

Measurement Note

Scratch edge chipping

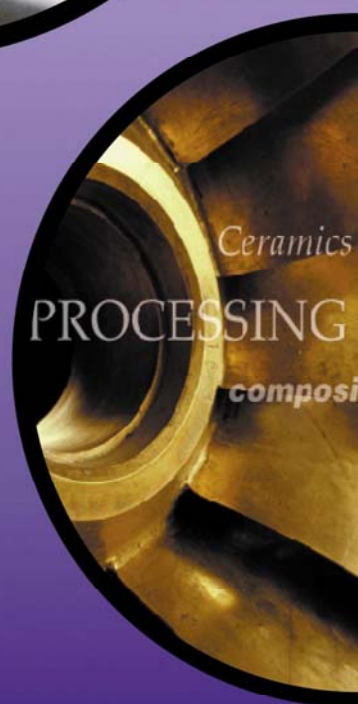
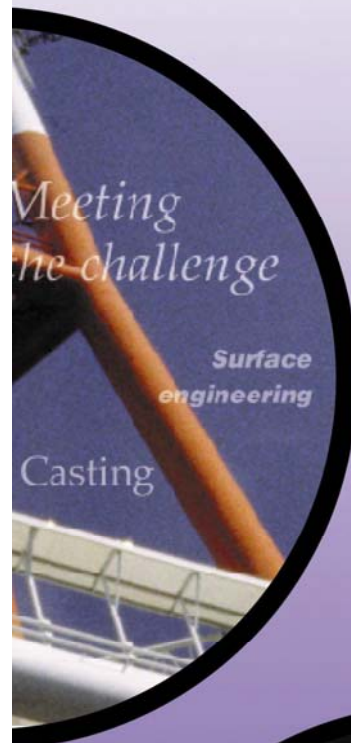
ABSTRACT

Scratch testing is a well-known technique for determining the resistance of surfaces to damage by hard localised contacts, such as abrasive particles. Less well-known is the extension of the test to the development of edge flakes in brittle materials. This measurement provides results of scratch edge chipping tests on a variety of ceramics, and compares the results with other tests designed to determine resistance to edge damage. The findings do not directly confirm preliminary evaluations by other researchers, and further work needs to be undertaken to understand the role of test system behaviour to avoid secondary damage to the edge.

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INTRODUCTION

The edges of brittle materials are prone to damage by localised contacts or impacts, and for this reason, components are usually chamfered or rounded to minimise the risks. In order to assess the resistance of an edge to damage, a controlled test method is needed. There are three simple ways to induce damage to an edge in a brittle material, by loading close to the edge using a hard sharp or blunt indenter [1-4], by impacting close to the edge with a similar indenter [5], or by dragging an indenter across the surface towards the edge until the edge gives way [6, 7]. In all three cases, an edge flake is formed. In the case of normal near edge indentation it has been found experimentally that the greater the distance of indentation from the edge, the greater the force required to form an edge flake. A similar situation occurs in impacting near the edge, the greater the distance of the impact site from the edge, the greater is the kinetic energy required to cause a flake to form. However, the situation is different with scratching towards an edge, because in this case the normal force is constant and the scratching action develops a significant lateral frictional force that is absent in normal indentation. It is therefore likely that it is the combination of the two forces that causes the edge to give way as the indenter approaches.

This Measurement Note describes experiments to investigate the utility of scratch edge chipping on a variety of advanced technical ceramics.

EXPERIMENTAL

Method

An instrumented scratch tester that has been described elsewhere [8] was employed. It was equipped with a Rockwell polycrystalline diamond (tip radius nominally 0.2 mm) to minimise risks of damage to the indenter. The scratch tester is programmed to make ten scratches separated by 1.5 mm towards the test-piece edge at a series of increasing normal force levels. This separation was mostly sufficient to avoid flake overlap. A sliding speed of 1 mm/s was used.

One of the principal problems to overcome was the avoidance of ancillary damage as the indenter drops over the edge. The most successful method was the use of a protective shoe around the indenter which touched down on the top of the test-piece as the indenter dropped during flake formation (Figure 1). However, this proved to be difficult to adjust to precisely the correct height relative to the indenter tip.

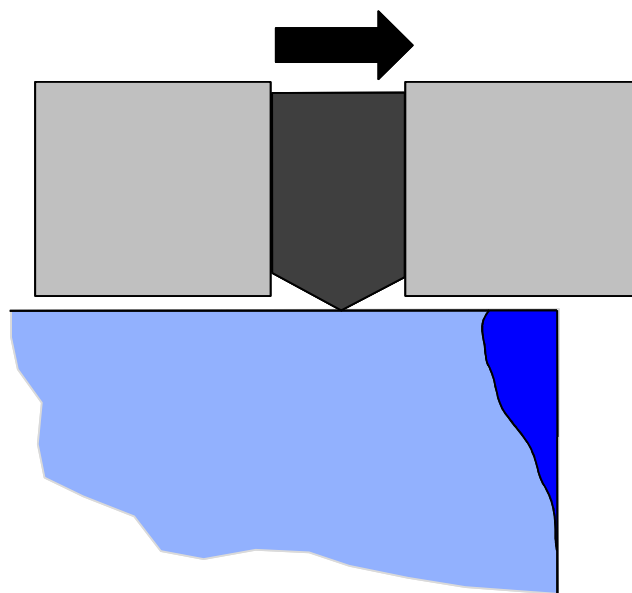


Figure 1: Schematic of loaded Rockwell indenter sliding towards an edge and producing a flake. The unit around the indenter is a shoe intended to minimise secondary edge damage.

The test sample was examined after scratching using a Nikon measuring microscope, and the following measurements were made:

- Scratch width;
- Distance from the edge of flake initiation measured on the centre-line of the scratch;
- Maximum flake width on side face;
- Maximum flake depth on side face.

