Tensile and Fracture Behavior of Wrought Al-6Mg Alloy with Ternary Scandium and Quaternary Zirconium and Titanium Addition

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Abstract: Effect of ageing on the mechanical properties of wrought Al-6Mg alloy with ternary scandium and quaternary zirconium and titanium is made. The results show that the influence of scandium is much pronounced on yield strength than on the tensile strength. The quaternary additions have negligible effect on the yield strength of the alloys. Elongation increases because of the fact that subgrains cannot coarsen so much. Strain hardening exponent 'n' values are found to comparatively low at higher ageing temperature. The room temperature values of strain rate sensitivity 'm' are comparatively high and hints upon its superplastic behaviour at elevated temperatures. The fracture of the experimental alloys occurs through microvoid coalescence.

Key words: Age hardening, precipitates, strain hardening exponent, strain rate sensitivity, microvoid.

1. Introduction

Aluminum alloys with magnesium as the major alloying element constitute a group of non heat-treatable alloys with medium strength. Unfortunately, the strength of such Al–Mg system alloys is lower than precipitation-hardening Al alloys. Aluminum alloys containing minor scandium is a new group of structure materials, which has low density, high strength, good weldability, high ductility and excellent corrosion resistance [1]. It is mainly used in aerospace, nuclear energy and ships industry. The simultaneous addition of scandium, Zirconium or Titanium elements is a popular trend for the development of aluminium magnesium alloys. Extensive research has been conducted on Al alloys containing scandium [2]-[6], but research on Al-Mg based alloys containing Sc, Zr or Ti are not common. The addition of a small amount of scandium to Al-Mg alloys causes a significant increase in the strength of the alloys. The reason is the existence of coherent and the fine LI_2 phase, Al_3Sc precipitates dispersed in the alloy matrix [2]. Finely dispersed Al_3Sc precipitates that can be obtained at a high number density, thus preventing the dislocation motion [7], [8]. Magnesium aluminide does not enter the precipitate structure and hence the strengthening effect of Al_3Sc is additive to the solid solution strengthening due to magnesium. Reportedly Al_3Sc is the most potent strengthener on equal atomic fraction basis [9]. These precipitates are also effective on stabilising substructure, thus allowing the use of strain hardening and stabilisation treatments to improve the strength properties quite considerably.

So efforts are made to determine if alloys of this type could be made competitive with other low-density systems for high performance applications. Zirconium and titanium also form their aluminides and are known to influence the precipitation behaviour of Al_3Sc in Al-Mg alloys [2]. Therefore it is thought to be interesting to study the achievable properties of Al-Mg-0.4 wt% Sc alloy with quaternary additions of zirconium and titanium. The ability of Al_3Sc precipitates to stabilise substructure envisages the use of strain hardening for enhancement of mechanical properties of the alloy. Superplasticity in metals and alloys are characterised by strain rate sensitivity values. The variation of strain rate sensitivity of the experimental alloys after thermal and mechanical treatments are also studied to get an idea of the possible deformation mode, operative in the experimental alloys.

2. Experimental

Melting was carried out in a resistance heating pot furnace under the suitable flux cover (degasser, borax etc.). Several heats were taken for developing base Aluminium-Magnesium alloy, Aluminium-Magnesium alloy containing 0.4 wt% scandium and with or without zirconium and titanium. In the process of preparation of the alloys the commercially pure aluminium (99.5% purity) was taken as the starting material. First the aluminium and aluminium-scandium master alloy (2%Sc) were melted in a clay-graphite crucible, then magnesium ribbon (99.7% purity) was added into solution. Zirconium and Titanium were taken in the form of powder (99.98% purity) with in a cover of aluminium foil and were then added by plunging. The final temperature of the melt was always maintained at 780±15°C with the help of the electronic controller. Casting was done in cast iron metal moulds preheated to 200°C. Mould sizes were 12.5 x 51.0 x 200.0 in millimetre. All the alloys were analysed by wet chemical and spectrochemical methods simultaneously. The chemical compositions of the alloys are given in Table 1. Cold rolling of the alloys in as cast state was carried out in a laboratory scale rolling mill of 10HP capacity at 75% reduction percentages. The sample sizes were 9 x 12 x 50 mm and the deformation given was about 1.25 mm per pass. Cold rolled samples were aged isochronally for 60 minutes at different temperature 25, 100, 200, 300, 400 and 500°C. Tensile testing was carried out in an Instron testing machine using different cross head speed to maintain the strain rate of 10^{-2}/s, 10^{-3}/s and 10^{-4}/s. The samples used were according to ASTM specification. Fractographic observations of the surfaces fractured by tensile testing were also carried out in a Jeol Scanning Electron Microscope. TEM studies of the alloy was carried out in Philips (CM12) Transmission Electron Microscope at an accelerating voltage 160 KV. TEM foils parallel to the sheet plane were taken at the sample's mid-thickness location. The samples were prepared by mechanical polishing of both sides and electro-polished using a double jet thinner with dilute solution of HNO_3 and methanol at −10°C under 10 V, until perforation occurred. The observations were carried out with sample tilts wherever needed. Both microstructures and selected area diffraction (SAD) patterns were studied.

