CO-EXTRUDED Al4.5Mg AND RAPIDLY SOLIDIFIED Al4.5Mg1Ag – STRUCTURE AND PROPERTIES

H. Kalager[1, a], K. G. Skorpen[1], C. J. Simensen[2], *X. Ma[2], R. Østhus[3], and *H. J. Roven[1]

[1]*Department of Materials Science and Engineering, Norwegian University of Science and Technology, 7491 Trondheim, Norway*
(*hans.j.roven@ntnu.no)

[2]*SINTEF Materials and Chemistry, P.O.Box 124, Blindern, 0314 Oslo, Norway*
(*xiang.ma@sintef.no)

[3]*SINTEF Raufoss Manufacturing AS, P.O.Box 163, Raufoss, 2831 Raufoss, Norway*

ABSTRACT

This work encompasses co-extrusion of alloy Al4.5Mg at the core and rapidly solidified Al4.5Mg1Ag forming the surface layer. The goal is to produce a multi-material by co-extrusion having a relatively hard surface as compared to a softer core. At the same time, work hardening in both alloys is expected to be significant due to Mg in solid solution. Precipitation hardening at 160°C is studied by applying solution heat treatments at 560°C. The hardness increased from 80 to 130 VH in the surface material after 236 h. Surprisingly, the Al4.5Mg alloy also shows pronounced aging at this temperature, i.e. from 70 to 90 VH. The relatively high solution heat treatment temperature introduces grain growth in the core, but this is insignificant for the RS surface alloy. Blisters forms at the surface and cracks follows large portions of the interface. However, the tensile ductility is significant in all conditions due to very pronounced work hardening, i.e. for the as co-extruded, T6, under- and over-aged. The elongation at fracture ranges between 40–45% and the tensile strengs 240–275 MPa. The tensile properties increase linearly with the thickness of the RS alloy, i.e. following the rule of mixtures.

KEYWORDS

Aluminium magnesium silver alloy, Rapid solidification, Co-extrusion, Heat treatment, Fracture

INTRODUCTION

Al-Mg alloys in 5xxx-series are classified as non-heat treatable wrought alloys (Polmear, StJohn, Nie, & Ma, 2017). The formation of the binary β-Al$_8$Mg$_5$ does not give a substantial increase in the strength of the alloys (Mondolfo, 1976). When a third element like Ag, Cu or Zn are added, G.P.–zones are formed at low temperatures and intermediate, partial coherent phases are nucleated somewhat higher temperatures and finally a ternary, equilibrium phase is formed (Auld, 1968; Cousland & Tate, 1986; Mondolfo, 1976). Both the formation of G.P.–zones and intermediate phases increase the hardness and strength of the materials. An intermediate, hexagonal T´–phase is formed in Al-5 wt%Mg-1wt%Ag, and finally the equilibrium, cubic phase (Al,Ag)$_9$Mg$_5$ is nucleated (Cousland & Tate, 1986; Mondolfo, 1976).

Al-Mg alloys are widely used in engineering due to its low weight compared to steel and good corrosion properties. The conventional process route is direct chilled (DC) casting followed by homogenizing, forming and occasionally (depending of the alloy composition), followed by precipitation hardening or a softening anneal. This process has its limitation related to segregation of alloying elements in highly alloyed materials. A rapid solidification process has been proposed as an alternative process route for such alloys. This process has a great potential to produce fine grained material and homogeneous microstructures in high alloyed aluminium. As a result of this, the material properties like mechanical strength, fatigue resistance, wear resistance and corrosion resistance may be improved. By the use of a coextrusion process, dual Al rods can possibly be made with a rapidly solidified material with improved mechanical and/or corrosion properties as the surface material, and a more ductile alloy as the base material (Ma et al., 2017). Such combination of dual materials and forming technologies can improve the production efficiency of lightweight components. In addition, the opportunity to use exotic or expensive alloying elements, such as silver, in the RS surface layer increases because the thickness of this layer is expected to be thin.

In this paper, coextruded rods with Al-4.5wt%Mg as the base material and rapidly solidified Al-4.5wt%Mg-1wt%Ag as the surface material has been characterized. That includes microstructure characterization, precipitation hardening, mechanical testing and corrosion testing.

EXPERIMENTAL

Materials

The examined materials are coextruded rods, consisting of an Al-4.5wt%Mg alloy as the base material and a rapidly solidified Al-4.5wt%Mg-1wt%Ag alloy as the surface material. Both alloys are made from the bolts of Al-4.5wt%Mg produced at Hydro Sunndalsøra. The composition of these bolts is given in Table 1.

Table 1. Element Composition [wt%]

Al	95.28	B	0.0030
Mg	4.53	Pb	0.0021
Si	0.075	Zr	0.0017
Fe	0.071	Mn	0.0007
Ti	0.017	Cu	0.0007
Ga	0.0087	Ca	0.0003
V	0.0059	Sn	0.0004
Zn	0.0034	Cr	0.0003
Ni	0.0031	Co	0.0002

The bolts are remelted to 1 kg bolts with a diameter of 40–44 mm at SINTEF in Trondheim. Addition of 1wt% Ag to the surface alloy is done during this remelting. Three bolts of Al-4.5Mg-1Ag are rapidly solidified in a melt spinner at NTNU. The rapidly solidified material from these bolts are labelled H1, H2 and H3, respectively. The RS ribbons are cut to small pieces at SINTEF in Trondheim and compacted to billets in Denmark. Cylindrical through thickness holes, with a 16mm diameter, are

machined through the billets of the RS material. The coextrusion are conducted at SINTEF Raufoss Manufacturing by pressing a billet of the base material together with a billet of the RS surface material, with the surface material in front. Five rods are made, and named P3, P4, P5, P6 and P7. Details of this procedure have been published elsewhere (Ma et al., 2017).

