PROCEEDINGS

OF

THE REGIONAL CONFERENCE ON

MATERIALS SCIENCE

MAKERERE UNIVERSITY,

KAMPALA, UGANDA

September 15th – 17th, 1999

THERMAL FATIGUE BEHAVIOUR OF A KENYAN KAOLINITE CLAY.

J.N Kimani and B.0 Aduda
Department of Physics, University of Nairobi
P.O Box 30197, Nairobi, Kenya
Tel: 254-2-447552, Fax: 254-2-449616
Email: Physics@Ken. Healthnet.org

ABSTRACT

The thermal fatigue resistance for a Kaolinite clay-based refractory used for thermal insulation in local (Kenyan) firewood cooking stoves has been studied by subjecting them to a water quench to predict their values for a stress-corrosive environment. Plots of speed of utrasonic pressure waves versus thermal shock severity and those of flexural strength versus thermal shock severity show retention of strength up to severi 'ties of about 480'c which is good for refractory materials for thermal insulation. Calculations were based on the assumption that the thermal fatigue of the ceramic material is mainly determined by the duration of time over which a crack reaches a small critical length. Prediction of the life was then made by application of fracture mechanics to the ceramics based on subcritical crack growth. Use was made of the expression In (-in P) = In V- + mln In N+m In (ATN) by Kamiya and Kamigaito, in which P is the survival probability, V' is a function of the stress volume V, N is the number of thermal stresses, ATN'Sthe thermal shock severity, m is the Weibuil modulus and n is a material constant. Experimental results showed that the formula proved to be applicable for the Kaoiinite clay-based refractory samples used. By making use of In N versus In (ATN) PlotS, the thermal fatigue life for different values of thermal shock seventies may be predicted. Further, values for the Weibuil modulus m and those of the material constant n(31 and 20 respectively), obtained from the In (-in P) versus In N plots, agreed quite well with those obtained from the mechanical test plots of In of versus In N where of is the flexural strength at cross-head speed ε.

Keywords: *Thermal fatigue, Kenyan Kaoiinite, Clay refractory, slow crack growth, Fracture strength, Thermal shock.*

INTRODUCTION

Ceramics exhibit a decrease in load bearing ability or strength under conditions of constant or cyclic load as well as repeated thermal shock (1,-4). These phenomena are referred to as static, cyclic and thermal fatigue respectively. Such fatigue behaviour is the result of slow crack growth at stress levels below those required for rapid fracture.

For reliable prediction of the behaviour of structural ceramics, a detailed understanding of their fatigue phenomena is imperative. This study has made use of the fact that consideration of the change in crack growth rate with crack length show that the fatigue life can be approximated by the duration over which the crack length reaches a certain critical value, beyond which the growth becomes relatively rapid. For a rough estimation of the life, only knowledge of the stress distribution in a restricted region of a ceramic body is needed. The study aims to investigate the thermal fatigue behaviour of a kaolinite clay based refractory used locally in Kenya as a thermal insulator for firewood cooking stoves and also for furnace linings. The material is observed to undergo spalling aftêr a number of thermal cycles or on repeated shocking from accidental spillage of fluids during cooking. In the firewood cooking stoves, the material undergoes a thermal cycle every time the fire is made for cooking and then put out after the exercise. The more severe temperature changes are those during the accidental spillage of cold fluids on the hot material during cooking. For the case of furnace linings, the thermal cycle is the usual firing cycle and the subsequent cooling to room temperature after firing.

THEORY

The time during which a sample is under stress is a very important parameter when concidering the strength of ceramics. For example it is observed that if a sample breaks in a time t, when stressed at a constant value, a, the same sample would fracture in a time 100t when stressed to approximately 0.75 σ. This sort of strength degradation with time is typical of oxide ceramics and is quite clearly of great significance to structural applications.
Figure 1 shows an idealized stress intensity factor versus crack velocity diagram.