Test materials

A series of nine commercial ceramic materials of different compositional types and grain sizes was selected, plus soda-lime glass. The scratch face was prepared by lapping using 3 μm diamond paste. The edge face was prepared by diamond sawing and hand lapping on a 320 grit diamond lapping disc, followed by 600 grit SiC paper. Best attempts were made to preserve the sharpness and squareness of the actual edge, although typically this was limited by the grain size of the material and the time spent in preparing the side face.

RESULTS

Preliminary experiments

These were conducted on yttria-TZP and soda-lime glass. Figure 1 shows a scratch made at 15 N indenter force towards an edge in soda-lime glass. There is a clear pattern of minor damage along the

sides of the scratch, but no major chipping damage until the edge is reached. Figure 2 shows a flake made by scratch edge chipping on soda-lime glass at 15 N indenter force. A symmetrical shell-shaped flake is formed, the ripple features of which are produced by acoustic noise of the chipping process. Note that the flake fractured leaving a tongue of material at the lower edge. This is

presumably caused by bending forces applied to the flake by the indenter continuing to move over the edge. In some other cases, it was noticeable that the developing flake undercut the top surface, similar to that seen previously in impact edge chipping [5], and probably a result of Hertzian type fracture initiation.

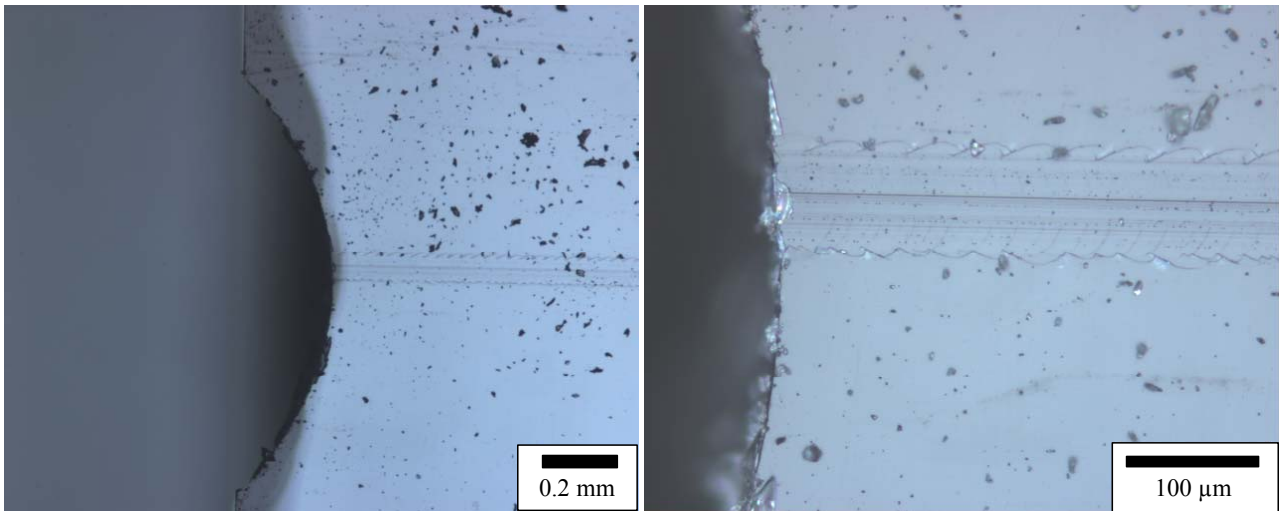


Figure 1: Scratch to the edge and flake formation in soda-lime glass at an indenter force of 15 N.

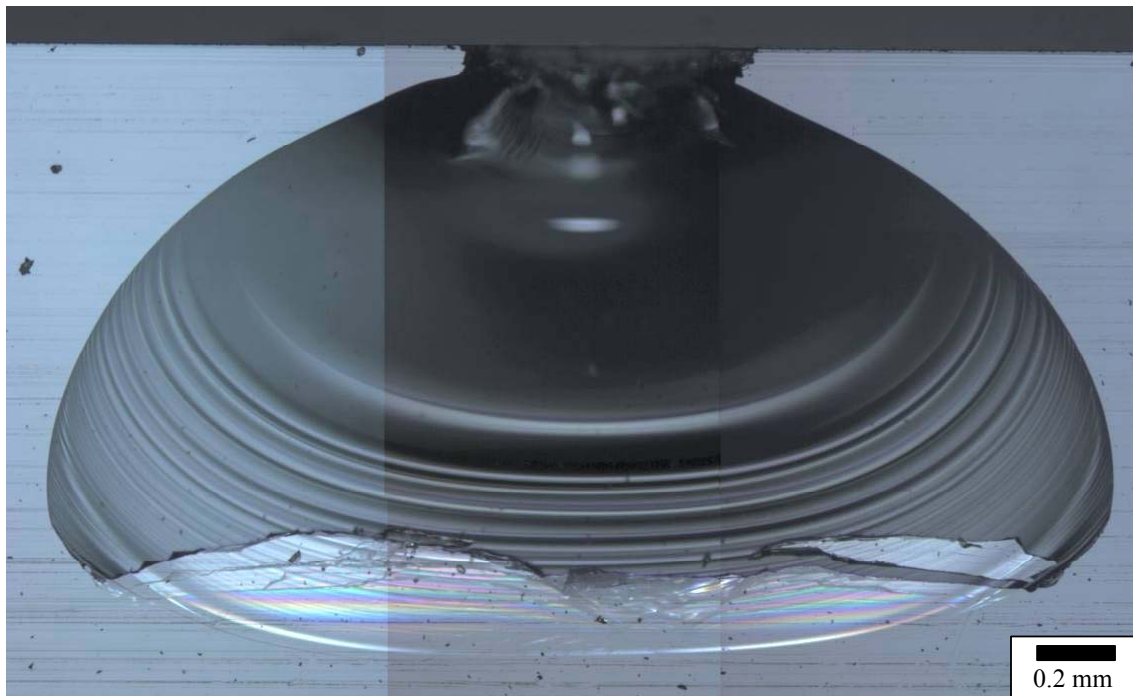


Figure 2: Photomontage of a flake made in soda-lime glass by scratch chipping at 15 N force.

