Hardness of alumina/silicon carbide nanocomposites at various silicon carbide volume percentages

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HARDNESS OF ALUMINA/SILICON CARBIDE NANOCOMPOSITES AT VARIOUS SILICON CARBIDE VOLUME PERCENTAGES

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ABSTRACT

Vickers indentation was employed to measure the microhardness of monolithic alumina and six alumina-based nanocomposites consisting of variable silicon carbide nanoparticle volume percentages of 0.3% to 20%. Indentation tests were performed over a broad range of loads from 0.5N to 40N. The resultant hardness-load curves exhibit cumulative increases in the apparent hardness based on the silicon carbide content and reveal each sample suffers from a prominent indentation size effect (ISE). Herein, we present a comprehensive analysis of this data using Meyer’s Law, the proportional specimen resistance model (PSR) and the modified proportional specimen resistance model (MPSR) and employ TEM imagery to detail potential mechanisms by which silicon carbide nano-reinforcements influence the “true hardness” and the ISE.

INTRODUCTION

For nearly a century, microindentation has been a fundamental and widely-accepted method for determining the mechanical property of hardness\(^1,\, 2\). In ceramics, irrespective of the indenter characteristics (Vickers, Knoop, Berkovich, Rockwell, Brinell, etc.), the technique is said to provide a quantifiable measurement of resistance to deformation, densification, displacement and fracture\(^3\). This is achieved by measuring the horizontal and vertical diameters of a diamond-shaped impression generated by a loaded indenter. The hardness \((H)\) is subsequently calculated as a ratio of load to resultant contact area, the equation of which is represented as follows:

\[ H = \alpha \left( \frac{P}{d^2} \right) \]  

where \(d\) is an average of the respective diameters of the indent, \(\alpha\) is the indenter constant, a number dependant on the geometry of the indenter employed, and \(P\) is the load.

Traditionally, during conventional testing both the indenter constant and load would remain as fixed parameters. However, it has been reported in numerous investigations that by increasing the load applied through the indenter a notable decrease in the calculated hardness can be observed\(^3,\, 4,\, 5,\, 6,\, 7,\, 8,\, 9,\, 10,\, 11,\, 12,\, 13,\, 14\). This apparent load-dependency in the eventual hardness values realised through microindentation is referred to as the indentation size effect (ISE) and was initially described through Meyer’s Law\(^15\)

\[ P = Ad^n \]  

where \(A\) and \(n\) are constants derived via regression analysis of experimental results.

Unfortunately, at present, because of the ISE phenomena, only a superficial hardness value can be obtained from the application of any microindentation technique and whilst Meyer’s Law aligns relatively well with the majority of hardness-load curves \((H-P)\), it is still insufficient and unable to address this issue.

Consequently, a range of alternative models have been proposed, including the Hays-Kendall approach\(^8\), the elastic recovery model\(^9\), the proportional specimen resistance model (PSR)\(^10,\, 11,\, 12\), the energy-balance approach\(^5,\, 11\), the modified proportional specimen resistance model (MPSR)\(^13\) and the fractal approach\(^14\).
For brittle ceramic materials, which do not experience the increased complexities of metal-related mechanics such as work hardening and high elastic strain, the PSR (Eq. 3) and MPSR models (Eq.4) have been adopted. These two models, when comparatively assessed against Meyer’s Law, exhibit not only an improved curve fitting against the experimental data, but also involve the separation of the load-dependent ISE and the load-independent hardness into two individual terms, $a_1$ and $a_2$ respectively. In doing so, what is known as and argued to be the “true hardness”$^{16}$ ($a_2$) can be determined.

$$P = a_1d + a_2d^2$$ (3)

$$P = a_0 + a_1d + a_2d^2$$ (4)

where $a_0$ is an additional constant that represents the indenter-induced residual surface stresses.

Whilst a number of comparative studies conducted on several single-crystal and polycrystalline ceramics have remained inconclusive as to the ascendancy of each model$^{6, 12, 13, 16, 17, 18, 19, 20}$ speculations concerning the cause of the ISE and the physical meaning associated with the “true hardness” have been widespread.

