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博 士 学 位 论 文

大型定向凝固设备非均匀温度场提纯多晶硅研究

Research on the Non-uniform Temperature Field for Purifying Polysilicon in Large Directional Solidification Equipment

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摘　要

太阳能资源分布广泛并可无偿获取，使光伏发电成为新型能源的重要发展方向之一。因其发电组件具备建设周期短、安全可靠、布置灵活等优点得到了人们的青睐。硅作为光伏发电主要的基础材料，已占据市场90%以上的份额。冶金法提纯多晶硅具备投产建设速度快、提纯工艺流程短、成本较化学法低等优点，近年来发展迅速，受到国内外的广泛重视。

定向凝固是冶金法的核心。与中小型提纯相比，大型定向凝固的生产效率高，综合成本较低，但固液界面较难维持水平，晶粒不易保持垂直方向上的整齐排列，凝固和退火过程中裂锭的概率也较大，对温度场构建、设备用材、控制策略均提出了更高的要求。本文针对提纯体积达1000㎜×1000㎜×450㎜硅锭的定向凝固提纯过程展开研究，提出一种适用于大型定向凝固的环形加热和非均匀散热方法，构建非均匀温度场，在保持界面水平的前提下加强自然对流，提高提纯效率，同时降低热应力。该方法不仅可以降低设备造价和提纯成本，还能减少加热元件对硅熔体的污染。本文以固液界面、硅温度场、流动场、热应力、凝固速率变化规律为研究对象，阐述该温度场的原理，并通过实验验证对提纯多晶硅的效用。具体内容如下：

定向凝固过程中，固液界面的形态对多晶硅晶粒尺寸和排列情况有直接的影响。水平上升的固液界面，不仅能提高杂质元素提纯效率，还能增加少数载流子寿命和电阻率，提高光伏电池的光电转换效率。通过数学建模求得界面形态的通用表达式，提出非均匀温度场定向凝固的理论基础，证明界面形态主要受坩埚侧壁热交换情况影响。通过实验研究不同热交换情况下的固液界面形态和硅锭质量指标。实验表明，水平界面的晶粒尺寸最大，微凸界面的杂质含量最低。

加热元件环形布置形成的温度场可加强自然对流，使富集在固液界面上的杂质加速扩散至液相表面，提高提纯效率并保持界面水平。通过建立温度场和流动场的数学模型，获得解析表达式，并采用数值研究验证和补充理论推导结果，阐述环形加热的原理，同时提出加热控制方法。

降低热应力是提高硅锭质量的直接手段。定向凝固和退火过程中如出现过大的热应力，将导致多晶硅缺陷增加，降低少数载流子寿命。通过研究凝固过程中硅锭热应

力分布和变化情况，提出一种非均匀散热方法用以疏散热应力。相对于固相底部均匀散热的常规做法，该方法可降低平均应力约30%，并可采用较高的凝固速率进行晶体生长。另外，还通过理论推导求得退火过程的通用表达式和函数图像。
以大型定向凝固设备作为实验平台，构建非均匀温度场，采用理论研究获得的温度场控制规律为基础进行实验。所获硅锭尺寸为1000mm×1000mm×450mm，晶粒排列齐整，平均晶粒尺寸≥4mm2；硼（B）、磷（P）含量达到国标GB/T25074-2010规定的1级品等级，主要金属杂质指标稳定在1~2级品等级之间；少数载流子和电阻率指标则略低于主流GT炉产品。通过实验还修正了控制曲线。

关键词：多晶硅；定向凝固；提纯；温度场

Abstract

Solar energy resources are widely distributed and can be obtained easily, so that the photovoltaic power generation has become an important direction of development of new energy. It has been widely accepted by people because of its short construction period, safety and reliability, flexible placement. Silicon is the main basis of photovoltaic power generation, has accounted for more than 90% of the market share. Due to the advantages of fast construction, short purification process, and low cost in the process of production and construction, the development of metallurgical technology for purifying polysilicon has been developing rapidly in recent years.

Directional solidification (DS) is the core of metallurgical process. Compared with tiny and medium purification, large directional solidification has high productivity, low cost, but the solid-liquid interface is difficult to maintain level, grain growth is not easy to keep perpendicular to the direction of neatly arranged, crack probability of the ingot is larger in solidification and annealing process, the higher requirements are put forward on temperature field, material, control strategies. The purification process of volume 1000mm×1000mm×450mm silicon ingot is researched, put forward a suitable to large directional solidification by annular heating and non uniform cooling method, constructing the non-uniform temperature field, is designed to keep the interface level, enhance the natural convection, reduce the thermal stress while reducing equipment and purification cost, reduce the pollution come from heating device. The solid-liquid interface, silicon temperature field, flow field and thermal stress, solidification rate changes are the research objects, the utility of purifying polysilicon and verified by theoretical deduction and the experiment.

In the process of directional solidification, the morphology of the solid-liquid interface has a direct influence on the grain size and the arrangement of the polysilicon. The solid-liquid interface can not only improve the efficiency of impurity elements, but also increase the lifetime of minority carriers and resistivity, and improve the photoelectric conversion efficiency of photovoltaic cells. The general expression of the interface form is obtained by mathematical modeling. The theoretical basis of the non-uniform temperature field oriented solidification is put forward. The solid-liquid interface is researched in the case of different heat exchange through the experiment. The experimental results show that the grain size of the horizontal interface is the largest and the impurity content of the micro convex interface is the lowest.

The temperature field and the natural convection caused by the annular arrangement of the heating device can accelerate the diffusion of the impurities in the interface to the liquid phase surface, and improve the efficiency of the purification. Through establish the mathematical model of temperature field and flow field, the analytical expressions are obtained. Using numerical studies verify and complement the theoretical results, and expounds the principle of annular heating. At the same time, the heating control method is put forward.

Reducing stress concentration is a direct method to improve the quality of silicon ingot. The thermal stress will lead to the increase of the silicon defect, and reduce the minority carrier lifetime in the process of directional solidification and annealing,. Through the research of directional solidification process in silicon ingot thermal stress distribution and variation, a non-uniform heat dissipation method is proposed for sparse heat stress. The average stress of the method can be reduced to about 30%, and can be used for the crystal growth with higher solidification rate. In addition, the general expression and function image of the annealing process are obtained by theoretical derivation.

Based on the experimental platform of large directional solidification equipment, the non-uniform temperature field is constructed, and the experimental results are based on the experimental platform. The ingot size reaches 1000mm × 1000mm × 450mm, and the arrangement of grains is neat, the average grain size is greater than or equal to 4mm2, B, P content reached 1 grade level base on GB/T 25074-2010, the metal impurities content is in 1~2 grade level, the lifetime of minority carrier and resistance rate is slightly lower than GT furnace. The control curves were corrected by experiments.

Keywords: polysilicon; directional solidification; purification; temperature field

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