Effect of Water Quenching Temperatures on the Hardness of Al-4.5%Cu

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Research Article

ABSTRACT

Precipitation kinetics of Al-4.5% Cu in water quench media at 0°C, 25°C, 40°C had been studied. The aluminum and copper ingots used as raw materials for furnace charge were melted in a tilting crucible furnace (electric) of 150 kg capacity. The molten alloy was thereafter, poured into metallic moulds directly from the crucible pot and allowed to solidify in the mould and cool at ambient temperature, after which the samples were removed from the mould. The cast samples were solutionized for one hour in a muffle furnace at about 545°C and plunged into the water at 25°C where it was held for 10 minutes. Precipitation heat treatment was carried out in the furnace at 130°C. The procedure was repeated but with water at 0°C, 40°C, and distilled water at room temperature at different aging times. As-quenched hardness and post aging hardness was determined using Rockwell hardness tester (Model HRS 150) in observance of ASTM D785. The results obtained showed that the peak hardness value of 23.8 was obtained at eighty minutes for the sample quenched in water at 0°C. The studied alloy may find application in development spare parts for automobile industries.

INTRODUCTION

Aluminum and Aluminum alloys have many outstanding attributes that lead to a wide range of applications, including good corrosion and oxidation resistance, high electrical and thermal conductivities, low density, high reflectivity, high ductility, reasonably high strength, and relatively low cost. Aluminum is a lightweight material with a density of 2.7 g/cm^3 (0.1 lb/in.3). Aluminum is the third most abundant element (after oxygen and silicon) and the most abundant metal in the earth's crust, making up about 8% by weight of the crust by mass [1].Pure aluminum and its alloys have the Face-Centered Cubic (FCC) structure, which is stable up to its melting point at 657°C (1215°F). Because the FCC structure contains multiple slip planes, this crystalline structure greatly contributes to the excellent formability of aluminum alloys [2]. Aluminum alloys display a good combination of strength and ductility.

The excellent mechanical properties of these materials and relatively low production cost make them very attractive for a variety of applications both from scientific and technological viewpoints. On a strength-to-weight basis, most of the aluminum alloys are superior to steel but wear resistance, creep, and fatigue properties are usually somewhat poorer [3].

Like aluminum, copper is 100% recyclable without any loss of quality, both from a raw state and from manufactured products. In volume, copper is the third most recycled metal after iron and aluminum.

It is well known that heat treatment is one of the important methods for improving the mechanical properties of aluminium alloys [4]. The heat treatment of age hard enable aluminium alloys involves solution zing the alloys, quenching, and then either aging at room temperature (natural aging) or at an elevated temperature (artificial aging) [5].

The precipitation hardening alloys can be formed in a relatively soft state and then heat treated to much higher strength levels after forming operations are complete. In addition, aluminium and its alloys are non-toxic and among the easiest to recycle of any of the structural materials. The final properties of an alloy are not due only to its chemical composition, but also to its metallurgical history [6]. It is possible to use different treatments to change the properties of an alloy. Thus finding the optimal treatment has a fundamental importance, in order to achieve the desired properties. Alloys based on the Al-Cu system have the advantage of superior creep strength at elevated temperatures [7].

Quenching is a crucial step to suppress precipitation to retain the super saturation of solid solution, control the distortion, and minimize the residual stress in aluminium alloys. Quenching media commonly used for aluminium alloys include brine solution, water, and polymer solutions [8]. Physical properties of quench bath directly affect the cooling rate of a quenched part. These properties include the type of quenchant, its temperature, concentration, and agitation level [8]. These parameters must be controlled to optimize the quenching process in terms of alloy microstructure, properties, and performance. Age-Hardening of aluminum alloys is basically the strengthening of a metal alloy by extremely small and uniformly distributed dispersed particles that precipitate from a supersaturated solid solution. The alloy becomes harder with time or as it ages it develops hardness and strength.

The hardness of the quenched alloy increases as a function of aging time. Aging of the quenched alloy at room temperature is known as natural aging while at elevated temperatures is known as artificial aging. Precipitation in solid solution occurs when the solubility of the solute decreases with decreasing temperature. The precipitate of the second phase should be coherent in nature. The objective of age hardening is to create in a heat treated alloy a dense of fine dispersions of precipitated particles in a matrix of deformable metal. The precipitate particles act as obstacles to dislocation motion and thereby strengthen the heat-treated alloy.

