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A STUDY ON FATIGUE PROPERTIES OF COMPACTS OBTAINED FROM READY-MADE AND POLYMER GRAFTED ALUMINA

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ABSTRACT

This study was devoted to outlining the fatigue behaviour of alumina as a widely used ceramic material and to predict their life under cyclic loads. Two types of α -alumina have been studied to predict their fatigue behaviour and the effects of physical properties, particularly, and porosity and their pore size; and mechanical properties such as fracture strength, modulus of elasticity, and toughness of materials; in addition to pressing loads. The first type was alumina made especially for pressing. The second type of alumina was commercial high purity grafted with random copolymer (n-butyl metacrylate-methacrylic acid) as a lubricant. Dry pressing forming technique with different loads has been used to form different alumina compacts. They were fired in two stages, 1200°C and 1600°C. It was found that porosity affects fatigue behaviour in a complicated manner. Firstly, it weakens the ceramic material. Secondly, pore sizes, particularly surface pores, have a great harmful influence on fatigue life. Therefore, reduction in porosity and good surface finishing can elongate the fatigue life. Furthermore, it was predicted that specimens of alumina made especially for pressing, which was pressed under relatively low loads (0.5, 1, 2 kN) were relatively better than those pressed under higher loads. That is due to the initiation of fractures in the specimens, pressed under higher loads with worst case for specimens, pressed under 9kN, on the contrary of their static mechanical behaviour. Gradual enhancement of fatigue properties for specimens, pressed under higher loads (11-16 kN) because of toughening mechanisms, which emerges due to the more closeness, higher internal friction, and meandrous paths of cracks. Whereas grafted alumina is better in static mechanical properties, and their fatigue properties are close to, or even better than their predecessors. Design and applications of ceramics should be based upon $K^{\bar{T}H}$.

Keywords: alumina, metacrylate-methacrylic acid, grafted, polymer

1. INTRODUCTION

Advanced ceramic materials are used in modern industries due to their unique properties such as chemical stability, thermal and electrical resistance, and mechanical stiffness. Therefore, these materials could withstand as superior alternatives to traditional materials (metals, etc.) in some modern applications such as shielding of space shuttles, blades of high speed turbines under high temperatures, and in highly corrosive environments. The serious drawback of ceramic materials is its brittleness. About 85% of mechanical failures of materials are ascribed to fatigue phenomena, emerging from dynamic loads. It is of great importance to thoroughly, investigate the ceramic behaviour under such conditions, to evaluate service life, to predict and to overcome or at least to avoid catastrophic failure of these materials [1].

From mechanical properties point of view, the applications are of more static rather than dynamic nature. The use of ceramics in situations where cantilever stresses exist has been so far largely avoided. The main drawback of ceramics is their inherent brittleness and the resulting low impact strength [2].

Due to the limited motion of dislocations in ceramics at ambient temperatures, strain hardening and consequent crack propagation under cyclic loads might not be expected; therefore, ceramics were not susceptible to fatigue damage. The properties of a ceramic are determined by its composition and its microstructure produced as a result of its fabrication. Unlike metals, the microstructure cannot in general be changed by working or further treatment. Most ceramics start as a powder or mixed of powders. Ceramics with homogeneous microstructures such as glass or very fine-grained, singlephase ceramics seem to be proof of fatigue damage.

At elevated temperatures, it seems that ceramics do not show degradation in fatigue resistance under cyclic loading. Conversely, their resistance to fatigue is enhanced due to bridging of the crack surfaces by grain boundary glassy phases [3, 4].

Fatigueis the process by which the strength of a structural member is degraded due to the cyclic or fluctuating load or strain. The fluctuated load that a structure can withstand is often significantly less than the static load that can withstand [5].

Fatigue failure is often catastrophic, insidious, and suddenly occurs without significant warning. Fatigue failure is brittle-like in nature, even in ductile metals, because there is no or little gross plastic deformation associated with failure. The applied stress may be axial (tension-compression), flexural (bending), or torsional (twisting) and modes of imposing fluctuating loads may be sinusoidal (symmetrical about a mean zero stress level or asymmetrical) or random fluctuating [6].