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

Alloy	Mg	Sc	Zr	Ti	Cu	Fe	Mn	Ni	Si	Zn	Cr	Sn	Al
1	6.10	0.000	0.000	0.001	0.081	0.382	0.155	0.003	0.380	0.136	0.002	0.002	Bal
2	5.97	0.400	0.000	0.002	0.071	0.314	0.107	0.002	0.335	0.124	0.002	0.002	Bal
3	5.85	0.400	0.185	0.003	0.069	0.335	0.112	0.001	0.345	0.170	0.003	0.002	Bal
4	6.06	0.400	0.000	0.175	0.080	0.306	0.104	0.002	0.335	0.170	0.002	0.002	Bal

Remarks:

Alloy 1 Al-6 wt% Mg Alloy 2 Al-6 wt% Mg-0.4 wt% Sc

Alloy 3 Al-6 wt% Mg-0.4 wt% Sc-0.2 wt% Zr Alloy 4 Al-6 wt% Mg-0.4 wt% Sc-0.2 wt% Ti

3. Results

3.1. Isochronal Ageing

The results of isochronal ageing of the cold worked alloys at different temperature for 1 hour are shown in Fig. 1. The base alloy (alloy 1) shows a continuous softening due to recovery and recrystallisation of the strained grains. All other alloys demonstrate age hardening response with peak hardness value at 300^{o}C. An initial softening to the tune of 5 VHN is noted in almost all the alloys. The extent of age hardening varies with the composition of the alloy; however alloy 2 shows maximum hardness. Most of the alloys show softening during initial period of ageing and increase in hardness after ageing finally enables the maximum hardness to reach a magnitude which is comparable to the initial hardness of cold worked alloys (Fig. 1). When the alloys are aged at higher temperature a sharp decrease in hardness is observed for all the alloys. Thus appreciable drop in hardness values are noted at ageing temperatures beyond 300^{o}C.

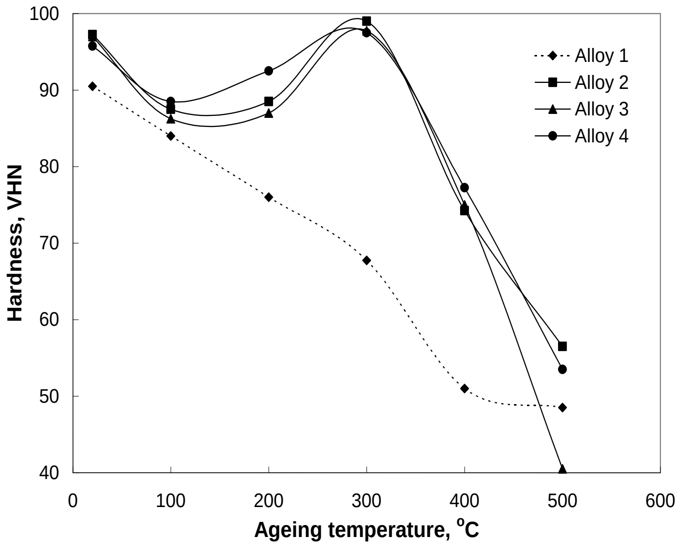

Fig. 1. Isochronal ageing curve of 75% cold rolled alloys. Aged for 1 hour.

