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Using Plasticity Values Determined From Systematic Hardness Indentation Measurements for Predicting Impact Behavior in Structural Ceramics: A New, Simple Screening Technique

by James W. McCauley and Trevor E. Wilantewicz

ARL-RP-268 **September 2009**

A reprint from the *Proceedings of the 26th Army Science Conference,* Orlando, FL, 1–4 December 2008.

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James W. McCauley
Weapons and Materials Research Directorate, ARL

Trevor E. Wilantewicz
Solyndra, Inc.

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14. ABSTRACT
In general, it has long been known that the hardness of ceramics correlates with gross impact performance, however, not to a degree useful for materials development. Wilkins, Cline and Honodel, 1969, were the first to point out the apparent importance of ceramic “plasticity” or inelastic deformation mechanisms in BeO and AlN in impact performance. More recently, Lundberg et al., 2000, have made compelling arguments that the compressive yield strength (related to hardness) augmented by the amount of “plasticity” in ceramics correlates well to transitional velocities (dwell), i.e. the velocity (or impact pressure) where penetration begins.
However, a direct measure of plasticity has not been determined. Hardness comparisons between materials are problematic since the values vary with the applied load, however, the full hardness-load curve can provide much more information on material behavior than hardness alone measured at a single load. In this work, several methods for curve fitting hardness-load data have been compared for both Knoop and Vickers hardness on several ceramic materials: aluminum oxynitride (AlON), silicon carbide, aluminum oxide and boron carbide. A power-law equation ($H = kF^c$) is shown to fit the Knoop data quite well. A plot of $\log_{10}$ (HK) vs. $\log_{10}$ (F) yielded easily comparable straight lines, whose slope and intercept data might be useful parameters to characterize the materials. It is shown on a series of hot pressed SiC variants that the absolute value of the reciprocal of the slope is a measure of plasticity and that the sum of this value with the calculated Knoop hardness at 1 N is a useful parameter to predict impact transitional velocity.

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USING PLASTICITY VALUES DETERMINED FROM SYSTEMATIC HARDNESS INDENTATION MEASUREMENTS FOR PREDICTING IMPACT BEHAVIOR IN STRUCTURAL CERAMICS: A NEW, SIMPLE SCREENING TECHNIQUE

James W. McCauley
U.S. Army Research Laboratory, APG, MD, 21005

Trevor E. Wilantewicz
ARL/ORAU Postdoctoral Research Fellow (now at Solyndra Inc., CA)

ABSTRACT

In general, it has long been known that the hardness of ceramics correlates with gross impact performance, however, not to a degree useful for materials development. Wilkins, Cline and Honodel, 1969, were the first to point out the apparent importance of ceramic "plasticity" or inelastic deformation mechanisms in BeO and AlN in impact performance. More recently, Lundberg et al., 2000, have made compelling arguments that the compressive yield strength (related to hardness) augmented by the amount of "plasticity" in ceramics correlates well to transitional velocities (dwell), i.e. the velocity (or impact pressure) where penetration begins. However, a direct measure of plasticity has not been determined. Hardness comparisons between materials are problematic since the values vary with the applied load, however, the full hardness-load curve can provide much more information on material behavior than hardness alone measured at a single load. In this work, several methods for curve fitting hardness-load data have been compared for both Knoop and Vickers hardness on several ceramic materials: aluminum oxynitride (AlON), silicon carbide, aluminum oxide and boron carbide. A power-law equation ($H = kF^c$) is shown to fit the Knoop data quite well. A plot of $\log_{10}$(HK) vs. $\log_{10}$(F) yielded easily comparable straight lines, whose slope and intercept data might be useful parameters to characterize the materials. It is shown on a series of hot pressed SiC variants that the absolute value of the reciprocal of the slope is a measure of plasticity and that the sum of this value with the calculated Knoop hardness at 1 N is a useful parameter to predict impact transitional velocity.

