Initial softening in some aluminium base precipitation hardening alloys

T. V. RAJAN and M. N. SAXENA

IT has been reported by previous workers[1,2] that some extent of softening is observed before setting in of the usual hardening process when ageing is carried out on the Al-Cu and Al-Mg precipitation hardening alloys. The possible reason for the initial softening has been suggested as relief of thermal strain. No experimental evidence in support of this postulate has been reported so far.

The present work was undertaken to make a systematic study of initial softening in certain Al-Cu and Al-Mg alloys. It was proposed to study the phenomenon of initial softening as a function of solute concentration, quenching medium and temperature of ageing. Hardness measurements were carried out to follow the process of softening and relief of thermal strain was studied by analysing X-ray line profile.

Experimental procedure

Preparation of alloys

Binary Al-Cu and Al-Mg alloys were prepared from super purity aluminium (99.9%) and high purity copper and magnesium. All melting was carried out in graphite crucibles placed in electrical resistance furnaces. Required quantity of magnesium wrapped in aluminium foil was added to molten aluminium. Loss of magnesium in the form of oxide was substantially reduced by keeping the metal dipped in the molten aluminium till all of it got melted. The alloys were chill-cast in mild steel moulds. Hexachloroethane was used as degassant.

The cast alloys were forged and then annealed at 350°C for three days to ensure removal of microinhomogeneity and cast structure. Annealing was followed by machining out disc shaped specimens 20mm dia. × 8mm thick. On one side of the specimens, numbers were punched for identification and the other side was polished so that subsequent to heat-treatment very little polishing was required for taking hardness values, thus reducing the handling of heat-treated specimens to a minimum.

Mr T. V. Rajan and Dr M. N. Saxena, University of Roorkee, Roorkee.

SYNOPSIS

Studies were carried out on relief of thermal strain that is produced by quenching precipitation hardening alloys from the temperature of solution treatment. Binary Al-Cu alloys containing 2 to 4.5 wt.% Cu and Al-Mg alloys containing 6 to 10 wt.% Mg were chosen for this purpose. Two types of quenching media viz. water at 20°C and brine water at 0°C were used. The quenched specimens were aged at various temperatures and hardness values were recorded with time. X-ray line profile analysis was also carried out for these specimens to estimate the lattice strain associated with them.

It has been observed that during the process of initial ageing there is softening followed by usual hardening. This phenomenon of initial softening is observable only over a definite range of ageing temperatures. The extent of softening has been found to be a function of (a) temperature of ageing, (b) solute concentration and (c) quenching medium. The time required for reaching minimum hardness has also been observed to be temperature dependant. X-ray line pofile analysis has revealed an initial sharpening of the lines followed by their broadening.

Above results can be explained in terms of two processes that take place simultaneously as ageing starts. One is the relief of thermal strains causing reduction in lattice distortion and consequent softening. The other process is clustering, zone formation of intermediate precipitate—all of which are responsible for hardening. Superimposition of these two phenomena leads to initial softening followed by hardening.

The nominal compositions of six binary Al-Cu alloys were from Al-2% Cu to Al-4.5% Cu at intervals of 0.5% Cu. The actual composition varied from the nominal within the limits of ± 0.05%.

The nominal composition of binary Al-Mg alloys were Al-6% Mg, Al-8% Mg and Al-10% Mg. The actual composition varied from the nominal within the limits of ± 0.1%.

> **1** *Quenched hardness values of Al-Cu alloys*
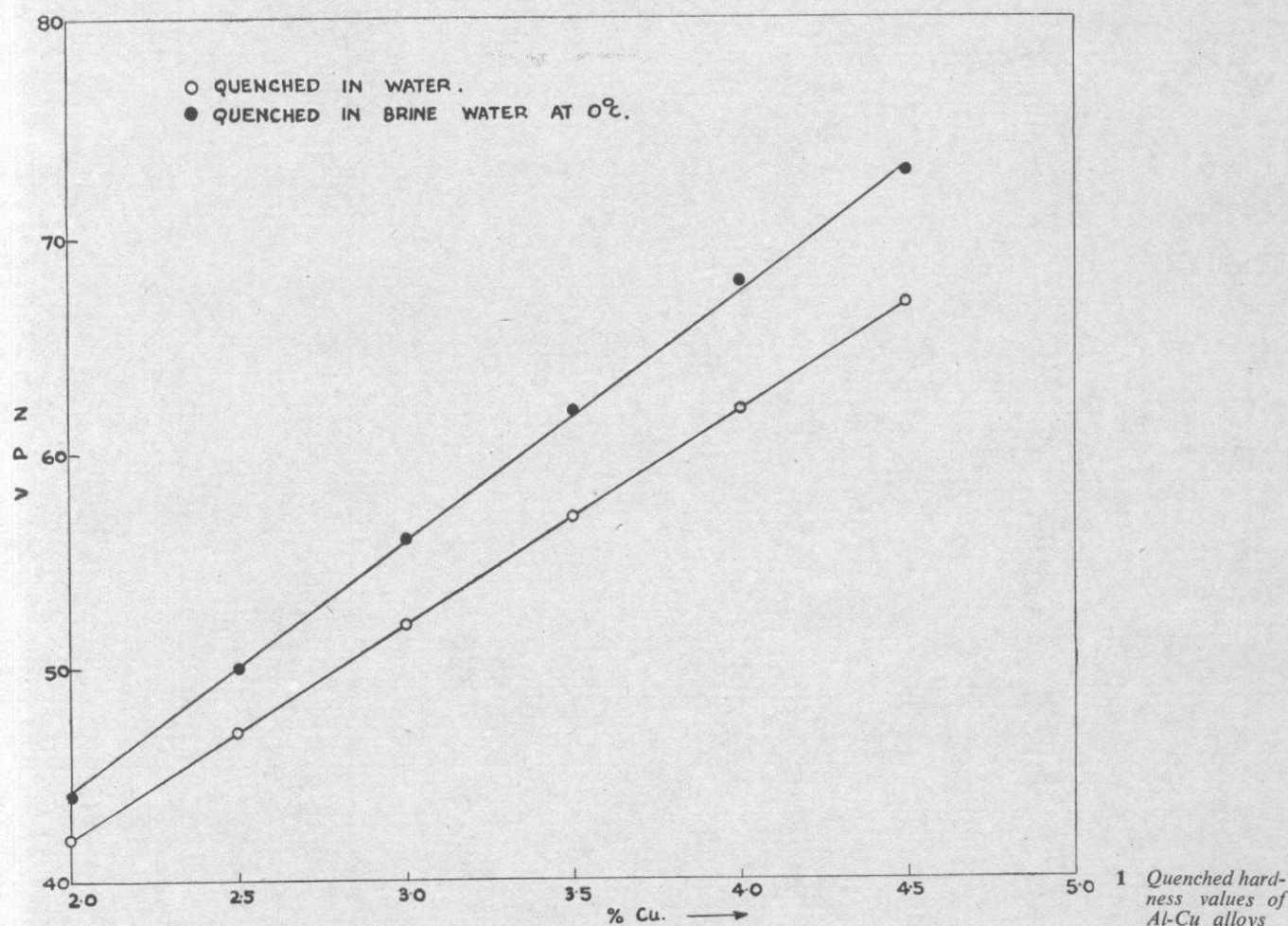
Heat treatment