The $a_1$ term has been related to various mechanisms, including the energy consumed in the creation of new surfaces$^{10, 11}$, the energy required for surface stretching prior to crack initiation$^{16}$, interfacial friction resistance$^{12, 21}$, bulk elastic recovery$^{9, 12}$ and subsurface cracking$^{22}$, either inherent or initiated concurrently with the indentation process.

Conversely, the $a_2$ term has just one interpretation, being ascribed to crack-free plastic deformation. Nevertheless, whilst this has been generally acknowledged, the supporting evidence is sparse. Furthermore, there is a lack of comprehension in regards to the governing factors which control the “true hardness”.

In this paper, we report the results of an extensive and meticulous Vickers microindentation study performed on ground and polished monolithic alumina (Al$_2$O$_3$) ceramics and alumina/SiC (Al$_2$O$_3$/SiC) nanocomposites: the use of which, can be rationalised by considering that one sole variable differentiates each sample, the volume percentage (vol%) of SiC dispersants. Using observational work from earlier studies by one of these authors, we propose that variations in the “true hardness” are dictated by the vol% of SiC and can be attributed to the nano-dispersants capacity to resist the motion of dislocations in the matrix.

EXPERIMENTAL PROCEDURE

In this investigation, a monolithic Al$_2$O$_3$ ceramic and six Al$_2$O$_3$-based nanocomposites containing 0.3, 1, 3, 5, 10 and 20vol%SiC were prepared via pressureless-sintering and hot-pressing respectively.

An in-depth methodology detailing the manufacture process in full is given in other publications$^{23}$. Briefly though, in order to control the growth of alumina grains and to ensure a mean grain size equivalent to that of the pure Al$_2$O$_3$ pressureless-sintered at 1500$^\circ$C (~3-4µm), all nanocomposites were hot-pressed in a graphite die at between 1550-1720$^\circ$C for 1hr under a pressure of ~25MPa in flowing argon. In doing so, full densification of all the samples was achieved. The Al$_2$O$_3$ powder used was a high purity (99.99% α- Al$_2$O$_3$) AKP53 (Sumitomo, Japan) with a sub-micrometre particle size. The SiC nanoparticles were α-SiC powder UF 45 (Lonza, Germany) with an average particles size of ~90nm.

After manufacture, the indentation surfaces of the monolithic Al$_2$O$_3$ and Al$_2$O$_3$/SiC nanocomposites were machined to produce a smooth surface-finish parallel with the base plane. The samples were positioned onto two flat mild steel blocks (4 samples on each) and held in place by a hard, high strength mounting wax (Testbourne Ltd, Basingstoke, UK). Initial levelling of the surface
was carried out using an Axminster Sieg U2 1000W drill (Axminster Tool Centre, Devon, UK) under the following conditions: Ø60mm diamond (100µm) core drill bit at a rotation speed 450rpm and a feed depth of 10µm per pass. Such coarse grinding was followed by lapping and polishing on a bench mounted KEMET 15 flat-bed diamond lapping machine (Kemet International, Maidstone, UK). Four grades of diamond slurry were applied to 4 separate wheels; 25µm iron wheel, 8µm and 3µm copper wheel, 3µm cloth polishing wheel, and a 1µm cloth polishing wheel. Depending on the wheel, a typical cycle lasted between 30-120 minutes. Once a shiny, polished surface-finish was achieved the samples were cleaned to remove any residual lubricant and/or diamond grit before indentation.

Two Vickers microindenters were employed to generate indents at the various loads. A Mitutoyo HM-124 (Mitutoyo UK Ltd, Andover, UK) was used at loads of 0.05kgf (0.49N), 0.1kgf (0.98N), 0.2kgf (1.96N), 0.3kgf (2.94N), 0.5kgf (4.9N), 1kgf (9.8N), and 2kgf (19.61N). An Innovatest Nexus 4503 (Innovatest Europe, Maastricht, The Netherlands) was then needed for higher loads of 2.5kgf (24.51N), 3.0kgf (29.42 N) and 4kgf (39.23N). Both machines boast a load accuracy of <1% at each load. 15 seconds was the elected dwell time. All samples remained on the mild steel blocks during indentation which was performed at room temperature.