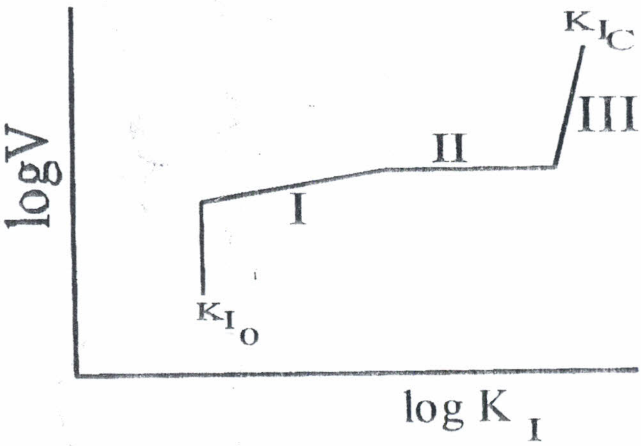

Figure 1. Idealised stress intensity factor versus crack velocity diagram.
From the figure, we see a number of features and different stages. There is sometimes a threshold value of stress intensity factor K, below which no crack growth occurs. It is quite clearly advantageous to establish the existence or not of such a critical value in order to define a possible perfectly safe region of operation. This is however difficult because the velocities involved are always particularly low. In region 1, V is related to K_I as.,

$$V = \alpha_1 K_I^n$$

where c(, and n are constants. A graph of log v versus log K, then yields a straight line from which the value of n , which is the slope of the graph may be calculated. This effect has been identified with a stress induced corrosion mechanism involving attack by water vapour. Thus the rate of crack growth in this region is reaction rate controlled.
In region 11, the crack growth velocity is constant and is given by

$$V = \alpha_2 \qquad (2)$$

Whereo(2 is another constant. The effects of this region has also been identified with stress induced corrosion mechanisms similar to those mentioned for region 1, which involves attack by water vapour. The rate of crack growth for equation (2) depends on the diffusion of the corrosive species to the crack tip.

The third region Ill occurs at higher stress intensity factor values and is normally of rather academic interest in materials which exhibit the corrosive effects mentioned for regions I and 1I as is the case for the materials studied here.

Under constant stress, the time to failure t, is given as;-

$$t = \int_{C_1}^{C_c} \frac{dC}{V}$$

where Ci is the initial crack size and C. is the critical crack size such that.,-

$$K_{I_c} = y\sigma C_c^{1/2}$$

Then

$$C = \frac{K^2}{Y^2\sigma^2}$$

and

$$\frac{dC}{dK} = \frac{2K}{Y^2\sigma^2}$$

or

$$dC = \frac{2K_1}{Y^2\sigma^2} dK_1$$

Substituting equation (5) into equation (3) gives

$$t = \frac{2}{\sigma^2 Y^2} \int_{K_{I_i}}^{K_{I_c}} \frac{K_1}{V} dK_1$$

and using equation (1) and equation (2) we have.,-

$$t = \frac{2}{\sigma^2 Y^2} \left\{ \frac{I}{\alpha_I} \int_{K_{I_i}}^{K_{I_*}} K_I^{(1-n)} dK_I + \frac{I}{\alpha_2} \int_{K_{I_*}}^{K_{I_c}} K_I dK_I \right\} + \text{Region III term}$$

region I term region 11 term

where K_{1*}.. is the K value between regions I and 11.
For ceramics, region I dominates and equation (7) becomes

$$t = \frac{2}{\sigma^2 Y^2 \alpha_1 (n-2)} [K_{I1}^{(2-n)} - K_{12}^{(2-n)}]$$

Furthermore, because n is large, typically greater than 1 0 in ceramics,

$$K_{I1}^{(2-n)} >> K_{12}^{(2-n)}$$

and

$$t = \frac{2K_{I_i}(2-n)}{\sigma^2 Y^2 \alpha (n-2)}$$

Substituting for K, from equation (4) shows that for a particular specimen, which implies a fixed c value, the product $t\sigma^n$ = constant. Thus the ration of the lifetimes $t_{\sigma i}$ and $t_{\sigma j}$ at two subcritical stresses σ_i and σ_j is given by.,-

$$\left\{\frac{\sigma_i}{\sigma_j}\right\}^n = \frac{t_{\sigma j}}{t_{\sigma i}}$$

This relationship permits the calculation of a strength probability time diagram by combining the statistical and the time dependent properties of ceramics.