3.2. Tensile

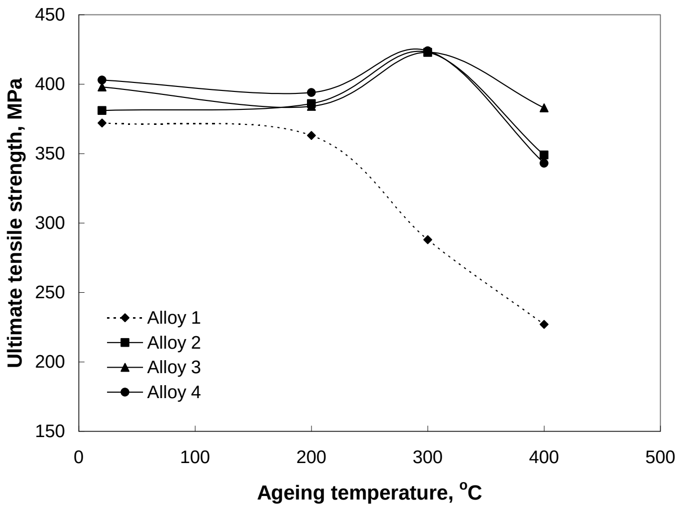

Fig. 2. Variation of ultimate tensile strength (10^{-3} s^{-1}) with ageing temperature of 75% cold rolled alloys isochronally aged for 1 hour.

When the cast alloys are cold rolled and aged, it is found that both the tensile and yield strength of the base alloy decrease with increasing ageing temperature (Figs. 2-3). After the onset of ageing of alloys 2-4 the tensile strength increases slowly and then reaches a peak at 300°C. A rise in tensile strength by about 40-50 MPa is noted due to ageing (Fig. 2). On the other hand the cold worked alloys do not practically exhibit any appreciable improvement in yield strength upon ageing (Fig. 3). The ductility values however are found to increase quite considerably upon ageing the cold worked alloys. An elongation value of about 16% is obtained in alloy 2 after ageing at 400°C. A benefit in elongation by about 4% is accrued if ageing temperature is increased to 400°C from the peak ageing temperature, 300°C (Fig. 4). In this process, there is however a significant sacrifice in strength properties. No significant improvement in the strength properties of the alloys with quaternary addition could be observed in the cold worked and aged condition. These quaternary alloys however show improved ductility at higher ageing temperature. The ductility value of titanium alloy reaches as high as 18% at an ageing temperature of 400°C.

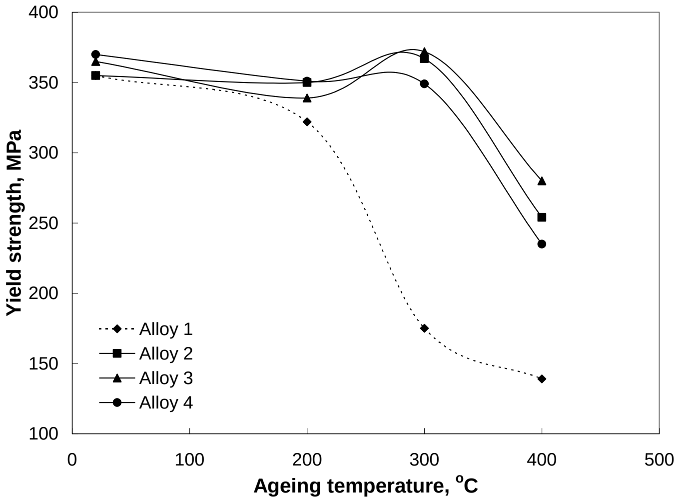

Fig. 3. Variation of yield (10^{-3} s^{-1}) with ageing temperature of 75% cold rolled alloys isochronally aged for 1 hour.

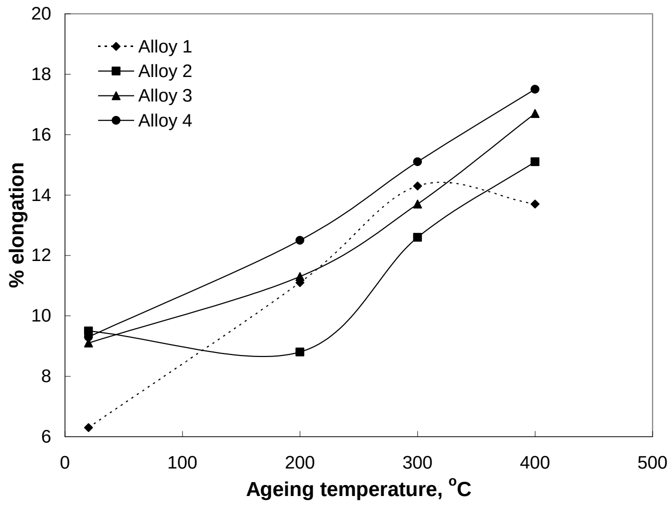

Fig. 4. Variation of percent elongation (10^{-3} s^{-1}) with ageing temperature of 75% cold rolled alloys isochronally aged for 1 hour.

