

SOME MECHANICAL AND THERMAL PROPERTIES OF MULLITE PREPARED FROM IRAQI STARTING MATERIALS

Salah Abdul Wahab Sheet *

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ABSTRACT

Some mechanical and thermal properties of mullite samples prepared by mixing different phases of alumina and silica powders have been studied according to ASTM methods. The cold crushing strength of the sintered bodies, with different porosity, at room temperature was in the range (18-54) Mpa. And the flexural strength was (3.8-26.1) Mpa. The thermal properties of samples. Specific heat, of thermal insulation was (19028(j/g C)), and the measured thermal expansion coefficient was between $(7.08-7.68) \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ at the temperature difference (20-100 C). (The thermal conductivity was measured and calculated for samples with different porosity and mullite content percentages.

INTRODUCTION

The aim of a ceramist in selecting or modifying a particular composition, fabrication methods, firing processes or heat treatment is to obtain product having certain useful properties. This requires good understanding of material properties. In this study we determined from the reaction of alumina with quartz, silicic acid, cristobalite. All these starting materials were manufactured or available as deposits in Iraq. The properties of the manufactured material are important in the selection of constructional refractors. The demands made on refractories are not only heat resistance but involves number of other factors depending on

their uses. This may be divided into three main types, mechanical strength, thermal expansion and confinement of heat. Properties of mullite depend on starting material and the impurities that greatly affect the equilibrium reaction, [1], also, the technique of producing mullite will affect the physical properties such as mechanical strength, and thermal conductivity, [2].

At normal service of refractories in furnaces, low strength is needed (98-980) Kpa. The strength will be retained up to the softening point, because of plastic deformation which occurs at high temperatures prevents brittle fracture, [3].

* Dr.- Assist. Prof.- Dept of Medical Physics- Al- kindy College of Medicine- Uni. of Baghdad.

Mullite strength is in very wide rang (20-700) Mpa, [4] and recent studies have shown that phase pure mullite can also retain its strength in temperatures up to 1500 C°, [4].

Ceramic materials in the annealed state are microscopically strain free when the temperature is uniform. In case if there is a Temperature gradient, hotter parts try to expand relatively to the colder ones which are subjected to tensile stress. The thermal expansion coefficient will directly determine the magnitude of the arising thermal strains. If the strain exceeds the fracture strain of the material, fracture will occur. Fenstermaier and Hummel in (1961) have pointed out that the mullite crystals do not have an exceptionally high degree of thermal expansion anisotropy. The linear expansion coefficient normal to c-axis is $4.5 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ and $5.7 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ parallel to c-axis, [5]. The thermal conductivity and the rate of applied heat determine the thermal gradient through the material. If conduction is not thermally steady, the rate of material heating and the thermal strains, will depend on the rate of heat absorption and hence on heat capacity, [6].

Pankatz et.al., (1963) has measured the low temperature heat capacity of mullite, prepared from pure alumina and quartz; it was $0.763 \text{ j/g } ^\circ\text{C}.$ For different refractories, the heat capacity ranges from 0.753 to 0.9209 j/g C, [3].

EXPERIMENTAL PROCEDURES

1-Mullite Samples:

The samples were prepared from the reaction of different phases of starting materials alumina and silica as shown as in (Table A).

Table (A)

Samples	Starting materials	Al/Si ratio wt/wt	Mullite content %
Grope A	m-alumina+Quartz	2.35	80.3
Grope C	m-alumina+Crystobalite	2.55	89.8
Grope D	α -alumina+crystobalite	2.4	95.3
Grope E	α -alumina+silicic Aside	2.63	90.5

2-Cold Crushing Strength:

The standard test method (ASTM C13384) for cold crushing strength is used, [7]. The cold crushing strength (com) calculated according to the following relation:

$$\sigma \text{ Max} = W / A \dots [1]$$

Where W= Total maximum load.

A = Average gross top to bottom areas of the Specimen

3- Flexural Strength:

The test method (ASTM C 133-84), [7], the flexural strength calculated using the relation:

$$\sigma_f = W / I \dots [2]$$

Where W is the applied force, l is the span times II is the depth, and I is the cross section moment of inertia, for a circular section rod of diameter (D) which is calculated according to the following relation:

$$I = (\pi D^4 / 64) \dots [3]$$

4- Specific Heat:

The test method (ASTM C 351-82) for mean specific heat determination by using calorimetry method. It was calculated according to the following equation:

$$C_p = (m_0 C_0 + m_w C_w) (T_f - T_1) / (m_2 (T_2 - T_f)) \dots [4]$$

Where C_0 , C_w and C_p are the specific heat of Copper, Water and the test sample respectively. m_0 , m_w , and m_2 are the mass of calorimeter, water used in the test, and the sample to be measured. T_1 , T_2 and T_f are the temperatures of water; sample after heating, and the final of the system respectively.

