

## PREPARATION OF GLASS-CERAMIC FROM SODA LIME WASTE GLASS

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### Abstract

The waste soda lime glass was converted to glass-ceramic by adding certain amounts of alumina, lithium oxide and titanium oxide. The effect of quenching in air on the microstructure and the mechanical properties were investigated. SEM observations clearly revealed that the presence of homogeneously dispersed tiny single crystals with average size of 4 $\mu$ m for quenching sample which increased with holding time at the nucleation temperature of 650°C to reach a maximum size of 10 $\mu$ m. The results of Vickers hardness tests indicate that the hardness value of the as quench soda lime based glass-ceramic is 724 kg/mm<sup>2</sup>. This value starts to decrease with increasing holding time at nucleation temperature to reach 687 kg/mm<sup>2</sup> after 96 h for the crystals growth.

### Introduction

Glasses are amorphous materials or materials with no ordered molecular structure. Glass-ceramic materials are solid materials with a crystalline structure obtained by the controlled diversification of glasses. To make a glass-ceramic product a glass is melted formed into the desired shape and heat treated to allow the molecules in the glass to connect and order them selves into a crystalline structure [1].

The main benefit of glass-ceramics is that they are easily made and shaped like traditional glass products, as well as having many of the desirable properties of ceramic materials (high strength, high resistance to thermal shock, hardness, etc). Other properties of glass-ceramic, such as thermal expansion can be varied over a large range (from negative to positive) making them ideal for many applications such a veneer for the metal frame work of crown, bridge, or inlay and in restorative dentistry [2].

The possibilities of obtaining glass-ceramic derived from soda lime waste glass were examined in this work. The formation of glass-ceramic affected by modifying the original composition of waste glass by addition aluminum oxide, lithium oxide in the present of titanium oxide as catalyst.

### Experimental work

The composition of the initial soda lime glass is shown in Table (1), was modified by increasing the aluminum oxide content to

9.8% and decreasing the sodium oxide concentration to 8.2%, while maintaining silicon oxide approximately constant as shown in Table (2).

Lithium oxide was added in amount of 1.3% to de-stabilize the glass [3]. 2.4% titanium oxide was added as a nucleation agent [4]. The sample were weighted by using electric balance four digits and milled for 6 h in ball milling and placed into an electrical tube furnace type HERZOG HAG 1215 and held at 1200°C for 6 h then the melts was quenching in air. Quenched glass was thermally treated at 650°C, maintaining this temperature for long time (24 to 96 h). The crystallized size of the heat treated samples were identified by SEM. Hardness measurement were then done using a Vickers hardness tester type (HD6330) from leitz Webzlar GMB at load of 100 g and a dwell time of 30 s.

**Table (1)**  
**Weight % of chemical composition of soda lime glass.**

Oxides	wt %
SiO <sub>2</sub>	71.75
Na <sub>2</sub> O	16.00
CaO	5.10
MgO	3.87
Al <sub>2</sub> O <sub>3</sub>	1.22
SO <sub>3</sub>	0.36
Fe <sub>2</sub> O <sub>3</sub>	0.08
K <sub>2</sub> O	1.60

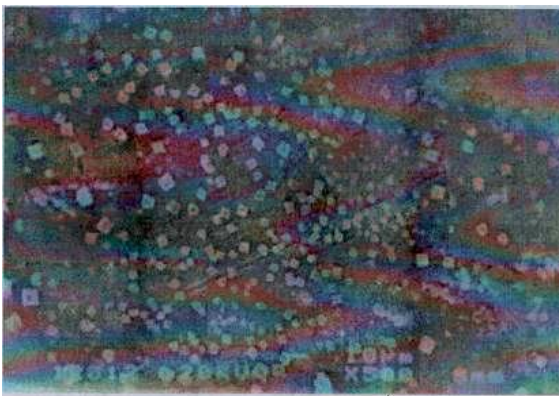
**Table (2)**  
**New weight % composition of soda lime glass.**

Oxides	wt %
SiO <sub>2</sub>	70.72
Na <sub>2</sub> O	8.15
CaO	2.55
MgO	3.33
Al <sub>2</sub> O <sub>3</sub>	9.75
SO <sub>3</sub>	20.18
Fe <sub>2</sub> O <sub>3</sub>	0.04
K <sub>2</sub> O	0.66

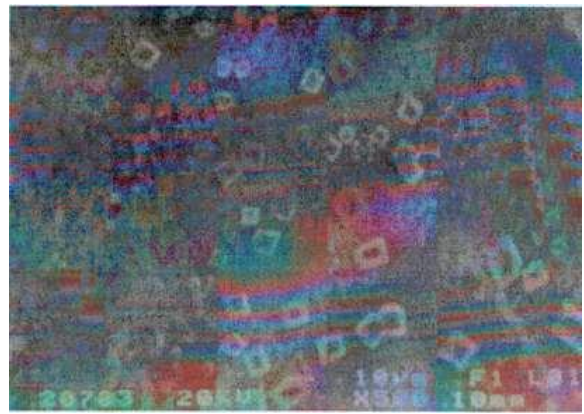
### Results and Discussion

Soda lime glass is very difficult to crystallize. The glass itself is very resistant to crystallization and will not nucleate from the bulk of the material by heat treatments. Addition of lithium oxide, aluminum oxide, and titanium oxide cause the glass to crystallize. The oxides addition were lower sodium oxide concentration to lower value and increase the alumina concentration while o kept the silicon oxide approximately constant as shown in Table (2). The affects off quenching of glass in air on the microstructure for samples were observed by SEM. From Fig.(1), it is clearly revealed that the presence of homogeneously dispersed tiny crystalline phase through the glass matrix with an average crystalline size of about 4  $\mu\text{m}$  (comparing with the standard line in the bottom of SEM photos ).