1. INTRODUCTION

Laboratory scale tests and/or mechanical properties that show good correlation with structural ceramic impact performance remain elusive to date. In order to expedite the further development of these materials (e.g. B_4C, SiC, Al_2O_3, AlN, AlON, glass ceramics, TiB_2 etc.), there is an urgent need to identify quasi-static and/or dynamic laboratory scale tests for rapid screening purposes, rather than having to utilize costly full scale tests early in the development of new and improved materials.

2. BACKGOUND

In general, it has long been known that hardness correlates with gross impact performance, however, not to a degree useful for materials development. Wilkins, Cline and Honodel, 1969, were the first to point out the apparent importance of ceramic "plasticity" or inelastic deformation mechanisms in BeO and AlN in impact performance. Later, Heard and Cline, 1980, carried out classic work quantifying the effect of confinement on the plasticity of BeO, AlN and Al_2O_3. Stress-strain curves for hot pressed AlN as a function of confining pressure (indicated on the curves in GPa) are presented in Fig. 1, showing that the "plasticity" of AlN is a function of the confining pressure; note that at about 0.5 GPa AlN behaves in a significantly plastic/ductile manner. Appropriately configured dynamic compression strength measurements in a Kolsky Bar (Split Hopkinson Pressure Bar - SHPB), have also been suggested (Pickup and Barker, 1997) as a way of determining the "plasticity"; McCauley and Quinn, 2006, have recently reviewed the determination of structural ceramic plasticity using this technique. More recently, Lankford, 2004, has reviewed these issues in the context of dynamic mechanical property measurements and recommended more systematic experimental work to clearly define the critical mechanisms. A new approach recently described by Wilantewicz and McCauley, 2008, using the exponent of a power law curve fit of the hardness – load curve, has been suggested as a quantitative representation of the amount of plasticity in structural ceramics. It was also proposed that the sum of the hardness and the quantitative plasticity value could be used to predict the transitional velocity (dwell) in an impact event.

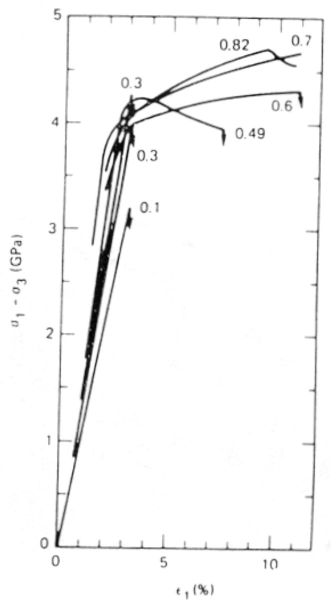

Figure 1. Stress-strain curves for hot pressed AlN as a function of confining stress; from Heard and Cline, 1980;

The theoretical strength of structural ceramics is generally estimated as the Young's modulus divided by 10. The actual strength of ceramics is controlled by the presence of processing defects in the form of pores, inclusions, microstructural inhomogeneities and residual stress. It is our contention that the stress to activate the inelastic deformation mechanisms or plasticity is larger than the actual strength in most structural ceramics.

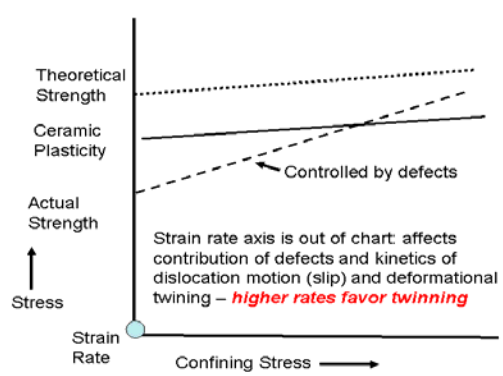

Figure 2. Confining stress effects on plasticity activation

In addition, we also posit that the actual strength can be significantly influenced by confining stress/pressure raising the actual strength to above the stress to activate plasticity mechanisms. This is schematically represented in Figure 2

The impact event can be simplified into two main stages: a dwell phase, where the impactor velocity is nominally zero at the ceramic front face, and a penetration phase through fragmented ceramic. If the impactor is completely stopped at the front surface, this is referred to as "interface defeat". More recently, Lundberg et al., 2000, have made compelling arguments that the compressive yield strength (related to hardness) augmented by the amount of "plasticity" in ceramics correlates well to transitional velocities (dwell), i.e. the velocity (or impact pressure) where penetration begins.

