Damage Accumulation and Fatigue Life of Particle-Abraded Ceramics

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Purpose: This investigation compared initial and fatigue strengths of particle-abraded ceramics to those of as-polished alumina and zirconia ceramics in crown-like layer structures. ***Materials and Methods:*** Alumina or zirconia plates bonded to polycarbonate substrates were subjected to single-cycle and multi-cycle contact (fatigue) loading. Cementation surfaces of the ceramic were damaged by controlled particle abrasion, indentation with a sharp diamond at low load, or a blunt indenter at high load. The stresses needed to initiate radial fractures were evaluated. ***Results:*** The strengths of specimens were lowered by fatigue loading. After the equivalent of 1 year of occlusal contacts, the strengths of undamaged specimens degraded to approximately half of their single-cycle values. In particle-abraded specimens, an additional 20% to 30% drop in strength occurred after several hundred load cycles. Particle abrasion damage was approximately equivalent to damage from sharp indentation at low load or blunt indentation at high load. ***Conclusion:*** Damage from particle abrasion, not necessarily immediately apparent, compromised the fatigue strength of zirconia and alumina ceramics in crown-like structures. In fatigue, small flaws introduced by particle abrasion can outweigh any countervailing strengthening effect from compression associated with surface damage or, in the case of zirconia, with phase transformation. *Int J Prosthodont 2006;19:442–448.*

The clinical survival of all-ceramic crowns is a complex combination of factors, including: material properties such as strength, modulus, and toughness; damage caused by shaping and handling procedures such as casting, machining, grinding, and particle abrasion; and cyclic loading from occlusal function. Particle abrasion is of special interest because of its countervailing influences on crown performance. It is an effective mechanism for removing residual investment associated with routine fabrication techniques,[1,2] can enhance the ceramic luting agent bond,[3–6] and can be used to prepare fractured porcelain for repair.[7] Particle abrasion can also introduce a protective layer of residual compressive stress into the surface, thus inhibiting fracture processes.[8–13] Such compressive layers are especially significant in yttria-stabilized zirconia (Y-TZP), because this material can undergo a stress-induced volume-expansion phase transition.[8] On the other hand, particle abrasion can also introduce surface flaws or microcracks that may accelerate failure.[14] There is a need to understand these various influences, especially strength degradation related to laboratory and clinical particle-abrasion treatments.

The objective of this investigation was to evaluate the effects of particle abrasion on the initial and fatigue strength of 2 clinically relevant ceramics: fine-grain alumina and Y-TZP. The working hypothesis was that flaws introduced by particle abrasion would degrade the strength of these ceramics, particularly when they are subjected to cyclic fatigue loading, potentially outweighing any benefits from damage-induced residual surface compression or phase transformation.

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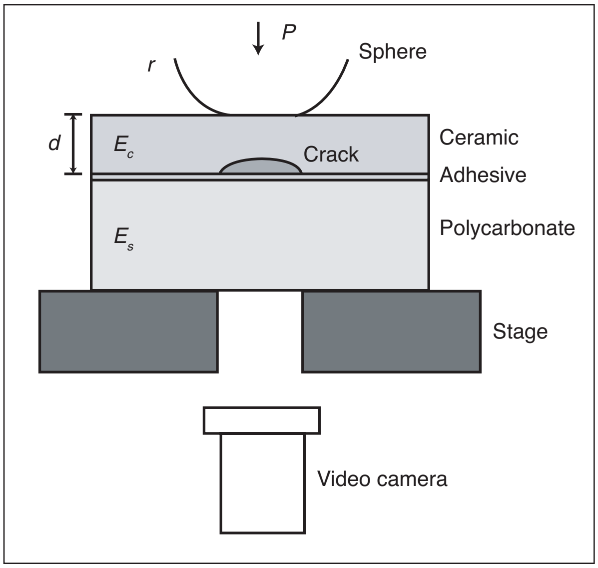

Fig 1 Schematic of the flat bilayer test specimen, consisting of a ceramic plate of thickness d and modulus E_c bonded with epoxy to a polycarbonate substrate of modulus E_s. Loading with the sphere flexes the ceramic layer and produces a radial crack at the cementation surface. This configuration simulates occlusal loading of a monolithic crown on dentin. Cyclic loading is used to determine fatigue properties of the bilayer system.

