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Fatigue of brittle materials—A critical appraisal

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Abstract. Recent demands for high performance ceramics and glass for various applications from bioceramics to cutting tools under fluctuating stress conditions has focussed attention of the scientific community towards fatigue behaviour of brittle solids. Attention to fatigue phenomena in alumina ceramics phenomenological to metals, having an endurance dependent on applied stress with a limit at around 50% of the single cycle fracture stress, was first drawn by the author in late sixties. Slip assisted fatigue process was not considered to be dominant in ceramic materials due to the absence of appreciable crack tip plasticity. With the background of this general survey of fatigue behaviour some fatigue studies based on mode of testing, theoretical and experimental analyses and fractographic evidence have been presented. Studies have shown that there is a dormant period between each successive crack advancement during which the residual stress and a plastic component is built up in a cumulative manner leading to eventual failure. During fatigue σ_{p_j} (plastic) and σ_{r_j} (residual stress) components are predominant for ductile metals and brittle glass/ceramics respectively.

It is also apparent that dislocation assisted plastic component as a contributing factor in the failure of brittle materials under fatigue cannot be ruled out.

Keywords. Fatigue; ceramics; glasses; space; power generation; machine tools; automobile; orthopaedic; earthquake; impact; endurance; elastic; plastic; residual stress; fractography.

1. Introduction

Since the dawn of industrial revolution man has been confronted with the subject on materials that will endure under repeated stressing conditions. Serious failures had occurred at the cost of human lives in aircrafts, railways, ships, bridges, etc where the material of construction had not been able to sustain the alternating stresses much below its critical failure stress. Failures of this kind has been coined as fatigue and arisen out of failures of railway axle in Germany in 1840. Since then volume of systematic studies were conducted to delineate the causes of such failures simulating actual operating conditions in the laboratory and characterization affecting life predictions and replacement of components before occurrence of a catastrophe.

Metals failure under fatigue is now well understood and attributed to dislocation assisted phenomena, but for brittle solids like glass and ceramics where dislocation assisted slip is not common the subject has remained in the realm of scientific curiosity since long. It is only during the last 25 years that scientists have taken serious look at this fatigue phenomena in such solids. The subject has drawn more attention in the last decade because of the immense potential and applications of glasses and ceramics as light weight, high strength, corrosion resistance and high temperature structural materials.

This paper is an attempt to critically appraise the fatigue behaviour of ceramics and glasses encompassing the state of the art knowledge applicable to its wider acceptance as an engineering material of next century.

2. Fatigue related applications

Applications of glasses and ceramics as an engineering material are being accepted much widely day by day and inspite of their being brittle without slip related plasticity, studies have been targetted towards understanding their behaviour under varying alternating stress conditions. The choice for such materials has been due to their inherent superior properties like corrosion resistance, ability to withstand high temperatures, relatively easy processing and cost effectiveness. The material being highly resistant to corrosion, corrosion fatigue phenomena as prevalent in metals and alloys does not bear much importance and is relatively neglected. In the last decade concentrated effort has been directed towards understanding the mechanism of fatigue failure in brittle solids affording life predictions of the components. Following applications will act as illustrations where the materials embrace alternating and fluctuating stresses of varying magnitude and frequency including temperatures.

2.1 *Space engineering*

Materials that are identified for use in space vehicles (satellites and rockets) are designed to withstand sudden shock, vibration and temperature fluctuations. The start of a rocket engine is a source of a severe shock and as it accelerates to supersonic speed, stresses of the order of 20 g is imparted on the materials of construction. Vibration during take off is equally severe. Many launch vehicle failures were attributed to this reason. The control systems are either monopropellant catalyst or bipropellant reactors that are switched on and off for a large number of times to steer the vehicle or satellites. Protective tiles in space shuttle are typical examples, and all these in their long service life have to endure millions of stress cycles.

Satellite components have to withstand severe thermal fluctuations of 150°C (day) and −70°C (night) temperatures and the rapidity of fluctuations are very often extreme. This imposes heavy repetitive stresses (thermal) on materials like camera lenses which were often seen to crack and destroy a mission. Stresses thus developed can almost be equal to the breaking strength of the materials.

Any failure of brittle materials under such repeated stressing conditions can be very expensive.

2.2 *Power generation*

Ceramics are increasingly used in power generation devices, thermal or nuclear, for their high temperature and corrosion resistance properties. Nickel and stainless steel based alloys are being replaced by alumina and zirconia based materials. There too it has to sustain repeated thermal cycle induced stresses with life beyond 10^6 cycles.

Nuclear fuel rods today are predominantly oxides or carbides. Mixed oxide fuels in fast breeder reactors have ushered in cost effective and cheaper generation of power. Repeated thermal stresses are the cause for failures in such fuel rods.

Porcelain based insulators in power transmission have been an accepted material since long. It has been observed that many of the failures in power transmission has been due to the failures of the insulators due to repeated fluctuating stress induced by sudden surge of currents. With the demand for 800–1000 MW power transmission, the problem of having right material to withstand such stresses over long periods of time has become acute.

2.3 *Machine tools*

Ceramic tools are rapidly replacing metals and alloys as cutting tools, bushes and bearings. The cutting tools made out of toughened alumina and zirconia as knives, scizzors, grinding, drilling tools etc encounter sudden shock and severe vibration induced stresses. TiC and TiN coated steels as drill bits are now widely accepted. The stress induced by the sudden contact can be as high as its breaking stress, and the life of the tool bit has to be predicted before irrepairable damage occurs.

Bearing and bushes too have to endure many cycles of operational stresses due to machine and operational vibrations. Attempts are being made to dampen the stress factors by proper design of the machine tools.