Initial solution treatment was carried out in muffle furnaces for at least 48 hours at 520°C for all the Al-Cu alloys except Al-4·5% Cu which was solution treated at 530°C. Al-Mg alloys were solution treated at 450°C. The solution treated specimens were quenched in (1) water at (20 ± 1)°C or (2) brine water at 0°C. Microscopic examination of as-quenched specimens revealed the complete dissolution of the second phase into the parent phase.

The solution treated, quenched specimens were aged at 110°C, 130°C, 150°C, 170°C, 190°C and 210°C for the binary Al-Cu alloys and 200°C, 250°C and 300°C for the Al-Mg alloys. Within 5 minutes of quenching, the specimens were put for ageing treatment. It has been shown by previous workers that heat treatment does not result in loss of either copper[1] or magnesium.[3]

Hardness measurements

Hardness values were determined on Vickers Hardness tester with 5 kg load ; 4 specimens were taken out at the end of each ageing period and 3 hardness values were determined on each specimen. Average of a set of 12 readings was taken for determining each hardness value on ageing.

X-ray diffraction studies

For the purpose of X-ray studies the specimens were mounted on perspex sheet after polishing them. A blank run with perspex sheet was carried out to ascertain that no peaks appear due to perspex. The target used in the X-ray tube was iron. Manganese filter was used to cut off $K\beta$ radiation. However, as no crystal monochromatizer was used, the radiation of $K\alpha$ consisted of doublet.

YPC–50 type X-ray diffractometer unit was used. The counter was run at the rate of $\frac{1}{2}$° per minute. Bragg angle vs intensity graph was plotted with automatic stripchart potentiometer. The graph was run for 2° on either side of the peak position to determine the background. The half width (β) of the diffracted line was evaluated by dividing the area under the curve by the peak height above the background.

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2 *Quenched hardness values of Al-Mg alloys*
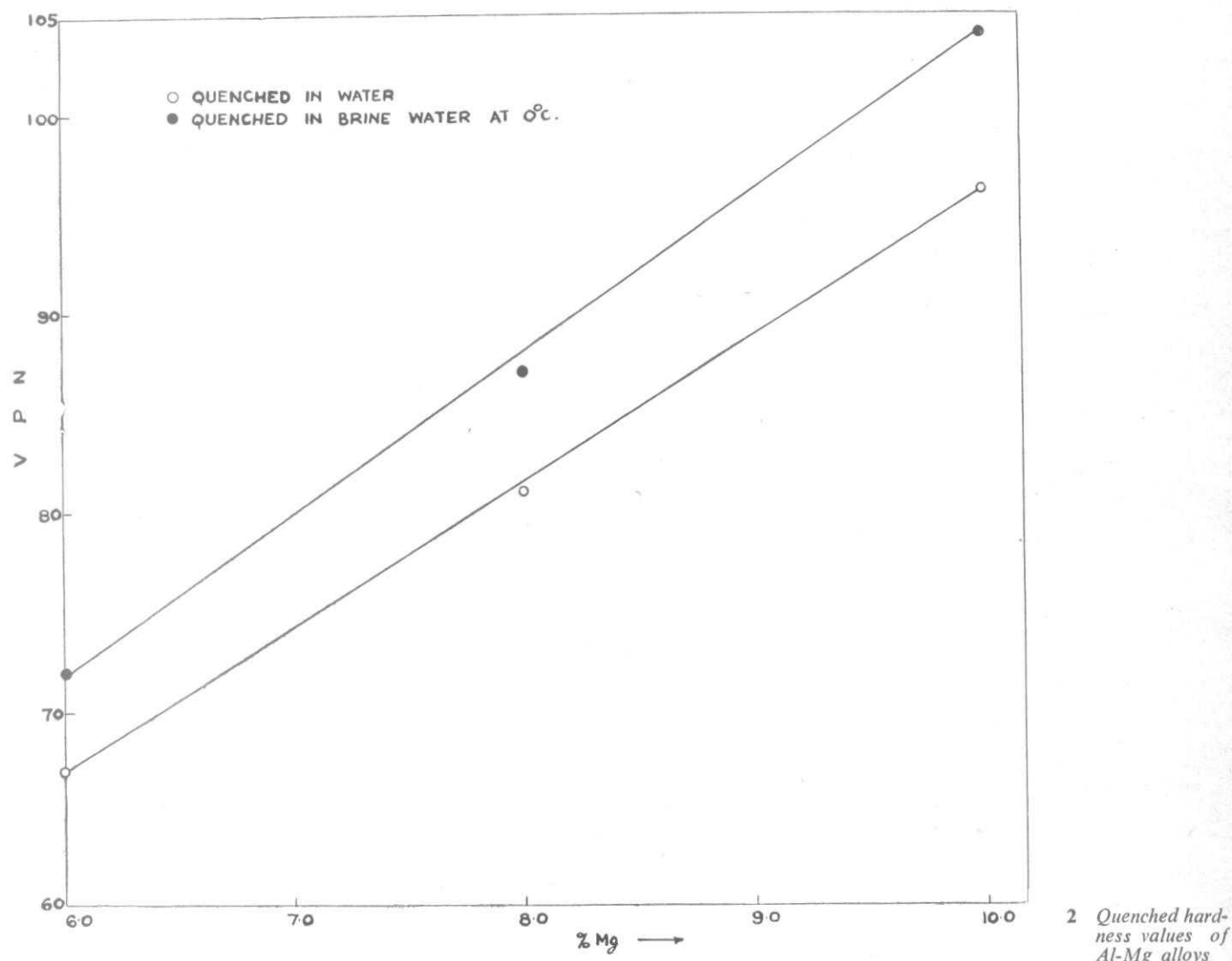
Results

Figures 1 and 2 show the quenched hardness values of binary Al-Cu and Al-Mg alloys with (a) water at 20°C and (b) brine water at 0°C as quenching media.

