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Microstructural impact on intergranular corrosion and the mechanical properties of industrial drawn 6056 aluminum wires

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ABSTRACT

The influence of individual manufacturing steps during industrial wire drawing processes on the mechanical and corrosion properties of the 6056 aluminum alloy was investigated. These steps demonstrated an essential influence on the microstructure, and thus, the susceptibility to intergranular corrosion (IGC). No clear correlation between IGC susceptibility and hardness was observed. Although the highest resistance against intergranular attack was determined for those alloys in the solution annealed condition, pitting corrosion was identified to occur. Subsequent artificial aging of the solution annealed and quenched wires reintroduced IGC susceptibility; this phenomenon was attributed to the occurrence of galvanic coupling between the noble Cu-phases, located on the grain boundary, and the anodic grain boundary area.

1. Introduction

The 6000 aluminum alloy series is generally considered rather insusceptible to intergranular corrosion (IGC), especially when compared to the 2000, 5000 or 7000 alloys [1]. Nonetheless, the literature confirms that certain factors can lead to IGC vulnerability, most particularly the Mg/Si ratio, the amount of Cu and the heat treatment condition [2–6].

Variations in the Mg and Si content, both major elements in the 6000 alloys, effect the manner and phases present in the microstructure of the alloy. The stoichiometric ratio of Mg to Si, 2:1, promotes the formation of the β-phase (Mg_2Si), which usually precipitates at the grain boundaries. According to Zeng et al. [2], these Mg_2Si particles, which are initially anodic to the matrix, begin to dissolve during corrosion. However, due to the preferential dissolution of Mg, these particles become enriched with Si until an overwhelming cathodic character evolves and prompts the anodic dissolution of the Al solid solution [2,7]. If an increase in Si content generates a mole ratio of Mg to Si less than 1.73 (2:1), any excess Si not consumed for the stoichiometric β-phase (Mg_2Si) may precipitate unalloyed [2,8]. Due to the strong cathodic character of pure Si particles, an excessive Si quantity increases the susceptibility to IGC by severe anodic dissolution of the adjacent periphery of the Si particles [2]. Controversially, other researchers propose that Mg_2Si particles on the grain boundaries do not contribute to IGC vulnerability [9].

Strong evidence on the dominant role of Cu on the susceptibility of 6000 aluminum alloys to IGC is present in the literature [5,6,9,10]; however, various critical values of Cu are proposed, ranging from 0.1 wt.% [11] to 0.4 wt.% [12]. This strong interdependency between IGC susceptibility and Cu content is attributed to the noble character of Cu and any Cu-rich phases, which cause highly effective galvanic cells with the adjacent matrix [5]. Svenningsen et al. observed a nanoscale Cu-rich film and coarse Q-phases ($Al_4Mg_8Si_7Cu_2$) on the grain boundary site and found microgalvanic coupling between these cathodic sites and the adjacent solute-depleted area [6]. Substantiating the relevance of Cu content, Larsen et al. [10] confirmed that merely small additions of Cu have more impact on IGC susceptibility than a large excess of Si. If a Cu-rich film is present on the grain boundaries, as observed by Svenningsen et al. [6], then artificial aging may improve IGC resistance due to a dual-faced factor. Not only does the Cu-rich film become interrupted due to a transformation to discontinuous coarsened particles, the interior Al grain matrix becomes depleted as a result of precipitation formation within the grain. Both aspects reduce the extent of microgalvanic activity along the grain boundaries [6,9].

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In addition to mechanical property improvements, heat treating 6000 alloys is known to dramatically influence the proneness to IGC through the formation of grain boundary precipitations. In general, solution annealed Al alloys are quite resistant to IGC due to the absence of precipitations [3]. During artificial aging, IGC susceptibility increases and reaches a maximum at the peak strength condition due to grain boundary precipitates formed during aging or slow cooling after solution heat treatments [3,6,9,13]. Overaging can reduce or delete IGC susceptibility due to precipitation coarsening, both on the grain boundaries and in the grain interior [6,13]; however, simultaneous strength reduction commonly occurs. Unfortunately, overaging has been observed to introduce pitting vulnerabilities [6], which can impact IGC due to the initiation of corrosion sites at pit walls [14]. According to Wang et al. [15], certain newer aging treatments are able to improve the intergranular corrosion resistance of Cu-containing 6000 alloys without severe strength loss.

The phases present in the 6000 aluminum alloy series containing Cu were investigated intensively. The quaternary Q-phase [5,6,8,16–19], binary θ-phase (Al_2Cu) [8,20], β-phase (Mg_2Si) [2,5,6,8,16] and metallic Si [2,8] are common phases that are present in 6000 aluminum alloys. Nevertheless, deepening the understanding of the correlation between these phases and IGC susceptibility is key to improve the performance of 6000 aluminum alloys.