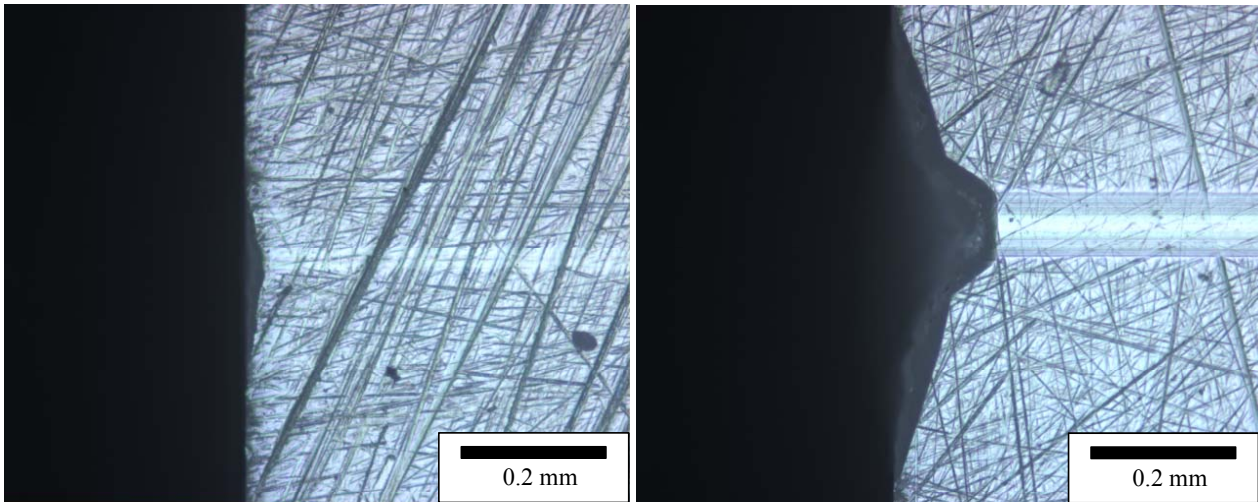


Figure 3: Scratches in Y-TZP at (left) 5 N, and (right) 70 N indenter force.

Figure 3 shows scratches in Y-TZP at 5 N and 70 N indenter force. Again, there is mostly plastic marking of the surface with no obvious cracking, and prior surface markings are still visible under the scratch. However, unlike the glass scratches, the formation of the flake is different, more noticeably at higher force levels. The appearance of the damage suggests that there may have been significant ‘follow-through’ of the indenter once the flake is formed, causing secondary damage and making the flake thickness measurement uncertain. It is this aspect of scratch edge chipping that creates the most uncertainty.

Figure 4 shows a plot of nominal flake thickness vs. indenter force. For this plot, the flake thickness has been taken as the distance of the end of the scratch track from the original edge, ignoring undercut in the glass and the possibility of follow-through in the Y-TZP. Although there is some scatter, there are broadly linear relationships between these parameters, similar to that found for edge chipping by quasistatic normal edge loading. There is a significant difference between the gradients of the relationships, indicating the much greater resistance to flaking of Y-TZP compared with soda-lime glass.

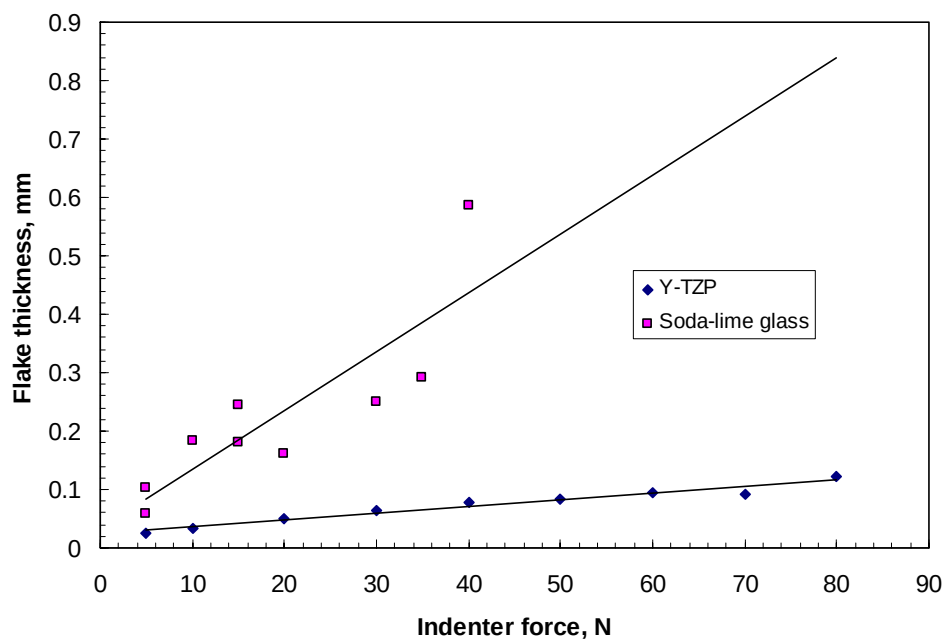


Figure 4: Nominal flake thickness as a function of indenter force.

Scratch width analysis

Figure 5 shows the results of scratch width measurements on a range of commercial ceramic materials. There is a general linear trend of increasing width with indenter normal force. The widest scratches at a given indenter force occur, somewhat surprisingly, with sintered silicon carbide and the 99.5% alumina, and the narrowest with Y-TZP and sintered reaction-bonded silicon nitride (SRBSN). This material ranking is clearly not in accordance with expectations based on conventional hardness data.