In other words, the general requirement for precipitation strengthening of the supersaturated solid solution involves the formation of finely dispersed particles [9]. The precipitate particle nucleates and grows; by the diffusion of solute atoms into it from the matrix phase. It is called precipitation because the small particles of the new phase are termed precipitates [10]. Artificial aging will be accomplished not only below the equilibrium solvus temperature but below a meta-stable miscibility gap called Guinier-Preston (GP) zone solvus line. Precipitation strengthening or Age hardening provides one of the most widely used mechanisms for the strengthening of metal alloys. The precipitation strengthening process involves three basic steps:

Solution Heat Treatment or Homogenization

During this step, an alloy of composition X1 is heated to a temperature T1, between solvus and solidus temperatures, in order to dissolve and disperse uniformly in the matrix the phases formed during the solidification and soaked there until all of the solute dissolves into the X phase and a uniform solid-solution structure is produced. The precipitates are dissolved and any segregation present in the original alloy is reduced.

Quenching

The solid solution is rapidly cooled forming a supersaturated solid solution. The rationale behind the quenching process is to preserve the high-temperature uniform solid solution structure of the alloy below the homogenization temperature **Figure 1**.
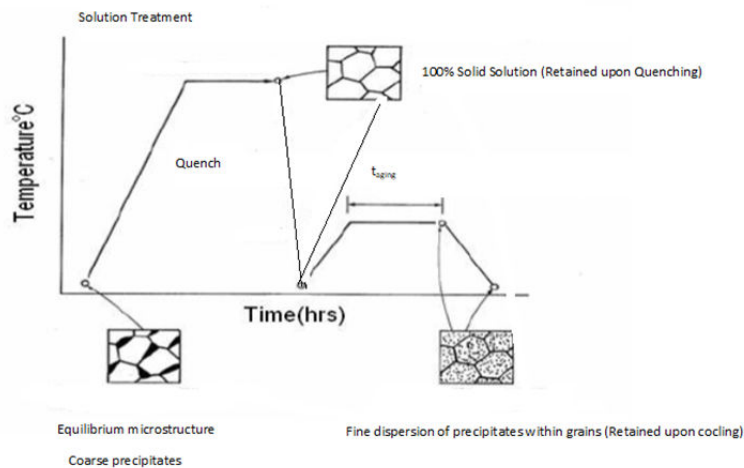

Figure 1. Schematic temperature versus time plot showing both solution and precipitation heat treatments for age hardening.

Ageing

This is the process of precipitating coherent or incoherent precipitates from a supersaturated solid solution. The supersaturated solid solution is heated below the solvus temperature to produce a finely dispersed precipitate **Figure 2.**
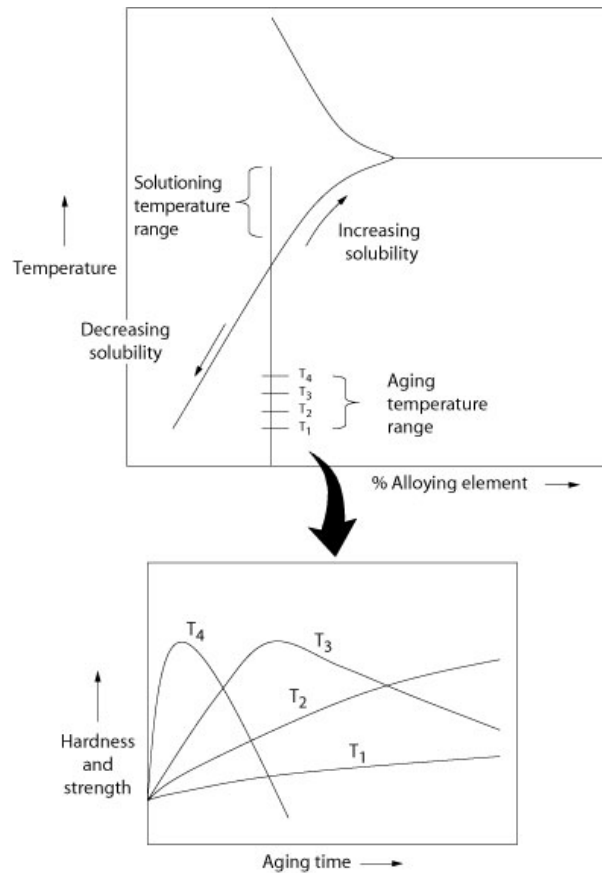

Fig. 3 Typical precipitation hardening heat treatment. Source: Ref 1

Figure 2. Typical Precipitation Hardening Heat Treatment source [2].