Sample Preparation

The cylindrical, coextruded rods are cut both along cylinder axis and normal to the axis. Then the samples are grinded and polished in several steps down to cloths with 1 µm diamond paste. Some samples were finally electropolished in order to get a smooth surface.

Heat Treatment and Analysis Procedure

Coextruded samples are directly solutionized. Then samples are aged at 125°C for times up to 2000 h. The next group of samples are solutionized for 30 min. at 560°C and then either aged different times at 125°C or at 160°C. Maximum aging time is 300 h at 160°C and 2000 h at 125°C. All samples are quenched in cold water after aging.

A Matsuzawa DVK-1S is used for the hardness measurements, with 1 kp (9.81 N) load, 15 s load time and 100 pm/s loading speed. Every time, there are four measurements in the surface material and three in the base material.

Two samples from approximately 25–30 cm along the rod P7 are solutionized at different temperatures. One of the samples is solutionized in a salt bath in two steps at 400 and 450°C. The sample is held at each temperature for 15 min., and the heating time between the two temperatures is 17 min. The other sample is solutionized in air at 560°C for 30 min. These samples are later examined in SEM.

Three tensile specimens are solutionized at 560°C in salt bath for 30 min., followed by aging at 160°C in oil bath. One of the specimens is from 20–30 cm along P5, and aged for 236 h to obtain the T6 condition. The second specimen is from 50–60 cm along the rod P5, and aged for 263 h to obtain an overaged condition. The third specimen is from 80–90 cm along P5, and aged for 6 h to obtain an underaged condition.

The procedure for all scanning electron microscopy (SEM) investigations, including energy dispersive spectrometry (EDS) are followed. A Zeiss Ultra 55 Limited Edition FESEM is used. Micrographs of solutionized and aged samples are taken, using signals from the backscatter electron detector. EDS line scans are performed near the surface of these samples. EDS maps are made around the interface. The EDS software that is used is Bruker Quantax Esprit. Micrographs are also taken of as-coextruded, solutionized and aged samples that are vibratory polished.

Fracture surfaces of an as-coextruded, an aged to T6, an overaged and an underaged tensile specimen are investigated.

RESULTS AND DISCUSSION

Heat Treatment

The hardness measurements are summarized in Figure 1. The material coextruded between 420 and 460°C obtains a hardness of 90VHN during aging at 160°C in the surface layer and no increase of the hardness (65 VHN) in the base material of Al-4.5 wt% Mg. The material solutionized at 560°C obtains a much larger hardness by aging. The maximum hardness at 160°C is 125 VHN in the surface layer of Al-4.5 wt% Mg-1 wt% Ag and 90 VHN in the base material of Al-4.5 wt% Mg. Thus small increase in hardness is also recorded in the base material. The material does not reach a maximum when it is aged at 125°C after solutionizing at 560°C. The largest value is 125 VHN in the RS-material in the surface and 85 VHN in the base material.
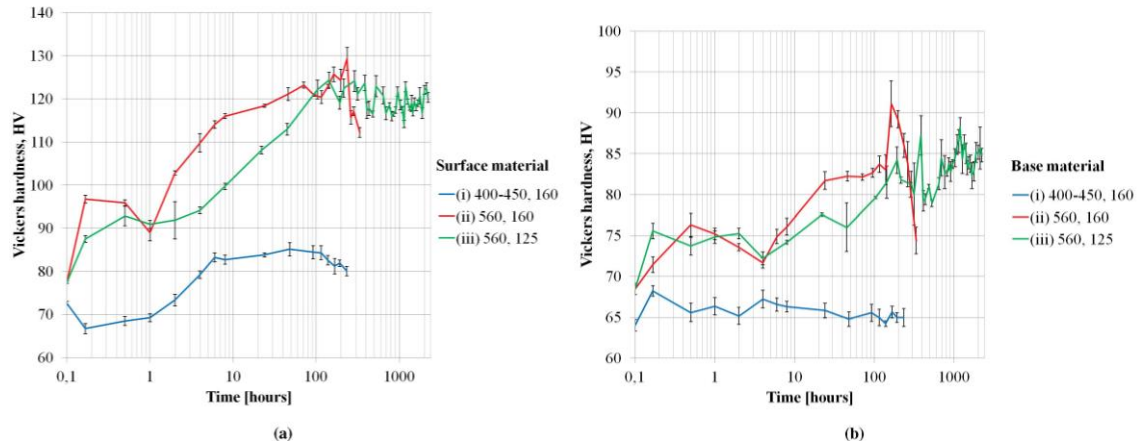
Figure 1. Vickers hardness in different co-extruded samples after three different heat treatments: (i) solutionized at 400–450°C and aging at 160°C, (ii) solutionized at 560°C and aging at 160°C, and (iii) solutionized at 560°C and aging at 125°C. (a) The surface material. (b) The base material.

