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Assessment of the recovery of photovoltaic cells cutting fluid by chemical pretreatment and ultrafiltration

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ABSTRACT

The main objective of the work was to regenerate a cutting fluid HS20 used in the manufacturing of silicon wafers. Centrifugation at ambient temperature is initiallyconsidered for the treatment of the cutting fluid HS20. However, the slurry being heavily loaded with mineral colloids, tests conducted following the use of this process, have proved its efficiency to be low. Indeed, the best results for colloidal matter abatement have never exceeded 30%. By contrast, an ultrafiltration through a polyethersulfone membrane with a cutoff of 1 kDa shows excellent efficiency and affinity towards the fluid (HS20) to be considered, allowing its full recovery by maintaining its original cutting fluid characteristics. However, this process does present some drawbacks. A strong resistance to flow across the membrane of up to 60% of the total resistance is observed and a drop in permeation flux of about 90% are observed. Given these results, reinforcement of ultrafiltration, under the same operating conditions, by chemical pretreatment is considered. Chemical pretreatment with ultrafiltration offers better regeneration efficiencies under same flow conditions through the membrane as compared to an ultrafiltration process. Indeed, the fouling index is significantly reduced to around $153 \times 10^{3+}\,\mathrm{s/L^2}$ and a permeation flux comparable to that observed for virgin cutting fluid (HS20) is obtained.

Keywords: Cells; Cutting fluids; Regeneration; Photovoltaic; Pretreatment; Ultrafiltration

1. Introduction

The photovoltaic (PV) industry is going through a rapid phase of growth. In 2006 alone, the global PV production was over 2 GW. The majority of PV cells are made of silicon, which is mainly produced during the energy-intensive Siemens process. As per the current status, the wafer shares more than 65% of the

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cost of solar cells, but on the other hand, more than 40% of the high-purity silicon is wasted during wafer slicing [1].

The manufacturing of photovoltaic cells involves a large number of complex and highly delicate processes including silicon growth, oxidation, doping, photolithography, etching, stripping, dicing, metallization, planarization, cleaning, etc. There are several types of organic and inorganic compounds involved in the manufacturing processes of cells. Some of the steps in the sequence are wafer backgrinding, sawing, die attachment, wire bonding, encapsulation, electroplating, trim and form, and marking [2]. Cutting fluids are a viscous slurry consisting of solid silicon carbide and fluid glycol. Silicon carbide is used as a sawing agent in the sawing process of wafers, where it is applied to the wire saw in order to provide to it the necessary specific hardness to saw wafers. The glycol acts a substrate for as well well as a coolant for the silicon carbide [3].

The effluent of cutting fluids called silicon kerf is formed as a slurry along with the cutting fluid, (usually polyethylene glycol) and other impurities, mainly from the broken particles of the SiC abrasive and iron from the wire. It has a non-negligible environmental and economic impact associated with the manufacturing process of PV systems. There are only a few published examples of technologies relating to the separation and purification of silicon powder kerf, and many of these lack significant detail [4]. During the cutting process, they carry silicon and iron particles respectively from the base material and sawing wire. Dumping of such waste in the environment is subject to specific regulations regarding disposal of hazardous waste, which depends on special industrial waste plans [5]. They must not be abandoned or burned in open air [6]. Because of their polluting nature, it is recommended to collect, concentrate and regenerate them. The separation of the solvent and particles is possible at about 80% and the rate of reuse can exceed 80% [7,8].

The regeneration of cutting fluids via simplest and least expensive processes should allow the reuse of used fluid through the recovery of its lubricating characteristics, thus reducing virgin fluid consumption while preserving the environment.

2. Experimental

2.1. Material and methods

The Amicon ultrafiltration cell 8200 is used for tests of solid–liquid separation (Fig. 1). The tested membrane, provided by Pall Company, is made of polyether sulfone with a cutoff of 1 kDa. The cutting fluid used for all tests is provided by the Unit of Silicon Technology Development of Algiers (UDTS). Used fluid is an average sample of the concentrate recovered within the manufacturing unit of silicon bricks. The cutting fluid used (slurry) is mainly composed of HS20 loaded with SiC and iron wire waste that are produced during the sawing process [9–12]. The pH of the slurry is around 6.9 and the whole operation is conducted at room temperature. The organic solvent used is dichloromethane of industrial quality provided by Panreac. The pretreatment performed consist in mixing the slurry with dichloromethane in a proportion of 10% followed by vigorous stirring for a minute and then settling. The resulting liquid phase is recovered and is made to undergo frontal

