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MICRO-HARDNESS OF RAPIDLY QUENCHED OF SOME ALUMINIUM ALLOYS (AL 14-16% CU AND AL 4% CU)

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ABSTRACT

Two simple apparatus based on the hammer and anvil principle have been constructed and used to study the micro-hardness of some AL-base alloys. Foils with thicknesses arranging from 20 µm up to 600 µm have been obtained. The cooling rate was estimated to be in the range of $1.6x10^4$:$7.5x10^4$ k sec^{-1}.
Rapid quenching of Al- 14-16% Cu showed the micro-hardness increases particularly in foils below 0.3 mm in thickness. Isochronal annealing of theses foils show that the highly supersaturated Al-14-16% Cu solid solution decomposes readily at relatively low temperature and short time intervals. The maximum hardness is obtained after annealing at 100 C for 30 minutes. However, with decreasing the Cu content of the foils the precipitation process is largely delayed. Eight hours of annealing at 100 ºC was not enough to achieve the maximum hardness in Al-4% Cu thin foils. The achieved hardness value was more than twice of the maximum hardness obtained in articles of similar composition but conventionally aged.

Keywords
Aluminium.
Copper.
Isochronal.
Micro-hardness.
Rapid quenching.

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1 INTRODUCTION

Rapidly quenching means, the change the crystal structure of the metal to be cooled. Rapidly quenching is used in order to produce or get some properties that cannot be achieved by normal cooling. Quenching is the rapid cooling of a metal to obtain certain properties such as microstructure, hardness, and residual stresses which in turn leads to certain strength characteristics, abrasion resistance. For example, rapidly quenching of steel produces a martensitic

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phase. This process produces a favorable compressive residual stress at the surface and significantly increases fatigue strength [6,7].

The aim of this study is to attempt to prepare some thin rapidly quenched discs of alloys of two binary systems based on aluminum by simple methods. The study of micro-hardness has been performed as-quenched conditions and after subjecting them to low-temperature annealing (aging). Retaining of high temperature phases and for obtaining new metastable phases by means of rapid cooling of metal alloys from high temperature, followed by heating at relatively low temperatures has been successfully used to regulate and control phase transformation processes and to achieve microstructure with optimal properties. However, such technology has been limited to the use of transformation in the solid state.

2 METHODS OF RAPID SOLIDIFICATIONS

Rapid solidification or rapid liquid quenching aims to achieve a metastable intermediate phase, high super-saturation or amorphous phases that cannot be obtained or achieved by conventional techniques. Rapid solidification (quenching) is a typical melt-quenching technique that can be classified into three categories:

- Spinning
- Droplet
- Surface melting

This requires that the cooling process should provide sufficiently high heat transfer coefficient at the liquid metal interface and sufficiently thin metal cross section that the heat conducted to the cooling medium is maximum. All the methods applied for rapid quenching from the liquid state can be classified into four groups:

I. Transformation of a drop or a thin stream into a foil and cooling by transfer to metal substrate.

II. Atomizing of liquid metal by gas jet and cooling of atomizing droplets in the gas stream, liquid quenching or on metal substrates

III. Pulling of micro-wires into glass shells and cooling them by the liquid stream.

IV. Melting of surface layers of materials by laser or electron beams and cooling by heat conduction to the bilk of un-melted material.

The majority of rapid liquid quenching techniques have utilized metal substrates due to high thermal conductivity. The molten metal spread in a thin layer on the substrates and subsequently solidifies. One of the widely used methods for liquid solidifications is the melt spinning technique, where a steam of molten metal is directed at rapidly moving substrate, the final product being ribbons 20 to 100 μm thick.

In the last two decades, another class of processes has been developed and referred to "laser glazing". The composition of this layer can be modified by local surface alloying using hydrodynamic processes taking place under the action of the beam. Relative movement of the sample and the beam removes the molten section from within the beam. The solid material beneath it then quenches the liquid. Thin surface layers of advantage properties can be produced while the article bulk retains its properties [1].

