



Full length article

Effects of trace elements (Y and Ca) on the eutectic Ge in Al–Ge based alloys



J.H. Li ^{a,*}, N. Wanderka ^b, Z. Balogh ^c, P. Stender ^c, H. Kropf ^b, M. Albu ^d, Y. Tsunekawa ^e,
F. Hofer ^d, G. Schmitz ^c, P. Schumacher ^{a,f}

^a Institute of Casting Research, Montanuniversität Leoben, Austria

^b Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Hahn-Meitner-Platz 1, 14109 Berlin, Germany

^c Institut für Materialwissenschaft, Universität Stuttgart, Heisenbergstraße 3, D-70569 Stuttgart, Germany

^d Institute for Electron Microscopy and Nanoanalysis, Graz University of Technology, Graz Center for Electron Microscopy, Austria

^e Toyota Technological Institute, Hisakata 2-12-1, Tempaku, Nagoya 468-8511, Japan

^f Austrian Foundry Research Institute, Leoben, Austria

ARTICLE INFO

Article history:

Received 1 February 2016

Received in revised form

11 March 2016

Accepted 14 March 2016

Keywords:

Al–Ge alloy

Segregation

Solute entrainment

Eutectic solidification

HAADF-STEM

Atom probe tomography

ABSTRACT

Effects of trace elements (0.2Y and 0.2Ca (wt%) on the eutectic Ge in high purity Al–20Ge (wt%) alloys were investigated by multi-scale microstructure characterization techniques. Particularly, the distribution of trace elements (Y and Ca) within the eutectic Ge and/or at the interface between eutectic Ge and eutectic Al was investigated by atomic resolution high angle annular dark field scanning transmission electron microscopy (HAADF-STEM) imaging and atom probe tomography (APT). The combined investigations indicate Al–Y and Al–Ca co-segregations. Such co-segregations change significant morphology and growth of the eutectic Ge. In addition, large Al₂Ge₂Y and Al₂Ge₂Ca phases were also measured. The modification of eutectic Ge is discussed in terms of previously postulated modification mechanisms: twin plane re-entrant edge growth mechanism, impurity-induced twinning, and growth restriction of eutectic Ge.

© 2016 Acta Materialia Inc. Published by Elsevier Ltd. All rights reserved.

1. Introduction

Trace elements may have a dominant effect on the microstructure evolution of Al based eutectic systems. For example, a trace addition of e.g. 200 ppm Sr in Al–Si alloys can modify the eutectic Si from plate-like to fibrous morphology and thereby greatly improve the mechanical properties [1]. Possible modification mechanisms of eutectic Si have been postulated [2–6]. It is generally accepted that impurity induced twinning (IIT) [3] and twin plane re-entrant edge (TPRE) growth mechanism [4,5], as well as poisoning of the TPRE [6] are effective under certain conditions. Among these mechanisms, the modifier agents (e.g. Sr) only within eutectic Si were assumed to be responsible for the modification of eutectic Si. However, the distribution of the modifier agents within eutectic Si has been observed to be inhomogeneous by using atomic

resolution high angle annular dark field scanning transmission electron microscopy (HAADF-STEM) imaging and atom probe tomography (APT) [7–10]. It has been clearly demonstrated that the modification of eutectic Si is related to the formation of the multiple Si twins with a high density [7,8] and/or the co-segregations of the modifiers together with eutectic Si and eutectic Al [9,10]. By contrast, in the case of eutectic Ge, it is not clear yet whether IIT, TPRE and poisoning of the TPRE mechanisms are still valid, although the TPRE mechanism was originally observed in the pure Ge [3–5]. Furthermore, effects of trace elements (e.g. Y and Ca) on the formation of co-segregations and thereby the growth of eutectic Ge also still remain to be explored.

