Dry Sliding wear behavior of spray formed Al20Mg_2Si2Cu alloy after two-step solution heat treatment and double age hardening at 150° and 210°C.

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Abstract

In the present work Al20Mg_2Si2Cu alloy was synthesized by spray deposition technique followed by hot pressed, heat treated and dry sliding wear behavior of double aged Al20Mg_2Si2Cu alloy was investigated. The alloy was solution treated in single step (1 hr at 480°C) single aged (3 hrs at 210° C) and the alloy sample was solution treated in two step (1 hr at 440° C +1.5 hrs at 480° C) double age (4 hrs at150° C + 210° C for three different periods of time 2,4,6 hrs) hardening. The microstructures of the aged samples were examined by scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS) in the whole process. The hardness values of the aged samples were measured after 70 days by micro hardness tests. Wear tests were carried out under different sliding velocities for variable loads with constant sliding distance of 1000m. As a result of this study the major precipitates for the alloy get formed under the two aged conditions. The hardness of the second phase precipitated samples was found to be higher than that of the single age samples and it is increased with increasing the solution temperature and aging period.

Key words Aging, Al20Mg_2Si2Cu, hardness, hot pressing , precipitation , SEM, spray deposition process , wear.

1. Introduction

The growing demand for reduction in weight, energy saving in the areas of automotive, aerospace and other structural applications has lead to the development of novel, lightweight automobile materials during the last decade. Aluminum alloyed with high content of Mg_2Si forms the fine microstructure by spray forming due to high cooling rate [1]. This alloy system has high potential as automobile brake disc material because the intermetallic compound Mg_2Si exhibits a high melting temperature, low density and high hardness [2]. These alloys are employed extensively in a wide variety of applications on account of their excellent castability, high thermal conductivity and low thermal expansion coefficient [3]. When it is further added with copper (2%wt), it leads to further increase in strength by heat treatment (age hardening) due to precipitation of Al_2Cu phase. The precipitation hardening is a thermal treatment, which consists of a heat treatment, quenching and artificial ageing process.

With convenient alloying and heat treatment, hardness increases 40 times compared to high purity aluminum. The main strengthening mechanism in the alloys is precipitation hardening by structural precipitates formed during artificial aging. It strengthens them through solid solution and hardening precipitation [4]. A two-step solution treatment, namely, conventional solution treatment followed by a high-temperature solution treatment, as suggested by Sokolowski et al. [5,6],produced the optimum strength compared to the traditional single-step solution treatment. The solution treatment process needs to be optimized, because too short a solution treatment time means that not all alloying elements added will be dissolved and made available for precipitation hardening, while too long a solution treatment means using more energy than is necessary. The solution heat treatment may be carried out in either a single step or in multiple steps.

The characterization of the precipitates is determined by the precipitation process during the ageing treatment. The precipitation is a complex process that may include several simultaneous reactions, depending on ageing conditions and pretreatments [7–9]. The usual precipitation sequences may be summarized as [10, 11]:

Supersaturated solid solution (SSS) → GP zones→ β' metastable phase →β stable phase.

In spray formed, the primary fine Mg_2Si (β) particles are interconnected with dispersoid distributed in α-Al matrix and intermetallic phases (θ and Q). Precipitation hardening phases are Al_2Cu (θ), Mg_2Si (β) and Al_2CuMg (Q).
Artificial ageing will be accomplished not only below the equilibrium solvus temperature, but below a meta-stable miscibility gap called Guinier- Preston (GP) zone solvus line.
The principal precipitation-hardening reactions and the phase reactions which occur between an

aluminum solid solution and the intermetallic phases are $CuAl_2$ and $CuMgAl_2$.

The precipitation hardening at high ratios of copper to magnesium is achieved in the sequence GP zones through a coherent phase (θ') to Al_2Cu (θ). Precipitation hardening at lower ratios of copper to magnesium is achieved in the sequence GP zones through a coherent phase to Al_2MgCu. These intermetallic precipitates are coherent with main structure and affects the mechanical and physical properties of the material.