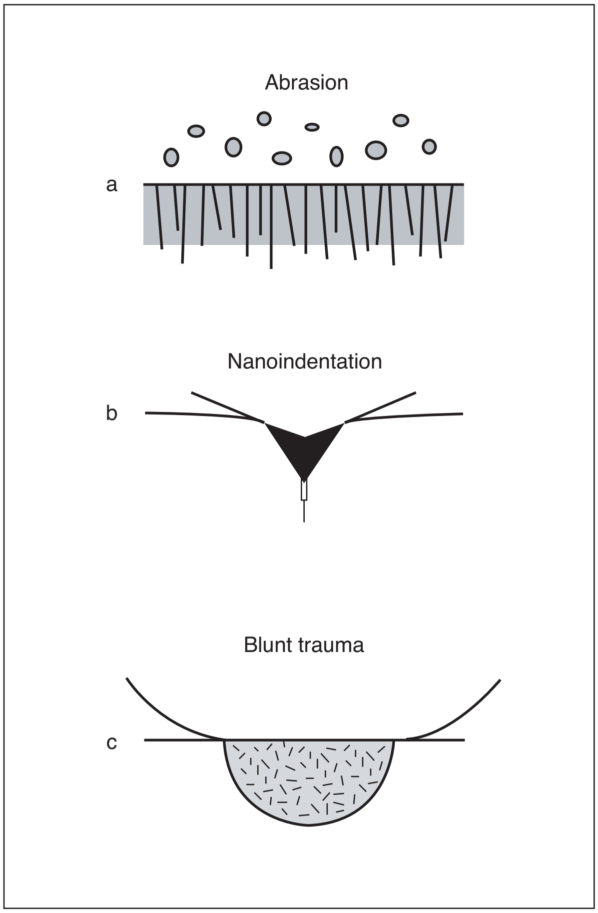

Fig 2 *(right)* Damage treatments in the ceramic undersurface: **(a)** particle abrasion, producing a quasiplastic damage zone of microcracks; **(b)** sharp diamond indentation at low load (1 N), producing an intense deformation zone with individual, well-developed cracks; and **(c)** WC sphere indentation, producing a blunt trauma quasiplastic zone with microcracks.

Materials and Methods

Flat bilayer specimens of ceramic plates supported by a polycarbonate base were prepared in simulation of monolithic ceramic crowns on dentin. The ceramic undersurfaces (cementation surfaces) were subjected to damage treatments before they were bonded to the base. The ceramic-polycarbonate bilayers were loaded at their occlusal surfaces with a hard sphere in single-cycle and multi-cycle fatigue loading. This contact loading, in addition to producing Hertzian stresses in the near-contact region, induces flexural tensile stresses in the lower half of the ceramic layer. These latter stresses result in radial cracking at the cementation surface.[15,16] The initiation and subsequent propagation of radial fractures were documented with a video recording system located beneath the specimen (Fig 1).

The tests were performed to evaluate the effects of particle abrasion on the initial and fatigue strengths of fine-grain alumina (AD995, CoorsTek) and Y-TZP (Pro-TZP, Norton). These materials are often considered to be structural ceramics. For comparison, the strengths of as-polished specimens of currently used dental ceramics (Procera Alumina and Procera Zirconia, Nobel Biocare; Lava Zirconia, 3M ESPE; and VITA Zirconia, VITA Zahnfabrik) were also measured.