2.4 *Automobile engineering*

With the advent of fuel crisis engineers are continually in search of material that can reduce fuel consumption and raise the thermal efficiencies of automobile engine components. Si_3N_4, toughened ZrO_2, and Al_2O_3, SIALON are few ceramic materials being seriously attempted to replace the alloys of engine components. Within a decade from now it will become a common feature in any automobile. In addition to withstanding high temperatures, repeated stress induced by vibration and shock during multiple ON-and-OFF periods imposes serious limitations on the life of a component. Few nations like Japan, USA and Germany are continually investigating the high temperature fatigue behaviour of ceramics. Ceramic processing being a powder metallurgy route the retention of pores of different sizes and shapes with widely varying distributions are inherent flaws that are common cause to failures at a much shorter life than predicted.

2.5 *Orthopaedic implants*

Mechanical integrity is a nearly universal requirement for implant materials. All materials must cohere or 'hold together' if they are to be expected to stay in one shape, in one location, and to perform their designed function. The requirement may be only that they withstand the various stresses that exist in the implant site. Table 1 gives a brief idea about the stresses endured in an anterior cruciate ligament replacement in a patient. A more rigorous requirement exists if part of the intended function for the implant is a mechanical one, such as a heart valve replacement or a fracture fixation device. Then the applications may require the preservation of a minimum value of a property, such as withstanding permanent deformation.

More than 1 lakh ceramic-to-ceramic total hip replacements (THRs) with design for press-fit or acrylic bone-cement fixation are reported to have been implanted till date. This figure can be estimated to, at most, about 3 to 5% of the more frequently used biomaterial combinations of polyethylene (UHHWPE) acetabular cups with alumina or metal balls fixed by conical clamping at the femoral metal stems in clinical

Table 1. Implant life history.

Activity	Peak load (N)	Cycles/year	Total cycles
Stairs			
ascending	67	$4\cdot2 \times 10^4$	$1\cdot7 \times 10^6$
descending	133	$3\cdot5 \times 10^4$	$1\cdot4 \times 10^6$
Ramp walking			
ascending	107	$3\cdot7 \times 10^4$	$1\cdot5 \times 10^5$
descending	485	$3\cdot7 \times 10^4$	$1\cdot5 \times 10^5$
Sitting and arising	173	$7\cdot6 \times 10^4$	$3\cdot0 \times 10^6$
Undifferentiated	<210	$9\cdot1 \times 10^5$	$3\cdot6 \times 10^7$
Level walking	210	$2\cdot5 \times 10^6$	$1\cdot0 \times 10^8$
Jogging	630	$6\cdot4 \times 10^5$	$2\cdot6 \times 10^7$
Jolting	700	$1\cdot8 \times 10^3$	$7\cdot3 \times 10^5$
Total		$4\cdot2 \times 10^6$	$2\cdot9 \times 10^8$

Mean age: 35–48 years
Mean life expectency: 40 years
Strain (range of maximum): 5–10%
Loads: moderate activity level including recreational jogging.
(data taken from Chen *et al* 1980)

applications. It has been ably demonstrated that alumina components of self-paired artificial hip joints, especially thin-walled, screw-in (bone) cups designed with sharp-edged anchorage profiles can fail by brittle fatigue fracture (Walter 1986).

A tooth is one of the most demanded material in a human body that requires to endure under varying stresses and strains. Filling a cavity and/or replacement by porcelain or zirconia based ceramics have been quite common since long. The fatigue stresses to be endured over 10^7 cycles in one's lifetime is quite a phenomena! The fatigue behaviour and life predictions are thus of very relevance and has been a subject of study on these substitute materials.

2.6 *Tectonic activity*

It is proposed that the entire surface of the earth is composed of series of internally rigid, but relatively thin (100–150 km) plates (figure 1). Although the size of the plates is variable, much of the earth's present surface is occupied by half a dozen or so large plates in motion both with respect to each other and to the earth's axis of rotation. Virtually all seismicity, volcanicity and tectonic activities are localized around plate margins and are associated with differential motion between adjacent plates.

All known earthquakes have characteristics which strongly suggest their generation by a double-couple mechanism (figure 2) i.e. by slip on some kind of fault plane or shear zone. Typically slip occurs on an existing plane when the stress difference across it is sufficient to produce rupture at the point where the two sides of the fault are locked. On failure at the locking point due to repeated stresses—a manifestation of cyclic fatigue—there is an explosive release of elastic energy in all directions, resulting in earthquakes. Release of molten magma across the fault areas might result in volcanic activity.

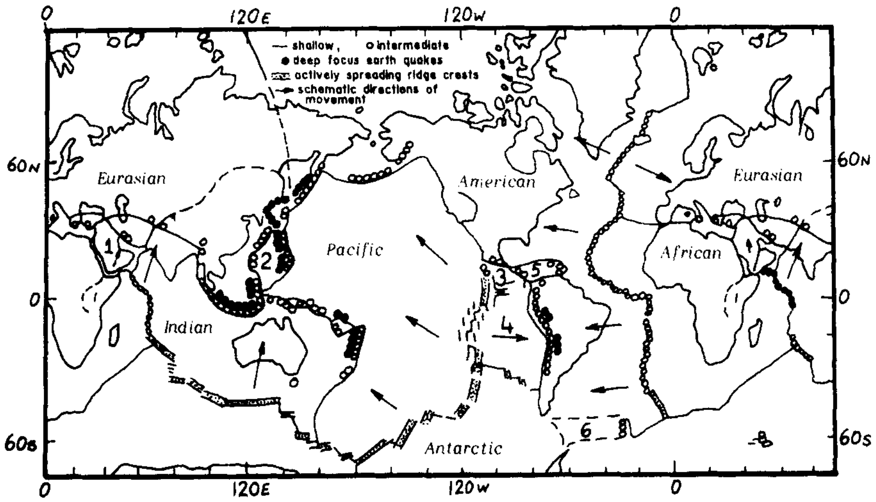

Figure 1. Summary of the seismicity of the earth. The six major aseismic crustal plates are named. Some minor plates are numbered: (1) Arabian; (2) Phillipine; (3) Cocos; (4) Nasca; (5) Caribbean; (6) Scotia.