The resultant indents were imaged in the SEM. All SEM observations were made using a Carl Zeiss (Leo) 1530 VP; samples were sputter-coated with a thin coating of gold-palladium to prevent charging. Meanwhile, graphic editing software (Adobe Photoshop, USA) was used to determine the horizontal \(d_1\) and vertical \(d_2\) diameters in mm. The hardness was calculated using Eq. 1, where \(\alpha\), the indenter specific constant = 1.8544 for a Vickers indenter and the load, \(P\), was in Newtons (N).

All cross-sectioned TEM samples were prepared by ion-milling\(^2\) and TEM analysis was carried out on JEOL200CX and JEOL4000 microscopes.

The load dependent nature of the measured hardness results for both monolithic Al\(_2\)O\(_3\) and each of the Al\(_2\)O\(_3\)/SiC nanocomposites was subsequently analysed against three models: Meyer’s Law, the PSR model and the MSPR model.

EXPERIMENTAL RESULTS AND DISCUSSION

The \(H-P\) curves for the monolithic Al\(_2\)O\(_3\) and the Al\(_2\)O\(_3\)/SiC nanocomposites are displayed in Fig. 1(a). For the purposes of clarity and conciseness, all error bars of plus and minus one standard deviation have been displayed in the adjoining bar chart in Fig. 1(b). As expected, maximum scattering, typically 7–8% of the hardness value, occurred in the fracture-prone unreinforced Al\(_2\)O\(_3\) and is particularly prevalent at the lower load ranges. As recommended\(^3\),\(^20\), in order to alleviate variance, each of the plotted data points is an average of at least 5 measurements. The typical conditions of the indents measured are presented in the SEM images in Fig. 2.

From Fig. 1(a), it is evident that both the monolithic Al\(_2\)O\(_3\) and the Al\(_2\)O\(_3\)/SiC nanocomposites experience an ISE of varying degrees. Additionally, each sample appears to encounter an abrupt, but continuous plateau in the apparent hardness values collected after ~20N. This is consistent with the hardness transition point hypothesised by Quinn & Quinn\(^3\).

Using the data in Table 1, confirmation that a levelling of the hardness has occurred is achieved by comparatively examining the mean hardness \(H_{\text{plateau}}\) across the four loads of the plateau (20N, 25N, 30N and 40N) against the minimum recorded hardness \(H_{\text{min}}\) for each sample. In all cases, these numbers are similar in value, despite the \(H_{\text{min}}\) being taken from various load points \(P_{\text{min}}\). Whilst not entirely conclusive, it is possible to deduce from this that across the loads of the \(H_{\text{plateau}}\) the measured hardness has begun to equalise. Meyer’s Law may also be employed to confirm this.
Figure 1: Hardness-load data for Al$_2$O$_3$ and Al$_2$O$_3$/SiC nanocomposites with different vol\% SiC dispersants; (a) a line graph with power trendlines highlighting the ISE (b) a bar chart with error bars of one standard deviation.
Another feature of the $H$-$P$ graph worth noting is the maximum recorded hardness ($H_{\text{max}}$) of the $\text{Al}_2\text{O}_3$/SiC nanocomposite. At 0.5N ($P_{\text{min}}$) the numerical values of the $H_{\text{max}}$ converge regardless of the SiC vol%, but immediately separate under greater loads. The circumstances of this have been rationalised by Lawn, Marshall and coworkers, who explain in a collection of papers that indents evolve from deformation-dominated behaviour to fracture-dominated behaviour above a certain critical load$^{24, 25, 26, 27}$. In conjunction with this theory, we further speculate that at loads as low as 0.5N, with a penetration depth of ~1-2µm, the range of the dislocation activity would be limited to one single alumina grain. This constrains dislocation gliding to short ranges which likely facilitates work hardening, especially if cracking is not available to release the strain energy. The introduction of SiC nano-dispersants imposes further resistance leading to a higher hardness for the nanocomposites. It is possible, that as the load increases, cracking is initiated through the interactions between slip and the grain boundary or among the slips themselves, dissipating the built-up strain energy and prohibiting work hardening. We suspect further nanoindentation studies will disclose more detail on the converging effect of the hardness values at low loads.