SEM reveals that the coextruded samples contained some small particles with diameters up to 2 µm (Figure 2). These are identified as the equilibrium T-phase (Ma et al., 2017). When the samples are solutionized at 560°C, almost all particles go into solution. We expect that all Ag and Mg will be in solid solution using longer solutionizing time. The sample heat treated at 160°C contains minute particles that are white in BSE. This is probably the intermediate T'-phase which are hexagonal (Cousland & Tate, 1986). The samples that are solutionized in air obtain an oxide layer that has not been observed on samples solutionized in salt bath. Figure 3 shows EDS line scans from the areas close to the surface of a sample that has been solutionized in air at 560°C and a sample that has been solutionized in salt bath at 400–450°C. It is seen from the line scans that there is a magnesium oxide layer on the surface of the sample that has been solutionized in air. The silver content in the oxide layer is lower than in the rest of the material. In the oxidized sample, there is a zone close to the surface with magnesium depletion. This zone starts approximately 0.8 µm into the material, and the magnesium content decreases towards the surface. In the sample that has been solutionized in salt bath, there is some magnesium depletion less than 0.02 µm from the surface. (Note that SEM analysis was made in the rod close to the start of the coextrusion where thickness of RS-material was 1 mm thick).

(a)

(b)
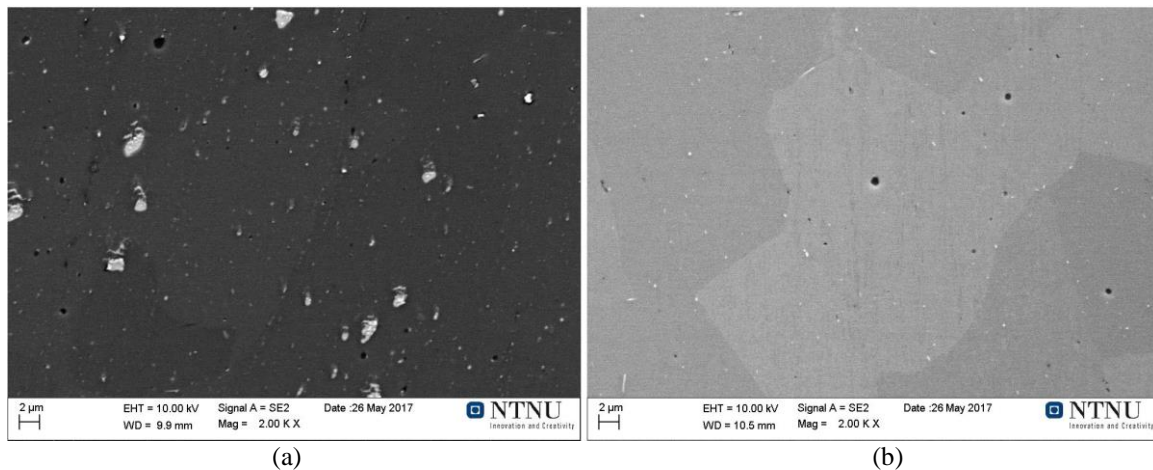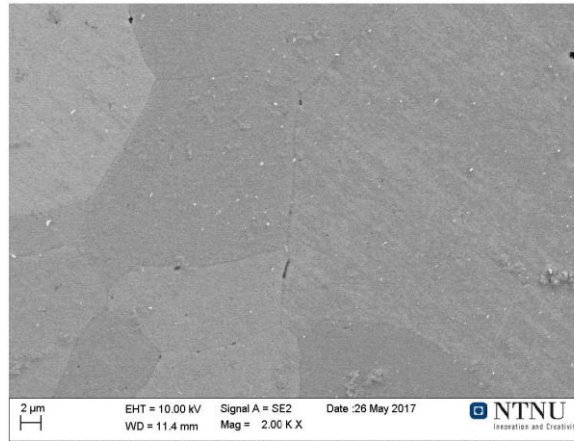
(c)

Figure 2. Micrographs of vibratory polished surfaces, showing the particle distribution in the surface material. (a) As-coextruded. (b) Solutionized at 560°C. (c) Solutionized at 560°C and aged at 160°C.

The EDS line scans in Figure 3 show original coextruded material contains two types of particles: Al-Mg-Ag particles, probably equilibrium T-phase, and an Al-Mg phase, probably Al_8Mg_5. The rods that have been solutionized at 560°C, have only a few Al-Mg-Ag particles (Figure 3b), and probably some minute Al_8Mg_5 as the SEM-scans show small peaks in Mg-curve.

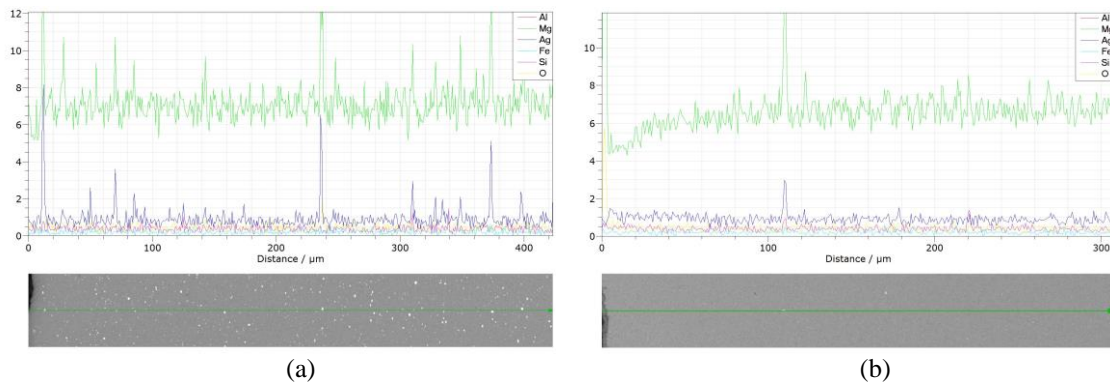(a)   (b)

Figure 3. EDS line scans from the surface (to the left) and into the surface material. (a) Solutionized in salt bath at 400–450°C. (b) Solutionized in air at 560°C.