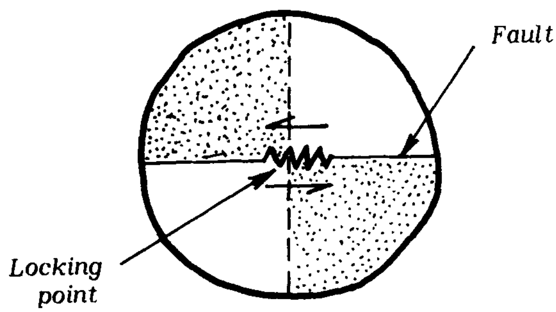

Figure 2. The double-couple mechanism. Stippled area first motions are compressional and other dilational.

3. Previous work

One of the earliest work on fatigue behaviour of sintered alumina after Williams (1956) was that of the present author (Sarkar and Glinn 1969, 1970). During the sixties agreement was lacking on the fundamental issue whether ceramics were susceptible to dynamic fatigue. One widely held view (Kingery 1959; Weil 1961) was that such brittle materials would survive alternating or repetitive stressing indefinitely if stressing was conducted below the single-cycle failure level and that, if damage was observed after repeated stressing, it may have resulted either from an accidental over-stressing at some stage or have been the result of growth of pre-existing flaws. The opposing view was that ceramics would exhibit damage and eventual failure owing to deformation occurring during repetitive stressing (Williams 1961) even though, after a single application of stress none had been observed. In spite of plastic deformation being observed in these materials (Congleton and Petch 1966), on the basis of the meagre existing evidence at that time it had been difficult to support or refute either view. The

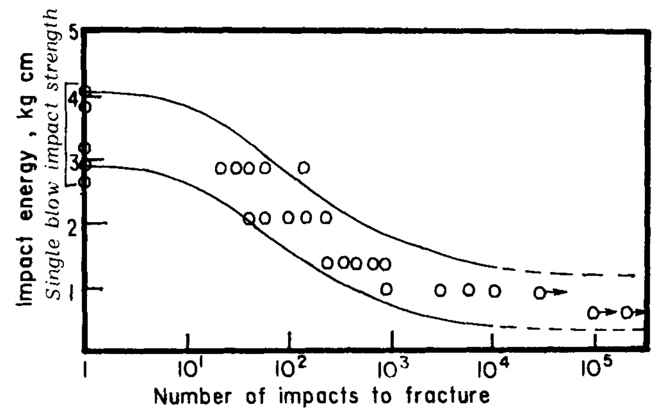

Figure 3. Impact fatigue of Sintox alumina.

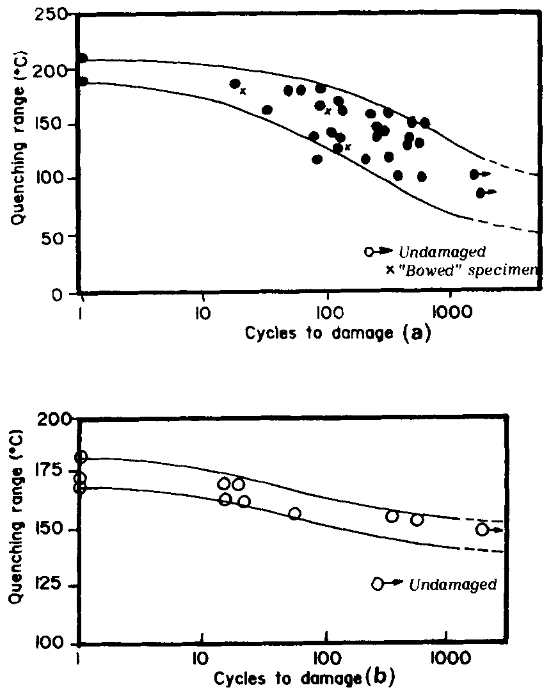

Figure 4. **a.** Thermal fatigue of Sintox alumina (cycle = 10 min) and **b.** thermal fatigue of Lucalox alumina (cycle = 10 min).

development of a new type of impact fatigue apparatus for determining single and repeated impact strength of ceramics was undertaken and demonstrated that for alumina a distinct fatigue behaviour existed (Sarkar and Glinn 1969) (figure 3) with progressive endurances at lower stress regimes.

Subsequently it was shown (Sarkar and Glinn 1970) that for alumina ceramics repeated thermal cycling (figures 4a, b) increased endurance with decreasing quenching-range, i.e. with decreasing applied stress. Failure at stress levels as low as 60% of the critical value of single-cycle precluded the possibility of failure by accidental over-stressing. A dynamic

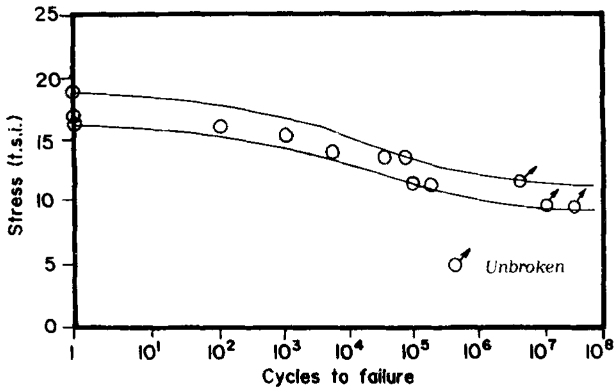

Figure 5. Mechanical fatigue of Lucalox alumina.

