

**NPL REPORT
DEPC-MPE 027**

**CHARACTERISING
SURFACE DAMAGE IN
ADVANCED TECHNICAL
CERAMICS**

**R MORRELL, A J GANT,
N JENNETT**

NOT RESTRICTED

April 2006



The DTI drives our ambition of 'prosperity for all' by working to create the best environment for business success in the UK. We help people and companies become more productive by promoting enterprise, innovation and creativity.

We champion UK business at home and abroad. We invest heavily in world-class science and technology. We protect the rights of working people and consumers. And we stand up for fair and open markets in the UK, Europe and the world.

The National Physical Laboratory is operated on behalf of the DTI by NPL Management Limited, a wholly owned subsidiary of Serco Group plc

NATIONAL PHYSICAL LABORATORY

**CHARACTERISING SURFACE DAMAGE IN ADVANCED
TECHNICAL CERAMICS**

R Morrell, A J Gant and N Jennett
Division of Engineering and Process Control

ABSTRACT

Two aspects of characterising the surface of advanced technical ceramics have been evaluated in this work: Hertzian indentation ring-cracking and surface acoustic wave (SAW) propagation. The purpose was determine the applicability of these techniques to technical surfaces, as opposed to highly polished ones.

Hertzian ring-cracking was performed using acoustic emission to detect the development of the surface ring crack. Several problems of consistency were obtained due, it is thought, to several factors including material inhomogeneity and tendency of coarser-grained materials to microcrack. Indentation force distributions have been examined and evaluated according to the technique of Warren which allows the apparent fracture toughness to be determined. Values of toughness obtained were of the correct order, but different (possibly appropriately) to expectations for larger planar cracks.

Surface acoustic wave (SAW) propagation was used to investigate the potential for characterising the depth of damage produced by machining processes, as well as the ability to detect additional damage, such as surface cracks produced by indentation. Appropriate values for the velocity of sound and hence elastic modulus could be obtained, but pulse transmission was not as good as in polished metals, and the frequency dispersion used as a measure of surface modulus was not consistent. Additional damage produced considerable scattering, restrictions of determinable frequency range, and difficulties with interpretation.

© Crown copyright 2006
Reproduced with the permission of the Controller of HMSO
and Queen's Printer for Scotland

ISSN 1744-0262

National Physical Laboratory
Hampton Road, Teddington, Middlesex, TW11 0LW

No extracts from this report may be reproduced without the prior written consent of the Managing Director, National Physical Laboratory; if consent is given the source must be acknowledged and the extracts may not be used out of context.

Approved on behalf of the Managing Director, NPL,
by Dr M G Gee, Knowledge Leader, Materials Performance Team
authorised by Director, Engineering and Process Control Division

CONTENTS

1.	INTRODUCTION	1
2.	HERTZIAN RING CRACK GENERATION	2
2.1	Background	2
2.2	Test equipment.....	3
2.3	Test materials	4
2.4	Test results	4
2.5	Optical examination	10
2.6	Discussion	10
2.7	Conclusions.....	11
3.	SURFACE ACOUSTIC WAVE CHARACTERISATION.....	12
3.1	Introduction.....	12
3.2	Preliminary evaluation	13
3.3	Systematic evaluation	13
3.3.1	Test-piece preparation.....	13
3.3.2	Tests performed and results	14
3.4	Discussion	19
3.5	Conclusions.....	20
	Acknowledgements	20
	References	20

1. INTRODUCTION

The surface quality of advanced technical ceramics is crucial for defining their strength and reliability. This arises because once significant internal defects are eliminated by careful processing, surface initiated flaws dominate fracture. However, the surface is vulnerable to external damage, both in its preparation and in its use. In many simple applications, the as-fired surface can be used but this tends to be relatively rough and of poor geometry, and is often inappropriate for engineering applications. In some cases, the surface roughness and any sharp corners may be removed by vibromilling with hard milling media, but this does not achieve accurate geometry. Most commonly, surfaces are machined and/or lapped to engineering tolerances suitable for mating to other engineering components. Examples include seals, bearings, pump parts and hip replacements.

The process of machining a ceramic surface requires diamond tooling. The grinding process is a severe one for the material, and involves the ploughing action of, usually, diamond abrasive particles producing a mixture of brittle chipping out of grains or grain fragments and plastic deformation through scratching. This process can produce residual stresses in the surface, often compressive in nature, and microcracks, often parallel to the direction of grinding or perpendicular to it. The extended nature of the former type of microcracking is often found when fractographic examination is made of fractured bar test-pieces ground across the direction of stressing. It has been well-established that there is a delicate balance between gainfully employing the compressive residual stresses to impede shallow surface flaws from growing, and aiding their propagation by driving the microcracks into the body of the material below the compressive residual stress layer, *i.e.* into the tensile zone beneath. However, understanding where the compromise is between these two conditions whilst maintaining cost-minimal grinding practices in industry is difficult, and probably material and machine specific.

The ability of a machined body to withstand further externally induced damage, such as from localised contact or indentation, leading to potential strength degradation, is an aspect that has not been studied in any detail. It is known that loading with a blunt indenter, such as a ball, produces a ring crack – often discontinuous in nature - which ‘pops-in’ a distance of a few micrometres [1,2,3]. Warren [4] successfully applied this to polished surfaces of glasses and fine-grained ceramics, and established a measurement of fracture toughness. In many cases, the pop-in distance is sufficient to reduce the residual strength of high-strength materials in which the critical Griffith flaw size has a similar value. There are practical limitations with this type of test.

Techniques for measuring residual stress are numerous. X-ray diffraction has commonly been employed, the peak shift obtained being related to the elastic modulus, and hence to the nominal stress in the surface. The difficulty with this is that the determined value is very much an average defined by the penetration depth of the X-rays at the particular diffraction angle used, and so is biased towards the immediate surface. Measurements using thin strip curvature have been demonstrated as capable of giving a total stress/depth integral effect, but it requires careful and tedious serial polishing to obtain depth profiles of residual stress. In contrast, non-destructively detecting the real damage in surfaces is difficult.

This report investigates two aspects of surface damage evaluation. Firstly, it reviews earlier unpublished data using the Hertzian loading technique to identify the potential for inducing

damage into conventionally machined surfaces of typical technical ceramics, as opposed to the polished surfaces of the ultrafine-grained ones used by Warren [4] and Roberts [5, 6].

2. HERTZIAN RING CRACK GENERATION

2.1 Background

When a sphere of radius R is pressed into a substrate under a force P , it is flattened, and the radius of contact a is given by [1, 2, 3]:

$$a = (3RP / 4E^*)^{1/3} \quad (1)$$

where E^* is the contact stiffness with a value given by:

$$1/E^* = (1 - \nu_1^2)/E_1 + (1 - \nu_2^2)/E_2 \quad (2)$$

where ν_1 , ν_2 and E_1 , E_2 are the Poisson's ratio and Young's modulus of the sphere and substrate respectively.

The peak pressure under the contact is:

$$p_{\max} = 3P / 2\pi a^2 = (3/2\pi)P^{1/3}(4E^*/3R)^{2/3} \quad (3)$$

The maximum tensile stress is radial along a ring just outside the radius of contact a , and it declines rapidly with distance from a and with depth into the material. It is this stress acting on surface flaws that initiates and propagates ring cracking. Warren [4] has shown that the fracture toughness of such flaws can be determined by establishing that it is related to the minimum force required to initiate cracking given by:

$$K_{Ic} = (E^* P_{\min} / CR)^{1/2} \quad (4)$$

where C is a dimensionless constant that depends (strongly) only on Poisson's ratio [5], and which has the value 3131 for glass on glass contact and 2790 for alumina on alumina contact. This technique has been promoted as a method for determination of the toughness originating from very small surface-connected flaws.

The application of this method has been extended to the case of residual surface stress. In this case the Hertzian contact stress is superimposed on the residual stress resulting from surface preparation, and this modifies the minimum Hertzian ring cracking force $P_{\min}$ [6].

Because any given surface has a wide distribution of sizes of surface flaws, a wide distribution of forces required to induce ring cracks will result from a series of Hertzian contact tests. An issue arising from the probabilistic nature of flaw distributions is that determination of the minimum force will be on a probabilistic basis. The more tests that are conducted, the lower will be the minimum force established. Nevertheless it provides a method for evaluating the proneness of different surfaces to damage by contacting objects.

The present work was conducted in order to gain experience with the technique, to determine what role the probabilistic nature of the flaw distribution has, and to ascertain whether consistent levels of toughness would be determined with different surface finishes.

2.2 Test equipment

The test facility used for this work was the CK10 instrument supplied on loan from the University of Oxford Materials Department through Engineering Systems (Nottingham) Ltd, the equipment manufacturer. Essentially, this instrument is a small compression tester with a flat test-bed equipped with an acoustic emission sensor which stops the crosshead and reverses it on detection of an acoustic event. It was designed specifically by the Oxford University research group of Dr S Roberts to perform the task of detecting ring cracking under Hertzian loading.

