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Precipitation Hardening of Semi-Finished Micro Components Made of Al-2Sc

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The continuous miniaturization of products needed e.g. in the automotive or the micro electronic sector requires process chains which allow the manufacturing of microscopically small components in high quantities. The development of the required processes and technologies is the aim of the Collaborative Research Centre 747 "Micro Cold Forming" of the German Research Foundation. A necessary step in the manufacturing process chain is the heat treatment, which enables the adjustment of the semi-finished micro components to cold forming. If necessary further heat treatment has to be performed after cold forming to reduce strain hardening. Finally the service properties have to be adjusted e.g. by precipitation hardening of aluminium alloys in order to increase the strength above the strain hardened level.

Within the Collaborative Research Centre an Al-Sc alloy with 2 mass-% Scandium was developed in order to achieve outstanding mechanical properties. In case of conventional casting techniques due to the high Sc content most of the alloying element would be precipitated as Al_3Sc during the solidification. In order to avoid these precipitations the material is produced as thin foils with a Physical Vapour Deposition (PVD) Magnetron Sputtering process at low temperatures which ensures the supersaturated solution. It is intended to age the material after cold forming to get a maximum material strength. Therefore the optimal aging parameters have been evaluated and characterized by ultra micro hardness measurements.

Keywords: *Al-2Sc, artificial aging, PVD, micro foils, CRC 747 "Micro Cold Forming".*

1. Introduction

The trend for micro components, such as connectors in mobile phones or fuel injectors, requires a steady decrease of size, weight and production cost per part and an increase in functionality. Additionally they have to be produced in a shorter time with a higher output regarding quantity. At the same time a precise and reliable function over the entire product life is required, which implies a constant or even improved quality. To fulfil all these requirements new production technologies, including a perfect adaption of materials and processes, are necessary because a direct downscaling of conventional processes into the micro scale is rarely possible. The most relevant difference is a significant higher impact of the local microstructure on the material and processing behaviour due to the very low ratio between for example grain size and material thickness. To deal with the challenges of downscaling the production processes the German Research Foundation initiated the Collaborative Research Center (CRC) 747 "Micro Cold Forming - Processes, Characterisation, Optimisation" at the University of Bremen in 2007. Overall 40 scientists from 8 different institutes participate in the interdisciplinary research in 14 different sub projects.

One important process step in the production process is the heat treatment. The heat treatment process is responsible for the adjustment of the material properties in order to ensure cold formability and furthermore to obtain the required mechanical properties for later applications. By e.g. recrystallisation annealing it is possible to achieve material properties, which enable an accurate and energy efficient cold forming process. In this context it is necessary to generate a homogeneous micro structure as far as possible regarding grain size and alloying element distribution. Particularly very thin components with wall thicknesses below 100 µm are very sensitive against inhomogeneities, because the number of grains in the cross section is usually very low. A higher portion of coarse grain leads to a lower number of activatable gliding planes at external load. Hence, an inhomogeneous and coarse grain size as well as other imperfections like defects or inclusions have a much greater impact on the failure behaviour of micro components the thinner the wall thickness is [1, 2]. The strain hardening by cold forming can lead to considerable material strength. However, in general a defined strength level is required. Therefore a final heat treatment is performed.

Age hardenable aluminium alloys have a beneficial combination of properties regarding their low weight combined with a high strength, which is widely used for macro scale components. For cold formed micro components with a thickness less than 100 µm these materials could be a very good opportunity as well. Unfortunately it is nearly impossible to produce the needed thin foils by conventional processes (e.g. milling, resp. rolling). Therefore another approach is used in the CRC 747 to produce these materials. It is based on a Magnetron Sputtering process that is conventionally used for the deposition of wear resistant layers on a substrate. In this special case a high adhesion between the deposed layer and the base material is not required because the layer is extracted and used as a foil like material [3] to produce micro-components. By this method very special alloys and properties can be produced that would be impossible to produce by conventional melting metallurgy.

