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Surface Properties and Weibull Modulus of Silicon Carbide Kaolin Composites Improved by Adding Alumina

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ABSTRACT

Refractory ceramics were prepared from Silicon Carbide (SiC) as a main phase bonded with different weight percentages of Iraqi White Kaolin (20, 40, 60 and 80 wt%). After that, two weight percentages of micro and Nano size of alumina (5wt %) were added to all SiC-Kaolin mixtures. The samples were shaped by the axial pressing method after mixing well, then dried and Fired at 13000°C. The structural and mechanical properties were measured such as atomic force microscope AFM and diametrical strength (Weibull modules). AFM showed that Adding 80wt% Kaolin without adding alumina leads to reducing the surface roughness, average grain size diameter, and Root main square (RMS)nm from 210.9nm to 93.30nm, from 814.175nm to 509.3nm and from 377.0 to 100 nm. Adding micro and Nano Al_2O_3 with a ratio of 5wt% led to a reduction the the surface roughness, average grain size diameter, and Root main square (RMS)nm for Micro alumina from 93.30 nm to 84.06nm, from 509.3 to 498.9 nm and from 100 to 87.15 nm. Also for Nano alumina from 93.30 to 35nm, from 509.3 to 498.9nm, and from 100 to 41.8 nm. Weibull modules showed The optimal values for the prepared specimens in terms of specimen homogeneity and the best mechanical properties were for the mixtures consisting of 20 wt% of kaolin, 80 wt% of silicon carbide, and 5 wt% of nano Alumina. The importance and aim of this work is to prepare of refractory ceramic for oil industry applications. and improve the properties of refractories by using silicon carbide as a base material bonded with Kaolin in different ratios, then studding the effect of Micro and Nano Alumina on the properties of the refractory to choose the ratio to optimum for this application.

Keywords: *Refractory, Ceramics, Silicon Carbide, Alumina, Kaolin*

1. INTRODUCTION

Refractory materials are those that can survive at high temperatures while retaining their physical and chemical properties in a furnace environment. Porous refractory ceramics are very important since has thermal applications can be attributed to their unique collection of properties, which include low density, high thermal insulation, good specific strength, and lightweight [1, 2,3,4].

Silicon carbide (α-SiC Carborundum) is classified as one of the most important advanced refractories, due to its properties that make it used in various parts of thermal systems. These distinctive properties are high resistance to thermal shock, high mechanical resistance, and good chemical resistance, These properties make SiC used in oil refinery applications. Covalent bonds make up most of the crystalline structure of SiC, making it difficult to sinter. Raising the sintering temperature to complete the process of granular diffusion and complete sintering leads to the oxidation of SiC and a thin layer of silica [5,6,7].

Clay minerals, particularly kaolin, are among the greatest resources for making refractories because they are readily available locally, inexpensive, and simple to produce. Natural deposits such as kaolin are created when feldspathic rocks, granite, and other materials are weathered [8]

Kaolin is the base material for the production of mullite refractories because it primarily contains silica (SiO_2) and alumina (Al_2O_3). Kaolin is therefore employed in the production of mullite refractories [9,10].

The (Al_2O_3-SiO_2) system's stable phase is mullite. Although it occurs infrequently as a natural mineral, the mullite phase is frequently seen in ceramics created by humans. A key part of aluminosilicate ceramics is mullite. Mullite's special qualities, including its exceptional high-temperature strength, low density, low thermal expansion, strong thermal shock resistance, extraordinary creep resistance, chemical stability, and great mechanical performance, have drawn a lot of interest to mullite ceramics Monolithic mullite [11,12,13,14].

Alumina is a ceramic oxide that can be produced by extracted from bauxite. Alumina (Al_2O_3) is found naturally in the mineral corundum. In the industrial world, a significant resource also contains iron (III) oxide (Fe_2O_3), silica (SiO_2), and 30–54% alumina. K. Bayer created the procedure, sometimes known as the Bayer process, in 1887. Alumina dissolves in a hot sodium hydroxide solution [15,16,17]

In 2018, Shihab A. Zaidan[18] Manufactured porous refractories from Iraqi Kaolin by adding expanded polystyrene waste the results show the addition of EPS showed good results in the formation of pores without distorting the dimensions of specimens and without any cracks. In addition, it is possible to use these thermal insulators at temperatures up to 1300 C.

In 2019, Enas Muhi Hadi, Sahar issa Hussain[19], prepared porous refractory ceramic from kaolin and Alumina by adding burned and raw wheat straw the results show that Adding alumina enhanced the formation of the mullite and which improved the physical and mechanical properties of the refractories, achieved the desired aim which is the formation of mullite in refractories at a higher rate.

In 2021 F. Hassan [20], Kaolin and bentonite were used to make refractory mortar. These refractories were made from Iraqi clay (kaolin or bentonite) with grog of medium alumina bricks that are suited for bonding medium alumina bricks. Then evaluated the bonding strength of the refractory, and also investigated the use of ready-made refractories as furnace lining. The results show that the bond strength increases when increasing the clay ratio from 2-3% (kaolin or bentonite).

In this work, SiC was used as a base material, and kaolin as a binding material using different ratios, to prepare ceramic refractories. Micro and nano alumina was supplemented with a mixture of ceramic to enhance the refractory properties.