5-Thermal Expansion) α)

Expansion data may be obtained by heating suitable marked pieces in a furnace with spy hole to observe the geometrical changes of the pieces with optical apparatus. The heating Microscope provides the observation and photographic recording of the various phases and dimensional change of the ceramic material. The mean thermal expansion coefficient (α) was calculated using the following equation $\alpha = (L_1 - L_0) / \{L_0 (T_2 - T_1)\} \dots\dots [5]$

Where L_0 is the length of the specimen at temperature T_1 , L_2 is the length of the specimen at temperature T_2 . And the percentage thermal expansion is $(L_1 - L_0) \times 100 / L_0 \dots\dots [6]$

6- Thermal Conductivity (K):

Thermal conductivity measurements were carried out by using Lees Disc method, [8], for samples with different apparent porosity at room temperature. In porous material, the gas Contained in the pores has much lower (K) than the solid phase. If pores from a dispersed phase then (K) of such system is $K = K_0 (1-V) / (1+V) \dots\dots [7]$

Where V is the volume fraction of porosity and K_0 is the thermal conductivity of full dense material. [3]. This shows that (K) is a strong function of microstructure features.

RESULTS AND DISCUSSION

i- Mechanical Strength

The cold crushing strength was calculated by Eq. (1), the results are shown in table (1). Comparing these results with the results obtained by many authors' work, there is an agreement with the average obtained from literature. The obtained range of cold crushing strength in this work shows that the product is applicable as a furnace constructional material. In 3-point test the flexural strength was calculated by using Eq. (2) and (3) table (1) shows the obtained results. These values are agreement with some authors work. The high flexural strength values obtained (or group (A) samples (8.37 – 26.18) Mpa. compared with author groups are due to the low apparent porosity of the samples and also to alumina / silica ratio. The effect of starting material ratio on mullite strength was studied by Miller and Davis (1996), they discovered that the mullite produced from (1.5) ratio of alumina to silica was stronger than that produced from the (0.3) ratio [9]. The presence of glassy phase surrounding the ceramic grain stress around the grain during material cooling, which is due to the difference in thermal expansion coefficients between the grain and the glassy phase. This stress in the glassy phase creates tensile forces perpendicular and compressive forces to the grain boundaries parallel to it. [10]. The mullite content in-group A samples was 80.3% it was the lowest compared with the other groups (C, D and E) samples, which showed low flexural strength value because they decrease the cross-sectional area in which the load is applied on, resulting a reduction in the body strength, they also act as stress concentrators. [3] The heat capacity of test samples in this work is within the range mentioned above.

ii-Thermal properties

The mechanical and thermal stability of mullite as a constructional refractory material at elevated temperatures is important as well as its thermal insulation. On these bases the thermal properties are presented as follows:

1-specific Heat capacity:

The result of meanspecific heat capacity was obtained by using the test method (ASTM C 351-82). [7]. and compared with some literature as shown in table (2).

Taking the average heat capacity of the measured samples and the average obtained from reference, the deviation was found to be (5%) this deviation may due to pores in the samples. Although the molar capacity for crystalline materials is not structure-sensitive, the volume heat capacity depends on the porosity, [6], the mass of material in a unit volume decreases in proportion with pore volume present. The heat capacity of the refractory materials, ranges from (0.754 to 0.92) J/g.C. [3].the heat capacity of test samples in this work is within the range mentioned above.

2-Thermal Expansion coefficient

The mean thermal expansion coefficient was calculated using eq. (5). For test samples (30A, 17G.132D, and 22E) with (26.72, 54.2, 46.7.and 50.32 % porosity) are shown in table (3). Comparing these values with those obtained from the work of many authors, a discrepancy is shown. These discrepancies may be due the measuring method used in this work and the high apparent porosity of the samples. Lower porosity can be achieved because of the difficulties associated with hand Pressing of the samples when using small diameter mold. The percentage thermal expansion values calculated by using eq. (6). Considering the values in the material

index. [11]. As a standard and comparing them with die values obtained in this work plotted in Fig(1).It can be noted that the behavior of the samples in temperatures between(20-100 C °) are similar to that of the standard and there is no sharp dimensional change.