![Figure 2: Representative SEM images of indents on samples containing 0, 1, 5 and 20vol%SiC at 0.2kgf (2N) and 2kgf (20N).](image)

<table>
<thead>
<tr>
<th>SiC (vol%)</th>
<th>$H_{\text{max}}$ (GPa)</th>
<th>$P_{\text{max}}$ (N)</th>
<th>$H_{\text{min}}$ (GPa)</th>
<th>$P_{\text{min}}$ (N)</th>
<th>$H_{\text{plateau}}$ (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>20.58</td>
<td>0.5</td>
<td>14.96</td>
<td>25</td>
<td>15.31</td>
</tr>
<tr>
<td>0.3</td>
<td>22.97</td>
<td>0.5</td>
<td>17.25</td>
<td>25</td>
<td>17.39</td>
</tr>
<tr>
<td>1</td>
<td>22.95</td>
<td>0.5</td>
<td>17.83</td>
<td>30</td>
<td>17.94</td>
</tr>
<tr>
<td>3</td>
<td>22.61</td>
<td>0.5</td>
<td>17.93</td>
<td>30</td>
<td>18.11</td>
</tr>
<tr>
<td>5</td>
<td>22.95</td>
<td>0.5</td>
<td>18.61</td>
<td>25</td>
<td>18.68</td>
</tr>
<tr>
<td>10</td>
<td>23.02</td>
<td>0.5</td>
<td>18.99</td>
<td>25</td>
<td>19.12</td>
</tr>
<tr>
<td>20</td>
<td>23.07</td>
<td>0.5</td>
<td>19.75</td>
<td>30</td>
<td>19.81</td>
</tr>
</tbody>
</table>
Figure 3: The log(load)-log(diameter) data for Al₂O₃ and Al₂O₃/SiC nanocomposites with different vol% SiC dispersants. Note the “kink” in the pure alumina curve across the higher load points.

(a) Meyer’s Law

The classical power law adopted to describe the $H$-$P$ curves can be further applied to approximately quantify the severity of the ISE based on the value of the exponent or Meyer Index, $n$. In the absence of an ISE $n = 2$. In this scenario, Meyer’s Law is modified to form Kick’s Law:\(^{28}\)

$$P = Ad^2$$ \hspace{1cm} (6)

where $P$, $d$ and $A$ are of equivalent significance as in Eq. 2. If $n < 2$ then the measured hardness decreases with an increasing load, indicating the presence of an ISE. In the rare case when $n > 2$ a reverse ISE behaviour is said to exist\(^{17,29}\).

In order to determine the $A$- and $n$-values through regression analysis, the Meyer’s Law, defined in Eq. 2, is expressed in a logarithmic relationship:

$$\log P = \log A + n \times \log d$$ \hspace{1cm} (7)

The resultant log$P$ vs. log$d$ graph is presented in Fig. 3 with the corresponding $A$- (intercept) and $n$-values (gradient) displayed in Table 2. By reviewing Fig. 3, a relatively high degree of linearity can be seen in each of the nanocomposites. However, the monolithic Al₂O₃ notably deviates at the higher applied loads of 20, 25, 30 and 40N. This may be attributable to measurement errors caused by the brittleness of Al₂O₃ which inhibited the precise dimensional assessment of the indents. Fig 1(b) supports this by highlighting the comparatively large error bars for pure Al₂O₃ at the aforementioned loads. Additionally, Fig. 2 visually illustrates the brittle nature of the unreinforced Al₂O₃, even at 2N. Nevertheless, overall, the results are consistent with previous studies\(^{19}\).