Blisters occur in the surface material during solution treatment at 560°C, but not when solutionizing at 400–450°C. Blisters are observed both after solution treatment in air and in salt bath. Figure 4 shows pictures of the blistered surfaces and SEM micrographs of a blister crack and its surrounding area. There is partly alignment of blisters along surface features from the extrusion. In the upper part of Figure 4c it can be seen voids that has nearly grown together to form a new big crack. The area close to the tip of the big crack, shown in Figure 4d, contains small intergranular cracks. There are also more large particles in the area close to the crack tip. SEM micrographs of the inner surface of a blister crack is shown in Figure 4d. There are smooth grain shaped features with rounded edges, and the size corresponds to the grain size.
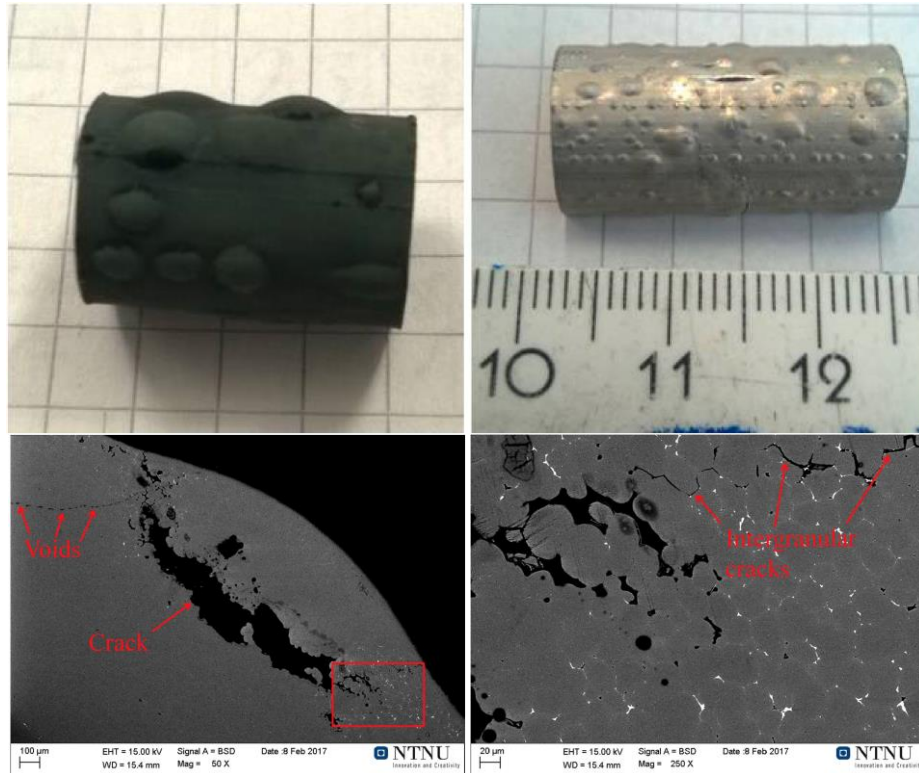
Figure 4. (a) Blistered and oxidized surface after solutionizing for 30 min. in air at 560°C. (b) Blistered surface after solutionizing for 30 min. in salt bath at 560°C. (c) and (d) SEM micrographs of a blister crack and the surrounding area.

To strengthen the material by precipitation hardening, the solution treatment is important to obtain a supersaturated solid solution prior to aging. This means that the solid solution temperature, holding time and heating medium should be optimized to be able to reach the material's strength potential. In this work, the precipitation hardening is carried out with a solutionizing temperature of 560°C. This temperature is chosen because it is right below the solidus line of the Al-4,5wt% Mg alloy, and also below the eutectic point in the Al-Ag system. The temperature is set as high as possible to still avoid melting of the materials. However, at temperatures that close to the solidus line, there is a risk of local melting if some areas have a higher magnesium concentration than the total concentration. The high temperature has the desired effect of dissolving silver in the surface material, and nearly all are dissolved after solutionizing for 30 min. (Figure 5b). A longer solution treatment time would probably dissolve even more. Some of the white particles that has not been dissolved might also be Al-Fe phases. The increase in dissolved silver is significant compared to the amount that is dissolved when solutionizing at 400–450°C, which is the solution treatment temperature in the previous work (Figure 5a, Ma et al., 2017).

