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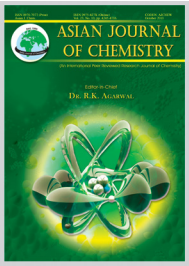

Study of Low Temperature Sintering Glass-Ceramic Using Fly Ash†

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By using orthogonal test, flexure strength and volume density as mostly performance index, by means of SEM and XRD, the glass-ceramic of the sintering and crystallization are investigated. The optimal parameters for the heat treatment of the glass is that the glass should be heated to 770 ºC, holding for 1h, then heated to 875 ºC, holding for 3 h. The crystallization temperature is the most important to the mechanical property of the glass-ceramics and holding time for nucleation is least. The main crystal phase is wollastonite by JSPDC cards and the crystal morphology of No. 4 is complete by SEM.

Key Words: Glass-ceramics, Orthogonal test, Fly ash, Sinter.

INTRODUCTION

Glass-ceramics, intended for building applications, constitute a well-developed and widespread way to absorb glasses obtained from treatment of several solid wastes(like fly ash)[1]. In addition to the environmental advantage of immobilizing wastes into materials with a generally high chemical resistance, a certain economic benefit may be found in entering the large market of construction materials.The first example was Russian Slagsitalls, later Japanese NEC company. Lately many papers have shown that several combinations of wastes have been proposed[2,3].

In Anhui province China, Huainan and Huaibie mining area of fly ash pile reach 45 million tons and increasing at a rate of 340 tons annually. How to effectively use fly ash is a very important issue, according to architectural glass performance and requirements, glass-ceramics is obtained by sintering fly ash in the paper.

EXPERIMENTAL

All chemicals used in present experiments were purchased and used as received without further purification and foundation glass was obtained by Applied Chemical Industry, 2011, 40(8):1331-1335. In a typical procedure, some amount of foundation glass were added into a refractory mold. The refractory mold was sealed and the temperature is heated to nucleation temperature with 10 ºC/min heating rate and maintained at 60 ºC for 1 h, then heated to crystallization temperature with 5 ºC/min heating rate ,and maintained at 760 ºC for 1 h and finally heated to 870 ºC, allowed to cool quickly to 750 ºC, then free cooling.

X-Ray powder diffraction patterns (XRD) of the products were obtained on a Japan Rigaku D_{max}-γA rotation anode X-ray diffractometer equipped with graphite monochromatized CuK_α radiation (λ = 1.54178 Å). The field-emission scanning electron microscope (FE-SEM) measurements were carried out with a field-emission microscope (JEOL, 7500B) operated at an acceleration voltage of 10 kV.

RESULTS AND DISCUSSION

The orthogonal experimental design and the experimental results were shown Table-1.

From Table-1 the flexure strength as performance index, Fig. 2 showed that primary and secondary relationships were: C (880 ºC) > A (760 ºC) > D (3 h) > B (1 h) and volume density primary and secondary relationships were: C (880 ºC) > D (3 h) > B (1 h) > A (770 ºC). The best heat treatment system is $A_2B_1C_2D_3$, that is the No. 4 in orthogonal experimental comprehensive flexure strength and volume density trend.

Fig. 1 shows the peaks of Nos. 3, 4 and 8 sharper than others relatively, may be their crystallization timewere longer, some of the crystal surface leading to fully developed.

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TABLE-1
ORTHOGONAL EXPERIMENTAL DESIGN

Sample number	Factors				Experimental index	
	A nucleation temperature (ºC)	B nucleation time (h)	C crystallization temperature (ºC)	D crystallization time (h)	Flexure strength (MPa)	Volume density (g/cm^3)
1	760	1	870	1	70.18	2.8159
2	760	2	875	2	79.80	2.7872
3	760	3	880	3	80.48	2.8211
4	770	1	875	3	64.78	2.8707
5	770	2	880	1	67.20	2.8365
6	770	3	870	2	63.92	2.7941
7	780	1	880	2	77.28	2.8069
8	780	2	870	3	74.52	2.7555
9	780	3	875	1	62.42	2.8730
T_{i1}	Flexure strength	220.46	212.24	208.62	199.80	
	Volume density	8.4242	8.4935	8.3655	8.5254	
T_{i2}	Flexure strength	195.90	211.52	197.00	211.00	
	Volume density	8.5013	8.3792	8.5309	8.3882	
T_{i3}	Flexure strength	214.22	206.82	224.96	219.78	
	Volume density	8.4353	8.4882	8.4645	8.4473	
$\overline{T}_{j1}$	Flexure strength	73.47	70.75	69.54	66.6	$\bar{\rho}$ = 2.8179 g/cm^3
	Volume density	2.8081	2.8312	2.7885	8.8418	
$\overline{T}_{j2}$	Flexure strength	65.30	70.51	65.67	70.33	
	Volume density	2.8338	2.7931	2.8436	2.7961	
$\overline{T}_{j3}$	Flexure strength	71.41	68.94	74.99	73.26	
	Volume density	2.8118	2.8294	2.8215	2.8158	
R_j	Flexure strength	24.56	5.42	27.96	19.98	
	Volume density	0.0257	0.0381	0.0551	0.0457	

