Age hardening and Thermomechanical Treatment of Stircast Iron Powder Reinforced Al Alloy Metal Matrix Composites

S. S. Sharma, Jagannath K., Chandrashekhar Bhat, P. R. Prabhu

Abstract - Metal matrix composites (MMC’s) consist of a metallic matrix combined with dispersed particulate phases as reinforcement. MMC’s with uniformly dispersed iron particles in a ductile matrix may be used for automotive, aerospace and structural applications. The semi-solid stir casting method is employed for composite preparation. Al-Zn-Cu alloy matrix is precipitation hardenable and therefore suitable for age hardening treatment and thermomechanical treatment (TMT). Cold deformation increases lattice defects due to strain hardening, which in turn improves the properties of solution treated alloy. In view of this, six different Al-Zn-Cu alloy composites reinforced with Iron powder (1 wt. % to 5 wt. %) are prepared by conventional semisolid stir casting process. The solution treated specimens are conventionally and thermomechanically treated. The hardness, strength, ductility and wear resistance are analyzed and compared with Iron free Al-Zn-Cu alloys. Higher peak hardness and lesser aging time are the characteristics of thermomechanically treated samples.

Keywords - Aging, Reinforcement, Precipitation, Stircast, Thermomechanical

I. INTRODUCTION

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VER the years, aluminum alloys have been the materials of choice for both military and commercial aircraft structures. 7xxx series Al alloys are used for structural applications in the aerospace and automotive industry [1]-[2]. Al–Zn–Cu alloys present a wide range of potential applications due to their high strength and low density [3]. Heat treatment of aluminium alloy is often employed in practice in order to strengthen the alloy via precipitation hardening. The strong age-hardening response in Al–Zn–Cu alloys is in most cases associated with fine-scale precipitation of the metastable Zn- and Mg-rich intermetallic phases and its precursors [4]-[5]. The secondary hardening response is accelerated by thermomechanical treatment. The stircast Al-

Zn-Cu alloy matrix, iron powder reinforced MMC’s also respond positively for these treatments. Generally Age hardening heat treatment is often used for sand and gravity die cast components. Age hardening treatment process consists of solution treatment and artificially ageing [6]. The solution treatment is carried out at a high temperature close to the eutectic temperature. The purpose of the solution treatment is to [7] obtain supersaturated single phase Al rich solid solution at room temperature. It also homogenizes the alloying elements in the matrix. Deformation is easy and high amount of cold deformation is possible due to the FCC structure of super saturated solid solution obtained on solutionising. The solution treatment process needs to be optimized because too less solution treatment temperature will not permit to dissolve all intermetallic phases for precipitation hardening, while very high solution treatment temperature consumes of more energy than is necessary. Other important factors include the negative influence of residual Cu-containing particles on the elongation to fracture, as well as the coarsening of Al rich solid solution grains.

The dissolution and homogenization processes are faster at high temperatures and more Cu and Zn can be dissolved in the matrix. The disadvantages of high solutionising temperatures are higher thermal stresses induced during quenching and the risk of localized melting of Cu-rich and Zn-rich phases. The choice of solution treatment temperature depends on the Cu and Zn concentrations of the alloy. Generally Cu-containing phases start to melt at 519°C [8]. So solution treatment temperature is kept below 500°C. Cold deformation is often carried out between the solution treatment and the aging treatment in thermomechanical processing of precipitation hardenable alloys. In composites, the effect of deformation on precipitation kinetics has often been attributed to an increase of dislocation density, interaction of these increased dislocations with macro particles (Fe powder), which increases the nucleation sites and provides easy diffusion paths for precipitation-forming elements in the material [9]-[10], leading to accelerated precipitation process as compared with the undeformed alloy. It is clear that cold working enhances precipitation of strengthening phases, which has been put to good use for many years for achieving superior properties in various Al alloys and composites [11]-[13]. Some experimental observations have suggested that deformation can cause the redistribution of the precipitate forming elements between the dislocation cell walls and the cell interiors [14]-[15]. Thus segregation of the solute
atoms to the potential nucleation sites may also contribute to precipitation kinetics and could provide an explanation for the observed acceleration of the precipitation process [16]-[17]. This relationship between cold working and precipitation process can be advantageously used in tailoring the properties of these materials.