In Figures 3 to 5 are plotted the relationships between the extent of softening and solute concentration in binary Al-Cu and Al-Mg alloys at various ageing temperatures. Figures 4 and 5 compare the extent of softening in Al-Mg alloys with the two types of quenching media. In case of binary Al-Cu alloys the extent of softening with water quench was very small, especially with lower copper contents (Cu less than 3·5%) and hence have not been plotted for comparison.

Figure 6 shows the temperature/time-to-minimum hardness relationship for the binary Al-Cu alloys.

Table I summarizes the results obtained on the X-ray diffractometer.

Discussion

Quenched hardness

From Figures 1 and 2, it is evident that quenched hardness increases with increase in solute concentration (viz. Cu or Mg as the case may be). The hardness of quenched alloy is attributable to :

(a) Solid solution hardening.
(b) Lattice distortion due to supersaturation.
(c) Lattice distortion due to quenching.

In comparing the quenched hardness of alloys quenched under similar conditions, it is only supersaturation that alters the hardness value. This explains the linear increase in as-quenched hardness with increase in (a) % Cu and (b) % Mg. The quenched hardness, with brine water at 0·C as the quenching medium, is higher

TABLE I Results obtained on X-ray diffractometer

Composition of alloy	Quenching medium	Ageing temperature (°C)	Ageing time		Indices of plane of reflection	β (Degrees)
Al–4·5% Cu	Brine water at 0°C	As quenched	—		(111)	0·41
,,	,,	,,	—		(200)	0·52
,,	,,	170	0·5	Tm*	(111)	0·38
,,	,,	,,		Tm	,,	0·29
,,	,,	,,	2·0	Tm	,,	0·36
,,	,,	,,	0·5	Tm	(200)	0·47
,,	,,	,,		Tm	,,	0·32
,,	,,	,,	2·0	Tm	,,	0·45
,,	,,	190	0·5	Tm	(111)	0·36
,,	,,	,,		Tm	,,	0·21
,,	,,	,,	2·0	Tm	,,	0·37
,,	,,	,,	0·5	Tm	(200)	0·41
,,	,,	,,		Tm	,,	0·25
,,	,,	,,	2·0	Tm	,,	0·48
Al–4% Cu	,,	As quenched	—		(111)	0·37
,,	,,	,,	—		(200)	0·46
Al–4·0% Cu	,,	150	0·5	Tm	(111)	0·31
,,	,,	,,		Tm	,,	0·32
,,	,,	,,	2·0	Tm	,,	0·40
,,	,,	,,	0·5	Tm	(200)	0·41
,,	,,	,,		Tm	,,	0·34
,,	,,	,,	2·0	Tm	,,	0·40
,,	,,	170	0·5	Tm	(111)	0·34
,,	,,	,,		Tm	,,	0·30
,,	,,	,,	2·0	Tm	,,	0·35
,,	,,	170	0·5	Tm	(200)	0·39
,,	,,	,,		Tm	,,	0·29
,,	,,	,,	2·0	Tm	,,	0·48
,,	,,	190	0·5	Tm	(111)	0·32

* Tm—Time to reach minimum hardness

TABLE I—*contd.*

Composition of alloy	Quenching medium	Ageing temperature (°C)	Ageing time		Indices of plane of reflection	β (Degrees)
Al–40% Cu	Brine water at 0°C	190		Tm*	(111)	0·24
,,	,,	,,	2·0	Tm	,,	0·35
Al–4% Cu	,,	,,	0·5	Tm	(200)	0·40
,,	,,	,,		Tm	,,	0·27
,,	,,	,,	2·0	Tm	,,	0·35
Al–3·5% Cu	,,	As quenched		—	(111)	0·32
,,	,,	,,		—	(200)	0·40
,,	,,	150	0·5	Tm	(111)	0·28
,,	,,	,,		Tm	,,	0·20
,,	,,	,,	2·0	Tm	,,	0·25
,,	,,	,,	0·5	Tm	(200)	0·32
,,	,,	,,		Tm	,,	0·26
,,	,,	,,	2·0	Tm	,,	0·43
,,	,,	170	0·5	Tm	(111)	0·26
,,	,,	,,		Tm	,,	0·19
,,	,,	,,	2·0	Tm	,,	0·34
,,	,,	,,	0·5	Tm	(200)	0·30
,,	,,	,,		Tm	,,	0·25
,,	,,	,,	2·0	Tm	,,	0·37
,,	,,	190	0·5	Tm	(111)	0·19
,,	,,	,,		Tm	,,	0·14
,,	,,	,,	2·0	Tm	,,	0·30
,,	,,	,,	0·5	Tm	(200)	0·28
,,	,,	,,		Tm	,,	0·22
,,	,,	,,	2·0	Tm	,,	0·42
Al–3% Cu	,,	As quenched		—	(111)	0·28
,,	,,	,,		—	(200)	0·35
,,	,,	130	0·5	Tm	(111)	0·28
,,	,,	,,		Tm	,,	0·20

* Tm—Time to reach minimum hardness

TABLE I—contd.

Composition of alloy	Quenching medium	Ageing temperature (°C)	Ageing time		Indices of plane of reflection	β (Degrees)
Al–3% Cu	Brine water at 0°C	130	2·0	Tm*	(111)	0·23
,,	,,	,,	0·5	Tm	(200)	0·31
,,	,,	,,		Tm	,,	0·27
,,	,,	,,	2·0	Tm	,,	0·37
,,	,,	150	0·5	Tm	(111)	0·25
,,	,,	,,		Tm	,,	0·20
,,	,,	,,	2·0	Tm	,,	0·31
,,	,,	,,	0·5	Tm	(200)	0·28
,,	,,	,,		Tm	,,	0·24
,,	,,	,,	2·0	Tm	,,	0·37
,,	,,	170	0·5	Tm	(111)	0·26
,,	,,	,,		Tm	,,	0·17
,,	,,	,,	2·0	Tm	,,	0·29
,,	,,	,,	0·5	Tm	(200)	0·27
,,	,,	,,		Tm	,,	0·22
,,	,,	,,	2·0	Tm	,,	0·36
,,	,,	190	0·5	Tm	(111)	0·23
,,	,,	,,		Tm	,,	0·18
,,	,,	,,	2·0	Tm	,,	0·26
,,	,,	,,	0·5	Tm	(200)	0·29
,,	,,	,,		Tm	,,	0·20
,,	,,	,,	2·0	Tm	,,	0·39
Al–2·5% Cu	,,	As quenched	—		(111)	0·24
,,	,,	,,	—		(200)	0·30
,,	,,	130	0·5	Tm	(111)	0·24
,,	,,	,,		Tm	,,	0·20
,,	,,	,,	2·0	Tm	,,	0·27
,,	,,	,,	0·5	Tm	(200)	0·28
,,	,,	,,		Tm	,,	0·24