3-Thermal Conductivity

The obtained values of thermal conductivity at room temperature for samples of each group. With different apparent, is shown in table (4) and in fig (2).were the thermal conductivity of solid material (with zero porosity) was calculated by using eq. (7). From these results we can noted that group(A) samples showed lower(K)values than other groups, this due to the relatively high content of unreacted silica(K for silica is 1.442w/m C)in the sample, and also due to increasing porosity percentage in the samples. This result is in agreement with Kingry (1966),who stated that, a anerly linear decrease in conductivity with increasing porosity, if found for dispersed pores in a solid.[12].

CONCLUSIONS

The mechanical and thermal properties of mullite samples were within limits of the values obtained by many others. On these basis the mullite as a constructional refractory material could be produced industrially from Iraqi row materials .The produced material could be utilized in glass tanks, crucibles steel industry as burner blocks , electrical furnace roof and in nonferrous metal industry for induction furnaces.

Table (1)
The strength of mullite samples for (A,C,D, and E) groups

Group	Apparent Porosity %	Cold crushing Strength Mpa	Flexural Strength Mpa
A	10-21	18.353- 50.293	8.369-26.184
C	23-31	24.36- 54.864	3.819-17.848
D	27-36	22.352-67.889	3.877- 14.738
E	25-35	22.223-59.842	4.148-16.671

Table (2)
Mean specific heat results

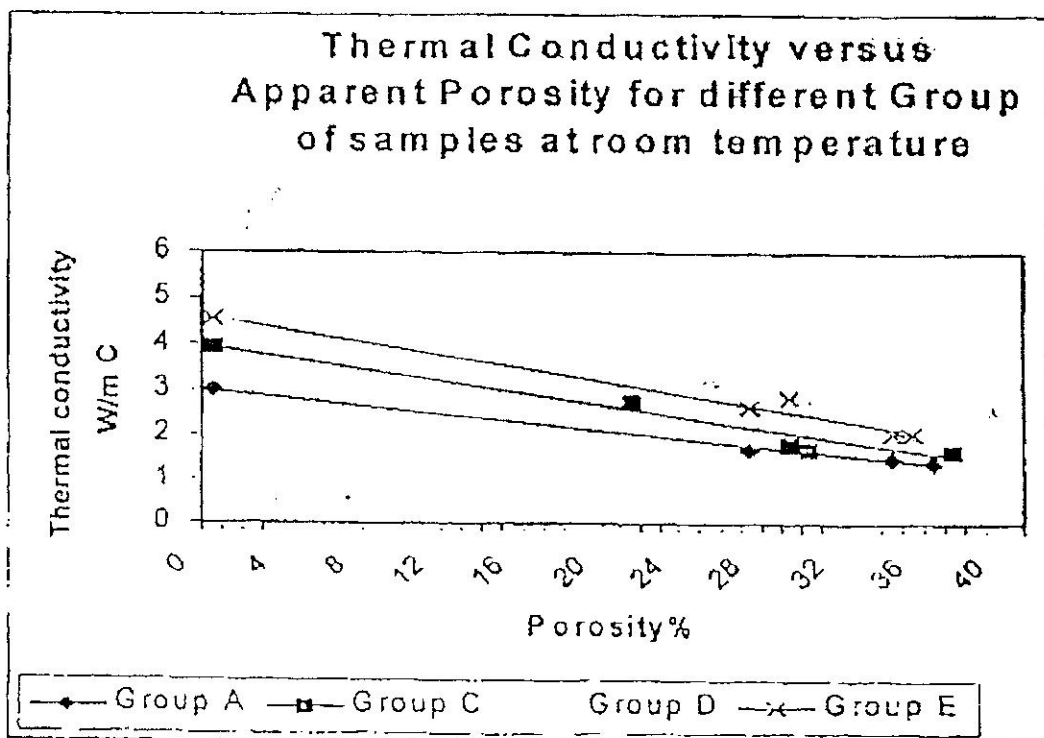
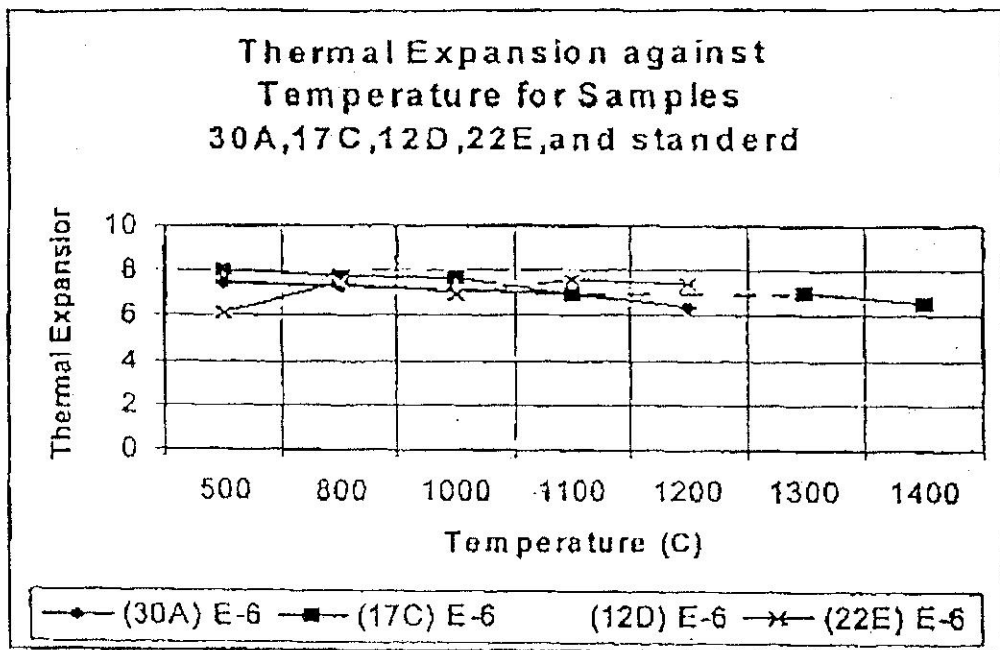
Group	Measured Mean Specific heat	Specific heat from some literatures (J/gC)
A	0.8999	0.733
C	0.90586	0.763
D	0.82098	0.88
E	0.8438	
<u>Average</u>	<u>0.86735</u>	<u>0.792</u>

