MATERIALS

# Investigations of some physical and mechanical properties of Alumina-Silicate Ceramic

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SUMMARY. – *The effect of the addition of some alkali oxides on physical mechanical properties for kaolin filled scrap glass powder and some alkali oxides were studied. Ceramic Filled Glass (CFG) technology has been used to prepare ceramic samples. The starting materials were composed of Iraqi-Duekhla raw kaolin, wasted soda-lime glass from industrial site, and small amounts of alkali carbonates. The sintering temperature was 950°C with a soaking time of 3 hr. Samples with a different composition of raw material have been prepared to obtain a disk shape using uniaxial pressure. Strength characteristic-hardness and diametrical strength depending on the alkali oxides concentration were determined. Present study showed the chemical composition of the kaolin, soda lime glass, and mechanical properties of prepared samples, while the phase transformations were investigated by XRD analysis. The prepared ceramics shows improved hardness and diametrical strength for ceramics prepared with $Na_2O$ and $K_2O$ alkali oxides except for ceramics samples prepared with $Li_2O$ which show small influence. The aim of this work was to evaluate the quality of the ceramic that can be made using kaolin, glass powder and some alkali oxides.*

## 1. Introduction

Ceramic -Filled -Glass (CFG) composite densify by a combination of glass redistribution, grain rearrangement, and viscous flow in a three stage process described as non-reactive liquid-phase sintering (NLPS) (1). As in all sintering processes, the driving force for densification during NLPS is the reduction of the free energy of the system. This is accomplished by replacing higher energy solid-solid and solid-vapor interfaces with lower energy liquid-solid and liquid-vapor interfaces during the initial and intermediate stages; and by reducing the minimizing the liquid-vapor interfacial area during the final stage .

Densification during initial-stage particle redistribution and intermediate-stage grain rearrangement occurs rapidly, within the first ~10 min of sintering (2).

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Final stage of NLPS is by far slowest stage of sintering, and consequently, it is the critical stage in the development of the microstructure and properties of a CFG composite.

The glass can be prepared by heating silica sand or quartz crystals to a temperature above the melting point of silica, 1725°C. Because of the three dimensional cross-linked nature of the network the resulting glass is so viscous (3,4). In order to decrease the viscosity of molten glass from that of silica, it is necessary to add flux, or network modifier. The alkali metal oxides are excellent fluxes. Since they are network modifiers, they soften the glass structure by generating of non-binding oxygen's (5,6).

Kaolin is used in ceramic whiteware products, insulators, and refractories. In whitewares, kaolin aids accurate control of molding properties, and adds dry and fired strength, dimensional stability, and a smooth surface finish to the ware. The excellent dielectric properties and chemical inertness of kaolin make it well suited for ceramic electrical insulators. In refractory applications, the dimensional stability, high fusion point, and low water content, along with high green strength, make kaolin an important constituent (7).

A lower softening point material is potentially capable of reducing the firing temperature of clay (kaolin) products such as bricks and tiles through a chemical reaction with the kaolin. The pioneer work done by M.E Tyrell et al at the Tuscaloosa Metallurgy Research Laboratories has shown the benefit of adding waste glass powder in clay (8). The addition of glass powder has been used as fluxing agent, and it showed to be efficient fluxing agent because this material can accelerate the densification process during firing. It presented the best mechanical and technological properties for ceramic bodies (9). Due to the requirement of using raw materials with very low oxides content, this possibility has particular importance for the production of hard ceramic. In this case, particular attention is paid to the rheological properties of the parent mass (kaolin), because the addition of the scrap glass might influence the forming process. This replacement, however, also leads to a variation of the phase composition of the green, which may influence the sintering process (10).

## 2. Experimental procedure

### 2.1 – *Sample preparation*

The Iraqi Kaolin (Duekhla Kaolin), Soda Lime Glass (SLG) taken from Al-Taji glass industry site, which is milled by blast method, have been used as a starting material, Lithium Carbonate ($Li_2CO_3$), Sodium Nitrate ($NaNO_3$) and Potassium Nitrate ($KNO_3$) as sources of $Li_2O$, $Na_2O$ and $K_2O$ respectively.

