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Grain Boundary Oxidation and Its Effects on High Temperature Fatigue Life*

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INTRODUCTION

Fatigue lives at elevated temperatures are often shortened by creep and/or oxidation. Creep causes grain boundary void nucleation and grain boundary cavitation. Grain boundary voids and cavities will accelerate fatigue crack nucleation and propagation, and thereby shorten fatigue life.

Gibb's free energies of metal oxide formation are negative. No metal or alloy is stable when exposed to an oxidizing environment. Grain boundary is a path of rapid diffusion. Therefore, grain boundary oxidation rate is higher and grain boundary oxide penetration is deeper. Oxide is brittle and fractures easily when a tensile stress is applied. Grain boundary oxide crack may serve as a nucleus of a fatigue crack and the crack nucleus will grow by the subsequent cyclic fatigue load. Therefore, grain boundary may shorten fatigue crack nucleation life. Oxidation also accelerates fatigue crack propagation. Hence, grain boundary oxidation will shorten fatigue lives at elevated temperatures.

Both oxidation and creep have been shown as possible mechanisms for high temperature fatigue damage. Grain boundary void formation and cavitation are the result of surface diffusion and/or grain boundary vacancy diffusion, while grain boundary oxidation is primarily caused by the diffusion of oxygen. The kinetics of the diffusions of vacancies and oxygen atoms is shown schematically in Figure (1). One mechanism dominates in the high temperature region and the other dominates in the low temperature region. Therefore, the question is not which one of these two mechanisms causes high temperature fatigue damage. The problem is to define the different regions dominated by these two different mechanisms.

The functional relationships between the damage rate of fatigue crack nucleation and propagation and the kinetic process of oxygen diffusion depend on the detailed physical processes. In this study, the kinetics of grain boundary oxidation penetration was invetigated. The statistical distribution of grain boundary penetration depth was analyzed. Its effect on high temperature fatigue life will be disscussed. A model of intermittent micro-ruptures of grain boundary oxide was proposed for high temperature fatigue crack growth. The details of these studies are reported in references 1 and 2.

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GRAIN BOUNDARY OXIDATION KINETICS AND ITS EFFECTS ON FATIGUE CRACK NUCLEATION

Cylindrical coupons of a nickel-base superalloy (TAZ-8A) were subjected to oxidation in air under the stress-free condition. The oxidized disk coupons were sectioned, each sectioned surface was examined under an optical microscope, and the maximum grain boundary oxide penetration depth, a_{mi} of the ith section was measured. Then a thin layer of coupon approximately 80μm was removed. Then the new surface was polished, and another a_{mi} of the new surface was measured. This process was repeated 12 times for each test coupon to collect enough data for the statistical analysis. Couling and Smoluchowski (ref. 3) and Turnbal and Hoffman (ref. 4) have found that grain boundary diffusion penetration is a function of the angle of (100) tilt boundaries. Therefore, it is expected that the grain boundary oxide penetration depth, a_m, is not uniform. It varies from one grain boundary to another. Figure 2 shows the Weibull plot of 480 data points for oxidation at 800°C for 500 hours. The data fits the Weibull distribution function very well.

$$[1 - P(a_{mi})] = \exp\left[-\left(\frac{a_{mi} - a_u}{a_o}\right)^b\right] = \exp\left[-\frac{(a_{mi} - a_u)^b}{\eta_a}\right] \qquad (1)$$

$P(a_{mi})$ is the probability of finding an oxide depth less than a_{mi} on a sectioned surface. The probability of finding a depth equal to or deeper than a_{mi} is $[1 - P(a_{mi})]$. For the data in Figure 2, b = 2.0, a_u = 40μm, and a_o = 31 μm.

Grain boundary diffusion is several orders of magnitude faster than bulk diffusion. If the flux due to bulk diffusion is neglected, grain boundary diffusion can be considered a channeled one-dimensional flow. With a constant oxygen concentration C_o at the "entrance" of the grain boundary, the oxygen concentration in the boundary is

$$C(x, t) = C_o\left[1 - \mathrm{erf}\left(\frac{x}{2\sqrt{D_{gb}t}}\right)\right] \qquad (2)$$

where erf is the error function, D_{gb} is the grain boundary diffusion coefficient. "Bulk" oxide will be formed when the oxygen concentration reaches a certain critical value, C_c. According to Equation (2),

$$C_c = C_o\left[1 - \mathrm{erf}\left(\frac{x_c}{2\sqrt{D_{gb}t}}\right)\right] \qquad (3)$$

x_c is the depth of the oxide penetration, where $C = C_c$. According to Equation (3), the quantity $(x_c/D_{gb}t)$ must be a constant, and x_c must be proportional to $\sqrt{D_{gb}t}$. Therefore, the grain boundary oxide penetration must be proportional to $\sqrt{t}$.