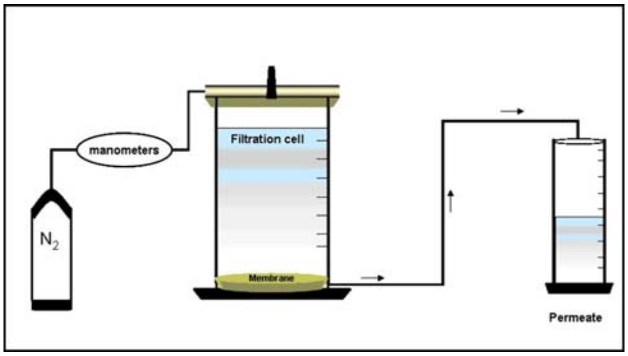

Fig. 1. Simplified diagram of the device for filtration.

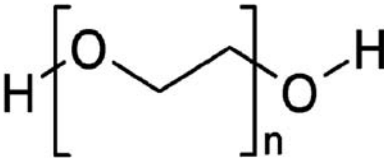

Fig. 2. Structure of HS20.

Table 1
Physicochemical characteristics of the HS20

Chemical proprieties	
Molecular formula	$C_{2n}H_{4n+2}O_{n+1}$, $n = 8.2$ to 9.1
Molecular weight	380–420/mol
Density	1.128 g/cm^3
Melting point	4–8 ℃
Viscosity	90.0 cSt at 25 ℃, 7.3 cSt at 99 ℃

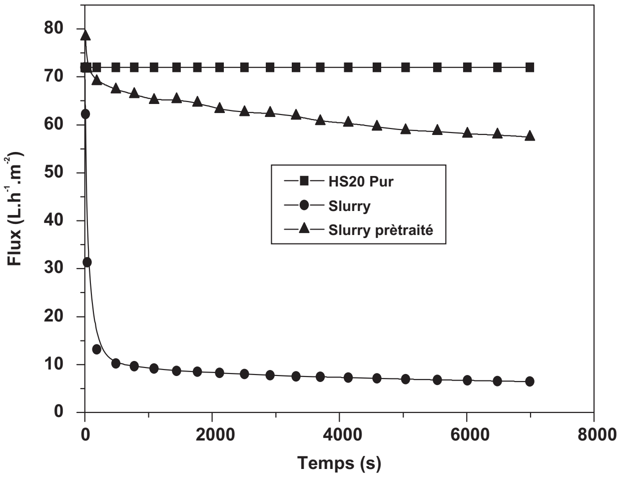

Fig. 3. Permeation flux vs. time at $p = 1$ bar.

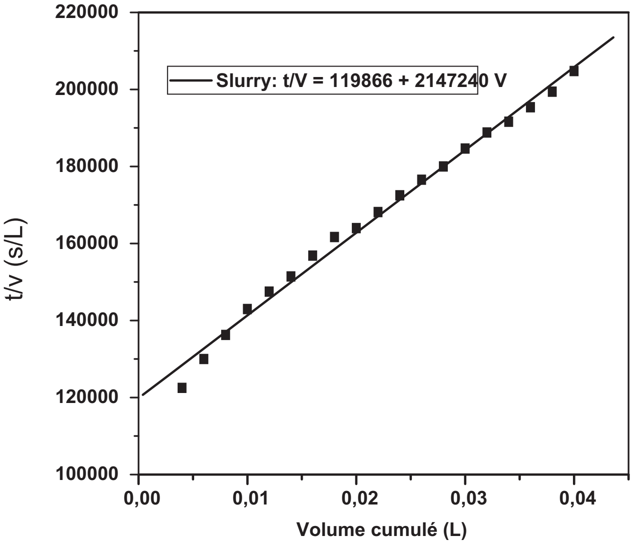

Fig. 4. t/v vs. v at $p = 1$ bar.