The instrument was equipped with various ball indenters of 2 mm and 5 mm diameter. All tests were performed with a nominal displacement rate of 0.5 mm/min.

It proved difficult in some cases to set the sensitivity of the acoustic detection to correlate with an obvious ring-cracking event. This appeared to be a problem mainly in coarse-grained alumina. It was suspected that crushing or microcracking of individual grains was relatively easily done compared with development of a continuous ring crack. It proved essential to ensure that the set detection level correlated with ring-crack production.

Table 1: Materials and surface conditions

Material	Source	Surface condition/process
Soda-lime glass	Float, unknown	As-received
		Annealed
SiC	Carborundum Hexolloy® SA	As-sintered, as-received
Alumina, 99.9%	Morgan, Vitox®	As-received, polished 0.25 µm
		As-received, annealed, polished 0.25 µm
Alumina, 95%	Morgan, Sintox® FA	Ground, polished
		Ground, annealed, polished 0.25 µm
Alumina, 88%	Royal Worcester, Regalox®	Ground, polished
		Ground, annealed, polished 0.25 µm
Sintered silicon nitride	Unknown	Ground, as-received
		Ground, polished, 6 µm
		Ground, polished, 0.25 µm
		Ground, vacuum annealed
		Ground, vacuum annealed, 6 µm polish
		Ground, vacuum annealed, 0.25 µm polished

2.3 Test materials

The variety of test materials shown in Table 1 were prepared as flat tiles with different surface finishes. Since ring-cracking is not visually obvious unless the surface is well-polished, the majority of surfaces were polished, either to a 6 μm diamond grit size finish or more often to 0.25 μm diamond finish, followed by relief polishing using OPS silica suspension.

2.4 Test results

Test outcomes were ranked according to the force at which the principal acoustic event occurred. These are shown as cumulative probability plots in Figures 1 to 6 in which the data are ranked in ascending value of force and assigned a probability of failure $P_f = (i - 0.5)/n$, where i is the rank and n is the total number of indentations. In most cases a minimum force threshold is seen, and is fairly clearly definable.

In the case of soda-lime float glass (Figure 1), the shapes of the distributions of fracture force are not particularly consistent. The minimum forces were 114 N and 140 N before and after annealing respectively, which indicates a change in the flaw distribution. As an experiment to see if friction affected the outcome, the test was repeated after lubricating the surface with petroleum jelly. The minimum forces were 86 N and 85 N for the as-received and annealed conditions respectively. Assuming $E = 70 \text{ GPa}$, $\nu = 0.29$ and $C = 3131$ [4], the average fracture toughness from these tests was $1.01 \text{ MPa m}^{1/2}$ for the unlubricated condition and $0.82 \text{ MPa m}^{1/2}$ for the lubricated condition, the latter being close to the recognised K_{Ic} figure of $0.70 \text{ MPa m}^{1/2}$ for glass.

In the case of sintered silicon carbide (Figure 2) there is a very clear and consistent distribution. The minimum fracture force with a 5 mm alumina ball was 75 N which corresponds with $K_{Ic} = 1.24 \text{ MPa m}^{1/2}$, assuming $E = 430 \text{ GPa}$, $\nu = 0.29$ and $C = 4280$ [4].

The testing of the 95% alumina material was particularly difficult, this is a relatively coarse-grained material, and appeared to suffer from acoustic noise during the early stages of loading, possibly due to the generation of internal microstructural damage rather than a localised ring crack. Similar microcracking behaviour has been seen in ball impact testing. Although the data appear to provide a reasonable shape of distribution both in the as-received and annealed conditions, there are uncertainties concerning the setting of the acoustic emission trigger thresholds in the test instrument. Assuming the results are genuine, and assuming values of $E = 350 \text{ GPa}$, $\nu = 0.25$ and $C = 3131$ [4], K_{Ic} is calculated as $1.49 \text{ MPa m}^{1/2}$. This is a relatively low value, and probably reflects the thresholding problem.

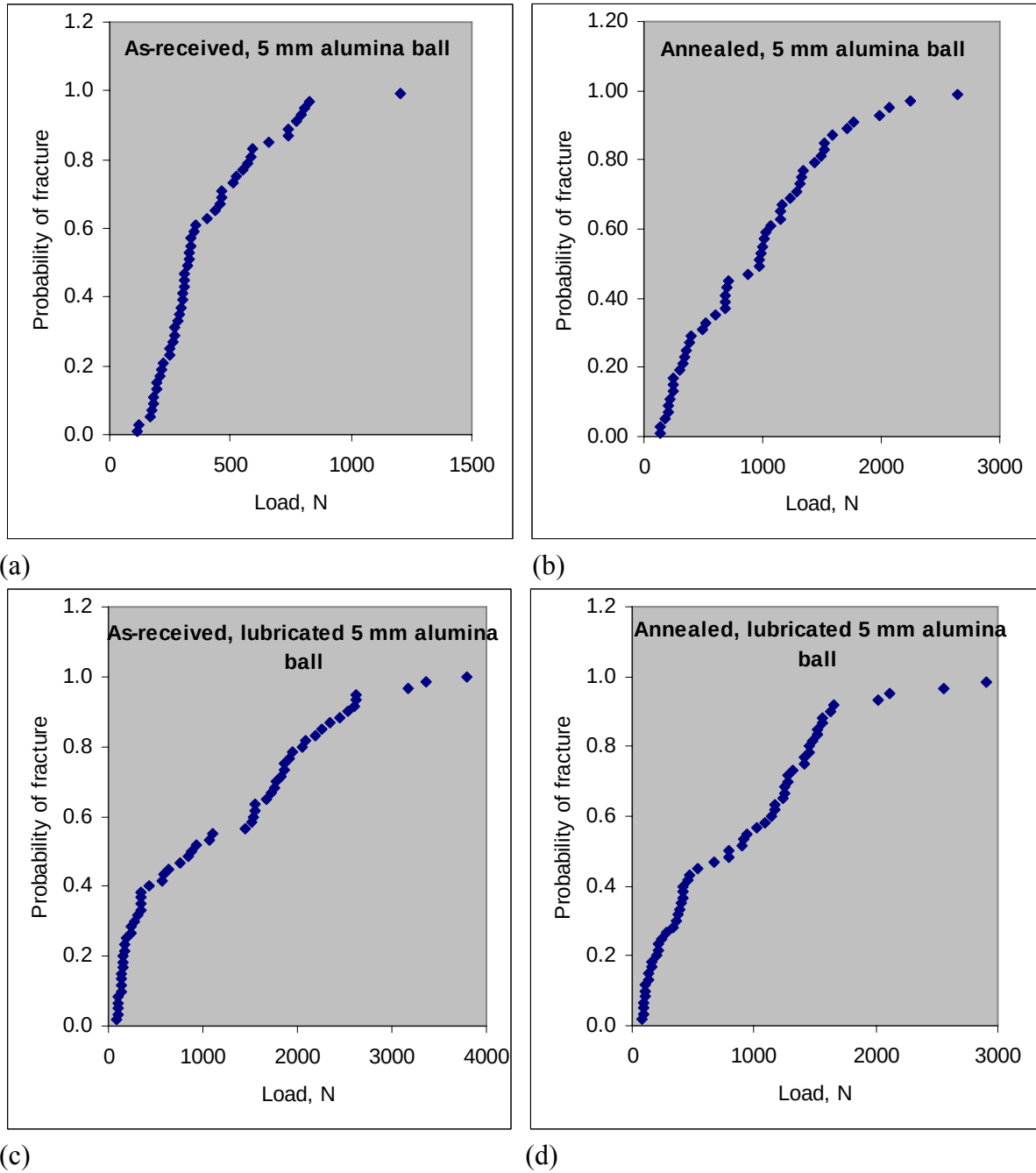


Figure 1: Hertzian indentation fracture initiation in float glass in the (a) as-received, (b) annealed, (c) as-received and lubricated, and (d) annealed and lubricated conditions.

