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DYNAMIC RESPONSE AND MICROSTRUCTURE OF COMMERCIAL ALUMINA

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Abstract - The dynamic response of commercial alumina discs, with an in-material manganin gauge, was determined in the 30 to 100 Kb range. The shock wave was composed of an initial elastic jump to about 60 Kb and a subsequent dispersive rise to the final shock level.

The manganin gauges were used to determine the Hugoniot curve and the spall strength of the material. Various features of the release wave were used to deduce the state of the material after passage of the shock wave.

The Hugoniot Elastic Limit could be varied from 6.0 to 7.5 GPa solely by varying the thermal history of the alumina specimens. These changes in dynamic properties were shown to correspond to microstructural changes and were well-correlated with corresponding changes in the static fracture toughness. This correlation was explained in terms of a model for dynamic yielding based on multiple unconnected microcracking on the scale of the microstructure.

1. INTRODUCTION

The dynamic response of brittle solids to impulsive loading has been the subject of many publications over the past twenty years. The article by Davison and Graham (1) reviews the important issues concerning stress-wave propagation in these materials. A single comprehensive model which will account for all the observed features is still lacking, although much progress has been achieved, for example in the thermomechanical description of Grady (2). In addition, the recent work of Munson and Lawrence (3) on the dynamic deformation of polycrystalline alumina demonstrated the dispersive nature of the wave profile above the Hugoniot Elastic Limit (HEL).

This paper presents experimental results on the dynamic behaviour of a commercial
alumina containing approximately 30% glass (AD-85, manufactured by Coors) with inmaterial manganin gauges and correlates changes in HEL with microstructural changes.

It is shown that this glass-containing alumina behaves similarly to polycrystalline alumina (3). In particular dispersive wave profiles are observed above the HEL and the leading edge of the release wave propagates at the longitudinal elastic velocity, which supports the assumption that the loss of shear strength during shock loading is only partial.

In addition, increases in dynamic yield strength (HEL) produced by heat treatment are shown to correspond to microstructural changes in the glassy intergranular phase and are associated with changes in density, elastic modulus and the state of microstress in this phase.

2. EXPERIMENTAL

2.1 Gun description

Planar impact experiments were conducted in a 64 mm gas gun (4), and 2.5" powder gun (5). Impact velocities were measured to 0.5% and ranged from 100 to 700 ms\(^{-1}\). For copper impactors and AD-85 disc targets, stresses were in the range from 20 to 100 kb. A schema of the impact assembly is shown in Fig. 1.

![Fig. 1. Schematic description of a planar impact experiment.](image)

Manganin gauges (Micro-Measurements, type LM-SS-125CH-048) were calibrated under both loading (5) and unloading (6) conditions. Gauge emplacement and data-reduction procedures were identical to those previously described (5,6). The 5 µm thick, grid-like, foil gauges mounted on a 0.04 mm epoxy sheet were embedded between two specimen discs, leaving a 0.05 mm intermediate layer of mostly epoxy resin, which has a much lower acoustic impedance than alumina. Any shock wave traversing the target reverberates in this layer several times before the stress reaches its final level. These reverberations degrade the rise time of the stress signal to of the order of 0.15 µs. The configuration shown in Fig. 2, in which the gauge records the stress-time history in a backing disc of poly(methyl-methacrylate) (PMMA), gives a much faster rise time. Since epoxy and PMMA have almost equal impedance, the gauge is effectively embedded in PMMA and the rise time of the stress signal is reduced to 5 ns (equal to a few reverberations across the gauge thickness). This configuration was also used to measure the spall strength of the alumina discs by means of a technique previously described (7).
2.2 Material specifications

The alumina discs were cut and lapped from rectangular tiles 6.2 and 12.4 mm thick. The material is mainly composed of corundum grains with some spinel (MgAl$_2$O$_4$). The grains of these two phases form a continuous network interlaced with a glassy phase. This glassy phase is of variable composition in the anorthite (Ca$_2$Al$_2$Si$_2$O$_8$) range and comprises about 30 wt.% of the total.