* Tm—Time to reach minimum hardness

TABLE I—*contd.*

Composition of alloy	Quenching medium	Ageing temperature (°C)	Ageing time		Indices of plane of reflection	β (Degrees)
Al–2·5% Cu	Brine water at 0°C	130	2·0	Tm*	(200)	0 32
,,	,,	150	0·5	Tm	(111)	0·26
,,	,,	,,		Tm	,,	0·17
,,	,,	,,	2·0	Tm	,,	0·21
,,	,,	,,	0·5	Tm	(200)	0·27
,,	,,	,,		Tm	,,	0·24
,,	,,	,,	2·0	Tm	,,	0·31
,,	,,	170	0·5	Tm	(111)	0·24
,,	,,	,,		Tm	,,	0·15
,,	,,	,,	2·0	Tm	,,	0·23
,,	,,	,,	0·5	Tm	(200)	0·25
,,	,,	,,		Tm	,,	0·20
,,	,,	,,	2·0	Tm	,,	0·29
,,	,,	190	0·5	Tm	(111)	0·22
,,	,,	,,		Tm	,,	0·11
,,	,,	,,	2·0	Tm	,,	0·34
,,	,,	,,	0·5	Tm	(200)	0·30
,,	,,	,,		Tm	,,	0·18
,,	,,	,,	2·0	Tm	,,	0·32
Al 10% Mg	,,	As quenched	—		(111)	0·48
,,	,,	,,	—		(200)	0·59
,,	,,	200	0·5	Tm	(111)	0·43
,,	,,	,,		Tm	,,	0·34
,,	,,	,,	2·0	Tm	,,	0·47
,,	,,	,,	0·5	Tm	(200)	0·59
,,	,,	,,		Tm	,,	0·51
,,	,,	,,	2·0	Tm	,,	0·51
,,	,,	250	0·5	Tm	(111)	0·46
,,	,,	,,		Tm	,,	0·37

* Tm—Time to reach minimum hardness

TABLE I—*contd.*

Composition of alloy	Quenching medium	Ageing temperature (°C)	Ageing time		Indices of plane of reflection	β (Degrees)
Al–10% Mg	Brine water at 0°C	250	2·0	Tm*	(111)	0·40
,,	,,	,,	0·5	Tm	(200)	0·54
,,	,,	,,		Tm	,,	0·44
,,	,,	,,	2·0	Tm	,,	0·56
,,	,,	,,		Tm	,,	0·39
,,	,,	,,	2·0	Tm	,,	0·50
,,	,,	300		Tm	,,	0·46
,,	,,	,,	2·0	Tm	,,	0·53
Al–8% Mg	,,	As quenched		—	(111)	0·44
,,	,,	,,		—	(200)	0 53
,,	,,	200	0·5	Tm	(111)	0·34
,,	,,	,,		Tm	,,	0·32
,,	,,	,,	2·0	Tm	,,	0·41
,,	,,	200	0·5	Tm	(200)	0·50
,,	,,	,,		Tm	,,	0·41
,,	,,	,,	2·0	Tm	,,	0·52
,,	,,	250	0·5	Tm	(111)	0·45
,,	,,	,,		Tm	,,	0·33
,,	,,	,,	2·0	Tm	,,	0·32
,,	,,	,,	0·5	Tm	(200)	0·50
,,	,,	,,		Tm	,,	0·40
,,	,,	,,	2·0	Tm	,,	0·45
,,	,,	300	0·5	Tm	(111)	0·48
,,	,,	,,		Tm	,,	0·44
,,	,,	,,	2·0	Tm	,,	0·58
,,	,,	,,	0·5	Tm	(200)	0·52
,,	,,	,,		Tm	,,	0·37
,,	,,	,,	2·0	Tm	,,	0·44
Al–6% Mg	,,	As quenched		—	(111)	0 39

* Tm—Time to reach minimum hardness

TABLE I—*contd.*

Composition of alloy	Quenching medium	Ageing temperature (°C)	Ageing time		Indices of plane of reflection	β (Degrees)
Al–6% Mg	Brine water at 0°C	As quenched	—		(200)	0·45
,,	,,	200	0·5	Tm*	(111)	0·39
,,	,,	,,		Tm	,,	0·25
,,	,,	,,	2·0	Tm	,,	0·33
,,	,,	,,	0·5	Tm	(200)	0·46
,,	,,	,,		Tm	,,	0·32
,,	,,	,,	2·0	Tm	,,	0·39
,,	,,	250	0·5	Tm	(111)	0·35
,,	,,	,,		Tm	,,	0·27
,,	,,	,,	2·0	Tm	,,	0·33
,,	,,	,,	0·5	Tm	(200)	0·42
,,	,,	,,		Tm	,,	0·35
,,	,,	,,	2·0	Tm	,,	0·48
,,	,,	300	0·5	Tm	(111)	0·37
,,	,,	,,		Tm	,,	0·29
,,	,,	,,	2·0	Tm	,,	0·29
,,	,,	,,	0·5	Tm	(200)	0·43
,,	,,	,,		Tm	,,	0·37
,,	,,	,,	2·0	Tm	,,	0·45
Al–10% Mg	Water at 20°C	As quenched	—		(111)	0·40
,,	,,	,,	—		(200)	0·52
,,	,,	200	0·5	Tm	(111)	0·38
,,	,,	,,		Tm	,,	0·27
,,	,,	,,	2·0	Tm	,,	0·34
,,	,,	,,	0·5	Tm	(200)	0·44
,,	,,	,,		Tm	,,	0·35
,,	,,	,,	2·0	Tm	,,	0·51
,,	,,	250	0·5	Tm	(111)	0·40
,,	,,	,,		Tm	,,	0·29

