Contents lists available at ScienceDirect

## Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

# Characterization of second-phase particles in two aluminium foil alloys

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#### A R T I C L E I N F O

Article history: Received 25 August 2015 Received in revised form 9 November 2015 Accepted 17 November 2015 Available online 22 November 2015

Keywords: Homogenization Precipitation Microchemistry Recrystallization Aluminium alloys Foil packaging

#### ABSTRACT

In commercial 8xxx series aluminium foil alloys several key materials properties, including formability, softening behaviour, strength and ductility, are influenced by alloying elements either in solid solution or in precipitated form, i.e. by the materials microchemistry. In the present paper, the development in microchemistry upon different homogenization treatments is tracked with a variety of experimental characterization methods in two 8xxx alloys which are differentiated by their Si-contents. Special attention is paid to the impact of homogenization on changes in the morphology of constituent particles as well as the formation of dispersoids from the supersaturated matrix. Furthermore, the changes in solute level and precipitation during homogenization annealing are simulated with a statistical micro-chemistry simulation tool termed ClaNG.

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#### 1. Introduction

The intrinsic properties of aluminium, in particular its impermeability and barrier properties together with its high level of corrosion resistance and high formability, make Al-foil an excellent material for packaging applications (e.g. Refs. [1-4]). With a variety of alloys and tempers plus a range of thicknesses varying from as little as 6 µm up to a (defined) upper limit of 0.2 mm, Al-foil may be used in numerous ways to package goods like food, dairy products, pharmaceuticals or industrial products. For many foil applications commercial purity aluminium is used, which consists of more than 99% Al with additions of up to 0.8% Fe and about 0.1–0.3% Si (all alloy compositions in this paper are given in wt.% unless stated otherwise). In the fully soft annealed condition (O-temper), such commercial purity Al of the AA 1xxx series, including the wellknown variants AA 1050 and AA 1200, have rather low strength. Higher strength – to allow for down-gauging for example – may be achieved through increased levels of Fe, for instance in the 8xxx series alloys such as AA 8079 containing 0.7-1.3% Fe.

recrystallization, the final gauge annealing is required to remove rolling lubricant from the foil. Proper control of the final materials properties requires detailed knowledge of the correlation of microstructure and processing parameters (e.g. Refs. [8–17]). In relatively clean Al foil alloys, especially the materials microchemistry, i.e. the solution/precipitation state of the alloying elements Fe and Si and other impurities, will affect both processing and foil properties at final gauge. Because of the very low solubility of Fe in Al, commercial Al alloys will always comprise large Fe-bearing phases. Type, volume, size and, especially, morphology of these so-called constituent particles have an impact on ductility and formability. Pre-heating or homogenization annealing prior to hot rolling will lead to the formation of fine secondary intermetallic phases usually called

Al-foil is produced in two different ways, either starting from a conventional direct chill (DC) cast ingot which is hot rolled to a strip

of about 2-5 mm, or from a 6-7 mm thick sheet produced by

continuous casting routes (twin roll casting or belt casting) [5-7].

This strip is then cold rolled to an intermediate (foilstock) gauge of

about 0.4-1 mm, followed by annealing, typically in the tempera-

ture range 350-400 °C. Then, the material is cold rolled to final foil

thickness. To achieve thinner foil gauges below say 100 µm, closed-

gap rolling must be used, which is performed in a dedicated foil

mill. Most Al-foil products will be used in the O-temper which

means that the foil must be soft annealed. In addition to the







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dispersoids, which likewise affect both processing and final foil properties [8-12]. For processing reasons, the solute level upon hot deformation must be kept low. This means that there is a limitation to the amount of strengthening through solid solution hardening, such that the dominant strengthening mechanism in dilute Al foil alloys is dispersion hardening with some contribution from solutes. Hence, volume fraction and size distribution of the dispersoids is an important factor in dispersion strengthening. Needle-shaped particles result in increased hardness but lead to reduced formability and fatigue resistance.

Grain size has a relatively small influence on strength, but largely impacts ductility, with finer grain size giving higher ductility. Thus, to achieve a good balance of strength and formability in the final gauge product, recrystallization to a fine, uniform grain structure is required which, again, is affected by solute concentrations of alloying elements as well as size and volume fraction of dispersoids (e.g. Refs. [13–17]). Solute elements will inhibit recovery, which generates recrystallization nuclei, thus tending to increase grain size. Dispersoids will tend to refine the grain structure in Al-foil alloys by inhibiting grain boundary movement during recrystallization.