2. EXPERIMENTAL PROCEDURE

The samples were created using a SiC (99.5 %) and (10 µm <particle size < 38 µm), supplied by Struers company. Kaolin was obtained from the Ministry of Industry and Minerals' Stat Establishment of Geological Survey and Mining provided the kaolin clay. According to Table 1, it has a high amount of silica and a low percentage of alumina. Jaw crushers were used to reduce kaolin stone to small particles, then a mortar was used to turn it into fine powder. A sieve shaker was then used to sieve the resulting powder (£ 45 µm). SiC/Kaolin mixtures were fabricated by different SiC/Kaolin ratios; (20/80, 40/60, 60/40, and 80/20) as shown in table 2. To increase the Mullite Phase after firing Kaolin, the 5 wt% of micro- and nano Al2O3 (particle size £ 30 µm and supplied by Riedel-de Haen) was added to the SiC/Kaolin mixtures (20/80 and 80/20).

Table 1 Chemical analyses of Kaolin

Oxide	**Wt%**
SiO_2	49.38
Al_2O_3	32.72
Fe_2O_3	2.07
CaO	1.19
TiO_2	1.08
K_2O	0.44
Na_2O	0.22
MgO	0.18
L.O.I	12.72

Table 2 Samples codes and weight percentages for all mixtures

Sample Code	**Silicon Carbide (SiC) wt%**	**Kaolin wt%**	**Alumina (Al_2O_3) wt%**
80S-20K	80	20	-----
60S-40K	60	40	-----
40S-60K	40	60	-----
20S-80K	20	80	-----
80S-20K-5A	95 wt% of 80S-20K		5
80S-20K-5 nA	95 wt% of 80S-20K		5
20S-80K-5A	95 wt% of 20S-80K		5
20S-80K-5nA	95 wt% of 20S-80K		5

The disc specimens were prepared by the dry pressing method, the pressing force (50 kN), and the pressing die diameter (20 mm). The samples were fired at a temperature of 1300 according to a specific program that included the stages of phase transformations of kaolin, as shown in Table 3.

Table 3 Stages of sintering

Stage	Temp. ^{o}C	Sintering time (hr)	Soaking time (hr)
First	25 – 700	3	2
Second	700 – 1000	2	1
Third	1000 – 1300	1	2

2.1. Testing

2.1.1 Atomic Force Microscope (AFM)

It's utilized for measuring the average roughness, buildup distribution, granularity, and size, granularity in nano-meter or even at the level of sub-nano. In the present project, a scanning probe microscope (CSPM-5000) instrument was employed for measuring the size of the grain as well as the film's roughness.

2.1.2 Weibull Modulus

When measuring the measured material strength of brittle materials, the Weibull modulus, a dimensionless parameter of the Weibull distribution, is employed to describe the variability. Even under the same testing conditions, the maximum stress that a sample of ceramic or another brittle material can bear before failing may vary from specimen to specimen. Since brittle failure processes start at these weak points, this has to do with how the surface or body of the brittle specimen is distributed in terms of physical faults. Samples will act more equally when defects are spread evenly and consistently than when they are clustered randomly. Strength is better represented as a distribution of values rather than a single value because this must be taken into consideration when discussing the material's strength. When measuring the measured material strength of brittle materials, the Weibull modulus, a dimensionless parameter of the Weibull distribution, is employed to describe the variability. Even under the same testing conditions, the maximum stress that a sample of ceramic or another brittle material can bear before failing may vary from specimen to specimen. Since brittle failure processes start at these weak points, this has to do with how the surface or body of the brittle specimen is distributed in terms of physical faults. Samples will act more equally when defects are spread evenly and consistently than when they are clustered randomly. Strength is better represented as a distribution of values rather than a single value because this must be taken into consideration when discussing the material's strength.

If the probability distribution of the strength, X is a Weibull distribution with its density given by:

$$f(x; x_0, \lambda, k) = \begin{cases} \frac{k}{\lambda}\left(\frac{x-x_0}{\lambda}\right)^{k-1} e^{-((x-x_0)/\lambda)^k} & x \geq x_0, \\ 0 & x < x_0, \end{cases}$$

k is the Weibull modulus.

The value of k shows the kind of failure being experienced. If k<1, then the failure rate decreases with time. This implies that the weak and defective parts fail in the beginning, with the harder sections surviving. If k=1, the rate of failure remains constant. This implies that there is random failure occurring. Thus, there should be some external factor strong enough to cause random failure irrespective of whether the section is strong or weak. If the value of k>1, the rate of failure increases over time. This points to some kind of aging process, the weakening of the material with time.

3. RESULTS AND DISCUSSION

This section shows the results of our prepared refractories.

3.1. Atomic Force Microscopy (AFM)

The topography images of SiC refractory taken by using AFM were illustrated in Fig Fig. 2, the measurement range for all specimens is (10×10 μm). This figure shows 3D topographies profiles under different additives, where the max high (peak-peak) of the grain size was decreased with Kaolin additions increases. The granular size depends on the nature of the materials used.

Figure (1) shows the Homogeneous and symmetrical distribution of particle size, as well as obtaining a uniform surface in granular distribution and free of agglomeration.

