

The Preparation of Color Glasses Using the Solid Waste Generated in Crystalline Silicon Cutting Process

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Abstract—Solid waste generated in crystalline silicon cutting process (referred to as the silicon waste) was used to prepared color glasses. Silicon carbide and silicon in the silicon waste acted as silicon source and iron fragments as coloring agent. All components in the waste were fully used by vitrification of the solid waste with addition of Na₂CO₃, CaO, etc. The highest content of the silicon waste was 44.97% in the glass batch. The prepared color glasses can absorb part of visible light and ultraviolet light.

Keywords—cutting of crystalline silicon; solid waste; color glass; absorption of ultraviolet light

I. INTRODUCTION

Silicon waste is generated during the cutting process of crystalline silicon. The waste mainly contains silica carbide, crystalline silicon, iron and its oxide, polyethylene glycol (PEG) [1-2]. There are many reports on the recycling of SiC and PEG in the waste. PEG is collected by dehydrating or distilling the liquid after solid-liquid separation of cutting slurry. Afterwards, the solid is dissolved in acid to remove iron and Si, SiC fine powder is left behind [3-7]. The recovery of crystalline Si is still a challenge. Alternatively, the silicon waste was used for the preparation of ceramics, building materials. The above studies provided directions for the reuse of the silicon waste. However, these processes are complex, high cost, time-consuming, and the waste cannot be fully utilized. Therefore, it is necessary to find a new recycling method for this silicon waste.

In this work, silicon waste was used for the preparation of color glasses. All components in the silicon waste were used without complex separation and purification. The prepared color glasses have high additional value.

II. EXPERIMENTAL SECTION

Chemical composition of the silicon waste is shown in Table I. Sodium carbonate, calcium oxide and other raw materials were analytical reagents. Silicon waste was ground in a planetary ball mill with 250rpm for 30min, and then was sieved to pass a 200 mesh sieve. In some cases, the waste was pretreated by calcined at 350 °C for 2h, and then at 1500 °C for 2h.

TABLE I. CHEMICAL COMPOSITION OF THE SILICON WASTE

Component	SiC	Si	Fe	Al ₂ O ₃	CuO	CaO	Un-known
wt %	62.51	27.49	8.58	0.38	0.31	0.25	0.48

The designed chemical composition of the glasses is shown in Table II. The preparation procedures were as follows. First, the silicon waste and other raw materials were weighed and uniformly mixed to form glass batches. The glass batches were melted in silica crucibles in an electrical furnace at 1350 °C-1550 °C for 2h. The melts were directly poured into a preheated steel plate for forming. Finally, the bulk glasses were annealed at 600 °C for 2h.

TABLE II. DESIGNED COMPOSITION OF THE GLASSES

The NO. Glass	Designed composition						
	SiO ₂	Na ₂ O	CaO	Fe ₂ O ₃	Al ₂ O ₃	MgO	else
1	66.08	16.60	9.06	1.89	1.51	3.02	1.84
2	57.22	20.95	11.43	2.38	1.90	3.81	2.31
3	55.87	20.47	11.16	4.65	1.86	3.72	2.27
4	54.10	21.47	11.71	4.50	1.95	3.90	2.37

The X-ray diffraction analysis (D8 ADVANCE, Bruker, Germany) was used for identifying the crystalline phase in the silicon waste before and after the pretreatment. The density of bulk glasses were measured by the Archimedes method using a density analytical balance (FA1004J, Xu hui, China). The spectra of the colored glasses were recorded on a visible spectrophotometer (721, Jing hua, China).

III. RESULTS AND DISCUSSION

A. The XRD Analysis of the Silicon Waste

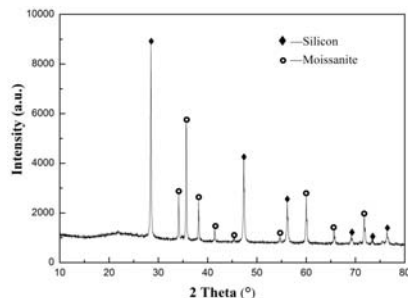


Figure 1. XRD pattern of the silicon waste

Fig. 1 shows the XRD pattern of the silicon waste. The main crystalline phases are SiC (JCPDF card No. 74-1302) and Si (JCPDF card No. 27-1402). The relative contents Si and SiC are respectively 30.54% and 69.46%, calculated by internal standard method using Si as the standard sample.

Crystalline silicon can be melted above 1410 °C [8]. However, the melting point of crystalline silicon carbide is above 2700 °C, which is difficult to melt alone.

B. The Formation of Glass

Silicon waste alone cannot be melt into a glass due to the refractory SiC. It should be mixed with other raw materials. From glass 1 to 3, the content of the silicon waste were 13.04%, 16.21%, 35.26% in the batch, respectively. The melting temperatures for preparing the glasses were 1350 °C, 1450 °C, 1500 °C, respectively, which are increasing due to the differences in the content of the refractory SiC brought by the silicon waste. When a small amount of silicon waste was added, refractory SiC can be slowly oxidized at high temperature [9-10]. In this case, this batch can be melted into glass. However, when silicon waste without pretreatment was used as the glass raw material, its content could only be up to 35.3% in the glass batch. It is difficult to form glass while the content of the silicon waste is higher.

C. Density

The densities of glasses No. 1, 2 and 3 are respectively 2.60 g/cm³, 2.64 g/cm³, 2.66 g/cm³. It can be seen that the density of the colored glass increases with the increase of the silicon waste. The higher the content of the silicon waste was used, the more Fe₂O₃ was formed in the glass (see the latter discussion). Therefore the glass density increased.