When the alloys are cold worked and aged at 200°C (Fig. 5) the strain hardening exponent values of the experimental alloys at $10^{-3}s^{-1}$are found to become lower (0.25-0.29). That the 'n' values of all the alloys lay within a narrow range implies that it is relatively independent of composition for an ageing temperature of 200°C. However after ageing the cold rolled alloys at 300°C (Fig. 6) the range of 'n' values is expanded. The strain-hardening exponent of the cold rolled alloy at the lowest strain rate (~ $10^{-4}s^{-1}$) is seen to increase with increasing ageing temperature. For scandium bearing alloys (alloys 2-4) 'n' values decreases with increase in ageing temperature up to 300°C and then it increases after ageing at 400°C (Fig. 7). The cold rolled alloys with minor additions show the least set of 'n' values after being aged at 300°C. The strain rate sensitivity 'm' values are found to be lowered and for various conditions of thermal and mechanical treatment its values lie within the range 0.02-0.036. The trend of variation of 'm' values with the ageing temperatures of cold rolled alloys is shown in Fig. 8. It is apparent from Fig. 8 that for all the alloys except base alloy, the strain rate sensitivity increases with the ageing temperature. For base alloy, 'm' value is seen to decrease with increase in ageing temperature. Moreover, for quaternary alloys 3 and 4 the 'm' values increase rather steeply beyond an ageing temperature of 300°C and a maximum 'm' value in alloy 2 is obtained at ageing temperature, 400°C (~0.036).

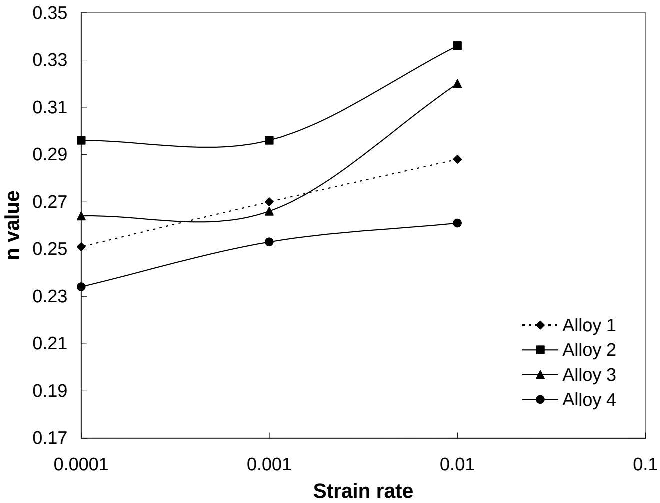

Fig. 5. Variation of strain hardening exponent with strain rate of testing of 75% cold rolled alloys aged at 200°C.

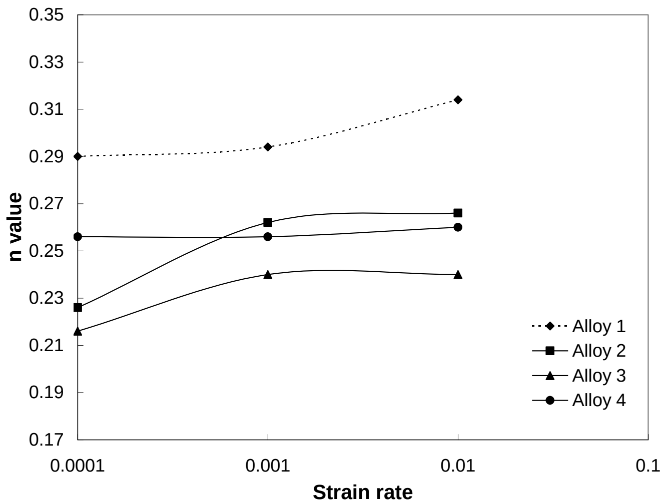

Fig. 6. Variation of strain hardening exponent with strain rate of testing of 75% cold rolled alloys aged at 300°C.