II. EXPERIMENTAL DETAILS

A. Alloy preparation

Six types of Al alloy composites are cast in the laboratory by stir casting technique and are solidified in the crucible itself. Pure iron powders of 300 microns are used as reinforcement. The Table 1 shows the composition of the specimens. Diffusion annealing treatment is given to all the specimens at 400°C for 18 hours to eliminate dendritic segregation. To minimize the oxidation of the alloy, the fusible salt bath of NaNO₂ and KNO₃ is used. At such a high temperature the solute clusters are eliminated and homogeneous chemical composition is obtained. First metal billets are rolled to 3mm by cold rolling with intermediate annealing treatment. Intermediate annealing treatment of 2 hours at 400°C was carried out to nullify the work hardening effect. For conventional age hardening, thickness of the final specimen is 3mm and for thermomechanical treatment initial specimen thickness is 4mm and 5mm for 25% and 40% deformation respectively. Later the thickness is reduced to 3mm by cold rolling without intermediate annealing by a number of passes with small amount of deformation per pass. The cast metal is cut into small test pieces of 30mm x 20mm x 10mm.

<table>
<thead>
<tr>
<th>Type</th>
<th>Chemical composition</th>
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<tbody>
<tr>
<td>A</td>
<td>Al–5Zn -1Cu</td>
</tr>
<tr>
<td>B</td>
<td>Al–5Zn -1Cu -1 Fe</td>
</tr>
<tr>
<td>C</td>
<td>Al–5Zn –1Cu - 2 Fe</td>
</tr>
<tr>
<td>D</td>
<td>Al–5Zn-1Cu - 3 Fe</td>
</tr>
<tr>
<td>E</td>
<td>Al–5Zn –1Cu - 4 Fe</td>
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<tr>
<td>F</td>
<td>Al–5Zn –1Cu - 5 Fe</td>
</tr>
</tbody>
</table>

B. Age hardening treatment

Specimens are first heated to 400°C in salt bath and quenched in cold water. This solution treated samples are aged at 60°C and 120°C and hardness versus aging duration graphs are plotted for all six types of alloys.

C. Thermomechanical treatment

All the specimens are cold rolled after solution treatment with 25 and 40 percentages of deformation to reduce the thickness to 3mm. At the end, rolling is performed through several passes giving only a small amount of reduction in each pass without any intermediate annealing treatment. These strained samples are aged at 60°C and 120°C.

D. Hardness measurement

All the treated and untreated specimens are subjected to Rockwell hardness test and the “B” scale hardness numbers are noted. The increase in hardness values are recorded for every one hour interval. The hardness versus aging duration graphs are plotted. The peak hardness obtained for each composition under treated and untreated specimens are also plotted.

E. Tensile test

First, tensile specimens are prepared for the test according to AISI standards. Size and shape of the specimens is shown in Fig. 1. Tension test is carried out on laboratory UTM of 100KN capacity (made by INSTRON) at cross head speed of 5mm/minute. Engg. stress versus Engg. strain graphs are plotted. The Yield strength, UTS and % elongation are found for all the specimens.

F. Wear test

The dry sliding wear behaviour is analyzed here. Before taking the reading, a trail run of one hour is provided to all the specimens to develop a perfectly flat and smooth contact surface. The experiment is conducted on pin-on-disc apparatus for 6.0 hours each on all specimens and the weight loss is noted at every 1 hour run of the specimen. Shape and size of the specimen used for wear test is shown in Fig. 2.

G. Microstructure analysis

The diffusion annealed specimens are polished and etched with Keller’s reagent [2ml HF (48% conc.), 3ml HCl (conc.),
5ml HNO₃ (conc.) and 190ml water]. Microstructures are recorded in metallurgical microscope at 200X magnification.

III. RESULTS & DISCUSSIONS

A. Hardness measurement

Fig. 3 shows the hardness versus aging duration graphs for “A“ type alloy. Similar graphs are drawn for all the other alloys and peak hardness values in different heat treatment conditions are noted. Peak hardness value increases with the increase in degree of deformation and decrease in aging temperature. In conventional aging, lower aging temperature increases the peak hardness value but with the longer aging duration. Increase in peak hardness value is due to the increase in the number of intermediate transition zones during the formation of intermetallic phases. The high peak hardness values in TMT are due to the combined effect of secondary precipitation of intermetallics and strain hardening. Fig. 4 explains the variation of peak hardness for different composites under different heat treatment conditions. As the iron percentage in the composites increases, the peak hardness value increases almost linear rate for age hardened specimens whereas for TMT specimens it increases slowly as the iron percentage increases, decreases for 3 percentage iron composites, further addition of reinforcement increases the peak hardness value. The rate of increase is higher for higher degree of deformation. It may be due to the synergetic effect of precipitation of fine metastable intermetallics with the increase in crystal defects. The difference in the distribution pattern of these peak hardness values may be the complex role of strain hardening and phase transformation process. In TMT, higher degree of deformation and lower aging temperature increases the peak hardness value to a larger extent. As the aging temperature increases, the time required for the attainment of peak hardness value decreases. Higher degree of deformation, lesser is the aging duration for the attainment of peak hardness value. An optimum number of optimum sized well distributed intermetallics in the matrix contribute a lot to the increased strength and hardness. Literature also indicates the presence of MgZn₂, Mg₃Zn₃Al₂ and CuAl₂ intermetallics in such alloy systems.