Specimen Preparation

Flat alumina plates measuring 20 × 20 × 1 mm and zirconia plates measuring 20 × 20 × 0.6 mm were ground flat and parallel, then polished to 1-μm finish. The ceramic plates were bonded onto clear polycarbonate substrates (Hyzod AIN Plastics) 12.5 mm thick with a thin layer (approximately 10 μm) of epoxy resin (Harcos Chemicals), with the damaged surfaces at the epoxy interface. The elastic properties of the epoxy were similar to those of the polycarbonate, so that the specimens could be considered to be bilayers.[17]

Five specimens of each alumina and zirconia ceramic were subjected to single-cycle loading to failure. Loading conditions are described below.

Polished plates of alumina and Y-TZP were subjected to different damage treatments, indicated schematically in Fig 2. One group of each ceramic was abraded with 50-μm aluminum oxide particles for 5 seconds from a standoff distance of 10 mm using 276 KPa compressed air pressure. This treatment created a damage zone of intense plasticity (known as quasi-

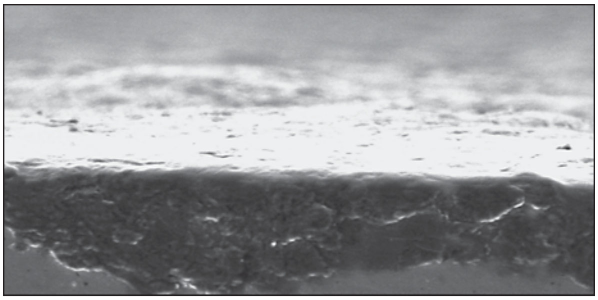

Fig 3 Particle-abrasion damage in a Y-TZP specimen. Substantial damage to a depth of approximately 4 μm in the ceramic is revealed. Section and part-surface views.

plasticity in the ceramics literature[18]) with associated deep microcracks (Fig 2a). An example of the severe damage layer created is shown in Fig 3. The second group was indented with a sharp diamond (Berkovich) indenter (Nanoindenter XP, MTS Systems) at a 1 N load. This produced individual sharp cracks of μm scale emanating from the indentation corners (Fig 2b). Such indents simulate inadvertent scratching damage from small, sharp particulates. A third group was indented with a 2-mm radius tungsten carbide (WC) sphere at a 3,000 N load, thus creating blunt trauma. This damage was a quasiplastic zone containing a distribution of small microcracks (Fig 2c). As a control, the fourth group of ceramic plates of both the alumina and Y-TZP were left as polished. Each group contained 5 specimens.

Loading

The specimens were loaded on their top surfaces with a WC sphere indenter with a radius of 3.18 mm (Fig 1). Loading was applied in 2 modes: *(1)* single-cycle (for all ceramics) in a screw-driven machine (Model 5500R, Instron) at a fixed rate of 1 mm/min, and *(2)* cyclic fatigue loading (for AD995 alumina and Pro-TZP zirconia only) at 10 Hz frequency in a hydraulic testing machine (Model 8500, Instron). In the fatigue tests, each specimen was loaded at 95% of the single-cycle load strength and fatigued to failure. If failure occurred before 1 million cycles, a new specimen was tested with a load of 90% of the single-cycle load strength, and fatigued to failure. Any time failure occurred before 1 million cycles, the load delivered to the next specimen was reduced by an additional 5% of the single-cycle load strength, until the specimens could withstand 1 million cycles.

To maintain alignment of the specimen and indenter during cycling, the minimum load was 2 N in the fatigue tests. Tests were performed in a laboratory environment with approximately 50% relative humidity at 20°C.

Fracture Detection

Radial fracture initiation at the ceramic cementation surface was imaged directly from beneath the transparent epoxy-polycarbonate base using a video camcorder (Canon XL1, Canon) equipped with a microscope zoom system (Optem, Santa) (see Fig 1).[19] The system simultaneously captured an image and the load-count indicator from the hydraulic testing machine.