The static compressive strength of the material quoted by the manufacturer is 19.3 kb and its density is 3.41 g/cc. Ultrasonic measurements gave a value for the longitudinal sound wave velocity of $C_L = 8.9$ kms$^{-1}$ and the bulk and shear moduli, as given by the manufacturer, are $K = 1380$ kb and $G = 960$ kb respectively, with a Poisson's ratio of $\nu = 0.22$.

2.3 Heat treatment

The alumina discs were impacted both as-received and after three classes of heat treatment:

a. Solution-treatment (1350-1600°C) followed by fast cooling.

b. Stress relief (ageing treatment) after solution treatment (750-900°C).

c. Precipitation treatment after solution treating and ageing (1050-1200°C).

The solution-treatment eliminated minor phases and increased the alumina content of the intergranular glass. Subsequent stress-relief reduced the alumina content of the glass without causing precipitation of additional phases. Stress relief is expected to have reduced the level of internal microstress. Precipitation treatment resulted in the formation of minor silicate phases (anorthite and some cordierite) in the glassy regions.

3. RESULTS AND DISCUSSION

3.1 The Hugoniot curve

A typical gauge record based on the configuration of Fig. 1, is shown in Fig. (3). The rise-time of the stress signal is rather large (about 0.2 $\mu$s), part of which is the result of the thickness of the gauge package. However, this relatively large rise time is also due to the dispersive nature of the wave at shock levels higher than the Hugoniot elastic limit.

The final stress states, determined from the gauge calibration curve (5), together with the measured impact velocity, determine a point on the Hugoniot curve of the
material by impedance matching. The measured points and the curve drawn through them are shown in Fig. (4).

Fig. 3. A gauge record for the configuration of Fig. 1.

Fig. 4. The measured Hugoniot curve of AD-85.

The back-surface configuration shown in Fig. (2) was used to determine the elastic limit of the Hugoniot curve more accurately. A typical gauge record for this configuration is shown in Fig. (5). The stress jump to the elastic limit is followed by a dispersive wave to the final stress value. The features of the dispersive wave are very similar to those observed with the VISAR interferometer (15). The elastic wave velocity was determined from the transit time through the specimen disc. The value obtained (8.9±0.1 ms⁻¹) is in very good agreement with the ultrasonic value. The Hugoniot elastic limit (HEL) is determined from the acoustic impedances of AD-85 alumina (Z₁) and PMMA (Z₂) in the relation:
where \( \sigma_E \) is the measured elastic jump in the stress signal transmitted to the PMMA. The calculated value of \( \sigma_{\text{HEL}} \) (60±1kb) is in good agreement with previous results (8). Combining this value for the HEL with the relation \( \sigma_y = (1-2)/(1-\nu)\sigma_{\text{HEL}} \), a value of \( \sigma_y = 43 \) kb is obtained for the dynamic uniaxial yield strength (Poisson's ratio \( \nu = 0.22 \)). This value is about 2.3 times larger than the static compressive strength given by the manufacturer. A similar result was also found for Lucalox (3) where the HEL value of 91 kb correspond to a dynamic uniaxial yield stress of \( \sigma_y = 60 \) kb, while the static compressive strength of a Lucalox is only 22 kb.

![Fig. 5. A gauge record for the back-surface configuration (Fig. 2).](image)

A possible explanation is that the compressive strength of the materials is pressure dependent. Hydrostatic pressure-dependent yield strengths have been observed for many materials, especially metals. For aluminum the yield strength increases by a factor of 4 for hydrostatic pressures of up to 100 kb (9-11). We can write a linear expression:

\[
\sigma_y = \sigma_{yo} + A \cdot P
\]  

(2)

for the compressive strength where \( A \) is an empirical constant, and \( \sigma_y \) and \( \sigma_{yo} \) are the dynamic and static strengths respectively.

The hydrostatic pressure in the specimen at the HEL point can be evaluated from the relation between the longitudinal stress and pressure under uniaxial elastic strain:

\[
P = \frac{K}{K + \frac{4}{3}G} \sigma_{1D}
\]

(3)

where \( K \) and \( G \) are the bulk and shear moduli.