However, the model does not take the bulk diffusion and the chemical process into consideration. Perhaps it is reasonable to assume that a_{mi} is proportional to $(D_{gb}t)^n$. Therefore a_{mi} must have the form

$$\frac{a_{mi}}{B} = \beta\left(\frac{D_{gb}t}{B}\right)^n \qquad (4a)$$

or

$$a_{mi} = \alpha_i\, t^n \exp\left(-\frac{n\Delta H}{RT}\right) = \alpha_i\, t^n \exp\left(-\frac{Q}{RT}\right) \qquad (4b)$$

B is the magnitude of the diffusion jumping vector or interatomic spacing.

The grain boundary oxide penetration depths were measured at the oxidation temperatures of 600, 800, and 1000°C at the exposure time from 100 to 1000 hours. The regression analysis of the data gives the following empirical relation

$$a_{mi}(cm) = 1.34 \times 10^{-3}\ t^{0.25} \exp(-4.26/RT) \tag{5}$$

where t is in seconds, the activation energy in kcal/mol, and T in ^{o}K. The coefficient of auto-correlation is 0.96.

Assume the relation between a_{mi}, t, and T is deterministic. The deviation of each measured penetration depth from the empirical relation can be lumped into the term α_i in Equation (4b).

$$\alpha_i = a_{mi}\ t^{-n} \exp(\frac{Q}{RT}) \tag{6}$$

At any temperature T and exposure time t, with the measure a_{mi} known, the value of α_i can be calculated from Equation (6). The data of 144 values of α_i fit well the Weibull distribution function as shown in Figure (3).

$$[1 - P_i(\alpha)] = \exp\ [-\ (\frac{\alpha - \alpha_u}{\alpha_o})^b] = \exp\ [-\ \frac{(\alpha - \alpha_u)^b}{\eta_\alpha}\ ] \tag{7}$$

$P_i(\alpha)$ is the probability of finding an α-value less than α. The value of b, α_u, α_o are 1.85, 0.53×10^{-3} and 0.51×10^{-3} respectively.

The maximum α-value along the periphery of the i'th sectioned surface can be considered as the α_i-value of an exposed area of $\pi\delta d$ of a test coupon. δ is the coupon diameter and d is the grain size. Another sectioned surface at a distance one grain diameter away contains an entirely different set of grain boundaries and it is another independent sample.

If $P_s(\alpha)$ is the probability of finding an α-value less than α on an exposed area s. $P_s(\alpha)$ is related to $P_i(\alpha)$

$$[1 - P_s(\alpha)] = [1 - P_i(\alpha)]^{s/\pi\delta d} \tag{8}$$

The value of $P_s(\alpha)$ might be taken as the value of $P_s(a)$, the probability of finding a penetration depth less than a on an exposed surface area s. Therefore the data measured from the test coupons can be used to extrapolate to a much larger surface area of an engineering component.

Oxide is brittle and fractures easily under a tensile stress. Once an oxide crack is formed, the crack will continue to grow under a cyclic fatigue load. The oxide crack can be considered as a precrack. The remaining fatigue life, N_{fa_o} of a precracked specimen or a precracked engineering component is a function of the precrack size, a_o

$$N_{fa_o} = f(a_o) \tag{9}$$

The probability of having fatigue life N_{fa_o} is also the probability of having an oxide crack size a_o. Therefore, the statistical scatter of the fatigue lives at elevated temperatures may reflect the scatter of the oxide penetration depth.

A fatigue crack is often nucleated by cyclic plastic deformation. This nucleation mechanism is cylcle dependent. The damage mechanism by oxide crack formation is time dependent. Therefore, for a very low cyclic frequency, the oxide crack formation may preceed the fatigue crack nucleation by the cyclic plastic formation process. Thus the fatigue nucleation life (in terms of number of load cycles) might be shortened. Perhaps, the shortened fatigue life at elevated temperatures and the wide scatter of the fatigue life of engineering components are caused by grain boundary oxidation.