From Table 2, a steady increase in the calculated $n$-values across all samples clearly demonstrates a substantial reduction in the ISE, particularly between the monolithic Al₂O₃ and the 0.3vol%SiC sample. More gradual changes are seen across the nanocomposites, with increasing SiC content correlating with a less pronounced ISE. The higher $n$-values also imply that the nanocomposites experience more ductile deformation during indentation with variations in the vol% of SiC nano-dispersants controlling the degree with which such deformation dominates. No direct relationship between the SiC vol% and the $n$-value is apparent, nor is there a synonymous relationship between $A$ and $n$ as described by Sargent and Page\(^{4,6}\).

Referring back to the $H_{\text{plateau}}$ in Table 1, Meyer’s Law can be employed to affirm the observed levelling of the measured hardness. In accordance with Eq. 6, if the $n$-value across the narrower load range = 2 it implies that the ISE has disappeared. From the results for the $n_{\text{plateaus}}$ presented in Table 2, it is apparent that the ISE has deteriorated to a negligible level in the SiC reinforced Al₂O₃ nanocomposites. However, the same cannot be said for the $n_{\text{plateau}}$ in the unreinforced Al₂O₃ which has
a value relatively similar to its original Meyer index and still significantly lower than the $n$-value of 0.3vol%SiC. Thus, it appears the ISE is still prevalent within the monolithic Al$_2$O$_3$ across the $H_{plateau}$ and that a levelling of the data collected is yet to occur.

Table 2: A summary of the $n$ and $A$ parameters determined using the Meyer’s Law and the $n$-values calculated across a narrower load range of 20-40N ($H_{plateau}$).

<table>
<thead>
<tr>
<th></th>
<th>0vol%SiC</th>
<th>0.3vol%SiC</th>
<th>1vol%SiC</th>
<th>3vol%SiC</th>
<th>5vol%SiC</th>
<th>10vol%SiC</th>
<th>20vol%SiC</th>
</tr>
</thead>
<tbody>
<tr>
<td>$n$</td>
<td>1.857</td>
<td>1.886</td>
<td>1.897</td>
<td>1.900</td>
<td>1.911</td>
<td>1.912</td>
<td>1.921</td>
</tr>
<tr>
<td>$A$</td>
<td>42.24</td>
<td>45.92</td>
<td>47.28</td>
<td>47.73</td>
<td>48.94</td>
<td>49.57</td>
<td>50.83</td>
</tr>
<tr>
<td>$R^2$</td>
<td>0.9998</td>
<td>0.9998</td>
<td>0.9999</td>
<td>0.9999</td>
<td>0.9999</td>
<td>0.9999</td>
<td>0.9999</td>
</tr>
<tr>
<td>$n_{plateau}$</td>
<td>1.869</td>
<td>2.013</td>
<td>2.000</td>
<td>2.010</td>
<td>2.017</td>
<td>2.035</td>
<td>1.985</td>
</tr>
</tbody>
</table>

(b) PSR Model

It is generally agreed that whilst Meyer’s Law provides a reliable indication of the existence of an ISE, it is insufficient in interpreting its origin and is incapable of determining the “true hardness” when $n$ does not equal 2. Consequently, the PSR model was composed\cite{10,11,12}, combining Kick’s Law (Eq. 6), known to be the load-independent hardness ($a_2d^2$), and a modified version of Hays and Kendall’s $W$ constant, representing a materials resistance to the initiation of plastic flow, but that increases with indentation size and is directly proportional ($a_1d$).

By rearranging the PSR equation (Eq. 3) into the following

\[ P/d = a_1 + a_2d \]  \hspace{1cm} (8)

regression analysis can be employed to acquire the $a_1$- and $a_2$-values. Such data is presented in both the $P/d$-d graph (Fig. 4) and in numerical format in Table 3. Table 3 also displays the “true hardness” values ($H_{PSR}$) calculated by combining Eqs. 1 and 3 to formulate Eq. 9:

\[ P = a_1d + (H/\alpha)d^2 \]  \hspace{1cm} (9)

where $\alpha$ is the indenter specific constant for a Vickers indenter and equal to 1.8544.