Al-2Sc is one of the materials produced by this new method. Scandium as an alloying element in commercially available alloys is normally added in very low concentrations which already has a great impact on the strengthening behaviour of the material [4, 5]. Therefore the effect of higher Scandium contents should be evaluated on the basis of the developed alloy Al-2Sc. Regarding the low process temperatures (<200 °C) and the fast cooling rate during the Magnetron Sputtering process, the produced material can be stated as solution annealed and quenched resulting in a supersaturated solution with a very high Scandium content. Therefore artificial aging treatments at different temperatures have been evaluated to determine the optimal time-temperature combination to achieve the maximum strengthening effect.

2. Materials and Methods

2.1 Material

The material used for the artificial aging treatments is an Al-2Sc-alloy with the distinct columnar morphology after sputtering shown in Fig. 1 and the chemical composition illustrated in Fig. 2. The mean Scandium content across the foil thickness is around 1.5 mass %.

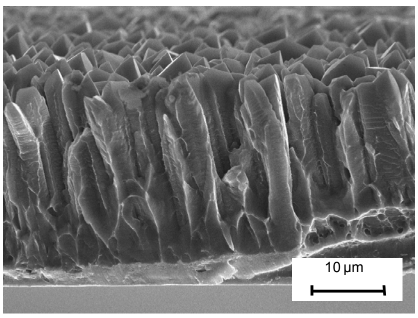

Fig. 1 Fracture surface (SEM) in as-sputtered condition

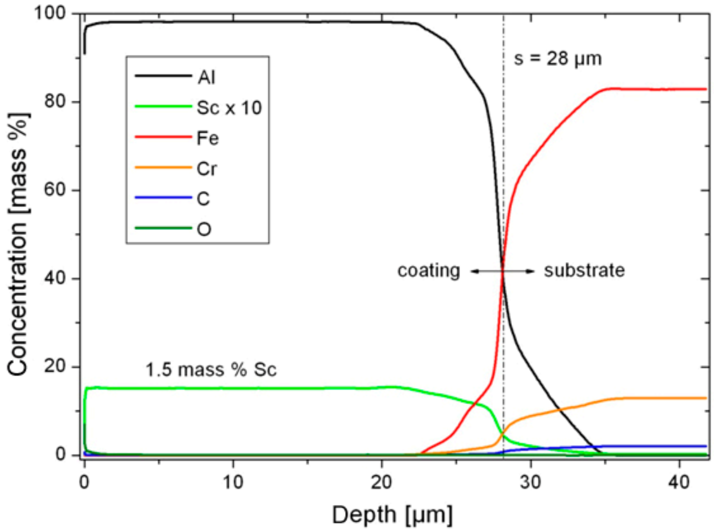

Fig. 2 Chemical composition of the foil, determined by optical glow discharge spectroscopy (GDOS)

2.2 Experiments

Artificial aging treatments were performed at three different temperatures (200 °C, 300°C and 400 °C). The samples were aged at each temperature over 1, 5, 10, 30, 60 and 120 minutes. After aging the hardness, as an easily measurable indicator for the material strengthening, was evaluated by ultra-micro hardness measurements. For this purpose the samples were glued onto a steel plate with a high strength two component glue to perform the hardness measurements on the surface.

3. Results

The results of the hardness measurements are illustrated in Fig. 3.