B. Tensile test

From the load versus percentage elongation graphs yield strength (YS) and ultimate tensile strength (UTS) are found out for different compositions under different heat treatment conditions. Figs. 5 & 6 show the YS and UTS variation of all the samples in treated and untreated conditions respectively. All the tensile graphs show the increase in tensile values with the increase in degree of cold deformation. This also supports the argument that fine, well distributed optimum number of particles is responsible to the increase in strength. The distribution pattern is similar to hardness distribution. Higher percentage reinforcement responds strongly to improve yield strength of the material in thermomechanically treated condition. Surprisingly, for 3% iron composite the TMT with
low temperature and 40 percentage deformation, UTS reduces approximately 10 MPa as compare to 2 percentage iron composites. This may be due to the abnormal behavior of precipitating intermetallics with discrete iron particles and increase in nucleation sites. So if the TMT is properly tailored in the Al composites with an optimum number of iron content, hardness and strength can be improved.

On the other hand, the TMT reduces the ductility considerably. Lower the degree of deformation and higher the aging temperature increases percentage elongation. The fractured surface of tensile specimen shows cup and cone type as well as pure catastrophic brittle type failures. Irrespective of the type of treatment, the increase in percentage iron in the composites reduces the ductility. Fig. 7 explains the pattern of ductility distribution in the composites with respect to the type of treatment and weight percentage iron in the matrix.

C. Wear test

Fig. 8 shows the cumulative wear versus running duration of the specimen graph for A type of alloy with different heat treatment conditions. Similar graphs are drawn for other compositions. Wear is generally the function of hardness. In all the cases, conventionally aged at 120°C specimens show least resistance to wear. Thermomechanically treated specimen with high degree of deformation and aged at lower temperature showed higher wear resistance. All the specimens contribute higher wear resistance when thermomechanically treated with high degree of deformation. During the initial period of run a severe wear mode is observed in all the composites irrespective of reinforcement density in the composites. As the running duration increases the wear rate decreases so that TMT samples show almost mild wear after 5 hours running of specimen. As the hardness increases, the wear resistance of the specimen also increases. Increase in wear resistance and the mechanical properties are due to the combined effect of dislocations with the precipitating secondary phases (intermetallics). Fig. 9 explains the total cumulative wear for 5 hours running duration with respect to type of treatment and weight % of iron in the composites. It is also seen that the weight percentage of iron greater than 3% in the composites will not contribute to increase the wear resistance if it is thermomechanically treated. On the other hand during longer running duration, the conventionally aged samples show higher wear resistance with the increase in weight percentage iron in the composites.

D. Microstructure analysis

Figs. 10 and 11 show the microstructure of homogenized B and D type composites at 200X magnification. Homogenized microstructures show equiaxial grains without any dendritic segregation. Iron particles are seen as discrete phases in the matrix.
Fig. 11 Microstructure of homogenized D type composite at 200X.

IV. CONCLUSIONS

- Compared to conventionally aged, thermomechanically treated specimens show higher peak hardness values with decreased peak aging duration.
- Lower aging temperature increases the peak hardness values with increased peak aging duration.
- In TMT, as the degree of cold deformation increases or aging temperature decreases, the peak hardness values increase.
- Tensile characteristics also improve with the increase in weight percentage of reinforcement in the composite.
- Yield strength and ultimate tensile strength are higher for thermomechanically treated specimens.
- Higher the degree of deformation and lower the aging temperature, greater is the tensile property.
- As the weight percentage of iron in the composite increases, the hardness and strength increase.
- In conventionally aged samples, higher the iron content in the matrix and lower the aging temperature, better is the wear resistance.
- Mild wear is observed for TMT composites after a considerable longer running duration.
- Thermomechanical treatment improves hardness and strength if treatment cycle is properly designed.
- Ductility reduction is drastic for TMT specimens of high iron content if it is aged at lower temperature.
- Homogenizing treatment is required to eliminate dendritic segregation.

REFERENCES