THE INTERMITTENT MICRO-RUPTURE MODEL FOR HIGH TEMPERATURE FATIGUE CRACK GROWTH

Figure (4) shows the frequency effect on fatigue crack growth rate for a number of high temperature alloys. For each data set, both ΔK and test temperature were maintained constant.

In the low frequency region, the fatigue crack growth rate, da/dN, of Inconel 718, Inconel X-750, Astroloy at 700 and 760°C, and Cr-Mo steels, are inversely proportional to frequency, ν. The time rates of the fatigue crack growth, $da/dt = (da/dN)(1/\nu)$ are constant. In this region, the fatigue crack growth is intergranular.

For constant-K tests at elevated temperatures, two crack growth features are common: (i) the time rate of crack growth is constant, (i.e. da/dt = constant) and (ii) crack growth is intergranular. Crack growth at constant-K is often referred to as creep crack growth. Fatigue crack growth in the low frequency region has these two same features. Therefore, fatigue crack growth in the low frequency region is often referred to as creep crack growth.

One question can be raised. Do the inverse relation between da/dN and ν and the intergranular crack growth preclude grain boundary oxidation as the underlying cause of the accelerated fatigue crack growth at elevated temperatures? In this section, a fatigue crack growth model, based on the fracture of grain boundary oxide, will be constructed. The model agrees with the inverse relation between da/dN and ν. The fracture path following the grain boundary oxide, is intergranular.

In Figure (4), the cyclic loading patterns are also shown. For Inconel 718 and Astroloy at 760°C, a hold time, Δt_H at K_{max} was applied. For Inconel X-750, Cr-Mo steels, and Astroloy at 700°C, a triangular loading pattern was used. The crack growth with a hold time will be analyzed first.

The oxygen arriving at a crack tip will have to diffuse into the region ahead of the crack tip in order to form oxide along the grain boundary. When the crack tip grain boundary oxide reaches a critical size, δa, the oxide will rupture and the crack will grow by the amount, δa. The critical size, δa, depends on the K-level during the hold time. Once the crack tip advances to its new position, this process of grain boundary diffusion, grain boundary oxidation, and the micro-rupture of the grain boundary oxide will be repeated again. This process of micro-rupture of crack tip grain boundary oxide can reoccur intermittently many times during a fatigue cycle.

During Δt_H at K_{max}, many micro-ruptures will take place. After each micro-rupture, the penetration of grain boundary oxide will have to start all over again

from "time" zero. Therefore, the time interval, δt necessary to reach the critical size δa is

$$\delta t = (B/D_{gb})(\delta a/\beta B)^{1/n} \tag{10}$$

The number of micro-ruptures during Δt_H is

$$m = \Delta t_H/\delta t = (\Delta t_H D_{gb}/B)(\beta B/\delta a)^{1/n} \tag{11}$$

m is linearly proportional to Δt_H and is inversely proportional to ν.

Fatigue crack growth per cycle is the sum of the micro-ruptures during Δt_H.

$$\frac{da}{dN} = m\delta a \tag{12}$$

From Equations (10, 11, and 12), we obtain

$$\frac{da}{dN} = \beta'\Delta t_H D_{gb} (B/\delta a)^{(1-n)/n} = \beta'(D_{gb}/\nu)(B/\delta a)^{(1-n)/n} \tag{13}$$

da/dN is inversely proportional to ν. For n=0.25, da/dN is inversely proportional to δa^3. Fatigue crack growth rate increases rapidly as δa becomes small. δa is smaller if the oxide is more brittle, if the flow stress of the material is higher, and if the crack tip stresses are in the state of plane strain.

Figure (5) shows the triangular loading patterns of two different frequencies. The time interval Δt_i at K_i is inversely proportional to ν. We treat Δt_i as the hold time at K_i. If the crack tip fields at K_i are the same at both of these two frequencies, the number of micro-ruptures during Δt_i is linearly proportional to Δt_i and is inversely proportional to ν. This must be true at every K_i-level. Fatigue crack growth is the sum of all of the micro-ruptures at all the K_i-levels during one fatigue cycle. Therefore da/dN is inversely proportional to ν. Fatigue crack growth follows the path of grain boundary oxide, therefore, it is intergranular.

We have shown grain boundary oxidation as a <u>possible</u> mechanism of high temperature damage that shortens fatigue life. Our work is only one of the many steps toward the construction of a quantitative model based on the physical damage processes caused by oxidation.