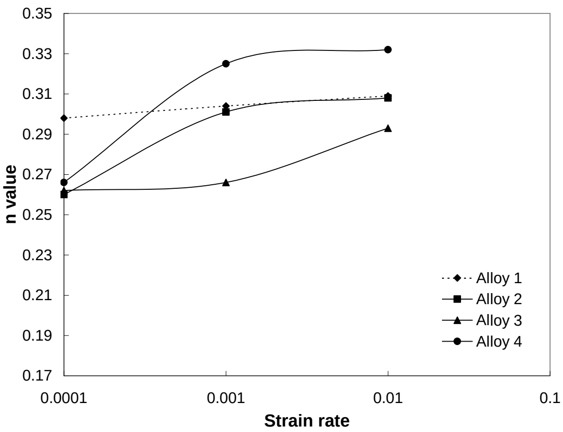

Fig. 7. Variation of strain hardening exponent with strain rate of testing of 75% cold rolled alloys aged at 400°C.

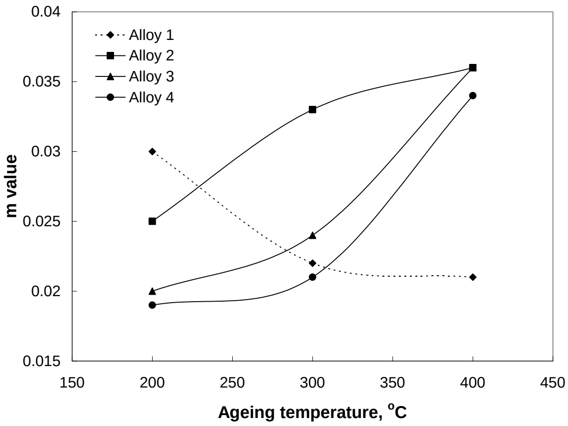

Fig. 8. Variation of strain rate sensitivity with ageing temperature of cold rolled alloys.

3.3. Fractography

The fracture mode of cold worked base alloy after annealing at 300°C seems to be cracks propagating along the grain boundaries (Fig. 9). When the alloys are cold rolled, with 75% deformation, at different strain rate the fracture is seen to be transgranular shear type (Figs. 10-11). In some cases delamination tearing is also visible. The fracture mode of cold worked base alloy after annealing at 400°C seems to be due to cracks propagating along the grain boundaries. When the quaternary addition alloy 3 is cold worked prior to annealing at 300°C the fracture surface shows elongated dimples as well as shearing ridges (Fig. 12). Similar features are visible in the fractured surface of the other alloys with quaternary addition alloy 4, in cold worked and annealed condition (Figs. 12 and 13).

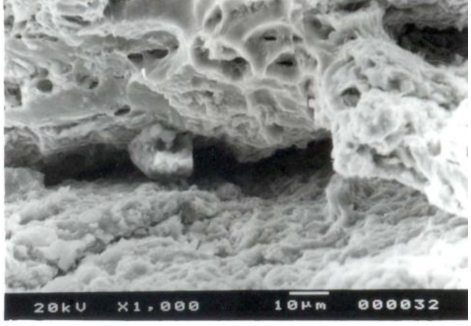

Fig. 9. SEM fractograph of 75% cold deformed alloy 1, aged at 300°C for 1 hour and tensile tested at strain rate of $10^{-4}s^{-1}$.

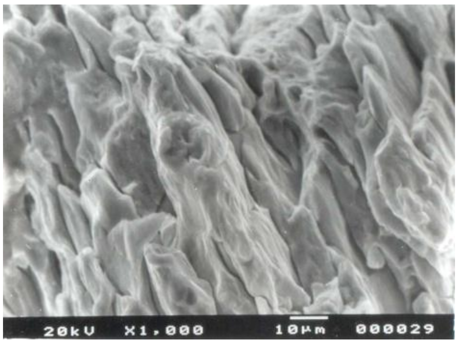

Fig. 10. SEM fractograph of 75% cold deformed alloy 2, aged at 300^{o}C for 1 hour and tensile tested at strain rate of $10^{-4}s^{-1}$.

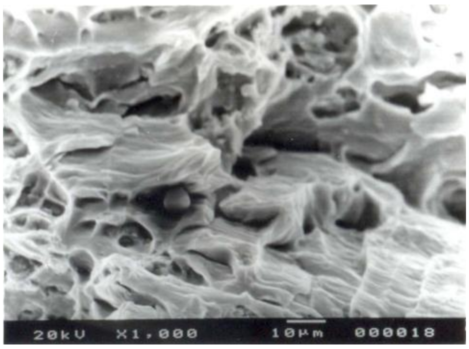

Fig. 11. SEM fractograph of 75% cold deformed alloy 2, aged at 400^{o}C for 1 hour and tensile tested at strain rate of $10^{-2}s^{-1}$.

