AGEING CHARACTERISTICS OF 7039 ALUMINIUM ALLOY

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submitted in partial fulfilment
of the requirements for the award of the Degree
of
MASTER OF ENGINEERING
in
METALLURGICAL ENGINEERING
(Physical Metallurgy)

By
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CERTIFICATE

Certified that the dissertation entitled " AGEING CHARACTERISTICS OF 7039 ALUMINIUM ALLOY", which is being submitted by Shri Sajal Kumar Gupta in partial fulfilment for the award of Degree of MASTER OF ENGINEERING in METALLURGICAL ENGINEERING (Physical Metallurgy) of University of Roorkee, Roorkee is a record of his own work carried out by him under my supervision and guidance from 6th January to 8th June 1976 at this University.

The matter embodied in this dissertation has not been submitted for any other degree.

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SAJAL KUMAR GUPTA

ABSTRACT

Ageing characteristics of 7039 Al-alloy were studied by hardness measurements and tensile testing at ageing temperature of 105°, 120°, 135° and 150°C. It was found that the peak - hardness values and corresponding tensile strengths decreases with increase of ageing temperatures. The best combination of strength and hardness was found with ageing at 120° for 23 hours.

Effect of multiple ageing was also studied by hardness measurements. It was found that with two step ageing, peak hardness values decrease slightly along with a decrease in total time to reach the same. 75% preageing at 135°C followed by final ageing at 120°C reduced the time to reach peak hardness to 10 hours from 23 hours, while the strength levels remained the same. Thus this treatment may be used commercially for industrial ageing treatment for the above alloy.

AGEING CHARACTERISTICS
OF
7039 ALUMINIUM ALLOY

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1. INTRODUCTION

The high strength aircraft and medium strength general purpose alloys of Al-Zn-Mg and Al-Zn-Mg(Cu) systems have 7XXX alloy designations. The highest room temperature strengths attained in wrought aluminium alloy products are developed by Al-Zn-Mg(Cu) alloys. In early 1960's several new weldable Al-Zn-Mg alloys were introduced. These new series (7XXX) alloy were different from older alloys 7075, 7079, 7178, in that the heat-affected zones (HAZ) were not embrittled by fusion welding. 7039 alloy is one of such new alloys.

Alloy 7039 is a relatively new high strength Al-Zn-Mg material. This alloy was developed to meet the need for weldable, heat-treated Al-base alloy. The alloy contains reduced amounts of Zinc and Magnesium, with small additions of Manganese, Chromium etc. Copper is generally eliminated or limited to very low amounts. This control of composition is reflected in complete solid solution of the soluble elements at much lower heat-treating temperatures in constract to the high-strength aircraft alloys. The optimum strength levels are secured even after relatively slow cooling rates. The low quench - sensitivity favours this alloy for massive forging or it permits slower quenching of thinner parts, thus reducing distortion and residual stresses.

The alloy can be strengthened by solution treatment and artificial ageing. Moreover this alloy exhibits appreciable precipitation hardening effect at room-temperature during natural ageing.

The alloy has good low-temperature toughness and good weldability which are required for pressure vessel construction. The material possesses sufficient ductility to form cylindrical segments. The alloy can also be hot formed and subsequently heat-treated to T61 or T64 tempers [illegible], as required for press forming parts such as elliptical heads and small diameter sphere segments. The alloy was originally developed for ballistic plates and is currently being used in armour vehicles. The toughness and ductility that give 7039 alloy its good ballistic impact-resistance, also make it a good alloy for pressure vessel constructions.

The alloy has been successfully used in both road and rail transport applications (1-3), for light weight military bridges(1,2), other structural purposes, and for armour-plates(1). It has excellent low-temperature properties (upto - 196°C) and appears to be ideally suited for cryogenic applications(1,4). In U.S.A. gas-metal arc welding was successfully used to produce first 7039 alloy pressure vessel(5) built under the requirement of ASME Boiler and pressure vessel code case 1363. Welding of the 9ft. diameter liquid oxygen sphere was also carried out without cracking difficulties.

The aim of the present work is to investigate the effect of ageing temperature and multiple ageing on time to reach peak-hardness and to determine the mechanical properties at the respective peak-hardness values on 7039 alloy.

CHAPTER - II

LITERATURE REVIEW

2.1 DEVELOPMENT OF Al- BASE 7039 ALLOY

Increased use of aluminium has been coincident with the development of alloys that presented increasingly higher aswelded strengths. At the time of second World War, high strength alloys were limited to the nonfusion weldable alloys, which were joined mostly by riviting. Even after Second World War and the increasing use of gas-shielded arc welding processes, the application of as-welded aluminium were limited by comparatively low strength alloys containing 1.0% Mn, 1.0 to 2.0% Si + Mg or 2 to 3% Mg. The strength of these alloys in the welded form ranged around 210 MN/m^2. In the mid 1950's, alloys 5086, 5083 and 5456 were introduced. These alloys contain 4 or 5 % Mg and are readily fusion welded by gas-shielded arc processes, giving aswelded joints strengths of 270 to 300 MN/m^2.

The expanded use of welded aluminium made possible by the later 5XXX series alloys, seem to call for a newer alloys with even higher aswelded strengths. The early 1960's saw the introduction of several weldable Al-Zn-Mg alloy(6). These new series (7XXX) alloy were different from older alloys 7075, 7079, 7178, in that the heat affected zones (HAZ) were not embrittled by fusion welding. The new alloy 7039 specially developed to meet the need for an alloy with as-welded properties superior to those 5XXX series Al-Mg alloys, as well as other 7XXX series alloys in present use. The typical mechanical properties at room-temperature and sub-zero temperatures are given in the Table 1(5) and Table 2(5) below for comparison. When welded with 5039 alloy

TABLE 1

Room temperature Mechanical Properties of ~~Alloy~~ 7039 alloy

Temper	Yield-stress (MN/m^2)	Ultimate Tensile Strength (MN/m^2)	Elongation(%)	DPN
T61	340	410	14	123
T64	390	460	13	133
0	106	230	22	61

TABLE 2

Mechanical Properties of 7039 alloy at low temperatures

Thickness (mm)	Temper	Test.Temp. (°C)	Yield-stress (MN/m^2)	Ultimate Tensile Strength (MN/m^2)	% Elongation
18.75	T61	24	370	440	13
		-196	440	580	14.5
34.50	T64	24	410	460	14
		-196	500	600	17
		-252	460	690	16

filler it gives crack free weldments with a minimum tensile strength of 340 MN/m^2 when naturally aged for atleast 15 days(5). This gives a desirable combination of tensile strength, yield strength and elongation values compared to typical weldment properties of other Al- alloys. The British alloy Hiduminium 48(7) is similar to American alloy 7039. But the former contain Mn, Cr and Zr whereas the latter Mn and Cr only . The other difference is that the Hinduminium 48 is welded with Al-5% Mg, while 7039 is by alloy 5039 filler to obtain a best combination of mechanical properties of the weldments.