Figure 6 shows an example of scratches made in sintered silicon carbide. In contrast to Figures 1 and 3, the traces show no plastic deformation, but indicate that much chipping and fracturing has occurred. There are also many side cracks, in one case with a significant flake-like propagation distance.

In Figure 7, the scratch widths have been converted into a scratch hardness using the formula

given in ASTM G171 which assumes that the indenter force, P , is borne by a half-cap of material of diameter equal to the scratch width, w :

$$H_s = \frac{8P}{\pi w^2}$$

For plastic materials, scratch hardness is related to the plastic properties of the material on the assumption that the track width supports the stress generated by the indenter, and one would expect the value to be independent of applied force. This assumption seems to apply reasonably well over most of the force range with Y-TZP and SRBSN from the scratch appearance, but with the remaining materials, there is a tendency for brittle fragmentation in ploughing which seems to correlate with grain size and brittleness. It is interesting that there is a consistent trend of reducing scratch hardness at higher indenter force levels at which there is a greater tendency for brittle fracture to occur.

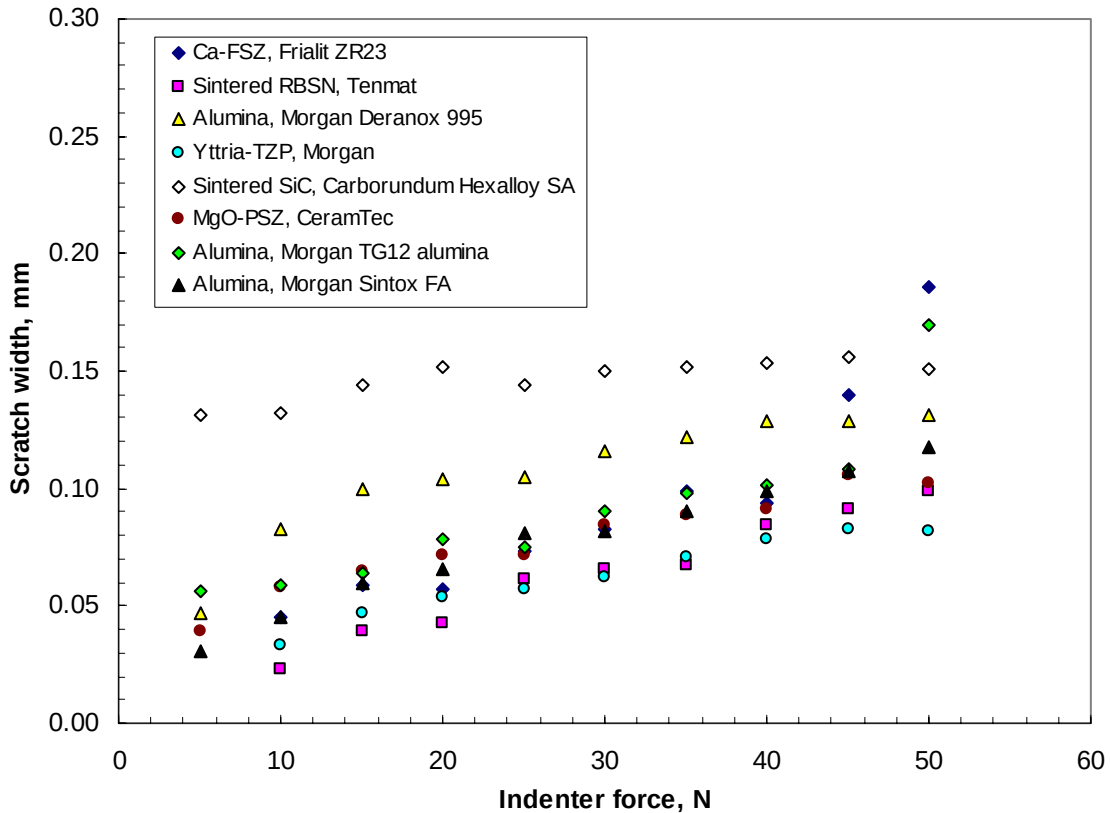


Figure 5: Scratch width as a function of indenter normal force for a range of ceramics.