Figure (1A) shows the Homogeneous distribution of granular size and high surface roughness due to the increased percentage of silicon carbide 80% with kaolin 20% and thus causes a low surface area.

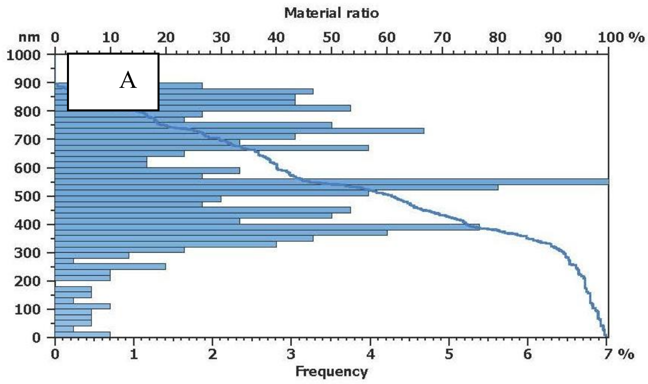
Figure (1 B, C) Adding 5% of micro- and nano-alumina to the silicon carbide refractory leads to a slight decrease in the roughness, and thus the particle size decreases, which leads to increases in the surface area.

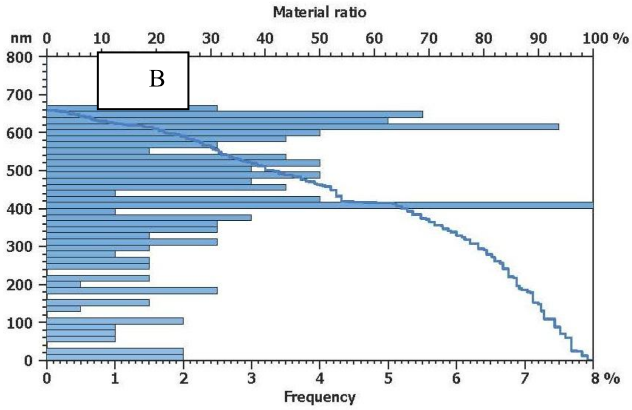
Figures (1 D, E, and F) Show that Adding 80wt% kaolin leads to reducing the surface roughness and obtaining a smaller grain size and a homogeneous distribution of the grains. Thus, causes increases in the surface area due to the increase in the number of grains per unit area, and the grain size affects the mechanical properties, especially fracture toughness.

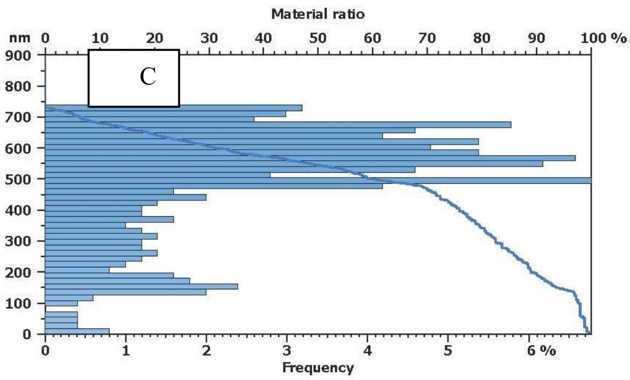
The addition of micro- and nano-alumina hinders the crystalline growth of the crystal phases resulting from firing kaolin, which are mullite, cristobalite, and the glassy phase.

Thus, the grain size and roughness of the surface decrease, and the surface area decreases due to the increase in the number of grains, and the resulting crystalline phases do not have crystal growth, which leads to improved mechanical properties.

The topography of the images AFM shows a decrease in the RMS with increasing addition rates of kaolin, Table 4 shows a decrease in the Root main square and this leads to an increase in the resistance of the sample surface to wear as the addition rate increases. [21]