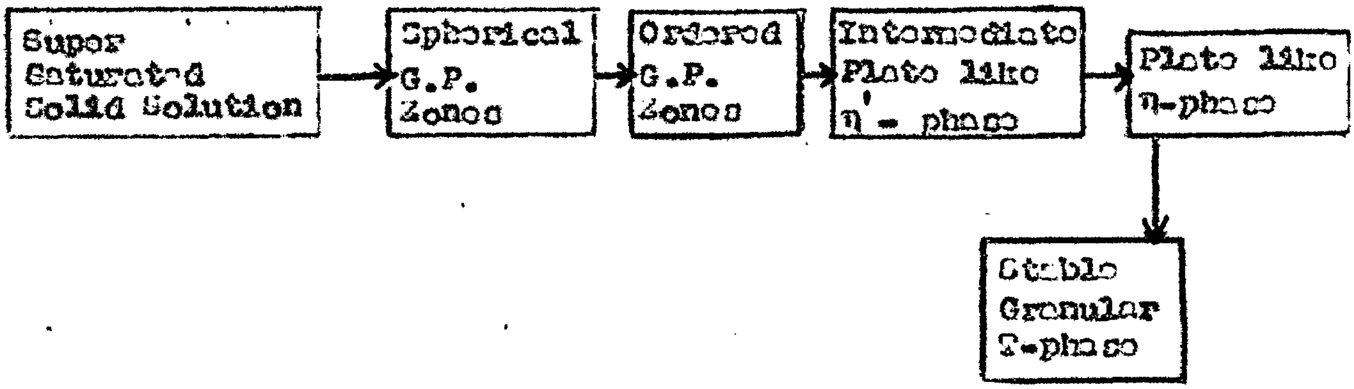
2.2 AGEING CHARACTERISTICS OF Al-Zn-Mg ALLOY

Aluminium has large high-temperature solubility for Zn which decreases to about 2 wt. % at room-temperature. This suggests that the binary Al-Zn alloys may have marked responce to age-hardening treatment, however the extent of hardening is small. The mechanism is thought to be that of decomposition of super-saturated solid solution in two stages. The first stage is the formation of spherical G.P. Zones. On further ageing small precipitate plates parallel to (111) planes can be detected with associated strain fields. Then the incoherent equilibrium precipitate forms by both continuous and discontinuous precipitation.

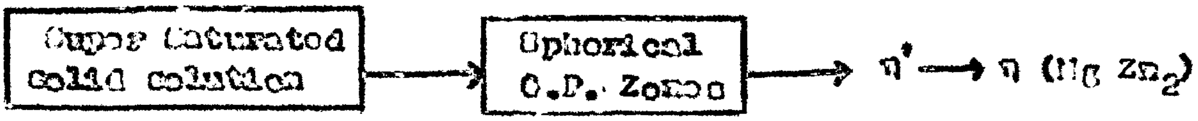
The ageing behaviour of ternary Al-Zn-Mg alloy is quite complex. Several workers have studied the ageing characteristics of this alloy systems. A brief review of which is given below.

Polmear(10) studied the ageing characteristics of a wide range of ternary Al-Zn-Mg alloys by determining the hardness/ageing-time curve at regular temperature intervals between -20°C to 240°C. It was found that small addition of Mg have a pronounced effect on ageing-response of Al-Zn alloys.

The precipitation process in Al-Zn - Mg alloys has been studied by X-ray methods(9), Electronmicroscopy(13), Specific-heat measurements(13) and Hardness-measurements(10,21,24). The general ageing sequence as observed through X-ray studies(8,9) are given below:


Super Saturated Solid Solution
Spherical G.P. Zones
Ordered G.P. Zones
Intermediate Plate like η' - phase
Plate like η-phase
Stable Granular T-phase

Thomas and Nutting(13) studied precipitation in the ternary Al-3.4% Zn - 2.5% Mg and the other more complex commercial materials based on this alloy. They found an ageing sequence:


Super Saturated solid solution
Spherical G.P. Zones
η' → η (Mg Zn2)

They concluded that η is formed at lower ageing temperature (upto 200°C), while T-phase is formed only at higher ageing temperatures (300°C and above).

In the ternary alloy the diameter of the G.P. zones is ~ 50 Å after short ageing time at 160°C and the density is only $\sim 10^{15}$ zones / cm^3. This suggests that most of the solute atoms remain insolution at this stage or are present as small clusters. The addition of Cu to the alloy decreases slightly the density and increases the size of GP.zones. The number of G.P. zones gradually increases during ageing even during the precipitation of η'-phase and the structure at the peak-hardness is a mixture of the two although η'-phase is predominant. Subsequently the G.P. zones dissolve as the precipitate coarsens. The η'-phase precipitate forms on $\{111\}$ planes and the electron diffraction pattern show that they have h.c.p. structure. The lattice parameters of η' are $a = 4.94$ Å and $c = 8.68$ Å (9) and the precipitates form with the orientation relationship :

$$(0001)_{\eta'} \parallel (111)_{Al}, \qquad [11\bar{2}0]_{\eta'} \parallel [1\bar{1}0]_{Al}$$

Mondolfo et al.(9) detected streaks in $\langle 111 \rangle$ directions in their X-ray diffraction patterns which they tentatively interpreted as an indication of G.P. zones on $\{111\}$ planes.

The η'-phase precipitates grow rapidly in the ternary alloy. The microstructure at peak-hardness shows the plates with ~ 50 Å thick and ~ 150 Å in diameter, having a density of precipitation around 2×10^{16} / cm^3. When the ternary alloy is overaged precipitates becomes coarser and their structure reverts to equilibrium low-temperature phase η ($Mg\ Zn_2$).

Additions of Cu, Mn, Mg, and Cr etc. has little effect on the morphology of η' precipitation but they influence the kinetics, e.g. Cu delays the rate of precipitation of η' but Mn and Mg have the opposite effect so that the hardness curve of complex alloy is similar to that of basic ternary alloy. Thomas and Nutting concluded that Cu, Mn and Mg must dissolve in η' without changing its structure. Another effect of increasing alloying additions is to increase the amount of precipitation on dislocations. There are probably two reasons for this:-

(1) The complex alloys contain large particles of intermetallic phases which are insoluble at the solution treatment temperature. These give rise to dislocations on quenching due to difference in thermal expansion.

(2) Secondly Cahn(15) has shown that the ratio of hetrogeneous to homogeneous precipitation will increase with increasing supersaturation, i.e. increasing alloying additions. However addition of

Cr to the complex alloy seems to decrease the amount of hetrogeneous precipitation and promote a uniform structure within the grains. It also inhibits recrystallisation at the solution treatment temperature, probably by the action of insoluble particles at the grain boundaries.

The lack of ductility of this alloy is normally associated with soft regions near the grain boundary.

Precipitation in alloys based on Al-Zn-Mg is complex and the results of Thomas and Nutting may only be true for alloys of certain Zn: Mg ratios. The effect of trace additions are qualitatively understood but their mode of action is not established.

2.3 STRUCTURE OF PRECIPITATION

Mondolfo et al.(9) and Graf(16,18) have investigated the structure of the intermediate precipitate η' in Al-Zn-Mg alloys. They found that it is formed after ageing at low temperatures, i.e. below 200°C and has a hexagonal structure with $a = 4.96$ Å, $c = 8.68$ Å compared with the lattice parameters of the equilibrium η ($MgZn_2$)-phase which are $a = 5.15$ Å, $C = 8.48$ Å. The change in lattice parameter of η' enables the phase to remain partially coherent with the matrix at small sizes. T-phase precipitates only at high-temperature i.e. 300°C. This phase has a b.c.c. structure with $a = 14.16$ Å and 161 atoms per unit cell.

2.4 EFFECT OF TRACE ELEMENTS

Many binary or ternary alloys contain either by accident or design a small quantity, e.g. 0.5 to 1.0 per cent of another element. This element may be precipitated as coarse particles of an intermetallic compound insoluble at the homogenisation temperature and therefore having little effect on the precipitation properties of the alloy. Alternately, the element may change the precipitation sequence by altering the solubility of the principal alloying elements or by promoting the formation of some precipitate particular to the ternary or quarternary alloy.

Hardy and co-workers (19,20) have extensively studied the effect of trace additions to Al-alloys. Hardy concluded that the so-called normal precipitation sequence in an alloy may be vitally dependent on the presence of some trace elements which is a standard impurity in the alloy.

Trace elements can have two distinct effects on precipitation :-

(a) Firstly their presence can alter the kinetics of zone formation so that the apparent rate of solute diffusion is greatly reduced.

(b) Secondly they can assist the nucleation of an intermediate precipitate.

Hardy also concluded that it is possible to predict whether or not an alloy will respond to trace additions. A detailed discussion of the effect of individual elements on the ageing sequence and related properties are given below.