D. Spectral Characteristics

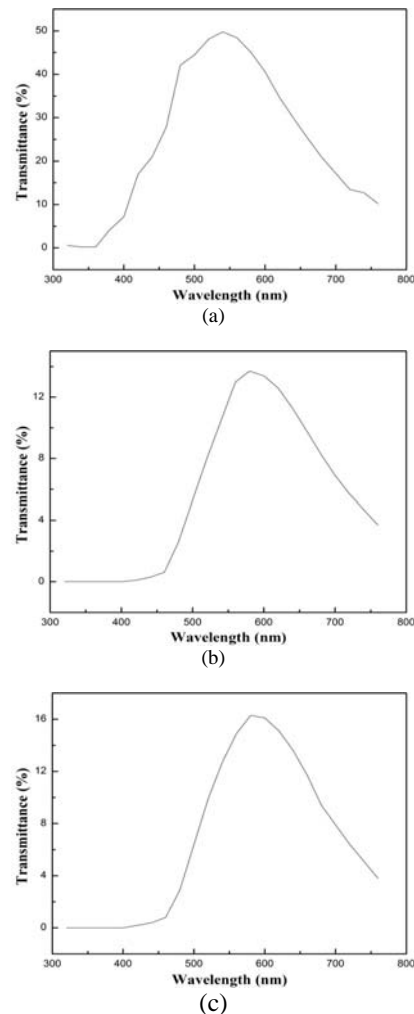


Figure 2. Spectra of the glasses 1 (a), 2 (b) and 3 (c)

The spectra of the glasses 1, 2, 3 are shown in Fig. 2. From the Fig. 2a, we can see that maximum transimission of glass 1 is at 540nm and the color of the glass 1 is light yellow-green. The glass absorbs ultraviolet light below 370nm. With increasing the content of silicon waste, glasses 2 and 3 shows maximum transimission at about 580nm (Fig. 2b, c) and the color is yellow-green.

E. The Glass Prepared with Pretreated Waste

SiC and Si in the silicon waste are not conventional glass raw materials. If the silicon waste was used in a large amount in the preparation of glasses, some of the components in the silicon waste cannot be easily melt and mixed with other glass components and hindering the formation of glass during the melting process. The above study has shown that use of the waste used to prepare glass is low. However, after the pretreatment of the waste, the amount of waste in the glass batch can be greatly increased.

F. Phase Transition

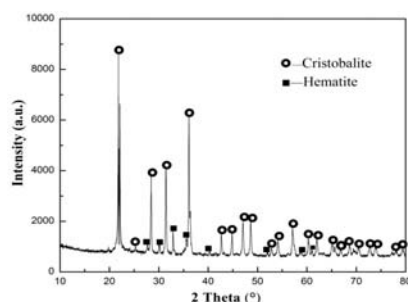


Figure 3. XRD patterns of the silicon waste after pretreatment

After the pretreatment, SiC and Si in the silicon waste were oxidized into SiO_2 (JCPDF card No. 82-1403) (Fig. 3). A new crystalline phase Fe_2O_3 (JCPDF card No. 99-0060) was found. The result suggests iron existed in the original silicon waste. It was brought by the saw line used in the cutting instrument. However, it was not identified in Fig. 1. The reason is that the iron content in the silicon waste was below the XRD detection limit. The chemical composition of the silicon waste after the pretreatment is presented in Table III.

TABLE III. CHEMICAL COMPOSITION OF THE SILICON WASTE AFTER PRETREATMENT

Silicon waste	Chemical composition after pretreatment				
	SiO_2	Fe_2O_3	Al_2O_3	Na_2O	Else
wt %	94.86	4.42	0.19	0.16	0.37

In Table III, we cannot found the refractory SiC in the silicon waste. It was oxidized into SiO_2 which is favorable for the glass formation. In addition, the oxidation degree of iron can affect the color of the prepared glass. The composition of the pretreated silicon waste conforms to the requirements of the oxide glass forming region. This makes it possible to use massive silicon waste in glass preparation.

G. The Formation of Glass

When the pretreated silicon waste was used as a glass raw material, its content can be increased to 44.97% in the glass batch. The utilization of the silicon waste is improved remarkably. Meanwhile, when the pretreatment silicon waste is used, glass can be prepared at a low temperature. Glass No.4 was melted at 1350 °C, although it was derived from a batch contained much higher content of silicon waste than glasses 1~3. The density of this glass is 2.70 g/cm³, larger than those of glasses 1~3 mainly due to the high inclusion of Fe_2O_3 .

H. Spectral Characteristics

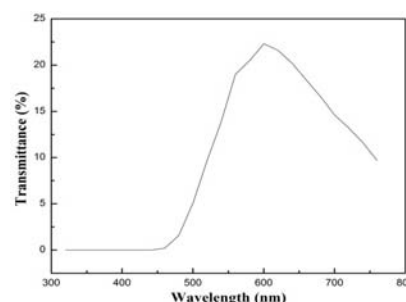


Figure 4. Spectrum of the glass 4

The maximum transmission of glass 4 (Fig. 4) is at about 600nm and its color is brown. The glass absorbs visible light and ultraviolet light below 460nm.

The prepared colored glasses can express different colors from yellow-green to brown -yellow according to the valence and content of iron ions. All the glasses absorb ultraviolet light.

IV. CONCLUSIONS

Silicon waste generated during the cutting process of crystalline silicon can be used to prepare color glasses. No other additional colorant is needed and the production cost is low. The obtained glass adsorbed ultraviolet light. Using the original solid waste, its content in glass batch can only be up to 35.3% and the melting temperature is up to 1500 °C. However, the pretreatment of the silicon waste turned refractory SiC into SiO_2 , which benefits the inclusion of silicon waste of in the glass batch up to 44.97% and the melting temperature is 1350 °C.

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