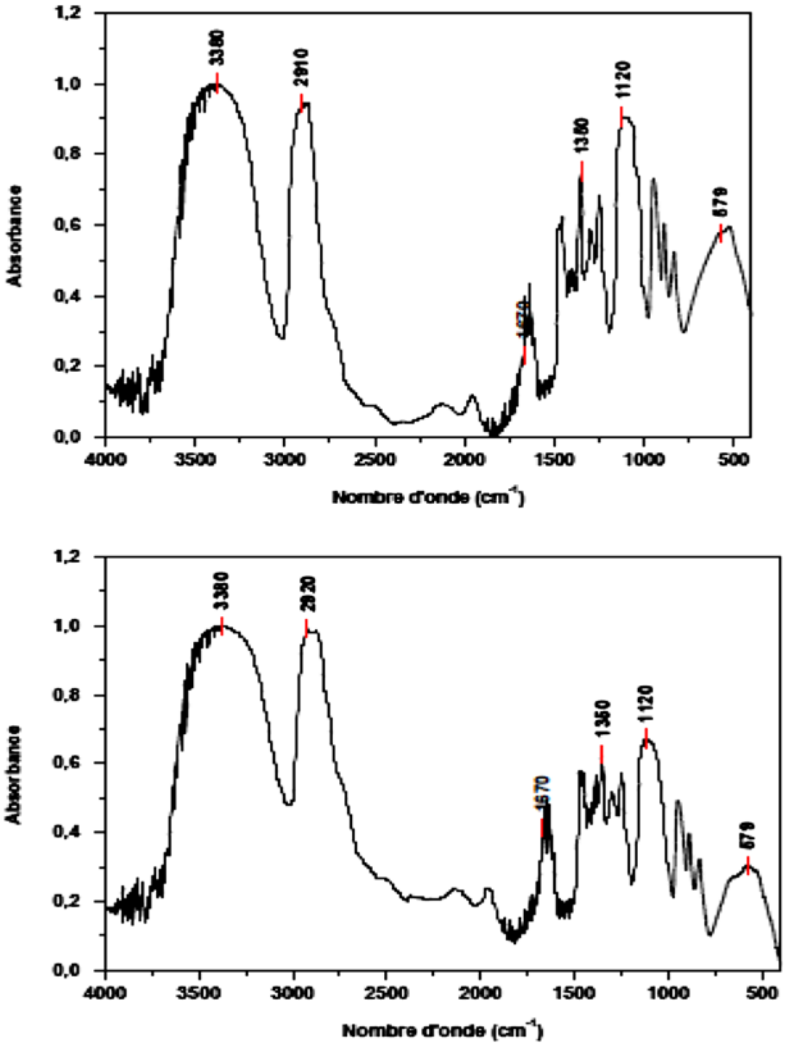

Fig. 5. FTIR spectrum for comparison of the regenerated oil and virgin oil.

ultrafiltration through a polyethersulfone membrane of 1 kDa. The comparison of the recovered fluid and virgin fluid is performed through an analysis made using infrared spectrophotometry. Table 1 represents the different physical and chemical properties of HS20, and the chemical structure is presented in Fig. 2.

3. Results and discussion

Fig. 3 exhibits the variation of permeate flux as a function of time at a constant pressure of 1 bar for pure HS20, the slurry and the slurry pretreated by dichloromethane. It appears that the flow of pure HS20 remains constant throughout the operation, indicating a macromolecule size of less than 1 kg Da. On the other hand, ultrafiltration of used cutting fluid reveals a rapid decrease in permeate flux right after the initiation of the filtration, eventually reaching a steady state that is maintained throughout the ultrafiltration and marked by a relative permeation flux decrease of up to 90%. For this case, the representation of t/v vs. the cumulative volume identifies a cake filtration mechanism (see Fig. 4). The curve indicates a non-negligible fouling index, with computations providing an Modified Fooling Index (MFI) of $2147 \times 10^{3+}\ \mathrm{s/L^2}$. Furthermore, Fig. 1 shows that under the same operating conditions, the values of permeation flux for the chemically pretreated cutting fluid are reasonably comparable to those obtained for pure HS20, with a maximal relative decrease of about 15%. For this case, the same method described above provides a much lower MFI of $153 \times 10^{3+}\ \mathrm{s/L^2}$. Moreover, comparison of the infrared spectra which shows (Fig. 5) a striking similarity between pure HS20 and the fluid obtained after ultrafiltration reinforced by a chemical pretreatment with dichloromethane indicates excellent regeneration.