(i) Effect of Cr and Mn

The presence of chromium or manganese in these alloys significantly changes the effect of quenching rate on their properties after artificial ageing. For many years, it was thought that this is resulted from accelerated precipitation of Zinc, Magnesium and Copper stimulated through nucleation provided by fine, previously precipitated particles of Chromium - containing or Manganese containing particles. These serve as nuclei for either zone formation or precipitation. Both zones and subsequently formed precipitates appear independent of these particles and with a random distribution in the matrix. Zone growth is accelerated by chromium; this effect is currently believed to be associated with the chromium atoms that remain in solid solution and presumably increase retention of vacancies during quenching. Polmear and Co worker(21) have concluded that Cr enhances the preferential precipitation within the grains rather than in the grain boundaries. This gives rise to an improved stress-corrosion properties of Cr bearing materials, whereas Mn suppresses the precipitation which would have been expected in the Mn free alloys. Other beneficial effect of Cr addition is that it modifies the recrystallisation behaviour(24) of the alloy.

(ii) Effect of Silver

Polmear(11,12) has shown that trace additions of Ag to an Al-Zn-Mg alloy result in an increase in peak-

peak -hardness of the alloy particularly at high ageing temperatures. The η' precipitate may be nucleated on some type of vacancy clusters whose distribution, particularly near grain boundaries, is affected by the presence of Ag atoms(22). Electron-micrographs show that the finer dispersion of precipitates occur with the addition of Ag. This dispersion is stable to a relatively high temperature, with the result that overageing is retarded(11). The precipitate free zone at the grain boundary also disappears with consequent change in the morphology of the precipitate in the grain boundary region, and a possible increased resistance to stress-corrosion-cracking.

A survey(23) of the influence of Ag on 23-ternary Al-Zn-Mg alloys has now proved that Ag may enhance the age-hardening of all these alloys in which it has adequate solid solubility. The magnitude of these response is a function of the saturation of the alloy and solubility of Ag. Experimental results suggest that the ability of Ag to stimulate nucleation of phase η' in many Al-Zn-Mg alloys originates in an interaction between silver atoms, magnesium atoms and vacancies. The presence of Ag promote high hardening in alloys with reduced Zn and Mg contents. These may have several practical implications, notably that it suggests new approaches to the problem of stress-corrosion cracking and low ductility in the high-strength alloys, and a method of improving strength of the range of weldable alloys.

(iii) Effect of Copper

Copper addition has greatest effect in the first stage of ageing in Al-Zn-Mg alloy(21). The important effect is the rapid hardening as soon ~~such~~ as the alloy are aged in the temperature range of 100° - 235°C. This effect increases with increasing Cu addition upto its solubility limit. This is considered to be associated with the formation of S-phase [Al_2CuMg](12). The early hardening is most marked in Al- 4% Zn - 3% Mg - 1.0% Cu which lies close to (α + S) - phase field. Addition of Cu to Al- 8% Zn-3% Mg does not change the form of ageing curve above 30°C. Early hardening described above is not observed since these alloys would probably lie in (α + T) - phase field at low-temperatures.

Cu does not affect the nucleation of intermediate precipitate. Once the early rapid hardening is over the quaternary alloy hardens in the same way as the ternary alloy. The addition of Cu has little effect on time to reach peak-hardness or the temperature at which the change from high to low level peak-hardness values occur.

Smith and Grant(25) concluded that the addition of Cu to Al-Zn-Mg alloy increases the rate of homogeneous precipitation at lower temperature by increasing the number of G.P. Zones. Thus Cu addition accelerates the formation and growth of G.P. zones at lower temperature.

Addition of Ag to high strength Al-Zn-Mg-Cu alloy promotes uniform precipitation during ageing and grain boundary precipitate free zone is greatly reduced. Polymear also suggested the Ag containing Al-Zn-Mg(Cu) alloy may be aged at temperatures above the normally used 135°C without sacrificing its tensile properties. Ellington et al.(26) showed that stress-corrosion resistance of Al-Zn-Mg(Cu) alloys can be improved by reducing Zn to Mg ratio and consequent reduction in quench sensitivity. This can improve the stress-corrosion resistance at the expense of its tensile strength . But with the addition of Ag and by ageing at appropriate temperatures stress-corrosion resistance gets improved without loss of strength levels.

(iv) Effect of Other Elements

(a) Gold : Gold has no observable effect on account of its negligible solid solubility in this system.

(b) Titanium : A minute addition of Ti was found(25) to retard the rate of precipitation and prevent solute atom aggregation at boundaries on air-quenching. Ti slows down diffusion of Zn and Mg atoms and has no effect on ageing characteristics of Al-Zn-Mg alloy because of its low solubility(12).

(c) Iron and Silicon : The effect of Fe and Si on the precipitate structure was studied by Thakory and Thomas(23). They found that both Fe and Si raises G.P. Zone solvus temperature. But presence of Fe and Si reduces the ductility of the alloy(29).

Fukui(30) studied the influence of Fe and Cr on toughness and fracture characteristics of Al-Zn-Mg alloys. It was found that the notch-toughness and tear-resistance of naturally aged and artificially aged Al-Zn-Mg alloy were markedly reduced with increasing amounts of Fe and Cr. However the reduction of toughness due to Cr was most marked.

2.5 MULTIPLE AGEING

Multiple ageing or double ageing is a process where the specimen is firstly preaged at some temperature followed by final ageing at some other temperature.

Cosmaro and Ciarda (31) investigated the influence of preageing at 0°C on ageing at 40°C of Al-10% Zn alloy by X-ray and resistivity measurements. They found that preageing effects size of G.P. zones. The size of G.P. zones on final ageing depends upon the preageing time. Size of G.P. zones increases with increasing preageing time.

In Al-Zn-Mg alloys preageing at room-temperature after solution heat-treatment leads to the formation of finer precipitate structure during artificial ageing([illegible],32).

Lorimer and Nicholson(32) studied the influence of quenching to below G.P. zone solvus temperature and holding at that temperature on final ageing of Al-Zn-Mg alloy.

They concluded that long preageing time gives a very fine precipitates and narrow precipitate free zone (PFZ) during final ageing. The rate of refinement and narrowing of PFZ is much less at lower ageing temperatures. However, no effect has been observed by preageing at or below -50°C.

Asano and Hirano(33) observed in the alloy of Al-5% Zn-1% Mg a considerable increase in quantity of G.P. zones during final ageing of pre-aged alloy but no influence of preageing on intermediate precipitates was observed. They concluded that the refinement of G.P. zones by preageing is responsible for improved properties of Al-Zn-Mg alloy.

Holl(24) studied the influence of pre-ageing at room-temperature of Al-5.3% Zn-1.8% Mg -0.09% Cr on artificial ageing by hardness measurements. In this alloy it is observed that preageing helps in the formation of η' precipitate even in the presence of stable grain-boundaries. The amount of η' precipitate formed during artificial ageing depends upon the pre-ageing time.

The effect of multistep-ageing on a Al-5% Zn- 2 to 4% Mg alloy have shown that greatest strength is obtained when the temperature of ageing steps are kept below 120°C(34) and the microstructure shows a minimum precipitate free zone near the grain boundary.

Sastry(35) studied the effect of multiple ageing on Al- base 7075 alloy (Al-Zn-Mg-Cu) and showed that the two stage-ageing reduced the overall time for full hardening with a little drop in peak-hardness values.

Stress-corrosion resistance of Al-7079 alloy can be improved by giving suitable multiple-ageing treatments (36). The resulting sacrifice in strength can be kept at low values either by raising final ageing temperature or accelerating quenching rate. Variations in quench rate and primary ageing temperature have effect on width of PFZ and precipitate distribution.

2.6 THERMOMECHANICAL TREATMENT

Thermomechanical treatment is defined(37) as " The use of deformation prior to or during an allotropic change so as to obtain an improvement in mechanical properties". Recently this treatment has been successfully applied to nonferrous alloys in which suitable transformation is observed during deformation.

The TMT has been clearly more popular with steels and comparatively little attention has been paid to the application of this important technique to modify the mode of decomposition of Al-base alloys. Some interesting developments of these type has been reported, which appear to improve the room temperature fatigue resistance of

7075 Al-alloys(38,39). The existing literature on the effect of dislocation on tensile properties is controversial.(40) It is not very clear whether TMT can be used as a basis for reducing heat treatment time in commercial practice and or obtaining improved properties.