3 COOLING RATES IN RAPID SOLIDIFICATION

In all liquid quenching techniques, the magnitude of the cooling rate is a major importance. The short time periods, small dimensions and possible instabilities inherent in most quenching processes which make it difficult to determine an accurate value of the actual cooling rate. The cooling rate may divide into three categories [2] based on the value of newton criterion N.

$$N = (h\ d)/k \tag{1}$$

where:

d= film thickness

k= thermal conductivity of the film

h= coefficient of heat transfer at the molten layer

I. Ideal cooling when the newton criterion $N>30$
II. Cooling regime at $N<0.015$ where the cooling process is completely controlled by the interface condition
III. Intermediate regime $0.015<N<30$

Table 1 Estimation of substrate foil contact conditions and cooling rates encountered during solidification of aluminium base foils of different thickness based on Newton criterion

Foil thickness(μm)	Newton criterion	The controlling step in heat transfer	Estimated cooling rate at the moment of solidification k sec^{-1}
10	0.005	Interface controlled	10^6
20	0.01	Interface controlled	$5x10^5$
50	0.024	Intermediate regime	$2x10^5$
100	0.048	Intermediate regime	10^5
200	0.096	Intermediate regime	$5x10^4$
300	0.144	Intermediate regime	$3x10^4$
400	0.192	Intermediate regime	$2.5x10^4$
500	0.24	Intermediate regime	$2x10^4$
600	0.288	Intermediate regime	$1.67x10^4$

4 EXPERIMENTAL PART

The hammer and anvil principle of rapid cooling has been selected as shown in figure (1). If properly applied, it gives cooling rate in the range 10^5-10^6 k/sec and foils of thickness ≤ 0.1 mm, both hammer and anvil have been fabricated from oxygen free of high conductivity (OFHC) red copper. The contacting surfaces were grinded to 1000 grit and mechanically polished by fine aluminum paste to achieve best contact and maximum heat transfer from the solidifying squeezed molten metal to the squeezing surfaces. The squeezing effect is achieved by the impact of the hammer part on the anvil part where the molten drop is located.

5 EXPERIMENTAL PROCEDURE

In this study, two alloy compositions have been investigated namely:

I. Al 14-16% Cu
II. Al ≈ 4% Cu

Figure 1 Hammer and anvil principle.

Re-melting of small pieces from each ingot for rapid solidification quenching has been performed in small alumina crucibles. Heavy-duty high heating rate furnace with SiC heating elements has been used for this purpose. The re-melting temperatures were selected to be ≈ 100°C above the liquidus (i.e. 730ºC and 760°C for Al 14-16% Cu and Al-4% Cu; respectively).

The parts of the apparatus (hammer and anvil) used for rapid quenching were kept for three hours in the freezer of a refrigerator (i.e. -12°C) before being used. After melting and stabilization of the furnace temperature, the crucible was left in the furnace for an additional 15 minutes to achieve full compositional and temperature homogenization of the melt. The crucible was then taken out of the furnace. A metal article to release the internal unoxidized melt from its thin sheath scratched the melted surface (mirror), and drop of melt was thrown into the anvil part. Depending on the amount (size) of the drop, different foil thicknesses could be achieved, the less the drop size the thinner the foil obtained.

6 SPECIMEN PREPARATION

The micro-hardness of quenched foils were measured, the foils were cut into specimens ≈ 10x10 mm, cold mounted in serifix cold mounting resin and subjecting to grinding by SiC abrasive papers of successive grit numbers 220, 320, 500, and 1000 by the conventional way. The specimens were further polished by diamond paste followed by 0.25 on semi-napped cloth. The micro-hardness values of the mounted specimen were measured on the finally polished surfaces as shown in figure (2).

Vickers and knoop micro-hardness measurements were performed. A special micro-hardness attachment Leitze- Aristomet camera microscope used. A unified load of 25 gram was selected for use during this study.

7 HEAT TREATMENT OF RAPIDLY QUENCHED FOILS

In order to study the effects of heat treatment on the micro-hardness of rapidly quenched Al-Cu alloys two series of experiments established.

I. Thin foils of as quenched Al-14- 16% Cu alloy of different thicknesses have been subjected to isochronal annealing at two different annealing times 10 min and 30 min.The annealing temperatures selected for this treatment were 100, 150, 200, and 300 °C. The treatment performed in the dry oven with the temperature control ± 2°C by means of the mercury thermometer, the specimens were put into the oven, when it was at required temperature, left for required time interval then taken out and air cooled.