Similar to the Al–Si alloy system, Al–Ge also represents a simple eutectic system, although the eutectic point and temperature of binary Al–Ge alloys (53 wt% at 424 °C) is different from that of binary Al–Si (12.7 wt% at 577 °C) [1]. Apart from these similarities, at least three differences should be highlighted. Firstly, compared to Si, Ge forms growth twins more easily and therefore a preferred <100> texture was not often observed. Instead, Ge grows with a <110> preferred orientation [1]. Secondly, it has been reported that

* Corresponding author. Institute of Casting Research, Montanuniversität Leoben, A-8700 Leoben, Austria.

E-mail address: jie-hua.li@hotmail.com (J.H. Li).

Na can modify eutectic Si in Al–Si alloy [2] and eutectic Ge in Al–Ge alloy as well. However, the modification effect and Ge twinning induced by Na is much less pronounced and decrease progressively with the Ge content in Al–Ge alloys [11]. Furthermore, the strong modifier (e.g. Sr) for eutectic Si cannot modify the eutectic Ge [11]. Thirdly, as illustrated in Refs. [4,5], the presence of two or more twinning events provides re-entrant grooves on at least two crystal faces which can act as preferential sites for molecular attachment and thereby favours the growth of the crystal in three dimensions. In the case of eutectic Si, the presence of modifiers at the twin re-entrant edges (for poisoning of the TPPE) has been experimentally supported. However, the distribution of other trace elements (e.g. Y and Ca) at the twin re-entrant edges was not investigated yet, in particular at an atomic scale. Furthermore, the distribution of trace elements at the interface between eutectic Ge and eutectic Al has been also proposed to affect the growth of eutectic Ge [1]. However, such type of investigations is still missing.

In this paper, the microstructure of eutectic Ge in high purity Al–20 wt% Ge alloys with the additions of 0.2 wt% Y and 0.2 wt% Ca was investigated by multi-scale microstructure characterization techniques, including scanning electron microscopy (SEM), HAADF-STEM and APT. In particular, the distribution of trace elements (Y and Ca) within the eutectic Ge and at the interface between eutectic Ge and eutectic Al was investigated using HAADF-STEM and APT, with the aim to elucidate the growth mechanism that controls the microstructure evolution of eutectic Ge.

2. Experimental

A series of Al–20 wt% Ge alloys (wt%, used through the paper in case not specified otherwise) with the additions of 0.2Y and 0.2Ca were prepared using arc melting. The cooling rate was evaluated to be about 200 °C/min. It is noteworthy that Ge was added by Ge (99.998). Y was added by an Al–4Y master alloy manufactured from (99.998) Al and (99.8) Y. Ca was added by an Al–20Ca master alloy produced from (99.998) Al and (99.8) Ca.

The specimens for SEM investigation were mechanically ground using standard metallographic procedures and finally polished with a colloidal silica suspension. For the microstructure characterization, a Zeiss 1540 EsB CrossBeam® workstation was employed. The imaging was performed with a low acceleration voltage of 5 kV using a secondary electron (SE) detector. The use of the low acceleration voltage enables to obtain high resolution images [12].

The samples for TEM investigation were mechanically ground, polished and dimpled to about 30 µm in thickness, and then ion-beam milled using a Gatan Precision Ion Polishing System (PIPS, Gatan model 691). A constant preparation temperature (about –10 °C) was maintained by using a cold stage during ion beam polishing. High resolution TEM was performed using an image-side Cs-corrected JEOL-2100F microscope operated at 200 kV. Atomic scale HAADF-STEM imaging and EDX investigations were performed using a monochromated and probe-corrected FEI Titan3TM G2 60–300 (S/TEM) microscope operated at 300 kV with an X-FEG high-brightness emission gun. The high-resolution images in STEM mode were recorded with a beam diameter of 0.1 nm and a current of 0.04 nA using the HAADF and dark field (DF) detectors. X-ray spectra were acquired by the SuperX detection system (Chemi-STEM™ technology) with a 120 mm acquisition area which reduces significantly the acquisition times. Elemental quantification of the EDX spectra was performed using the K-factor method [13]. The images and spectra were recorded by a Gatan Digiscan unit and Digital Micrograph software, and were corrected for dark current and gain variations.