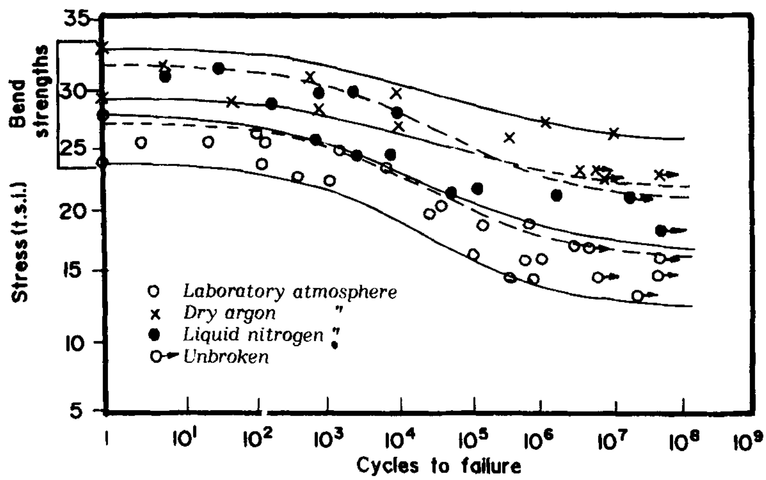

Figure 6. Comparison between mechanical fatigue behaviour of Sintox alumina in various environments.

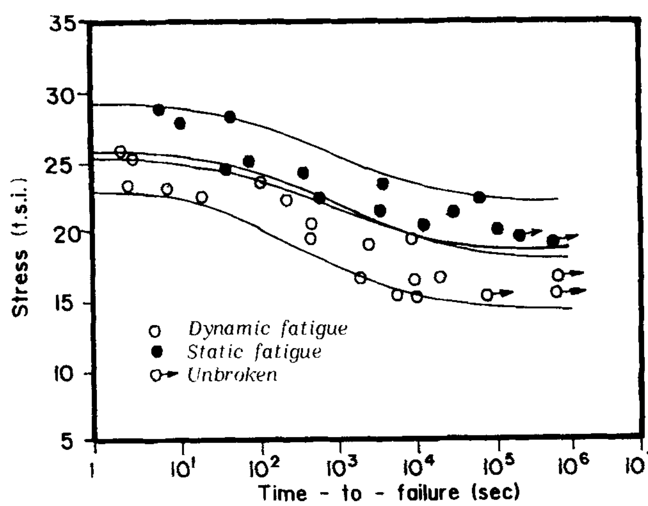

Figure 7. Comparison between dynamic and static fatigue of Sintox alumina.

fatigue effect was also apparent, though it might have been due to the growth of pre-existing flaws. Figure 5 (mechanical stressing) showed that the high-alumina ceramics examined were susceptible to fatigue failure. Testing them in dry argon and liquid nitrogen (figure 6) and comparing dynamic and static stressing behaviour on a time-to-failure basis (figure 7) indicated that such materials, unlike glass (Gurney and Pearson 1948), failed in normal loboratory atmospheres substantially earlier under repeated stress than under static stress. Eliminating conditions of either dynamic or static failure, resulted in improved resistance to fatigue indicative of both dynamic and static effects being operative under normal laboratory testing conditions.

4. Present work

4.1 *Impact fatigue of a hard porcelain*

After the work of Sarkar and Glinn (1969) the only reported work on impact fatigue of ceramic materials was by Huffine and Berger (1977). Since then static and cyclic fatigue behaviour of ceramics have been extensively studied where materials were tested under various types of loading such as plane bending (Mizushima and Knapp 1956), cantilever bending (Kossowsky 1973), pull-push loading (Guiu 1978), pull loading (Ohji *et al* 1990), four-point bending (Horibe and Hirahara 1991), repeated indentation (Reece and Guiu 1990), rotary bending (Ko 1992) to mention a few. Increasing use of ceramics where frequent stress oscillations were encountered compelled the author to reopen the investigation on impact fatigue of brittle materials. A case study on a hard porcelain has been presented here (Maity *et al* 1994).

4.1a *Repeated impact equipment*: For the impact fatigue tests a simple machine based on earlier work (Sarkar and Glinn 1969) was devised and constructed. Details of the machine are given elsewhere (Maity *et al* 1994). The machine was essentially a modified Charpy type impact tester with a cylindrical hammer mounted at the extreme end of the pendulum arm. The machine was instrumented for repeated blows, up to failure. An electronic circuit was so designed that it triggered off the system upon specimen failure. Resulting number of impacts were stored in a permanent storage electromagnetic counter.

4.1b *Repeated impact tests*: For repeated impact tests a pendulum length of 28·3 cm and a hammer weight of 0·189 kg were used. The single impact fracture value was obtained by reducing the angle of swing starting from high angle to a value where the material sustained the impact without failure. For repeated impact tests, the angle of pendulum swing was set at 5° intervals below the angle at which single impact failure occurred. The angle of swing was progressively reduced at intervals of 5° till the material sustained impacts beyond 3×10^4 cycles.

4.1c *Analysis of results*: From figure 8 (Maity and Sarkar 1995) it is evident that a fatigue behaviour exists in the porcelain material tested. Two distinct regions were defined from the curve. With decrease in applied stress there was progressive increase in endurance of the material. An asymptotic nature of the curve with furthur decrease in applied stress determined infinite endurance at least up to the number of cycles (3×10^4) tested. This threshold in the impact stress was defined as the endurance limit and was 36·11 MPa for the present material. The 'fatigue ratio' was 40·03% of the single