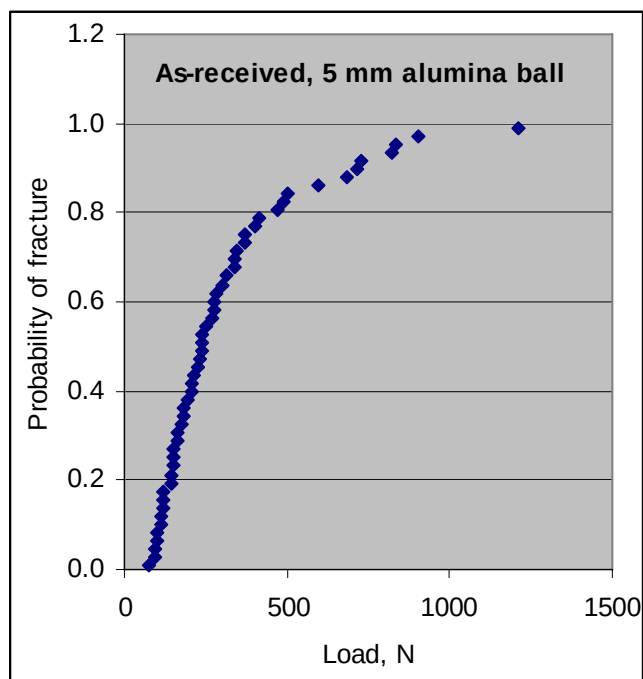
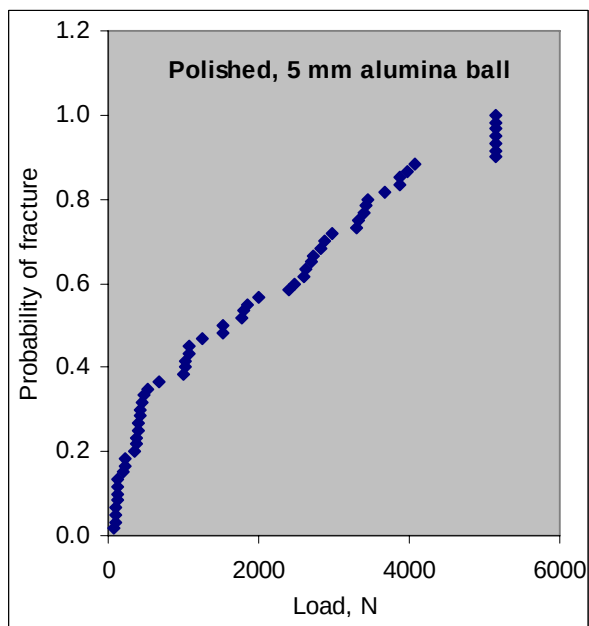
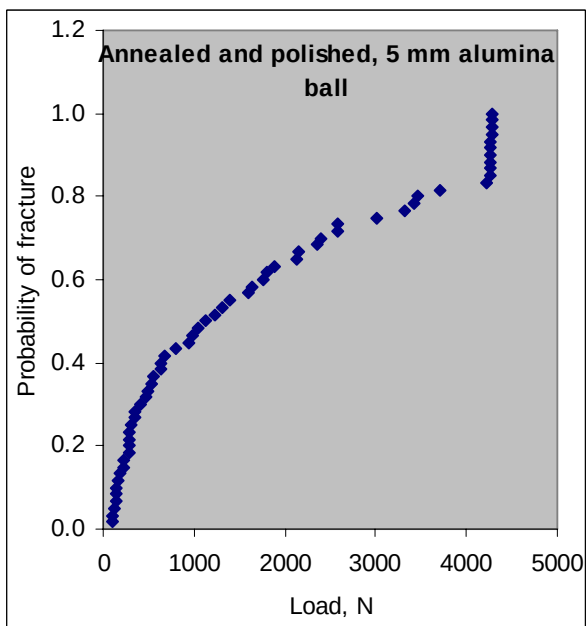


Figure 2: Hertzian indentation fracture in sintered silicon carbide in the as-fired, as-received condition.



(a)



(b)

Figure 3: Hertzian indentation fracture in 95% alumina in (a) as-received and polished condition and (b) annealed and polished condition.

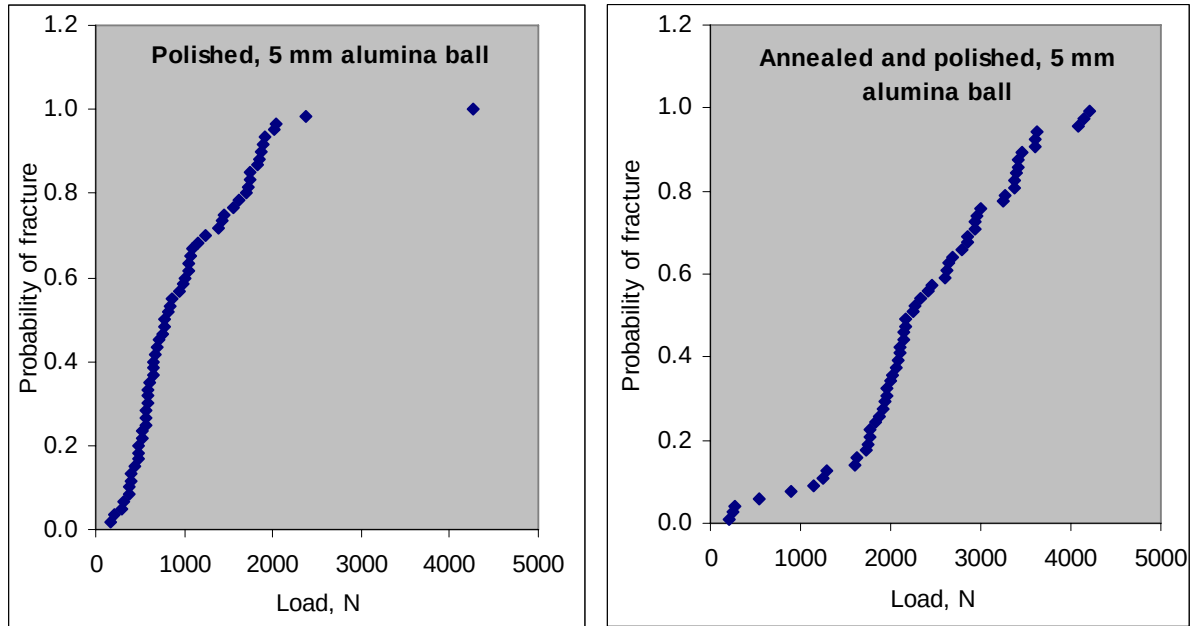


Figure 4: Hertzian indentation initiation in 88% alumina (a) ground and polished, (b) ground, annealed and polished.

Distribution data for the 88% alumina in the as-received/polished and annealed/polished conditions (Figure 4) seem to be very different for reasons which are not clear (Figure 4), although the threshold loads for the two conditions are rather similar at 164 and 208 N respectively. Assuming $E = 290$ GPa, $\nu = 0.25$, and $C = 3131$ [4], the average K_{Ic} is calculated as $2.04 \text{ MPa m}^{1/2}$.

The distributions for the 99.9% alumina were obtained using both 5 mm and 2 mm diameter alumina ball indenters (Figure 5). This material has a very fine-grained consistent pore-free microstructure compared with the other aluminas, and this is reflected in the consistency of the distribution shapes. However, the force threshold levels and overall ranges established are not consistent. There is a large difference between the values for the as-received/polished and the annealed /polished conditions with the 5 mm ball, while the difference for the 2 mm ball is small. There is currently no explanation for this. Using the 2 mm ball, the threshold forces were 73 N and 70 N respectively, giving an average K_{Ic} of $2.18 \text{ MPa m}^{1/2}$.

