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Influence of Copper and Nickel Addition on Microstructure and Artificial Ageing of Al-6Si-0.5Mg Alloys

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Abstract

The effects of Cu and Ni on the artificial age hardening behaviour and microstructure of Al-6Si-0.5Mg alloys were investigated. Different amounts of Cu and Ni were added to molten Al-Si-Mg master alloys. The molten alloys at 700°C were cast in metal molds. The solution treatment was performed by heating at 540°C for 2h followed by quenching in iced salt-water. The samples were aged isochronally for 60 minutes at temperatures up to 400°C and isothermally at 225°C for different periods in the range 15 to 360 minutes. Age hardening behaviour was followed by hardness measurements. Resistivity changes with annealing time at 225°C were measured to understand the precipitation behaviour of the alloys. The microstructure was examined using an optical microscope and a scanning electron microscope. Maximum hardness could be achieved by artificial ageing for 1h at 225°C. Cu was found to be more effective than nickel in increasing the hardness and addition of both Cu and Ni resulted in still higher hardness. The hardness of the matrix increased due to precipitation. The particle distribution including eutectic silicon and intermetallic compounds was found to be more homogenous after heat treatment.

***Key words:** Al-Si-Mg-alloys, Heat-treatment, Age hardening, Resistivity, Microstructure*

1. Introduction

Al-Si-Mg wrought and casting alloys have found extensive use in a variety of applications in the automotive, aerospace and defence industries due to their wide range of mechanical and physical properties [1-2]. The accelerated need for weight reduction however calls for improved properties in these alloys. Among the elements added to Al–Si base alloys for increasing strength and to control grain-size, copper has arrested considerable attention. Cu addition reduces the natural ageing rate of Al-Mg-Si alloys but generally increase the kinetics of precipitation during artificial ageing [3]. Addition of Cu to Al-Si alloys leads to the formation of Al_2Cu phases and other intermetallic compounds, which influences the strength and ductility [4,5].

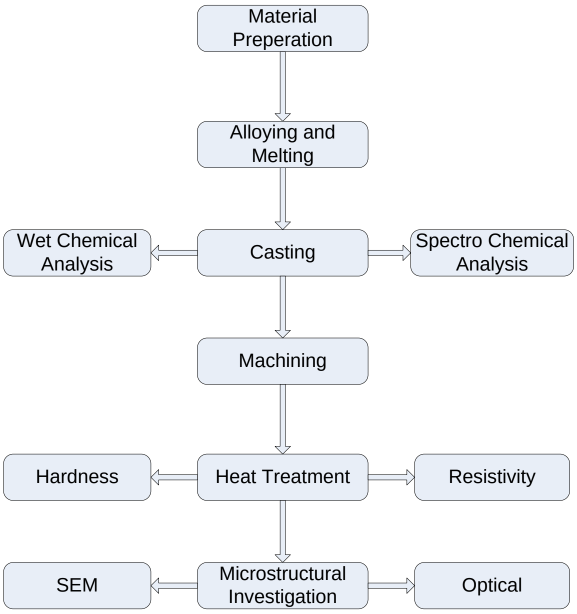
The castings are usually heat treated to obtain the desired combination of strength and ductility. The most common is the T6 heat treatment, which consist of a solution heat treatment, water quench, natural and artificial ageing. A solutionizing treatment in the range 400-560°C dissolve hardening agents in the Al matrix, homogenizes the casting, and modifies the morphology of the eutectic silicon. Castings are quenched from the solution treatment temperature to suppress the formation of intermetallic phases, retain alloying elements in solution to form a supersaturated solid solution and limit their diffusion to grain boundaries, undissolved particles or other defect locations. [6]. A report regarding the properties of the Al_3Ni system indicates that an increased amount of Al_3Ni reinforcement reduces the time of peak ageing [7].

Thus Cu and Ni could strengthen aluminium alloys through precipitation and dispersion hardening respectively. The object of this study is to investigate the individual and combined effects of Cu and Ni on the age hardening properties and microstructure of Al-Si-Mg alloys.

2. Experimental Procedures

Calculated amounts of commercially pure aluminium (99.7% pure) and Al-7.1Si-0.3Mg master alloy were melted in a gas fired clay-graphite crucible under suitable flux cover (degasser, borax etc). Magnesium ribbon (99.7% pure) was added to the molten solution. Copper, in the form of wire (99.98% purity), Ni in the form of chips were then added by plunging. The final temperature of the melt was always maintained at 950±15°C with the help of an electronic temperature controller. Before casting, the melt was homogenized by stirring at 700°C. Casting was done in cast iron metal moulds preheated to 200°C. Mould sizes were 15mm x 150mm x 300mm.