II. Very thin specimens of Al 4% Cu (less than 100µm) alloy were subjected to isothermal annealing at 100°C for time intervals ranging from 15 min up to 8 hours. The specimens were sheeted in an aluminium packing foil, treated in boiling water (100°C) for required time intervals and air-cooled.

The heat-treated specimens were cold mounted; grinded, polished, and etched as mentioned earlier followed by micro-hardness measurements.

Figure 2 Grinding, Polishing and Micro-hardness measurements

8 RESULTS AND DISCUSSION

The thickness measurement of the quenched foils obtained by using a hammer and anvil is ranging from ≈ 20 µm to ≈ 650 µm. The most important factors that effect on the thickness are the volume of the molten drop, the temperature of the melt and the impact of the hammer part on the drop. In case of Al 4% Cu the minimum obtained thickness is 100 µm.

An attempt has been undertaken to evaluate the cooling rates (table 2&3) encountered in this study based on the homogeneity of the resulting thickness and the smoothness of the foil surface. By considering the thermal conductivity of aluminium (K) is 209 w $m^{-1}k^{-1}$ and the heat transfer coefficient (h) is $\approx 10^5$ w $m^{-2}k$ (1)[3-4]. The results achieved in tables (2) and (3) by substituting the values of (h), (k) and thickness in equation (1).

The cooling rate for Al 14-16% Cu increases from $1.6x10^4$ to $5x10^4$ k sec^{-1}, for Al 4% Cu, the cooling rate is $7.5X10^4$ k sec^{-1} (estimation). However, the prediction of further increase in the cooling rate with further decrease in the foil thickness, below 50μm is questionable, since the heat transfer condition have been changed and the heat transfer is no longer controlled by the melt. Under such conditions, it is more likely that the cooling rate in this investigation is estimated in the range of 10^4-10^5 k sec^{-1}.

As shown in figure (3), the foils of different thicknesses have been subjected to isochronal annealing at two time levels (10 min and 30 min) for increasing temperatures.

Table (4) introduces the summary of micro-hardness measurements carried out on the annealed specimens.

Table 2: estimation of cooling rate based on Newton criterion for Al 14-16% Cu

Thickness (m)	K(wm^{-1} k^{-1})	h($wm^{-2}k^{-1}$)	Newton(N)	Estimated cooling rate ($ksec^{-1}$)
0.0002	209	100000	0.0956938	$5X10^4$
0.00027	209	100000	0.1291866	$3X10^4$
0.00035	209	100000	0.1674641	$2.75X10^4$
0.00055	209	100000	0.2631579	$2X10^4$
0.00065	209	100000	0.3110048	$1.6x10^4$

Table 3: estimation of cooling rate based on Newton criterion for Al 4%

Thickness (m)	K(wm^{-1} k^{-1})	h($wm^{-2}k^{-1}$)	Newton(N)	Estimated cooling rate ($ksec^{-1}$)
0.0001	209	100000	0.04784	$7.5X10^4$

Table 4 Micro-hardness measurements of rapidly solidified foils of different thicknesses annealed at different temperature for different time intervals (Al 14-16% Cu).

Annealing temperature ºC	Annealing time (minutes)	**Foil thickness (mm)**				
		0.2 mm	0.27 mm	0.35 mm	0.55 mm	0.65 mm
				H_v kg/mm^2		
RT	0	205	193	183	174	170
100	10			242		

	30	254				
150	10		222			
	30	220				
200	10			203		
	30				171	
250	10			168		
	30	170				
300	10					161
	30				153	

Figure (3) shows the effects of increasing annealing temperature on the micro-hardness values of foils of two different thicknesses keeping the annealing time constant. The introduced data indicates that the annealing for 30 minutes at such a low temperature as 100ºC effects on the incremental in hardness in ≈ 50 kg/mm^2. It is probably connected with sub-microscopic precipitation in the cell bulk [5]. With further increase in the annealing temperature the micro-hardness values decreases the reaching value as low as 176 kg/mm^2. Such micro-hardness decreases is probably caused by the coarsening and changes in the nature of precipitates rejected from the highly saturated solid solutions. The results of isochronal annealing of foils ≈ 350µm in thickness are similar to those of foils ≈ 200 µm thick.