The majority of the $a_1$-values obtained are in good agreement with the $n$-values derived using Meyer’s Law. In both models, the largest sequential change is between the pure Al$_2$O$_3$ and 0.3vol%SiC nanocomposite. Similarly, incremental reductions in the ISE can be identified across all samples. However, magnified displays of nonlinearity result in a relatively lower correlation coefficient and irregularities in the $a_1$-values are seen at 3 and 10vol%SiC. At these points $a_1$ actually increases with further additions of SiC, rather than decreasing. The reason why this occurs is unknown.

Conversely, the $a_2$ term exhibits a favourable trend with regular increases in value related to higher vol% of SiC nano-dispersants.
Comprehension of the “true hardness” ($H_{PSR}$) determined from $a_2$ is exceedingly difficult as the numbers are lower than the $H_{min}$ values displayed in Table 1. Several authors have produced complementary data$^{19, 20, 30, 31}$ with some concluding that it is only logical to reject “true hardness” values that are below experimental measurements$^{44}$.

Table 3: A summary of the $a_1$ and $a_2$ terms according to the PSR model in conjunction with the “true hardness” values.

<table>
<thead>
<tr>
<th>SiC (vol%)</th>
<th>$a_1$</th>
<th>$a_2$</th>
<th>$H_{PSR}$ (Gpa)</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>36.34</td>
<td>7648.53</td>
<td>14.19</td>
<td>0.9964</td>
</tr>
<tr>
<td>0.3</td>
<td>25.26</td>
<td>8957.12</td>
<td>16.61</td>
<td>0.9990</td>
</tr>
<tr>
<td>1</td>
<td>24.90</td>
<td>9255.86</td>
<td>17.16</td>
<td>0.9988</td>
</tr>
<tr>
<td>3</td>
<td>25.45</td>
<td>9344.53</td>
<td>17.33</td>
<td>0.9984</td>
</tr>
<tr>
<td>5</td>
<td>22.55</td>
<td>9696.22</td>
<td>17.98</td>
<td>0.9990</td>
</tr>
<tr>
<td>10</td>
<td>22.89</td>
<td>9931.37</td>
<td>18.42</td>
<td>0.9987</td>
</tr>
<tr>
<td>20</td>
<td>21.51</td>
<td>10299.41</td>
<td>19.10</td>
<td>0.9993</td>
</tr>
</tbody>
</table>

(c) MPSR model

In a series of papers, Gong reported various problems with the PSR model beyond those documented in this paper. These included the nonlinearity of the $P/d$-$d$ curves and the “true hardness” across wider and narrower load ranges$^{13}$, and the inability to determine agreeable reverse ISE results$^{17}$. As such, Gong deemed the PSR model unsatisfactory for explaining the ISE and made appropriate adjustments to form the MPSR model, defined in Eq. 4.

The application of this equation to the $H$-$P$ data in Fig. 1 produces the $P$-$d$ curves illustrated in Fig. 5. Values for $a_0$, $a_1$ and $a_2$ are evaluated through second order polynomial regression analysis and presented in Table 4.

The “true hardness” values for each of the samples have been estimated by substituting Eq. 1 into Eq. 4 generating Eq. 10 shown below:

$$P = a_0 + a_1d + (H/\alpha)d^2$$ (10)

In both of the MPSR equations (Eq. 4, Eq. 10), the addition of the $a_0$ term is said to account for the residual surface stresses caused by abrasive grinding/polishing and are strictly compressive. Hence, as Gong experienced, values of $a_0$ should be negative. In Table 4, $a_0$ for pure Al$_2$O$_3$ conforms to this assumption and even yields a numerical value consistent with Gong’s initial study ($a_0 = -0.3N$)$^{13}$.