Strength variations are conveniently generated by breaking a number of samples at a specific strain rate. In this case it is necessary to first consider the connection between constant strain tests and delayed fracture tests.

If the fracture time under constant strain rate conditions is t_ε and had this same sample specimen been stressed instantaneously to σ_f it would have survived a much shorter lifetime t_σ

The ratio of these lifetimes is given by

$$t_\varepsilon = (n+1)t_\sigma$$

For specimen failing at $\sigma_{\varepsilon i}$ and $\sigma_{\varepsilon j}$ in times $t_{\varepsilon i}$ and $t_{\varepsilon j}$ when tested at ε_i and ε_j, respectively, we have

$$\sigma_{\varepsilon i} = E\varepsilon_i t_{\varepsilon_i}$$

$$\sigma_{\varepsilon j} = E\varepsilon_j t_{\varepsilon_j}$$

Eliminating time from equation (11) and equation (12) and using equation (10) gives.'

$$\frac{\sigma_{\varepsilon_i}}{\sigma_{\varepsilon_j}} = \frac{E\varepsilon_i}{E\varepsilon_j}(\ )$$

Using equation (13) we may write

$$\frac{\sigma_f}{\sigma_f} = \frac{\varepsilon^{\frac{1}{n+1}}}{\varepsilon'} \qquad (14)$$

where σ_f and σ' are the flexural strengths at cross head speeds ε and ε respectively.

The low crack growth of ceramics can be described by the following equation

$$\frac{dC}{dt} = A\exp(-Q/RT)K_I^n \qquad (15)$$

where C is the crack length, T the temperature, R the gas constant and Q a material constant.

Substituting equation (4) into equation (1 5) gives

$$\frac{dC}{dt} = A\exp(-Q/RT)Y^n\sigma^n C^{n/2} \qquad (\text{}$$

Equation 16 may be integrated on the assumption that

$$\sigma_T = \Delta Tf(t) \qquad (\text{}$$

$$T = T_o + \alpha\Delta T$$

which yields the following

$$C_i^{(2-n)/2} - C_f^{(2-n)/2} = \frac{(2-n)}{2}(\Delta T)^n G(T_o + \alpha\Delta T)$$

where

$$G(T_o + \alpha\Delta T) = A\exp\{-Q / RT(T_o + \alpha\Delta T)\}\int_o^W Y^n f^n dt$$

where C_i and C_f are the crack lengths before and after one thermal shock.
For ceramics of low 0 or for a small variation in AT, equation(1 9) becomes

$$C_i^{(2-n)/2} - C_f^{(2-n)/2} = \frac{(n-2)}{2}\Delta T^n G(T_r)$$

Where $G(T_r)$ has a similar meaning to $G(T_o, +\alpha\Delta T)$. T_r, is a respective value of the temperature over the duration of the crack growth in a series of experiments. Thus when $C = CC_c$..after N cycles of thermal stressing, the following equation is given from equation (20).

$$C_i^{(2-n)/2} - C_c^{(2-n)/2} = \frac{(n-2)}{2}N(\Delta T_N)^n G(T_r)$$

where AT_N is the temperature difference under which the lifetime of the ceramic is N and the initial crack length of the ceramic before N thermal stresses are given.
In equation (21), C_c is much greater than C_i and n is much greater than 1. On assuming occurrence of crack growth in stage I of the k-v diagram of figure 1, the following approxima can be made.