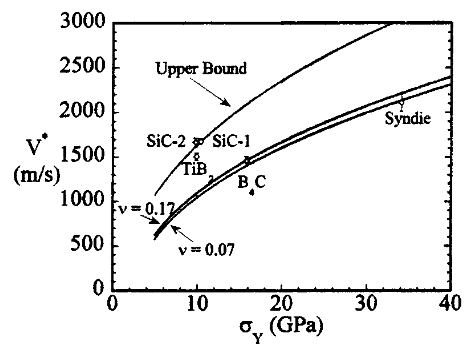

Figure 3. Transitional Velocities from Lundberg et al. 2000.

In Figure 3, V* is the transitional velocity (or pressure) and σ_y is compressive yield strength. The lower curve represents a purely brittle response, whereas the upper bound curve represents a perfectly plastic response, under triaxial stress conditions. If the analysis proposed by Lundberg et al., 2000, to explain their results is correct, then a small amount of "plasticity" can have a significant effect on the transitional velocity (dwell) of the ceramic. However, it is our contention that hardness - load curves may be a much more simple way to determine the plasticity of structural ceramics. Heard and Cline, 1980 used an imposed external confinement to activate plasticity, however, there is an intrinsic or self confining stress environment in hardness indentation measurements which should result in similar effects.

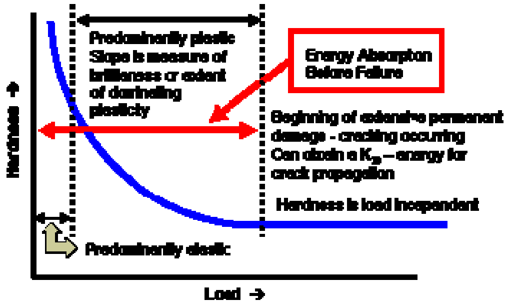

Figure 4. Conceptualized load – hardness curve

In hardness-load curves (Figure 4) there are, conceptually, three main regions: a predominantly elastic region, a predominantly plastic region and a region of extensive permanent damage (fracture) where extensive crack initiation and crack growth is occurring. The total work of indentation W_t (Bull, 2006), includes a number of energy dissipation mechanisms and can be formulated as follows:

$$W_t = W_{plas} + W_{fracture} + W_{elastic} + W_{thermal} + W_{\Delta\ phase} \quad (1)$$

If $W_{thermal}$ and $W_{\Delta\ phase}$ are very small:

$$W_t = W_{plas} + W_{fracture} + W_{elastic} \quad (2)$$

And the total work before failure (fracture) would reduce to:

$$W_t = W_{plasticity} + W_{elastic} \quad (3)$$

It is our hypothesis that the transitional velocity (dwell) is controlled by the hardness and the energy absorption in the predominantly elastic and predominantly plastic region, and that an appropriate power law equation describing the data in this region can be used to determine the amount of plasticity in the ceramic and, therefore, used to readily determine subtle differences in the ceramics. A fracture toughness value (K_{IC}) can be determined in the region of permanent damage, but this is not a measure of plasticity.

3. RESULTS

In previous work, Wilantewicz et al., 2008, fit Knoop data on a series of materials using the following equations:

$$HK = (a/F) + b \quad (4)$$

where,

a,b = constants

F = indentation load

and

$$HK = (a_1'/d) + a_2' \quad (5)$$

where,

a_1', a_2' = constants

d = diagonal length

Regression analysis using these equations did not exhibit consistently high correlations to the data.

