Hierarchical investigation of 3D microstructure at three different length scales – towards quantitative understanding of microstructure formation for the example of lightweight Al-Si alloys with complex morphology

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Abstract
Three dimensional microstructure characterizations at different scales play a key role for the quantitative understanding of the relationship between processing, microstructure and properties. However, it could not be fully exploited so far due to the lack of adequate 3D characterization techniques in all relevant scales. Recent progress in atom probe tomography, serial sectioning techniques combining FIB and SEM, as well as the submicron precision of mechanical serial sectioning has led to new insights into the evolution of microstructure of many engineering materials. We present such a hierarchical investigation for very complex microstructure morphologies in an important lightweight material for automobiles – the Al-Si alloys.

Keywords: FIB/SEM Tomography, Atom Probe Tomography, Serial Sectioning, Al-Si alloys

1 Introduction
Hypoeutectic Al-Si alloys are widely used cast materials in lightweight car structures, e.g. for most parts of the combustion engine, but also wheel rims and wheel suspensions. The microstructure of these alloys consists of primary solidified Al-dendrites and an interdendritic Al-Si eutectic. The eutectic phase consists of a complex, needle shaped Si-network embedded in an aluminium matrix. The microstructural constituents have very different properties, so that their three-dimensional shape and arrangement plays an essential role for the mechanical behaviour of the alloy, especially in terms of toughness and strain at fracture. In order to improve these parameters, the needle like Si structure can be changed towards a fine, coral like morphology with less curvature by a so called modification treatment with ppm-amounts of Sr. Although modification treatments are already widely used in industry, the microstructural mechanisms of modification are still under discussion and the materials potentially still far away from the optimum.

2 Experimental
In this work, the three dimensional microstructures of modified and unmodified Al – 7 wt% Si alloys have been analyzed by three different tomographic techniques in the micro, nano and atomic scale: By means of tomography based on mechanical serial sectioning, by FIB/SEM tomography as well as by atom probe tomography correlated with HR-TEM imaging. Why is the application of all these techniques useful and relevant?

(1) The coarse dendritic structure and shape and arrangement of the interdendritic eutectic, as well as shape and distribution of coarse intermetallic phases can well be studied in a mechanical sectioning process combined with light microscopy which delivers the largest field of view.
(2) The fine eutectic structure and its local morphology are analyzed by means of FIB/SEM tomography [1]. In the unmodified alloy, tomography reveals a plate like, well interconnected structure of the eutectic silicon (Figure 1, left), which - in a 2D section - seems to be needle shaped and partly isolated. The modified alloys shows highly branched Si colonies with a rounded shape (Figure 1, right).

Figure 1: Reconstruction from FIB/SEM tomography of the eutectic silicon in an unmodified AlSi7 alloy, approx. 70x100x40 µm³ (left) and reconstruction of an modified AlSi7 alloy, approx. 37x17x35 µm³ (right).

(3) In order to identify the role of the very few strontium atoms in the modification process, atom probe tomography combined with HR-TEM and STEM imaging was used to study distribution and segregation of Sr in the eutectic silicon [2]. Sr segregations of special morphologies were found (Figure 2) and could be correlated with the formation of the Si crystal lattice.

Figure 2: Reconstruction from atom probe tomography of the eutectic silicon of a Sr-modified AlSi7 alloy (300nm in length and 120nm in diameter). Al atoms are shown in green, Sr atoms in red, Si atoms (matrix) are not shown. Nanosized Al and Sr rich structures can be found inside eutectic silicon

3 Summary
The example of Al-Si cast alloys impressively shows, that the combination of different tomographic techniques at different length scales can lead to a better understanding of microstructure formation and its influence on the eutectic morphology and the global materials mechanical properties.

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References