

Investigate the Influence of Boron Removal by Multi-Fluxes Addition in Metallurgical Grade Silicon

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ABSTRACT

The main purpose of refining the solar-grade poly-silicon studies are following the direction of cost down, reducing air pollution and energy consumption. Therefore, this research adopted the process of oxide fluxes with low cost and low pollution, to eliminate the boron impurities from metallurgical grade silicon. This research program will formulate different types of multi-fluxes and process with Induction Heating Method for slag smelting, then, analyze the content of boron impurity from silicon ingots. Therefore, we can figure out the relations between the types and quantity of fluxes, isothermal holding time, instalment adding times, and the residual boron impurity. From experimental results shown that the calcium oxide will decompose easily and provide more oxygen than alumina, and the oxygen will react with boron to form the boron oxides floating on silicon liquid and separated it. During the smelting of refining, increase the quantity of flux and the holding time, do benefit the decomposed oxygen reacted with boron to form oxides and help to remove the boron impurity. Also increasing the instalment adding times, it also helps the boron impurity continuing removal. If add whole quantity of flux at a time, will not be able to adequate and effective reacting with silicon liquid. However, further increasing the number of adding times, the removal ratio of boron impurity still having a certain limit. If wish to further reduce the boron content, the amount of flux added is required to achieve the goal of low-boron content poly-silicon. Based on the overall experimental results suggested that the best combination of multi-fluxes is 5SiO₂-4CaO-1Al₂O₃, and using 120 wt.% flux divided into five times' addition, with 10~15 minutes smelting holding time, should have the most significant results of boron impurity removal in this metallurgical grade silicon. Boron content can be reduced from 8.4 ppm. to 1.7 ppm., the removal rate reached up to 79%.

Keywords : Metallurgical grade silicon、Multi-Fluxes、Boron impurity、Refining

Table of Contents

封面內頁 簽名頁 中文摘要.....	iii
ABSTRACT.....	iv 誌
謝.....	v 目錄.....
錄.....	xix 表目錄.....
第一章 前言.....	1 第二章 文獻回
顧.....	3 2.1 砂純化簡介.....
2.1.1 砂資源及提煉.....	3 2.2 砂的應用領
域.....	4 2.2.1 單晶砂應用領
域[5].....	4 2.2.2 多晶砂應用領域[5].....
2.2.3 單晶與多晶砂差異[5].....	5 2.3 砂純化製
程.....	6 2.3.1 三氯矽烷氫還原法(西門子法)[7].....
) [7].....	6 2.3.2 流體床反應法[7].....
狀沉積法[6].....	7 2.3.3 管
法.....	8 2.3.5 定向凝固.....
造渣原理.....	10 2.4.1 造渣純化
矽.....	11 第三章 實驗方法.....
3.1 實驗材料.....	23 3.2 實驗規
劃.....	23 3.3 實驗步驟.....
3.4 造渣劑的選擇.....	26 3.4.1 造渣劑的配製比例和添加
量.....	26 3.4.2 造渣劑添加量增加.....
3.4.3 持溫時間增長.....	29 3.4.4 造渣劑分次添
加.....	29 3.5 實驗器具與設
備.....	30 3.5.1 熔煉坩堝.....