Only after the quantitative models of the physical processes of fatigue damage due to creep and oxidation are completed, we will be able to predict high temperature fatigue life accurately and with confidence. The quantitative relations between the diffusion rates of vacancies and oxygen, the rate of oxide rupture, the rate of nucleation and growth of voids and cavities, and the rate of fatigue damage have not yet been established.

Without a clear understanding of the underlying physical processes for the observed fatigue behaviors, it is difficult and unsafe to extrapolate a limited amount of experimental data for fatigue life predictions. For example, to extrapolate the crack growth rate in Figure (4), from the low frequency region into the high frequency region or vise versa will underestimate the growth rate. Therefore it is unsafe.

REFERENCES

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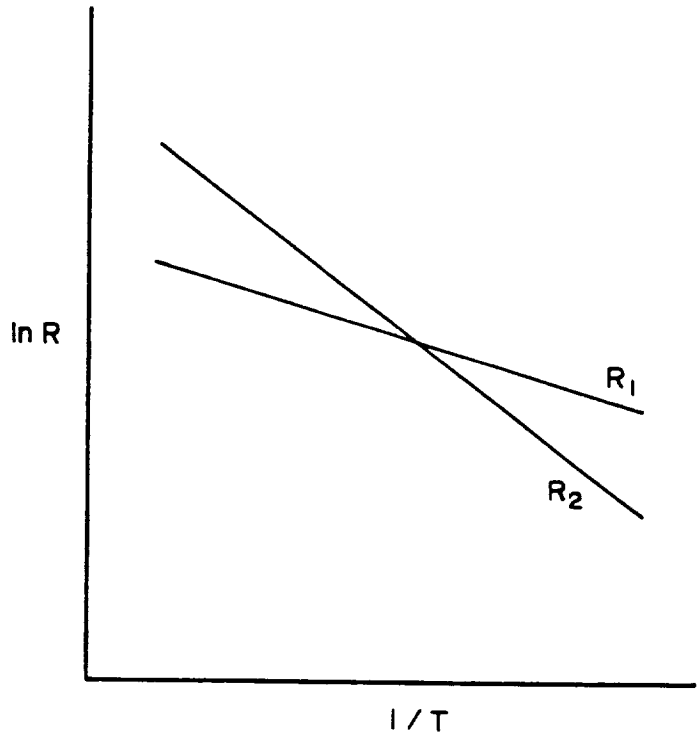

Figure 1. Schematic illustration of diffusion kinetics of vacancies and oxygen atoms

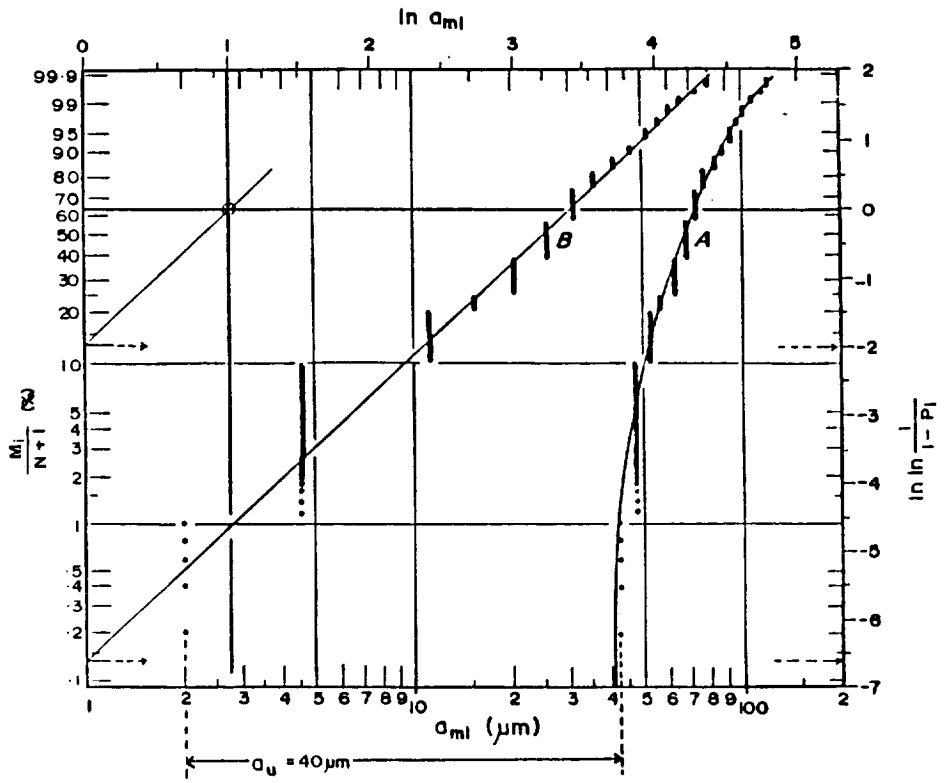

Figure 2. Weibull plot of a_{mi} (i = 480)