The fatigue of ductile and brittle materials differs only in the relative importance of the intrinsic and extrinsic mechanisms. In metallic materials, intrinsic damage mechanisms involve processes of creating microcracks or voids, e.g. by dislocation pileups or

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interface decohesion, in the highly stressed region ahead of the tip, causing failure by cleavage, intergranular cracking, or micro void coalescence. These mechanisms under cyclic loads involve the repetitive blunting and resharpening of the crack tip. Conversely, extrinsic shielding mechanisms result from the creation of inelastic zones surrounding the crack wake or from physical contact between the crack surfaces by wedging, bridging, sliding or combinations thereof [7, 8].

The intrinsic mechanisms are an inherent property of the material, and thus are active irrespective of the length of the crack or the geometry of the test specimen. Conversely, extrinsic mechanisms, act in the crack wake and are thus strongly dependent on crack size and (to a lesser extent) geometry. They are responsible for the development of resistance-curve (R-curve) behaviour, thus have an effective influence on the driving forces required for continued growth of the crack [9].

Glasses and un-toughened ceramics are essentially immune to cyclic fatigue, the generation of a nonlinear stress-strain curve with toughened ceramics results in their susceptibility to premature fatigue failure under cyclic loading. Whereas the cyclic processes in metal fatigue are predominantly intrinsic in nature, the cyclic fatigue processes in ceramics are extrinsic. The mechanism by which the crack advances is thus identical under cyclic loading as it would be in a single overload cycle. This is associated with the clear dependency of growth rates on K_{max} rather than ΔK , and a similar appearance of fracture surface under cyclic and monotonic loading [7]

This study is intended to predict fatigue behaviour of alumina ceramics quantitatively. This includes fatigue life prediction, threshold stresses and stress intensity estimation, below which fatigue failure is unexpected, and effects of pressing loads and grafting with polymers on the fatigue behaviour of such materials. It is of high importance to point out that the results of this study do not necessarily agree with the actual results, which should be conducted. This study tries to incept mathematical modeling for the fatigue behaviour of ceramic materials.

2. EXPERIMENTAL

The main aim of this study is to predict the fatigue behaviour of alumina ceramics on a quantitative base and to obtain some useful data, which can be used in design with such materials under cyclic loading conditions.

Care should be taken that ceramics are sensitive materials external influences (mechanical, environmental, etc.) and internal defects as well. Therefore, an extensive amount of experimental work should be accomplished to obtain reliable data. In addition, statistical approach should be taken into account due to the wide distribution of inherent defects, which possibly propagate until failure.

3. MATERIALS

Two types of alumina were used in this work. The first type of alumina was alumina made especially for pressing; it was purchased from the Alcan Company of England. It was dry pressed under loads of 0.5, 1, 2, 4, 7, 9, 11, 13, and 16 kN.

The second was aluminatype Reynolds R172 DPM obtained from MIT (USA). It was grafted with random copolymer (n-butyl methacrylate-methacrylic acid) as a lubricant. The specimens were pressed under 9kN.

The specimens of the two types were fired in two stages (1200°C and 1600°C). The final phase was αalumina (corundum).

4. FATIGUE LIFE PREDICTION

Basically, physical and mechanical data on the work of Al-Lami [10, 11] were adopted in this study, in spite of the static nature of its tests. Recalling Paris-Erdogan Equation and its modification [7]:

$$da/dN = C (\Delta K)^{m}$$

$$da/dN = A (K_{max})^{n}. (\Delta K)^{p}$$

n+p=m, in ceramics $m\approx 15\sim 50$ (or even 100), and n>>p. $\Delta K = K_{max} - K_{min}$. Indeed K_{max} and K_{min} may take any value {positive (tensile) or negative (compressive) stress intensities}, provided that $K_{max} \ge K_{min}$. Under compressive stress, fatigue is not expected [2], provided that the stress does not cause crushing. Therefore, we can propose that $\Delta K = K_{max} - K_{min} = K_{max} - 0 = K_{max}$, and this is the worst case. Knowing that $R = K_{min} / K_{max}$, then $\Delta K = K_{max}$ (1-R) and $(1-R) \le 1$. Therefore:

$$da/dN = A (K_{max})^m (1)$$

$$dN = A (K_{max})^m da = A [Y\sigma\sqrt{(\pi a)}]^m da$$
 (2)

Taking the integration of both sides the left-hand side from 0 to N and the right-hand side from $a=a_i$ to

$$N_f = 2 \left[\pi^{0.5} Y \sigma_f \right]^{-m} \left[a_i^{1-m/2} - a_c^{1-m/2} \right] / A (m-2)$$
 (3)

Furthermore, Y is a dimensionless geometric factor that depends on the geometry of the slit. It takes values around unity. If a/b is very small Y=1. For threepoint bending, an acceptable value of Y is (1.1) [5], and plotting the relationships between da/dN and ΔK estimate the value of $A = 2 \times 10^{-16}$, and $m \approx 19$ for alumina [7].

$$(a_i)_{ii} = 0.25 \,\mu m$$
 $(a_i)_{ii} = 25 \,\mu m$ $(a_i)_{iii} = 200 \,\mu m$ $a_c = (K_{Ic}/\sigma \, Y)^2/\pi [1],$

where

initial surface crack length or half-length of an a_i : internal crack (m).

length of critical surface crack or half-length of a_c : an internal crack (m).