The long heat treatment at the nucleation stage of 650°C lead to increasing the crystalline size to approach an average size of 10  $\mu\text{m}$  after 24 h, Fig.(2).

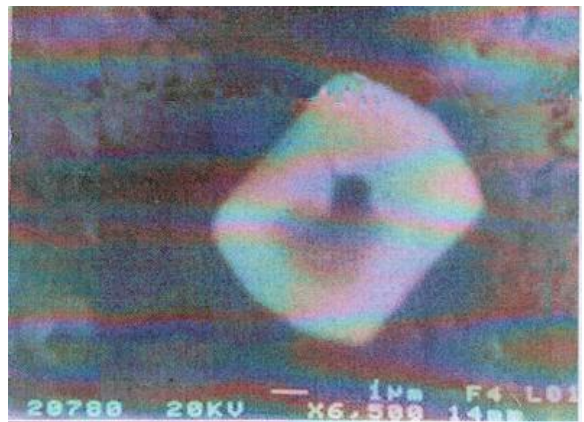


**Fig. (1) : SEM of as quenched glass-ceramic in air.**



**Fig. (2): SEM of glass-ceramic heat treated at 650 °C for 48 h.**

No farther increase in dimension size for the single crystal up to 96 h of heat treatment as shown in Fig. (3). This size is depending on the viscosity of the sample, since the viscous flow strongly dependent on the chemical composition [3].



**Fig. (3):SEM of glass-ceramic heat treated at 650 °C for 96 h.**

To determine the kind of tetragonal crystals in the scanning electron microscope (SEM), X-ray diffraction was chosen to examine the samples treated at 650 °C for 96h, Fig. (4), so the sharp peaks refers to Cristobalite phase.

Comparison of hardness values of the various samples measured at the same load of 100 g and dwell time of 30 s, shows that the quenched glass-ceramic have the maximum hardness values of 724  $\text{kg}/\text{mm}^2$  since its crystals size have the smallest size of all glass-ceramic samples produced. These values were decreased with heat treatment time to reached about 680  $\text{kg}/\text{mm}^2$  as shown in Table (3) and Fig.(5) which is acceptable compared to other

[5],[6] and to the original soda lime glass of  $4.6 \text{ kg/mm}^2$  [7].

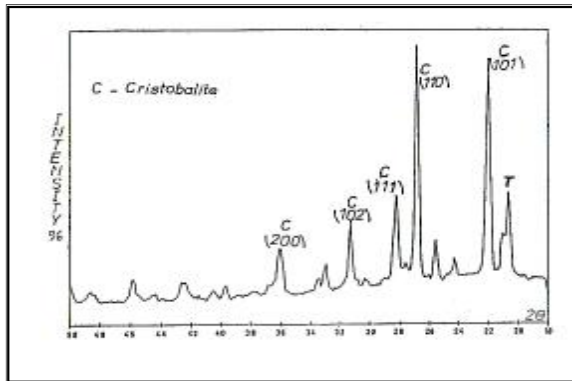


Fig. (4): X-ray pattern shows Cristobalite phase.

Table (3)

The hardness in  $\text{kg/mm}^2$  change with long time heat treatment.

Holding time	Quench in air	24 h	48 h	72 h	96 h
Hardness	724	724	296	562	687

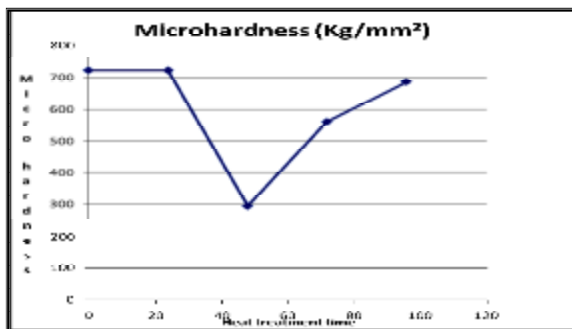


Fig. (5): the behavior of hardness with heat treatment time.

### Conclusions

Soda lime glass is very difficult to crystallization. The best way to get the material to crystallize in an acceptable manner is to shift the composition of the soda lime glass by added  $\text{Li}_2\text{O}-\text{Al}_2\text{O}_3-\text{TiO}_2$  in different concentrations. This composition which was shift the composition of soda lime on the phase diagrams show more desirable results with heat treatment at nucleation temperature of  $650^\circ\text{C}$  at different times.

### Acknowledgment

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### الخلاصة

تم تحويل حطام زجاج جبر الصودا الى زجاج سيراميكي بإضافة كميات محددة من اكاسيد الالمنيوم والليثيوم و التيتانيوم . فحص تأثير الاخمد السريع في الهواء على التركيب الدقيق وكل الصفات الميكانيكية. اظهرت نتائج المجهر الالكتروني الماسح وجود بلورات احادية صغيرة منتشرة بشكل متجانس داخل الزجاج بحجم  $(4\mu\text{m})$ ، ويزداد حجم البلورات بزيادة زمن التبلور عند درجة  $650^\circ\text{C}$  لتصل الى  $(10\mu\text{m})$ . ان نتائج فحص الصلادة بطريقة فيكرز اعطت صلادة عالية للعينة سريعة الاخمد تقدر بـ  $724 \text{ كغم/ملم}^2$ . ان هذه القيمة تأخذ بالنقصان لتستقر عند صلادة مقدارها  $687 \text{ كغم/ملم}^2$  عند تعرضها لزمن تبلور مقداره 96 ساعة.