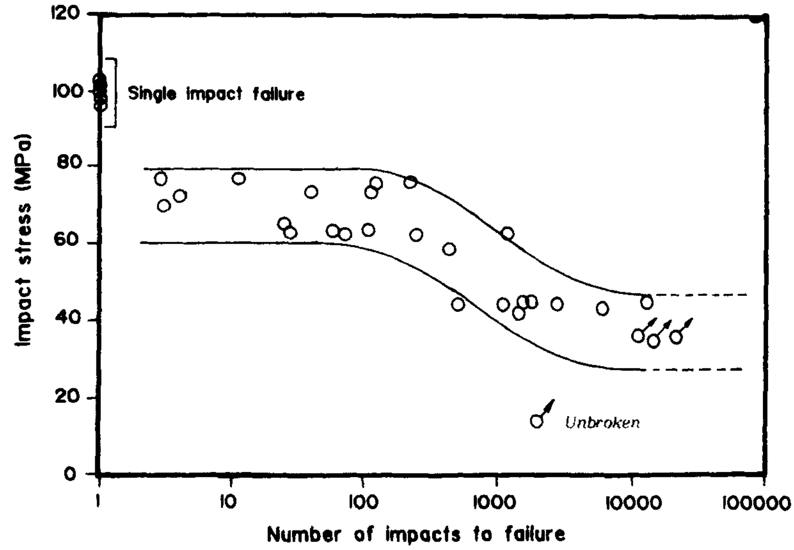

Figure 8. Impact fatigue of a porcelain ceramic.

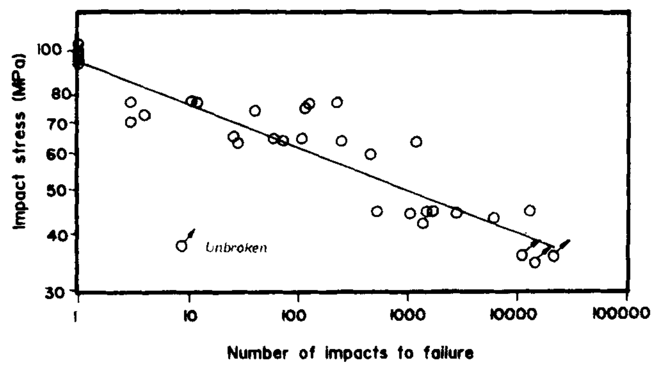

Figure 9. S–N curve including single impact strength.

impact breaking strength. This data is important from the engineering point of view for designing structural ceramic components.

To examine the correlation between single impact strength and fatigue strength, test results were plotted together on a double logarithmic graph (figure 9). In the figure the S–N curve could be expressed by linear regression analysis to be a simple power law of the form

$$\sigma^n N = A,$$

where σ is the impact stress, N the number of cycles to failure and A a constant. The exponent n obtained from the graph was about 10·01 and indicated fatigue resistance of the material. The value of n depend on frequency and amplitude of the applied stress (Ko 1992). Also during impact fatigue testing, impact on the specimen produced a shock wave which induced a greater damage than the slow cyclic stressing to the same level as performed in conventional fatigue tests, defining the value of n.

4.1d *Fractographic analysis*: Stereomicrographs of fractured surfaces of fatigue failured samples showed a smooth surface in the mirror region (figure 10). Dominantly rougher surface in the area away from the mirror region (figure 11) commonly referred to as hackle, was a result of crack path deflection and crack branching. Evidence of mist region could not be clearly identified due to heterogeneous microstructure of the porcelain.

Fractographic analysis of mirror and hackle regions under SEM are shown in figures 12–13 respectively. The mirror regions showed a dominantly transgranular crack path. Beyond the mirror region increasing intergranularity was a result of the meandering nature of the propagating crack.

Expansion of the crack during impact fatigue loading is related to resistance at the crack front. When residual stresses at the crack tip surpasses the work which resists crack-tip expansion, the crack expands. Capability of resisting crack expansion is related to microstructure of the material (Jin 1986). It is suggested that the dominant factor that determines the tortous course of crack propagation in a glassy-crystalline matrix is interaction of the crack with crystalline particles in the matrix. These particles

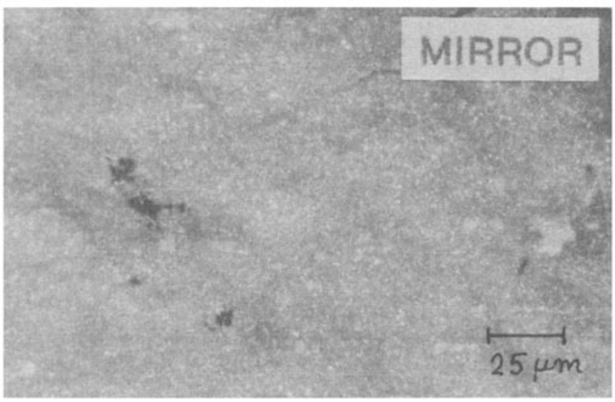

Figure 10. Stereomicrograph of mirror region.

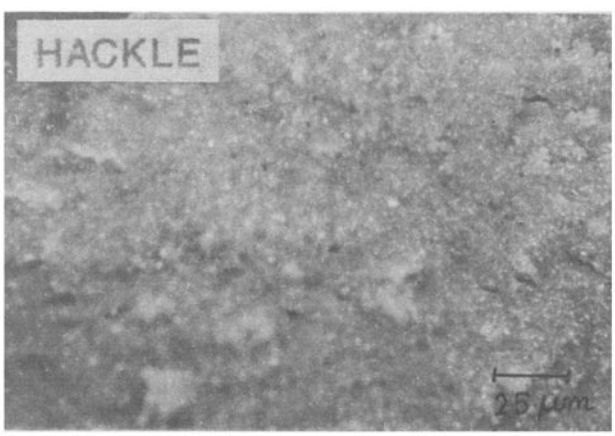

Figure 11. Stereomicrograph of hackle region.