* Tm—Time to reach minimum hardness

TABLE I—*contd.*

Composition of alloy	Quenching medium	Ageing temperature (°C)	Ageing time		Indices of plane of reflection	β (Degrees)
Al-10% Mg	Water at 20°C	250	2·0	Tm*	(111)	0·42
,,	,,	,,	0·5	Tm	(200)	0·47
,,	,,	,,		Tm	,,	0·37
,,	,,	,,	2·0	Tm	,,	0·48
,,	,,	,,		Tm	,,	0·31
,,	,,	,,	2·0	Tm	,,	0·46
,,	,,	,,		Tm	,,	0·43
,,	,,	,,	2·0	Tm	,,	0·55
Al–8% Mg	,,	As quenched	—		(111)	0·35
,,	,,	,,	—		(200)	0·44
,,	,,	200	0·5	Tm	(111)	0·32
,,	,,	,,		Tm	,,	0·21
,,	,,	,,	2·0	Tm	,,	0·27
,,	,,	,,	0·5	Tm	(200)	0·39
,,	,,	,,		Tm	,,	0·36
,,	,,	,,	2·0	Tm	,,	0·51
,,	,,	250	0·5	Tm	(111)	0·34
,,	,,	,,		Tm	,,	0·24
,,	,,	,,	2·0	Tm	,,	0-31
,,	,,	,,	0·5	Tm	(200)	0-37
,,	,,	,,		Tm	,,	0·37
,,	,,	,,	2·0	Tm	,,	0·47
,,	,,	300	0·5	Tm	(111)	0·30
,,	,,	,,		Tm	,,	0·22
,,	,,	,,	2·0	Tm	,,	0·36
,,	,,	,,	0·5	Tm	(200)	0·40
,,	,,	,,		Tm	,,	0·35
,,	,,	,,	2·0	Tm	,,	0·41
Al–6% Mg	,,	As quenched	—		(111)	0·28

* Tm—Time to reach minimum hardness

TABLE I—concld.

Composition of alloy	Quenching medium	Ageing temperature (°C)	Ageing time		Indices of plane of reflection	β (Degrees)
Al–6% Mg	Water at 20°C	As quenched	—		(200)	0·34
,,	,,	200	0·5	Tm*	(111)	0·26
,,	,,	,,		Tm	,,	0·22
,,	,,	,,	2·0	Tm	,,	0·27
,,	,,	,,	0·5	Tm	(200)	0·34
,,	,,	,,		Tm	,,	0·25
,,	,,	,,	2·0	Tm	,,	0·34
,,	,,	250	0·5	Tm	(111)	0·29
,,	,,	,,		Tm	,,	0·21
,,	,,	,,	2·0	Tm	,,	0·27
,,	,,	,,	0·5	Tm	(200)	0·31
,,	,,	,,		Tm	,,	0·22
,,	,,	,,	2·0	Tm	,,	0·28
,,	,,	300	0·5	Tm	(111)	0·26
,,	,,	,,		Tm	,,	0·23
,,	,,	,,	2·0	Tm	,,	0·32
,,	,,	,,	0·5	Tm	(200)	0·32
,,	,,	,,		Tm	,,	0·24
,,	,,	,,	2·0	Tm	,,	0·37

* Tm—Time to reach minimum hardness

than with water at 20°C as the quenching medium. This is true for every alloy of Al-Cu and Al-Mg and the difference in the as quenched hardness values increases with increase in solute concentration.

The extra hardness with brine water quench is attributable to (i) higher lattice distortion due to greater severity of quenching and (ii) creation of dislocation loops due to possible collapse of vacancy clusters[4] created due to large number of trapped thermal vacancies.

The greater effectiveness of rate of quenching to alter the as-quenched hardness, with increase in solute concentration may be attributed to the following reason.

When the solute concentration is higher, larger number of solute atoms are available in the matrix and they are helpful in setting up lattice distortion during the process of quenching.

Extent of softening

From Figure 3, it can be observed that the extent of softening increases with increase in ageing temperature for Al-Cu alloys. The obvious reason is that, with higher ageing temperatures, the process of relief of thermal strain is nearer to completion. It is also seen from Figure 3 that larger the solute concentration greater is the degree of softening for Al-Cu alloys. With larger solute concentration, larger amount of lattice distortion is introduced while quenching (as discussed already) and relief of this distortion lead to a larger degree of softening.
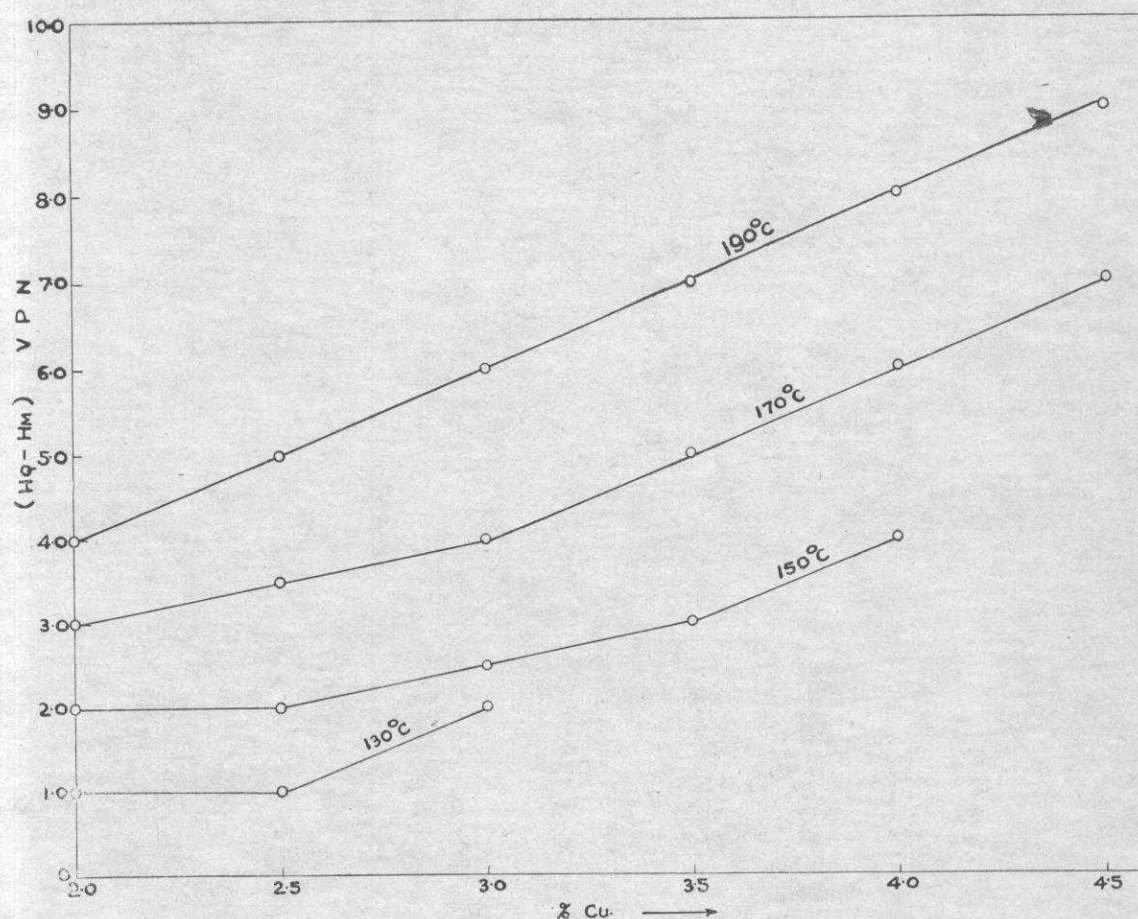
3 Extent of softening in alloys quenched in brine water at 0°C and aged at various temperatures
H_Q-Quenched hardness
H_M-Minimum hardness values observed during initial ageing