These observations enabled direct measurement of the critical loads for the onset of radial fracture as a function of number of loading cycles. Magnification of the specimen with the zoom system was 10× and the video capture rate was 30 frames/second. Each loading cycle was captured in 3 sequential frames. Frame-by-frame postfracture analysis of the images allowed the number of cycles prior to crack onset to be accurately determined. Corresponding critical stresses were calculated using the relationship for plates on compliant supports[20–22]:

$$S = 0.75\ (P/d^2)\ \log\ (E_c/E_s)$$

where P is the load to initiate fracture, d is the ceramic thickness, E is the elastic modulus, and subscripts c and s refer to ceramic and polycarbonate substrate, respectively. In this analysis, $E_c = 205$ GPa for Y-TZP, $E_c = 372$ GPa for alumina, and $E_s = 2.3$ GPa for polycarbonate. For specimens with controlled indentations, special care was taken to align the indentation site along the loading axis.

Statistical Analysis

Differences between the strengths of ceramics subjected to single-cycle loading were analyzed using simple *t* tests. For fatigue tests, 2-way analysis of variance was used to compare materials and the strength of specimens with polished versus damaged surfaces.

Results

Single-Cycle Loading

Table 1 compares the critical stress levels needed to form radial cracks in as-polished and particle-abraded Y-TZP and alumina plates in single-cycle loading. Values represent means and SDs for 5 measurements for each data set. The strengths of Y-TZP specimens are statistically lower for the abraded surfaces ($P < .05$), but remain in excess of 1 GPa. The strengths of alumina are not statistically different in the as-polished and abraded states.

Table 1 Material Strength in Single-Cycle Loading*

Material class	Strength (MPa) As polished	Strength (MPa) Particle abraded	Brand name	Manufacturer
Alumina	754 ± 60	691 ± 69	AD995	CoorsTek
	706 ± 46		Procera Alumina	Nobel Biocare
Zirconia	2486 ± 88	2078 ± 60†	Pro-TZP	Norton
	1654 ± 152		Procera Zirconia	Nobel Biocare
	1475 ± 92		Lava Zirconia	3M ESPE
	1267 ± 116		VITA Zirconia	VITA Zahnfabrik

*As-polished strength of commercially available dental ceramics is provided for comparison.
†Significantly different from as-polished strength.

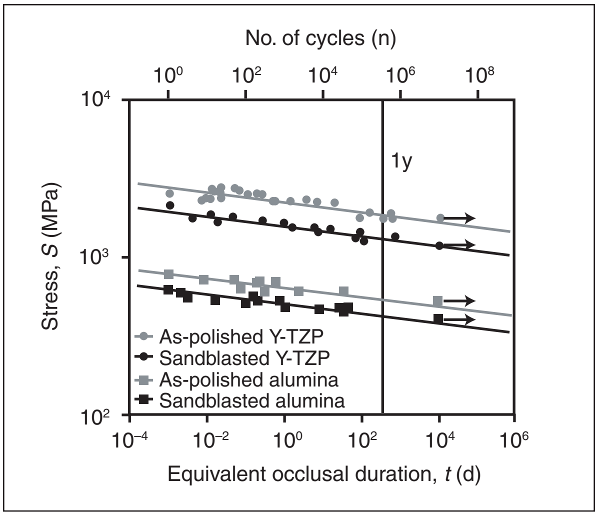

Fig 4 Flexural stress needed to produce radial cracks as a function of number of cycles n or equivalent occlusal contact cycles in fatigue tests, comparing Y-TZP and alumina bilayer specimens (see Fig 1) with as-polished and particle-abraded surfaces. Data are individual test results. Solid lines are linear best fits.[14] Arrows indicate test "runouts."