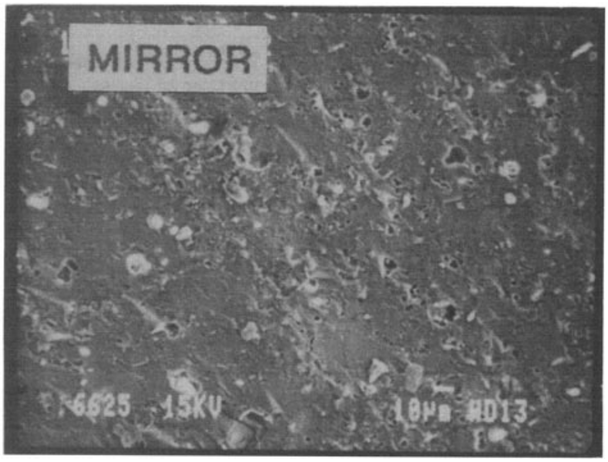

Figure 12. SEM of mirror region showing transgranular fracture.

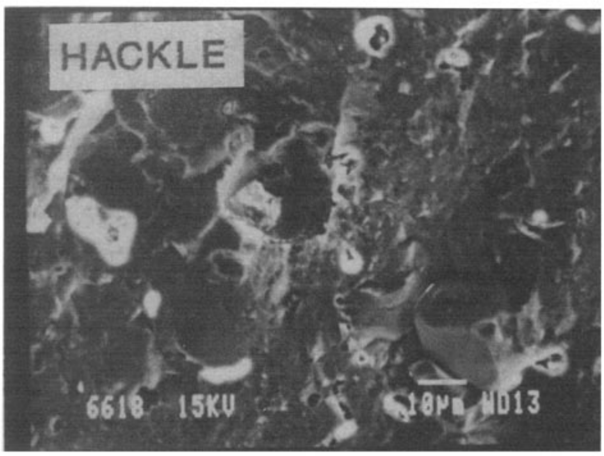

Figure 13. SEM of hackle region showing intergranular fracture.

appear to block the crack motion resulting in local barriers that must be overcome for crack motion to continue. Stress fields due to thermal expansion anisotropy of particles and thermal expansion mismatch between particles and surrounding matrix can also be a contributing factor to the interaction (Pletka 1978). However this type of interaction does not occur in a relatively homogeneous microstructure. Air is a mild corrosion agent. When air, together with impact stress acts on the crack, destructibility is more severe than when either of them acts alone on the crack (Weiderhorn 1974).

4.2 *Indentation fatigue on glass*

Indentation fatigue is an important technique to study the failure mechanism of glass under alternating load since long. Its interest has become more prominent recently due to large application of such glasses in different areas encountering fluctuating stress. Since a brittle material like glass is primarily elastic in nature so it gets fractured while applying a stress higher than the elastic limit.

Workers (Taylor 1950; Ainsworth 1954) as early as 1950 had shown the formation of plastic deformation in glass under stress by indentation technique. But their sudden failure under low stress prompted others to undertake a detailed fatigue study (Lawn *et al* 1981; Vaughan *et al* 1987; Reece and Guiu 1991; Sparks and Hutchings 1992). The process involved application of ball, knoop or diamond indenter on a prepared surface of a glass with a critical load creating an impression with surface cracks. Indentation was then repreated with several subcritical loads for the crack to grow leading to chipping of the material which is technically termed as fatigue fracture. Sparks and Hutching (1992) however did not follow the conventional rule of earlier workers. They applied a load above critical stress of the glass to initiate a crack first followed by repeated indentation at the same point with the same load to propagate the crack to chipping.

Contrary to the earlier works, even with repeated application of very small amount of stress by indentation, the initiation of cracks on the surface of the glass and its subsequent failure could be observed (Banerjee and Sarkar 1995). Also increase in plastic cavity was established at the vicinity of indentation to a limiting number of cycles after which surface cracks occurred leading to eventual chipping. This phenomena was observed on sodalime glass with subcritical loads of 0·1 N, 0·15 N, 0·25 N, 0·50 N, and 1·0 N where cracks were found to initiate at 65, 60, 30, 15, 2 cycles respectively (figure 14). Load was plotted against number of indentation cycles needed to initiate radial crack and is shown in figure 15. Susceptibility of sodalime glass to fatigue failure was imperative. Static fatigue behaviour cannot be ruled out though the time between two consecutive cycles were very small. However, an interesting feature of this experiment was the increase in the diagonal lengths of the impression in each

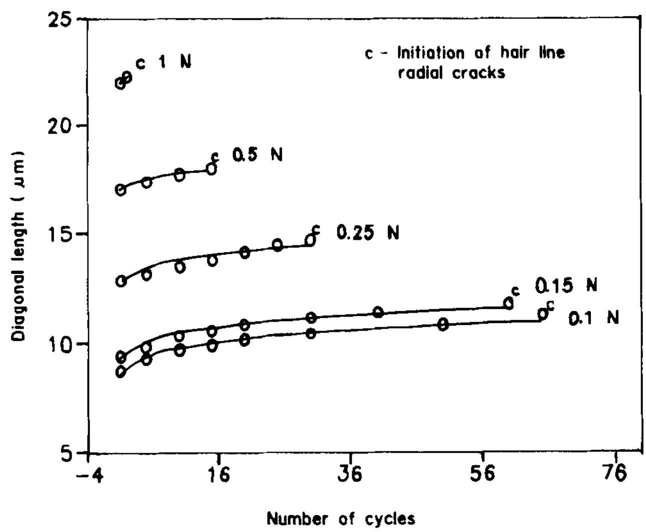

Figure 14. Gradual increase in the diagonal length with repeated cycles at loads 10, 15, 25, 50 and 100 g where 'c' indicates the point of cracking.