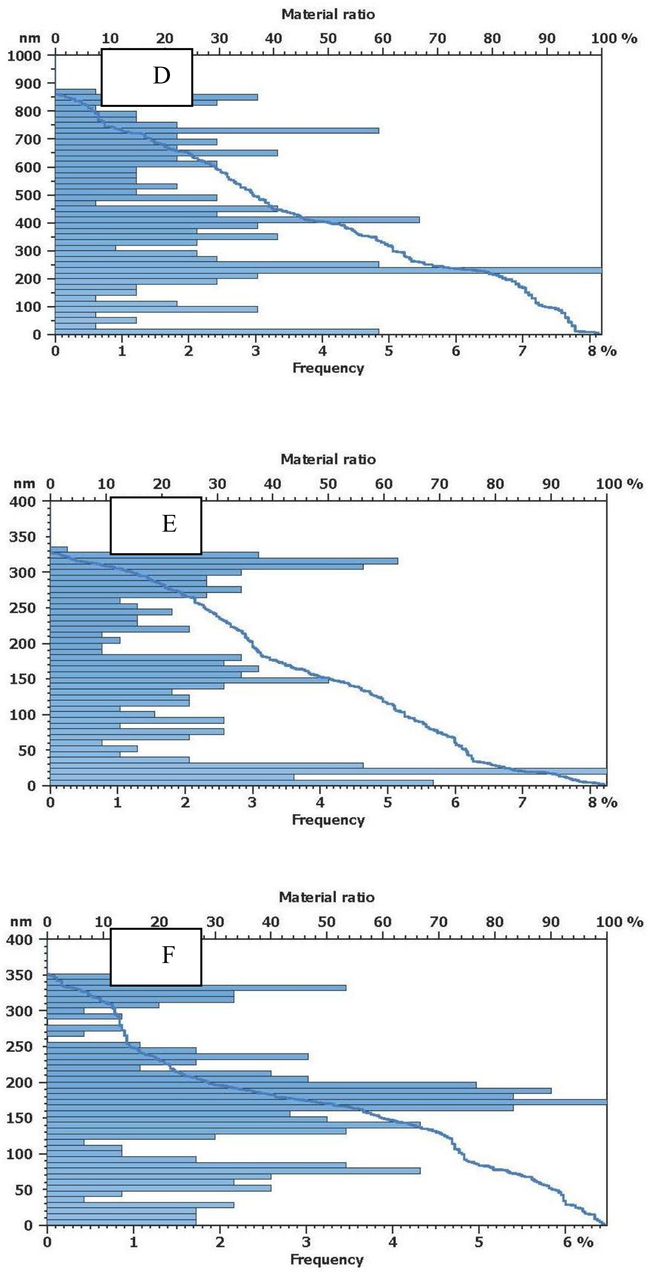


Figure 1. Statistical distribution of grain size for refractory ceramic with different addition.

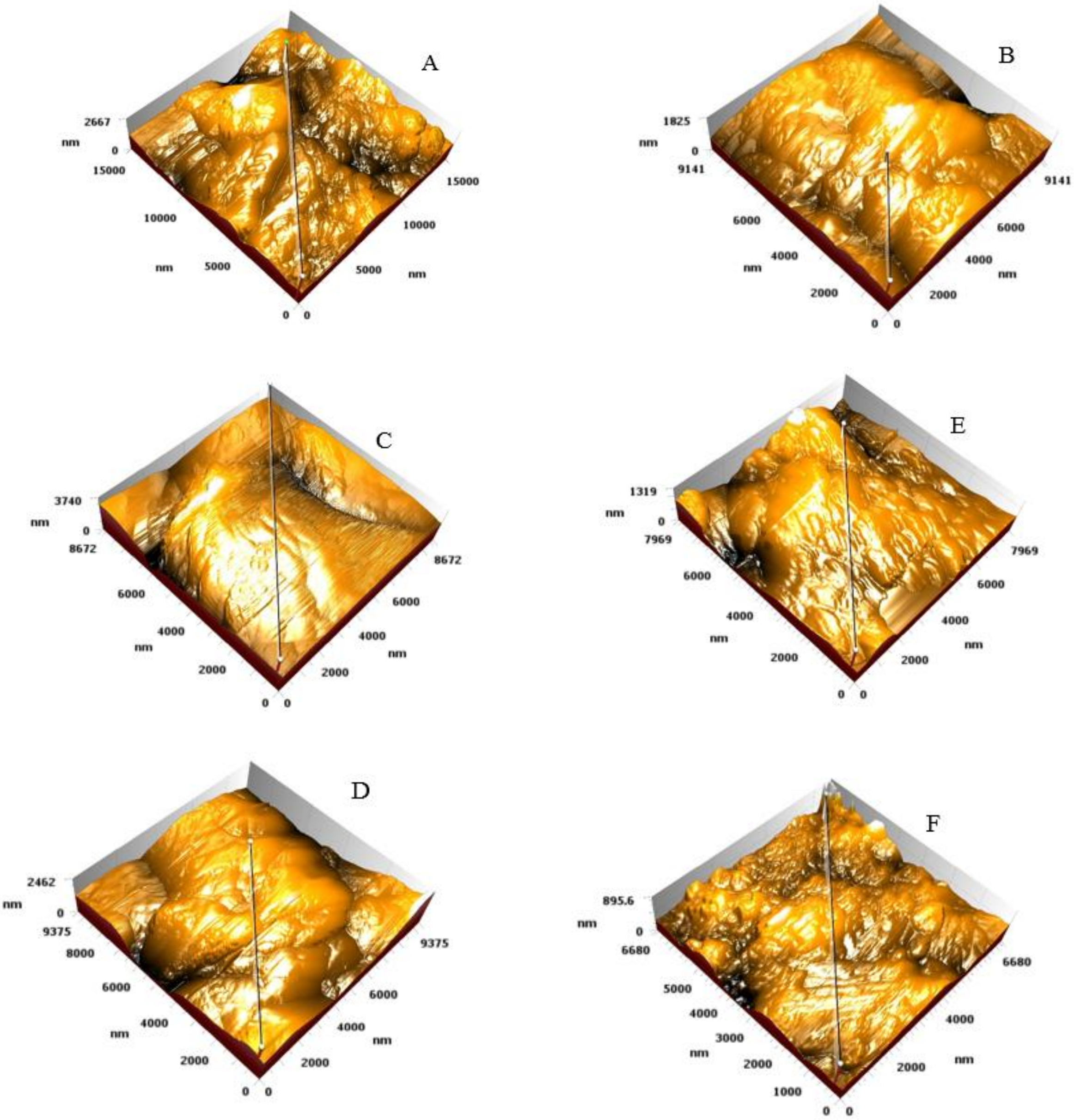


Figure 2. 3D AFM scan data for SiC refractory (A)S 80wt%+K 20wt%, (B) S80wt%+K20wt%+5mAwt%, (c) S80wt%+K20wt%+5nAwt%, (D) S 20wt%+ K 80wt%, (E) S 20wt%+ K 80wt%+5mAwt% and(F) S 20wt%+ K 80wt% +5nAwt%.