All the alloys were analysed by wet chemical and spectrochemical methods simultaneously. The chemical compositions of the alloys are given in Table 1. The cast samples were first ground properly to remove the oxide layer from the surface. All the alloys were then homogenised by heating in an electric muffle furnace for 24 hours at 500^{o}C. The homogenised samples were solution treated at 540^{o}C for 120 minutes and quenched in salt iced water solution. As-cast and quenched samples were aged isochronally for 60 minutes at different temperature up to 400^{o}C. The samples were also isothermally aged at 225^{o}C for different ageing times ranging from 15 to 360 minutes. Hardness of different alloys processed with different schedules and aged at different temperatures was measured in Rockwell hardness testing machine with 60 kg load and 1/16$^{//}$ steel ball indenter [F scale]. An average of seven consistent readings was accepted as the representative hardness value of an alloy. Electrical conductivity of the heat treated alloys was determined with an Electric Conductivity Meter, type 979. Pieces of the alloy measuring 15 mm x 20 mm x 5 mm were ground and polished prior to the electrical conductivity measurement. Electric resistivity was calculated from the conductivity data. All the samples were observed under an optical microscope. The specimens for metallographic observation were prepared using standard techniques, final polishing was done with very fine alumina. The etching was done in Keller's reagent (HNO_3 – 2.5 cc, HCl – 1.5 cc, HF – 1.0 cc and H_2O – 95.0 cc). The washed and dried samples were observed carefully in an OPTIKA Microscope (Model: B-600 MET) at different magnifications and some selected photomicrographs were taken. Scanning Electron Microscopy of the selected samples was carried out by a Scanning Electron Microscope type Inspect^{TM} S50 FEI Quanta at various magnifications.



Alloy synthesis and production of samples for aging studies

Table 1. Chemical Composition of the Experimental Alloys (wt %)

Alloy	Si	Mg	Cu	Ni	Fe	Zn	Mn	Pb	Cr	Sn	Ti	Sb	Al
1	5.902	0.461	0.007	0.005	0.146	0.004	0.002	0.003	0.001	0.004	0.099	0.008	Bal
2	5.801	0.497	1.980	0.003	0.300	0.004	0.004	0.002	0.005	0.001	0.094	0.005	Bal
3	5.965	0.454	0.007	2.202	0.141	0.006	0.003	0.002	0.005	0.001	0.088	0.008	Bal
4	5.760	0.501	1.968	2.001	0.265	0.001	0.004	0.001	0.010	0.001	0.081	0.005	Bal

Remarks:
Alloy 1: Al-6 wt% Si-0.5 wt% Mg
Alloy 2: Al-6 wt% Si-0.5% Mg-2 wt% Cu
Alloy 3: Al-6 wt% Si-0.5 wt% Mg-2 wt% Ni
Alloy 4: Al-6 wt% Si-0.5 wt% Mg-2 wt% Cu-2 wt% Ni

3. Results and Discussion

3.1. Optical and SEM micrographs:

Figures 1 to 4 show the optical micrograph of the alloys, solution treated for 120 minutes at 540^{o}C. α (Al) face centered cubic solid solution is the predominant phase in the microstructure of the alloys. The matrix of the specimens show equiaxed grain structure. During solution heat treatment primary particles Si- and Cu-containing phases: Al_2Cu and Al_5Cu2Mg8Si6 dissolve in the α -Al matrix. The subsequent aging heat treatment leads to formation from the supersaturated solid solution fine intermetallic strengthening particles of α-Mg_2Si and Al_2Cu.