It may be observed from Figures 4 and 5 that in contrast to Al-Cu alloys, degree of softening decreases with increase in ageing temperature in the case of Al-Mg alloys. It has been reported[2] that in Al-Mg alloys, there is first discontinuous precipitation of β' followed by continuous precipitation. The discontinuous precipitation starts at the grain boundaries and then proceeds into the interior of grains. The precipitation at the grain boundaries hardly contributes to any hardening and it is only after precipitation within the grains has taken place that increase in hardness results. At low temperature, the grain boundaries act as potential sites for precipitation of β'. However with increasing ageing tempetatures, imperfections like dislocations within the grains also assume importance as potential sites for precipitation of β'.

In other words, with increasing ageing temperature the process of hardening is accelerated doubly fast (i) due to faster precipitation of β' and (ii) due to precipitation of β' taking place within the grains simultaneously with precipitation at grain boundaries. The process of softening also gets accelerated due to faster rate of relief of thermal strain. It is suggested that hardening process is accelerated to a greater extent than the softening process and hence the observed decrease in the

extent of softening with increase in ageing temperature :

From Figures 4 and 5, it is also clear that higher the solute concentration greater is the extent of softening. The explanation is the same as for Al-Cu alloys.

It is also revealed from Figures 4 and 5 that the extent of softening is higher for specimens quenched in brine water than for specimens quenched in water. Specimens quenched in brine water have undergone more severe quenching and hence the process of relief of thermal strain is faster and more appreciable. The result is the greater extent of softening.

Time to reach minimum hardness

In discussing the time to reach minimum hardness, the major difference in the precipitating phases in the Al-Cu and Al-Mg alloy in the range of ageing temperatures under study is to be considered.

Ageing at temperatures from 110°C to 230°C in Al-Cu (Cu-2 to 4·5%) proceeds with precipitation of either G. P. Zones or direct θ' depending on Cu content.[5] With lower ageing temperature and higher Cu content i.e. with increasing degree of supersaturation, the initial hardening constituents are zones whereas with lower Cu content and higher ageing temperature (i.e. with

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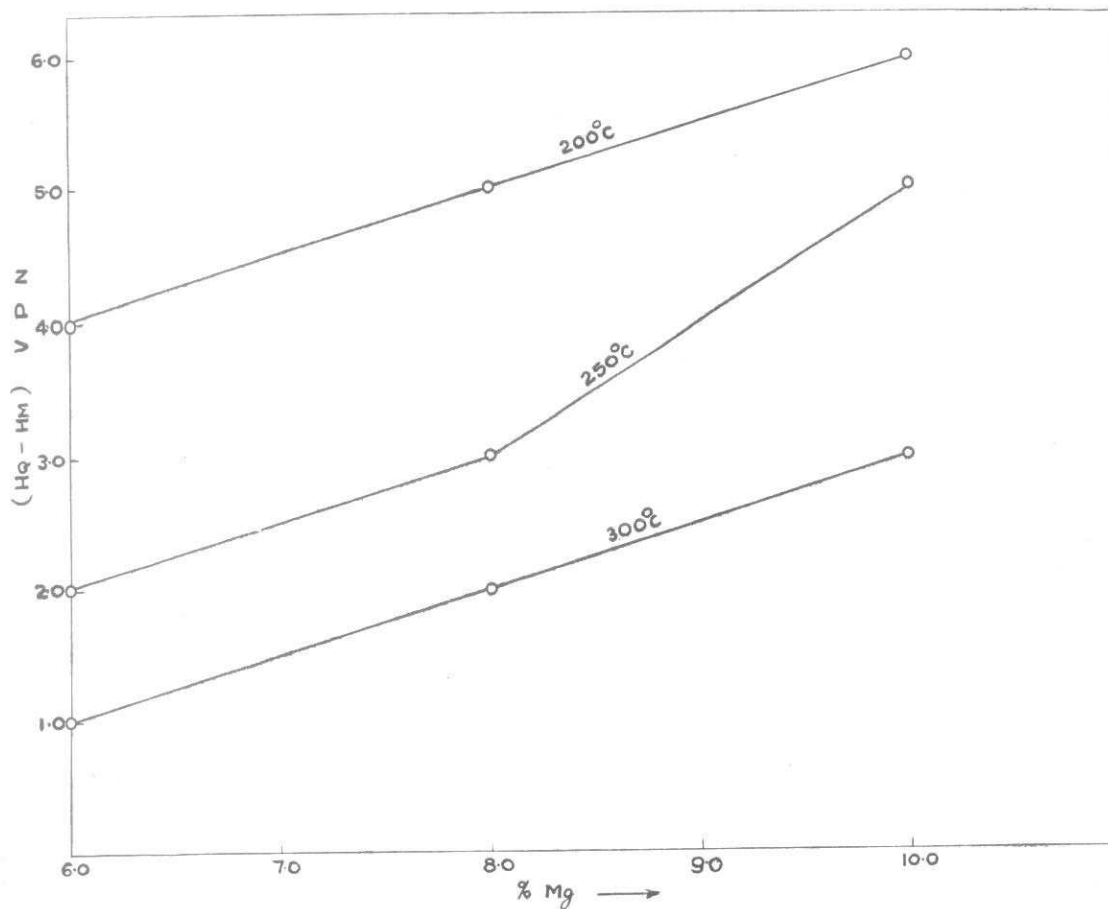
4 *Extent of soften-ing in alloys quen-ched in water at 20°C and aged at various tempera-tures*
H_Q-*Quenched hard-ness*
H_m-*Minimum hard-ness values ob-served during ini-tial ageing*

decreasing degree of supersaturation), there is direct precipitation of θ'.