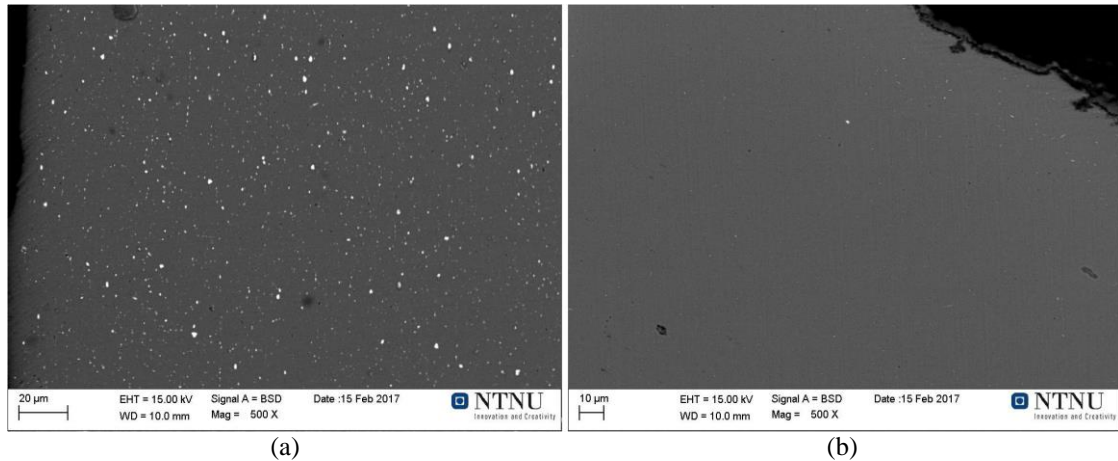(a)   (b)

Figure 5. SEM images of the RS Al-4.5Mg-1Ag surface material after solution treatment at different temperatures. (a) solutionizing at 400–450°C for 15 + 15 min. (b) solutionizing at 560°C for 30 min.

The increase in dissolved silver at the higher temperature affects the aging response in both materials. First of all, the strength contribution of silver in solid solution gives a higher hardness after solution treatment and after short aging times. The effect of this is so pronounced that the hardness of the surface material, when solutionized at 560°C, after only 10 min. of aging exceeds the maximum hardness obtained after solution treatment at 400–450°C and aging at 160°C. This is the case both when aging at 160°C and 125°C. Secondly, the increased dissolution of silver leads to more precipitation of strengthening GP-zones and coherent particles during aging. Therefore, a higher maximum hardness is obtained with the higher solution treatment temperature.

Solution treatment is done both in air and in salt bath. Of these, solutionizing in salt bath is preferable because of the magnesium oxide layer (Figure 3b) that is formed in air. The oxidation of magnesium on the surface results in a zone with magnesium depletion in the outer part of the material. To avoid oxidation on the surface and depletion of magnesium, an inert gas, such as Argon, may also be an alternative as the heating medium. The solution treatment at 560°C also caused blistering on the surface, which was not seen after solutionizing at the lower temperature (Figure 6).
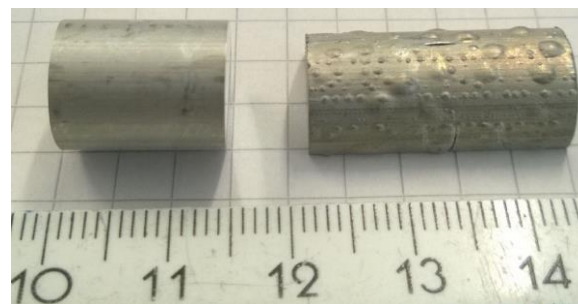

Figure 6. The sample to the left has been solutionized at 400–450°C for 15 min. at each temperature, with 17 min. heating time between the temperatures. The sample to the right has been solutionized at 560°C for 30 min. Both samples were heat treated in salt bath. Blisters are only seen after solution treatment at 560°C.

The blisters appear on the surface because of cracks in the surface material beneath the surface. These cracks are most likely caused by hydrogen, in the surface material. In aluminium alloys, the hydrogen can be trapped on interstitial lattice sites, dislocations, vacancies, solute atoms, precipitates and high-angle grain boundaries (Toda et al., 2009). The gas may have been introduced to the material during