After artificial aging the GPZ (Mg, Si) and β'phase are formed; it is due to refining and increasing the precipitation density of these β' particles (Mg_2Si) and therefore, improves the strength [12] upto the moment when the concentration of copper becomes sufficient to form its own GP (Al, Cu).

Many works have been done on microstructure control of the $AlMg_2SiCu$ earlier [13,14], but less work has been carried out on the wear behavior. The wear behavior of aluminum alloys has been considered substantial. By most estimates, improved attention to friction and wear would save developed countries up to 1.6% of their gross national product, or over $100 billion annually in the USA alone [15,16].

In the present study, Al-20Mg_2Si-2Cu alloy was spray formed and hot pressed and aimed at investigating the effects of single and two-step solution heat treatment followed by quenching and the effect of single age and Double aging on the precipitation in order to achieve higher strength. The precipitation-hardening response depends on the temperature of aging, the degree of deformation and the composition and to investigate dry sliding wear behavior on a single step single aged and two step solution heat treated double age hardened on sprayed and hot pressed Al20Mg_2Si2Cu alloy.

2. Experimental methods

The chemical composition of Al-20 (wt %) Mg_2Si2 (wt %) Cu alloy is shown in Table 1. The details of spray forming set up employed in the present study, have been described elsewhere [17]. Spray forming processes were employed to produce the Al20Mg_2Si2Cu alloy. During the spray forming process, the molten metal was heated to 800^{0}C, and then flowed through the nozzle and atomizer. The nitrogen gas was used to atomize the molten metal into droplets and these droplets were deposited on a substrate to form a near net shape preform and the process parameters were listed in Table 2.

The preform, was cut to the size 100 x 30 x 20 mm and was hot (temperature of 480°C) pressed at a pressure of 55 MPa for porosity reduction.

Single Aging

After hot pressing, a solution heat treatment was carried out in a muffle furnace at 480^{0}C for 1h followed by water quenching. Aging was carried out at 210 °C for 3 h.

Double Aging

In Double age hardening the low temperature is followed by high temperature aging, In this hardening, samples were solution heat treated for 1 h at 440 °C and for1.5 h at 480 °C followed by water quenching and two stage artificial aging for 4h at 150^{0}C and 2h,4h & 6h at 210^{0}C.

Samples were extracted from the above aged pieces for microstructural examination. The samples were prepared by polishing using standard metallographic techniques of grinding on emery paper with 1/0, 2/0, 3/0 and 4/0 specifications. Final polishing was done on a wheel cloth using aluminium oxide powder. The polished samples were etched with Keller’s reagent (1% vol. HF, 1.5% vol. HCl, 2.5% vol. HNO3 and rest water). Its microstructural evolution was investigated by scanning electron microscopy (Model: S-3400N Hitachi Model), with energy dispersive spectroscopy (EDS) in the whole process.

Dry sliding wear tests were carried out on all the above aged specimens on a pin-on-disc wear testing machine (Model: TR-20, DUCOM). The counterpart disc was made of quenched and tempered EN-32 steel having a surface hardness of 65 HRC. Specimens of size Ø8×30 mm were machined out from all the aged specimens. The specimens were polished and then cleaned with acetone before conducting the test. The wear tests were conducted over a range of loads (9.81–68.67 N) and a sliding velocities (1.0, 1.5 and 2.0 m/s) at a constant sliding distance of 1000 m. The worn surfaces of wear specimen after the test were examined under Scanning Electron Microscopy. The hardness measurement was carried out using Vickers Hardness Tester (Mattoon ATK-600) at an applied load of 300g.

Scanning electron microscopy (SEM) with EDS was employed to analyze the intermetallic compounds and precipitates

3. RESULTS AND DISCUSSION

3.1Microstructure

Fig. 1 shows the morphology and distribution of precipitates in the samples after single aging and double aging. Most of the precipitates appear round in the image, and others appear elongated, as shown in Fig.1 (a). In double aging (150 ◦C/4 h + 210 ◦C/2 h)Fig.1(b) the precipitates exhibit a little higher number density as compared with that of the single aged 3 h at 210 ◦C. The size of the precipitates in the sample of the double aging is smaller than that of single aging.

When the double aging temperature was 210 ◦C/4h, Fig 1(c) the precipitates exhibits higher density than 210◦C /2h.