The strengthening mechanisms in wrought, heat-treated Aluminium alloys can be demonstrated using the Al-Cu alloy as shown in **Figure 1.**
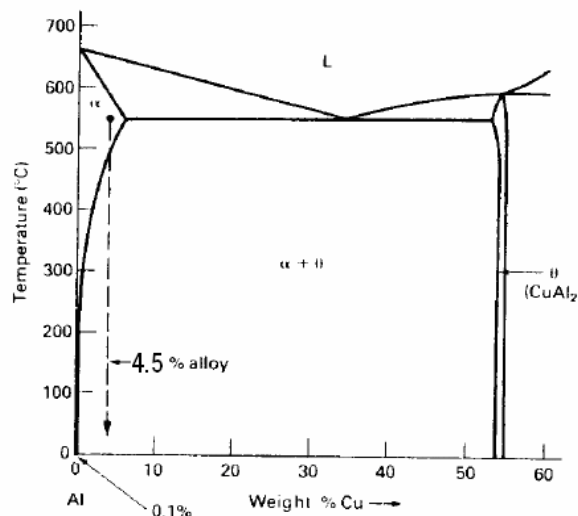

Figure 3. The binary phase diagram for Aluminum-Copper alloy [11].

Precipitation hardenable aluminum alloys are heat treated in a three-step process: solution heat treatment, quenching and age hardening. The solution treatment aims to dissolve the soluble phases. Quenching aims to preserve the solid solution formed by rapidly cooling to some lower temperature, usually room temperature. Age hardening aims to precipitate out the strengthening phases [3]. Overheating above or under heating below a specified range of temperatures in the solution treatment and age hardening steps as well as inadequate quenching may cause degradation of mechanical properties.

Hardening is achieved by the controlled rejection of copper from a supersaturated solid solution. The solubility of copper in α-Aluminum increases with increasing temperature up to the eutectic temperature of about 540°C. The equilibrium microstructure below the eutectic temperature is a two-phase mixture of α-Aluminium and the CuAl2 intermetallic phase. Rapid quenching from the solution temperature prevents the kinetically slow precipitation of θ, forming a highly supersaturated solid solution of copper **Figure 3**.

Al-4.5% Cu alloy has a wide range of applications in the aerospace and automotive industries [12]. This can be attributed to its high strength to weight ratio and also its characteristic increase in tensile strength at elevated temperatures.

In this work, an investigation has been done to study the effect of water quenchant at different quenching temperatures on the precipitation kinetics in Al-4.5% Cu.

MATERIALS AND METHODS

Material Sourcing

Pattern making materials i.e. wood, nails etc., Silica sand from Akwuke river Coal dust, Bentonite, Aluminum Ingot, Copper Ingot, Pyrometer, Ladle Crucible furnace, Heat treatment furnace, water, Distilled water, Emery Papers, Etchants, Nikon camera microscope, Hardness testing machine.

Melting Procedure

The required composition for each melt was prepared by weighing their percentage by weight. This was slightly exceeded in each case after charge calculation to make room for oxidation losses. The furnace was thoroughly inspected and ignited afterward. The materials were melted in a tilting crucible furnace (electric) of 150 kg capacity. The raw materials used for furnace charge were; aluminium ingot and copper ingot. The materials to be charged into the furnace were weighed correctly according to the charge make-up, as an error in weighing would render the product out of specification and it becomes a reject. The copper ingot was first charged into the furnace because of its higher melting point. Though the melting point of copper, 1083°C is higher than that of aluminium, 660°C, the melting point of aluminium was high enough to cause diffusion of copper atoms in the melt.