4. Conclusion

The main objective of this work was to regenerate a cutting fluid HS20. It is used in the cutting of silicon ingots into thin slabs at the UDTS. Such a regeneration presents a double interest, economical and environmental, by reducing the consumption of virgin cutting fluid and wastage of highly polluting slurry. The present preliminary study demonstrates that ultrafiltration reinforced by a chemical pretreatment based on dichloromethane is a very feasible method for regeneration of the cutting fluids studied.

References

[1] T.Y. Wang, Y.C. Lin, C.Y. Tai, R. Sivakumar, D.K. Rai, C.W. Lan, A novel approach for recycling of kerf loss silicon from cutting slurry waste for solar cell applications, Journal of Crystal Growth (2008) 3403–3406.

[2] Fatehah Mohd Omar, Nik Norulaini Nik Abdul Rahman, Anees Ahmad, COD reduction in semiconductor wastewater by natural and commercialized coagulants using response surface methodology, Water Air Soil Pollut. 195 (2008) 345–352.

[3] N. Drouiche, M.W. Naceur, T. Ouslimane, H. Boutoumi, N. Aitmessaoudene, R. Henniche, M. Dziril, Preliminary Study of the Regeneration of Photovoltaic cells Cutting Fluid by Ultrafiltration Reinforced by a Chemical Pretreatment, Economic Sustainability and Environmental Protection in Mediterranean Countries through Clean Manufacturing Methods, Advanced Training Course: Economic Sustainability and Environmental Protection in Mediterranean Countries through Clean Manufacturing Methods (NATO Course Ref no: 984226), Huelva, 2011.

[4] Final Report on EC Project, RE-Si- CLE, “Recycling of Silicon Waste from PV Production Cycle”, Project funded by the European Community under the “Competitive and Sustainable Growth Programme”, 2002 to 2004, Project No. NNE5-2001-00175.

[5] M.J. de Wild-Scholten, E.A. Alsema, E.W. ter Horst, M. Bächler, V.M. Fthenakis, A cost and environmental impact comparison of grid-connected rooftop and ground-based PV systems, in: 21th European Photovoltaic Solar Energy Conference, Dresden, Germany, 2006.

[6] E.A. Alsema, M.J. de Wild-Scholten, V.M. Fthenakis, Environmental Impacts of PV Electricity Generation – A Critical Comparison of Energy Supply Options, in: 21th European Photovoltaic Solar Energy Conference, Dresden, Germany, 2006.

[7] Décret n°2002-540 du 18 avril 2002 relatif à la classification des déchets [Decree No. 2002-540 of 18 April 2002 relating to the classification of waste]. Journal officiel du 20 avril [Official Journal of 20 April] (2002).

[8] Décret n°77-254 du 8 mars 1077 relatif à la réglementation du déversement des huiles et lubrifiants dans les eaux superficielles, souterraines et de mer [Decree No. 77-254 of March 8, 1077 relating to the regulation of spill of oils and lubricants in superficial waters-75 they, ground and sea]. Journal officiel du 19 mars [Official Journal of 19 March] (1977).

[9] D. Ouadjaout, Y. Gritli, H. Rahab, A. Manseri, I. Hmadas, A. Ahmanache, R. Bendir, F. Kerkar, M. Boumaour, Growth by the heat exchanger method of multi-crystaline silicon ingot, in: Proceedings of the Twentieth European Photovoltaic Solar Energy Conference, Barcelone, Spain, 2005, pp. 6–10.

[10] H.A. Eltawahni, M. Hagino, K.Y. Benyounis, T. Inoue, A.G. Olabi, Effect of CO_2 laser cutting process parameters on edge quality and operating cost of AISI316L, Opt. Laser Technol. 44 (2012) 1068–1082.

[11] Toshiro K. Doi, Ioan D. Marinescu, Syuhei Kurokawa, Advances in CMP polishing technologies, Chapter 3 – The current situation in ultra-precision technology—silicon single crystals as an example, Adv. (2012) 15–111, doi: 10.1016/B978-1-4377-7859-5.00001-6.

[12] Toshiro K. Doi, Ioan D. Marinescu, Syuhei Kurokawa, Chapter 4 - Applications of ultra-precision CMP in device processing, Adv. CMP Polis. Technol. (2012) 113–228.