> N_f : number of cycles, at which fatigue failure takes place.



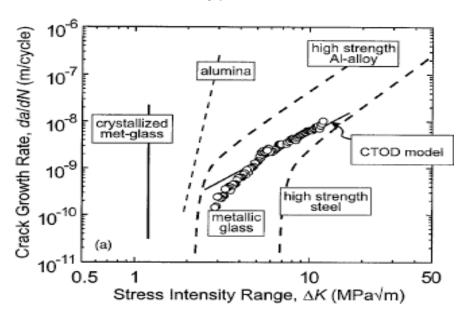


Figure-1. Crack growth rate da/dN versus ΔK , both on logarithmic scales [7].

Using Equation (3), for various samples of alumina, N_f under maximum applied stresses $\sigma_{max} = 40$, 30, and 20 MPa where $(\sigma_{max} < \sigma_f)$. Also σ_{max} for each specimen to achieve $N_f = 10^8$ cycles was computed using Equation (3).

Critical surface energy (G_c) was obtained from the following equation [14]:

$$K^2 = E G_c / (1-v^2)$$
 or $G_c = K^2 (1-v^2) / E$

where v is Poisson ratio, which for alumina is 0.21[1].

For alumina grafted with random copolymer, σ_f was estimated by Ryskewitch Equation. Calculations were based upon n=4 and n=7, than the average values were taken. The differences from the average values were relatively small (< 2.5 %).

5. RESULTS

Theresults obtained are shown in the Tables (1-8) and Figures (1-4) to shed light upon the fatigue behaviour prediction of commercial high purity α -alumina (corundum) having theoretical density 3.987g/cm³, with mean particle size about 0.3 µm[13], and grafted alumina with a random copolymer of n-butyl methacrylatemethacrylic acid having an average molecular weight of 24600[10,11].

Tables (1) and (2) exhibit some physical and mechanical properties for ready-made alumina and grafted alumina with random copolymer (n-butyl methacrylate-methacrylic acid) as a lubricant, respectively.

Tables (3) and (4) show calculated critical surface crack length (a_c), K^{TH} , σ_{max} , and critical surface crack energy for ready-made alumina and grafted alumina with random copolymer (n-butyl methacrylate-methacrylic acid) as a lubricant, respectively.

Tables (5) and (6) list predicted fatigue lives under different cyclic stresses ($\sigma_{max} = 20$, 30, and 40 MPa respectively).

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Table-1. Some physical and mechanical properties of ready-made alumina for pressing [10, 11].

Load (kN)	Bulk density (g/cm³)	App. solid density (g/cm³)	True Porosity (%)	σ _c (GPa)	Hv (GPa)	Hk (GPa)	E (GPa)	Kıc (MPa√m)
0.5	3.015	3.785	24.550	54.000	14.836	15.671	244.703	3.341
1	3.355	3.720	15.890	59.000	14.975	15.818	246.997	3.373
2	3.537	3.587	11.332	69.157	15.253	16.112	251.583	3.436
4	3.602	3.604	9.716	89.402	15.809	16.700	260.756	3.563
7	3.619	3.622	9.290	126.112	15.994	16.985	263.804	3.605
9	3.628	3.631	9.054	132.506	16.599	17.534	273.784	3.742
11	3.639	3.640	8.791	132.498	17.218	18.189	284.006	3.878
13	3.649	3.650	8.528	106.373	17.977	18.189	296.510	4.049
16	3.653	3.653	8.445	88.856	18.225	19.284	301.108	4.950

Table-2. Some physical and mechanical properties of alumina grafted with random copolymer ($Mw_{avg}=24000$), (Load = 9 kN) [10, 11].