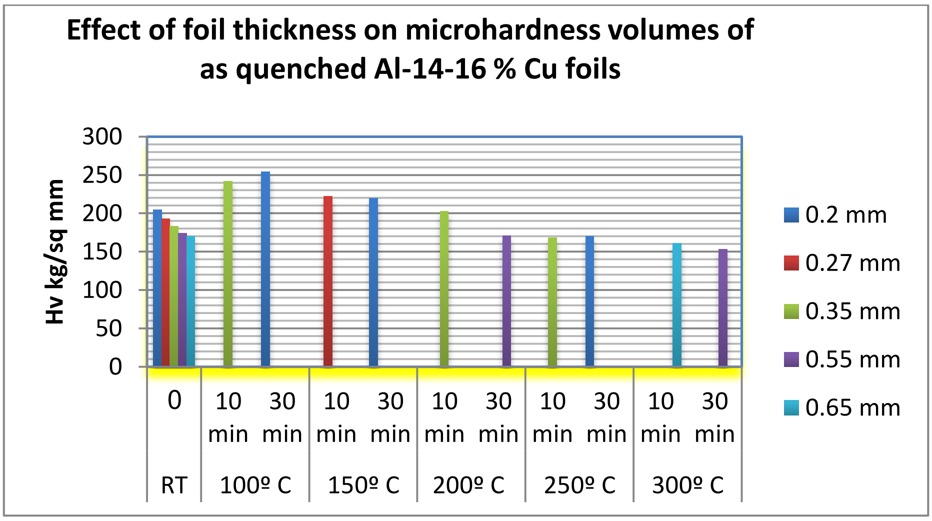

Figure 3 Effect of foil thickness on micro-hardness volumes of as quenched Al-14-16% Cu foils.

The effect of how temperature isothermal annealing (100ºC) for increasing time intervals on the micro-hardness of Al 4 wt. % Cu has been performed with foils of a thickness below 100 μm table(5).

Table 5 Micro-hardness of rapidly solidified Al 4 wt. % Cu specimens of < 0.1 mm thickness, aged at 100ºC.

Aging time Hrs	**Hv kg/mm2**					**Average Hv kg/mm2**
0	259.8	259.8	241.3	259.8	268.5	258
0.25	222.3	229.1	241.3	233.8	246.3	235
1	233.8	259.8	241.3	233.8	236.3	241
2	259.8	274.5	268.5	259.8	294.5	267
4	274.5	268.5	290.3	300.7	259.8	279
6	286.3	290.3	304.5	300.7	326.7	302
8	360	326.7	395	347.4	360	358

The results of knoop micro-hardness measurements carried on the specimens annealed at 100ºC for the time interval from 15 minutes up to 8 hours as shown in figure (4). In the first portion of annealing, the micro-hardness slightly decreases probably due to annihilation of some of the excess vacancies that have been frozen in during rapid quenching. With a further increase in annealing time, the micro-hardness starts to increase, first slower, then faster. After 8 hours of annealing at 100 º C, the incremental increase in micro-hardness constitutes 100 kg/mm^2. In order to compare the results obtained in this investigation on rapidly quenched foils with results obtained for conventionally solidified articles the Vickers micro-harness values were determined for the foil annealed for 8 hrs. The average of five measurements was 256 kg/mm^2.

Eight hours of annealing at 100°C were not enough to achieve the maximum hardness in the foils. However, the absolute values measure are approximately twice those obtained in articles of similar compositions aged to their maximum hardness by conventional age-hardening methods, comparing the results of isothermal annealing of thin rapidly quenched Al-4% Cu foils at 100°C with those of isochronal annealing of rapidly quenched Al 14-16% Cu.

Eight hours of annealing in the first case were not enough to achieve the maximum hardness, while about thirty minutes at the same temperature in the second case were enough to achieve the maximum hardness. The measured hardness values in both cases were almost identical. Such great difference in kinetics of precipitations is probably caused by much larger super-saturation caused by rapid quenching in Al 14-16% Cu foils.