Table 4 Avg.Grain size Diameter, Roughness Average, and root main square value for SiC Refractory

SiC Refractory Specimen	AVG. Grain size Diameter (nm)	Roughness average(nm)	Root main square (RMS)nm
80wt%SiC+20wt%Kaolin	814.175	210.9	377.0
80wt%SiC+20wt%Kaolin+5wt% micro Al_2O_3	528.525	190.6	280.9
80wt%SiC+20wt%Kaolin+5wt% nano Al_2O_3	516.8	111.1	124.5
20wt%SiC+80wt%Kaolin	509.3	93.30	100
20wt%SiC+80wt%Kaolin+5wt% micro Al_2O_3	498.9	84.06	87.15
20wt%SiC+80wt%Kaolin+5wt% nano Al_2O_3	442.0	35	41.8

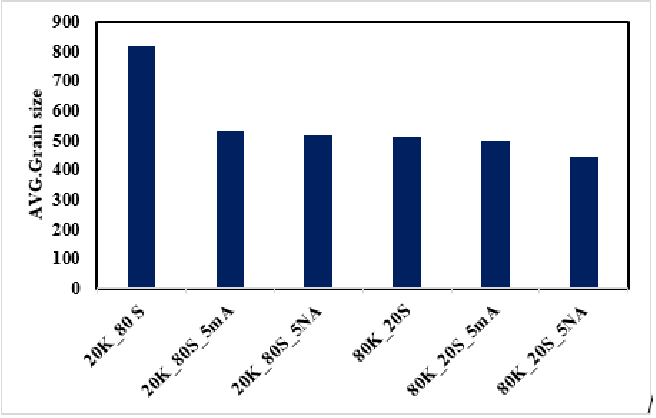


Figure 3. Average grain size for SiC refractory with different addition.

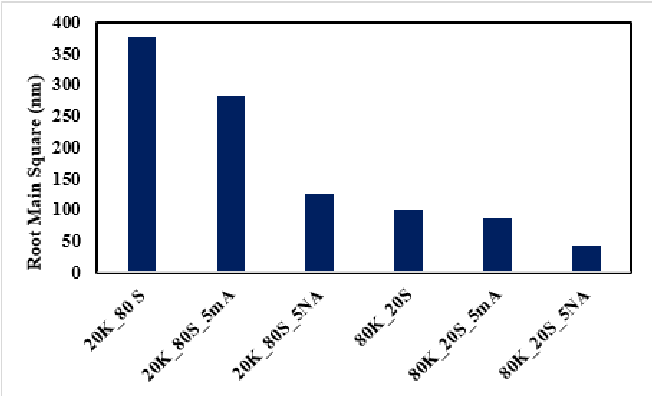


Figure 4. Root Main Square for SiC refractory with different addition.

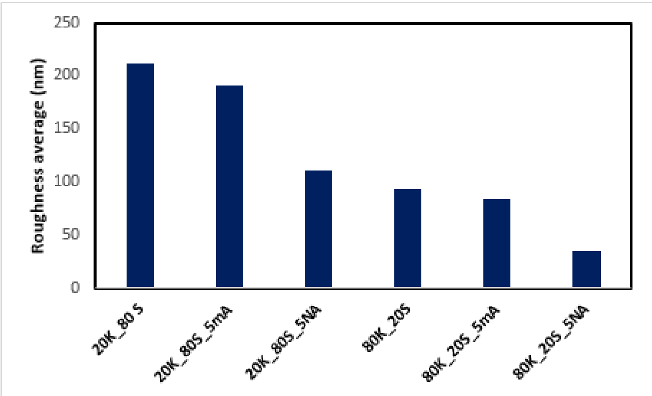


Figure 5. Roughness average for SiC refractory with different addition.

3.2 Mechanical Properties: Weibull Modules

The Weibull modulus is one of the best statistical methods to describe this randomization, and it is possible to find variations in measurements. Fig6 shows the change in the values of the characteristic mechanical strength (x-axis), which represents the logarithm of the greatest mechanical stress (ln|σEmax|) where the mechanical breakdown occurs. The y-axis represents the logarithm-logarithm inverted probability of survival of the sample without breakdown (lnln|1/Ps|), note that the probability of failure can be found from the relationship (Pf = 1 - Ps).

Figure 4 shows a change in the probability of survival with mechanical strength, that mains the Weibull modules associated with the normalized strength. The Weibull modulus was calculated from the slope of the straight line.