The charging of the ingots into the furnace was done gently and in a batch process, which helps to eliminate the possibility of damage to the lining as well as metal-bridge forming in the furnace. The molten alloy was thereafter, poured into the metallic moulds directly from the crucible pot. It solidified in the mould and was allowed some time to cool at ambient temperature, after which the casts/samples were removed from the mould. The cast round bars were milled on the lathe to obtain a diameter of 20 mm and length 140 mm. Each of the bars was cut into 12 pieces with a hacksaw, a piece measuring 20 mm Ч 20 mm.

They were machined to the desired shape and sizes for the various tests using standard specification.

Experimental Procedure

7 round bars of Al-4.5% Cu alloy measuring 20 mm in diameter and 140 mm long obtained from the cast, were cut into 12 pieces of 20 mm diameter Ч 20 mm disks gave a total of 84 disks. The first 12 disks were put in the muffle furnace at about 545°C and allowed to solutionize for one hour and then plunged into the water at 25°C where it was held for 10 minutes. One out of the 12 disks was brought out and it's as quenched hardness and microstructure taken. The remaining 11 disks were put in the furnace at 130°C for precipitation heat treatment. The choice of 130°C is to ensure that coherent precipitation is first formed because it is below the solvus lines for both GP1, and GP2 zones. After 15 minutes one out of the 11 specimens was removed and its hardness and microstructure taken, followed by bringing out three specimens at 15 minutes intervals. The remaining 7 specimens were brought out at 20 minute intervals bringing the total ageing time to 200 minutes. For each of these specimens, hardness reading was taken.

These experiments were repeated with another four sets of 12 disks but plunged in water at 0°C, 40°C, and distilled water at room temperature after solution zing, held for 10 minutes and the ageing steps above were repeated.

Hardness Test

The type of hardness carried out on the samples was the Rockwell hardness test as it gives a quick and direct reading of the hardness values. In the Rockwell hardness tester (Model HRS 150) the test piece was placed on the machine and the indenter (steel ball) was brought into contact with the prepared surface of the test piece under an initial load of 100N and an original load of 1371N given a total load of 1471N. The start button was now pressed and the reading automatically shows on the screen. For each test piece, three indentations were made, and the average value is taken. The symbol HR is supplemented by a letter (B or C), which denotes the hardness scale used. The scale used in this test was the C scale which made the value to be in HRC.

RESULTS AND DISCUSSION

Rockwell Hardness Test

Table 1. Hardness test results for samples quenched in water at 0°C.

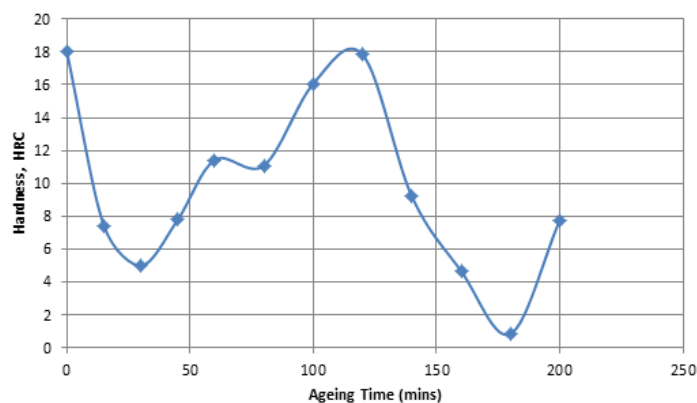| Ageing Time (mins)	Hardness Value(s) HRC Load 1471N			
	1	2	3	Mean HRC
As quenched	16.50	16.50	20.50	12.2
15	07.30	07.30	07.70	12.33
30	04.90	05.10	05.00	12.4
45	09.40	06.80	07.20	15.9
60	10.70	11.30	12.30	20.13
80	10.50	11.00	11.80	23.8
100	15.80	16.20	16.20	16.8
120	12.20	25.00	16.80	12.49
140	14.60	11.90	01.10	7.6
160	04.30	05.00	04.70	7.53
180	00.70	01.00	00.90	9
200	07.30	07.80	08.20	2.7

Figure 4. Graph of hardness versus ageing time for samples quenched in water at 0°C.