When the second stage aging temperature was 210 ◦C, after aging for 6h, the precipitates were observed with a wide size Fig. 1(d). The precipitate number density decreases greatly as compared with that of 210◦C/ (4h).

In single step, single aging treatment of 3h at210 ^{0}C, dark spherical particles in fig.1 (a), were identified as solute clusters of GP zones (precipitates of the β and metastable β').

In the two step, double aged for 4h at150^{0}C plus 2h at 210^{0}C the precipitates are distributed homogeneously and their volume fraction is very high.

It shows that the precipitate particles continued coalesce as aging progress caused the particle size to increase and thus decreased the degree of complication for the dislocations to break the Mg-Si bonds when they pass through the precipitates. Thus, it was concluded that the higher the aging the softer the sample which looses some mechanical properties such as tensile strength and hardness. Therefore, aging at temperatures which are too high must be avoided

Table 1 Chemical composition of Al-20 (wt %) Mg_2Si alloy

Material	Si	Mg	Fe	Ni	Zn	Mn	Cu	Ti	Cr
Al20 (wt%)Mg_2Si	8.06	14.43	0.15	0.01	0.01	0.01	1.78	0.01	0.01

Table 2 Process parameters

Process parameter	Value
Atomization pressure Mpa	0.7
Super heat temperature ^{o} C	100
Gas-metal mass flow rate ratio	2.36
Deposition distance (mm)	380

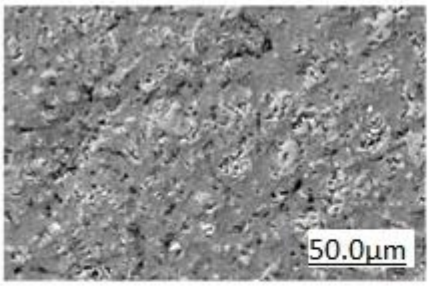

Fig1(a). SEM micrograph of Al20Mg_2Si2Cu single aged 210^{0}C /3h.

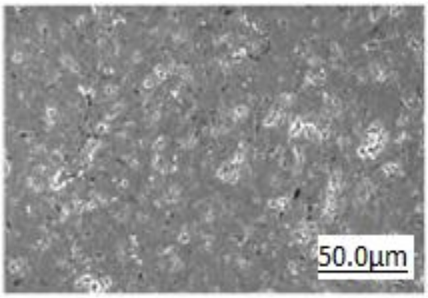

Fig.1(b). SEM micrograph of Al20Mg_2Si2Cu double aged 150^{0}C/4h+ 210^{0}C/2h.

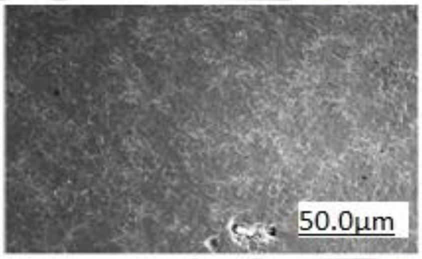

Fig1(c) . SEM micrograph of Al20Mg_2Si2Cu double aged 150^{0}C/4h+ 210^{0}C/4h.

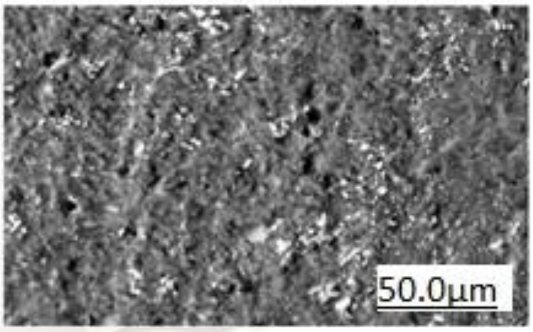

Fig1(d). SEM micrograph of Al20Mg_2Si2Cu double aged 150^{0}C/4h+ 210^{0}C/6h.

3.2. Hardness

The aged samples were measured after 70 days on micro hardness tester and the results of hardness measurement are shown in table 3. It is observed that the hardness of two-step solution treatments of 440^{0}C for 1h followed by 480^{0}C for 1.5 h are higher than the traditional single-step solution treatment of 480^{0}C for1 h due to finer dispersion of precipitates.