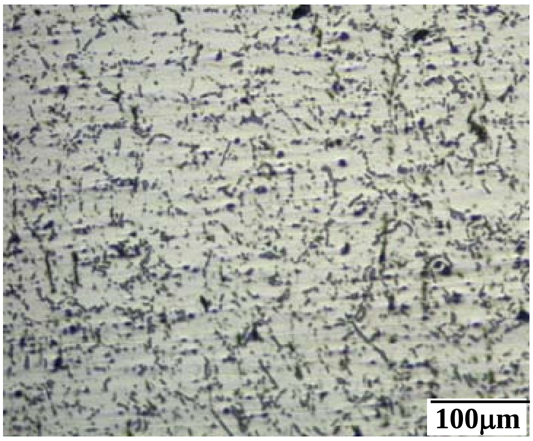


Fig. 1. Optical micrograph of solution treated alloy 1

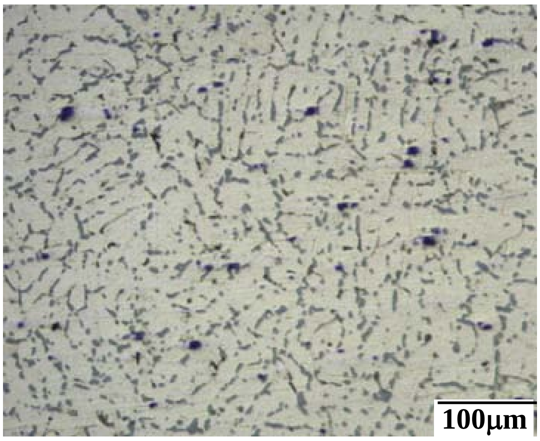


Fig. 2. Optical micrograph of solution treated alloy 2

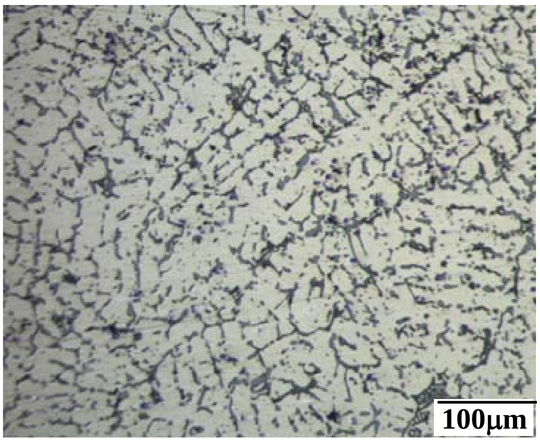


Fig. 3. Optical micrograph of solution treated alloy 3

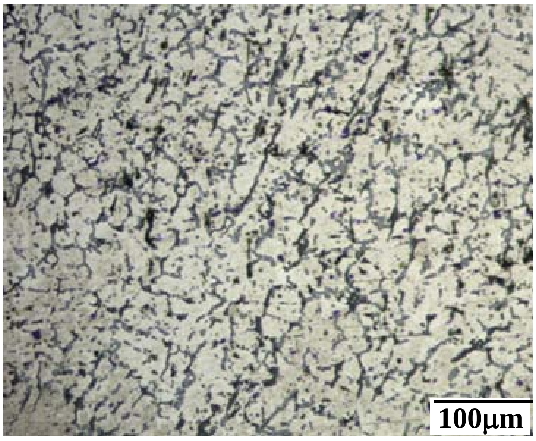


Fig. 4. Optical micrograph of solution treated alloy 4

The scanning electron micrographs of the aged samples show the presence of the intermetallic compounds (Figures 5-6). According to the chemical compositions of the samples the precipitated particles should contain Cu, Mg and Si. It can thus be inferred that the hard phases in Alloy-1 is Mg_2Si, in Alloy -2: Mg_2Si and Al_2Cu, in Alloy-3: Mg_2Si and Al_3Ni and in alloy-4 : Al_2Cu, Mg_2Si and Al_3Ni.