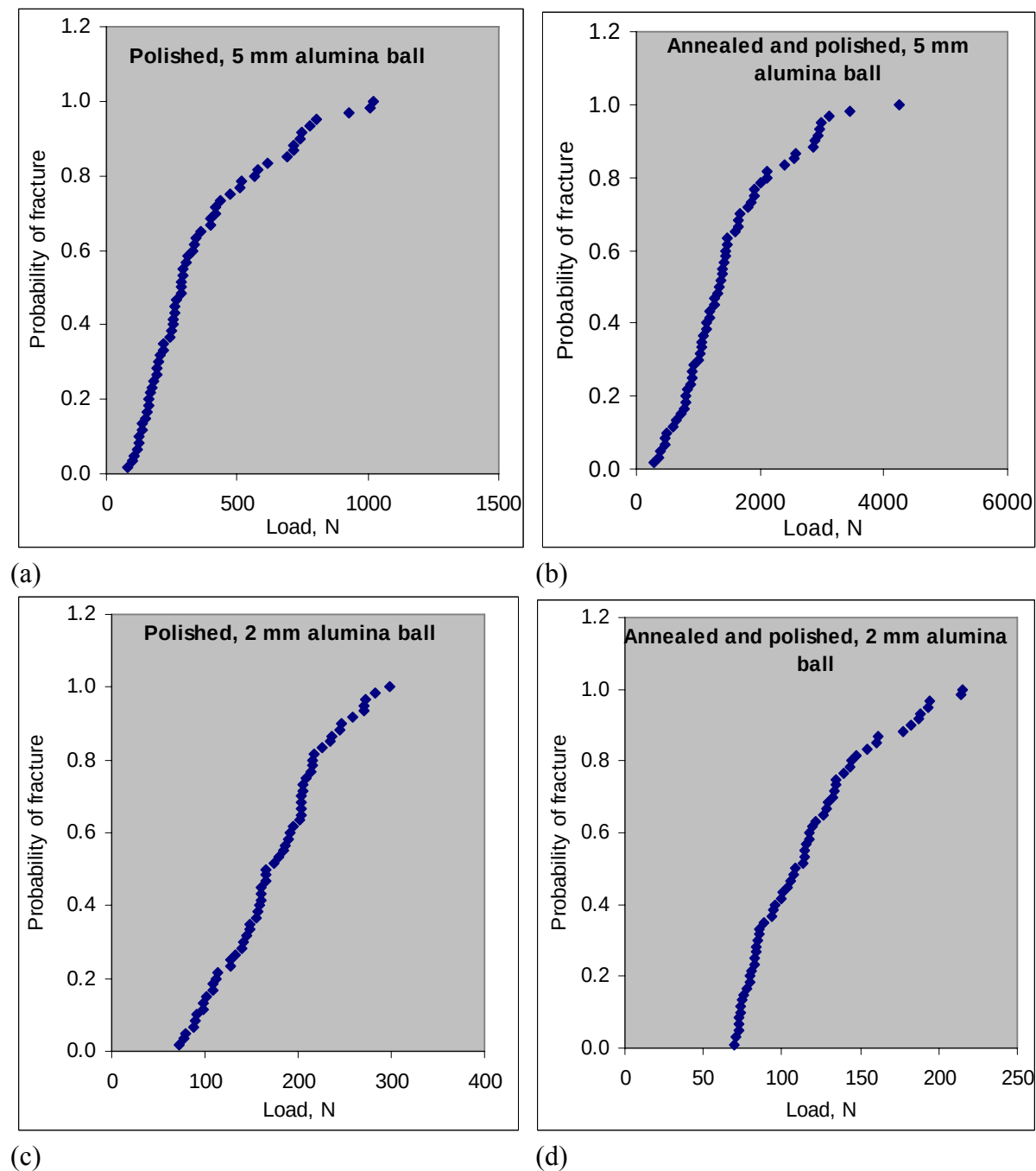


Figure 5: Hertzian indentation fracture in 99.9% alumina in (a) the as-received and polished, and (b) the annealed and polished conditions indented with a 5 mm alumina ball; (c) the as-received and polished, and (d) the annealed and polished conditions indented with a 2 mm alumina ball.

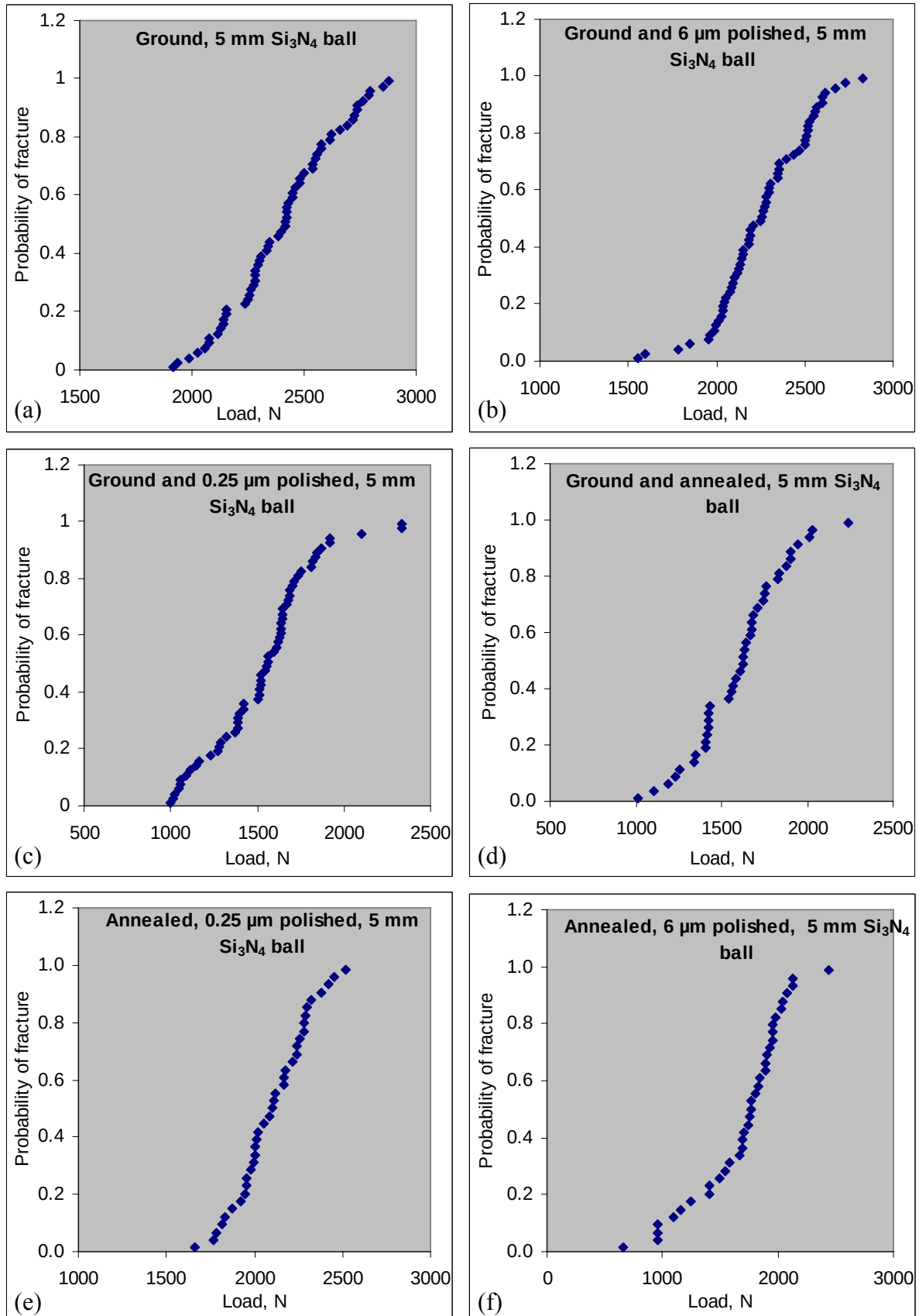


Figure 6: Hertzian indentation fracture in sintered silicon nitride in (a) ground surface, (b) ground and polished to 6 μm finish, (c) ground and polished to 0.25 μm finish, (d) ground and annealed, (e) and (f) as (b) and (c) but annealed before polishing.

Table 2: Hertzian indentation results for sintered silicon nitride

Condition	Threshold force, N	Assumed E , GPa	Assumed ν	Assumed C value [4]	K_{Ic} , MPa m ^{1/2}
Ground	1919	310	0.275	4280	5.74
Ground, 6 μ m polish	1558				5.17
Ground, 0.25 μ m polish	1001				4.14
Ground, annealed	1012				4.17
Ground, annealed, 6 μ m polish	659 (957)*				3.36 (4.05)*
Ground, annealed, 0.25 μ m polish	1663				5.34

* Second result, treating the first as an outlier.

The distributions for the three surface finishes in the sintered silicon nitride material with and without annealing of the as-received condition are shown in Figure 6. A summary of the threshold loads and the calculations of apparent fracture toughness is given in Table 1. It is immediately clear that this material is much more resistant to ring-cracking than any of the others. However, there are some inconsistencies in the results which are not fully explainable. Residual stresses in the ground, as-received condition may contribute to a raised threshold level, but this does not explain why vacuum annealing and fine polishing also produces a high threshold. The data may contain some outliers, notably the individual low threshold result for the ground/annealed/6 μ m polish condition.

2.5 Optical examination

Some of the ring cracks were examined in the optical microscope and their sizes measured and compared with estimates based on the contact diameter computed using equation 1. Generally there was a good correlation. However, within the timeframe available for this work, only a small number of ring cracks were evaluated in this way. One notable feature was the discontinuous nature of the cracks, often being only short crack arcs, not complete rings. It is probable that the inhomogeneity of the test materials on the scale of the ring crack diameters (0.3-0.5 mm) is a contributory factor.

2.6 Discussion

Despite the use of typically 60 indentations for each surface condition on each material, the ring-crack force distributions obtained are not particularly consistent. In part this may be due to the acoustic detection capability of the system on which the experimenter is reliant for identification of the ring-cracking initiation. As a consequence, it has not been possible to provide a clear demonstration that the extent of prior machining damage systematically changes the threshold force, nor that the existence of residual stress restricts the ability to ring-crack.

Another issue of concern is that the determination of apparent fracture toughness from the ring-cracking threshold is subject to the chance that an appropriate optimum size flaw is present at one of the sites of indentation. In some cases, the threshold seems quite well defined, with several data points defining it, and the probability distributions becoming near

vertical at this point. In other cases, the probability curves are more sigmoidal in shape, and in such cases pin-pointing the indenter force threshold is less reliable. This may be linked with material inhomogeneity.