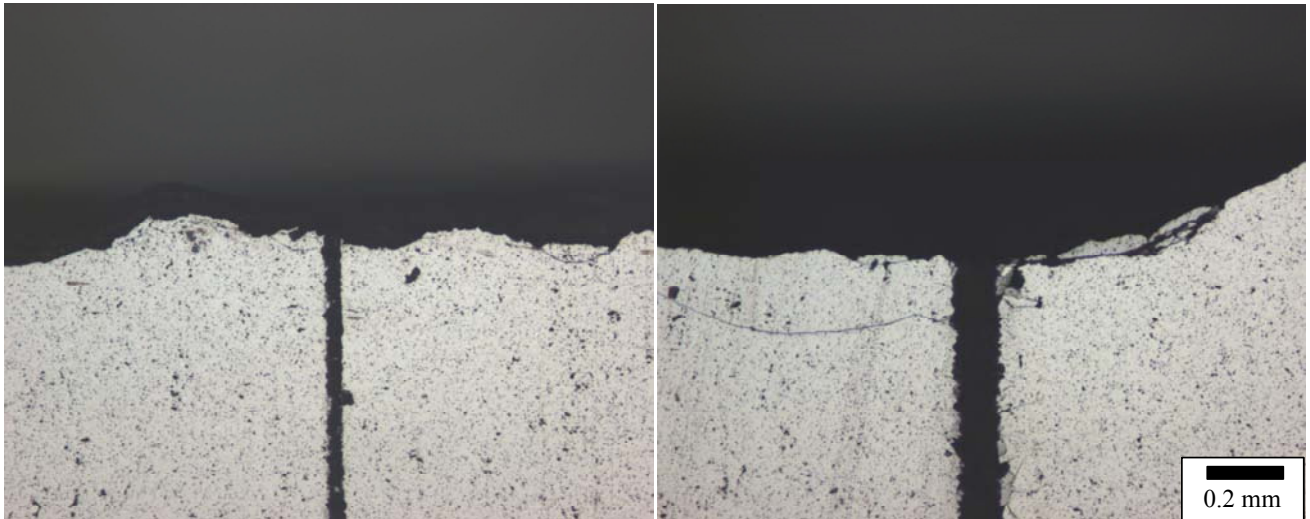


Figure 6: Scratches in Hexalloy SA sintered silicon carbide at (left) 10 N, and (right) 50 N.

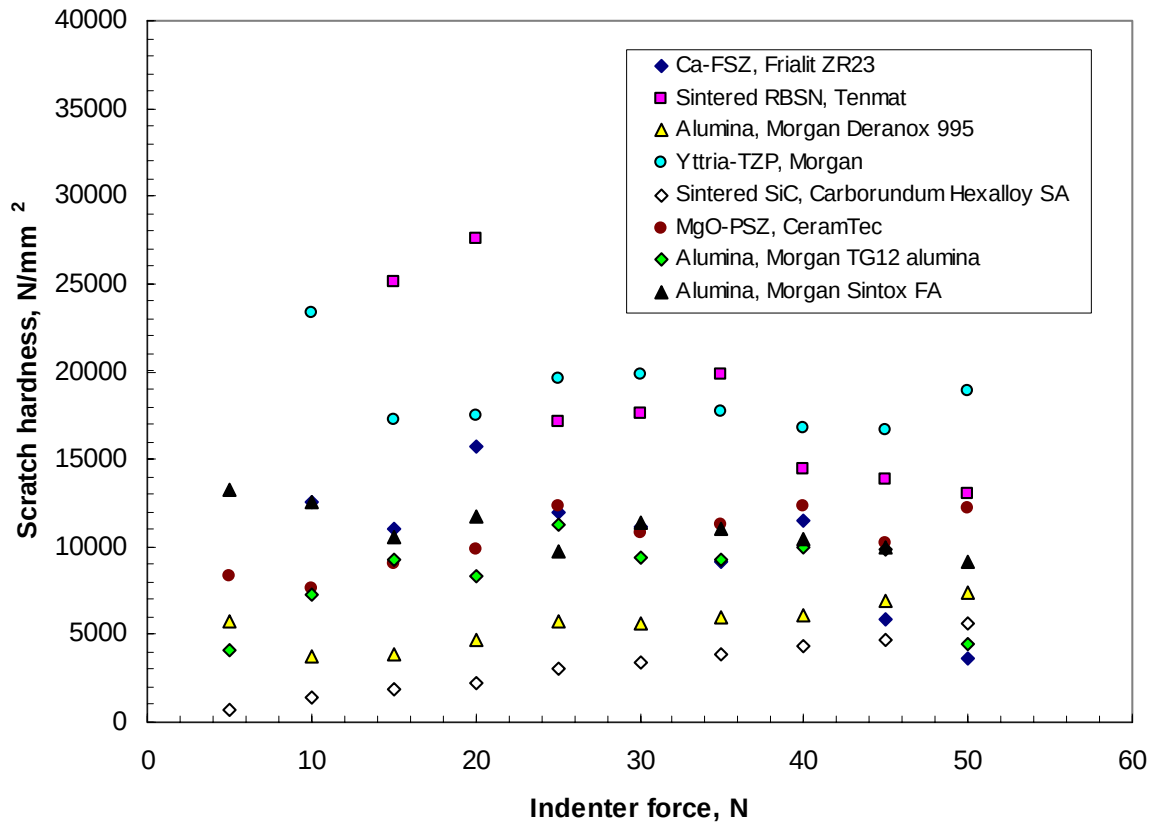


Figure 7: Scratch hardness as a function of indenter force.

Edge chipping behaviour

As noted above, flake thickness was recorded as the distance between the end of scratch centre-line and the original edge, which in most cases could be readily determined using the X-Y stage of the measuring microscope. The trends with normal indenter force are shown in Figure 8. It can be seen that Y-TZP produces by far the thinnest flakes,

with Mg-PSZ the next thinnest, while Ca-FSZ produced the largest and most variable sizes. Inspection of the microstructure of the last of these (Figure 9) shows that the grain size is very large, and a considerable amount of fragmentation occurs, which will limit the reproducibility of cracks of the scale of a few grains only.