Weight of grafted polymer (mg/g)	Bulk density (g/cm³)	App. solid density (g/cm³)	True Porosity (%)	σ _c (GPa)	Hv (GPa)	Hk (GPa)	E (GPa)	K _{Ic} (MPa√m)
12.3	3.863	3.867	3.110	143.69	21.64	22.84	356.86	4.373
13.5	3.867	3.871	3.009	152.00	22.30	23.53	367.65	4.626
16.6	3.887	3.891	2.508	160.67	23.62	24.90	389.20	4.890
18.0	3.906	3.912	2.032	162.84	24.04	25.30	396.00	4.956

Table-3. Critical surface crack length (a_c) , fatigue stress intensity threshold and related maximum stress (σ_{max}) , and critical surface energy (G_c) for ready-made alumina for pressing.

Load (kN)	True Porosity	a c (mm)	K^{TH} (MPa. \sqrt{m})	σ _{max} (GPa)	K^{TH}/K_{Ic}	G _c (J/m ²)
0.5	24.550	1.007	2.648	42.80	0.79	43.60
1.0	15.890	0.860	2.546	44.53	0.75	44.03
2.0	11.332	0.649	2.382	47.96	0.69	44.86
4.0	9.716	0.418	2.169	54.41	0.61	46.86
7.0	9.290	0.215	1.923	67.27	0.53	47.09
9.0	9.054	0.210	1.916	67.81	0.51	48.89
11.0	8.971	0.225	1.937	66.23	0.50	50.62
13.0	8.528	0.381	2.129	55.94	0.53	52.85
16.0	8.445	0.563	2.308	49.89	0.56	53.68

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Table-4. Critical surface crack length (a_c) , fatigue stress intensity threshold and related maximum stress (σ_{max}) , and critical surface energy (G_c) for grafted alumina with random copolymer (Mw_{avg}=24000), (Load = 9 kN).

Weight of grafted polymer (mg/g)	True Porosity (%)	a c (mm)	K^{TH} (MPa. \sqrt{m})	σ _{max} (GPa)	K^{TH}/K_{Ic}	G_c (J/m ²)
12.3	3.110	0.221	1.932	66.66	0.44	51.22
13.5	3.009	0.244	1.964	64.49	0.42	55.64
16.6	2.508	0.262	1.988	62.99	0.41	58.73
18.0	2.032	0.251	1.973	63.87	0.40	59.29

Table-5. Predicted fatigue lives based on Paris-Erdogan Equation modified by Liu and Chen [15] for ready-made alumina for pressing.

Load (kN)	True Porosity (%)	N_f (x10 ¹⁵ cycles); (σ =20 MPa)	N_f (x10 ¹² cycles); (σ =30 MPa)	N_f (x10° cycles); (σ =40 MPa)
0.5	24.550	9.59	4.3	18.29
1.0	15.890	9.59	4.33	18.29
2.0	11.332	9.59	4.33	18.29
4.0	9.716	9.57	4.3	18.26
7.0	9.290	4.4	1.99	8.4
9.0	9.054	3.25	1.95	6.21
11.0	8.971	6.065	2.7	11.57
13.0	8.528	9.55	4.3	18.22
16.0	8.445	9.59	4.33	18.29

Table-6. Predicted fatigue lives based on Paris-Erdogan Equationmodified by Liu and Chen [15] for grafted alumina with random copolymer (Mw_{avg} =24000), (Load = 9 kN).

Weight of grafted polymer (mg/g)	True Porosity (%)	N_f (x10 ¹⁵ cycles); (σ =20 MPa)	N_f (x10 ¹² cycles); (σ =30 MPa)	N_f (x10° cycles); (σ =40 MPa)
12.3	3.110	5.49	2.48	10.46
13.5	3.009	7.82	3.53	14.92
16.6	2.508	8.63	3.89	16.45
18.0	2.032	8.20	3.70	15.64

Predicted stresses, which result in fatigue lives of 10³, 10⁴, 10⁵, 10⁶, 10⁷, 10⁸, and 10⁹, respectively for each specimen of alumina are shown in Tables (7) and (8). Figures (2) and (3) exhibit predicted fatigue behaviour (S-

N curves) for studied two types of alumina. Figures (4) and (5) show comparisons between static and fatigue properties for both types of alumina, i.e. ready-made and grafted ones respectively.



Table-7. Predicted stresses, which result in fatigue lives of 10⁰, 10¹, 10², 10³, 10⁴, 10⁵, 10⁶, 10⁷, 10⁸, and 10⁹ cycles respectively, for ready-made alumina for pressing.