From the results in **Table 1** the as-quenched condition had a hardness value of 12.2HV. After this, the hardness value increased, peaking at eighty minutes with a peak hardness value of 23.8HV. This may likely be attributed to sufficient time for diffusion of the atoms of the alloy system thereby, leading to precipitates attaining the required critical size and distribution for precipitation hardening to occur. As a result, fine precipitates that were well dispersed were formed. These

precipitates were effective in retarding dislocation motion, hence, the observed increase in hardness value. As the ageing time increased, as was observed between one hundred minutes and one hundred twenty minutes, the hardness value decreased which could be attributed to the transformation of the fine precipitates to coarse precipitates as ageing progressed. These fine precipitates in the alloy will impede dislocation motion in the alloy system, thereby, hardening the alloy **Figure 4**. Also, because of the temperature of the system i.e. 0°C, the rate of diffusion of atoms of the alloy were too slow, since temperature affects the rate of diffusion corroborating the works of Ilangovan and Sellamuthu [12].

Table 2. Hardness test results for samples quenched in water at 25°C.

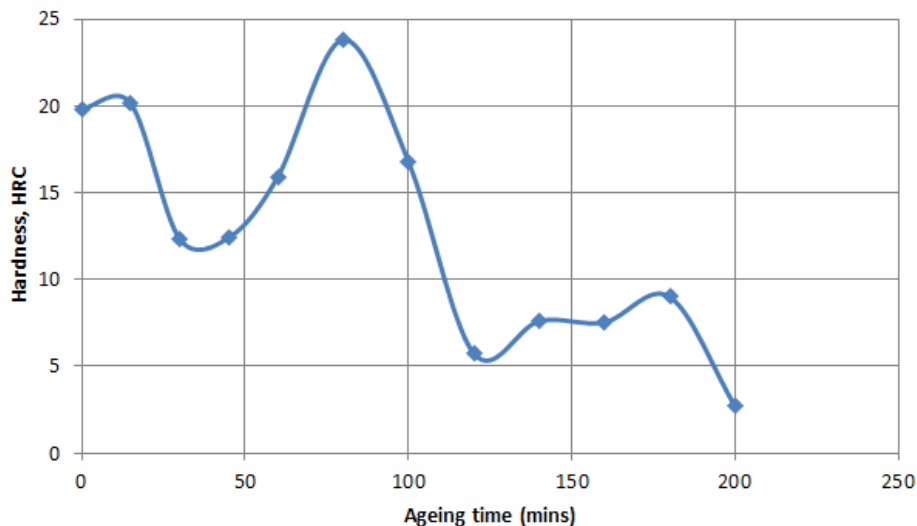| Ageing Time (mins)	Hardness Value(s) HRC			
	1	2	3	Mean HRC
As quenched	20.40	11.10	28.00	4.65
15	25.40	19.90	15.10	6.1
30	19.90	05.60	11.50	7
45	01.60	20.10	15.50	9.48
60	06.60	19.00	22.40	14.6
80	15.50	27.90	28.00	16.07
100	19.00	14.60	00.00	15.5
120	05.50	06.00	00.00	14.8
140	05.70	09.50	00.00	12.20
160	06.10	07.20	09.30	10.96
180	07.00	09.90	10.10	9.71
200	02.70	00.00	00.00	7.32

Figure 5. Graph of hardness versus ageing time for samples quenched in water at 0°C.

The results of the Rockwell Hardness test for samples quenched in water at 25°C are as presented in **Table 2** and the plot in **Figure 5**. The Rockwell hardness value of the test specimen in as-quenched state is 4.6. Maximum Rockwell hardness value of 16.07 was obtained at one hour twenty minutes i.e. eighty minutes while the lowest hardness value of 6.1was obtained at fifteen minutes. After fifteen minutes of aging, the hardness of the material increased, reaching a maximum of 16.07 at eighty minutes. Thereafter, it continued to decrease again from one hundred minutes to two hundred minutes. In Al-Cu binary alloys, the precipitation sequence can be represented as: supersaturated solid solution —• GP zones —• θ'' —• θ' —• θ (Al_2Cu). In these alloys, the hardening observed at room temperature is attributed to