However, successive results for the nanocomposite shift from negative to positive to negative, or compression to tension to compression. This is inconsistent with our own surface residual stress measurements performed using luminescence spectroscopy$^{37}$. Although, the data from the investigation...
found significant fluctuations between compression and tension, it showed that Al₂O₃, with the same 1µm finish, has a net tensile residual stress whilst the Al₂O₃/SiC nanocomposite has a net compressive residual stress. This is contrary to the \( a_0 \) values obtained using the MPSR model.

It should be noted that Gong has since revised his original interpretation of the physical meaning of \( a_0 \) and currently associates it with experimental errors caused by the optical resolution tolerances of the objective lens used and/or the load accuracy of the indenter equipment. Regardless, the addition of the \( a_0 \) term achieves its intended purpose, to improve the correlation coefficient over varying load ranges. Evidently, there is scope for a further investigation into whether a physical meaning exists for \( a_0 \).

For \( a_1 \), a distinct differential between the unreinforced Al₂O₃ and the nanocomposite is apparent. However, as with \( a_0 \), the actual numerical values follow no discernable pattern.

As expected, the behaviour of \( a_2 \) is in accordance with the exponent, \( n \), in Meyer’s Law and the \( a_2 \) term in the PSR model. Once again, a substantial increase between the monolithic Al₂O₃ and the 0.3vol%SiC nanocomposite occurs with small, accumulative increases across the nanocomposites. Similarly, as observed in the PSR model, the calculated “true hardness” (\( H_{MPSR} \)) values determined from the \( a_2 \) data are less than experimental results.

Table 4: A summary of the \( a_0 \), \( a_1 \) and \( a_2 \) terms in accordance with the MPSR model together with the “true hardness” values and corresponding correlation coefficients.

<table>
<thead>
<tr>
<th>SiC (vol%)</th>
<th>( a_0 )</th>
<th>( a_1 )</th>
<th>( a_2 )</th>
<th>( H_{MPSR} ) (Gpa)</th>
<th>( R^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>-0.3291</td>
<td>72.72</td>
<td>7091.85</td>
<td>13.15</td>
<td>0.9995</td>
</tr>
<tr>
<td>0.3</td>
<td>0.1453</td>
<td>13.62</td>
<td>9115.74</td>
<td>16.90</td>
<td>0.9997</td>
</tr>
<tr>
<td>1</td>
<td>0.0202</td>
<td>26.28</td>
<td>9209.61</td>
<td>17.08</td>
<td>0.9997</td>
</tr>
<tr>
<td>3</td>
<td>0.0714</td>
<td>22.29</td>
<td>9364.11</td>
<td>17.36</td>
<td>0.9996</td>
</tr>
<tr>
<td>5</td>
<td>0.0725</td>
<td>18.45</td>
<td>9736.41</td>
<td>18.06</td>
<td>0.9998</td>
</tr>
<tr>
<td>10</td>
<td>0.1384</td>
<td>12.23</td>
<td>10074.92</td>
<td>18.68</td>
<td>0.9997</td>
</tr>
<tr>
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<td>29.76</td>
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</table>

(e) Physical Interpretation of the Data

Having calculated the values for \( a_1 \) and \( a_2 \) using the PSR and MPSR models, it would seem appropriate to attribute some sort of physical significance to the terms.

For \( a_2 \), two key observations were made:

1. The initial introduction of SiC nanoparticles to the alumina matrix generated a large discrepancy between the \( a_2 \) values of the monolithic Al₂O₃ and the 0.3vol%SiC nanocomposite.

2. The subsequent additions of SiC nano-dispersants produced smaller incremental increases in the nanocomposite. We believe that two interrelated mechanisms are capable of controlling this trend.