Table (3)
Thermal Expansion coefficient of Mullite Samples

Temperature C ^o	Samples (30A)x10 ⁻⁶ C ^o	Samples (17C)x10 ⁻⁶ C ^o	Samples (12D)x10 ⁻⁶ C ^o	Samples (22E)x10 ⁻⁶ C ^o
20 - 500	7.4925	7.995	6.32	6.1575
20 - 800	7.3275	7.74	6.725	7.447
20 - 1000	7.08	7.6875	7.225	6.975
20 - 1100	6.975	6.975	7.75	7.523
20 - 1200	6.3825	-----	6.925	7.425
20 - 1300	-----	7.05	-----	-----
20 - 1400	-----	6.5475	-----	-----

Table (4)
The Thermal conductivity of different group sample of Mullite

Group	Porosity %	Thermal Conductivity W/m C
A	0.0	3.0222
	27.632	1.724
	34.83	1.482
	<u>36.44</u>	<u>1.4079</u>
C	0.0	3.958
	21.402	2.772
	29.417	1.825
	30.04	1.6857
D	<u>36.962</u>	<u>1.656</u>
	0.0	5.397
	25.017	3.237
	28.921	2.976
E	35.753	2.654
	<u>39.036</u>	<u>2.5055</u>
	0.0	4.566
	26.718	2.641
	29.08	2.832
	34.316	2.0735
	<u>35.208</u>	<u>2.0624</u>



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الخواص الميكانيكية و الحرارية للمولايث المحضر من الخامات العراقية

صلاح عبد الوهاب شيت خطاب

أستاذ مساعد - شعبة الفيزياء الطبية
كلية طب الكندي - جامعة بغداد

الخلاصة

تمت دراسة بعض الخواص الميكانيكية للمولايث المحضر من أطوار مختلفة للالومينا وأظهرت الفحوصات للنماذج بان الصلادة كانت بحدود (١٨-٥٤) ميكاباسكال والتباين ناتج عن تأثير نسبة الفجوات في النموذج. أما مقاومة التثبي فكانت بحدود (٢٦,١-٣,٨) ميكاباسكال. الخواص الحرارية التي تم قياسها للنماذج أظهرت بان الحرارة النوعية كانت بحدود (١٩,٢٨) جم.م^٣/جول، أما معامل التمدد الحراري للعينات فكانت بحدود (٧,٦٨-٧,١٨) $\times 10^{-6}$ لكل درجة سليزية، وبطرف حراري (٢٠-١٠٠) درجة سليزية وهذه النتائج بينت إمكانية تصنيع واستخدام المولايث في الصناعات الحرارية المختلفة تطابق النتائج مع الدوريات المنشورة.