The needle-like samples of a radius less than 50 nm for APT

analysis have been prepared by site-specific FIB milling in the Zeiss 1540 EsB CrossBeam® workstation. The FIB preparation method comprises many steps: (i) cutting the lamella of the eutectic including the area of interest with the interface between eutectic Al and eutectic Ge; (ii) welding the lamella to the micromanipulator; (iii) attaching the lamella to support pillars (pre-sharpened Mo tip) and welding it; (iv) cutting a portion of the tip (one lamella can be prepared into about five tips); (v) ion etching to achieve a desired shape of 50 nm apex radius, and (vi) finally cleaning up to remove Ga implantation with a low kV mode (2 kV). APT measurements were carried out in a local electrode APT instrument build at the University of Münster (now University of Stuttgart), Germany [14]. Field evaporation of atoms from the apex were performed by femtosecond UV laser pulses of 343 nm wavelength with a repetition rate of 200 kHz and a pulse energy of 60 nJ. APT analyses were performed at a tip temperature of 45 K in an ultra-high vacuum (10^{-8} Pa).

3. Results

3.1. SEM

Fig. 1 shows typical microstructures of Al–20Ge alloys without and with the additions of 0.2Y and 0.2Ca, respectively. The microstructure consists of primary aluminium, eutectic Ge and eutectic Al as well as $\text{Al}_2\text{Ge}_2\text{X}$ type (X: Ca, Y) intermetallic phases. All these phases show different contrasts when imaged with the SE detector. The eutectic Ge can be distinguished by its bright contrast. The intermetallic phases can be identified not only by their light grey contrast but also by their morphology, which is rod-like in two dimensions. Finally eutectic Al as well as the primary Al phase is imaged by dark grey contrast.

In the Al–20Ge alloy, eutectic Ge was observed as coarse bright lamellas and fibres, as shown in Fig. 1a and b. In the Al–20Ge–0.2Y alloy, a much finer eutectic Ge structure was observed when compared with the binary Al–20Ge alloy. A deeper insight in the eutectic Ge structure shows an interconnected network of eutectic Ge, as shown in Fig. 1c and d. Similarly, in the Al–20Ge–0.2Ca alloy, a much finer eutectic Ge structure was observed, as shown in Fig. 1e and f. However, in contrast to the Al–20Ge–0.2Y alloy, some spherical eutectic Ge particles were observed in the Al–20Ge–0.2Ca alloy. In addition, intermetallic $\text{Al}_2\text{Ge}_2\text{Y}$ and $\text{Al}_2\text{Ge}_2\text{Ca}$ phases were also observed, as marked by arrows in Fig. 1d and e. These intermetallic phases are similar to the $\text{Al}_2\text{Si}_2\text{Sr}$ phase existing in Al–Si based alloys [8,9].

3.2. TEM

To obtain structural and compositional properties of the eutectic Ge, different imaging TEM techniques were combined. Fig. 2 shows a bright field (BF) TEM image as well as a high resolution TEM (HRTEM) micrograph of a eutectic Ge particle in Al–20Ge alloy. Several parallel arranged {111} twin traces in Fig. 2b were observed in the eutectic Ge phase of a sample aligned with [011] zone axis parallel to the electron beam. Twin traces are marked by a white solid arrow. Accurate crystallographic information about the planar defects of twins and stacking faults can be revealed by electron diffraction [15]. Fig. 2c shows the corresponding selected area electron diffraction (SAED) pattern of Ge from Fig. 2b. This SAED pattern is typical of a diamond crystal in [011] zone axis orientation containing {111} planar defects of twins. The additional spots located at one third between the main diffraction spots along $\langle 111 \rangle$ directions represent the presence of {111} twins. This presence of twins along the Ge plates confirms the TPPE growth mechanism of eutectic Ge reported in Refs. [5,6].

Download English Version:

<https://daneshyari.com/en/article/7878160>

Download Persian Version:

<https://daneshyari.com/article/7878160>

[Daneshyari.com](https://daneshyari.com)