The first mechanism involves the dislocation density. In unreinforced Al₂O₃, the primary mode of deformation under external loading is basal twinning⁴², ⁴³. Here, twins initiate at points of high stress, typically on the contact surface or grain...
boundary, and grow through the grain until they hit the adjacent grain boundary. In the nanocomposite, nanoparticles prevent twins gliding whilst simultaneously acting as nucleation sites for dislocations\textsuperscript{34, 35}. This is evidenced in TEM images given in earlier publications\textsuperscript{34, 36} which show extensive basal twinning in pure Al\textsubscript{2}O\textsubscript{3}, whereas, the subsurface of the nanocomposite is dominated by dislocations without basal twins.

However, the mere presence of high dislocation densities at the surface is not enough to substantiate the previously described behaviour of the $a_2$ term. Thus, a second mechanism is necessary.

We propose that the nano-dispersion’s capacity to resist the motion of dislocations is the underlying cause of variations in the measured hardness values displayed in Fig. 1. TEM examinations have demonstrated how SiC nanoparticles impede dislocations, causing them to bow, as indicated by the arrows in Fig. 6. The critical shear stress needed for this to occur is dictated by the lattice spacing of the nanoparticles within the Al\textsubscript{2}O\textsubscript{3} matrix and controlled by the SiC particle size (inversely-proportional) and vol\% (proportional). The maximum critical shear stress needed to bypass silicon carbide nanoparticles is given by\textsuperscript{36}:

$$\Delta \tau = \frac{71.4 f^{1/3}}{(1.6 - 2 f^{1/3})r}$$

where $f$ represents the volume fraction and $r$ is the radius of the particles. Following Tabor’s approximation:

$$H \approx 3\sigma_y = 6\tau = H_0 + 6\Delta \tau$$

where $H_0$ is the true hardness of alumina and $\sigma_y$ is the yield stress. The predicted $H$ values of Eqs. 10 and 11 are plotted in Fig. 7 for dispersants sized between 25-200nm. The “true hardness” results generated by the MPSR model have been applied to determine their correlation.

If we postulate that the SiC nanoparticles are equally-sized and homogenously distributed throughout the matrix, then we would expect the “true hardness” data to fit the predictions. However, we must address the realities of ceramic powder processing. It is well known that lower vol\% SiC dispersions are far more suitable for producing a uniform distribution throughout matrix and have a higher probability of maintaining a particle size close to that of the original crystallites. Whereas, in higher vol\% dispersions, nanoparticles have a tendency to agglomerate into substantially larger, irregular clusters. This is illustrated in Fig. 6, which highlights nanoparticles of both 50-100nm and 200-300nm (circled) within the same microstructure. Consequently, the “true hardness” values derived from the MPSR model do not satisfy the predictions. Nevertheless, they act as a base from which the SiC dispersions distribution and population throughout the nanocomposite can be characterised.

For the $a_1$ term, little can be concluded based on the numerical values obtained from the PSR and MPSR models, particularly across the Al\textsubscript{2}O\textsubscript{3}/SiC. However, the randomness of the results combined with the notable difference between pure Al\textsubscript{2}O\textsubscript{3} and the nanocomposite tends to suggest that fracturing is the direct cause of the ISE. The contrasting fracture mode of the Al\textsubscript{2}O\textsubscript{3} in comparison with
the Al₂O₃/SiC could justify the large gap in the $a_1$ values and the unpredictable nature of crack initiation and crack propagation could explain the inconsistencies in the data. Future work will concentrate on validating this theory.

CONCLUSION

Hardness-load plots for ground and polished monolithic Al₂O₃ and Al₂O₃/SiC nanocomposites show that with further additions of SiC nano-reinforcements increases in the measured hardness and reductions in the severity of the ISE are to be expected. Meyer’s Law, the PSR model and the MPSR model have all been proven insufficient in determining an acceptable “true hardness”. Increases in dislocation density and the nano-dispersants capacity to resist the motion of dislocations are two mechanisms that have been proposed to explain apparent hardening of Al₂O₃/SiC nanocomposite.

REFERENCES

28 Kick, F. *Das gesetz der proportioanlen widerstände und seine anwendung*. Felix, Leipzig, Germany, 1885.