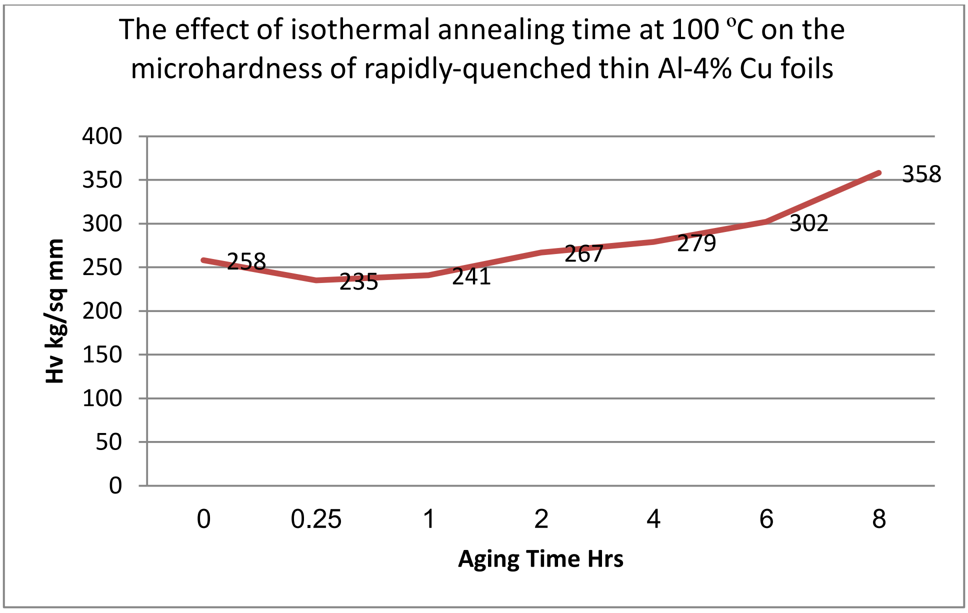

Figure 4 The effect of isothermal annealing time at 100 ºC on the micro-hardness of rapidly quenched of thin Al 4% Cu foils

9 CONCLUSION

With decreasing the foil thickness, the micro-hardness of the Al 14-16% Cu foils increases, the most remarkable inverse being observed in the thickness below 0.3 mm, highly supersaturated solid solution of Al 14-16% Cu foils show maximum hardness on annealing at relatively low temperature for relatively short time intervals (30 min at 100ºC).

The micro-hardness values obtained because of isothermal annealing of rapidly quenched Al 4% Cu thin foils are more than twice of the maximum hardness values obtained in conventionally aged articles of similar composition.

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الصلادة الدقيقة لبعض سبائك الالومنيوم المبردة تبريدًا سريعًا

(Al 14-16% Cu and Al 4% Cu)

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الملخص

تم بناء جهازين بسيطين يعتمدان على مبدأ المطرقة والسندان واستخدامهما لدراسة الصلادة الدقيقة لبعض سبائك الالومنيوم AL-base. تم الحصول على رقائق بسمك يتراوح من 20 ميكرومتر إلى 600 ميكرومتروتم تقدير معدل التبريد في حدود $1.6x10^4$: $7.5x10^4$كلفن/ثانية أظهر التبريد السريع لـ Al- 14-16٪ Cu أن الصلادة الدقيقة تزداد خاصة في الرقائق التي يقل سمكها عن 0.3 مم ويظهر التلدين المتزامن للرقائق أن محلول النحاس شديد التشبع Al-14-16٪ Cu يتحلل بسهولة في درجات حرارة منخفضة نسبيًا وفترات زمنية قصيرة. يتم الحصول على أقصى صلادة بعد التلدين عند 100 درجة مئوية لمدة 30 دقيقة. ومع ذلك ومع انخفاض محتوى النحاس في الرقائق تتأخر عملية الترسيب إلى حد كبير. ثمانية ساعات من المعالجة عند 100 درجة مئوية لم تكن كافية لتحقيق أقصى صلادة في رقائق Al-4٪ Cu الرقيقة. كانت قيمة الصلادة المحققة أكثر من ضعف الحد الأقصى للصلادة التي تم الحصول عليها في أصناف ذات تركيبة مماثلة.

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متساوي الزمن.
الصلادة الدقيقة.
التبريد السريع.