According to binary phase diagram of Al–Cu, Mg and Si [18–20], the solubility of Cu, Mg and Si in aluminum is 4.5%, 10.8% and 1% at solution temperature of 500±5^{0}C respectively., Therefore, the low melting point phases of $CuAl_2$ and partial eutectic phases (Mg_2Si, Si) dissolve into aluminum matrix during the longtime solution treatment. The eutectic phase (Mg_2Si, Si) transforms to fine dot-like Fig.1(c). After water-quenched to room temperature, the aluminum matrix transforms to supersaturated solid solution. During the aging process, metastable phase and solute atoms are separated to form a structure of saturated solid solution combining with precipitated phase. The diffuse precipitated phase leads to the increase of hardness.

Copper atoms influence the nucleation and stability of GP (Mg, Si) zones in such a way that these zones become the nuclei for the coherent phase upon artificial aging. The high nucleation rate, which occurs during the low temperature pre-ageing treatment, is responsible for the precipitate refinement. This fine precipitate dispersion is then retained at the high aging temperature. These fine precipitates then transform to the final precipitates during final aging. The higher hardness of the sample given the double aging treatment is due to the higher precipitate density giving rise to a smaller interprecipitate spacing and thus higher stress is required for dislocation bowing.

The hardness of the second phase precipitated samples was found to be higher than that of the single step single aged sample and it increased with increasing the solution temperature and aging periods.

Table 3

S.N	Sample	Vickers hardness(Load 300gms)
01	Single step solution heat treated single age hardening(SSH Sage)	170.4
02	two step solution heat treated double age hardening (150^{0}C/4h+210^{0}C/2h) (DSH,Dage(4+2))	194.8
03	two step solution heat treated double age hardening (150^{0}C/4h+210^{0}C/2h) (DSH,Dage(4+4))	212.6
04	two step solution heat treated double age hardening (150^{0}C/4h+210^{0}C/2h) (DSH,Dage(4+6))	198.3

3.3. Wear studies

Wear tests were carried out on the pin-on-disc (Model: TR-20, DUCOM) wear test apparatus under sliding distance of 1000m

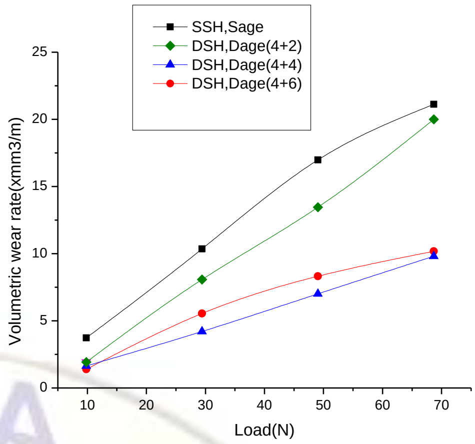

Fig.2 Variation of volumetric wear rate with applied load

Experimental data, i.e. frictional force for every 0.5min at each load (9.81,29.43,49.05and 68.67N), at different sliding velocities (1.00,1.50 and 2.00 m/sec)and at constant sliding distance of 1000 m , the experiment was stopped, then wear samples were removed and measured the mass-loss values of the aged samples during the wear tests and were recorded continuously.