Wilantewicz and McCauley, 2008, fit the same data using a power-law equation of the form:

$$HK = kF^{c} \quad (6)$$

where HK is the Knoop hardness (N/m^2), F indentation load (N), and k,c are constants determined by a computer regression analysis; c is dimensionless and k has unusual units of $N^{(1-c)}/m^2$. Taking the $\log_{10}$ of both sides of Eq. 1 yields:

$$\log_{10} HK = \log_{10} k + c \log_{10} F \quad (7)$$

If Eq. 6 accurately describes the data, then a plot of $\log_{10}$HK vs. $\log_{10}$F will yield easily comparable straight lines with slope c. Knoop hardness data was also measured on two hot-pressed boron carbide materials, and was collected using the same instrument, and careful procedures, described in the previous work.

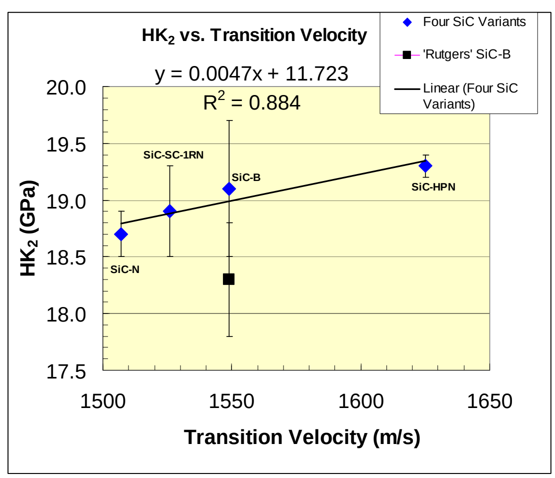

Figure 5. Rutgers SiC-B HK2: 18.3 ± 0.2 GPa (20 tests); original Lundberg and Lundberg (2005) SiC-B 19.1 ± 0.6 GPa (11 tests).

In addition, the Knoop hardness (Figure 5) of five hot-pressed silicon carbide materials was measured at 19.6 N and compared to the transition velocities measured in the reverse impact experiments conducted by Lundberg and Lundberg (2005). Two SiC-B materials were tested: a random one (Rutgers) and one from the original set of Lundberg tested samples. It is important to note that there is enough very subtle differences in the SiC-B samples to effect the hardness measurements. (Note: the hot pressed SiC variants were produced by BAE Advanced Ceramics, formerly Cercom)

Vickers hardness data were also generated on the same specimens, using the same instrument and procedures. A 15 second hold time at the maximum load was used. For these data, in addition to using Eq. 6, an additional equation was utilized:

$$HV = \frac{a}{F} + b \qquad (8)$$

where HV is the Vickers hardness (N/m^2), F indentation load (N), and a,b are constants determined by a computer regression analysis; b has units of N/m^2 and a has units of N^2/m^2. Although hardness can be plotted as a function of the diagonal size of the indentation, it is believed more appropriate to plot data as a function of load, since load is the independent variable that is under direct control. The constant 'a' gives the magnitude of the ISE (Indentation Size Effect), with larger values indicating a greater change in hardness for a given load interval. Unfortunately, excessive cracking at loads greater than about 30 N prevented the collection of reliable data.

Fig. 6 compares the power-law curve fit (Eq. 6) with the curve fit using Eq. 4 for the Knoop hardness of SiC-N. Note how Eq. 6 fits the data over the entire load range much better than Eq. 4, particularly at the higher loads, as shown by the much higher R^2 for the former than the latter i.e.,. 0.96 compared to only 0.67, respectively. Table 1 lists the R^2 values of the curve fits from Eq. 6, and those obtained from Eq. 4; columns 2-4 for Eq. 6 and column 5 for Eq.4. The Knoop hardness data for all the materials is shown in Fig. 7, plotted using Eq. 7. The slope, c, is indicative of the rate of change of the Knoop hardness with load. A negative value indicates the hardness decreases with load, as expected.