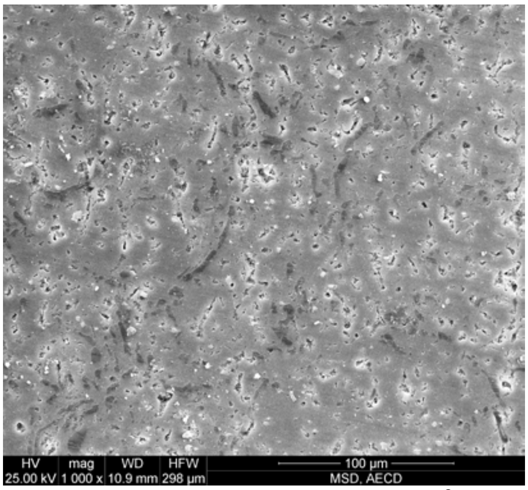


Fig. 5. SEM micrograph of alloy 1 aged at 300^{o}C for 1 hour

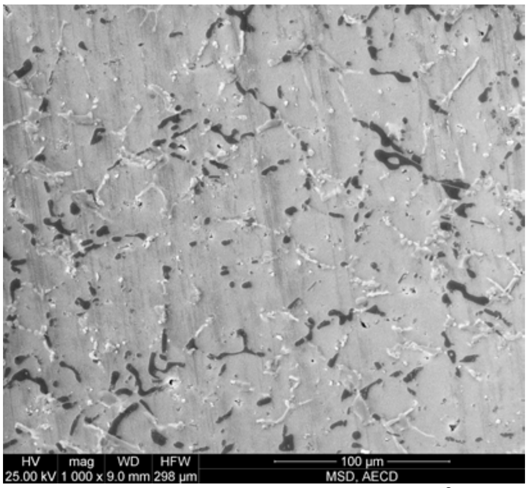


Fig. 6. SEM micrograph of alloy 4 aged at 300^{o}C for 1 hour

3.2. Isochronal Ageing:

Fig. 7 shows the plots of hardness against ageing temperature during isochronal ageing for 1h of the as-quenched samples of Al–6Si–0.5Mg with additions of Cu or of Ni or of both the elements. The addition of both Cu or of Ni to the base alloy (alloy-1) strongly increases the hardness in both the as-quenched and the peak aged conditions. Peak aged (maximum hardness) condition is achieved at 225 ^{o}C for 1h artificial ageing for all alloys. Hardness value of Alloy-2 (2% Cu) is greater than that of Alloy-3 (2% Ni). Thus the effect of Cu on precipitation hardening of the base alloy is higher than that of Ni. When both Cu and Ni has been added (Alloy-4), the hardness value is slightly higher than that of Alloy-3 (containing 2%Ni).

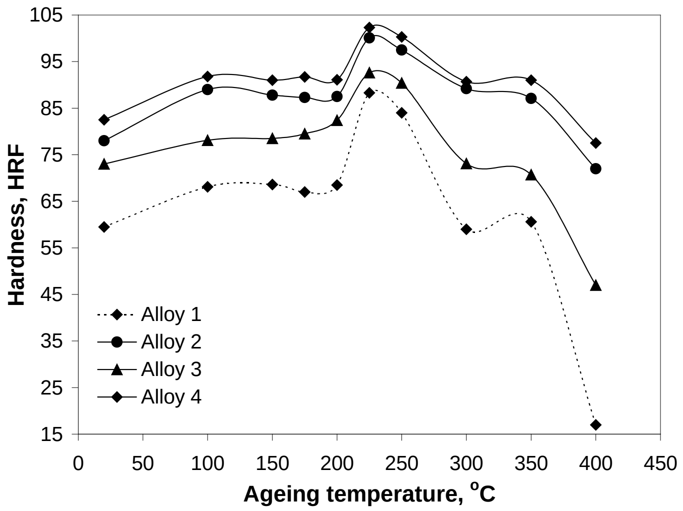


Fig. 7: Isochronal ageing curve of the alloys, aged for one hour

For Al-Si-Mg alloys, the precipitation sequence is α_{ss} ⟶clusters/GP zones⟶ β''⟶ β'⟶ β (Mg_2Si) [8]. The age hardening is caused by the precipitation of β'' and/or β' phases (precursor of Mg_2Si phases) [9]. For Al-Si-Mg-Cu alloys, the precipitation behaviors are rather complicated and several phases such as β (Mg_2Si), θ($CuAl_2$), S($CuMgAl_2$) or Q($Cu_2Mg_8Si_6Al_5$) in metastable situations may exist [10]. If Ni is added to the Al–Si–Mg alloy or Al–Si–Mg-Cu alloy systems at first the eutectic Si and Al_3Ni hard phases are formed [7]. The hard phases in the eutectic is completely continuous for the as cast and as well as for the solution treated samples and increases the hardness of the alloy.

3.3. Isothermal Ageing:

Hardness of all the alloys (Alloys 1, 2, 3 and 4) was found to vary with time of isothermal ageing at 225^{o}C (Figure 8). All the alloys, except Alloy 3, attained the peak hardness after 60 minutes of ageing. Softening due to overageing was not very pronounced in alloys containing copper. Noticeable decrease in hardness in alloys containing copper (Alloy 2 and Alloy 4) could not be detected during aging for 90 – 360 minutes. Attainment of peak hardness in Alloy 3 (containing 2% Ni) required a little more time. At the same time softening due to overageing was found to begin earlier in Alloy 3 than in alloys containing copper.

With continued ageing beyond peak aged condition hardness in age-hardenable alloys decreases due to growth of the precipitate particles. Softening in alloys containing copper could not be noticed within the time period of investigation. The hardness of the alloy containing only 2% Ni, on the other hand, showed significant decrease in hardness. It thus appears that addition of copper not only enhance the maximum attainable hardness, but also retards overageing.

The chemical compositions of the sample indicate that the precipitate particles contain Cu, Ni and Mg. Therefore the hardening was caused by the precipitation of the Al_2Cu, Al_3Ni and Mg_2Si particles. From the experimental results it can be state that artificial ageing of 60 minutes is sufficient to achieve the hardness and microstructure compared to the other ageing times.