$$C_i^{(2-n)/2} = \frac{(n-2)}{2}N(\Delta T_N)^n G(T_r)$$

Using equation (21) and equation (22) and for ceramics having common Ci value, we have

$$\frac{N}{N'} = \left\{\frac{\Delta T'_N}{\Delta T_N}\right\}^n$$

For individual ceramics, a statistical treatment is needed. As well known, the short time fracture strength of ceramics the Weibuil statistics,

$$P = \exp\{-V(\sigma_f / \sigma_o)^m\}$$

where P is the survival probability under the fracture stress σ_f m is the Weibull modulus, σ_f normalization constant and V is the stress volume. The fracture stress σ_f of is related t through equation (4) as

$$\sigma_f = K_{I_c} Y^{-1} C_i^{1/2}$$

Substituting equation (25) into equation (24) gives

$$P = \exp\{-V(C_i / C_o)^{-m/2}\}$$

where

$$T = T_o + \alpha\Delta T \tag{18}$$

which yields the following

$$C_i^{(2-n)/2} - C_f^{(2-n)/2} = \frac{(2-n)}{2}(\Delta T)^n G(T_o + \alpha\Delta T) \tag{19}$$

where

$$G(T_o + \alpha\Delta T) = A\exp\{-Q / RT(T_o + \alpha\Delta T)\}\int_o^W Y^n f^n dt$$

where C_i and C_f are the crack lengths before and after one thermal shock.
For ceramics of low 0 or for a small variation in AT, equation(1 9) becomes

$$C_i^{(2-n)/2} - C_f^{(2-n)/2} = \frac{(n-2)}{2}\Delta T^n G(T_r) \tag{20}$$

Where $G(T_r)$ has a similar meaning to $G(T_o, +\alpha\Delta T)$. T_r, is a respective value of the temperature over the duration of the crack growth in a series of experiments. Thus when C = CC_c..after N cycles of thermal stressing, the following equation is given from equation (20).

$$C_i^{(2-n)/2} - C_c^{(2-n)/2} = \frac{(n-2)}{2}N(\Delta T_N)^n G(T_r) \tag{21}$$

where AT_N is the temperature difference under which the lifetime of the ceramic is N and C is the initial crack length of the ceramic before N thermal stresses are given.
In equation (21), C_c is much greater than C_i and n is much greater than 1. On assuming the occurrence of crack growth in stage I of the k-v diagram of figure 1, the following approximation can be made.

$$C_i^{(2-n)/2} = \frac{(n-2)}{2}N(\Delta T_N)^n G(T_r) \tag{22}$$

Using equation (21) and equation (22) and for ceramics having common Ci value, we have

$$\frac{N}{N'} = \left\{\frac{\Delta T_N'}{\Delta T_N}\right\}^n \tag{23}$$

For individual ceramics, a statistical treatment is needed. As well known, the short time fracture strength of ceramics the Weibuil statistics,

$$P = \exp\{-V(\sigma_f / \sigma_o)^m\} \tag{24}$$

where P is the survival probability under the fracture stress σ_f m is the Weibull modulus, σ_f is a normalization constant and V is the stress volume. The fracture stress σ_f of is related to C_i through equation (4) as

$$\sigma_f = K_{I_c} Y^{-1} C_i^{1/2} \tag{25}$$

Substituting equation (25) into equation (24) gives

$$P = \exp\{-V(C_i / C_o)^{-m/2}\} \tag{26}$$

where

$$C_o = (K_{I_c} Y^{-1} \sigma_o^{-1})^2$$

Substituting equation (22) into equation (26) give

$$\ln(-\ln P) = lmV' + \frac{m}{n}\ln N + m/n(\Delta T_N)$$

where

$$V' = V.C_o^{m/2}\left\{\frac{n-2}{2}\right\}^M /(N-2)\frac{m}{a}/(n-2)\{G(T_r)\}^{m/n}$$