Harendra-Nath et al.(40) studied the behaviour of an Al-4.5 % Zn - 2.0% Mg alloy under the influence of plastic deformation. They have made an effort to establish the conditions in which the dislocations introduced by plastic deformation interact most favourably with the age hardening processes to produce alloys that have improved properties compared to conventionally aged alloys.

They have used two TMT cycles as noted below:

TMT 1 : Solution treatment for 30 minutes at 450°C → plastic deformation by 5% at room-temperature and finally ageing above the G.P. zones solus-temperature T_{GP}.

TMT 2 : Solution treatment → preaged below T_{GP} → plastic deformation by 5% and final ageing above T_{GP}.

They concluded that the dislocations act as potential sites for precipitate of second phase from the parent-matrix. Thus increase in dislocation density produced by plastic deformation should increase the number of nucleation sites and thus accelerate the process of precipitation. There is little evidence of the dislocations to influence the formation of zones. This is due to the fact that the dislocation produced by plastic deformation prior to ageing acts as sinks for quenched-in vacancies. Thus removal of vacancies by plastic deformation delays the zone formation$^{(45)}$. In this investigation with the two treatments as above, there is no change in time to reach peak-hardness but there is very significant increase in peak-hardness values as compared to that for conventionally aged materials. It is observed that TMT also increases the value of UTS by 100 MN/m^2 with little loss in ductility.

Earlier Di Russo et al$^{(41)}$ determined the metallurgical properties of Al-Zn-Mg and Al-Zn-Mg(-Cu) alloys products processed by newly developed "Final Thermomechanical treatments of T-AHA type," which is illustrated in the block diagram below.

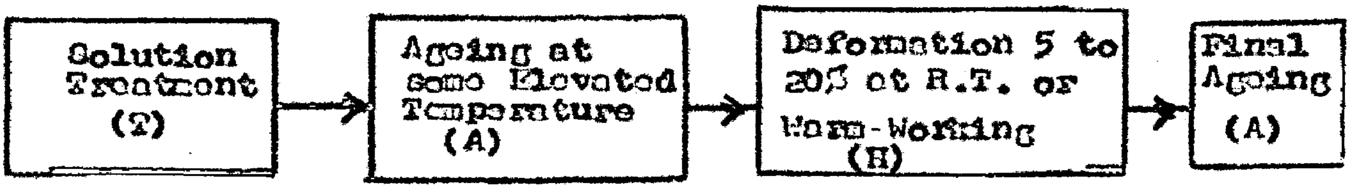

Solution Treatment (T)
Ageing at some Elevated Temperature (A)
Deformation 5 to 20% at R.T. or Warm-Working (H)
Final Ageing (A)

Fig. 1 "FTMT of T-AHA TYPE FOR Al-Zn-Mg Alloys.

The combined effects of plastic deformation and precipitation processes give new strength-structure relationship, even though Al-alloys do not exhibit polymorphic transformation as in the case of steel.

DI Russo(42) et al. obtained various combinations of strength and ductility by controlling the solidification conditions, chemical compositions and homogenisation solution treatments and ageing cycles. Flemings et al(43) obtained similar results with the alloys which are solidified under controlled conditions, completely homogenised and then cold worked after normal solution treatment (T) and ageing treatments (A). The different combinations of ageing and work hardening, was tested by Mercier and his co-worker(44) using process of plastic deformation after T6 - followed by partial recovery.

Paolov studied the effect of solution treatment (T) warm-working (H_c) followed by artificial ageing (A). It was observed that strength increases with a loss in ductility . The T- AH cycles have no significant relations, while in T-HA cycles reduces response to artificial ageing(45,46). But properties of materials were not substantially different from TA-state(21,47-48).

The initial ageing at lower temperature before plastic deformation and final ageing (i.e. T-AHA cycles) reduces the inhibiting effects of dislocations on ageing reactions. This treatment is markedly increase in UTS and Y.S. values compared to conventional tempers, while maintaining adequate ductility(41).

In their experiments with alloys 7039 and 7075 DI Russo et al.(41) found that (i) if deformation is applied at room-temperature before artificial ageing (T-HA cycles). The peak strengths are lower, other conditions being equal, than those obtained with T-AHA cycles. Thus the intermediate deformation increases the ageing rate during the final artificial ageing. They also found that this deformation should not be higher than 30% for sheets, 20% for plates and 10% for extrusions. Since higher degrees of deformations cause only a slight increase in strength accompanied by a large decrease in elongation value. They also studied the effect of warm-working in between the two ageing steps and found that in 70-39 alloy with deformation (H) in the temperature range of 130° to 170°C, the strengthening achieved by last ageing step is higher in the case of warm-working than for the cycles with deformation (H) at room-temperature. With alloy 7075 it was found that the deformation in the temperature range of 170° to 220°C, increases the plasticity with a better strength-properties than that obtained with room-temperature deformation. Thus it is found that a good strength property is obtained without any appreciable loss in ductility by "FTMT-T-AHA Type " treatment. It has been proposed that the increase in strength is due to superposition of strengthening effects from dislocation and precipitation. There were about 20% increase in Y.S. and UTS with better ductility, notch and fracture-toughness values with this treatment than that of conventionally treated alloys. But there is no significant increase in stress-corrosion resistance though a slight decrease in fatigue-resistance was noted. In an independent work earlier Mc-Evily et al.(49) have concluded that thermomechanical processing on Al-Zn-Mg alloy has small beneficial effect on fatigue resistance, but a major beneficial effect on resistance to stress-corrosion cracking.

Thomson(50) studied the effect of thermomechanical treatment on newly developed alloys 7475 and 7175 (Al-Zn-Mg-Cu system). These alloy have better fracture-toughness than 7075 alloy at the same strength levels. By a suitable ageing treatment, a good resistance to stress-corrosion and exfoliation-corrosion can also be obtained with these materials. But there was no observable improvement in fatigue - resistance. It was found that the effect of under-ageing followed by cold-work prior to ageing increases the strength level, the hardness values being same. The toughness of the initial over-aged materials is always found to exceed the toughness of initially underaged material at an equivalent yield strength level.

2.7 WELDABILITY

In a recent review on weldable Al-Zn-Mg alloys Kent(51) showed that weldable alloys contain 3-5% Zn and 1.2 to 2.8% Mg i.e. total Zn+Mg is 5 to 7% with Mn, Cr, Zr and Ti as minor additions. These contain no copper as it has adverse effects towards weldability.

Weldable alloys possess the advantage that during welding, the HAZ (Heat Affected Zone) close to the weld undergoes solution treatment in usual ways, but reversion occurs in the remainder of the HAZ accompanied by insignificant overageing.

Maximum strength in this system is obtained when Zn:Mg is in between 2:1 to 4:1. Higher the Zn+Mg content greater is the strength but lower is the stress-corrosion resistance. The alloys with lower Mg content can be easily extruded under lower pressure and higher speeds(52,53). These are also low quench-sensitive. But these having greater hot cracking tendencies and must be welded by filler rods with higher Mg content(54).

Additions of Mn, Cr and Zr are made to these alloys to inhibit recrystallisation and to obtain good mechanical properties. Zr has additional benefits over Cr and Mn, since it is also capable of refining the grain size of welded pool and so reduces the hot-cracking tendencies(55,56,57). It is also claimed that Zr addition increases the flow of the molten weld metal (57).

Ti-in the order of 0.01 to 0.04 may be added to refine the cast structure. It does not reduce the hot - cracking tendency, but reduces the general corrosion

-resistance in the HAZ.

Cu is omitted as it increases the hot-cracking tendencies and increase HAZ embrittlement. Both these tendencies may be reduced if Zr is added . Although stress-corrosion resistance of high strength alloy is improved by Cu additions.