Pres.					N_f (cycle	s)				
Load (kN)	×10°	×10¹	×10 ²	×10 ³	×10 ⁴	×10 ⁵	×10 ⁶	×10 ⁷	×10 ⁸	×10 ⁹
0.5	54.000	54.000	54.000	54.000	54.000	54.000	54.000	54.000	52.620	46.610
1.0	59.000	59.000	59.000	59.000	59.000	59.000	59.000	59.000	52.620	46.610
2.0	69.157	69.157	69.157	69.157	69.157	69.157	67.050	59.400	52.620	46.610
4.0	89.402	89.402	89.402	89.402	85.430	75.680	67.040	59.400	52.620	46.610
7.0	126.112	117.970	104.500	92.580	82.000	72.650	64.360	57.000	50.510	44.740
9.0	132.506	116.110	102.850	91.120	80.720	71.500	63.340	56.100	49.710	44.040
11.0	132.498	119.970	106.289	94.150	83.400	73.880	65.450	57.980	51.360	45.500
13.0	106.373	106.373	106.373	96.420	85.420	75.670	67.030	59.380	52.610	46.600
16.0	88.856	88.856	88.856	88.856	85.440	75.690	67.050	59.400	52.620	46.610

Table-8. Predicted stresses, which result in fatigue lives of 10⁰, 10¹, 10², 10³, 10⁴, 10⁵, 10⁶, 10⁷, 10⁸, and 10⁹ cycles respectively, for grafted alumina with random copolymer.

Weight of	$N_f(\text{cycles})$									
grafted polymer(mg/g)	×10°	×10¹	×10 ²	×10 ³	×10 ⁴	×10 ⁵	×10 ⁶	×10 ⁷	×10 ⁸	×10
12.3	151.000	119.340	105.720	93.650	82.960	73.500	65.110	57.680	51.090	45.260
13.5	152.000	121.590	107.710	95.420	84.530	74.880	66.330	58.760	52.060	46.110
16.6	155.000	122.210	108.270	95.910	84.960	75.270	66.680	59.070	52.320	46.350
18.0	160.400	121.890	107.980	95.650	84.740	75.070	66.500	58.910	52.180	46.230

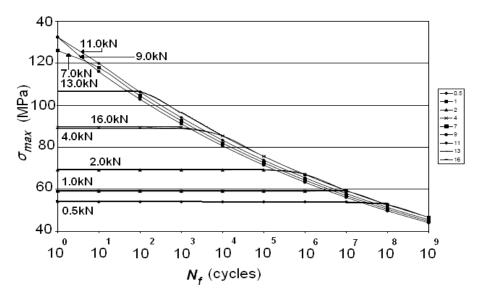


Figure-2. Predicted fatigue behaviour (S - N curves) for studied types of alumina ready-made for pressing, pressed under various loads.



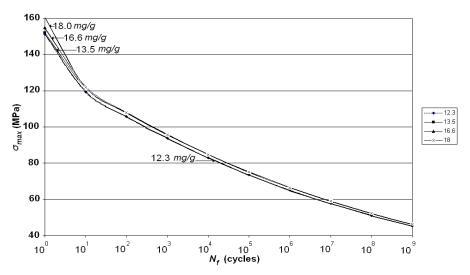


Figure-3. Predicted fatigue behaviour (S - N curves) for studied types of grafted alumina. Pressing load = 9kN.

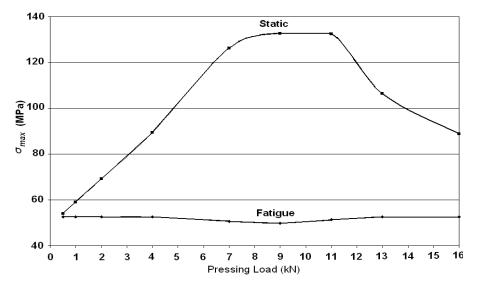


Figure-4. Comparison between static and fatigue properties of ready-made alumina, pressed under various loads.

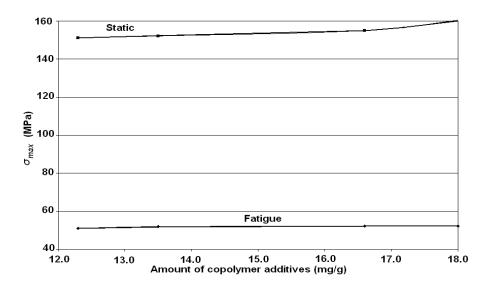


Figure-5. Comparison between static and fatigue properties of grafted alumina. Pressing load = 9kN.