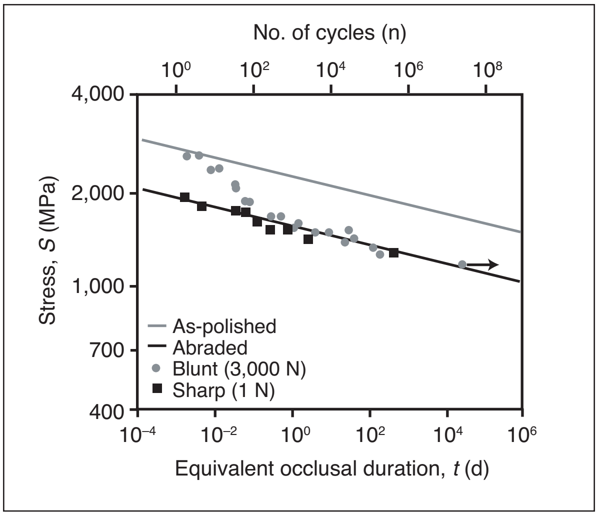

Fig 5 Flexural stress as a function of number of cycles n or equivalent occlusal contact duration needed to produce radial cracks in Y-TZP for bilayer specimens with different ceramic cementation surface treatments. Data points are individual test results. Solid lines are linear best fits from Fig 4 and Zhang and Lawn.[25] Arrow indicates test "runouts."

Cyclic Fatigue Loading

Figure 4 shows the results from cyclic fatigue tests of particle-abraded specimens compared to as-polished specimens for Y-TZP and alumina, plotted as stress *S* versus number of loading cycles n. The plot is given in logarithmic coordinates to accommodate data over several orders of magnitude of load cycles. An equivalent "occlusal" test duration t is plotted on the lower axis, based on $t = n/1{,}000$ days (corresponding to a nominal 1,000 occlusal contacts/day). As previously reported, the strength degradation of ceramics from radial crack initiation in single-cycle or multi-cycle loading results primarily from moisture-assisted slow crack growth (see Discussion).[14] For polished specimens of either ceramic, the stress required to initiate fracture is reduced by a factor of nearly 2 over an occlusal contact time of approximately 1 year. With particle-abraded surfaces subjected to cyclic fatigue, stress to initiate fracture is *further* reduced by 30% for Y-TZP and 20% for alumina.

Figure 5 compares critical stresses in cyclic loading for Y-TZP specimens with different surface-damage treatments. The strength degradation for blunt trauma and sharp indentation is similar to that for particle-abraded surfaces. The data for blunt trauma initially show little degradation, but quickly fall off after a few hundred cycles to a level similar to that for particle abrasion or sharp indentation, indicating an additional stage in flaw evolution.

Statistical analysis showed that Y-TZP was significantly stronger than alumina, and that the polished materials were stronger than the particle-abraded materials ($P < .001$). The particle abrasion produced a significantly greater degradation in the strength of Y-TZP than alumina ($P < .05$).

Discussion

The principle finding of this study is that the strength of ceramic materials may be reduced by particle abrasion. Such degradation arises because most particle-abrasion treatments introduce deep surface microcracks, which can act as sources of failure. The deleterious nature of these microcracks needs to be balanced against the potentially countervailing surface compression forces that this same treatment can create. Such compression forces are a manifestation of an outward expansion of cumulative quasiplastic deformation against the surrounding elastic material. Any compression could, in principle, act to inhibit mirocrack extension, thus serving to strengthen the material.[11–14,18,23–25] In Y-TZP, the outward compression is augmented by volume-expanding phase transformations.[8] The results of the present study indicate that these compressive stresses are not sufficient to overcome the strength loss from the microcracks. Moreover, plasticity-induced compressive stresses could be relieved by heat treatment, such as that associated with application of a porcelain veneer to the core ceramic, or by aging.[26]