localized concentrations of copper atoms forming Guinier-Preston zones, designated GP (1). The atoms do not have sufficient time to diffuse to potential nucleation sites and thus θ precipitates do not form, hence, the decrease in hardness value. Also, when the precipitates become too coarsened, they are too dispersed to be as effective in retarding dislocation motion, and hardness decreases. At eighty minutes, it reached a peak value of 16.07; it decreased again after this time, reaching the lowest hardness value of 7.3 at two hundred minutes. As was noted by Sheng, the maximum hardness value, which is known as the peak aging point or T6 temper, is associated with precipitates attaining a critical size and distribution. The formation of a finely dispersed precipitate in the alloy is the objective of the precipitation-hardening process. The fine precipitates in the alloy impede dislocation movement by forcing the dislocations to either cut through the precipitated particles or go around them. By restricting the dislocation movement during deformation, the hardness of the alloy is improved. Optimal mechanical properties are achieved by growing the θ" phase as large as possible without beginning to form the intermediate θ' phase. The observed decrease in hardness value after the peak hardness value of 16.07 at eighty minutes is likely due to the formation of intermediate 'θ' phase which caused recrystallization, softening, and a decrease in hardness; this is known as over-aging. Therefore, as was noted by Ilangovan and Sellamuthu [12]. Hardness increases with increasing aging time up to a certain limit which depends on the alloy composition and aging temperature.

Table 3. Hardness test results for samples quenched in water at 40°C.

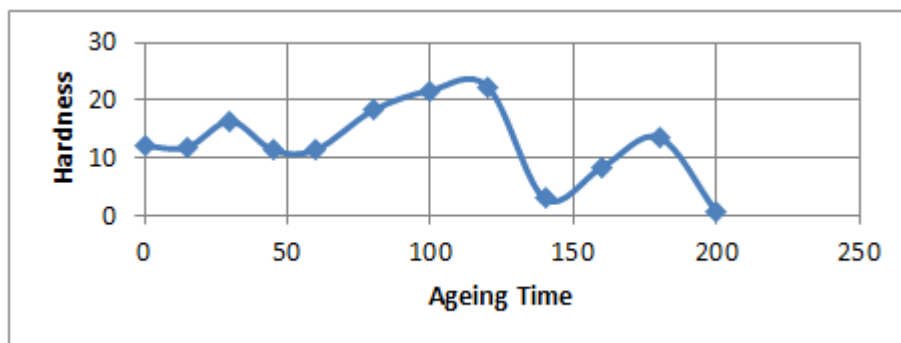| Ageing Time (mins)	Hardness Value(s) HRC			
	00.00	11.40	12.60	7.83
As quenched	14.90	13.30	07.10	8.5
15	10.60	21.00	17.30	8.92
30	14.60	25.40	27.00	18.9
45	00.00	15.90	27.40	15.23
60	00.00	18.90	17.50	12.3
80	04.70	10.20	10.20	18.5
100	10.40	03.40	20.10	18.2
120	01.90	00.00	03.90	16.34
140	00.00	00.00	08.30	12.3
160	17.80	16.40	06.30	11.2
180	00.00	00.00	00.60	8.7
200	00.00	11.40	12.60	7.83

Figure 6. Graph of hardness versus ageing time for samples quenched in water at 40°C.

Hardness test results for samples quenched in water at 40°C are as presented in **Table 3** and plotted in **Figure 6**. In water quenching, as was observed by Shen, cooling rates can be reduced by increasing water temperature. The formation of the precipitates to an optimum size and distribution will be more rapid at a higher temperature so that the peak hardness occurs at the shorter time as the aging temperature increases. From **Table 3**, this was evident as the peak

hardness of 18.9HV was achieved at forty-five minutes aging time. However, the amount of the precipitate formed decreased with increasing temperature (the degree of supersaturation decreased), and the magnitude of the peak hardness decreased. At 40°C, Table 3, the peak hardness is lower compared to that where the water temperature is lower. This is in line with the work of Kadhim, where it was noted that the increase in water temperature lowered the hardness of water-quenched alloy. The quenching characteristics of water are significantly changed by heating the water (http://www.croucher.us/water-quenching). Also, it is expected that at a given aging temperature, the higher the solute content, the faster the peak hardness will be attained, because the supersaturation will be greater, and therefore diffusion will be faster. Increasing the water temperature can result in a significant loss in hardness and tensile properties when the part is later aged especially when heat treating the more quenches sensitive 2000 and 7000 series alloys (http://www.croucher.us/water-quenching).