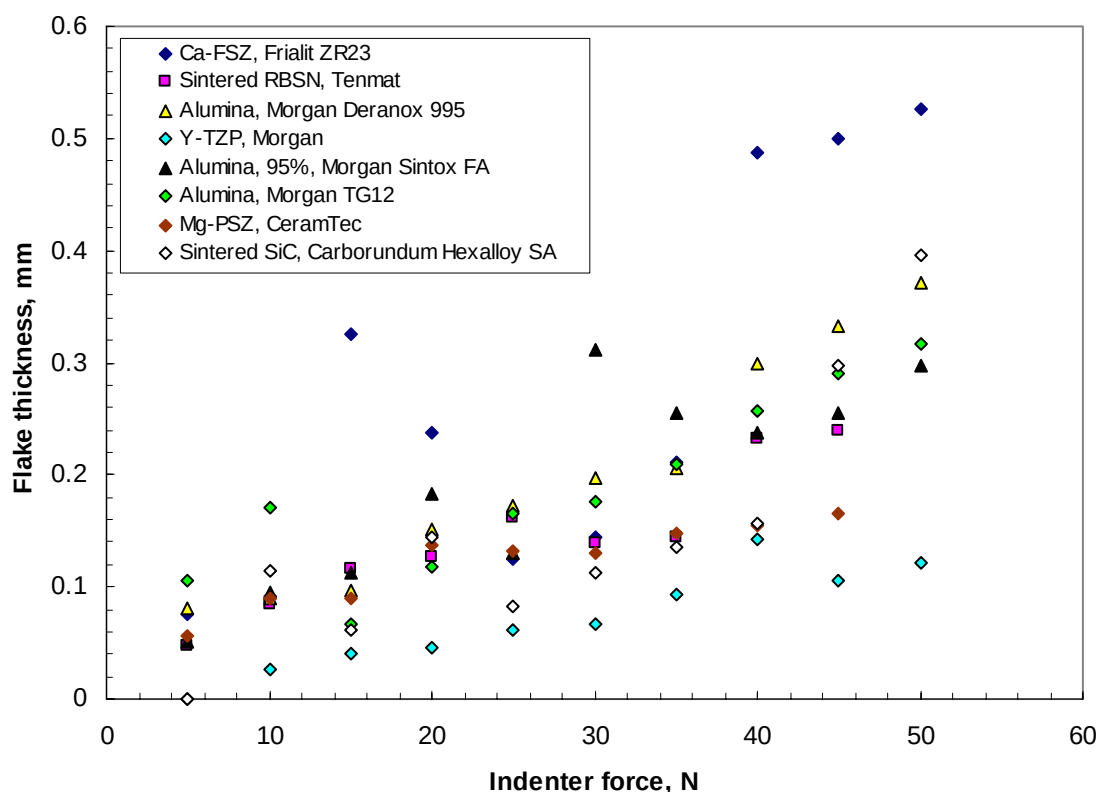


Figure 8: Nominal flake thickness as a function of indenter normal force.

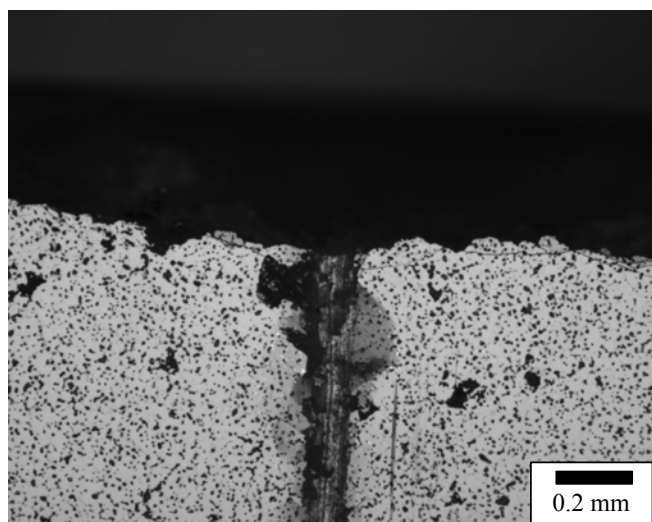


Figure 9: Scratch in Ca-FSZ showing grain damage and pluck-out. Note that the average grain size is about 100 μm .

Viewing the side face of the test-pieces, it was found that in most of the tests there was marked secondary damage to either side of the flake centre. However, it was usually possible to identify the trace of the scratch, or to determine its position from the constant

relative spacing of scratches. Based on this it was then possible to identify secondary damage and ignore that in measuring the flake dimensions. The formation of flakes in the ceramic test-pieces was much more variable than in glass. While there was a general tendency to increase in size with increasing indenter normal force, there was no consistency of shape or size except in the finest grained materials. In addition to the shape seen in Figure 2, thin feather-edged flakes were commonly seen in the same test-piece. In Y-TZP, unlike other materials, the flakes were triangular in shape, and so the maximum width was taken at the edge, while in most other cases the widest part was some distance below the edge. In view of the scatter, correlations were made using only the flake thickness.

Correlations of the inverse slopes of the data sets shown in Figure 8 (the 'edge toughness', units $\text{N/mm} [1]$) with nominal values of fracture toughness K_{Ic} and strain energy release rate $G_{Ic} = K_{Ic}^2/E$, are given in Figure 10. The results fall with a broad band as indicated by the dashed lines, and there is no clear single correlation.

