considered that the effect

(26) S.N. Buckely and K.M. Endwhistle, Acta Met., **4** (1956), 352.
(27) S. Kumakura, Meeting of Japan Soc. Mech. Eng., Sept., (1961), at Nagaoka.
(28) R.W. Wooley, Phil. Mag., **44** (1953), 597.
(29) W.Z. Koester and K.Z. Rosenthal, Z. Metallk., **30** (1938), 345.

of alloying on the strain age hardening is to form strong barriers and piled-up dislocations in a large number during cold working, rather than to take short range ordering or to segregate into stacking faults during low temperature annealing.

4. Strain age hardening seems to be a general phenomenon in metals and alloys.

In conclusion, the author expresses his cordial thanks to Dr. Y. Imai and Dr. S. Kôda, professors of the Tohoku University, for kind direction and encouragement.

(30) H.J. Seeman and F. Stavenow, Z. Metallk., **52** (1961), 667.
(31) P.W. Swann and J. Nutting, J. Inst. Metals, **90** (1961–62), 133.
(32) K. Nishino, Sci. Rep. RITU, **A 15** (1963), 253.