The degree of strength degradation of the particle-abraded surfaces is similar to that induced in surfaces subjected to controlled pre-indentation damage (blunt or sharp contact). All such surface damage processes introduce some form of quasiplasticity accompanied by microcracks.[14,25,27] Blunt trauma at high loads introduces a relatively large damage zone with many isolated microcracks. Individual microcracks are initially too small to degrade the strength, but under cyclic loading they grow and coalesce with their neighbors. The blunt trauma specimens shown in Fig 5 may have undergone such coalescent degradation, with attendant acceleration of strength loss within a few load cycles. It is sobering that the same level of degradation can be achieved almost instantly by a single indentation with a sharp indenter at a dramatically lower load of 1 N. This highlights the susceptibility of ceramics to contact with sharp particles, as is well documented in the ceramic literature.[28–31] The message to clinicians and laboratory technicians is to minimize the severity of particle-abrasion treatments whenever possible. The use of softer, rounder abrasives (eg, silica microspheres instead of hard, sharp alumina) may be an avenue worth exploring. Ultimately, the avoidance of particle abrasion altogether in favor of other surface-modification treatments for divesting or adhesive bonding of crowns to tooth structure would appear to be an important factor in extending crown life.

There are 2 features of the cyclic fatigue results (Figs 4 and 5) that warrant further comment. First, strength losses up to a factor of 2 may occur over a year or more of occlusal function, as a result of simple progressive extension of otherwise innocuous surface flaws, *even in polished specimens*. In specimens damaged by particle abrasion, the strength is further degraded by an additional 20% to 30% (in alumina and Y-TZP, respectively). Thus, single-cycle strength testing procedures can grossly overestimate the stress-bearing capacity of crown-like structures. Second, notwithstanding the general susceptibility of ceramics to degradations from damage and/or cyclic fatigue, alumina and (especially) Y-TZP nevertheless remain strong. The stresses for Y-TZP remained above 1 GPa, even after cycling over the equivalent of an occlusal year or more. For reference, a 1 GPa stress would be created at the ceramic cementation surface by a 250 N occlusal force acting on a 0.5-mm-thick Y-TZP plate supported by dentin (E-15 GPa). The load needed to initiate fractures is highly dependent on thickness.[19,32,33] The performance of even the strongest materials, such as Y-TZP, can be compromised by the combined effects of damage from particle abrasion and fatigue loading, especially in thin-crown areas.

Although this study has clearly simplified clinical conditions in several respects, it nevertheless provides valuable physical insights into the potential performance of all-ceramic crowns. Still, it is worth discussing the potential limitations of the study design:

1. This study investigated simple bilayer specimens consisting of a single-ceramic core material on a relatively compliant polycarbonate base. This configuration offers simplicity in testing and analysis. The choice of flat specimens should not be considered restrictive. The main purpose was to demonstrate the effect of particle-abrasion damage, without geometrical complications. Extension to specimens with curved surfaces suggests the same qualitative features in strength degradation from imposed damage, with only a slight elevation of the critical loads required to initiate fracture at the ceramic interfaces.[34] Thus, it is unlikely that there would be a substantial difference in the results with anatomically correct dental specimens.
2. Our tests were conducted in air and not in water. Radial fractures develop at the ceramic cementation surface, well away from the outer surfaces and margins, and are not immediately exposed to any external aqueous environment. However, the presence of some moisture at the ceramic/epoxy/polycarbonate interface is inevitable, even if only in trace quantities, from the epoxy or polymeric base, if not from the ceramic surface itself.[35] Trace moisture has been shown to strongly exacerbate damage in a rate-dependent manner, accounting for the fatigue effect. In a clinical situation, moisture from the dentin, and leakage

and water uptake by the luting agent, will expose the cementation surface to a moist environment.