The present investigation aims to elucidate the microstructural impact on intergranular corrosion and the mechanical properties of industrial drawn 6056 aluminum wires. Corrosion was investigated according to the german standard DIN EN ISO 11846 Method B by measuring the penetration depth as well as the pH and mass change during testing. Mechanical evaluation comprised of micro- and macrohardness measurements while microstructural characterization was conducted using thermodynamic calculation, metallographic examinations and microscopic methods. Analysis of intrinsic corrosion mechanisms focused on the T4 (solution annealed) and T6 (artificial aged) conditions in accordance with [21]; both conditions represent different corrosion mechanisms.

2. Experimental methods

2.1. Materials

The wire material was provided by Drahtwerke Elisental W. Erdmann GmbH & Co.; each wire was associated to a specific manufacturing step within the industrial drawing process. Table 1 presents an overview of the investigated material, with step (a) representing the as-cast material produced by direct strand reduction. Wire from step (b) was subsequently homogenized at 550 °C for 20 h and cooled in air. Steps (c), (d) and (e) executed the drawing process at room temperature and resulted in three different diameters, 13.00 cm, 11.50 cm and 10.25 cm, respectively. Wires with the 10.25 cm diameter were subsequently solution annealed at 545 °C for 6 h and quenched in water (step f, T4 condition). Finally, some solution annealed wires were artificially aged at 172 °C for 12 h to the final T6 condition (step g).

The investigated material was provided from two batches. The as-cast material was taken from batch 2 while all other conditions were produced utilizing batch 1. However, the chemical compositions of both batches, as given in Table 2, reveal only marginal differences.

Table 1
Overview of the investigated 6056 aluminum wires.

N	Specimen designation	Batch	Condition	Diameter (mm)	Deformation φ_{axial} (related to initial diameter)
(a)	6056-AC-15.20	2	As-cast	15.20	0
(b)	6056-H-15.20	1	Homogenized	15.20	0
(c)	6056-D-13.00	1	Drawn	13.00	0.312
(d)	6056-D-11.50	1	Drawn	11.50	0.558
(e)	6056-D-10.25	1	Drawn	10.25	0.788
(f)	6056-SA-10.25	1	Solution annealed (T4)	10.25	0.788
(g)	6056-AA-10.25	1	Artificially aged (T6)	10.25	0.788

2.2. Thermodynamic calculation

Thermodynamic calculations were used in order to obtain information about the microstructural constituents of the T4 and T6 conditions. The calculations were based on the thermodynamic equilibrium at the annealing temperatures, 545 °C (T4) and 172 °C (T6). For thermodynamic calculations, ThermoCalc® software with the database TTAL6 was utilized. Calculations were conducted exclusively for batch 1, including all elements and amounts, which are listed in Table 2. Regarding the diffusion model diffusion was defined to occur in the liquid as well as in the solid.

2.3. Microstructural analysis

The microstructures as well as the corrosion phenomena were analyzed utilizing an optical (Zeiss Apollo) and scanning electron (Zeiss 1540 XB) microscope. The chemical compositions of the phases were determined via an Oxford energy-dispersive X-ray spectroscopy (EDX) facility attached to the scanning electron microscope. Grain boundary investigations as well as element mappings were obtained by using high angle annular dark field scanning transmission electron microscopy (Zeiss Libra 200Cs with an Oxford EDX facility).

Analysis of grain shape and orientation was completed using longitudinal sections of the various wires. The samples were mounted in cold-setting resin and subsequently ground to 2400 level grit and finally polished. In order to increase the visibility of the grain structure, Barker etching was utilized. An electrical voltage of 20 V was applied to the embedded specimens, while they were exposed to a mixture of 950 ml bi-distilled water and 50 ml HBF_4 for 2 min at room temperature.

2.4. Hardness testing

The hardness was tested according to DIN EN ISO 6507-1 (microhardness) and DIN EN ISO 6506-1 (macrohardness). The microhardness testing was according to Vickers (HV0.1, $F = 0.9807$ N) and the macrohardness was evaluated according to Brinell using a hard metal bowl with a diameter of 2.5 mm, a pressure of 62.5 N and a holding time of 10 s. The indentation positions are illustrated in Fig. 1. Nine measurements were conducted on each sample.

2.5. Intergranular corrosion (IGC) analysis

The material was tested in the as-received condition. Only the cutting etches of the wires were ground to 1000 grit using SiC paper. IGC testing was performed according to DIN EN ISO 11846 Method B. This method involves the subsequent steps of degreasing in acetone, etching in 5% NaOH at 60 °C for two minutes, rinsing with deionized water, dipping in concentrated nitric acid (1.4 g/ml) for two minutes, rinsing with deionized water and finally immersion in an aqueous solution consisting of 30 g NaCl and 10 ml concentrated hydrochloric acid (1.19 g/ml) per