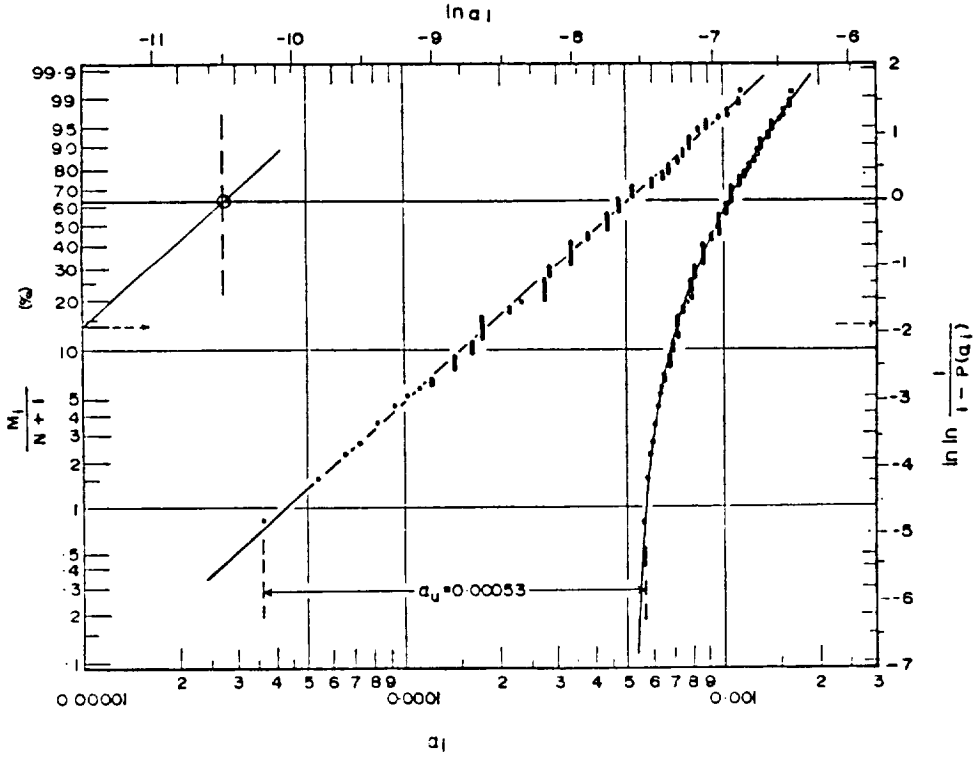

Figure 3. Weibull plot of α_i (i = 144)