The Weibull modulus depends largely on the history of the specimens: the properties of the raw materials, the manufacturing processes, and the type of phases emerging after the sintering processes. As is known, silicon carbide is one of the advanced ceramic materials, while kaolin clay is classified as a traditional ceramic material. The addition of clay came as a necessity to bind the silicon carbide powder by employing the plasticity property of the clay to achieve compaction after pressing. Therefore, according to the classification of composite materials, clay is the base (matrix), while silicon carbide is the powder dispersed in the composite. During firing processes, many phase changes occur in the clay composition, perhaps the most prominent of which is the emergence of glassy phases of silicates. These phases generate many microscopic defects and microcracks in the basic structure of the composite. Therefore, we notice from the results of calculating the Weibull modulus that with small percentages of clays added (specimens 20K-80S), the Weibull modulus is high, reaching a value of approximately (m=50). This high value came due to the lack of glassy phases and the defects resulting from the small amount of clay addition. An increase in clay to more than 20% led to a deterioration in the values of the Weibull modulus and its decline to (m=16) when 80% clay was added (specimens 80K-20S), as shown in Figure 7.

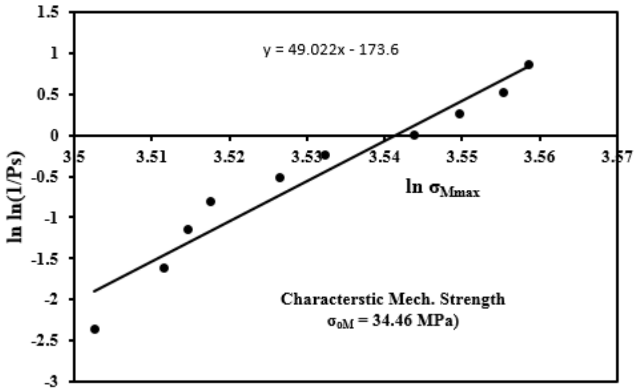
Adding an amount of micro alumina powder at a rate of 5% (specimen 80K-20S-5 μA), led to a slight improvement in the Weibull modulus, reaching (m=20) compared to (m=16) for specimens (80K-20S). This is a result of the decrease in the glassy phases and defects with the increase of the mullite phase (3Al2O3.2SiO2).

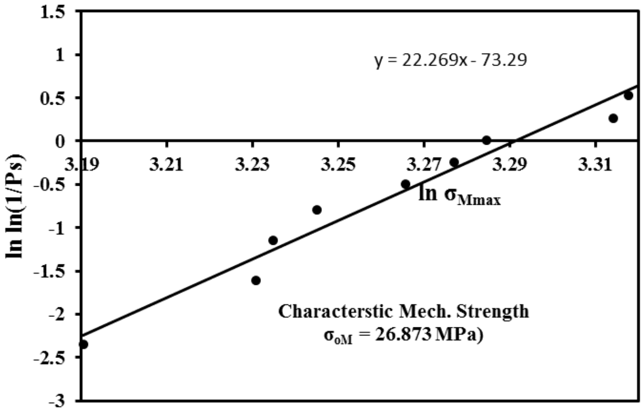
While increasing the percentage of micro alumina added to 10% (80K-20S-10μA), led to better properties improvement and the structure of the specimens became more homogeneous. This explains the increase in the value of the Weibull modulus to reach (m=24), compared to the specimen (80K-20S).

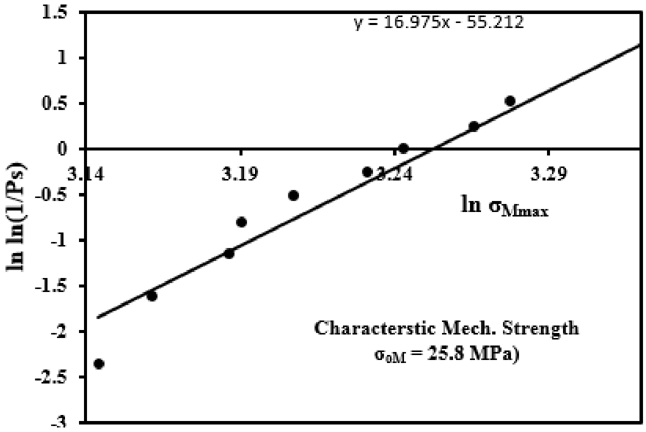
Adding 5% micro-alumina (specimens 20K-80S-5μA) did not lead to an improvement in the Weibull modulus, and this means the emergence of defects in the structure of the composite material and thus inhomogeneity. While increasing the addition rate to 10% micro alumina, the Weibull coefficient improved compared to the addition of 5%. [22]

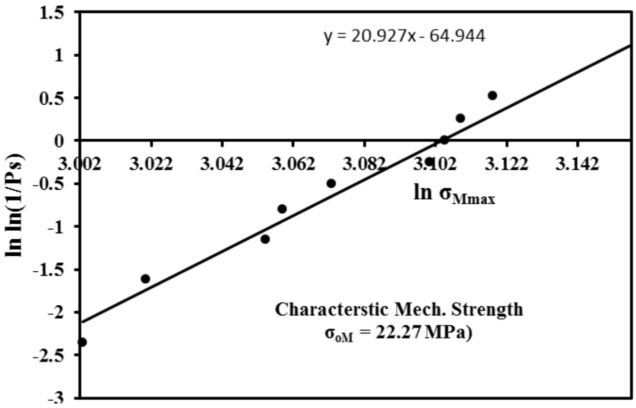
As for nano-alumina additives, they have been characterized by their large spread in the ceramic body because nano-powders are characterized by an increase in surface area. Therefore, the value of the Weibull modulus increased when adding 5% nano-alumina (specimens 20K-80S-5nA), maintaining its value compared to without addition, despite the greater formation of the mullite phase. While the value of the Weibull modulus improved slightly when 5% nano-alumina was added to the specimens (80K-20S-5nA), reaching m=18.