2.7 Conclusions

An evaluation of the use of the Hertzian indentation technique with acoustic ring crack detection has been made on a variety of typical advanced technical ceramics, including silicon carbide, alumina and silicon nitride, compared with behaviour on a homogeneous material, float glass. It is concluded that:

- Using the CK10 instrument, the Hertzian ring-cracking test is apparently easy and quick to perform. However, a clearly defined threshold of detection is not always possible, notably in materials which suffer multiple localised microcracking. These materials typically have relatively coarse microstructures with grain sizes in the range 10-20 μm .
- The distributions of ring-crack loads determined are not especially consistent. In some cases a clearly defined threshold force can be determined, in others not.
- Most of the tests were performed on samples that had previously been polished, in order that ring cracks could more readily be seen. Prior residual damage from previous grinding operations may have influenced the results through the existence of surface cracks or residual stress. Annealing surfaces before polishing generally produced changes in the force threshold.
- Computations of fracture toughness from the ring cracking thresholds according to the procedure of Warren [4] produced data of approximately the correct order for float glass, but lower than typically reported values for most of the other materials. It may be the case that short-crack fracture toughness as determined in this test is lower for most materials than longer-crack toughness determined in typical conventional fracture toughness tests. The ranking of fracture toughness amongst the materials tested appeared to be broadly correct.
- Most of the tests were conducted with a ball that was dissimilar to the test surface. It is unclear how this should be managed in the calibration factor C which is sensitively determined by Poisson's ratio.
- More evaluation of actual ring crack diameters and their correlation with the elastic properties of ball and test surface needs to be undertaken.

3. SURFACE ACOUSTIC WAVE CHARACTERISATION

3.1 Introduction

Surface acoustic waves (SAW, or Rayleigh waves) are waves which travel along the surface of an object at a different speed to those propagating through the bulk. The phase velocity of this surface wave is given by:

$$c = \frac{0.87 + 1.12\nu}{1 + \nu} \sqrt{\frac{E}{2\rho(1 + \nu)}} \quad (5)$$

where ρ is the density. In contrast the faster, bulk-propagating compressional and transverse (shear) wave velocities are respectively given by:

$$v_l = \sqrt{\frac{E(2 - \nu)}{\rho(1 + \nu)(3 - 2\nu)}} \quad (6)$$

$$v_t = \sqrt{\frac{G}{\rho}} = \sqrt{\frac{E}{2\rho(1 + \nu)}} \quad (7)$$

The speed of Rayleigh waves is influenced by the nature of the surface, and in particular by interruptions or damage in the surface which causes them to deviate deeper. Their penetration depth decreases with increasing frequency. The surface properties affect the higher frequencies more strongly than the lower frequencies. The phase velocity of the surface wave thus depends on frequency, termed ‘dispersion’. A measure of the frequency dispersion provides information on the mechanical nature of the surface layer. If there is no dispersion, the surface is the same as the bulk. If the surface is mechanically damaged, the surface wave velocity is decreased with increasing frequency. If the surface is reinforced, *e.g.* has a high modulus coating, then the wave velocity increases with increasing frequency. Thus a measure of the phase velocity dispersion provides information on the integrity of the surface, and can be used to measure the thickness of a coating of different elastic properties.

The surface acoustic wave (SAW) instrument at NPL was devised and supplied by IWS, Dresden, Germany [7]. A UV laser is pulsed onto the surface to be investigated, and the resulting stress pulse is detected by a piezoelectric film sensor positioned a few mm away. By recording the received pulse shape as a function of distance from the pulse site, the dispersion of the phase velocity can be determined, and the elastic character and thickness of any surface layer, *e.g.* a coating, can be estimated.

This type of instrument is widely used within the electronics industry to estimate coating thickness and surface quality. It is known that grinding damage on single crystals, such as Si and Ge, can be characterised by the SAW methods. However, the instrument has not commonly been applied to typical engineering ceramic materials, and it is unclear whether it is feasible to detect localised damage using this tool. The present work was conducted to evaluate the potential of the SAW technique to discriminate differing levels of machining damage in advanced technical ceramics, and to identify whether individual large cracks could be detected.

3.2 Preliminary evaluation

Some preliminary trials were conducted to investigate whether surface acoustic waves could be generated by the system. As-fired discs 35 mm diameter and 2 mm thick of Vitox® (99.9% alumina) and Zyranox® (ZrO₂ with 3 mol% Y₂O₃, so-called 3Y-TZP) supplied by Morgan Advanced Ceramics Ltd, Rugby, were employed.

One of each type of disc was placed in turn on the bed of the SAW apparatus, and the focal site of the laser suitably positioned. The piezo-foil (PVDF) sensor was placed adjacent to the focal site, and the knife-edge contact lowered to force it against the test surface. The laser was regularly pulsed, and attempts were made to detect the surface acoustic wave at a distance of 5 mm. Despite several attempts, running through the laser line focus position, no signal above background could be detected. It was concluded that the problem lay principally with the fact that both of these ceramics must have been sufficiently optically transparent at the laser wavelength and energy (337 nm, 400 mJ, 0.5 ns pulse) to have scattered rather than absorbed the radiation, thus not developing a sufficient mechanical pulse to allow the surface propagation to be detected.

3.3 Systematic evaluation

3.3.1 Test-piece preparation

Each of the test materials was prepared in three surface finishes:

- As fired;
- Heavily machined using a single 20 µm final cut on a surface grinder using a 180 grit bronze-bonded diamond wheel rotating at 2880 rpm giving a surface speed of 1550 m/min, with 1 mm step lateral rasters across the surface; the lubricant was a light machining oil;
- Lightly machined, using a final sequence of cuts of 10 µm, 5 µm, 2 µm, 1 µm, 0 µm, 0 µm, using a 320 grit wheel operating as above.

The surfaces were cleaned of residual machining oil by baking at 400 °C in an oven for 1 h.

One quadrant of the test surface of each test-piece was coated with a ~200 nm layer of sputtered 60Au-40Pd to provide a target for absorbing the laser radiation in an attempt to overcome the transparency problem.

Adjacent to the corner of the quadrant (Figure 7), a row of eight Vickers indentations was made at a force of 49.6 N (5 kgf), spaced approximately 0.2 mm apart. The indentations were lined up such that the radial cracks emanating from the indentation corners would nearly overlap and line up to form an extended surface flaw.

This arrangement permitted tests to be performed within the area of coating ((1) in Figure 7) and from the area of coating to the uncoated region, with (3) or without (2) the extended crack feature being in the path of the pulse. The directionality of the surface damage could also be investigated. Before testing, surfaces were re-cleaned with isopropanol.

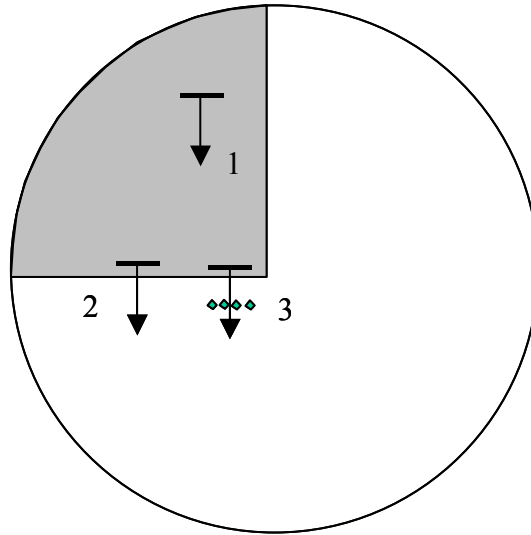


Figure 7: Layout of test-pieces with one quadrant coated with Au-Pd, arrows indicate SAW direction from line laser source to the detector location; (1) test with coating, (2) test off coating, (3) test off coating across additional indentation damage zone.

3.3.2 Tests performed and results

Initial trials were made on the coated area of each test-piece to evaluate the instrument response compared with previous experience. It was found that the coating was transiently successful in creating pulses large enough to detect, but was soon spalled away unless precautions were taken to reduce the intensity of the laser through the use of neutral density filters.

Series of tests were then made on the as-fired (minimal mechanical damage, but rough) surfaces and the heavily machined surfaces. When the evaluation was performed entirely on the coated region of each test-piece, and the elastic parameters associated with both the coating and the ceramic material were input into the software, the best fit thickness determined for the coating was consistently and appropriately ~200 nm.

Figure 8 shows the dispersion curves for two experiments on the as-fired alumina surface within the coated region. There is a moderate degree of dispersion suggesting a lower stiffness surface, which would accord with the presence of the coating and of a relatively rough surface. The slopes of the fitted lines are near parallel and, using a least-squares linear fit to the entire frequency range, similar intercepts with the ordinate were obtained, indicating similar surface acoustic wave velocities averaging 5846 m/s. Assuming a Poisson's ratio of 0.275 and a density of 3.95 Mg m^{-3} a bulk Young's modulus of 403 GPa is computed, very close to the expected value for this type of fine-grained high-density polycrystalline product.