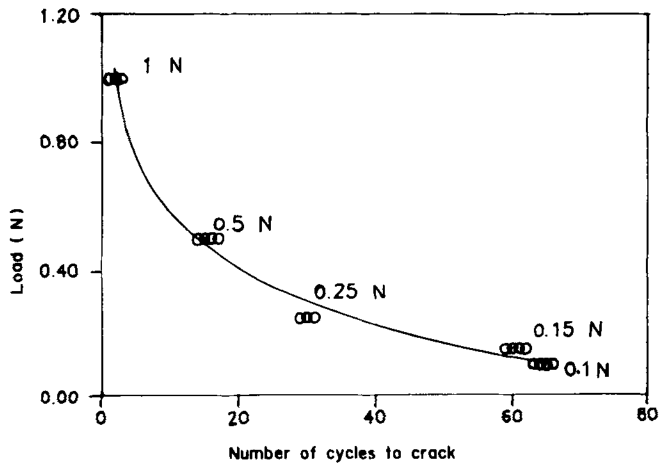

Figure 15. Fatigue graph showing load to number of cycles at cracking.

cycle before hair line radial cracks appeared which showed that glass do undergo continuous plastic deformation under subcritical loads before its failure.

To analyse the above phenomena, let us consider a stress σ_a applied on a prepared surface of a glass at a localized zone. This stress being greater than the yield stress (σ_y) but less than the critical stress (σ_c) of glass, a small localized zone gets plastically deformed. Consequently a plastic strain develops. Since elastic zone lies below the plastic zone and stress being a continuous vector, the plastic stress slowly decreases and at the boundary of the elastic zone becomes

$$\sigma_{p_{(r\ b)}} = \sigma_{c_{(r\ b)}},$$

where ($a \leqslant r \leqslant b$), a, b are the radius of plastic zone and elastic zone respectively. Thus during loading the equilibrium condition can be quantitatively defined as

$$\sigma_a = \sigma_p.$$

As the indentor was unloaded the material got partially recovered thus reducing the diagonal length from the original length when the indentor was fully within the formed cavity. Thus at the surface around the point of application the stress was zero but due to elastic recovery the stress concentration around the stressed zone was given by a coupled equation

$$\sigma_r = (\sigma_p + \alpha\sigma_e),$$

where σ is a scalar quantity within the domain ($0 < \alpha < 1$) and σ_r a residual stress of the material. The direction of this residual stress is towards the surface.

On imposing a new impression during the second indentation when the indentor meets a new surface at the cavity produced by the first impression the diagonal length increases but not to the same dimension as in first cycle due to the residual stress opposing the applied stress.

This process was repetitive thus increasing the digonal lengths at each cycle but in decreasing magnitude due to cumulative residual stress built up at each cycle. The magnitude of this residual stress at n number of cycles can be calculated by

$$\sigma_r^* = \sum_{j=1}^{n} (\sigma_{p_j} + \alpha_j \sigma_{e_j}),$$

here σ_r^* denotes the total residual stress. Neglecting other components when the sum total of applied stress and residual stress exceeds the fracture stress of glass, surface cracks get generated. With further cycle chipping would occur. It is thus imperative that glass do undergo fatigue failure under subcritical loads.

4.3 *Granulation of ceramic powders by repeated impact*

It had been shown that high energy impact vibrational ball milling (HEIVBM), apart from particle fracture, may induce lattice strain in hard metal carbides and ceramic oxides (Lewis and Lindley 1964; Cutter and McPherson 1969; Lewis and Wheeler 1969). The effect was studied by X-ray line broadening techniques, where the broadening of the line profile obtained by milling was interpreted in terms of crystal size, strain and stacking faults. Since ceramics are known to be brittle, serious doubts have been raised regarding the interpretation of the results, and the broadening of the line profiles has often been attributed to particle size reduction alone. Sarkar and Towner (1971) have shown by X-ray line broadening and electron microscopy studies, that ball milling introduces a strain in ceramic powders in the form of dislocations. However, formation of a high pressure phase by high energy repeated impact has not previously been reported.

A high pressure polymorph of titania (HPPT) has been developed from an anatase phase using HEIVBM. Thermal results strongly suggest that HPPT is a metastable phase at room temperature and transforms slowly to rutile phase on heating above 500°C. The polymorph transforms completely to rutile at 900°C.

Titanium dioxide being a polymorphic compound occurs in nature in three crystalline forms, namely, rutile (tetragonal), anatase (tetragonal) and brookite (orthorhombic). A fourth polymorph, having α-PbO_2 structure, had been developed from the anatase, brookite and rutile phases of titania by static high pressure and shock wave techniques (Dachille and Roy 1962; McQueen *et al* 1967; Simons and Dachille 1967; Linde and DeCarli 1969). This polymorph has been developed by the authors for the first time using a new technique of HEIVBM from anatase phase (Chaudhuri *et al* 1994).

Anatase titania powder of high purity (99%) from B.D.H. was dry ball milled for various time periods up to 100 h in a Glen Creston M280 vibratory mill. XRD spectra of the powders were recorded in a PHILIPS X-ray Diffractometer (Model PW-1730).

Figure 16 shows the various XRD spectra of titania powder after different milling times. The XRD pattern (figure 16a) of the unmilled titania powder showed prominent peaks of the tetragonal TiO_2 anatase phase. In the XRD pattern (figure 16b) of the 32 h milled titania powder, a diffuse broad band centred around $2\theta = 31{\cdot}5°$ was found to have developed along with the other lines of anatase TiO_2. The XRD spectra (figures 16c, d) of higher milling time samples, 64 h and 100 h, confirmed the appearance of a broad band in the system. The observed broad band at $2\theta = 31{\cdot}5°$ neither correspond to the diffraction lines of anatase phase nor to the α-Al_2O_3 lines of possible contaminated material from the alumina ball and vial. It was clear from the XRD spectra that the observed band around $2\theta = 31{\cdot}5°$ was a new phase originating