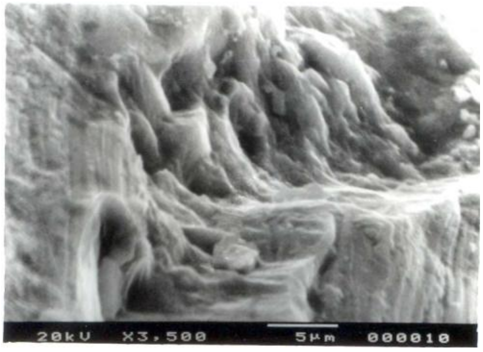

Fig. 12. SEM fractograph of 75% cold deformed alloy 3, aged at 300^{o}C for 1 hour and tensile tested at strain rate of $10^{-2}s^{-1}$.

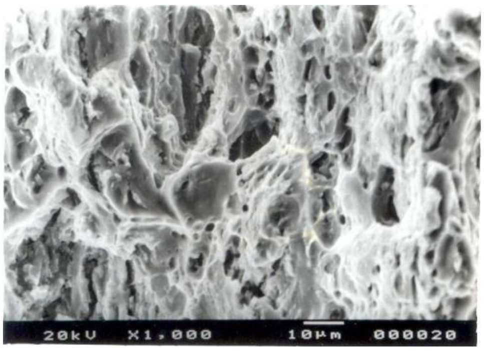

Fig. 13. SEM fractograph of 75% cold deformed alloy 4, aged at 300^{o}C for 1 hour and tensile tested at strain rate of $10^{-2}s^{-1}$.

3.4. Transmission Electron Microscopy

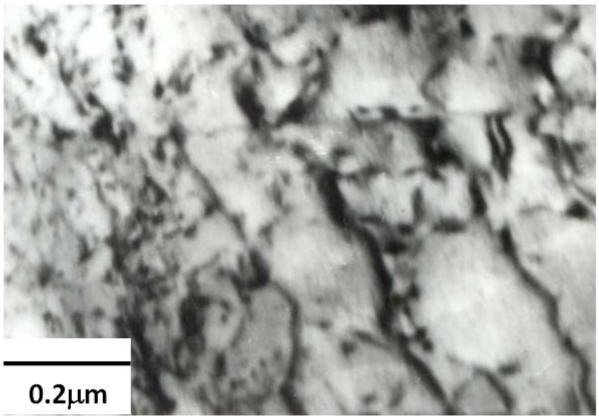

Fig. 14. TEM micrograph of alloy 3 aged at 300°C for 1 hour.

TEM image of the same alloy at a higher magnification shows irregular boundaries with high amount of precipitates (Fig. 14). Fig. 15 shows the SAD pattern of the precipitates as well as of the matrix.

Fig. 15. SAD pattern of the matrix and the precipitates of alloy 3 aged at 300°C for 1 hour.

4. Discussion

The initial softening of the cold worked alloys during isochronal ageing is thought be due to rearrangement of dislocations at the ageing temperature. The age hardening of the alloys containing scandium is attributable to the formation of Al_3Sc precipitates [10]. The strengthening is found to be greater for alloys with higher deformation because a higher degree of strain hardening resulted from higher dislocation density. But the extent of age hardening has not improved [7]. This means that extra advantage is not accruable by working scandium treated alloys. Moreover there has not been any change in the peak hardness temperature due to cold working. This signifies that scandium precipitation is not dislocation induced. However peak temperature in zirconium bearing alloy is responsive to cold working. It is conjectured that Al_3Zr is formed at dislocations. The Al_3Zr being isomorphous with and soluble in Al_3Sc, the nucleation of Al_3Sc is facilitated indirectly by the presence of higher dislocation density. Moreover extensive cold working also generates large number of vacancies, which form vacancy-scandium atom complexes of high binding energy. The vacancy-solute atom complexes reduce the mobility and availability of solute atoms at low temperature to form G P zones. Hence hardening takes place only at a temperature high enough to decompose the complexes thereby making solute scandium atoms available for precipitate formation. Beyond peak hardness, over ageing effect due to coarsening of the precipitates is seen to have taken place. At higher ageing temperature there is ample scope for dislocation annihilation and this softens the material. In respect of resisting softening due to overageing titanium is found to be more effective than zirconium.