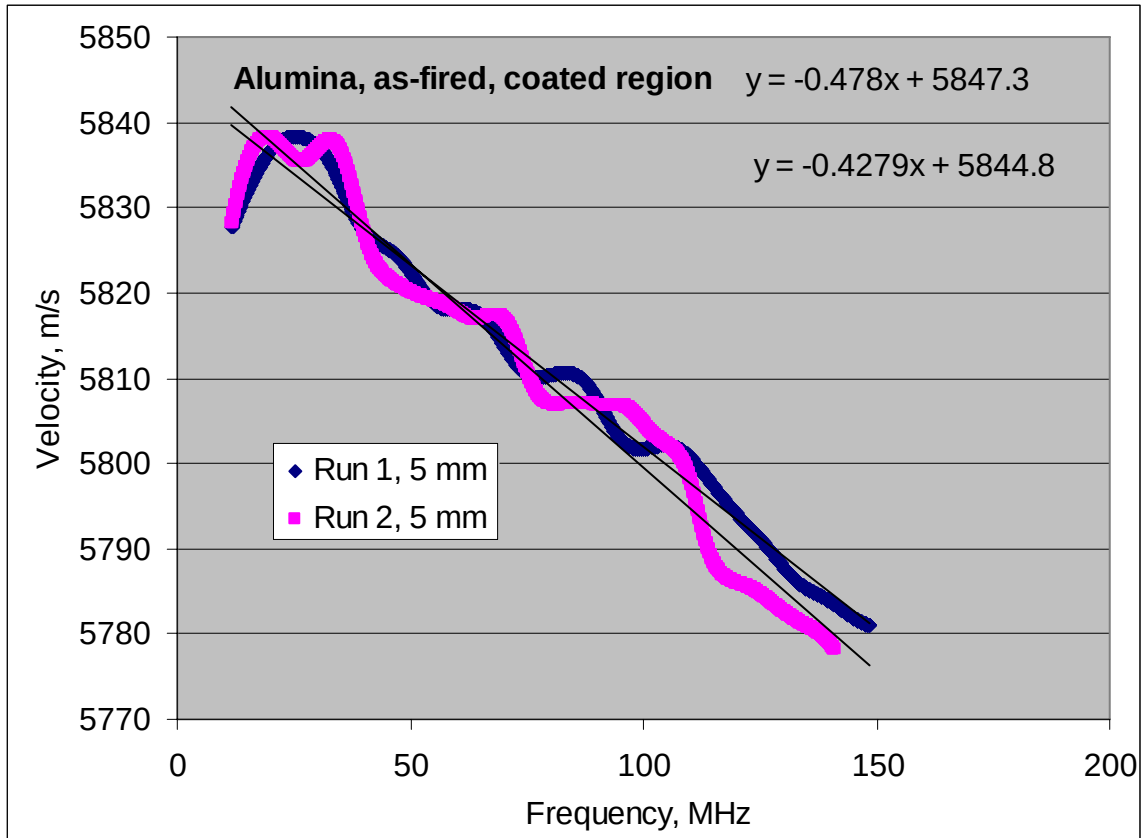


Figure 8: SAW dispersion diagram for as-fired and coated Vitox® alumina.

It can be seen that the slope of the dispersion curve is sensitive to the region in which the linear fit is made, but that the two curves essentially overlap, so the difference between them cannot at this stage be taken as significant.

In the tests with the laser striking near the edge of the coating and the detector in the uncoated region, the results shown in Figure 9 were obtained. In this case there is more deviation from straight-line behaviour, but the SAW wave velocity determined at the intercept is almost identical to that from the tests wholly within the coated region. The fitted linear slopes of the dispersion curves are similar also, suggesting that what is seen is a result of near-surface differences in behaviour.

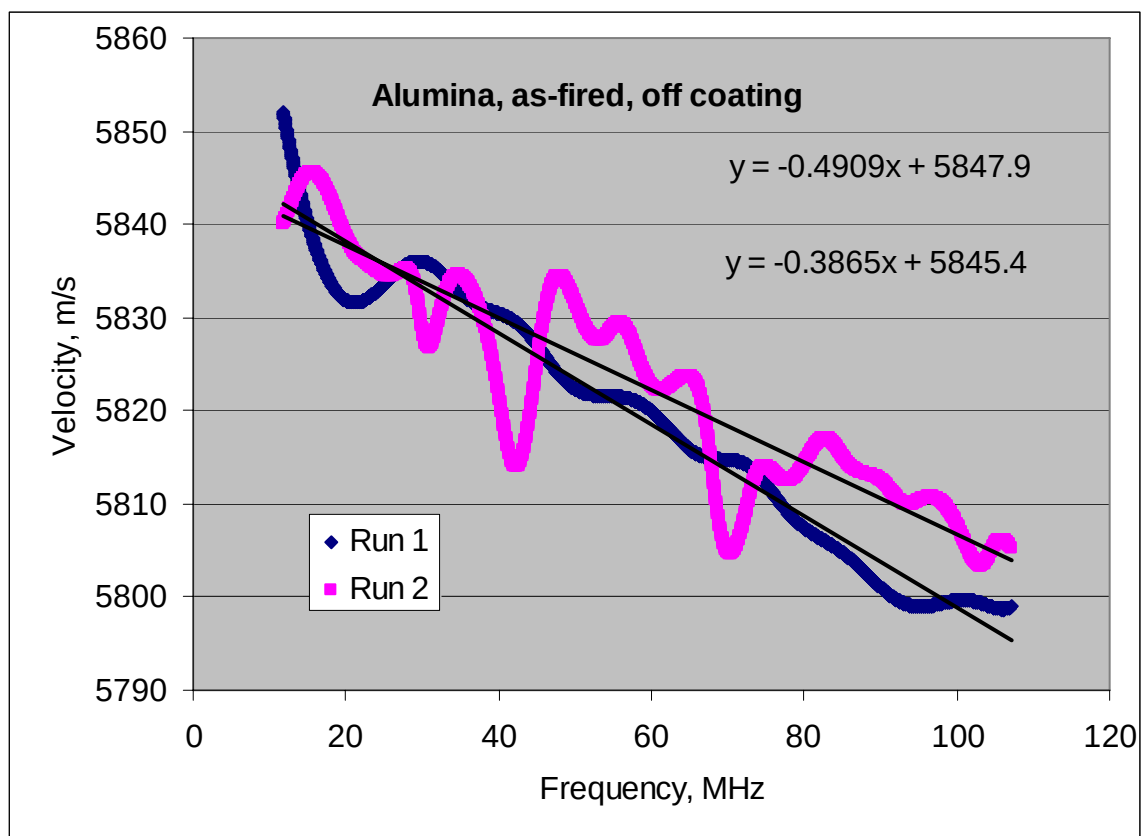


Figure 9: SAW dispersion diagram for as-fired alumina in the coating-free region.

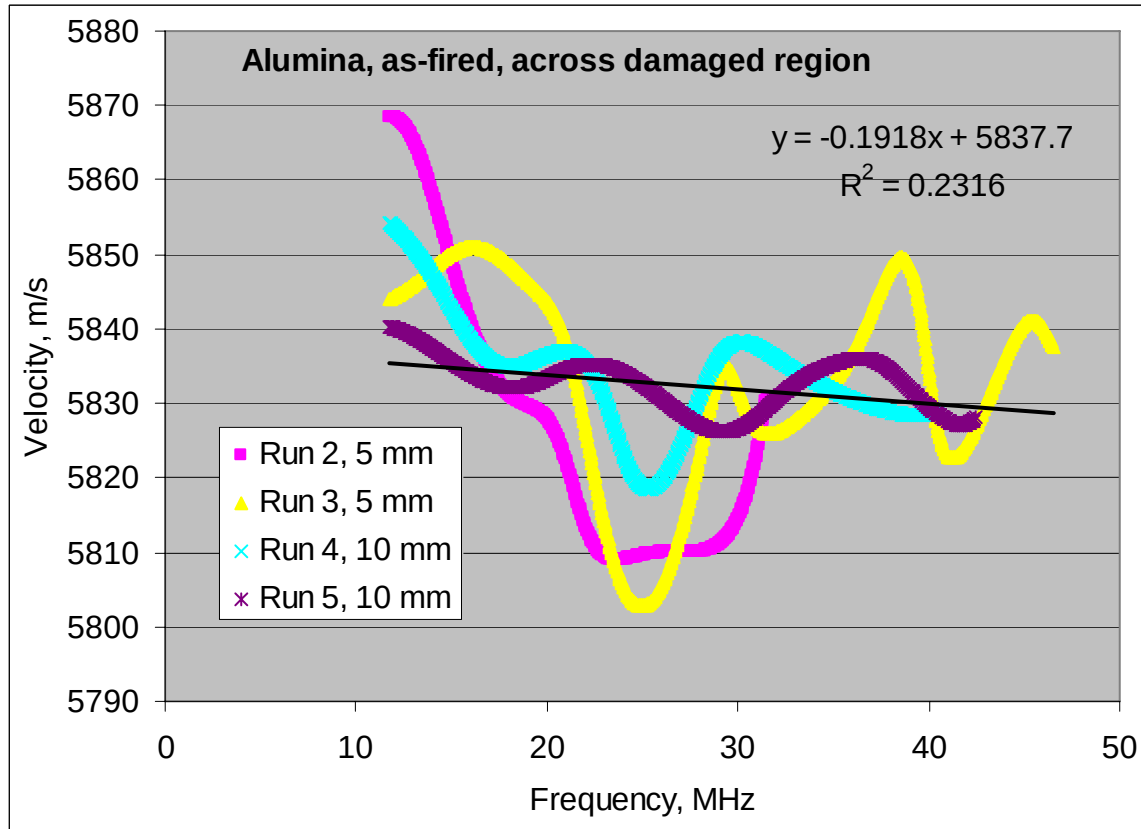


Figure 10: SAW dispersion diagram for as-fired alumina across the indented region showing very variable behaviour.