After the raw materials are selected and the desired amounts of batches have been weighed, they go through a series of preparation steps. First, the raw material particles are produced to the desired size (> 53) which involves using a variety of

equipment during crushing and grinding steps. This process leads to mixing the batches compositions by using paddle mixer type (willy-Wab-T2) for 2 hr to get a more homogenized mixture. Mixtures of Kaolin, glass powder and Alkali oxides are used to prepare 9 samples, in addition, kaolin alone and 95% kaolin and 5% scrap glass powder were taken as an extra samples. The amounts of materials have been weighted using a sensitive four-digit balance type (Precisa Instruments Ltd.). The compositions of the prepared samples are listed in Table 1.

**Table 1**
Composition of the prepared samples in wt %.

| Sample No. | Kaolin wt% | S.L.G wt% | $Li_2O$ wt% | $Na_2O$ wt% | $K_2O$ wt% |
|---|---|---|---|---|---|
| 1 | 100 | | | | |
| 2 | 95 | 5 | | | |
| 3 | 92 | 5 | 3 | | |
| 4 | 90 | 5 | 5 | | |
| 5 | 88 | 5 | 7 | | |
| 6 | 92 | 5 | | 3 | |
| 7 | 90 | 5 | | 5 | |
| 8 | 88 | 5 | | 7 | |
| 9 | 92 | 5 | | | 3 |
| 10 | 90 | 5 | | | 5 |
| 11 | 88 | 5 | | | 7 |

Distilled water was added to the mixture. It is found that 5 wt % of distilled water give the best compaction results. Batch of 2 gm powder was pressed to disk shape with diameter of 1.5 cm using stainless steel cylindrical die. It is found that 30 Kgf/cm$^2$ for 1 min press duration is the best to obtain crack free green samples. The prepared green samples have been dried in the air for 72 hr. The samples are further dried in oven operating at 110°C for 2 hr. The prepared samples have been sintered at 950°C for 2 hr at a heating rate of 10°C/min, using programmable furnace type Carbolite. The samples then left over night for cooling and collected next day.

**2.2** – *Mechanical properties*

2.2.1 – Hardness test

Hardness is the resistance to penetration. Various procedures are used to measure hardness. They are essentially divided into two classes, commonly called microhardness (Vickers, Knoop) and macrohardness (Brinell, Rockwell) tests. The comparison between the two methods occurs for a load of approximately 200 gf (2N). These depend on the material, its thickness, the indenter used, and the load applied. Hardness value helps to characterize resistance to deformation under load, and fracture (11.12).

Selected specimens have been tested by Vickers indenter. The indenter is attached with optical microscope (Model Micromit, ADOL PHI. BUEHLER INC., optical and Metallurgical instruments, U.S.A., 60204). All the samples were ground and polished to mirror like surface. The indentation load was 9.8 N for loading time of 20 sec. Three indents were made on each sample surface and the average diagonal dimensions of these indent was calculated to find the Vickers hardness using Eq.[1].

$$W.H.N. = \frac{2P\sin(\alpha/2)}{I_{av}^2} = \frac{1854.4F}{I_{av}^2}\left[\frac{Kgf}{mm^2}\right] \quad [1]$$

where 1.854 is a constant, $F$ = load [Kgf], $I_{av}$= average of indentation diameter [mm] = $(I_1+I_2)/2$

*2.2.2* – Fracture test

This test was developed originally in Brazil by Carnieo and Borcellos as long as 1953 and has recently come into rather general use. It has been standardized as ASTM C496. PHYWE test machine (model 1757793, Japan) has been used in its compression mode. The applied load can be varied up to 30 tons. The crosshead speed has been fixed to 0.07 mm/sec. The samples in form of discs have been used. The diameter and thickness for each sample have been measured and the force at fracture point (at which the sudden decrease in the load force is observed) is obtained. Eq.[2] has been used for calculation of the splitting tensile strength.

$$\sigma_D = \frac{2F}{\pi Dt} \quad [2]$$

where $\sigma_D$ is the splitting tensile strength measured in Pascal (Pa), $F$ is the maximum applied load in Newton (N), $D$ and $t$ are the cylinder diameter and height. The results of the chemical analysis for Kaolin and SLG are shown in Table 2.

## 3. Results and discussions.

It have been seen that kaolin contains some oxides in the higher level, such as $Al_2O_3$ (34.84%), and S.L.G. have $SiO_2$ (72.54%) in higher amount. These oxides may play a significant role towards vitrification, phase transformation, and growth of mullite crystals in soda lime glass, and then have an influence toward improved strength. (5,11,13).

