学校编码：10384

学号：20720131150088

硕 士 学 位 论 文

多晶硅定向凝固过程典型杂质的迁移机理与分布规律研究

Study on the Transfer and Distribution Mechanism of Typical Impurities During Directional Solidification ofMulti-crystalline Silicon

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专业名称：工程硕士(材料工程)

答辩日期：2016年5月

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摘　要

定向凝固方法是去除多晶硅中典型金属杂质的有效途径。本研究通过实验探索，并结合热力学和动力学，研究多晶硅中的金属杂质、夹杂相杂质和气泡在定向凝固过程的迁移机理与分布规律；探究典型杂质的传输与分布规律、夹杂相杂质的形成机理以及气泡的形核和生长机理。主要研究结果如下：

（1）研究冶金级硅定向凝固过程铁杂质的传输与分布规律。实验发现铁杂质含量沿晶体生长方向并未完全符合Scheil 定理，而是保持恒定，达到某一凝固分数后快速递增；晶格与晶界的杂质比较，晶界处铁杂质含量比晶内的高出一个数量级；硅熔体对流对杂质传输与分布起到重要作用，使得杂质含量呈现出中间多、边缘少的规律。根据质量守恒定理，计算得到铁杂质有效分凝系数为2.98×10-4，溶质边界层有效厚度为4 mm。

（2）钙杂质赋存于原料中的化学状态对其在定向凝固过程的传输与分布有重要的影响。实验发现钙杂质不仅以固溶态赋存于原料中，还以夹杂相CaO 形式赋存。由于 CaO 在硅熔化和凝固过程仍保持难熔颗粒物状态，当CaO沉降后，包覆在固相硅中使杂质钙的含量偏高。CaO 在定向凝固过程的沉降行为从热力学角度得到了很好的揭示。

（3）硅原料中的夹杂相杂质 SiC、Si_3N_4 在多晶硅定向凝固过程沉降在硅锭底部，从热力学和动力学角度分析了沉降机理与规律，并发现SiC和Si_3N_4对多晶硅锭的少子寿命几乎无明显影响。

（4）石英坩埚在硅熔化的高温条件下会分解出氧，而氧会经坩埚涂层扩散至硅熔体，并与硅反应生成气态SiO。SiO 聚集并借助难熔物颗粒SiC等异质形核成气泡。从热力学角度探讨了气泡形核与生长的过程以及在固相硅中的分布。

关键词：多晶硅；定向凝固；杂质迁移

Abstract

Directional solidification is an effective method to remove typical impurities in multi-crystalline silicon. In the present paper, multi-crystalline silicon growth was carried out by directional solidification method. Transfer and distribution mechanism of metallic-impurities, insoluble inclusions and bubbles were investigated by experiments during the directional solidification of multi-crystalline silicon, as well as analysis from the perspective of thermodynamics and kinetics. The law of transfer and distribution of typical impurities, mechanisms of insoluble inclusions formation and mechanisms of nucleation and growth of bubble during directional solidification were studied in detail, and the main results of research were shown as follows:

(1) Law of transfer and distribution of iron impurity during the directional solidification of multi-crystalline silicon were studied. It was found that the content of iron kept constant and increased dramatically at last along crystal growth direction, but not completely meet the law of Scheil equation. Compared the content of iron in grain with the content of iron at grain boundary, the content at boundary was higher than that in grain with an order of magnitude. Convection in silicon melt played an important role in the transfer and distribution of impurities, resulting in the content of iron in the middle was higher than that in the edge. According to the law of mass balance, the effective distribution of iron was obtained as 2.98×10-4, and effective solute boundary layer was further got as appropriately as 4 mm.

(2) The chemical state of calcium impurity in the feedstock has a significant impact on the calcium removal during the directional solidification of multi-crystalline silicon. It was found that calcium not only existed as solid solution impurity, but also as inclusions, CaO, in the feedstock. Since CaO transfer as refractory particle during the melting and solidification procedure, it naturally

settled down at solid-liquid interface, and was engulfed at solid silicon at last. In consequence, the content of calcium was much higher than the normal position with an order of magnitude. The sedimentation behavior of CaO particle was thermodynamically analyzed in detail.

(3) Insoluble inclusions, SiC and Si3N4, as foreign materials in the feedstock, and they settled down at solid-liquid interface during directional solidification of multi-crystalline. The mechanism of sedimentation of insoluble particles and distribution were analyzed from the perspective of thermodynamics and kinetics. Meanwhile, it was found that SiC and Si3N4 did not have significant impacts on the minority carrier lifetime of multi-crystalline silicon ingot.

(4) Quartz crucible could break out free oxygen during directional solidification of multi-crystalline silicon, and oxygen diffused through coatings into melt silicon. Oxygen reacted with silicon to produce gaseous SiO. Gaseous SiO aggregated together and heterogeneously nucleated against insoluble particle, SiC, resulting in bubble. The nucleation and growth of bubble were thermodynamically analyzed in detail.

Keywords: Multi-crystalline silicon; Directional solidification; Impurity transfer

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