From Figure 6, it may be observed that in certain Al-Cu alloys, there is steady increase in time required to reach minimum hardness with decrease in ageing tem-perature. This is so because both relief of thermal strain and precipitation of intermediate phase are slowed down with decrease in ageing temperature. However, below a particular temperature of ageing, the time required to attain minimum hardness suddenly drops down instead of increasing. It is suggested that this point of change over denotes that temperature below which there is first zone formation followed by preci-pitation of θ' rather than the direct precipitation of θ'. Since formation of zones has different kinetics than formation of θ' from the supersaturated matrix and since the former pocess is much faster, it is natural to expect a break in the time to minimum hardness vs. ageing time relationships. The break in the above straight lines are in good agreement with the tempera-tures below which zone formation has been reported in the given alloys.[5]

On the other hand, in certain Al-Cu alloys with lower Cu contents, it is found that there is steady increase in time to reach minimum hardness with decrease in ageing temperature. In these alloys[5] there is direct

precipitation of θ' over the complete range of tempera-ture of ageing under consideration.

In all the Al-Mg alloys, there is steady decrease in time to reach minimum hardness with increasing tem-perature because the kinetics of precipitation has remained unchanged due to precipitation of intermediate phase β' as established by earlier workers.[2]

On comparing the time to reach minimum hardness between Al-Mg alloys specimens quenched in water (at 20°C) and brine water at 0°C, it was found that the former always takes longer time than the latter for a given alloy at a given temperature of ageing. It is possible to explain the above observation in terms of faster rate of ageing process in latter case due to:

(i) Presence of strains which facilitate precipitation.
(ii) Presence of large number of thermally trapped vacancies which
  (a) act as potential sites for precipitation of zones[6]
  (b) increase effective rate of diffusion.
(iii) Possible collapse of cluster of vacancies to form dislocation loops[4] or condensation of cluster of vacancies to form voids[7] each of which can act as potential site for precipitation of intermediate phase.
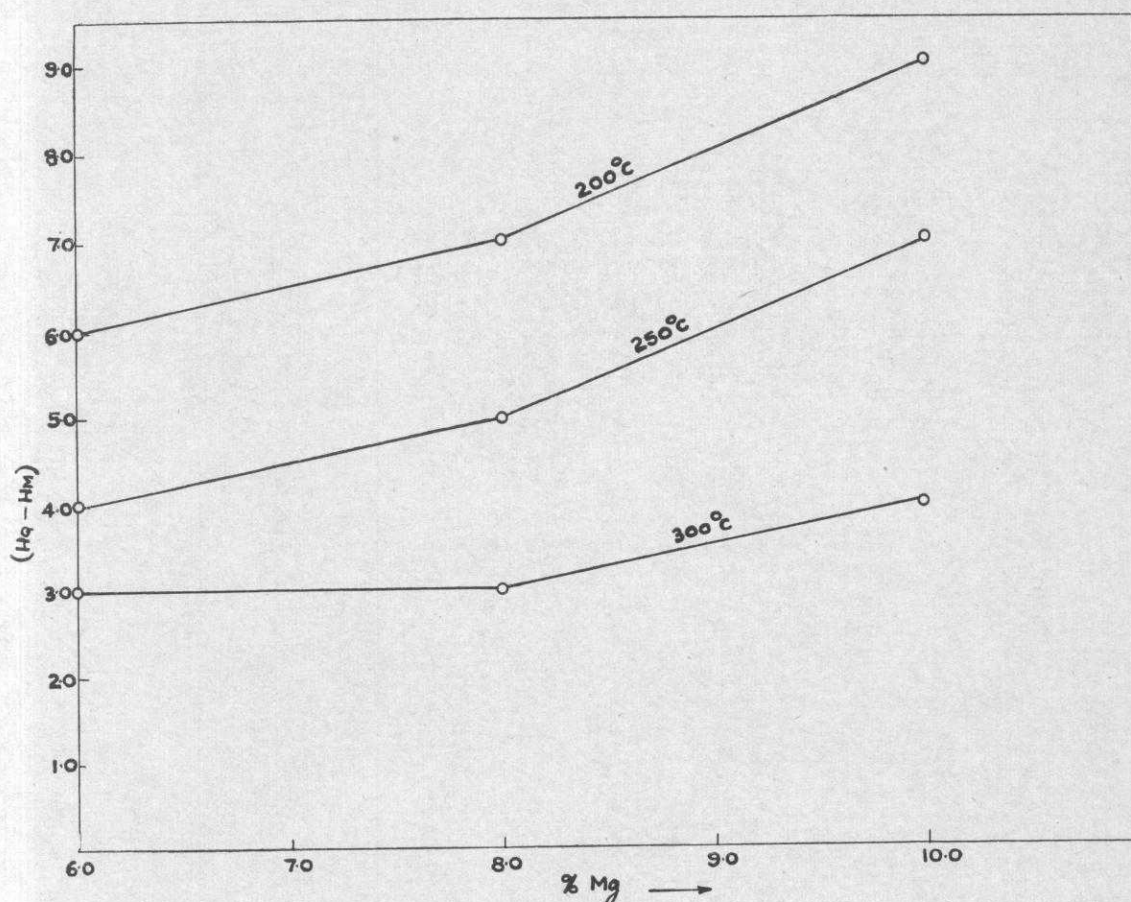
5 *Extent of softening in alloys quenched in brine water at 0˚C and aged at various temperatures*
H_Q-*Quenched hardness*
H_M-*Minimum hardness values obtained during initial ageing*

It was also observed that for Al-Cu as well as Al-Mg alloys the time to reach minimum hardness at a given temperature decreased with increase in solute concentration (Fig. 6). The explanation lies in the fact that higher the solute concentration in the alloy, greater the degree of supersaturation, higher the resulting driving force for precipitation and consequently faster the rate of hardening.

Range of softening

From Figures 3 and 5, it can be seen that for each Al-Cu alloy there is a range of temperature only within which the initial softening can be observed. Existence of such temperature range for softening may be explained as follows :

Below the lower limit, the slow rate of stress relief is overcompensated by zone formation. Hence no effective softening is observable. Above the upper limit, the formation of intermediate phase is so fast that the softening effect is completely masked. It is also possible that softening might have occurred within the first five minutes when hardness values were not recorded.

It can also be noted from Figures 3 and 6 that the temperature range of softening narrows down with increase in copper content. With increase in copper

content there is increase in supersaturation and resulting increase in driving force for (a) zone formation and (b) precipitation of intermediate phase. Also with increase in Cu content, the zones appear at higher ageing temperature.[5] The net result is raising of lower limit and lowering of upper limit i. e. narrowing down of temperature range of softening.

X-ray line width

The line broadeding[8] that takes place in X-ray diffraction pattern is due to (1) particle size and (2) microstresses. If the line broadening is due to particle size alone, then the line width β_P is related to the effective particle size ϵ, the X-ray wave length and Bragg angle θ by the equation :

$$\beta_p = \frac{K}{\epsilon \cos \theta}$$

Where K is a constant nearly equal to unity.