The improvement of tensile strength by about 45-50 MPa is explained by the fact that precipitation strengthening takes place due to the formation of ageing precipitates Al_3Sc over and above the usual dislocation hardening due to cold rolling. This is partially true in the present case, as the alloys aged up to 400°C do not practically undergo any recrystallisation. The insignificant benefit in yield strength due to ageing is to be explained by taking several points in consideration. Just after cold rolling fine subgrains are formed. These describe the effective grain size, which determines the yield strength values in terms of Hall-Petch equation. On ageing, certain degree of recovery reduces the yield strength due to increase in effective grain size. Then the precipitation sets in. This tends to increase the yield strength. Thus a hump is observed in the ageing curve. But advantage of dislocation cutting is compensated by increase in subgrain size at higher ageing temperature. It appears that these two opposing factors have fixed the peak aged yield strength at a value either close to or marginally lower than the YS values obtained just after cold rolling. Elongation however increases because of the fact that subgrains cannot coarsen so much on appearance of Al_3Sc precipitates because the sub-boundaries are pinned by the fine precipitates. At the ageing temperatures higher than 300°C, precipitate coarsening contributes to the ductility enhancement and this is why no drop in ductility is recorded for alloy 2, which are capable to produce a good amount of precipitates. In the alloys with quaternary additions no extra advantage is obtained because so far as strengthening is concerned, the aluminides of zirconium or titanium do not have much influence since these are associated with Al_3Sc. But the fact that the precipitate size in the alloys is large enough for bypassing mechanism to be operative and also higher degree of precipitate coarsening are seemingly responsible for ductility enhancement in quaternary alloys which are seen to record higher percentage elongation than the ternary alloys under identical ageing situations.

The strain hardening exponent of the cold worked alloys are found to be less even when ageing is done after cold rolling. This observation corroborates the earlier finding [11] and is ascribed to the hardening already experienced in the cold rolled alloys which do not undergo recovery process even after ageing at 400°C. Thus the highly dislocated materials are not capable to sustain the levels of hardening demonstrated by the non-worked material. The decrease in ‘n’ value of the cold rolled scandium alloys due to ageing up to 300°C is supportive of the above conjecture. Ageing at 300°C maximizes the precipitate density and therefore precipitation hardening becomes maximum as already demonstrated by the ageing curves. Thus further hardening to a large extent due to straining is improbable. When the alloys are aged, partial recovery and precipitate coarsening take place. These create extra provision for dislocation-dislocation interaction during straining. This is reflected by an increase in the ‘n’ values of the scandium alloys aged at 400°C. Velocity of mobile dislocations is statedly linearly proportional to strain rate. Again, velocity of dislocations is strongly dependent on flow stress. It may be deduced that for a specific strain increment an increase in the velocity of mobile dislocations would lead to an increase in ‘n’ values as it increases the flow stress. Now increasing strain rate leads to an increase in the velocity of dislocations. This is why the ‘n’ values of the experimental alloys are seen to increase with increasing strain rate of testing. However for alloys 3 and 4 the microstructure after ageing at 300°C contains maximum amount of fine precipitates of Al_3Sc. This precipitate formation is not dislocation induced. Hence during tensile straining of materials with this type of microstructures, dislocation motion is greatly inhibited by the precipitate particles. Thus within the range of variation in strain rate of testing, the velocity of mobile dislocations cannot appreciably increase due to the inhibitions stated above. Therefore 'n' value remains essentially independent of strain rate of testing (Fig. 6). The cold worked alloys exhibit 'm' values lower than the non-worked alloys. At room temperature although grain size is fine enough, so far as the deformation behaviour is concerned grains are primarily the subgrains. However the sub-boundaries are generally immobile with respect to grain boundary sliding. As a result deformation is primarily controlled by dislocation slide through the lattice. Because the sub-boundaries are easily pinned by the dislocation aided precipitates formed in the alloys 3 and 4 they are made further immobile as corroborated by appreciably lower 'm' values in alloys 3-4 than the base alloy. However there is a general trend of increase in 'm' value with the temperature of ageing for the cold rolled alloys 2-4. This effect is more pronounced in alloys 3 and 4. The increasing 'm' values of the cold rolled alloys with ageing temperatures are consistent with the corresponding enhancement of tensile ductility as evidence in Fig. 3. Again the highest attainable 'm' value in alloy 4 aged at 400oC corresponds to the highest value of tensile ductility achieved in the present set of experiments. This is presumed to be due to the advent of power law break down as enumerated earlier [6]. The fracture mode of cold worked base alloy after annealing at 300^{o}C seems to be due to cracks propagating along the grain boundaries. Alloy 1 contains high amount second phase constituents at the grain boundaries. These have initiated cracks and decohesion along the grain boundaries has led to the failure (Fig. 9). When alloy 2 is cold worked and annealed at 300^{o}C the fracture of tensile specimens at slower strain rate ($10^{-4}s^{-1}$) is found to be transgranular shear type. It is due to the fact that the grains are unrecrystallised and have the propensity for planer slip. Such delamination tearing is noticed in Fig. 10. When annealing is done at 400^{o}C, the fractures at different strain rates are shown in Fig. 11. A mixed mode of fracture is in general noticed. Big voids due coalescence of microvoids are noticed along with longitudinal shear. The ageing precipitates have assumed large size after annealing at higher temperatures. These precipitates might have helped to reduce planarity and promoted microvoid coalescence. On the contrary when the alloy 3 is cold worked prior to annealing at 300^{o}C the fracture mode becomes a mixed type. While the elongated dimples are observed in Fig. 12, the presence of shearing ridges is indicative of overload failure through longitudinal delamination tearing. When the cold worked alloy 4 (Fig. 13) is annealed, the tensile sample is found to fracture by both transgranular shear and microvoid coalescence. With the above kind of processing this alloy remains partially recrystallised with the presence of fine precipitates Al_3Sc and Al_3Ti in microstructure. The presence of longitudinal shear ridges in its fractograph accounts for transgranular shear arisen out of planer slip. However precipitates have been able to reduce planarity of slip in the recovered grains. As a result ductile dimples are also formed [9].