the rapid solidification melt spinning process or the compaction of the rapidly solidified material. The melt spinning is conducted in air, but if it had been done in an inert atmosphere the possibility of hydrogen absorption would be reduced. The appearance of blisters after solution treatment at high temperature correlates with the findings (Toda et al., 2009), who shows that hydrogen micropores initiate and grow in an Al-5.5mol%Mg alloy at high temperature exposure. The cracks seem to be intergranular. As can be seen in Figure 5d there are intergranular cracks in front of the tip of the large blister crack. The features at the inner crack surface look like grains with rounded edges, as a consequence of the high pressure during the growth of the crack. Cracks have also been observed along the interface after solution treatment. When fractured, this caused more separation of the two materials as-aged than as-coextruded. The fracture surface of the aged specimens also contained larger voids, in both materials, than the as-coextruded specimen (Figure 7). Even though these specimens also are aged, the larger voids are probably explained by the increased pore growth at high solution treatment temperatures. The reason for this is that the solution temperature is much higher than the aging temperature, and the larger voids are also seen in the underaged specimen that only are aged for 6 h.
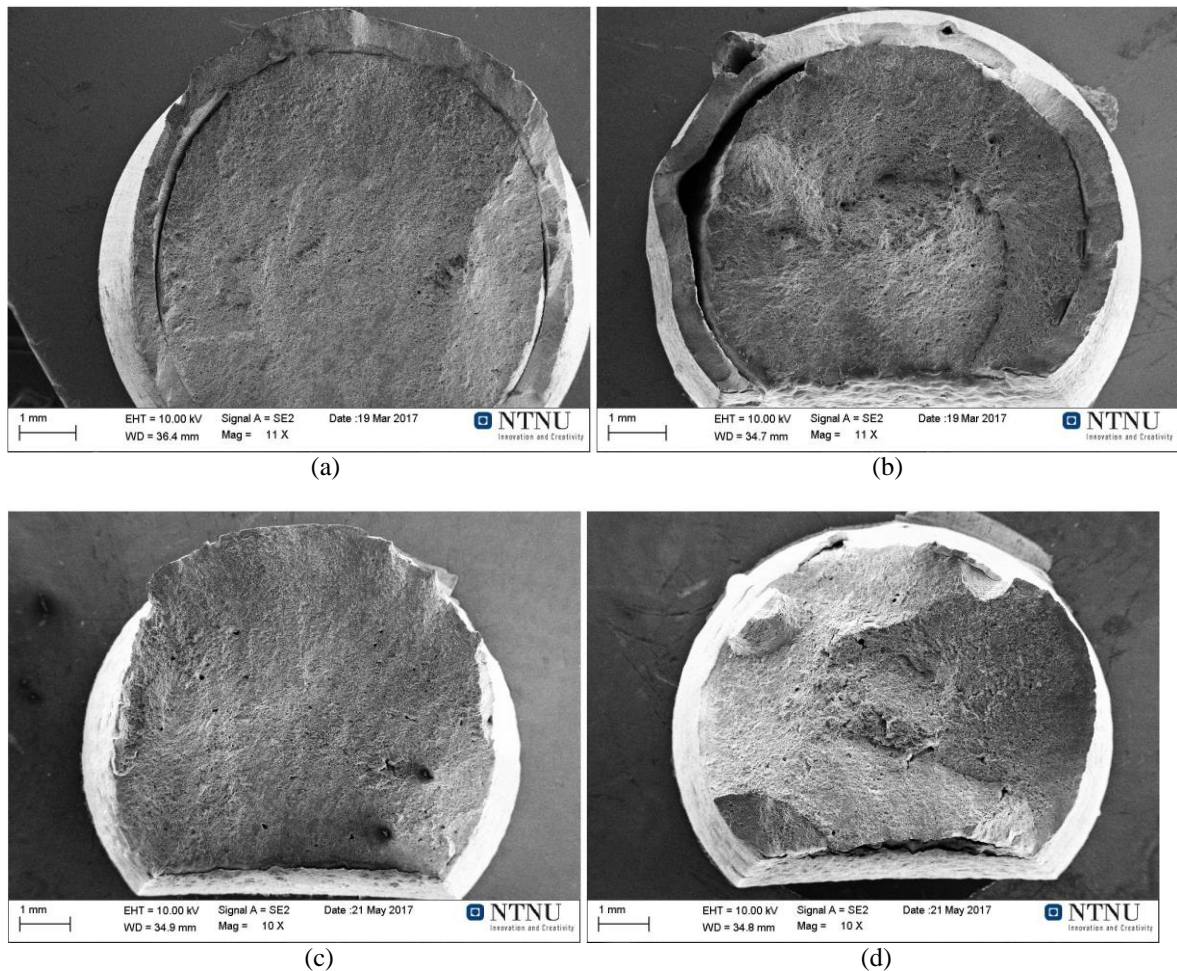

Figure 7. Fracture surfaces in four different conditions: (a) as-coextruded, (b) aged to T6, (c) underaged and (d) overaged. Larger voids are seen in the three aged specimens.

Aging

The positive effect of adding 1wt% silver to Al-4.5wt%Mg on the precipitation hardening properties has earlier been documented by others (Auld, 1968; Cousland & Tate, 1986; Kubota, Nie, &

Muddle, 2004), and is confirmed in the previous work (Ma et al., 2017). In this work, aging temperatures of 160°C and 125°C has been compared, both after solution treatment at 560°C. The surface material has a drop in hardness after 1 h of aging, at both aging temperatures. Before this point, the silver in solid solution contributes to the hardness, but this contribution decreases when the silver precipitates. The drop occurs at a point where the lost hardness contribution from silver in solid solution is greater than the hardness increase from the early precipitates. After the drop, the formation of strengthening GP zones and coherent particles is faster at higher temperature, as expected. At both temperatures there is first a steady increase in hardness, followed by a flat-out and then some peaks and dips. These different parts of the aging curve represent the different stages of GP zone and particle precipitation. The relatively flat part, and the small peaks and dips before the maximum hardness may indicate that there are several precipitation reactions leading to a proximately the same hardness. This is also suggested by others (Kubota et al., 2004). Another explanation for the flat part is that the hardening precipitates are relatively stable, and the peaks and dips also represent a kind of stabilization in a narrow value range. The aging at the lowest temperature is expected to give the highest maximum hardness. However, when the aging at 125°C is terminated after 2229 h, the overaged condition was not yet reached and the maximum measured hardness is lower than for aging at 160°C.

This could be due to that the precipitation of the T'-phase has not started yet (Auld, 1968). The peaks and dips, from 150 h to termination, might correspond to the peaks and dips before maximum hardness when aging at 160°C. If so, the maximum hardness when aging at 125°C has not been reached. Anyhow, it is a very slow precipitation hardening system. If the maximum hardness not have been reached, it might be practical reasons, related to aging time, for choosing a higher aging temperature, even though it turns out that 125°C give a higher hardness. That being said, the best compromise between maximum hardness and aging time might be at temperatures between 125°C and 160°C.