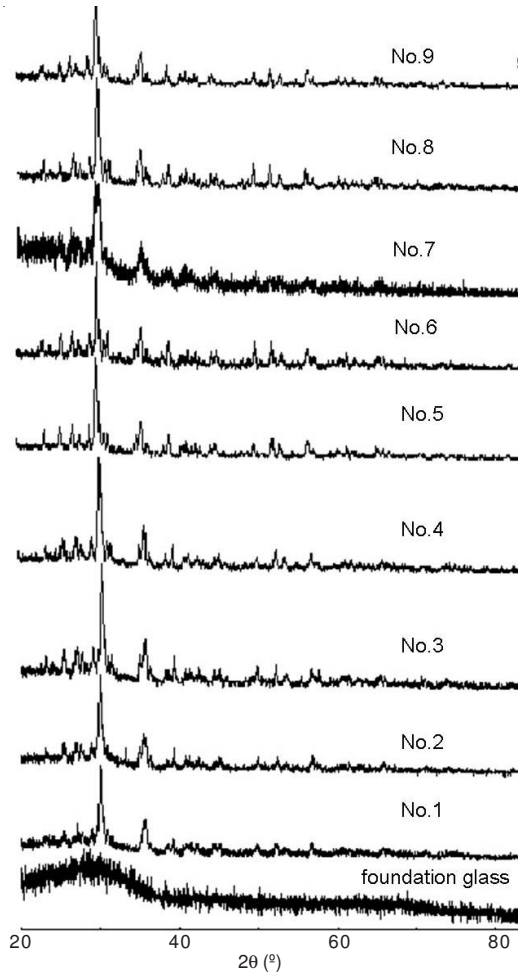

Fig. 1. XRD curves of different samples

Fig. 2 shows the crystal morphology of No. 4 is more complete than others.

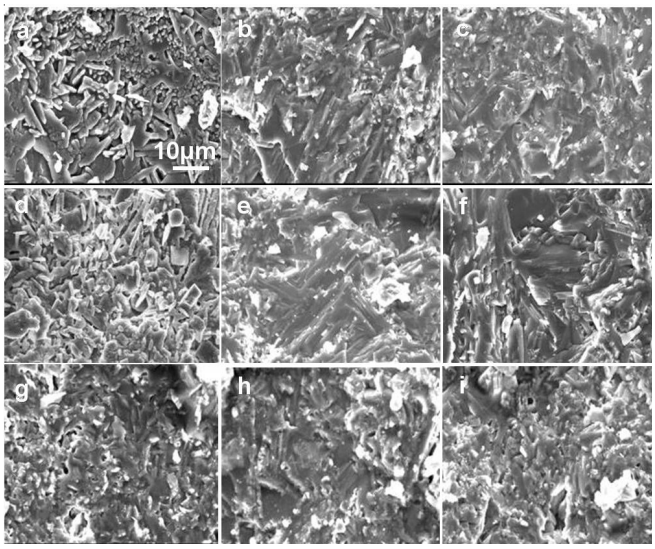

Fig. 2. FE-SEM images of sample cross-section, from a to i is from Nos. 1-9 accordingly and all rulers is same to a

Fig. 3 shows the surface of No. 4 is not very uniform which were composed of granular crystal and the distribution of scattered clusters on the glass body.

Conclusion

In summery, we have shown that the optimal parameters for the heat treatment of the glass is that the glass should be heated to 770 ºC, holding for 1 h, then heated to 875 ºC, holding for 3 h.

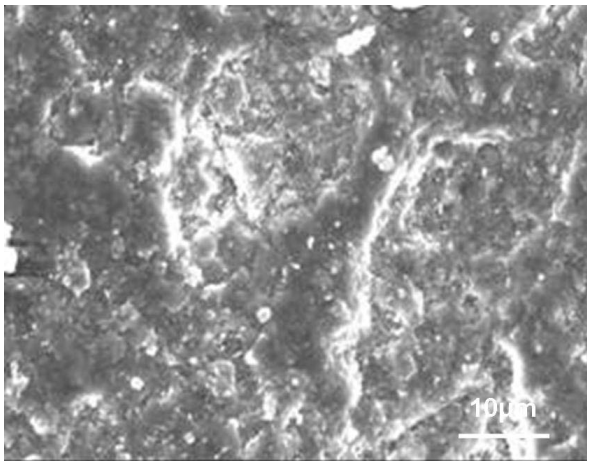

Fig. 3. FE-SEM image of No. 4 surface

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