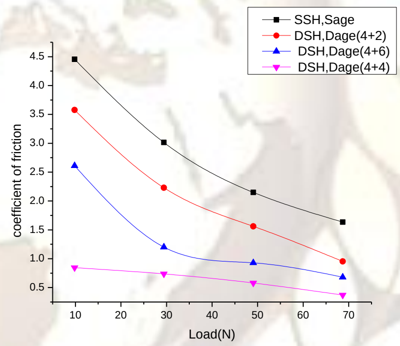

Fig3 Coefficient of friction Vs applied load

The wear rate Al20Mg2Si2Cu alloy for the range of loads and sliding velocity of 2 m/sec at a constant sliding distance of 1000m are shown in fig2. The effect of applied load on the Volumetric wear rates of the single step solution treatment single age hardening[SSH Sage], hardening process as involved are: Two step solution treatment double age[DSH,Dage(4+2)], Two step solution treatment double age [DSH,Dage(4+4)] & Two-step solution treatment double age[DSH,Dage(4+6)] hardening in the load range of 9.81–68.67 N. Obviously, the wear rate of the alloys increases with increasing load. In the entire applied load range, the two step(1h at 440^{0}C,1.5h at 480^{0}C) double age [DSH,Dage(4h at 150^{0}C+4h 210^{0}C)]hardening shows better wear resistance than the others due to fine distribution of approximately spherical precipitates. The precipitates exhibit higher density than the two step solution treatment double age

(150^0C/4h + 210^0C /2h.) hardening and the single step solution treatment single age hardening.

It also reveals that the wear rate of two step double aged (150^0 C/4h+210^0C/6h) is little higher than the two step double aged (150^0C/4h+210^0C/4h) due to the particle coarsen or wide size precipitate and the precipitate number density decreases greatly i.e. precipitate particles continued coalesce as aging progress cause the particle size to increase and thus decrease the degree of complication for the dislocations to break the Mg-Si bonds when they pass through the precipitates. Thus, it can be concluded that the higher the aging the softer the sample which looses the hardness. This study gives the optimum ageing time for second phase precipitation and temperature is determined.

Fig.3 shows the coefficient of friction of the single step solution treatment single age hardening [SSH Sage], Two step solution treatment double age[DSH,Dage(4+2)], Two step solution treatment double age [DSH,Dage(4+4)] & Two-step solution treatment double age[DSH,Dage(4+6)] hardening at sliding speed of 2m/sec in the load range of 9.81–68.67N. In the above alloy two step double ageing (4+4) indicate a reduction in value of coefficient of frwith small size precipitate. It can be seen from the figure that coefficient of friction decreases initially with increase in further load, and increase in load, the coefficient of friction is more or less constant irrespective of processing condition but it is invariably low in Two step solution treatment double age [DSH ,Dage(4+4)]

3.4 Worn surface morphology

In order to investigate the wear mechanism, the surfaces of the worn samples were examined under SEM. Fig4 shows typical worn surface of the single step solution treatment single age hardening [SSH Sage], at the applied load of 68.67 N and sliding velocity of 2m/s. It indicates that the width of fine grooves increases with increasing load and with small size dimples . The worn surface of the alloys has a smooth appearance because of uniform and homogeneous wear mainly by plastic deformation. This kind of worn surface is typical of the abrasive wear. The visible deformed materials at the ridges that remain adhered to the surface due to low speed and thus a low agitation on the surface. The spheroidized Mg_2Si phase, Al_2Cu precipitates are formed.

Fig5 and Table4 shows that EDS result of wear surface of the above alloy and the composition of the alloy. It reveals that the surface exhibits small amount of oxygen besides Cu Mg ,Al and Si. It indicates that an oxidative wear at high load.

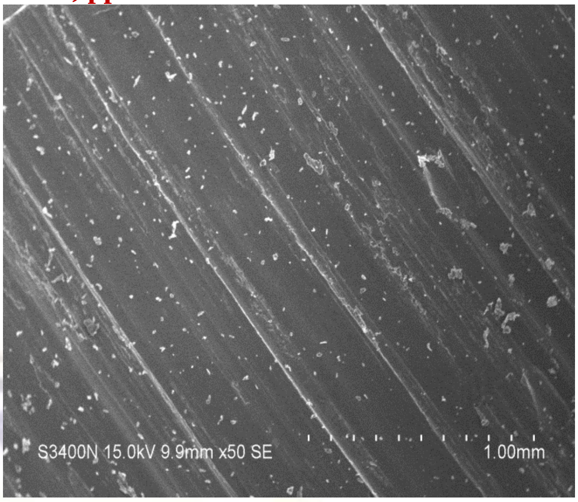

Fig4 SEM morphology of worn surface of single step solution treated single aged alloy at the load 68.67N