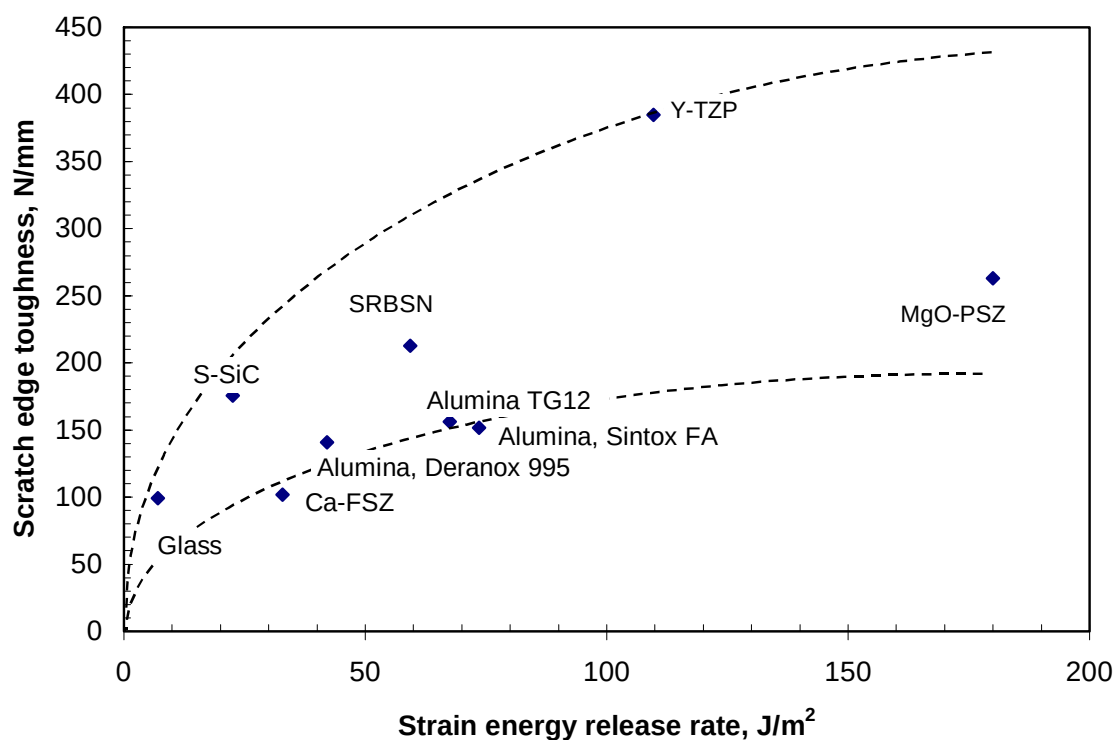
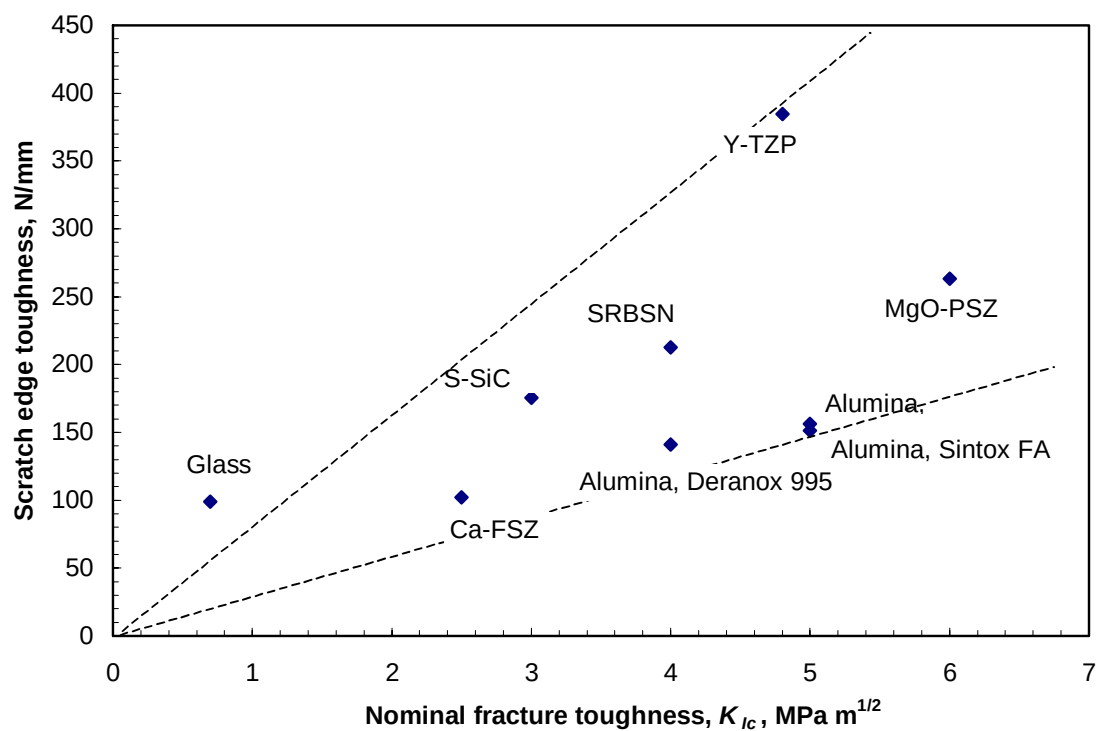


Figure 10: Correlations of scratch edge toughness with nominal values of (top) K_{Ic} and (bottom) G_{Ic} .