Typical weldable alloys are British Hiduminium 48(7) and American 7039 alloy(5). The difference is that the former contains Mn , Cr, and Zr as minor alloying additions where the latter Mn + Cr only. Moreover Hiduminium 48 is welded with Al-5% Mg, while 7039 with 5039 alloy filler rods. MIG - welding is used for section thickness of 6 mm and above and for thickness below 6 mm TIG or PIG welding technique is used. Welding structures in this higher strength alloys are generally left to age at ambient temperatures. But if higher strength are required these may be aged artificially at some suitable higher temperature. Alloy 7039 when welded with 5039 alloy filler, gives rise to a minimum weldment strength of about 340 MN/m^2 in naturally aged weld conditions.

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CHAPTER III

EXPERIMENTAL PROCEDURE

3.1 ALLOY PREPARATION

The component materials used for preparing the alloy were E.C. grade aluminium (99.7% pure), Zinc(99.9% pure), Magnesium (99.9% pure), Chromium (99.9% pure) and electrolytic manganese (99.9% pure).

20 kgs of aluminium were melted in a graphite crucible.Weighed quantity of other alloy additions were made to this as per calculations to prepare the specific alloy. First manganese and chromium were added. The melt was stirred with a graphite rod. Low-melting Zinc and Magnesium of weighed quantities were wrapped in Al-foil and dipped well inside the molten metal till complete dissolution. This was done to avoid oxidation and vapourisation losses. The alloy then chilled cast in a thin walled cylindrical steel mould of height 100 mm. and 350 mm. diameter.

A quantitative analysis of the casting was under taken to confirm the composition of the alloy which is given in Table 3(58).

TABLE 3

CHEMICAL COMPOSITION

Elements	Nominal Composition (wt. %)	Composition as analysed (wt. %)
Si	0.30 Max.	0.13
Fe	0.40 Max	0.38
Ti	0.10 Max.	-
Mn	0.10 to 0.40	0.27
Cr	0.15 to 0.25	0.26
Mg	2.30 to 3.30	2.71
Zn	3.5 to 4.5	4.04
Al	Balance	Balance

The cylindrical casting was then cut into pieces and final casting was made by melting the alloy in graphite-crucibles in muffle furnace at 740 ±10°C. Cryolite was used as flux to minimise oxidation and the melt was degassed with hexachloroethane just before the pouring. The alloy was finally casted in mild-steel moulds of 40 mm x 40 mm x 160 mm size.

3.2 SPECIMEN PREPARATION

Castings were homogenised for 16 hours at 450° ±5°C and hot rolled to 20 mm square at 460°± 5°C and machined to 18 mm square. From the rolled-stock hardness specimens of 18 mm x 18 mm x 25mm were cut and ground and polished. Bars of 18 mm x 18 mm x 250mm were also cut for tensile specimens. Hardness samples and the bars for tensile specimen were homogenised for 56 hours at 460°C plus 94 hours at 480°C. The microstructure of hardness specimens showed single-phase structure after the above homogenisation treatment and average grain size as measured was found to be equal to 0.1465 mm.

3.3 HEAT TREATMENT

Hardness specimens were solution treated 1 hour at 480° ± 5°C in a muffle furnace and then quenched in water at room temperature. Solutionised samples were immediately subjected to further ageing treatment. All elevated temperature ageing treatments were carried out in silicone oil bath furnace with a temperature accuracy of ± 2°C. The tensile specimens were made from homogenised bars as per IS specification: 1816-1961 as shown in Fig.2. Finished specimens were then given the requisite heat treatments in the oil bath before the final testing.

3.4 Hardness Testing

Hardness testing were made with Vickers Pyramidal Diamond Indentor with 5 kgs of load. The hardness values of the alloys as quenched were determined by averaging five readings on each of three specimens. Average value of as quench hardness was found to be equal to 50 VPN.

For ageing treatments, duplicate specimens were used for hardness measurements. The results reported are average of six indentations.

3.5 TENSILE TESTING

Tensile testing were carried out, in a Universal Tensile Testing Machine at a load range of 0-10 metric-tonnes. Two specimens were tested for each of the heat-treatment cycles and the reported results are average of them.

3.6 SCHEME OF EXPERIMENTS:

TABLE - 4

A. DETERMINATION OF AGEING CURVES BY HARDNESS MEASUREMENTS :-

Experiment	Preageing Temperature (°C)	Percentage preageing	Final Ageing Temperature (°C)
i. Effect of Temperature of Ageing	-	-	105
	-	-	120
	-	-	135
	-	-	150
ii. Effect of multiple-ageing	105	25	120
		50	120
		65*	120
	135	25	120
		50	120
		75	120

* For 75% preageing at 105°C takes 30 hours which is greater than the time for 100% ageing at 120°C namely 23 hours. So 65 % preageing has been given at 105°C.

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TABLE - 5

B. TENSILE TESTING **

Experiments	Preageing Temperature (°C)	Percentage preageing	Final ageing temperature (°C)
i. Effect of Temperature of Ageing	-	-	105
	-	-	120
	-	-	135
	-	-	150
ii. Effect of multiple ageing	105	50	120
	135	50	120
		75	120

** Tensile testing was carried out corresponding to peak-hardness values alone.

CHAPTER- IV

RESULTS AND DISCUSSION

4.1 RESULTS

Basic ageing curves were plotted by measuring hardness / ageing time data at ageing temperatures (T_A) of 105°, 120°, 135°, and 150°C . The curves are shown in Figs 3 and 4. Table 6 shows the effect of ageing temperature on peak-hardness and the time to reach the same. Table 7 shows the tensile-properties at peak-hardness at these ageing temperatures.

The effect of multiple-ageing has also been ~~studied by recording hardness/ageing has also been~~ studied by recording hardness/ ageing time data and is shown in Figs. 5 and 6 . In Tables 8 and 9 are shown the effect of preageing at 105°C and 135°C respectively on the time to reach peak-hardness at the final ageing temperature of 120°C. From Table 10 and 11 the effect of preageing at 105° and 135° C on tensile properties associated with peak-hardness values at final ageing temperature of 120°C can be noted.

4.2 DISCUSSION

Table 6 shows the peak-hardness values and the time to reach the same at different ageing temperatures as obtained from Figs. 3 and 4 . It is found that peak-hardness values decrease with increase in temperatures of ageing along with the time to attain the peak-hardness value. The decrease in peak-hardness with increase of ageing temperature is due to :

(i) Coarser nature of precipitate(s) associated with peak-hardness at higher temperatures of ageing.

(ii) Lower volume fraction of precipitate formed at higher ageing temperatures as given by the metastable phase boundaries.

Ageing at 105°C for 56 hours gives the highest peak-hardness value of 135 VPN. Ageing at 120°C reduces the peak value by only about 7% than that at 105°C. But the time to reach peak hardness reduces drastically from 56 hours to 23 hours i.e., with about 60% reduction of time.

Ageing at 135°C for 16 hours gives a peak-hardness value of 110 VPN. This is about 20% less than that of peak-values at 105°C. The reduction in time to reach the peak-values is about 70% with respect that at 105°C. Ageing at 150°C gives the lowest peak - value in the shortest possible time. The peak value of 80 VPN is attained by ageing for 12 hours with only about 60% increase in peak-hardness value with respect to its quenched hardness. Ageing at 120°C gives rise to a good peak hardness of 125 VPN within a reasonable time of 23 hours. So this ageing treatment appears to be the optimum ageing temperature for the above alloy.

The effect of ageing temperature on mechanical properties at fully hardened condition is given in Table 7. Values of U.T.S. decreases with increase in ageing temperature. Maximum UTS of 402 MN/m^2 is obtained with ageing at 105°C for 56 hours and a minimum of 274 MN/m^2 at 150°C for 12 hours. But the ductility as measured by percentage elongation values increases with increase off temperature from 105° to 135°C. However 0.2% proof-stress values are found to be almost same for all in the above cases.

