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ScienceDirect

Energy Procedia 136 (2017) 53–59

Energy Procedia

www.elsevier.com/locate/procedia

4th International Conference on Energy and Environment Research, ICEER 2017, 17-20 July 2017, Porto, Portugal

Recovery of silicon carbide from waste silicon slurry by using flotation

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Abstract

The amount of waste silicon slurry increases as the wafer production raises. The slurry is generally disposed using incineration or land-filling. Separating high-purity SiC from waste silicon slurry can reduce costs for enterprises and assist in waste reuse and recycling. In this study, flotation was applied to separate SiC and Si from waste silicon slurry through hydrophilicity and hydrophobicity of the particle surface. By controlling the concentration of hydrofluoric acid and the oxidation reduction potential of the two stages flotation, the SiC can be separated from Si. The optimal condition of first stage flotation is 0.8 mol/L of HF at -400 mV, while 0.6 mol/L of HF and -300 mV was applied in the second stage. Under these conditions, approximately 52.8% of SiC was recovered with the grade of 98.1 %.

Keywords: Flotation; recycling; silicon carbide; silicon; waste silicon slurry

1. Introduction

The amount of waste silicon slurry increased as the wafer production raised since the wafer slicing primarily accompanied cutting of silicon ingots into thin wafers. This process requires a steel wire saw, grinding media, and coolant and thus caused the impurities in the slurry. According to Taiwan Environmental Protection Administration (EPA), approximately 20,000 tons of waste silicon slurry was produced annually in the solar industry in Taiwan. The

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10.1016/j.egypro.2017.10.281

waste silicon slurry was generally disposed using incineration or land-filling and therefore leads to environmental damage and wasting substantial resources. The mainly components of the slurry are silicon carbide (SiC) and silicon (Si) mixed with a small amount of iron and alcohol compounds like PEG, PG and DEG (T. Surek, (2005)). It is expected that SiC and Si can be recycled from the waste slurry and reused in the related industry such as wafer production and steel casting to reduce the costs and particularly protect the environment.

Waste silicon slurry can be separated using physical, chemical methods or flotation. Physical separation mainly includes centrifugation, electrical field and magnetic separations. Wang et al. (2009) used heavy-fluid high-gravity centrifugation with temperature treatment and directional solidification to separate SiC from Si recovering 86% of SiC with 87% of grade. Lin and his team (2010, 2010) combined centrifugation with phase-transfer separation to recover Si from silicon slurry. The phase-transfer separation improved the grade of recovered Si from 90.8% to 99.1%. By controlling the surface potential through centrifugation, Lin et al. (2013) recycled 90.8% of SiC with 95.2% of grade. Wu and Chen (2009) separated SiC and Si through different particle velocity under different electrical field. The grades of the recycled SiC and Si were 90% and 92.4%, respectively. Tsai et al. (2011, 2013) investigated the efficiency of electrical field separation under different electric field strength, baffle plate. The grade of SiC and Si could be improved to 95.2% and 92.9%, respectively. For magnetic separation, Sergii et al. (2014) experimented high-gradient magnetic separation combined with hydrocyclone and sedimentation. With different specific weight, 95% grade SiC was recycled from the slurry. Nishijima et al. (2003) removed iron from waste silicon slurry by employing super-conduction magnetic separation and recycled SiC by centrifugation. The recovery efficiency of SiC was 80%. The main chemical separation method is liquid-liquid extraction. Wei et al. (2015) separated SiC and Si via different hydrophobic characteristics of particle surfaces, and established a silicon regression model from the results of the research.

The influences of activator, collector and frother on flotation were investigated separately (Iskra, 1997, Lin et al., 2002, Sahoo et al., 2016). Shibata (2006) conducted flotation separation using various cationic surfactants to separate SiC and Si. Instead of directly separating the waste silicon slurry, Shibata recovered SiC through flotation after silicon oxidation into SiO_2 and obtained a 99.7% grade SiC with a recovery efficiency of 96.7%. As regards previous research, the hydrofluoric acid (HF) dissolved the SiO_2 layer on the surface of particles in waste silicon slurry (Eq. 1) and made SiC a hydrophilic polar sinking mineral (Larsen and Kleiv, 2016). In addition, the floatability of silicon in the slurry increased since the Si-F bonding on the surface of Si particles (Ernsberger, 1960, Guo et al., 2005, Lin et al., 2008).

$$SiO_2 + 6HF \rightarrow H_2(SiF_6) + 2H_2O \tag{1}$$

This study focus on the flotation separation of SiC and Si rather than SiO_2. In order to raise the recovery efficiency and grade of recovered SiC, a surface activator was applied. The concentration of surface activator was investigated to achieve the optimal condition leading to high recovery efficiency and grade. The oxidation reduction potential (ORP) of the system was also controlled to verify the optimal potential for silicon removal. High grade of SiC can be recovered as concentrate by controlling the oxidation-reduction potential and the hydrophobicity of Si particles.

2. Experimental

2.1. Composition and properties

The sample for this study is the powder dried from the waste silicon slurry produced from the wafer fabrication. The SiC grade of the waste silicon slurry was determined by acid leaching, the waste silicon slurry was leached using hydrofluoric, nitric, sulfuric, and hydrochloric acids, after which the sample was filtered and subjected to high-temperature drying. An X-ray diffractometer (XRD, Bruker AXS-D8A) was used to analyze the crystalline phase of the sample; the particle size distribution was determined using a laser particle size distribution analyzer (LS, Beckman Coulter LS230); and the particle morphology was observed using a scanning electron microscope (SEM, Hitachi S3000-N).