For pure stress broadening the width β_S is related to the effective strain and the Bragg angle by the equation :

$$\beta_s = \eta \tan \theta$$

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6 *Tempetature/ Time-to-Minimum hardness relationships*
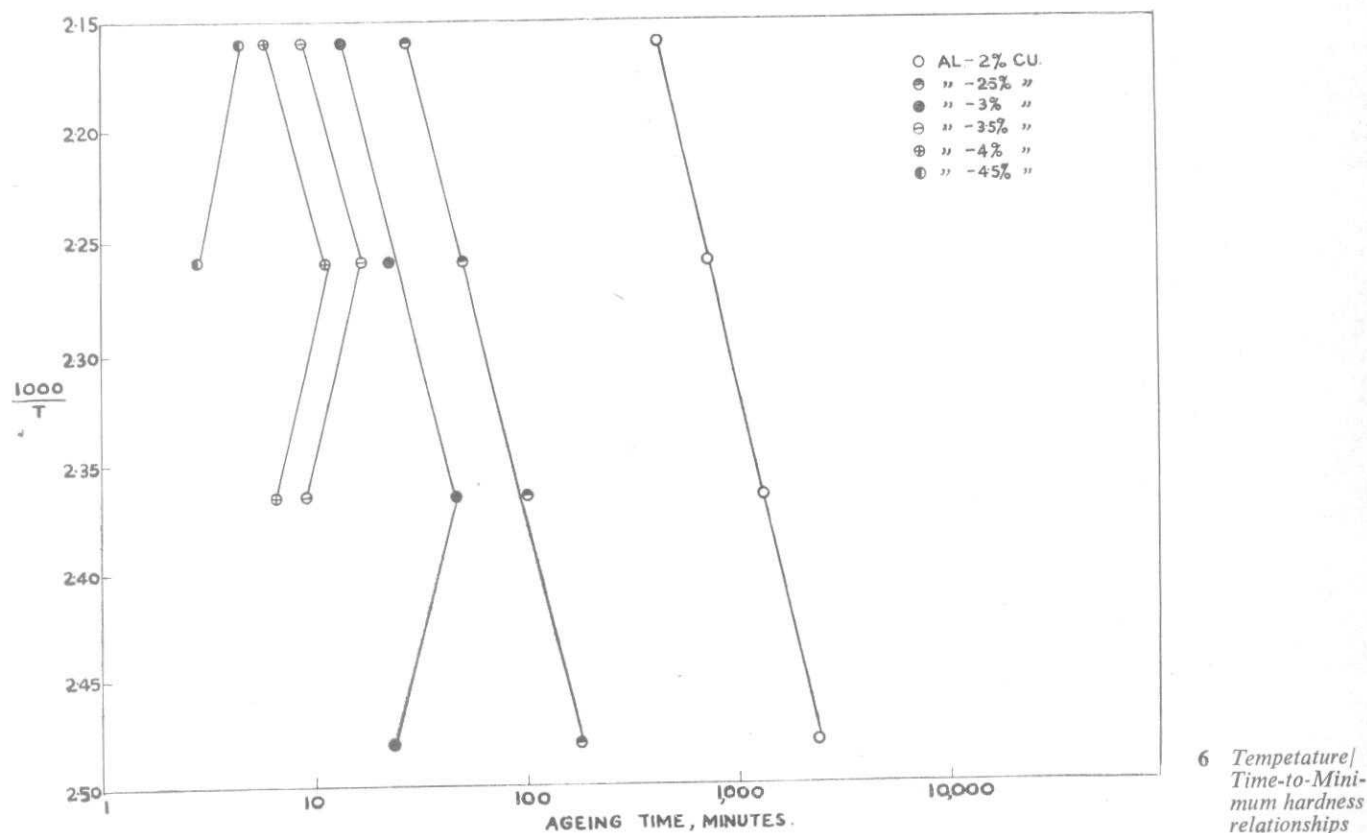
If both types of broadening are present then,

$$\beta = \beta_p + \beta_s, \text{ or}$$

$$\frac{\beta \cos \theta}{\lambda} = \frac{1}{\epsilon} + \frac{\eta \sin \theta}{\lambda}$$

In a distorted lattice, the effective particle size ϵ must be interpreted as a measure of the volume of region in the lattice which diffracts coherently.

Extending the analysis of Wheeler and Jawson[9], it may be concluded that the line broadening observed in the initial stages of hardening in Al-Cu and Al-Mg is due to balanced internal stresses rather than small particle size. That is,

$$\beta = \beta_s \pm \eta \tan \theta$$

If the microstress in the same material under two different conditions are to be compared, it is sufficient to compare the β values of the lines due to same set of planes.

As Jones[10] has pointed out, the observed line widths can be influenced by a number of factors other than particle size and microstress in the specimens. Even a fully annealed specimen, which is relieved of all microstresses and which has "infinite particle size" (i. e., particle size greater than 10 microns), will produce X-ray diffracted lines of finite width. Hence a correc-

tion is to be made to the observed line width before proceeding to estimate particle size or microstress. The correction to be applied is

$\beta^2 = B^2 - b^2$ (assuming Gaussian distribution for the profile of the lines)

where

β is pure diffraction broadening.

β is the observed width with given specimen, b is the observed width when the specimen is in standard condition i.e. in the fully annealed condition.

Yet another correction is to be applied for non-homogeneity of the radiation used for X-ray diffraction work. The Kα radiation used consists of a doublet which causes broadening of the lines since in general the two components are not resolved. Necessary correction has been carried out on the basis of the method suggested by Jones[10].

The values of β so obtained and shown in Table I are in accordance with hardness values. Initially the value of β is large and there is sharpening when the hardness value attains a minimum, followed by broadening of further ageing. The plausible explanation is that the initial width is due to distorted lattice produced as a result of quenching. Subsequent broadening can be attributed to coherency strains developed between the matrix and precipitating zones or precipitating phases,

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Conclusion

The study of initial ageing indicates that :

1. There is initial softening followed by usual hardening.
2. Only within a definite range of temperature of ageing, initial softening can be observed. With increase in solute concentration the range narrows down.
3. The extent of softening increases with (a) severity of quench and (b) solute concentration.
4. For Al-Cu alloys, the extent of softening increases with increase in ageing temperature in contrast to Al-Mg alloys where the reverse is found to be true.
5. Time to reach minimum hardness increases with decrease in temperature and there is a reversal in this trend for many Al-Cu alloys.
6. X-ray line profile analysis reveals an initial sharpening followed by the broadening of the lines.

Acknowledgement

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