Equation (28) relates statistically the results of the relatively short term tests of a group of ceramics to those of a long term practice. For ceramics having a common P value, we obtain from equation (28)

$$\frac{N}{N!} = \left\{\frac{\Delta T_N'}{\Delta T_N}\right\}^n$$

Equation (29) is identical to equation (23) which was obtained for ceramics having a common C value. This identity is due to the fact that a common value of P correspond to a common value of initial crack legth in a group consisting of a large number of ceramics.
According to equation (28), a graph of In(-InP) plotted against InN yields a straight line for a given value of AT_N. The lines run parallel to one another because their slopes have a common value of m/n. Such a plot is referred to as thermal shock severity probability time (T-SPT) diagram.
Alternatively, value of the constant n, may be determined by plotting InN against $\ln(AT_N)$ for a fixed value of survival probability P. From equation (28), this yields a straight line of slope equal to the constant n.

EXPERIMENTAL PROCEDURE

The kaoiinite clay samples moistened with five weight percent water were formed into cylindrical rod samples using a mould designed to produce samples of length 15cm and 1 cm in diameter. After oven drying at 6O°C for24 hours followed by 1OO°C overnight, the diameters of the green samples were measured using digital callipers. The samples were then fired in sets from room temperature in the following sequence, 2°C per minute to 220°C, 5°C per minute to 56O°C and 7°C per minute to the final temperature. The final temperatures were between 6OO°C and 1300°C. The soaking time at each final temperature was 3 hours and after soaking, the sets of samples were allowed to cool overnight in the furnace to room temperature.

For the thermal fatigue test, the samples were transferred from the hot zone of an electric resistance furnace into water, and then returned to the hot zone. This process was repeated until the number of cycles reached a given specific value. The specimens were held in the hot zone for 30 minute, and in water for five minutes. The time for transfer from the hot zone to water was about 2 seconds and that from water to the hot zone about 16 seconds. The temperature of the hot zone was controlled to within ± 0.5° C. The temperature difference AT, in the formulae was taken as the difference between the temperature of the hot zone and that of the water. The

failure of a specimen was determined by the occurrence of visible crack. The detection of the crack in a specimen was made at a given number of thermal cycles, N.
The survival probability, P, was taken as the fraction given by dividing the number of specimens surviving after a given number of thermal cycles, by the total number of specimens, which was 20 in our case.
To estimate n, and m, by a mechanical method, the flexural strength was measured by the three point bending test, in which the span was 10 cm. the cross-head speed was varied from 0.5 to 50 mm per minute. For a given value of cross-head speed, 20 specimens were used.
Using an ultrasonic tester, the velocity, v, of propagation of sound waves through the thermally shocked specimen was determined.

RESULTS AND DISCUSSION

Table 1 shows the chemical composition and the physical properties of the kaolinite clay used in the study. Figure 2. shows values of the fracture strength of the kaolinite clay specimens after water quenching at different thermal shock seventies, AT. Up to values of about 480°C, the flexural strengths of the samples is about 60 MPa. Between thermal seventies of 480°C and 580°C, there is a sudden drop in the flexural strengths of the specimens, down to values of about 18MPa.