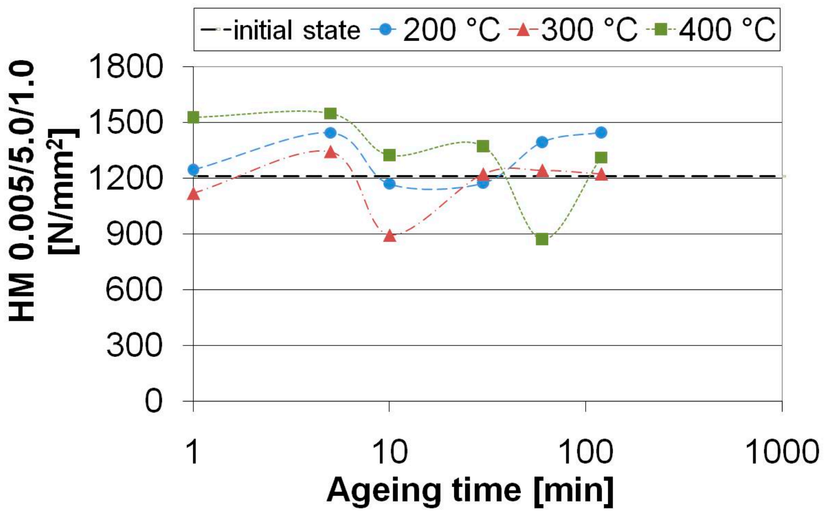

Fig. 3 Results of ultra-micro hardness measurements after artificial ageing

At first the hardness increases for all ageing temperatures, already after 1 min, above the initial state of 1200 HM 0.005/5.0/1.0 [N/mm^2] up to 1550 HM 0.005/5.0/1.0 [N/mm^2] after 5 min at 400 °C. At 200 °C and 300 °C the increase in hardness is less than the one at 400 °C. After this first maximum all curves descend, partially even below the initial state. The minimal hardness is reached for 300 °C after 10 min of ageing. For 400 °C the curve is slightly increasing again, but then descends strongly down to 890 HM 0.005/5.0/1.0 [N/mm^2] after 60 min of ageing. Finally the hardness is increasing up to 1250 HM 0.005/5.0/1.0 [N/mm^2] at 300 °C for 60 min and 1310 HM 0.005/5.0/1.0 [N/mm^2] at 400 °C. At 200 °C a maximum hardness of 1450 HM 0.005/5.0/1.0 [N/mm^2] is reached after 120 min.

4. Discussion

The presented results do not match the assumed curve progression for artificial ageing processes of traditionally produced aluminium alloys. Conventionally this would be an ascending curve up to a maximum, characterized by a specific precipitate phase and diameter, with an ensuing descent caused by the overageing. Only the hardness curve for the sample aged at 400 °C is close to the expected progression. The other two curves and even more the fact that the 200 °C curve is above the 300 °C regarding the hardness level is unexpected. One reason for this could be the inhomogeneous structure of the material in as sputtered condition. As indicated in Fig. 1 the material consists of columnar grain structures. The height of most of the columns is equal to the material thickness. The properties of the grain boundaries in comparison to the ones of the base material are unknown. Therefore the hardness measurement could be highly affected by the indentation point, the local material properties and the surface roughness of the material.

Besides this the accomplished strengthening effect is only 300 HM 0.005/5.0/1.0 [N/mm^2] which is small compared to the differences that are detectable with commercial available materials. Nevertheless the presented material is specific in its structure as well as in its properties and therefore a stronger strengthening seems feasible. Furthermore the thickness of the material has a strong influence on the properties, as mentioned before, because of the greater influence of imperfections and inhomogeneities.

5. Conclusion

Aluminium alloys with a high Scandium content can now be produced by a magnetron sputtering process as thin foils which enables them as a potential material for micro scale components that are produced by micro-cold forming. Furthermore artificial ageing treatments allow a significant strengthening after the forming process to ensure the required material properties for service. The presented results show an increase in hardness by 300 HM 0.005/5.0/1.0 [N/mm^2] up to 1550 HM 0.005/5.0/1.0 [N/mm^2] for an ageing treatment at 400 °C for 5 minutes on the alloy Al-2Sc. For longer aging times or lower temperatures the increase in hardness is smaller.

Regarding the relatively small increase in hardness combined with the unexpected curve progression further experiments combined with a detailed analysis (e.g. hardness measurements on polished cross sections of the samples) are necessary. To determine the precipitation size, phase and distribution for example TEM analysis will be performed.

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