Thus at the HEL point, \( \sigma_{1D} = 60 \) kb, we obtain a value of \( P = 31 \) kb and from equation (2) we get \( A = 0.76 \) after using \( \sigma_y = 43 \) kb and \( \sigma_{yo} = 19.3 \) kb. The data for Lucalox (\( \sigma_y = 60 \) kb, \( \sigma_{yo} = 22 \) kb) give the same value for \( A \) (\( P = 50 \) kb at the HEL point of 91 kb).

3.2 The Spall strength

The configuration shown in Fig. 2 can be used to determine the spall strength of the specimen (7). When a free surface is generated inside the target, due to spallation, part of the relief wave reverberates between this new surface and the specimen - PMMA interface. These reverberations are recorded by the gauge at the
interface and their amplitude can be used to estimate the spall strength of the material. A similar approach was used by Johnson (12).

Fig. 6 shows gauge records from experiments with increasing impact velocities. Only for the lowest impact velocity, in which the shock stress was well within the elastic range, is there some indication of spall strength. Using a 1-D code, as previously described (7), a value of 3kb is obtained for the spall strength of AD-85 alumina in this range. This spall strength decreases with increasing impact velocity and at a shock level of 60-70kb is practically zero. These results are consistent with the values of less than 0.5kb reported for Lucalox (3).

![Gauge records](image)

Fig. 6. Spall signals from experiments with increasing impact velocity.

The very small spall strength for these materials near the HEL point is not surprising, since their static tensile strengths are also very small (1.5kb for AD-85 as reported by the manufacturer).

3.3 Release behaviour

The velocity of the leading edge of the release wave was found to correspond to the elastic longitudinal velocity. This may not be surprising for the low stress experiments, but at higher impact stresses this can be considered as evidence that the material does not completely lose its shear strength above the HEL point. The Lagrangian velocity of the leading edge of the release wave was found to be the same as the longitudinal elastic wave velocity (8.9mm/μS).

The nature of the release wave is also direct evidence that the material retains some shear strength, and the records show that the release path has an elastic part characterised by a very short falltime similar to the loading rise times. The elastic portion of the release path decreases as the shock stress increases. This can be seen by comparing the gauge record in Fig. 7 (for which the shock stress was 85kb) with that of Fig. 3 (for a shock stress of 65kb). The smaller elastic
portion of the high stress experiment indicates clearly that the residual shear strength of the material decreases after the passage of a strong shockwave.

Further confirmation for this decrease in strength can be gained by observing the Hugoniot curve itself (Fig. 4). We see that the curve departs from the elastic line, consistent with a decreasing shear strength with increasing shock amplitude. Similar observations have been reported for other ceramics (1), although in most cases the evidence for decreasing shear strength comes from a flattening of the Hugoniot and its approach to the hydrostatic compression curve.

3.4 Mechanical Properties

Values of through-thickness fracture toughness ($K_c$) and transverse rupture strength (TRS) could be correlated with the dynamic yield strength (HEL). Furthermore, these mechanical properties could be correlated with the fracture morphology (13). Fig. 9 shows the observed correlation between $K_c$ and HEL. The fracture morphology at low values of $K_c$ was characterised by a rough, inter-granular failure confined to the glassy phase. At high values of $K_c$ the fracture surface was smooth, and included extensive cleavage of alumina grains. Similar observations were made on conoid fragments recovered from projectile penetration tests: tiles exhibiting poor penetration resistance yielded conoid fragments with roughened fracture surfaces while those from tiles with good penetration resistance yielded fragments with smooth fracture surfaces (Fig. 10). Values of $K_c$ and TRS could be combined to yield an estimate of the characteristic critical crack length $C_0$.

$$\pi C_0 = (K_c^{0.5}/\text{TRS})^2$$

Maximum values of $C_0$ were also correlated with maximum values of $K_c$. Table 1 summarises these correlations and also demonstrates that heat-treatments for the highest strengths are associated with solution-treatment and rapid cooling of the tiles, while the lowest strengths correspond to fully-precipitated, equilibrated tiles.