The bright field image of alloy 3 at a higher resolution shows irregular boundaries with a huge amount of intragranular precipitates. The deformation contrast around the precipitates is indicative of coherency of the precipitates. The solidification strain has resulted into dislocations. A large number of precipitates are seen to have been formed at the dislocations as the strain field near them assist nucleation by reducing strain energy required to form a nucleus. However the dislocation induced precipitation in alloy 3 is to be explained on the basis of the role played by zirconium in the process of nucleation of precipitates. Al3Zr and Al_3Sc are both known to be of LI_2 crystal structure. They are known to be mutually soluble in each other and are isomorphous [4]. Thus it is conjectured that Al_3Zr is formed first at dislocations in alloy 3 as it is already reported earlier that Al_3Zr precipitation is facilitated at dislocations [6]. In order to decrease nucleation energy Al_3Zr particles are formed at dislocations already available in the alloy. These particles induce the formation of Al_3Sc which gets dissolved thereby to form precipitates of the type $Al_3(Zr_xSc_{1-x})$. Whether the process of nucleation is insitu or not remains unresolved at this stage. The precipitates are uniformly distributed within a size range of 15-20 nm with an average interparticle distance of around 30 nm. The larger size of these precipitates seemingly indicates that there is separate formation of Al_3Zr first and subsequently the formation of Al_3Sc and its dissolution into Al_3Zr leads to the formation of $Al_3(Zr_xSc_{1-x})$; the chance of direct nucleation of $Al_3(Zr_xSc_{1-x})$ cannot be concluded from the results of the present experiments. The SADP (Fig. 15) analysis shows that this cubic phase of LI_2 structure bears a perfect orientation relationship with the matrix which is found to be (100)m||(100)p. This observation corroborates with the existing knowledge [12] about the structure of Al_3Sc and $Al_3(Zr_xSc_{1-x})$.

5. Conclusion

Improvement in strength during ageing is due to the formation of Al_3Sc precipitates. The improvement in yield strength of the alloys due to ageing is more than that for ultimate strength. The quaternary additions have negligible effect on the yield strength of the alloys. Elongation however increases because of the fact that subgrains cannot coarsen so much. ‘n’ values are found to comparatively low at higher ageing temperature. The room temperature values of ‘m’ are comparatively high and hints upon its superplastic behaviour at elevated temperatures. The fracture mechanism of the cold worked alloys a mixed mode of fracture with transgranular shearing as well as void coalescence is visible.

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