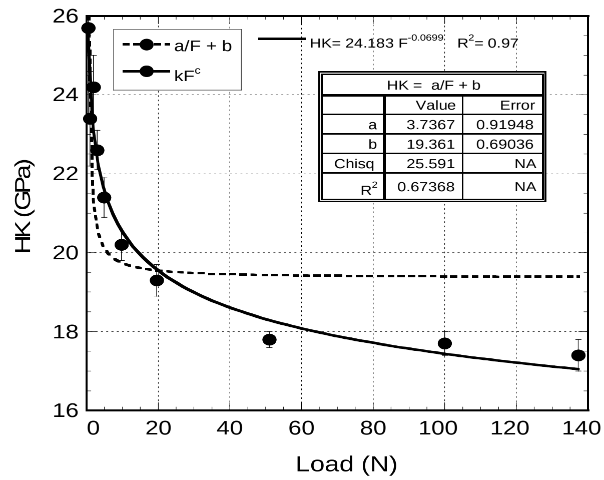

Figure 6. Knoop hardness data for SiC-N; two calculated curves using equations 6 and 4.

Table 1. Knoop Hardness Curve Fit Data

Material	Y-int. HK (GPa)	Slope, c	R^2	R^2 from (a/F + b) fit
SiC-N	24.1	-0.0699	0.97	0.67
SiC-B	23.8	-0.0728	0.98	0.67
PS-SiC*	23.8	-0.0845	0.96	0.51
AlON	16.0	-0.0565	0.98	0.65
AD995 CAP3 Al_2O_3	17.9	-0.0832	0.96	0.81
Cercom B_4C	29.6	-0.1536	0.98	0.90
Ceradyne B_4C	28.1	-0.1499	0.96	0.91

* PS-SiC is a pressureless sintered material.

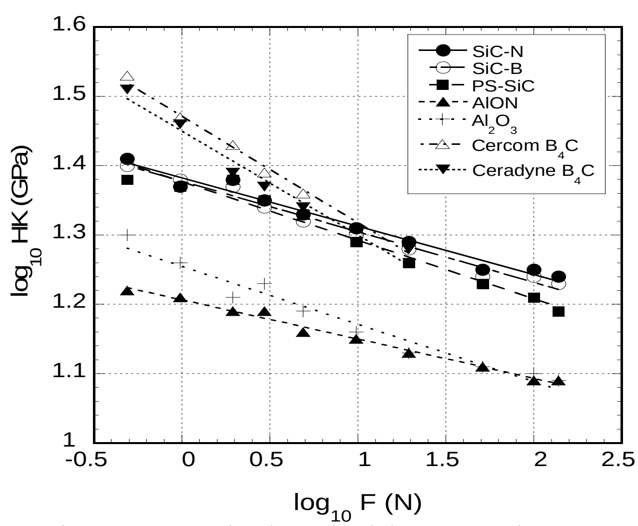

Figure 7. Knoop hardness-load data on a series of ceramics using equation 7

The y-intercept hardness, i.e., HK at $\log_{10}F = 0$, corresponds to the predicted Knoop hardness at F = 1 N, which is very close to the hardness measured at F = 0.98 N. The slope and intercept data from the Knoop tests are also summarized in Table 1. Note in Fig. 7 that the materials with the steeper slopes are more brittle and exhibit less plasticity.

4. DISCUSSION

It is proposed that the absolute value of the reciprocal of the slope (c) is a semi-quantitative measure of plasticity and that the sum of this value with the measured Knoop hardness at 1 N may be a useful parameter to correlate to transitional velocity. Simplistically, the transitional velocity should depend on the hardness and the plasticity as follows:

$$\text{Trans. Vel.} = \text{HK (1N)} + [-(1/c)] \qquad (9)$$

Using this equation and the experimental transitional velocity data from Lundberg and Lundberg, 2005, for SiC-N, SiC-SC-1RN and SiC-HPN and HK(1N) and (1/c) data from our measurements, a least squares analysis resulted in the following equation:

$$\text{Trans. Vel.} = 28.24\,[(\text{HK(1N)} - (1/c)] + 448.856 \qquad (10)$$

Experimental transitional velocities and [(HK(1N) – (1/c)] are plotted in Figure 8. Using this equation, transitional velocities can be predicted for other materials which are listed in Table. 2. Comparing the calculated transitional velocity with the average measured transitional velocity (standard deviation in parentheses), the match is quite remarkable: SiC-N – calculated = 1509 m/s, actual 1507 (±5); SiC-SC- 1RN – calc = 1524, act = 1526 (±25); SiC-HPN - calc = 1625, act =1625 (±12). In addition, transitional velocities can be predicted for other materials: e.g. AlON = 1401, B_4C = 1438-1469.

The plasticity is seen to vary from the most brittle boron carbide materials - 6.51, 6.67 to AlON -17.70 and SiC-HPN(L) -19.16. However, when the hardness is added to these values it results in a parameter that seems to correspond more closely with the transitional velocities of the SiC samples tested by Lundberg and Lundberg, 2005.

Finally, these are preliminary results and clearly should be followed up in more detailed investigations. In addition, since the data was obtained using quasi-static hardness measurements, extrapolation to dynamic environments may not be totally valid and would need to be confirmed by additional systematic work. Measurement of transitional velocities is an extremely complex experiment with specialized equipment. The results in this work should be taken in the context of the experimental procedures used by Lundberg and Lundberg, 2005, to measure transitional velocities.

5. CONCLUSIONS

We believe that this is the first time that the amount of plasticity before failure has been determined in structural ceramics. Other work on describing the “brittleness” of ceramics by Quinn and Quinn, 1997, and others, has been carried out, but those indices do not seem to equate to the “amount” of plasticity.

In summary:

- Knoop hardness-load data for structural ceramics are well described by a power-law curve fit – which can be conveniently plotted as straight lines using log-log plots
- The resulting slope is indicative of the amount of plasticity of the materials, which may be a useful parameter for materials screening
- There appears to be a correlation of the combination of hardness and the slope (plasticity) to transition velocities

Table 2. Calculated plasticity values and transitional velocities of selected ceramics.

Material	HK (GPa)	Act. Trans. Vel.	Slope (c)	-(1/c)	HK1N + plasticity	Calc Trans. Vel.
	Load = 1N	(m/s)		Plasticity		(m/s)
SiC-N (R)	24.1	-	-0.0699	14.31	38.41	1533.55
SiC-B (R)	23.8	-	-0.0728	13.74	37.54	1508.98
PS-SiC	23.8	-	-0.0845	11.83	35.63	1455.04
AlON	16	-	-0.0565	17.7	33.7	1400.54
AD995 CAP3	17.9	-	-0.0832	12.02	29.92	1293.79
Cercom B_4C	29.6	-	-0.1536	6.51	36.11	1468.60
Ceradyne B_4C	28.1	-	-0.1499	6.67	34.77	1430.76
SiC-N (L)	22.4	1507	-0.0661	15.13	37.53	1508.70
SiC-B (L)	-	1549	-	-	-	-
SiC-SC-1RN (L)	22.9	1526	-0.0659	15.18	38.08	1524.23
SiC-HPN (L)	22.5	1625	-0.0522	19.16	41.66	1625.33

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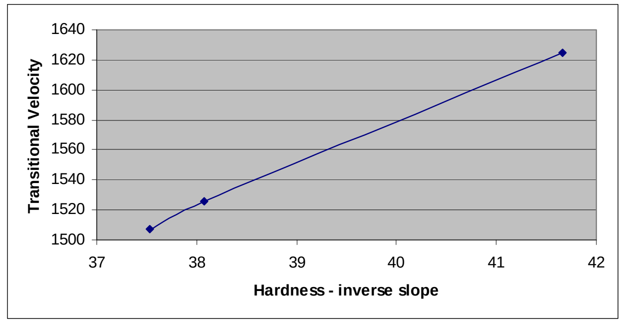

Figure 8. Experimental transitional velocity data plotted against [HK(1N) – (1/c)]

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