Table 4. Hardness test results for samples quenched in distilled water.

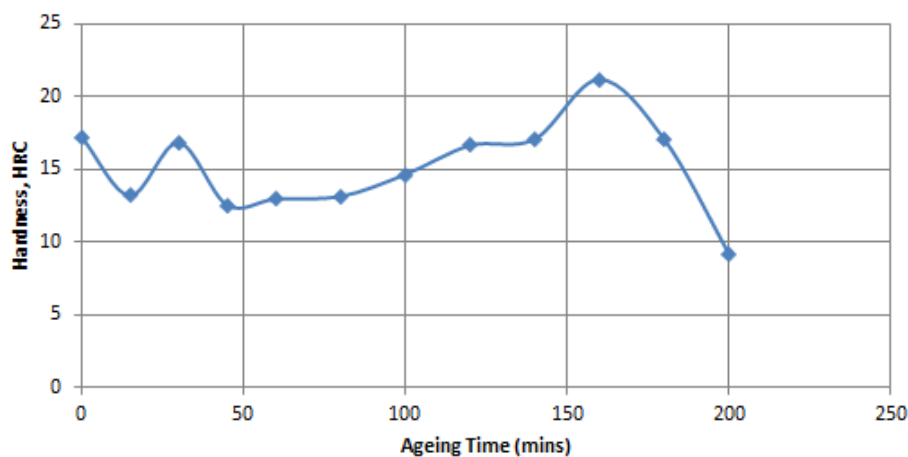| Ageing Time (mins)	Hardness Value(s) HRC			
	1	2	3	Mean HRC
As quenched	08.00	17.30	26.40	12.5
15	04.40	20.50	14.70	12.55
30	00.40	27.70	22.40	13.13
45	20.70	04.40	00.00	14.62
60	10.90	15.00	00.00	16.63
80	01.20	17.70	20.50	17.07
100	03.60	26.30	20.00	12.95
120	14.70	23.90	11.30	13.21
140	16.20	22.20	12.80	21.16
160	12.60	25.40	25.50	16.83
180	08.40	26.30	00.00	17.07
200	04.40	15.60	07.60	12.4

Figure 7. Graph of hardness versus aging time for samples quenched in distilled water.

The results of Rockwell Hardness test for samples quenched in distilled water is presented in Table 4 and plotted in Figure 7. The hardness value of the test specimen in the quenched state was 12.55HV. Maximum Rockwell hardness value of 21.16HV was obtained at one hundred and forty minutes while the lowest hardness value of 12.4HV was obtained at two hundred minutes. The long time to reach peak hardness is likely due to delay in the formation of a finely

dispersed precipitate in the alloy system responsible for precipitation-hardening process and may be attributed to the low rate of diffusion of copper atoms in distilled water. As the concentration of Cu atoms in the alloy system gradually decreased [13], observed that precipitation hardening is delayed, leading to copper leaned area and peak hardness at larger aging times. Under all the heat treatment conditions, the micro-hardness increases with the aging time to a peak value and then decreases with prolonged aging time. This can be explained by the evolution of $CuAl_2$ precipitates with the aging time and the interaction between the precipitates and dislocations. Water quenched samples usually show peak hardness at later stages of aging time and this can be attributed to the diffusion of copper atoms from the base metal to the inter-metallic region of the alloy thus reducing the copper concentration required for precipitation hardening. Water quenched samples produce more dislocations.

CONCLUSION

Precipitation kinetics of Al-4.5% Cu in water quench media at 0°C, 25°C, 40°C had been studied. Water at 0°C and 25°C recorded peak hardness values after the same aging at one hour twenty minutes, although water at 0°C had the highest hardness value of 23.8. Samples quenched in water at 40°C had its hardness value at a shorter aging time of forty-five minutes. Distilled water 0°C had the samples quenched in it attain peak hardness after the ageing time of one hour fifty minutes. Therefore, water temperature and the state in which the water exists affects the precipitation kinetics of Al-Cu alloys quenched in water as was shown by the hardness values developed in the samples studied [14].

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