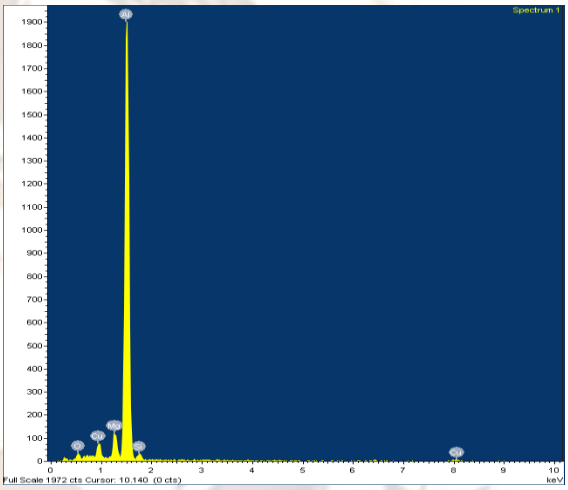

Fig 5: EDS result of wear surface of single step solution treated single aged (210^0C /3h) Al-20Mg2Si 2Cu

Table 4.

Element	Weight%
O K	3.88
Mg K	4.44
Al K	82.61
Si K	2.39
Cu L	6.68
Totals	100.00

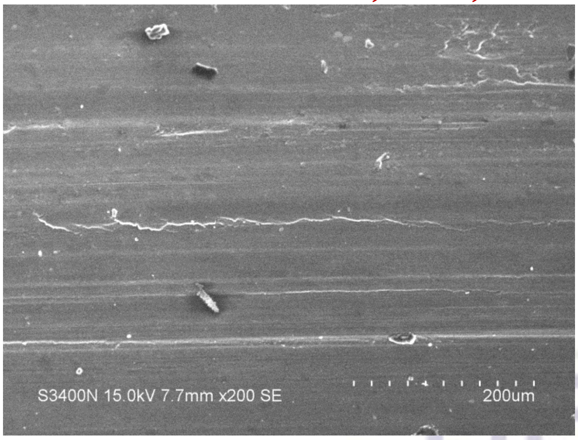

Fig6 SEM morphology of worn surface of two step solution treated Double aged (4+2) alloy at the load 68.67N

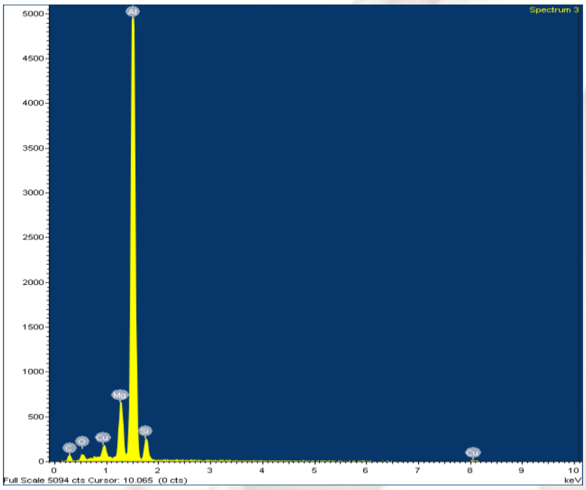

Fig 7: EDS result of wear surface of two step solution treated double aged (150^0 C/4h+210^0C /2h) Al-20Mg2Si 2Cu

Table5.

Element	Weight%
C K	12.74
O K	3.44
Mg K	7.31
Al K	66.37
Si K	5.67
Cu L	4.48
total	100.00

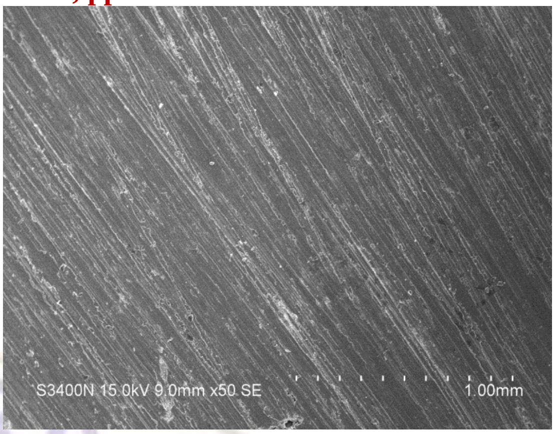

Fig8 SEM morphology of worn surface of two step solution treated Double aged (4+4) alloy at the load 68.67N