The Al-4.5wt% Mg base material is not considered as a precipitation hardenable alloy. Nevertheless, there is a hardness increase during aging, both at 160°C and 125°C. This indicates that some alloying elements are dissolved during solution treatment, and precipitated as hardening phases during aging. However, the hardness increase is small compared to the increase in the surface material. The aging response of the two materials are represented in the same plot in Figure 8.
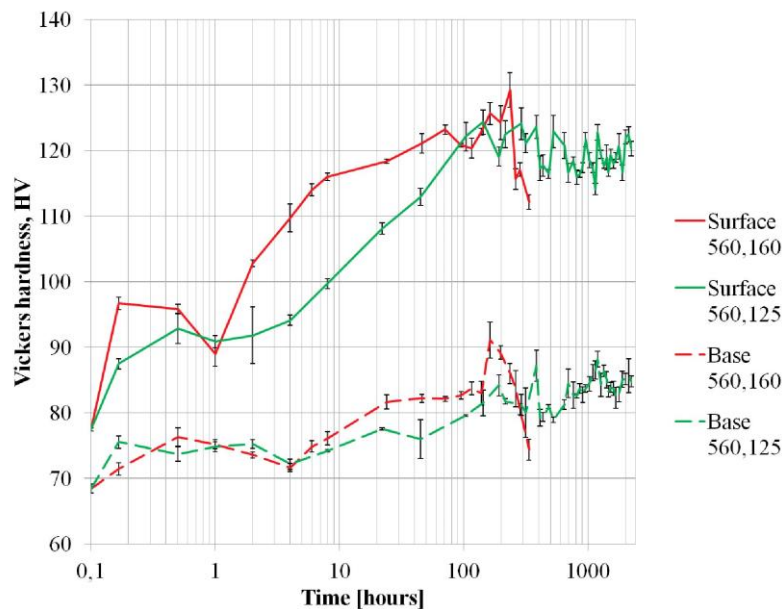

Figure 8. Comparison of the aging response of the surface material and the base material. Curves are shown for aging at 125°C and 160°C, both after solution treatment at 560°C.

Tensile Behaviour

Tensile testing and deformation analysis are performed at four heat treatment conditions: (1) As-coextruded, (2) Aged to T6 at 160°C, (3) Aged to an underaged condition at 160°C and (4) Aged to an overaged condition at 160°C. A special tensile specimen geometry is designed in a way that makes it possible to study the deformation behaviour along the interface during tensile testing. 100 mm long rod parts are used, and the middle 30 mm is thinned 2 mm from one side. This resulted in a flat rectangular area in the middle of the specimens, with the surface material along the edge and the base material in the central part. The geometry of the tensile specimens is shown in Figure 9. The tensile testing is performed with a MTS 810 Material Testing System, with a strain rate of 1 mm/s.
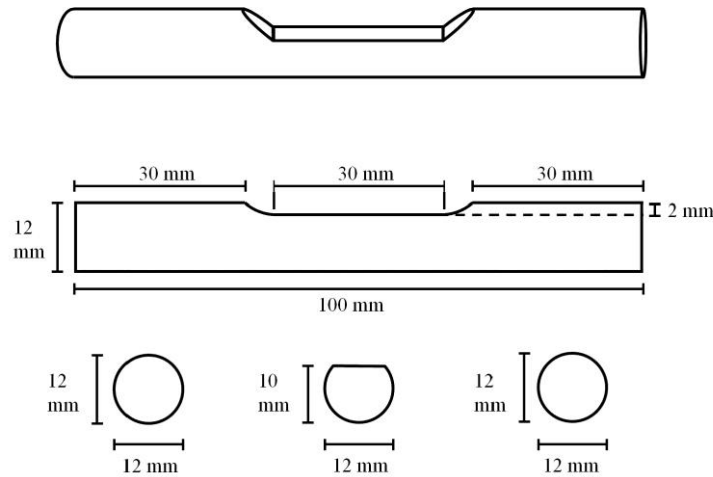

Figure 9. The geometry of the tensile specimens. In the bottom, the transverse cross sections along the specimens.