Fig. 1 shows the Al-rich corner of the ternary phase diagram Al-Fe-Si as a function of the Si content, computed for the average Fe-content of 0.8% of the present alloys AA 8079 and AA 8011 [18]. The equilibrium phase diagram displays a total of three species of second-phase particles in equilibrium with the Al-matrix (Al), the stability ranges of which depending on temperature and the exact Si-content. The maximum solubility of Fe in Al is very low, viz. max. 0.05% at 650 °C [19]. Accordingly, alloys based on commercial purity Al will always comprise Fe-bearing constituent phases. Alloys with low Si-content usually contain rather large fractions of the monoclinic phase Al<sub>3</sub>Fe (or Al<sub>13</sub>Fe<sub>4</sub>). This phase usually forms needle-like particles which adversely affect formability of Al-foil alloys [20]. Al-alloys with increased Si content exceeding 0.5% display significant portions of ternary AlFeSi-phases [19–23]. At medium Si-contents and/or high temperatures the cubic  $\alpha$ -AlFeSi phase is obtained. The chemical composition of this phase shows rather large scatter and is described as Al<sub>8</sub>Fe<sub>2</sub>Si or Al<sub>12</sub>Fe<sub>3</sub>Si. That is to say, the ratio of Fe:Si (in at.%) may vary between 2:1 and 3:1 or, in wt.%, between 4:1 and 6:1. Increased Si-contents and lower temperatures tend to stabilize the monoclinic phase β-AlFeSi. For this



Fig. 1. Al-rich corner of the ternary phase diagram Al–Fe–Si, computed for 0.8% Fe (FactSage).

phase a stoichiometric composition of Al<sub>5</sub>FeSi is reported; thus, the Fe:Si ratio (in at.%) is approximately unity. Expressed in wt.% this ratio is very close to 2:1.

Because of the quite high cooling rates prevailing during industrial DC-casting the conditions for equilibrium are not fulfilled in practice (e.g. Refs. [21–25]). Accordingly, the as-cast ingot will be supersaturated by Fe and other slow-diffusing species as Mn and Cr, if present. Under these conditions, besides the stable monoclinic Al<sub>3</sub>Fe phase, there is a metastable phase Al<sub>6</sub>Fe. This phase is isomorphous to the orthorhombic Al<sub>6</sub>Mn phase which is regularly encountered in Mn-containing alloys, e.g. in 3xxx or 5xxx series alloys [26,27]. Furthermore, the literature describes other metastable phases Al<sub>m</sub>Fe where m varies from 4 to 5 [20–25]. Homogenization at high temperatures leads to a transformation of the metastable Fe-bearing phases towards stable Al<sub>3</sub>Fe [12,28–30]. In the presence of Mn the  $\alpha$ -AlFeSi phase is replaced by an isostructural quaternary  $\alpha$ -Al(Mn,Fe)Si phase which, again, may show large variation in exact chemical composition [2,11,20,31].

In the present study the microstructural evolution of two typical dilute Al-Fe-Si foil alloys during homogenization was investigated using optical and electron microscopy as well as measurements of specific electrical resistivity. The materials studied included an alloy AA 8079 with 0.8% Fe and very low Si (i.e. ratio Fe:Si >> 1) and an alloy AA 8011 with similar Fe and Si (Fe:Si ~ 1). The effect of different homogenization practices on the formation of intermetallic phases and solid solution levels was analysed. Especially, we examined the impact of homogenization on changes in the morphology of plate-like constituent particles which are known to adversely affect formability of 8xxx series alloys [20]. Moreover, the formation of dispersoids from the supersaturated matrix (type, volume, size) was tracked. The evolution in microchemistry, i.e. variation in solute level and precipitation, upon homogenization annealing was further simulated with a statistical microchemistry model termed ClaNG [32] in order to aid the interpretation of the experimental findings. Note that some of the results have already been presented at a recent conference [33].

#### 2. Experimental procedures

#### 2.1. Material and heat treatments

Slices were cut well below the top of two conventionally directchill (DC) cast ingots of two different AA 8xxx foil alloys which were produced on industrial scale at the Alunorf rolling and remelt plant in Norf, Germany. All samples for the present study were taken close to the half width at quarter thickness of the as-cast ingots. This position is commonly considered to be representative of the product properties at final gauge. Two different 8xxx series alloys were probed, viz. AA 8079 and AA 8011, the chemical composition of which is given in Table 1. It is seen that the two alloys are quite similar in their Fe-content of about 0.8%, but vary in their Sicontent. Alloy AA 8079 has a very low Si-content of 0.056%, which is typical of commercial purity Al. Alloy AA 8011 has Si added to a content of 0.66% to provide some extra strength and ductility required for specific packaging applications [5,34].

Samples from these two as-cast ingots were subjected to

 Table 1

 Chemical composition of the two AA 8xxx alloys analysed in the present study (all in wt%; rest: Al).

Alloy	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti
AA 8079	0.056	0.838	0.0016	0.019	0.001	0.0010	0.007	0.020
AA 8011	0.660	0.736	0.0017	0.020	0.016	0.0014	0.006	0.026

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