3.5.2 熔煉狀態.....	31	3.6 試件取樣分
析.....	32	3.6.1 消化、取樣與分
析.....	33	3.7 感應耦合電漿放射光譜
儀(ICP-OES).....	34	第四章 實驗結果分析與討論
論.....	37	4.1 實驗用矽料與造渣劑之ICP成份分析
析.....	38	4.2 二元造渣劑化驗分析
4.2.1 二氧化矽與氧化鈣配比變動分析.....	38	4.3 三元造渣劑化驗分析
析.....	39	4.3.1 固定60%二氧化矽含量之複合式造渣劑化驗分析
析.....	39	4.3.2 固定50%二氧化矽含量之複合式造渣劑化驗分析
析.....	42	4.3.3 固定40%二氧化矽含量之複合式造渣劑化驗分析
析.....	44	4.3.4 固定30%二氧化矽含量之複合式造渣劑化驗分析
析.....	46	4.3.4 固定40%氧化鈣含量之複合式造渣劑化驗分析
析.....	48	4.3.5 固定30%氧化鈣含量之複合式造渣劑化驗分析
析.....	50	4.3.6 固定20%氧化鈣含量之複合式造渣劑化驗分析
析.....	52	4.3.8 固定10%氧化鋁含量之複合式造渣劑化驗分析
析.....	57	4.4 造渣劑最佳配比探討.....
渣劑添加量與矽錠硼雜質含量關係.....	59	4.5 造
去除之影響	60	4.6 熔融持溫時間與造渣劑添加量對硼元素
影響.....	62	6.4.7 造渣劑分次添加對硼元素去除之影
響.....	65	第五章 結論.....
獻.....	69	參考文
	71	

REFERENCES

- [1] 郭博堯， “全球化石能源危機時代與我國所面臨挑戰”，永續(研)091-029號。
- [2] 陳子秦， “太陽能電池產業製程及污染防治簡介”，財團法人台灣產業服務基金會，2009年。
- [3] 袁華堂， “新能源材料”，五南圖書出版股份有限公司，2004年。
- [4] 王旭昇， “太陽能光電產業二”，台灣工業銀行，2007年。
- [5] 香港矽片回收網， <http://www.gdfpw.com/show.asp?id=271>。
- [6] 梁智恆， “太陽能級多晶矽製程技術探討”，DIGITIMES中文網，2009年。
- [7] 新餘日報，2008年11月1日。
- [8] J. Dietl, "Hydrometallurgical Purification of Metallurgical Grade Silicon," "Solar Cells", pp.145~154, 1983.
- [9] 黃瑩瑩，郭輝，黃建明，沈樹群，精煉法提純冶金矽至太陽能級矽的研究發展，“動能材料”，2007年第9期。
- [10] 黃平平，吳浩，傅翠梨，張蓉，李錦堂，羅學濤，“CaO-SiO₂基造渣劑除硼的研究進展”，材料導報A:綜述篇，第25卷，2011年第10期。
- [11] 伍繼君，戴永年，馬文惠，楊斌，劉大春，王燁，魏奎先，“冶金級矽氧化精煉提純制備太陽能級矽研究進展”，真空科學與技術學報，第30卷，2010年第1期。
- [12] 王新國，丁偉中，沈虹，張靜江，“金屬矽的氧化精煉”，中國有色金屬，2002年第4期。
- [13] Jijun Wu, Wenhui Ma, Binjie Jia, Bin Yang, Dachun Liu, Yongnian Dai, "Boron removal from metallurgical grade silicon using a CaO – Li₂O – SiO₂ molten slag refining technique", Journal of Non-Crystalline Solids. 358, pp.3079-3083, 2012.
- [14] Cai Jing, Li Jin-tang, Chen Wen-hui, Chen Chao, Luo Xue-tao, "Boron removal from metallurgical silicon using CaO-SiO₂-CaF₂ slags", Trans. Nonferrous Met. Soc. China 21, pp.1402-1406, 2011.
- [15] Zhao Ding, Wenhui Ma, Kuixian Wei, Jijun Wu, Yang Zhou, Keqiang Xie, "Boron removal from metallurgical-grade silicon using lithium containing slag", Journal of Non-Crystalline Solids. 358, pp.2708-2712, 2012.
- [16] Da-wei Luo, Ning Liu, Yi-ping Lu, Guo-liang Zhang, Ting-ju Li, "Removal of boron from metallurgical grade silicon by electromagnetic induction slag melting", Trans. Nonferrous Met. Soc. China 21, pp.1178-1184, 2011.