Tables 8 and 9 show the effect of percentage pre-ageing at 105° and 135°C respectively on the time to reach peak-hardness at final ageing temperature of 120°C. In all the above treatments total time to reach peak-hardness is found to decrease with some decrease in peak-hardness value. From point of view of economy of ageing time following cycles are found to be most beneficial:

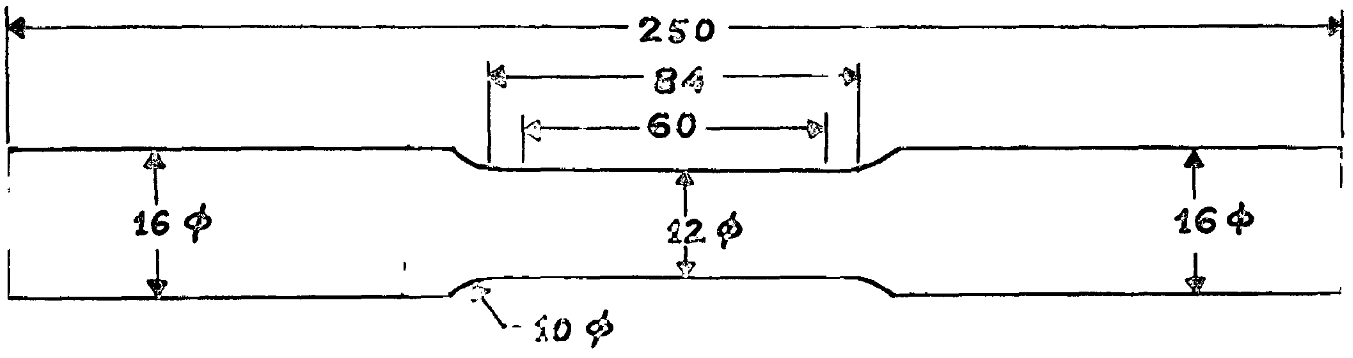
(i) 50% preageing at 105° followed by final ageing at 120°C, and

(ii) 50% or 75% preageing at 135°C followed by final ageing at 120°C.

In the two steps ageing of 50% preageing at 105°C plus final ageing at 120°C, the time to reach peak-hardness is reduced from 23 to 19 hours i.e. about 20% with a drop in hardness value of 5%. While 50% and 75% preageing at 135°C followed by final ageing at 120°C reduces the time to 13 hours(about 45% reduction) and 10 hours (i.e. about 60%

reduction) respectively with about 20% drop in peak-hardness values in both the treatments with respect to that at 120°C.

Tables 10 and 11 show the effect of multiple ageing on tensile properties. Table 10 shows the effect of 50% preageing at 105°C followed by final ageing at 120°C. It gives UTS of 280 MN/m^2 at peak-hardned condition (reduction of about 10% with respect to simple ageing at 120°C). Table 11 shows the effect of preageing at 135°C followed by final ageing at 120°C . Both 50% and 75% preageing at 135°C give rise to nearly same strength levels. With the latter treatment a strength level of 296 MN/m^2 is obtained at peak-hardness with 10% of elongation. This value of U.T.S. lies in between the values that obtained with 100% ageing at 120° and 135°C. Thus for about equal strength levels a combination of 75% preageing at 135°C followed by final ageing at 120°C is to be choosen to economise the time for ageing cycles in this alloy.


250
84
60
16 φ
12 φ
16 φ
10 φ

ALL DIMENSIONS IN MM.

FIG. 2 TENSILE SPECIMEN

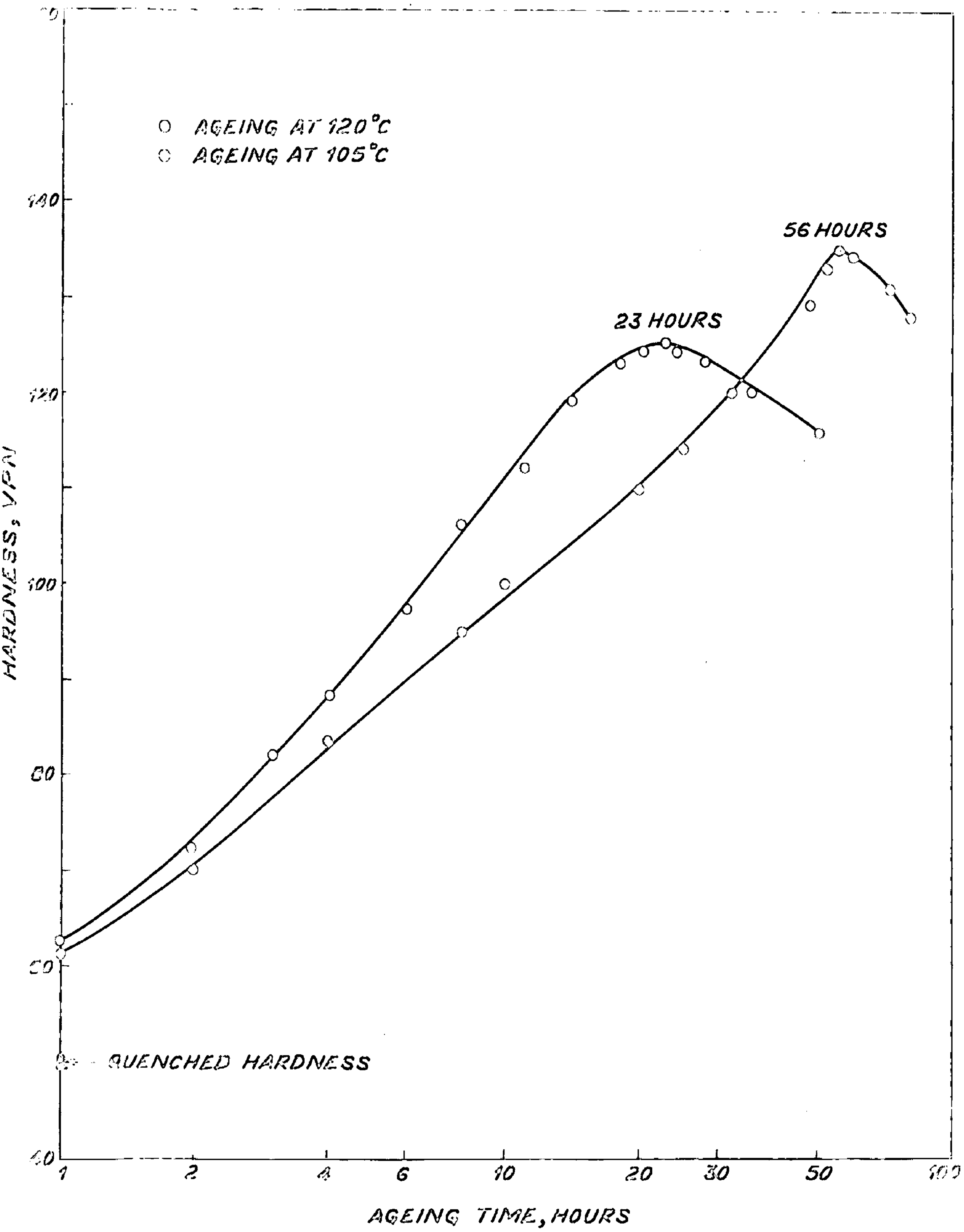


FIG.3 AGEING CURVES AT 105° AND 120°C FOR 7039 ALLOY.