In tests in which the detection path includes the line of indentations, the data in Figure 10 were produced using two ranges of distance. It is clear that there is much less certainty concerning detected response, which has given widely non-linear behaviour. It is clear that there is significant disruption to the surface acoustic wave in this case. The extrapolated intercept of the straight-line fit to the straightest of the dispersions gives a very similar velocity and Young's modulus value.

Equivalent experiments using the severely ground surface are shown in Figures 11 to 13. Although all these tests show similar outcomes for the surface acoustic wave velocity, and hence for the apparent modulus, there is not only variability in outcome, but also no convincing differences in behaviour between the different conditions.

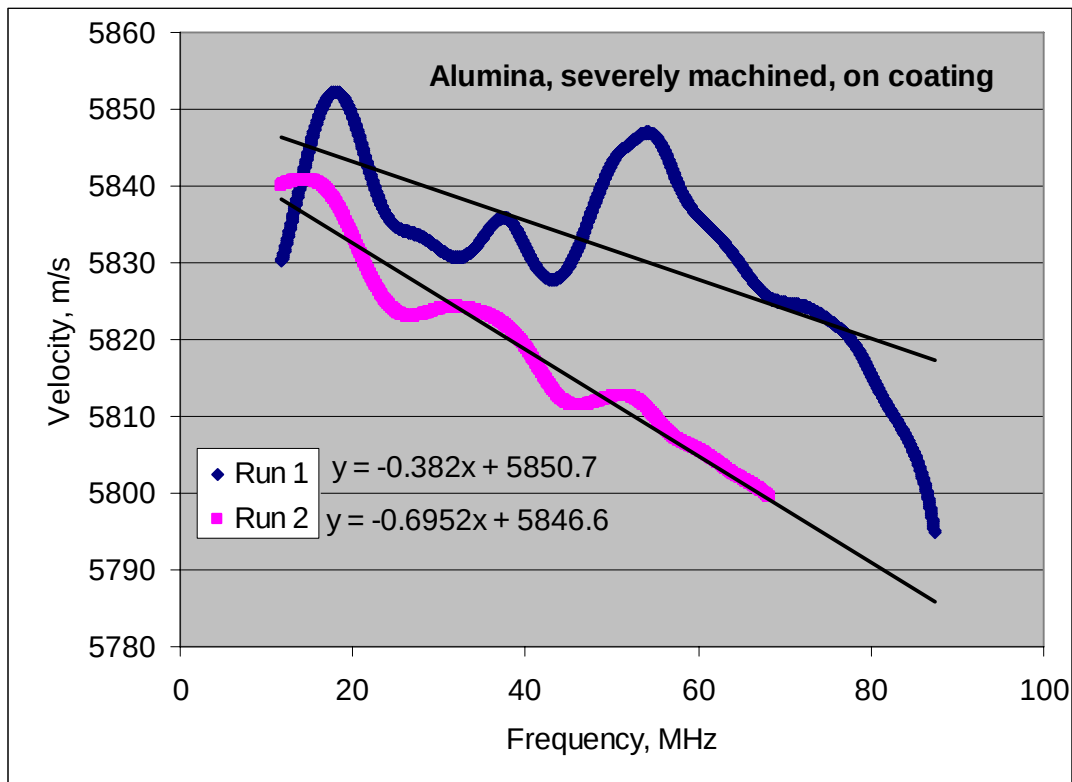


Figure 11: SAW dispersion diagram for severely machined Vitox® alumina pulsed on the coating.

Figure 14 summarises attempts to measure the equivalent behaviour in the Zyranox zirconia. This material proved more difficult. The detected pulse was weak and difficult to obtain, and was restricted to a smaller frequency range. The coating was ablated more quickly, probably because of the low thermal conductivity permitting the pulse temperature to be significantly higher than in the case of alumina. The mean surface acoustic wave velocity was approximately 3410 m/s which computes to 210 GPa, an appropriate value for the type of material with density 6.1 Mg m⁻³ and Poisson's ratio approximately 0.26. It is unclear whether the additional damage is being detected or not.

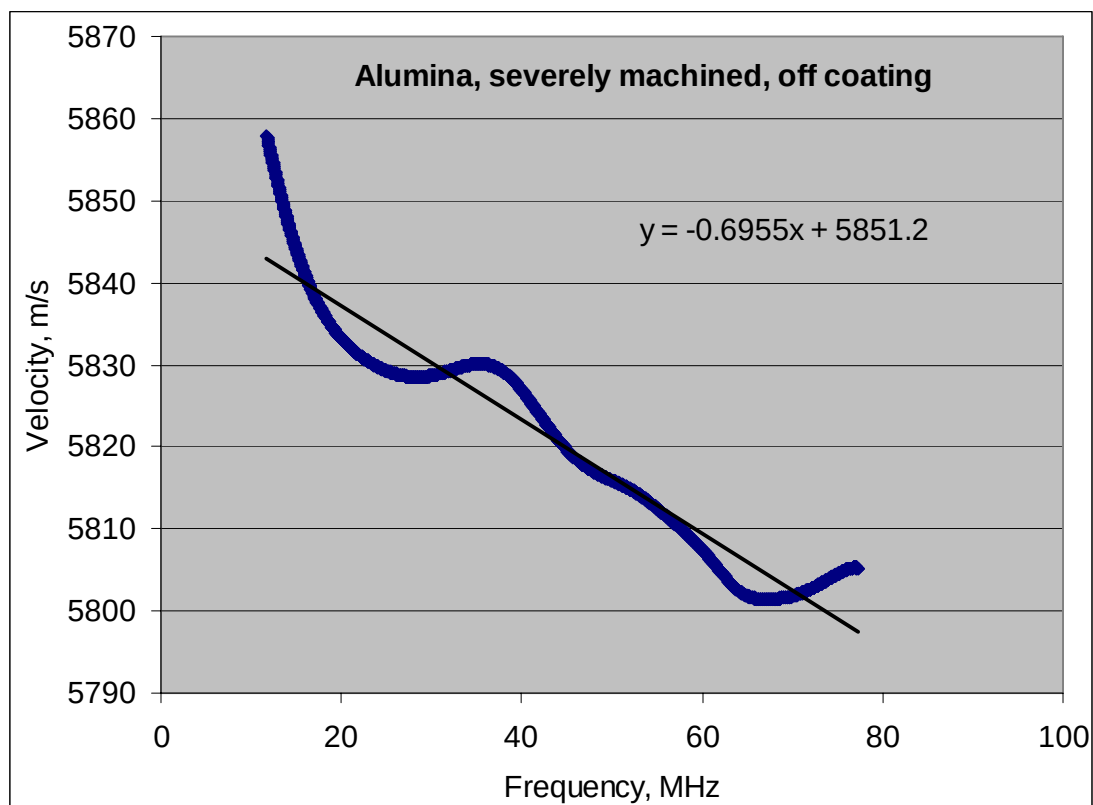


Figure 12: SAW dispersion diagram for severely machined Vitox® alumina pulsed on the coating but with pulse detection off the coating.