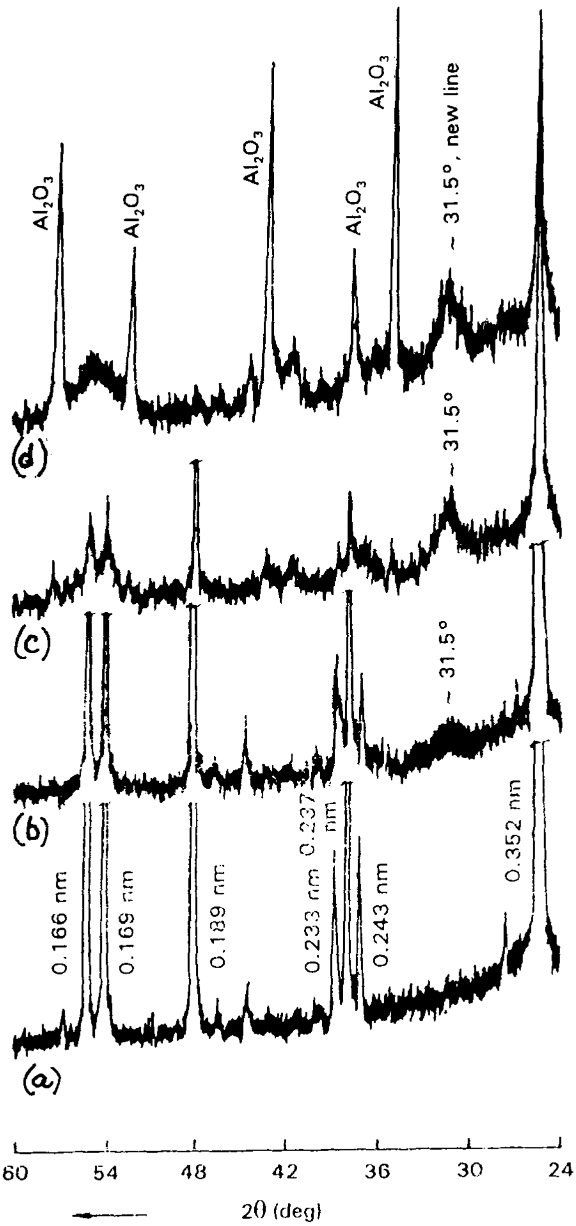

Figure 16. XRD spectra of titania powder milled for various times: (**a**) 0 h; (**b**) 32 h; (**c**) 64 h and (**d**) 100 h.

from the anatase phase of titania by HEIVBM. This new developed phase had been characterized by the authors (Chaudhuri *et al* 1994) to be a high pressure polymorph of titania.

For sequential thermal study, 64 h milled powder was selected because of less contamination of Al_2O_3 and greater amount of HPPT phase developed. The heat treatment was done in air for 1 h at different temperatures starting from 300–900°C. The XRD spectrum (figure 17a) of 64 h milled without heat treated sample showed

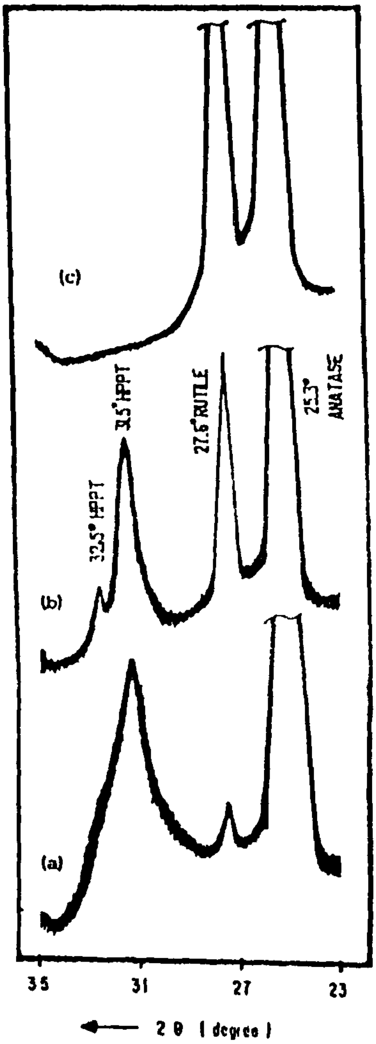

Figure 17. XRD spectra of 64 h milled sample; (a) RT; (b) 600°C and (c) 900°C.

a broad band around $2\theta = 31{\cdot}5$°C which was due to HPPT discussed earlier. The band was highly asymmetric in nature showing the presence of another peak around $2\theta = 32{\cdot}5°$ which was clearly discernible in the XRD spectrum of figure 17b. The HPPT was highly stable at room temperature even on heating up to 500°C. On heat treatment at 600°C (figure 17b), it converted slowly to rutile· phase with greater X-ray intensity. The complete transformation of HPPT to rutile was observed at 900°C (figure 17c).

By analysing the experimental results it was concluded that prolonged HEIVBM created sufficient impact shock on the particle causing fracture to a limit beyond which the stress enforces the anatase-titania to grow in a particular fashion, like the high pressure modified phase of titania. The polymorph was a metastable phase at RT and gets converted slowly to rutile above 500°C.

5. Conclusions

Various fatigue related experiments on ceramics and glasses carried out so far by the author and his colleagues have revealed that brittle solids are susceptible to failures under repetitive loads having an endurance limit depending on the applied stress. Whilst static fatigue phenomena cannot be ruled out totally, predominant factors that have emerged are a cumulative residual stress component and a plastic component, the magnitude of which depended upon the material, its microstructure and environment.

It is evident that the cumulative residual stress component, per cycle, around the crack front plays a contributory role in fatigue failures according to

$$\sum_{J=1}^{N} (\sigma_{p_j} + \sigma_{r_j}) = \sigma_f,$$

where σ_{p_j} is the plastic and σ_{r_j} is the residual stress component at the Jth cycle, N the total number of cycles, n material constant and σ_f the fracture stress. During fatigue σ_{p_j} and σ_{r_j} components are predominant for ductile metals and brittle glass/ceramics respectively.

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