3. The loading frequency used in our fatigue testing (10 Hz) is considerably greater than that of occlusal function. It was selected simply to keep the tests to a reasonable duration. However, this high frequency is not anticipated to have an influence on the results, since previous studies[14] have demonstrated, at least for radial fracture, that fatigue-related strength degradation depends only on test duration and not the frequency of loading.
4. The materials subjected to damage and fatigue in this study were similar but not identical to those used in modern all-ceramic crowns. The strengths of the as-polished alumina and Y-TZP are somewhat higher than those in clinical use (see Table 1). Consequently, the loads needed to initiate failure (and the resulting stresses reported in Figs 4 and 5) likely overestimate the performance of actual dental materials, especially Y-TZP. However, the authors anticipate that all clinical ceramics will be similarly vulnerable to fatigue degradation, because of the pervasive influence of moisture on crack growth. If true, the residual strengths after cycling will tend to be lower than those reported here.
5. The focus of this investigation was initiation of radial fractures from the cementation undersurface of the ceramic. There is the potential, however, for a number of competing fracture modes to cause crown failure, depending on the relative thickness of the ceramic and porcelain layers, and the properties of the materials selected (crown, luting agent, and dentin or foundation restoration).[16,36,37] Ultimately, these competing modes must be considered in any design considerations. Nevertheless, radial cracking is a serious threat to crown failure, and the bilayer test used in this study provides a direct route for investigation of this fracture mode.

Conclusions

In summary, the damage created by particle abrasion can reduce the strength of both alumina and Y-TZP ceramics. In fatigue cycling, the extent of this reduction can be 20% to 30% greater than for as-polished specimens, and is equivalent to the strength reduction of either a 1-N sharp indentation or a 3,000-N load blunt indentation. Subjecting specimens to single-cycle testing does not adequately reflect the extent of the damage introduced. Laboratory and clinical procedures should be evaluated relative to the extent and nature of the damage introduced into ceramic layers. In vitro test screening of new ceramic materials should include fatigue testing. The use of sharp-particle abrasion on ceramics in laboratory and clinical procedures should be minimized.

Acknowledgments

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Literature Abstract

Delay in referral of oropharyngeal squamous cell carcinoma to secondary care correlates with a more advanced stage at presentation and is associated with poorer survival

The purpose of this retrospective case notes–based study was to investigate delay in referral, defining this as the time from symptom onset to the date of the general practitioner's referral letter to secondary care, and the effect of that delay. Patients diagnosed with squamous carcinoma of the oropharynx were identified from the Department of Oncology computerized database from June 1995 to June 2005. The following information was obtained: age, gender, site of primary cancer, date of symptom onset, date of referral from primary care, TNM stage at presentation, presenting symptom(s), any treatment given in primary care, and outcomes. Of the 110 patients found, only 69 patients' data were used. The following patients were excluded: 19 patients' information could not be obtained, 17 patients' case notes could not be located, and 4 patients were not referred by primary care. The time difference between the date of first symptom and the date of general practitioner's letter referring the patient to secondary care was necessary. Several sources had to be used to obtain the date of the first symptom, since it was a challenge to determine this information. A log transformation was applied to the data for delay in referral, as it was skewed to the right. Spearman rank correlation and PLUM ordinal regression were used to check for any association between delay in referral and stage at presentation. Survival was assessed using Kaplan-Meier survival analysis and the log-rank test. The mean age was 57.5 years old, with 54 males and 15 females. The patients presented in the following stages: II (6), III (16), IVA (35), and IVB (10). There were no stage I or IVC tumors. Forty-three were tonsil tumors, 19 posterior tongue tumors, and 7 were other sites (uvula and palate). Spearman rank correlation showed a positive, but not necessary, linear correlation ($P = .011$) between an increased delay in referral and more advanced state at presentation. Log-rank test showed that the group with less than 6 weeks delay had significantly better survival ($P = .032$). Limitations of this study included the restricted range of the patients in the late stage category, small number of total patients, and mixture of oral and pharyngeal tumors.

Pitchers M, Martin C. *Br J Cancer* 2006;94:955–958. **References:** 11. **Reprints:** Dr M. Pitchers, School of Biological Sciences, University of East Anglia, Norwich NR4 7TJ, United Kingdom. E-mail: m.pitchers@uea.ac.uk—*Alvin G. Wee, OSU College of Dentistry, Columbus, OH*