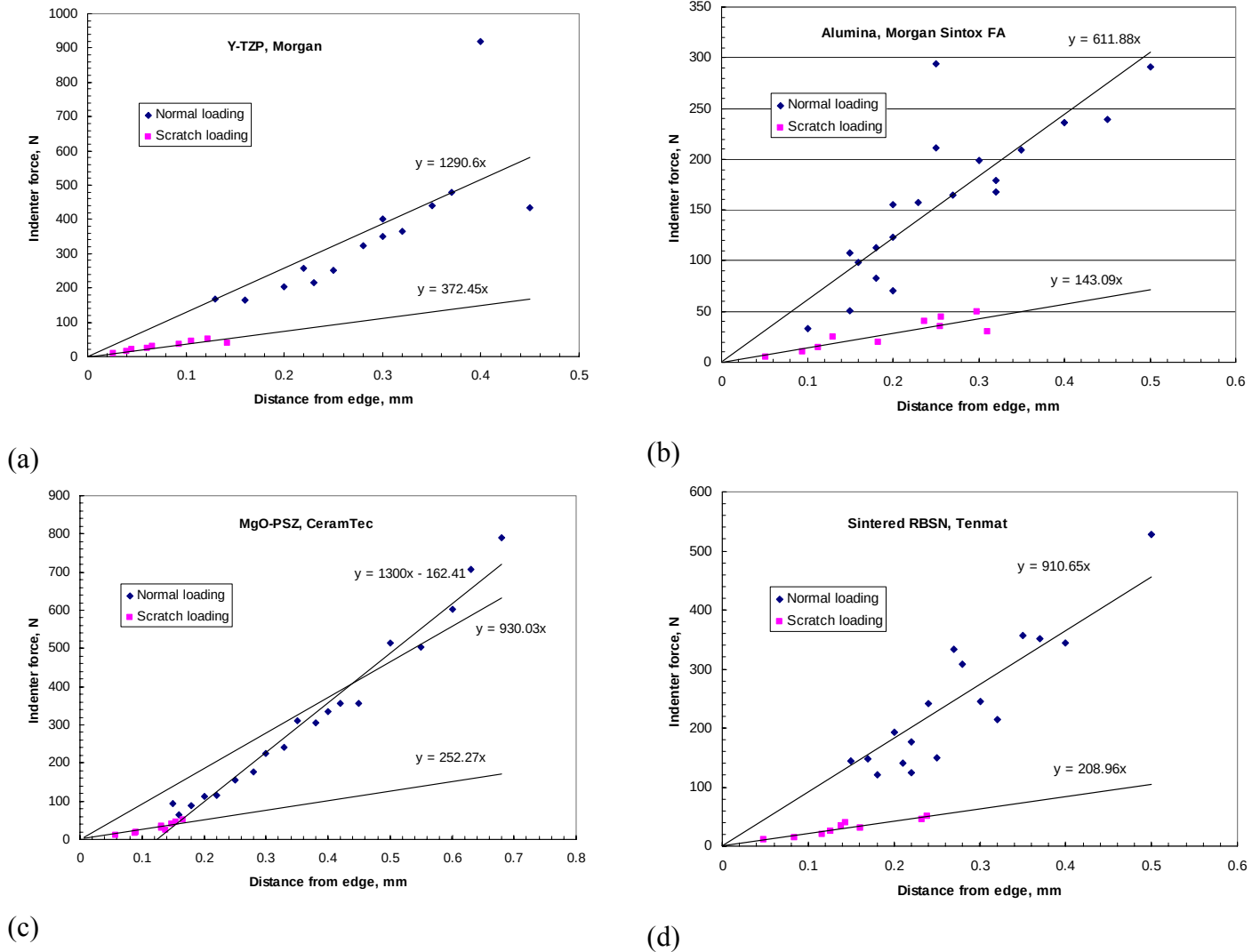


Figure 11: Comparisons of normal indentation edge flaking with scratch edge flaking for (a) Y-TZP, (b) 95% alumina, (c) MgO-PSZ, and (d) sintered RBSN.

DISCUSSION

In this investigation of semi-automated scratch edge chipping it has been found that there are some experimental difficulties associated with control of the indenter as the edge gives way. While it could be argued that this is a realistic representation of engineering situations where a loaded contact reaches an edge, the ancillary damage produced at the immediate edge makes measurement less certain, resulting in poorer correlations than would be the case if the indenter were prevented from dropping. Unfortunately this proved not to be possible with the present machine, and there are some suspicions that the flake depth measured is greater than the true depth. It proved difficult to retrieve intact flakes for later examination to verify this.

The resistance of Y-TZP to scratch edge damage clearly stands out in both these plots.

In order to compare the scratch results with the normal indentation method, a series of normal indentation edge chipping tests were made using the ET500 tester¹ equipped with a polycrystalline diamond Rockwell indenter. The indenter displacement rate was 0.5 mm/min, and a series of distances of indentation from the edge was preset over the range 0.1 to 0.7 mm.

Figure 11 shows examples comparing the data for four of the test materials, Y-TZP, MgO-PSZ, SRBSN and Sintox FA alumina. In each case the gradient of

¹ ET500 Edge toughness tester, manufactured by Engineering Systems (Nottingham) Ltd, UK.

the distance from edge plot vs. normal indentation force is significantly steeper than in the scratch edge chipping case. The ratio of slopes (the ‘edge toughness’) lies between 3.5 and 4.4, assuming the linear data fit is made through the origin (note that in the case of MgO-PSZ, the data are better fitted by a line not through the origin). Even if correction is made for the possibility that the thickness of scratch edge flakes is larger than the true thickness owing to indenter follow-through, the two sets of data remain distinctly separate. This agrees with the findings of other researchers [9] who found ratios between 1.6 and 3.3 for various ceramics. However, the present results are not in accordance with those of reference [6] where a similar comparison was made for a lithium disilicate glass-ceramic, with the finding that the two sets of data overlapped. Further investigation of this issue needs to be made.

The authors of reference [7] correlate their scratch edge chipping test results with fracture toughness K_{Ic} . There is an assumption that the indenter causes half-penny cracks to form, and by correlating the functional form of quasistatic indentation fracture with the known toughness of the material from single edge vee-notched beam (SEVNB) tests, they find a relationship of the form:

$$K_{Ic} = C \left[\frac{E}{H} \right]^{1/2} \left[\frac{P}{l^{3/2}} \right]$$

where E is Young’s modulus, H is hardness, l is the flake thickness, P is the indenter force, and $C \approx 0.054$. These authors were also able to demonstrate that a $3/2$ power law relationship exists between force and flake thickness, something alluded to also in [6]. In the present work, most of the data sets have been found to exhibit too much scatter to confirm that the power law, rather than a linear relationship, holds for this test. Attempts were made to force fit a $3/2$ power law to the data in order to compute toughness values, but the outcomes were not comparable to the fit shown in reference [7]. It was concluded that further testing needed to be performed to improve upon the quality of the data sets, especially the concerns about secondary damage modifying the apparent flake thickness determined subsequently to the test.

CONCLUSIONS

Scratch edge flaking is a convenient way of producing edge damage in materials, and thereby ranking the resistance of materials to this process. It has been shown to require a lesser indentation force to chip the edge than in the case of quasistatic

indentation, a factor which is ascribed to the additional frictional forces adding to the crack tip stress intensity once a crack is nucleated. Improvements in the ability to prevent secondary damage by the indenter are needed before correlations with material toughness can be developed further.

ACKNOWLEDGMENTS

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Further information

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