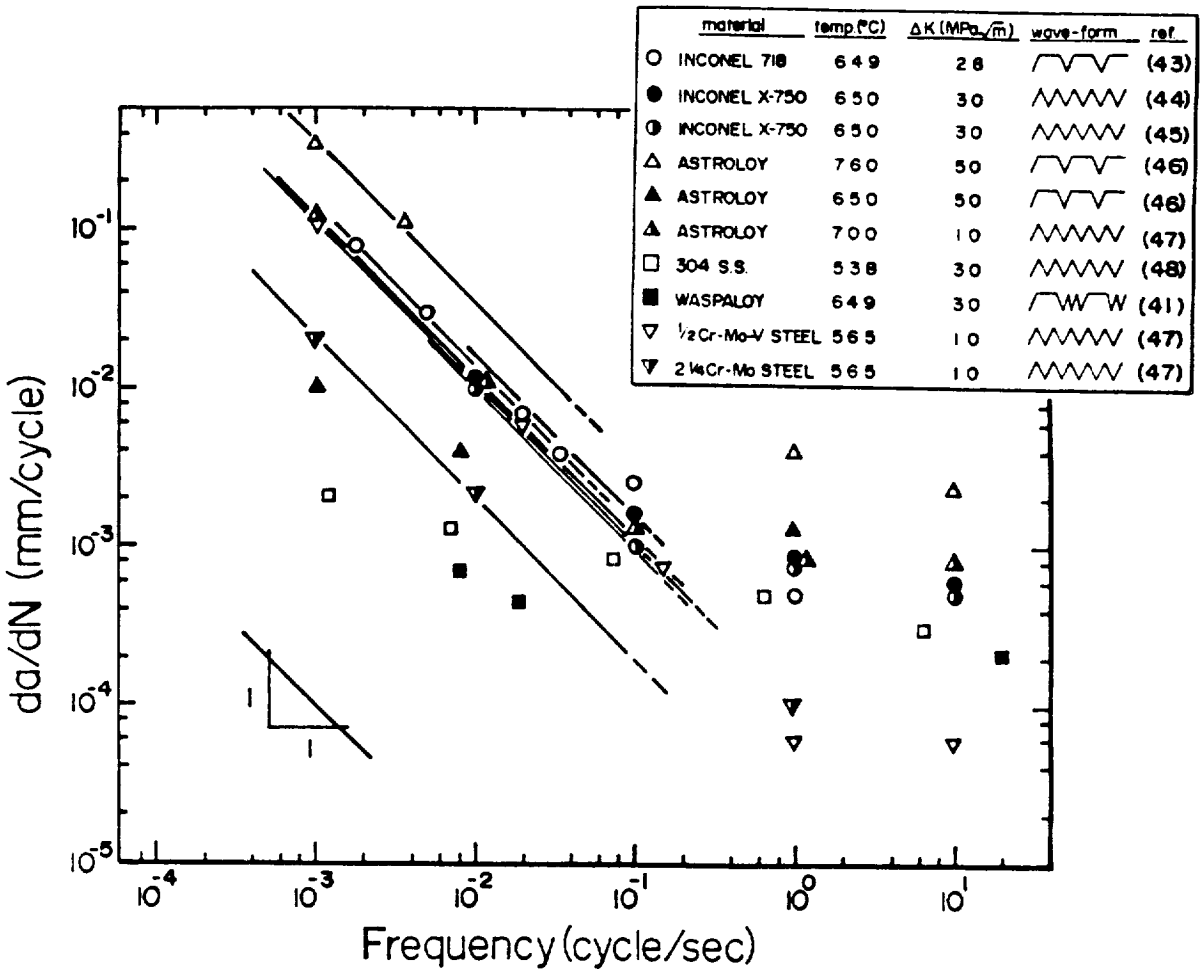

Figure 4. Frequency effect on fatigue crack growth

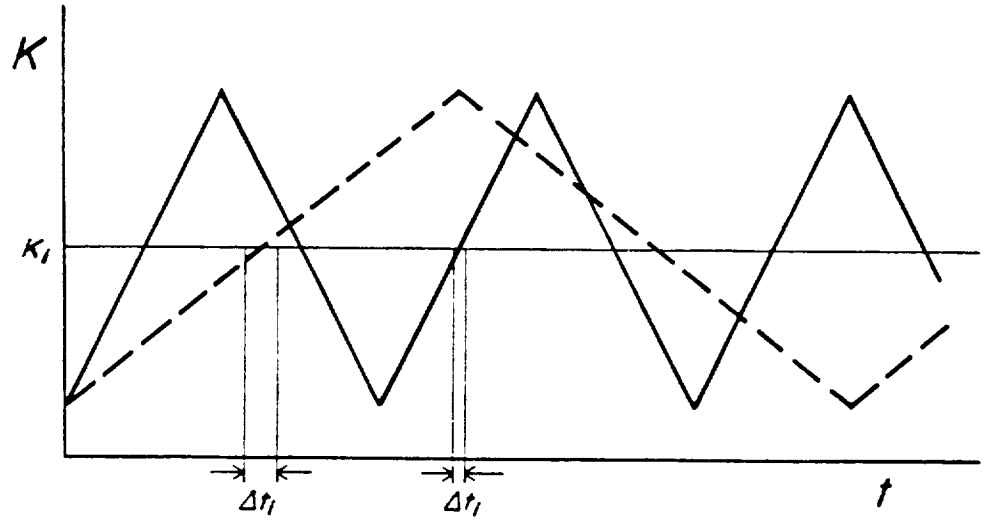

Figure 5. Triangular loading patterns of two different frequencies