Figures 1,2 and 3 show the XRD analysis for pure kaolin, scrap soda lime glass, and sampled prepared with kaolin and 5% of S.L.G., all samples were sintered at 950°C. It have been seen that the XRD for kaolin show the presence of kaolin and quartz. The soda lime glass analysis shows the amorphous phase that reveled in XRD pattern. Fig. 3 shows the presence of quartz and cristobalite phases, which means, new phase has been growth.

**Table 2**
Results of the chemical analysis for Kaolin and SLG

| Kaolin Content | Weight % | S.L.G. Content | Weight % |
|---|---|---|---|
| $Na_2O$ | 0.25 | $Na_2O$ | 16.4 |
| CaO | 0.15 | CaO | 5.0 |
| $K_2O$ | 0.61 | $K_2O$ | 0.35 |
| MgO | 0.38 | MgO | 3.45 |
| $Al_2O_3$ | 34.84 | $Al_2O_3$ | 1.45 |
| $Fe_2O_3$ | 1.32 | $Fe_2O$ | 0.6 |
| $SiO_2$ | 47.26 | $SiO_2$ | 72.54 |
| $TiO_2$ | 1.4 | $TiO_2$ | 0.01 |
| L.O.I. | 13.79 | $SO_3$ | 0.15 |
| | | Cl | 0.05 |

L.o.I: Loss on Ignition.

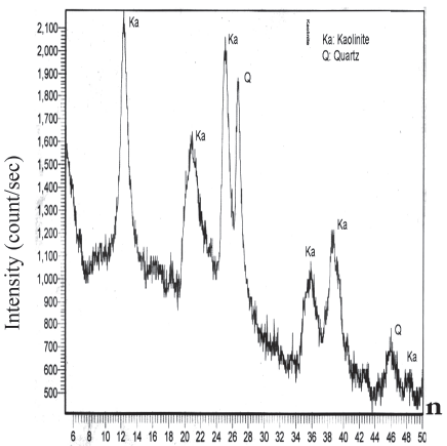


FIG. 1

XRD pattern of Kaolin

Vickers microhardness test for samples prepared of alkali oxides were shown in Figs. (4a, 4b and 4c). Fig.4a, shows that the addition of $Li_2O$ gives good values of hardness with increasing $Li_2O$ content, but these values were very close to one another, that means that the increasing of this oxide did not improve the hardness properties. Fig 4b shows the V.H.N. versus the $Na_2O$ content. It can be clearly seen that the increasing of this oxide in prepared samples have increased the hardness very well. This result was similar for the samples prepared with addition of $K_2O$, however these samples give the best values for microhardness test, and the hardness is improved with higher alkali oxide inclusion, Fig 4c. The increase of microhardness strength with increasing of potassium and sodium carbonate con-

tent may be due to the new phases originated from the existence of potassium and sodium oxides, otherwise it can be explained by the fact that samples sintering temperature are given enough time and temperature to arrange its microstructure. This is not the case when lithium carbonate is introduced. It seems that the phases those lithium oxide shares to are rather brittle (3, 4).

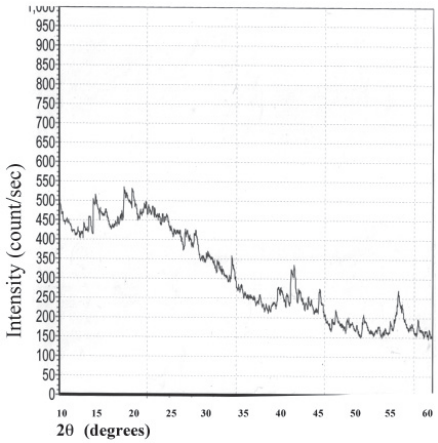


FIG. 2

XRD patterns for SLG at 950°C.

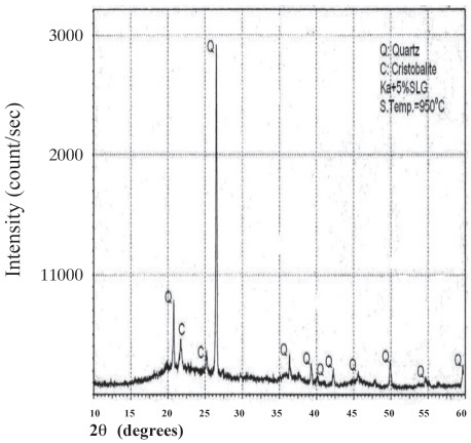


FIG. 3

XRD pattern (Cu-Kα) for sample prepared of Kaolin + 5% SLG and sintered at 950°C.