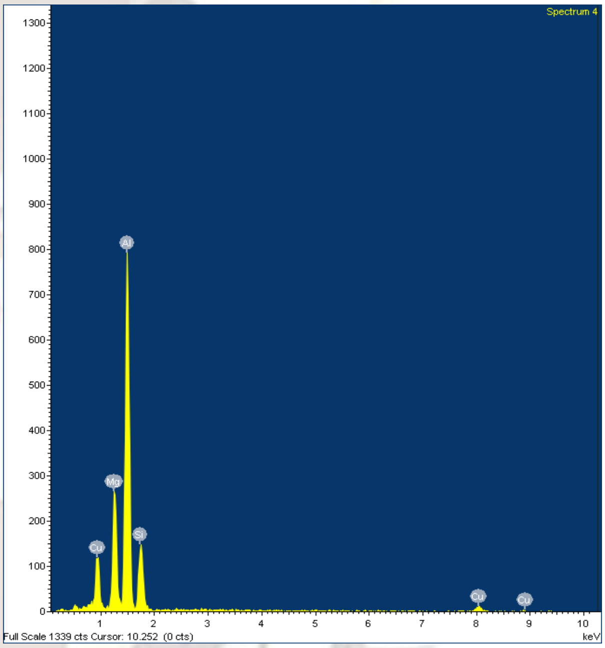

Fig 9: EDS result of wear surface of two step solution treated double aged (150^0 C/4h+210^0C /4h) Al-20Mg2Si 2Cu

Table 6

Element	Weight%
Mg K	15.34
Al K	53.79
Si K	16.04
Cu L	14.83
Totals	100.00

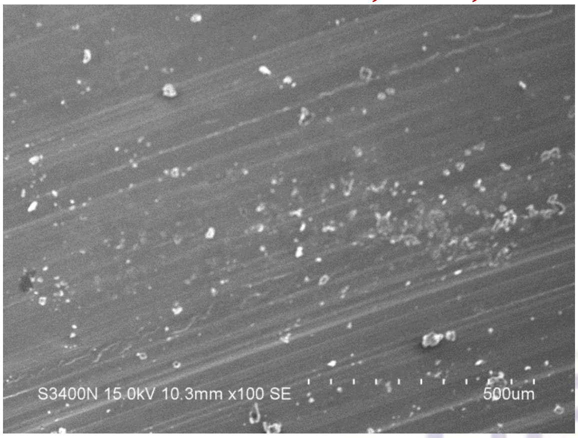

Fig10 SEM morphology of worn surface of two step solution treated Double aged (4+6) alloy at the load 68.67N

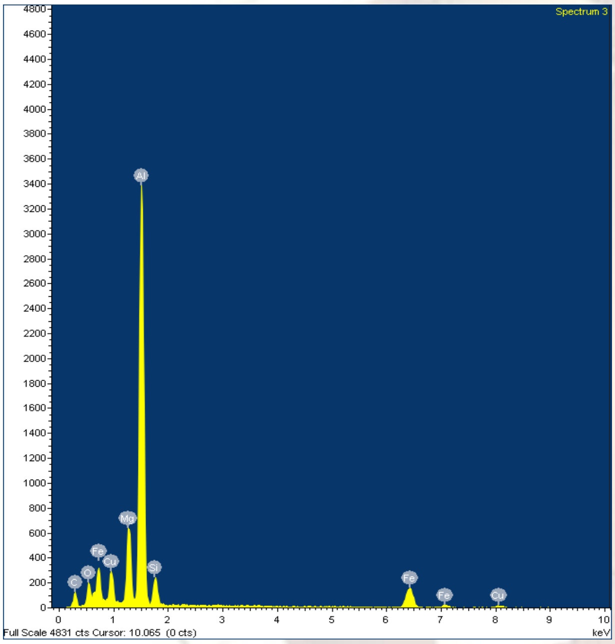

Fig 11: EDS result of wear surface of two step solution treated double aged (150^0 C/4h+210^0C /6h) Al-20Mg2Si 2Cu

Table 7

Element	Weight%
C K	13.29
O K	6.47
Mg K	7.90
Al K	42.97
Si K	3.96
Fe K	15.89
Cu L	9.52
Totals	100.00

The secondary phases in matrix or the surfaces of the worn samples were examined under SEM. Fig6 shows typical worn surface of the two step solution treated double age hardening (4+2), at the applied load of 68.67 N and sliding velocity of 2m/s. The worn surface have wider, thin grooves with few fine scoring marks. Scoring is due to the abrasion caused by the entrapped debris particle.