Figure 9 shows the changes of electrical resistivity during aging at 225^{o}C in all the alloys under investigation. It also shows that the electrical resistivity in the solution treated condition depends on the chemical composition of the samples. The resistivity increases as the amount of alloying element increases. It can also be seen that the electrical resistivity of the alloys decreased rather rapidly during the initial period of ageing. The most significant decrease in the electrical resistivity occur during the first 2 hours of ageing. The electrical resistivity shows a step decrease and then an increase leading to peak or a plateau followed by a decrease until it reaches a nearly constant value.

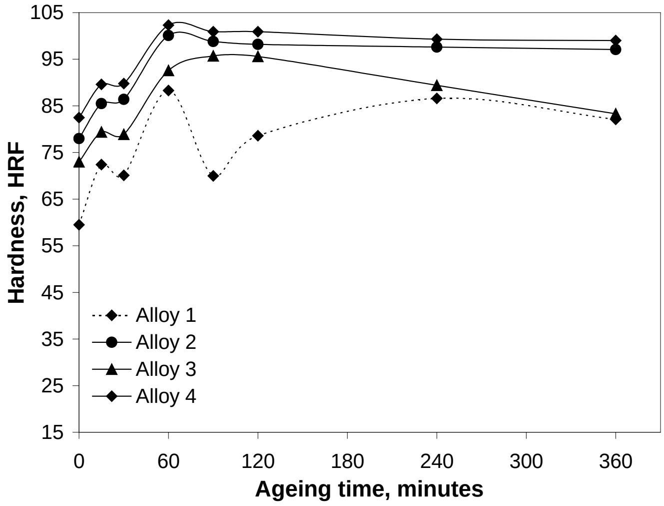


Fig. 8. Isothermal ageing curve of the alloys, aged at 225^{o}C

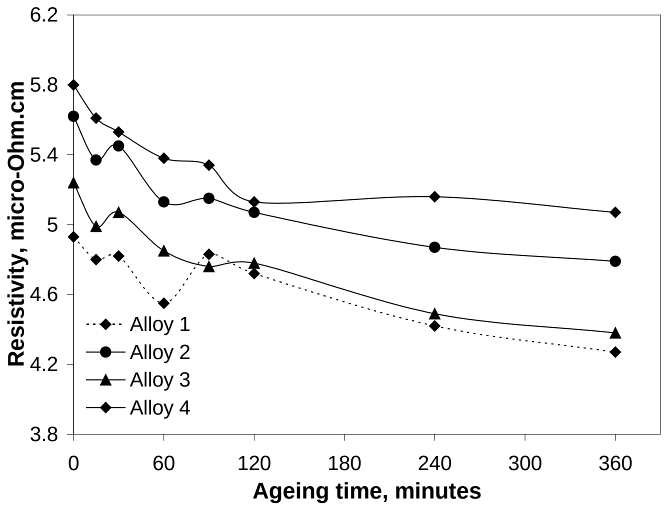


Fig. 9. Variation of resistivity of the alloys, Isothermally aged at 225^{o}C

In the presence of secondary particles, the primary effective parameters on the electrical resistivity are as follows: (1) the volume fraction of fine and coherent particles in the structure, (2) the particle interspacing, and (3) the concentration of elements in solid solution. It is well-known that the presence of alloying elements or impurities in solid solution increases the electrical resistivity of aluminum alloys. The formation of dispersoids leads to a decrease in the concentration of the corresponding elements in the matrix. If the elements precipitate out during a thermal process, the change in the electrical resistivity of the material depends on the size and the interface of the newly formed particles. Regardless of the interface of the dispersoids formed, it is expected that, if the particle interspacing is greater than the required free-passing distance of the electrons (i.e., for precipitate interspacing of 100 nm and larger in aluminum alloys), the effect of particles is negligible. The distances between the dispersoids vary depending on the extent of growth of the precipitate particles. The hardness in the copper containing samples remained more or less unchanged indicating that the size and distribution of the precipitate particles did not vary much. Thus the inter particle spacing and also the resistivity did not changed.

4. Conclusions

Maximum hardness could be achieved by artificial ageing for 1h at 225^{o}C. Cu was found to be more effective than nickel in increasing the hardness and addition of both Cu and Ni resulted in still higher hardness. The hardness of the matrix increased due to precipitation. The particle distribution including eutectic silicon and intermetallic compounds was found to be more homogenous after heat treatment.

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