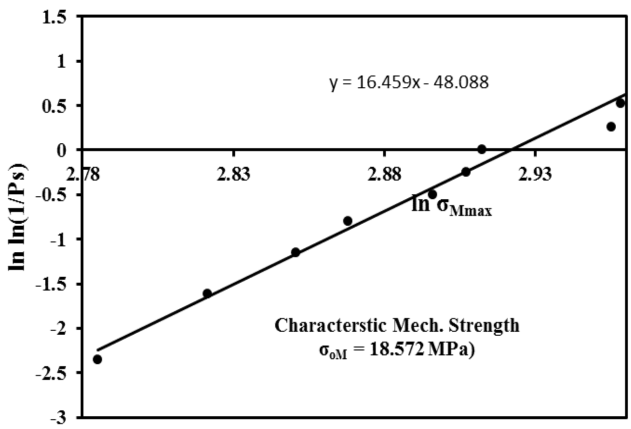
The characteristic strength values shown in Figure 8 represent reliable values for calculating strength. It is possible for a decrease in properties to occur at lower values and by a specific percentage depending on the required design. After determining the value of the characteristic strength from the straight line equation (equation no.), it was noted that increasing the percentage of clay addition relative to silicon carbide led to a deterioration of the characteristic strength and reaching its lowest value at (80K-20S) and reached 18.5 MPa. The micro-addition of alumina slightly improved the characteristic strength, and it improved even better with the nano-addition, reaching its best value for specimens (20K-80S-5nA) to reach 45 MPa. These specimens represent the optimal values for the two cases: the Weibull modulus and the characteristic strength.[23,24]

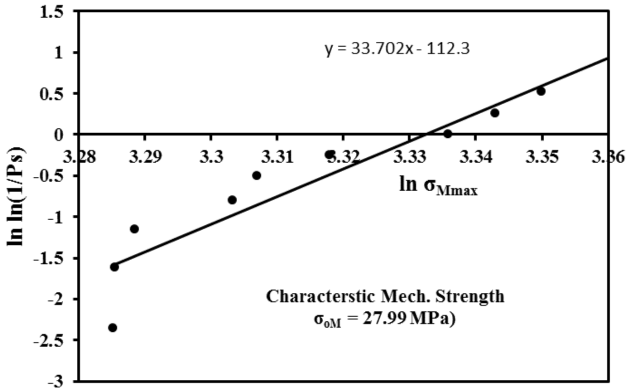


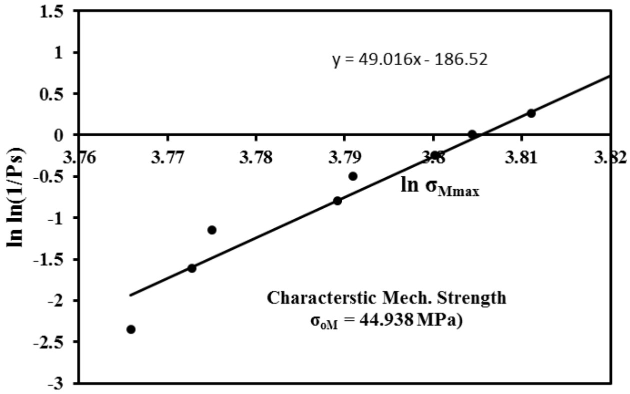

y = 22.269x - 73.29
ln ln(1/Ps)
ln σMmax
Characterstic Mech. Strength
σoM = 26.873 MPa)

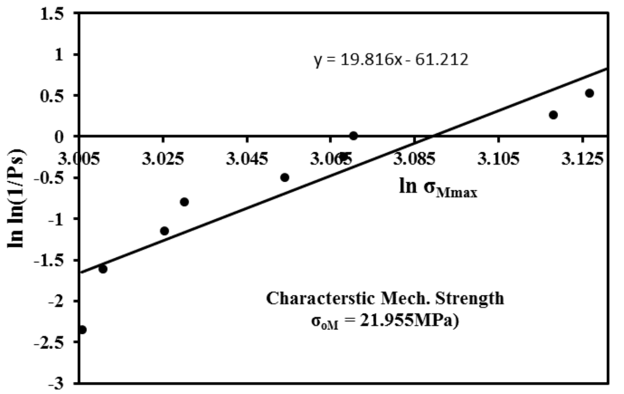

y = 16.975x - 55.212
ln ln(1/Ps)
ln σMmax
Characterstic Mech. Strength
σ0M = 25.8 MPa)