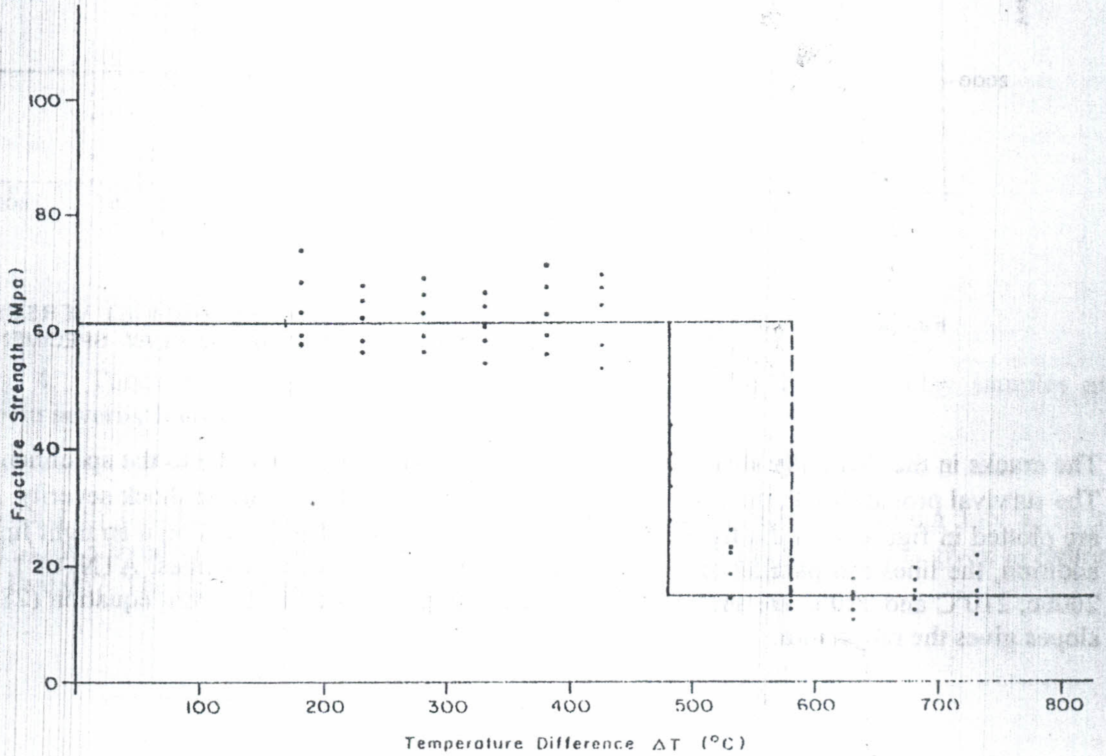

Figure 2 : FRACTURE STRENGTH OF THE KAOLINITE CLAY SPECIMENS AFTER WATER QUENCHING AT DIFFERENT THERMAL SHOCK SEVERITIES.

The variation of the speed of the ultrasonic waves of the thermally shocked specimens reveals a similar trend as is apparent from figure 3. Up to a thermal shock severity of about 480°C, the velocities have an average value of about 2170 ms^{-1} Between thermal shock seventies of 480°C and 630°C the velocities drop to values of about 2000ms^{-1}.