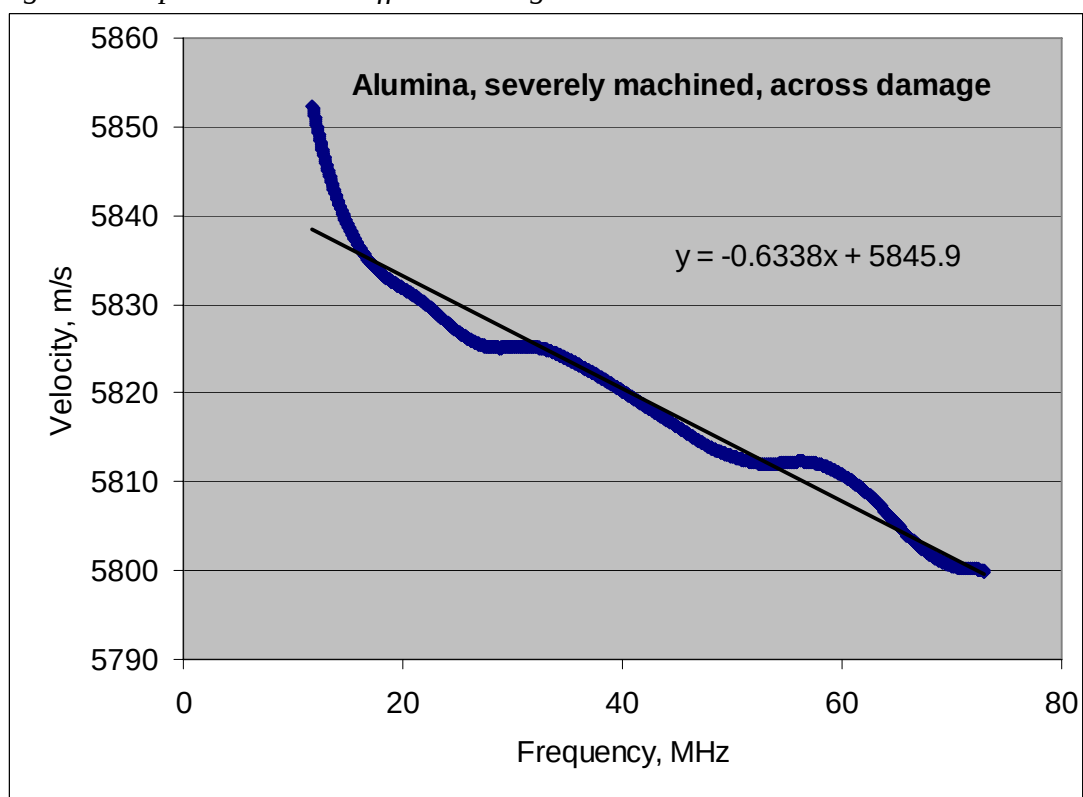


Figure 13: SAW dispersion diagram for severely machined and indented Vitox® alumina pulsed on the coating but with pulse detection off the coating after the indentation damage.

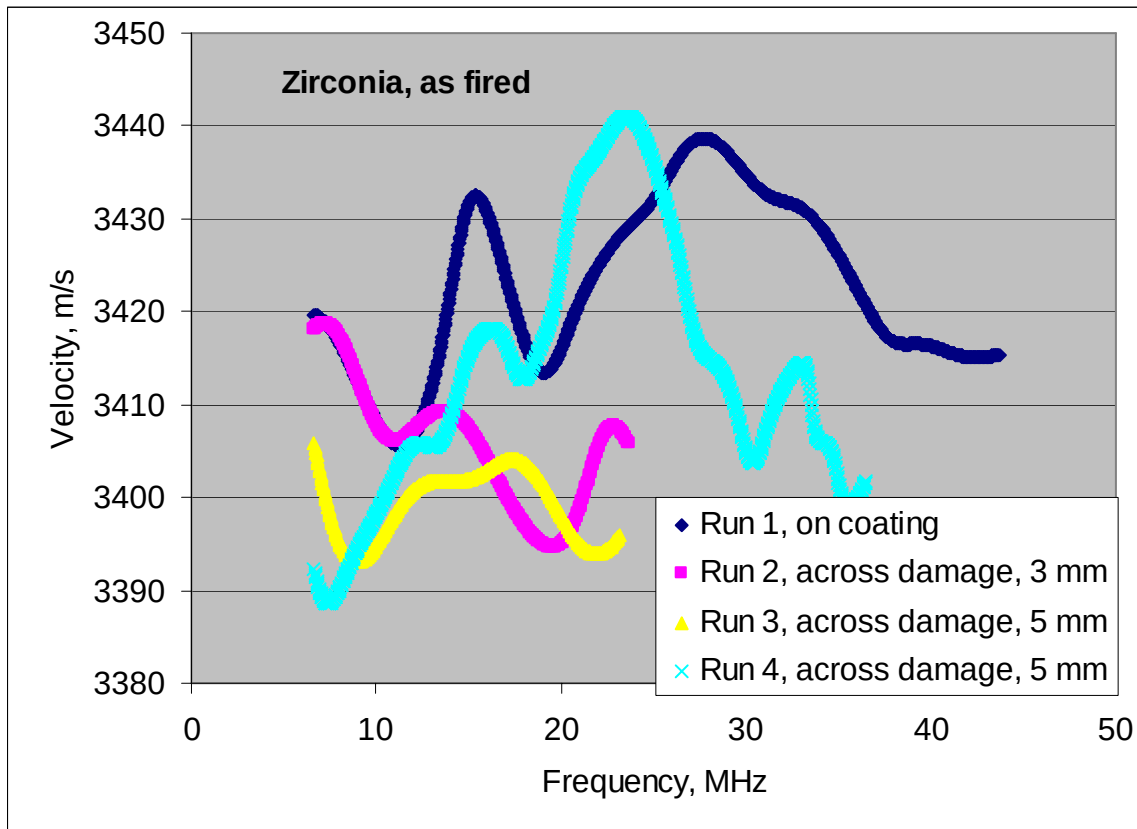


Figure 14: SAW dispersion diagram for as-fired Zyranox® zirconia pulsed on the coating and off the coating across the indentation damage.

3.4 Discussion

It is clear from the experimental results that although surface pulse transmission and detection in translucent ceramics is made possible by the use of a coating to intercept to the laser pulse and provide a means of inducing a localised stress pulse, it is not a reliable method in the sense that coating ablation takes place, and this leads to a lack of repeatability of outcome of the analysis. There is therefore poor discrimination of the effects of applying the coating or of the nature of the ceramic surface. The presence of the additional deep damage in the form of the extended flaw produced by the line of Vickers indentations did seem to reduce the pulse transmission significantly, as might be expected, but also introduced additional variability and lack of discrimination.

A further factor is the topography of the surface. Most published evaluations have been made on relatively smooth polished surfaces, with or without thin flat coatings. In hindsight, it may well have been better to commence the present studies on flat polished and annealed surfaces to provide a baseline, rather than on relatively rough as-fired and machined surfaces.

It also remains unclear as to what extent the experimental practice affects the reliability of the results. When tests are made on conducting metallic materials, the outcome seems to be more consistent, and it is thought that this may be related to the ability to maintain a good electrical earth on the test-piece to stabilise the behaviour of the piezo-foil sensor. This is of course impossible on an insulating ceramic, and it remains unclear whether the application of aluminium foil between the sensor and the test-piece is a sufficient mechanism to provide this stability.

3.5 Conclusions

SAW measurements on advanced technical ceramics, high-purity alumina and 3Y-TZP zirconia with technically prepared surfaces have been found not to be straightforward. It has been found that:

- A sputtered coating is required in order to provide a target for the laser pulse and to avoid scattering of the input energy as a result of UV translucency.
- The signal to noise ratio of the received SAW pulse at the piezo-sensor is poor compared with the response on polished surfaces of metals.
- An electrically earthed layer needs to be placed under the piezo-sensor in order to stabilise the received signal.
- The surface wave velocity extrapolated to zero frequency correlates with the correct value of Young's modulus of the substrate.
- The frequency dispersion of the surface waves, used as a measure of the nature of the surface layer, has poor consistency, and does not readily correlate with the degree of damage. Further more systematic tests would be needed using a stress-free polished surface as a baseline rather than an as-fired surface with significant roughness.
- The introduction of additional damage in the form of a line of Vickers indentations with associated radial cracks between the laser pulse target and the detector produced absorption or scattering of the surface acoustic waves and reduced the ability to properly resolve dispersion. Thus an influence could be seen, but could not be quantified.

Acknowledgements

Morgan Advanced Ceramics Ltd (Steve Hughes) is thanked for supply of characterised test materials for this work.

References

1. Hertz, H., "On the elastic contact of solids", *Z. Reine Angew. Math.*, 1881, **92**, 156-71.
2. Frank, F.C., Lawn, B.R., "On the theory of Hertzian fracture", *Proc. Roy. Soc. Lond.*, 1967, A229, 291-306.
3. Warren, R., "Measurement of the fracture properties of brittle solids by Hertzian indentation", *Acta Metall.*, 1978, **26**, 1759-69.
4. Warren, P.D., "Determining the fracture toughness of brittle materials by Hertzian indentation", *J. Eur. Ceram. Soc.*, 1995, **15**, 201-7.
5. Warren, S.G. "Hertzian testing of ceramics", *Brit. Ceram. Trans.*, 2000, **99**(1), 31-8.
6. Bisrat, Y., Roberts, S.G., "Residual stress measurement by Hertzian indentation", *Mater. Sci. Eng.*, 2000, **A288**, 148-53
7. Schneider, D, Schwarz, T., *Laser acoustic thin film analyzer handbook*, v5.02, Fraunhofer Institute for Material und Beam Technology (IWS), Dresden, Germany.