6. DISCUSSIONS

If the surface crack is too small ($a_i << a_c$, $a_i = 0.25$ μm , and $a_i = 25 \mu m$), the applied stresses will make no difference on N_f . The predicted N_f in this case will be very large ($x10^{120}$ to $x10^{160}$ cycles).

It is obvious that that specimen of alumina made especially for pressing, which was pressed under relatively low loads (0.5, 1, 2 kN) and those pressed under high loads (16 kN) were little better in fatigue properties than the others. Static strengths of the former group were low in comparison with the latter group. Gradual enhancement of fatigue properties for specimens, pressed under medium to higher loads (9-16 kN) indicates that higher pressing loads (> 16 kN) could result in better fatigue properties.

In comparison with specimens of alumina specially-made for pressing, which are pressed under (9kN), alumina grafted with random copolymer, which had been pressed under the same load were superior in static properties to their former analogues. Indeed, their fatigue properties are close to those of their analogues from alumina especially made for pressing and superior in static mechanical properties.

With the increase of pressing loads from 4kN to 9kN, fatigue life decreases. This may be ascribed to the deterioration of toughening mechanism in alumina under cyclic loading and this agrees with what was pointed out in the literature [16]. For pressing loads above 9kN, fatigue life increases. This could be ascribed to the higher closeness of grains, which result in the development of grain bridging, which in turn results in toughness enhancement. Higher toughness without significant increase in fracture strength means longer critical crack length. Al-Lami pointed out that pressing load higher than 9kN had resulted in some internal cracks [9]. It could be concluded that those internal cracks result in an increase of impediment to crack propagation due to the crack deflection and meandering (an extrinsic toughening

mechanism) [7]. It may expected that higher loads (>16kN) could result in better static and fatigue resistance as well. An alternative method for enhancement of the aforementioned properties is grafting of alumina with random copolymer due to the lower porosity and more efficient compactness [13, 14].

Porosity detracts both static and fatigue resistances of a material. In case of fatigue, it acts in two ways. Firstly, it weakens fracture strength and toughness. Secondly, pores could be possible regions for crack initiation and propagation. The probability of failure increases with increase in porosity. Indeed, pore size; particularly surface pores have significant influences on fatigue crack propagation, being already initiated flaws. Critical surface energy is enhanced by increasing pressing load and by grafting of alumina better compactness.

Design and applications of alumina ceramics (and ceramics in general) should be based upon K^{TH} , beneath which no fatigue crack could propagate unstably. Unfortunately, the value of this parameter is relatively low. The ratio K^{TH}/K_{Ic} or $\sigma_{max}/\sigma_f = 0.4$ to 0.8.

7. CONCLUSIONS

Several parameters are of concernment in predicting fatigue lives:

- a) A (constant): N_f varies inversely linearly with this factor.
- b) Y (geometric factor), which takes values around unity. In case of ceramic materials, this parameter has little influence upon fatigue life. For simplicity, it can be considered equal to unity.
- c) m, which takes values ranging from 15-50 or even 100, is a highly effective parameter. Its high values indicate high sensitivity of da/dN to stress intensities.
- d) For highly polished high toughness ceramics, effect of m on $(a_i$ and $a_c)$ in increasing N_f , is greater than its decreasing effect on the applied stress σ to decrease N_f ,

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especially, under low stresses and great differences between a_i and a_f . As a_i approaches a_c , or σ approaches σ_f , N_f drops down to low values. A value of $a_i/a_c = 0.58$ make little difference in the term $[a_i^{1-m/2} - a_c^{1-m/2}]$ in Equation (3). For m = 19, this difference is $\leq 1\%$, and a value of $a_i/a_c = 0.66$ makes difference of about 5%.

- e) K^{TH} : is the governor parameter, upon which design of ceramic parts depend. Below K^{TH} , fatigue failure is not expected. At or above K^{TH} , ceramic material could suddenly fails under cyclic loading. The relationship of K^{TH} with K_{Ic} is $K^{TH} \le K_{Ic}$.
- f) Moderate pressing loads, which result in crack initiation, could result in fatigue failure susceptibility in the straight propagation path, whilst higher pressing loads, which result in the meandrous path of crack, together with better compactness and closeness of grains, which in turn result in grain bridging, could result in little enhancement fatigue resistance. Compression and high internal friction will enhance fatigue resistance.

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