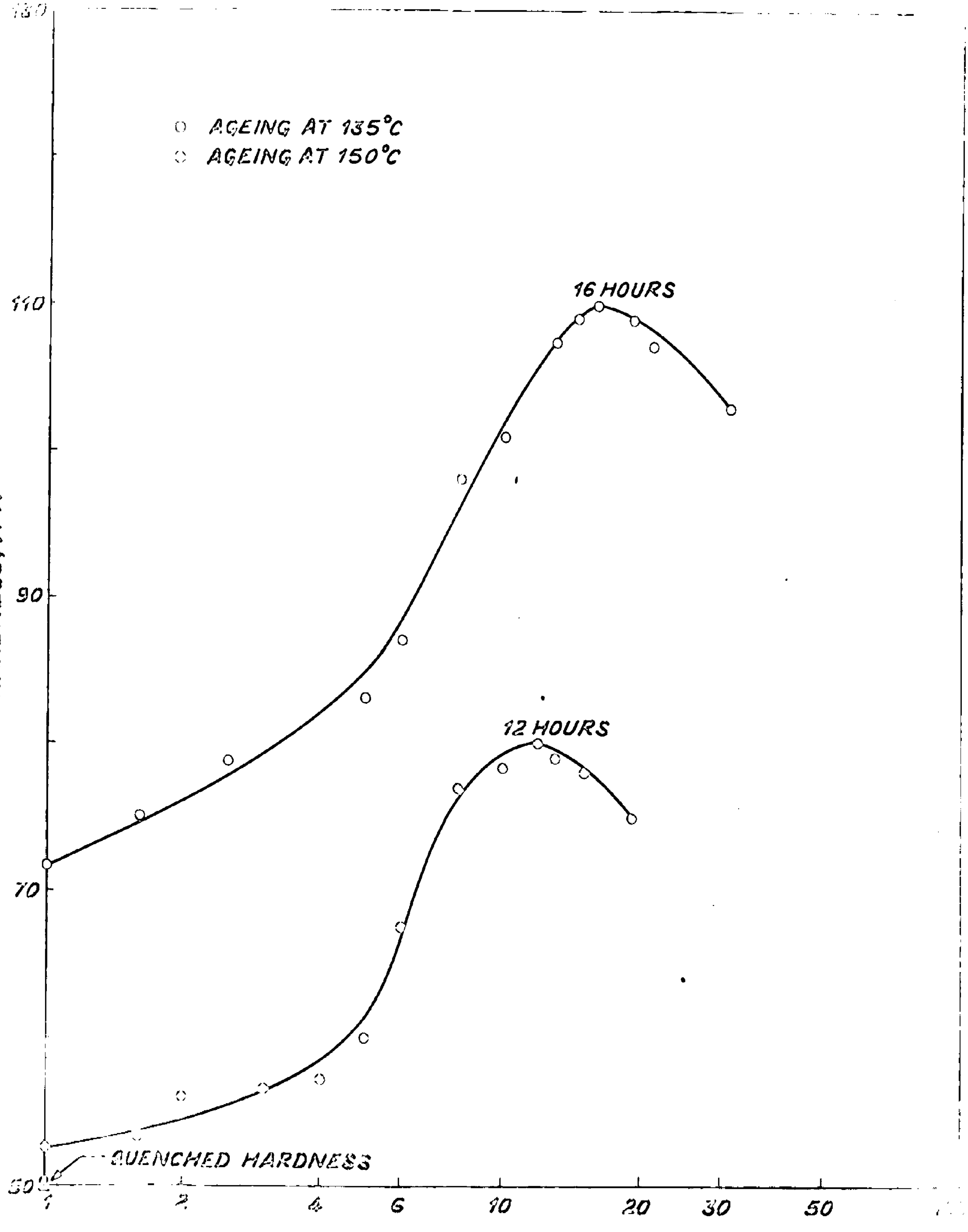


FIG.4 AGEING CURVES AT 135° AND 150°C FOR 7039 ALLOY.

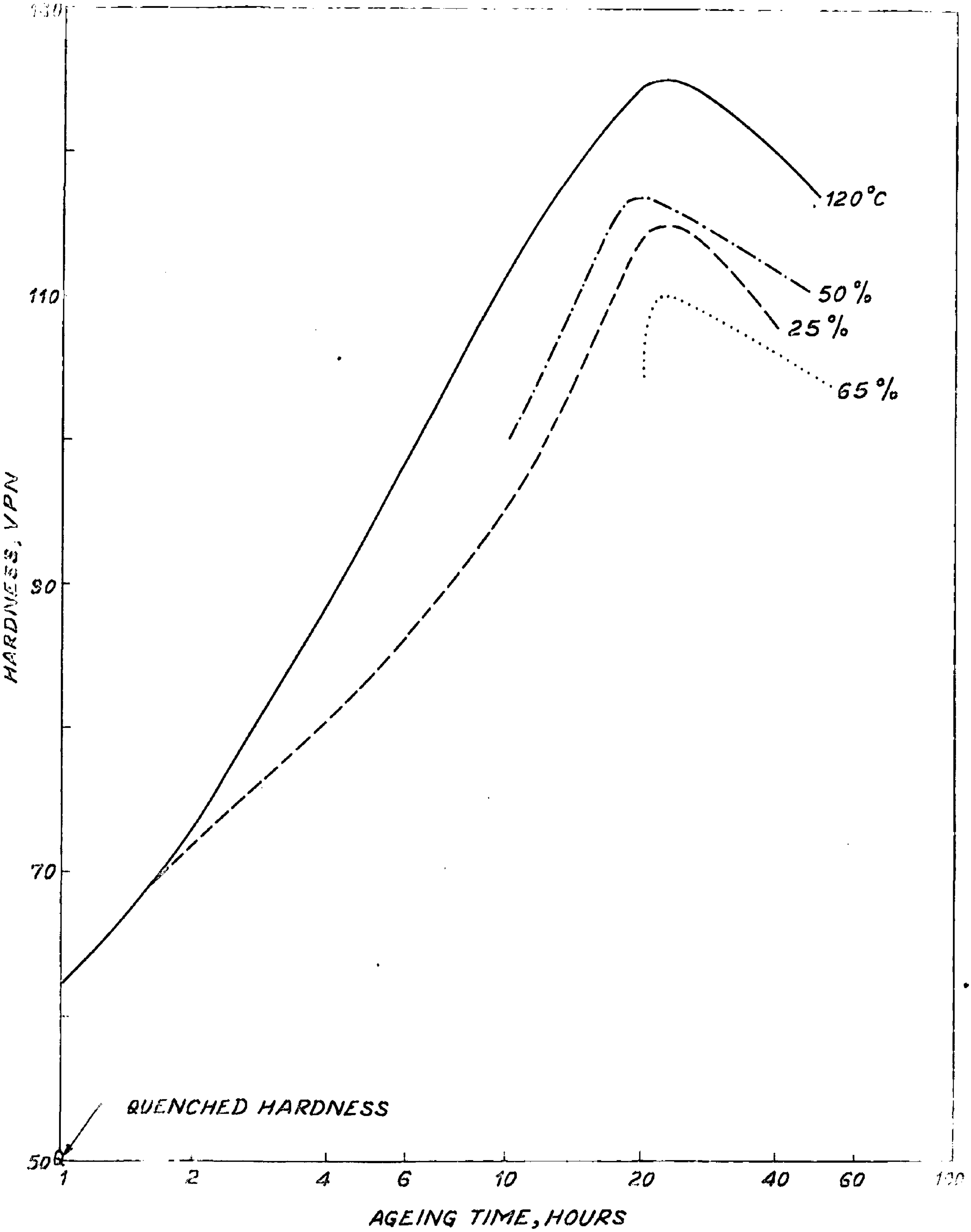


FIG. 5 EFFECT OF PERCENTAGE PRE-AGEING AT 105°C ON FINAL AGEING AT 120°C.