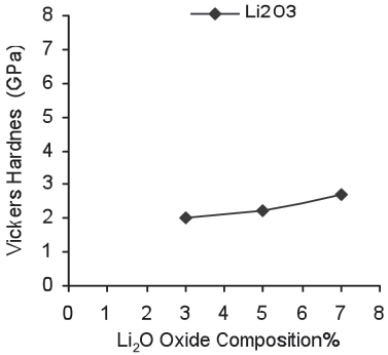


FIG. 4A

Vickers microhardness versus wt% of $Li_2O$ content.

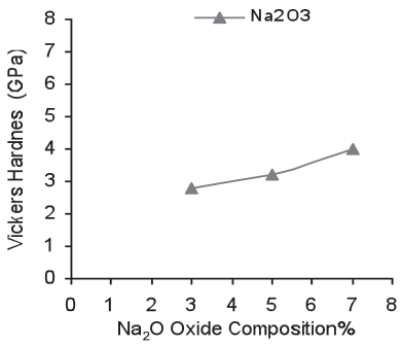


FIG. 4B

Vickers microhardness versus wt% of $Na_2O$ content.

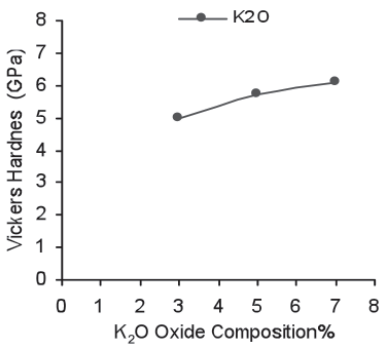


FIG. 4C

Vickers microhardness versus wt% of $K_2O$ content.

Figures 5a, 5b and 5c show the splitting strength versus alkali oxides inclusion. The noteceable feature of diametrical strength curve for $Li_2O$ specimen sintered at 950°C was that the strength was increased with increasing this oxide content (Fig. 5a). But the splitting effect has very close results. The diametrical strength values obtained for the other prepared samples show similar behavior that examined for Vickers microhardness tests. The specimen with $Na_2O$ content exhibited the same tendency as the specimen including $K_2O$ content (Figs. 5b and 5c). Also here we can see that the addition of the alkali oxides gives rise to the diametrical strength. in addition, sample prepared with $K_2O$ gives the best values.

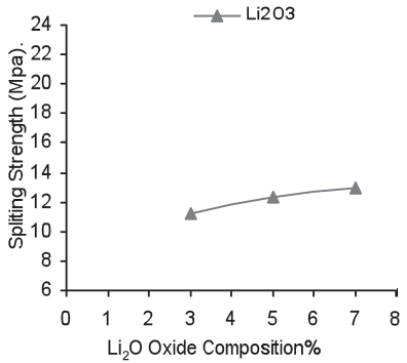


FIG. 5A

Diametrical strength Vs $Li_2O$ wt% content

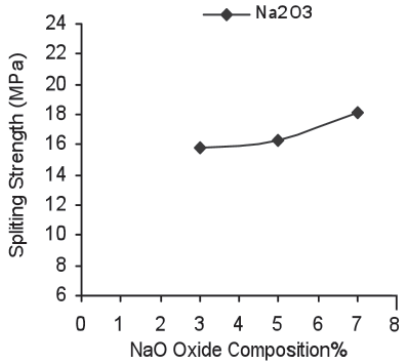


FIG. 5B

Diametrical strength Vs $Na_2O_3$ content %

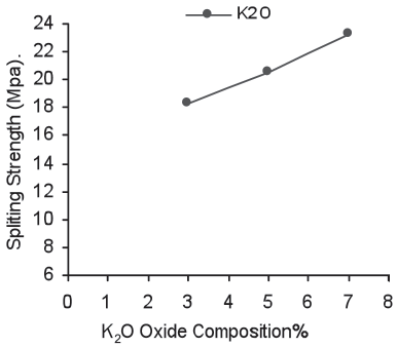


FIG. 5C

Diametrical strength Vs $K_2O$ content %

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