Stress-strain curves for the coextruded rods at four heat treatment conditions are shown in Figure 10a. It should be noted that the tensile specimen geometry is not conventional (Figure 9), so that the values are not comparable to values from the literature. The aged specimens are solutionized at 560°C and aged at 160°C. The thickness of the surface layer is approximately 500 pm in the as-coextruded and aged to T6 specimens, approximately 150 pm in the overaged specimen and approximately 70 pm in the underaged specimen. The T6 and overaged rods have a yield strength around 155 MPa and 130 MPa respectively, while the underaged and as-coextruded rods have a yield strength around 100 MPa. The T6 and as-coextruded rods have approximately the same ultimate tensile strength, at around 270 MPa. The as-coextruded specimen reaches the tensile strength at a higher strain than the T6 specimen, thus the ductility is higher without heat treatment. The stress-strain behaviour of the underaged specimen is quite similar to the non-heat treated, but with an ultimate tensile strength of 245 MPa. The overaged specimen has a quite similar stress- strain behaviour as the T6 specimen, but with a little higher ductility and an ultimate tensile strength just above 250 MPa. The heat treated samples, especially the underaged and overaged, experience some load drops between the yield point and the ultimate tensile strength. All the stress-strain curves have serrations.
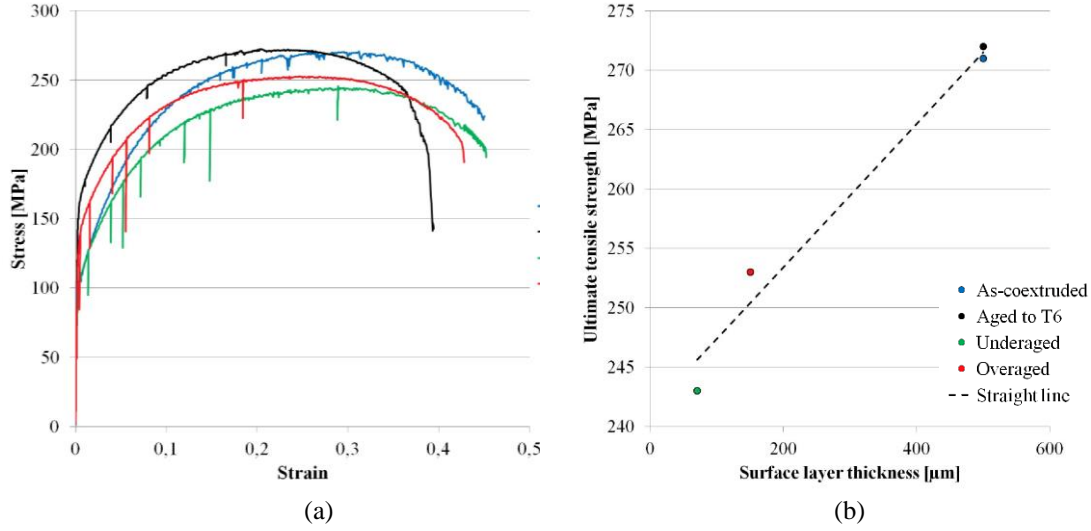
(a)   (b)

Figure 10. (a) Stress-strain curves for four different heat treatment conditions; (b) Tensile strength as a function of the thickness of the RS-surface layer.

As seen in the stress-strain curves in Figure 10a, the aging increases the yield stress of the coextruded rods. However, the ultimate tensile strength is not much affected by the aging, but has a nearly linear relationship with the thickness of the surface layer. This relationship is plotted in Figure 10b. The coextruded material can be treated as a fiber composite material, with the surface layer as a continuous and aligned fiber in the longitudinal direction. The contributions from the two materials to the ultimate tensile strength are assumed to follow the rule of mixtures. From this rule, the maximum ultimate tensile strength in the longitudinal direction of a composite material is given by Equation 1:

$$UTS_{tot} = f_s \times UTS_s + (1 - f_s) \times UTS_b \tag{1}$$

where UTS_{tot}, UTS_s and UTS_b are the ultimate tensile strength of the coextruded material, the surface material and the base material respectively, and f is the volume fraction of the surface material. The thickness of the surface layer varied from approximately 500 pm to 70 pm. Since the diameter of the rods are 12 mm, the volume fraction of the surface layer is small in all the tested specimens. Thus, the contribution from the base material dominates, and changes in the ultimate tensile strength of the surface material due to aging have little influence on the total ultimate tensile strength. However, changes in the volume fractions of the materials have more influence. The differences in ultimate tensile strength that have been observed indicate that the strength of the surface material is significantly higher than the strength of the base material.

The ductility seems to be the same in both materials as-coextruded, since the as-coextruded specimen and the underaged specimen reach both the ultimate tensile strength and fracture at the same strain, despite the difference in surface layer thickness. On the other hand, the specimen that is aged to T6 has the lowest ductility. The surface layer of the T6 specimen also tends to have a lower strain than the base material during the test. Except for this little difference, the deformation of the two materials is very similar, indicating that they have the same strain hardening. That is logical, since the two materials are based on the same Al-4.5Mg alloy, and magnesium contributes to the strain hardening properties.

The drops that are seen in the stress-strain curves of the aged specimens, are probably due to voids, blisters, separation of the two materials or other defects caused by the heat treatment. The larger drops in the overaged and underaged material, might be because of the thinner surface layer, and less strength to withstand it.

CONCLUSIONS

Coextruded rods with Al-4,5wt%Mg as the base alloy and rapidly solidified Al-4.5%Mg-1wt%Ag as the surface material has been characterized. The results are promising with respect to obtain a material with excellent surface properties.

Precipitation hardening is performed with a solution treatment temperature of 560°C. At this temperature nearly all the particles in the surface material are dissolved after 30 min., giving an excellent basis for strength increase during consecutive aging. A hardness increase from 77 HV to 129 HV is obtained in the surface material when aging at 160°C for 236 h. The high solution treatment temperature causes blister formation on the material surface, because of expanding gas in the rapidly solidified material.

Tensile testing confirms a nearly linear increase in ultimate tensile strength with increasing surface layer thickness, despite different heat treatments. In an underaged condition with a surface layer thickness of 70 pm, the ultimate tensile strength is 243 MPa, while it is 271 MPa in the as-coextruded condition with a surface layer thickness of 500 pm. This indicates that the strength of the surface layer is significantly higher than that of the base material. The ductility of the rods decreases with precipitation hardening, and is a little lower in the surface material than in the base material. Nevertheless, the deformation behaviour is approximately the same in the two materials due to similar strain hardening, and the separation of the two materials at high strains is ductile.

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