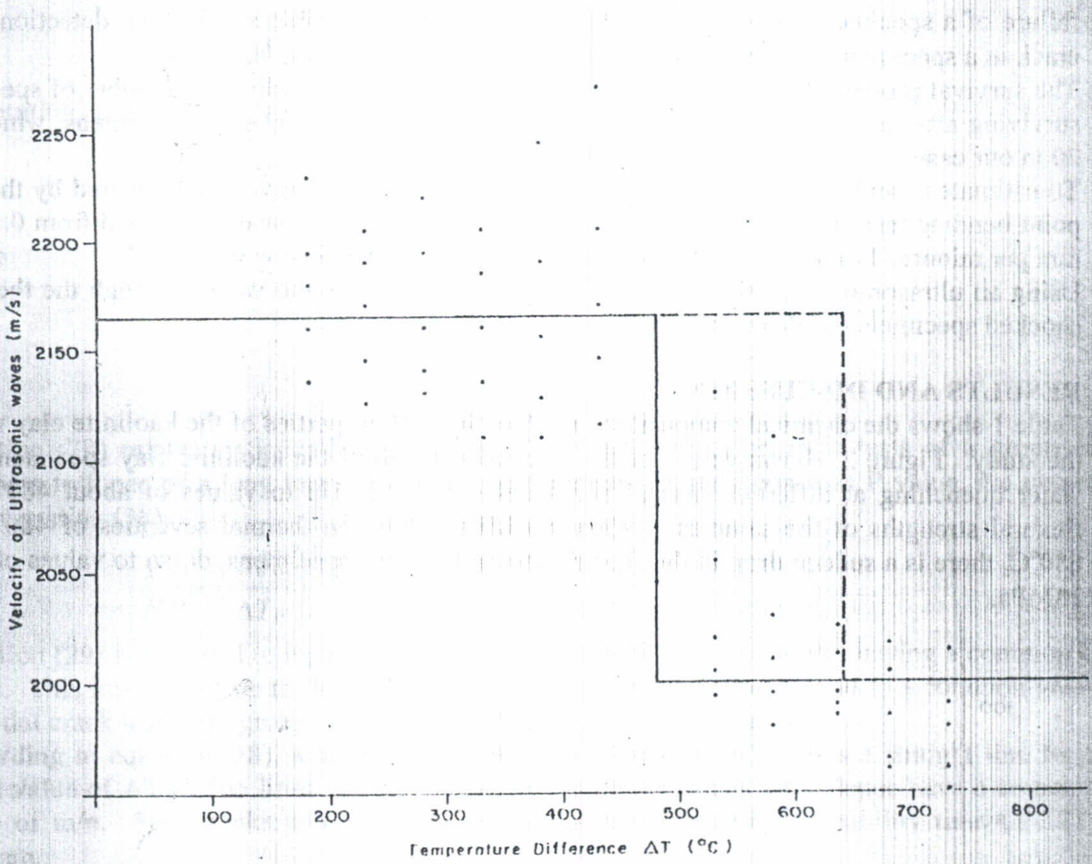

Figure 3 SPEED OF ULTRASONIC PRESSURE WAVES (40+12) VERSUS THERMAL SHOCK SEVERITY FOR KAOLINITE CLAY SPECIMENS.

The cracks in the thermally shocked specimens were noted to grow parallel to the specimen axis. The survival probability P, the fatigue life thermal cycles **N** and the thermal shock severity, ΔT_N, are plotted in figure 4. In the figure, In (-In) for a fixed value of ΔT_N fails on a straight line. In addition, the lines run parallel to one another. Thermal shock severity values, ATN, of 1 90°C, 2000c, 210°C and 220°C are shown. They all yield slope vales of 1.55. From equation (28), the slopes gives the ration m/n.

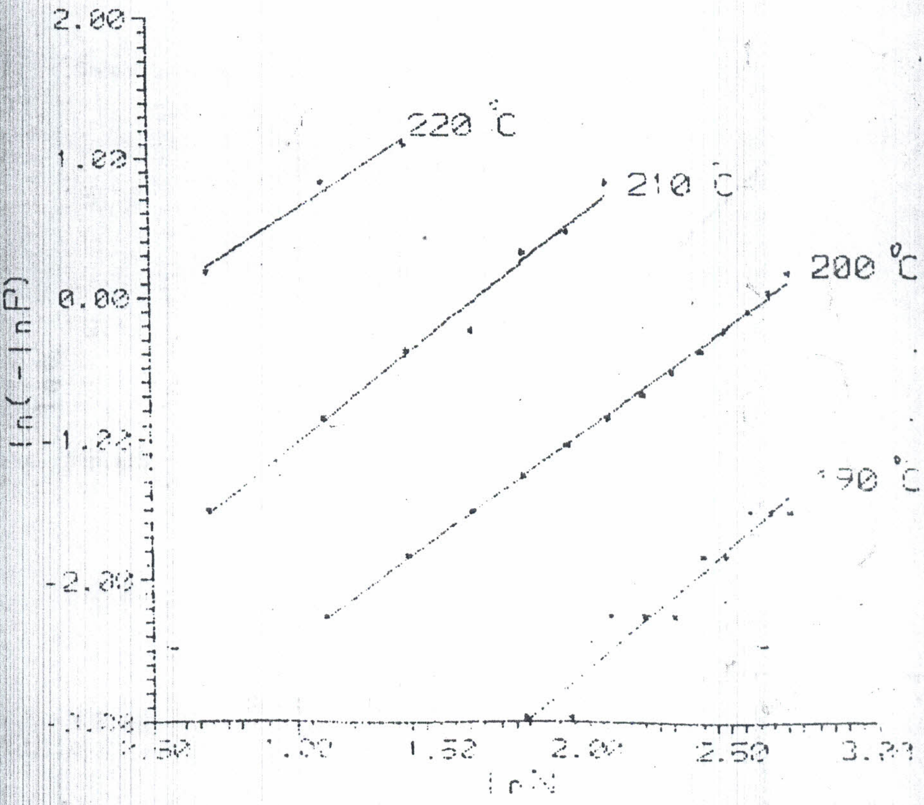

Figure 4: Thermal shark probability time (T-SPT) diagram for Kaolinite clay samples at different severity levels.