. With the fact that the pre-ageing at lower temperature has a strong effect on the size, distribution and density of more stable Al_2Cu precipitates during the subsequent ageing at higher temperature, and assists the formation of β' precipitates [21]. The size of the precipitates in the sample of the two step ageing treatment is smaller than that of one step ageing treatment. This kind of worn surface is typical of the abrasive wear.

Fig7 and Table5 shows that EDS result of wear surface of the above alloy and the composition of the alloy. . It reveals that the surface exhibits small amount of oxygen besides Cu Mg ,Al and Si. It indicates that an oxidative wear at high load.

Fig8 shows typical worn surface of the two step solution treated double age hardening (4+4), at the applied load of 68.67 N and sliding velocity of 2m/s.

Fig9 and Table 6 shows that EDS result of wear surface of the above alloy and the composition of the alloy.

Worn out surface is characterized by a highly smooth surface with fine grooves due to the broken fine hard particles entrapped between the counter face and the alloy may act as third-body abraders and be responsible for the fine grooves on the worn surface. After pre-aging at 150 ◦C, it can be noted that the peak hardness of the sample aged at 210 ◦C increases greatly. This is attributed to the precipitation of GP zones at 150 ◦C, which lead to a more homogeneous precipitation at 210 ◦C. The size of the precipitates in the sample of the two step double ageing treatment (4+4)is again smaller than that of two step double ageing treatment(4+2), This kind of worn surface is typical of the abrasive wear. The hard and spheroidized Mg_2Si phase, Al_2Cu precipitates resulted in a more mechanically stable structure providing an excellent wear resistance.

Fig10 shows typical worn surface of the two step solution treated double age hardening (4+6), at the applied load of 68.67 N and sliding velocity of 2m/s. The worn surface have wider thin grooves with fine scoring marks. The precipitates were observed with a wide size (coarse) and the precipitate number density decreases greatly as compared to double aging (4+4). This kind of worn surface is typical of the abrasive wear

Fig11 and Table 7 shows that EDS result of wear surface of the above alloy and the composition of the alloy, the result shows that the surface of two step double aged (4+6) alloy contain a certain amount of Fe , oxygen, Al, Mg and Si elements. It indicates that an oxidative wear and at high load, the Mg_2Si phase particles are fractured in the deformation layer. The worn surface of the alloys has a smooth appearance because of uniform and homogeneous wear mainly by plastic deformation.

4. Conclusions

It was also determined in wear tests that the wear rate is increased with increase in the normal load irrespective of the alloy processing condition and sliding speed.

It was observed that the amount of mass loss increased with increasing the load, and the amount of the maximum wear loss was determined to be in 49.05and 68.67N load.

The study also reveals that the wear rate and friction coefficient can be decreased with increasing the solution temperatures and aging periods.

The precipitation during the two step ageing process is greatly influenced by the GP I zones.

It was determined that two step double age heat treated $Al20Mg_2Si_2Cu$ alloys had higher hardness values than single step single aged alloys.

The highest hardness value of 212.6Hv is obtained when the samples were double aged ($150^0C/4h+ 210^0C/4h$). A hardness increased with the aging process, due to the formation of higher number density of CuAl2 , Mg_2Si and $CuMgAl_2$ precipitates.

The Investigation shows an excessive aging in double age at ($150^0C/4h+ 210^0C/6h$) due to coarser distribution of precipitates. Due to excessive ageing the hardness value and wear resistance is lesser.

The precipitate number density decreases greatly as compared with that aged at ($150^0C/4h+ 210^0C/4h$).

The precipitation and hardness are strongly influenced by ageing process.

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