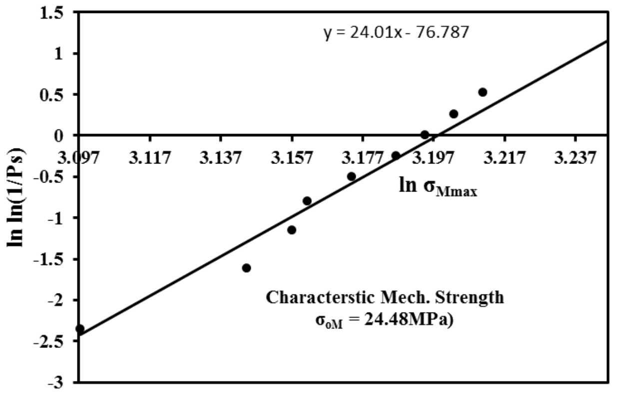

y = 20.927x - 64.944
ln ln(1/Ps)
ln σMmax
Characterstic Mech. Strength
σoM = 22.27 MPa)


y = 16.459x - 48.088
ln ln(1/Ps)
ln σMmax
Characterstic Mech. Strength
σoM = 18.572 MPa)


y = 33.702x - 112.3
ln ln(1/Ps)
ln σMmax
Characterstic Mech. Strength
σoM = 27.99 MPa)


y = 49.016x - 186.52
ln ln(1/Ps)
ln σMmax
Characterstic Mech. Strength
σoM = 44.938 MPa)


y = 19.816x - 61.212
ln ln(1/Ps)
ln σMmax
Characterstic Mech. Strength
σoM = 21.955MPa)


y = 24.01x - 76.787
ln ln(1/Ps)
ln σMmax
Characterstic Mech. Strength
σoM = 24.48MPa)

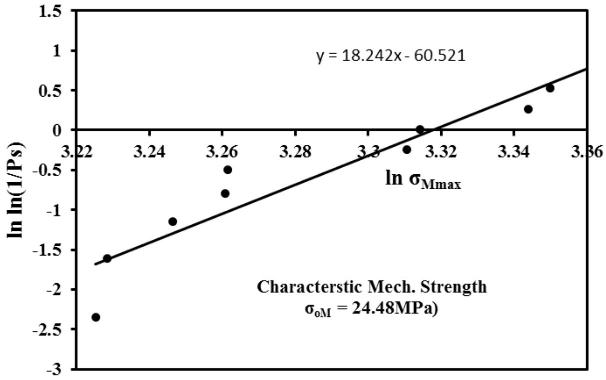


Figure 6. Weibull distribution of the mechanical strength with different samples.

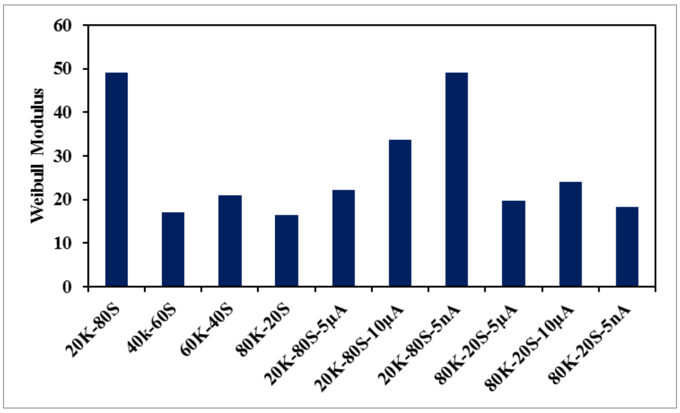


Figure 7. Weibull modules depend on characteristic mechanical strength.

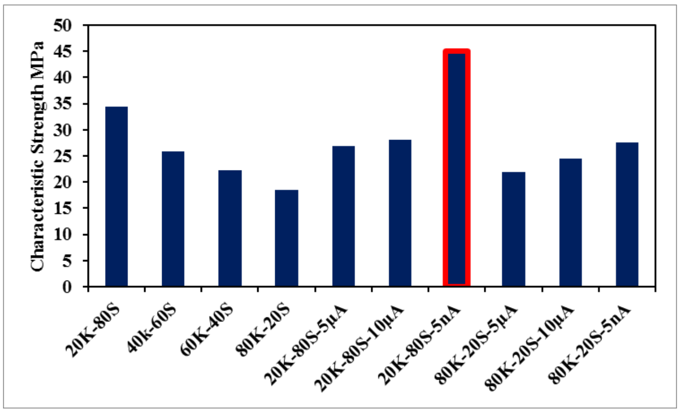


Figure 8. Weibull modules depend on characteristic mechanical strength.

4. CONCLUSION

Despite the big difference between the properties of traditional kaolin clay and advanced silicon carbide, there is compatibility between them when mixed in refractory composites (SiC/Kaolin). This was evident in the absence of any macroscopic defects in the prepared samples.

The addition of alumina contributed to the increase of the mullite phase through interaction with cristobalite resulting from firing kaolin, or through interaction with silicon oxide resulting from the oxidation of SiC within the boundaries of the sample surface.

Kaolin provides the plasticity necessary for the success of the dry pressing process and for achieving maximum compaction of powders and high homogeneity in the dimensions of samples.

The decrease in the values of the Weibull modulus came as a result of the increase in the addition of clays to silicon carbide, and this means the non-homogeneity of the distribution of the emerging phases, especially the effect of the glassy phases resulting from the phase transformations of the kaolin clays. The addition of alumina modified the values of the Weibull modulus due to the formation of the mullite phase.

The optimal values for the prepared specimens in terms of specimen homogeneity and best mechanical properties were for the mixtures consisting of 20 wt% of kaolin, 80 wt% of silicon carbide, and 5 wt% of nano Alumina.

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