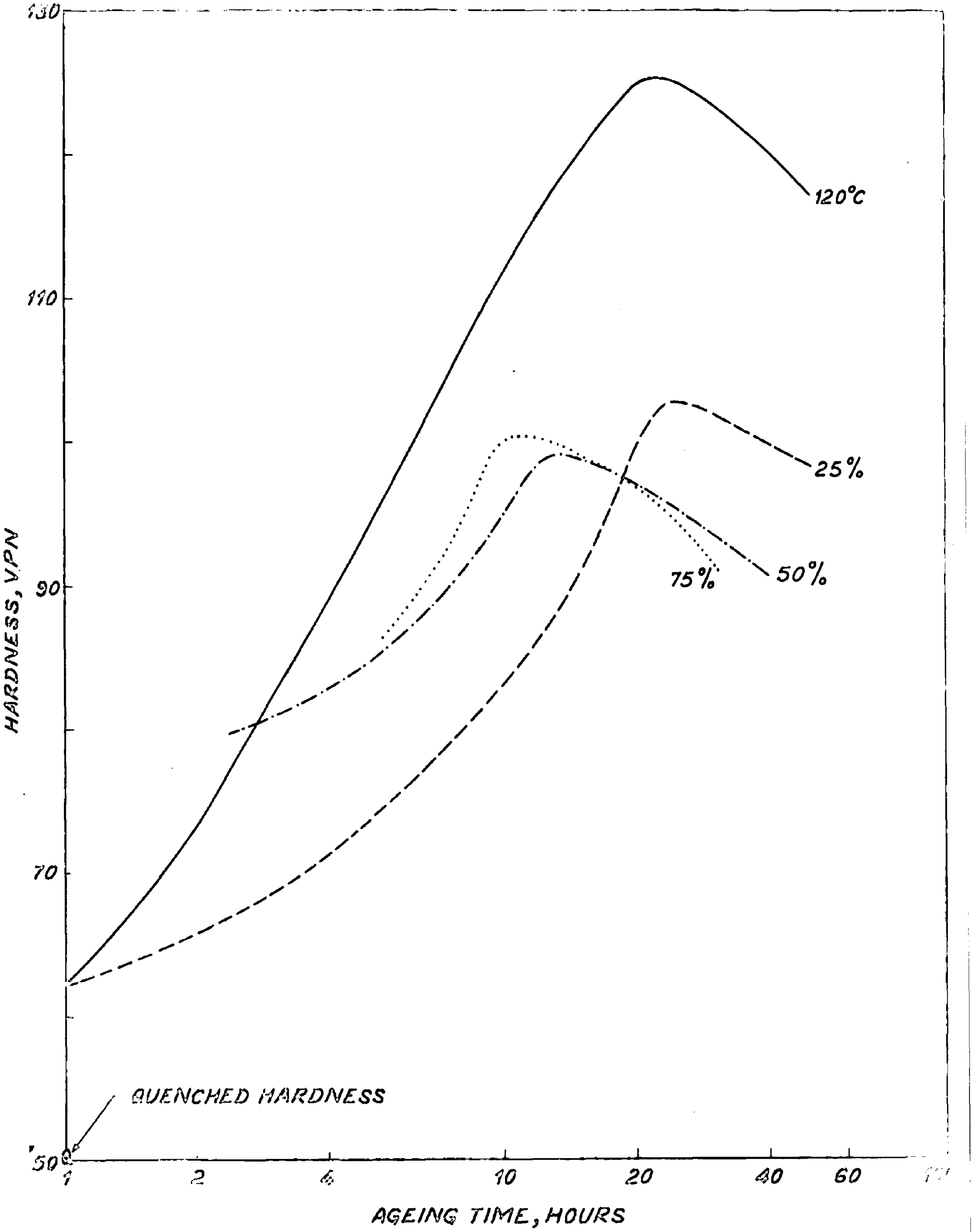


FIG. 6 EFFECT OF PERCENTAGE PRE-AGEING AT 135°C ON FINAL AGEING AT 120°C.

TABLE - 6

EFFECT OF AGEING TEMPERATURE ON THE TIME TO REACH PEAK-HARDNESS

Ageing Temperature (^{o}C)	Time to reach peak-hardness (Hours)	Peak-hardness value (VPN)	Percentage increase in VPN
105	56	135	170
120	23	125	150
135	16	110	120
150	12	80	60

TABLE - 7

EFFECT OF AGEING TEMPERATURE ON MECHANICAL PROPERTIES

Solution Treatment	Aged to Peak-hardness at Temperature (^{o}C)	0.2% proof-stress (MN/m^2)	Ultimate tensile strength (MN/m^2)	Percentage Elongation	Percentage in reduction in area
480°C for 1 hr.	105	160	402	12.5	22.04
"	120	154	320	12.5	21.30
"	135	-	287	15.9	41.08
"	150	150	274	10.00	11.52

TABLE 8

EFFECT OF PERCENTAGE PREAGEING AT 105°C ON THE FINAL AGEING AT 120°C

Preageing temperature (°C)	Percentage Preageing	Final ageing Temperature (°C)	Total time to reach peak-hardness (Hours)	Peak-hardness value (VPN)	Percentage increase in VPN
105	0	120	23	125	150
	25	120	22	115	130
	50	120	19	117	136
	65	120	21.5	110	120

TABLE 9

EFFECT OF PERCENTAGE PREAGEING AT 135°C ON FINAL AGEING AT 120°C

Preageing temperature (°C)	Percentage preageing	Final ageing Temperature (°C)	Total time to reach peak-hardness (HOURS)	Peak-hardness value (VPN)	Percentage increase in VPN
135	0	120	23	125	150
	25	120	23	105	106
	50	120	13	99	96
	75	120	10	100	100

TABLE 10

EFFECT OF PREAGEING AT 105°C ON TENSILE PROPERTIES

HEAT TREATMENTS				MECHANICAL PROPERTIES				
Multiple Ageing								
Preageing Temperature (°C)	Percentage preageing	Final ageing Temperature (°C)	Total time to reach peak-hardness (HOURS)	Peak-Hardness (VPN)	0.2% Proof-stress (MN/m²)	Ultimate tensile strength (MN/m²)	Elongation percentage	Reduction in area (%)
105	0	120	23	125	154	320	12.5	21.30
	50	120	19	117	147	280	10.0	11.55

TABLE -11

EFFECT OF PREAGEING AT 135°C ON TENSILE PROPERTIES

HEAT TREATMENT				MECHANICAL PROPERTIES				
Multiple Ageing								
Preageing Temperature (°C)	Percentage preageing	Final ageing Temperature (°C)	Total time to reach peak-hardness (Hours)	Peak-Hardness (VPN)	0.2% Proof-stress (MN/m²)	Ultimate tensile strength (MN/m²)	Elongation percentage	Reduction in area (%)
135	0	120	23	125	154	320	12.5	21.30
	50	120	13	99	142	270	11.0	15.00
	75	120	10	100	152	296	10.0	11.25

CONCLUSIONS

1. 7039 type Aluminium alloy can be made from indigenous available raw materials.
2. The optimum hardening was obtained with this alloy when aged at 120°C for 23 hours which gives a tensile strength of 320 MN/m² and hardness 125 VPN with percentage elongation of 12.5.
3. The strength levels and hardness values were found to increase with decrease in ageing temperature.
4. Maximum hardness (135 VPN) was obtained when aged at 105°C for 56 hours with a tensile strength of 402 MN/m².
5. Ductility was found to increase with increase in ageing temperature upto 135°C.
6. Two step ageing was found to reduce the overall time for reaching the peak-hardness with a drop in peak hardness value.
7. Best combination of two-step ageing was found to be that with 75% preageing at 135°C followed by final ageing at 120°C. This treatment reduced the total time to reach peak-hardness to 10 hours while maintaining the same strength levels as obtained with other combination of ageing cycles.

SUGGESTIONS FOR FUTURE WORK

The following investigations may be undertaken:

1. Effect of other combinations of two step ageing on mechanical properties.
2. Effect of ageing temperature and two step ageing on other properties such as fatigue strength, fracture-toughness and stress-corrosion resistance,
3. Influence of quenching rate on artificial ageing and corresponding mechanical properties.
4. Effect of direct quenching to final ageing temperature (i.e. quench-ageing) and corresponding mechanical properties at peak-hardness values.
5. Effect of intermediate warm-working in between two-step ageing on time of heat treatment cycles and corresponding mechanical properties.
6. Effect of minimum copper addition to this alloy which will give better stress-corrosion resistance without deteriorating its good weldability.
7. Transmission electron microscopic (TEM) studies may be carried out to investigate the precipitation sequence, precipitate distribution, width of precipitate free-zone near the grain boundary, and morphology of precipitate in the vicinity of grain boundaries produced by different ageing treatments and in thermo-mechanically treated samples.

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