According to equation (28) also, the constant n can be determined by plotting InN against $\ln(\Delta T_N)$ for a fixed value of survival probability. The In **N** versus In (ΔT_N) Plot is given in figure 5 for P=0.057. The slope gives a value of n=20.

Table 1. Chemical composition and physical properties of the kaolinite clay used in the study.

Compounds	Quantity(%tage)
SiO_2	44.81
Al_2O_3	36.79
Fe_2O_3	1.58
MgO	0.21
CaO	0.71
Na_20	0.42
K2 0	0.39
TiO_2	0.98
Loss on iginition	13.91
Major mineral constituent	$Al_2(Si_2O_5)(OH)_4$
Description of raw material	White greyish powder
Modulus of elasticity	10.74 GNm^{-2}
Poissons ratio	0.25
Specific heat capacity	931 $Jkg^{-1}K^{-1}$
Coefficient of expansion	6 x $10^{-6}K^{-1}$
Specific gravity	2580

TABLE 2. Values of m and n obtained from the ln N plots and the In N-In ΔT plots respectively and those obtained from the mechanical method tests. In

	From lnN-lnΔT plots	From ln(-lnp)-lnN plots	From lnσ_f-lnε plots (flexu based)
m	31		30.5
n	20	20	20.5

CONCLUSION

The thermal fatigue resistance for a kaolinite clay-based refractory used for thermal insulation i local (Kenyan) firewood cooking stoves was studied by subjecting them to a water quench.
Plots of speed of ultrasonic waves versus thermal shock severity and those of flexural strengt versus thermal shock severity show retention of strength p to seventies of about 480°C.
The thermal fatigue life is predicted by making use of the In N versus In (ATN) PlotS, fo different values of thermal shock seventies. A thermal shock severity ΔT_N 18O°C result with thermal fatigue life N=75 cycles.
The thermal shock probability time (T-SPT) graphs of In N versus In (-lnP) and the mechanic method test plots of In **of** versus In t gave values of the crack growth parameter n= 20 and of th Weibull modulus m = 31.

REFERENCES

1. Awaji H., Takahashi T. , Yamamoto N. And Nishikawa T. "Analyss o temperaturelstress distributions in thermal shocked ceramic disks in relation t temperature dependent properties". Journal of the ceramic society of Japan PP 358-36 (1998).

2. Wang P.C, Her Y.C and Yang J.M. "Fatigue behaviour and damage modeling of SCS-6/titanium/titanium aluminide hybrid laminated composite". Materials science and Engineering PP 100-1 08 (1997).
3. Ashizuka M. Nakamura S. And Kubota Y. "Fatige behaviour and fracture toughness of mullite ceramics containing 7ro2 at 1200°C". Journal of the Ceramic society of Japan. PP 460-464 (1998).
4. Kamiya N. And Kamigaito O. "Prediction of thermal fatigue. Lif of Ceramics." Journal of materials Science PP 212-214 (1978).
5. Grimshaw W. "The chemistry and physics of clay and allied ceramic materials." 4th Ed. Errest Been Ltd, London (1971).
6. Davidge R.W. Mechanical Behaviour of Ceramics. Cambridge University press, Cambridge (1986).
7. Hasseiman D.P.H, Badaliance R., Mckinney and Kim C.H. Failure prediction of the thermal fatigue resistance of a glass. Journal of materials science Vol 11 PP 458-464 (1976).