3.5 Microstructural Changes

The as-received material contained small quantities of anorthite and cordierite, in addition to the alumina, spinel and glassy phases. After solution treatment at 1350 to 1550°C the minor precipitate phases were observed to have dissolved in the glass (Fig. 8), and both the volume fraction of glass and the alumina content of the glass increased (to 35.3%, as compared to 30.2% in the as-received material). Furthermore, the bulk density was increased by solution treatment (13) as was the elastic modulus (245 GPa, as opposed to 219 GPa for the as-received material). These results imply that solution treatment removes the minor embrittling phases, and leads to the formation of a higher density glass with an increased elastic modulus.
Stress-relief reduced the bulk density, the volume fraction of the glassy phase and the alumina content of the glass (which returned to the as-received value), but no precipitation of the minor phases was observed. Fully-precipitated tiles (1050°C heat-treatment) showed higher volume fractions of the minor phases than were observed in the as-received material. Further details of the time and temperature dependence of these changes in structure are given elsewhere (13).

4. DISCUSSION

The correlation of $K_c$ with the HEL (Fig. 9) is by no means perfect (especially for the lower values of $K_c$) and does not necessarily imply that the loss of shear strength associated with dynamic compressive yielding occurs by the same mechanism as the crack propagation which controls the toughness in tension. Neither are differences in fracture morphology, noted in specimens of high toughness (smooth morphology) as opposed to specimens of low toughness (rough surfaces), necessarily indicative of radically different mechanism of crack propagation.

Fig. 9. Correlation between $\sigma_{HEL}$ and fracture toughness.
Three distinct models of dynamic yielding in brittle materials have been proposed:

1. Viscous flow associated with local "melting".
2. Plastic deformation by dislocation multiplication and glide.
3. Multiple microcrack formation, with a corresponding increase in elastic compliance.

The theory of local "melting" was proposed to account for yielding at very high pressures. If the dynamic yield strength is controlled by local melting, it is difficult to understand how it could be affected by heat-treatment, as reported here, or how it could be correlated with fracture toughness in tension. If the primary processes responsible for dynamic yielding are dislocation multiplication and glide, it would seem equally unlikely that heat treatment should have a major effect, since the primary crystalline phase, corundum, is unaffected by heat-treatment. This leaves only the third mechanism, multiple microcracking.

The observed fracture morphologies and the calculated values of $C_\tau$, noted in the mechanical test results (Table 1), strongly suggest that in all cases the critical crack length exceeds the microstructural dimensions, and that the primary morphological differences are due to differences in the preferred crack path. In the solution-treated tiles it is clear that the glassy phase has been toughened, probably not only by the composition changes, but also by compressive microstresses. Crack propagation in these toughened tiles occurs at higher stresses and traverses both the crystalline and the glassy phases. On the other hand, the stress level for crack propagation in the fully-precipitated tiles is much lower and the crack path is confined to the intergranular regions and the glassy phase.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>$K_{C1/2}$</th>
<th>$K_{C1/2}^*$</th>
<th>T.R.S.</th>
<th>T.R.S.*</th>
<th>E</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solution treatment</td>
<td>8.0</td>
<td>7.0</td>
<td>450</td>
<td>280</td>
<td>245</td>
</tr>
<tr>
<td>Stress relief</td>
<td>4.5-6.0</td>
<td>-</td>
<td>$\sim$300</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Precipitation treatment</td>
<td>3.0-4.0</td>
<td>-</td>
<td>200-250</td>
<td>200</td>
<td>-</td>
</tr>
<tr>
<td>As received</td>
<td>3.7</td>
<td>3.7</td>
<td>230</td>
<td>198</td>
<td>219</td>
</tr>
</tbody>
</table>

*Perpendicular to cooling direction.

If the microstructural dynamic yield mechanism is basically the same as the failure mechanism under static loading, then the dynamic time to failure $\tau$ should be related to the fracture toughness. A lower limit to $\tau$ can be estimated from the relation:

$$K_C = \sigma_y d \sqrt{\pi C_L \tau_{\min}}$$

This relation assumes that the dynamic fracture toughness is identical to the static fracture toughness, and that the "time to failure" is really a critical crack length $C_\tau \tau$, where $C_L$ is the longitudinal velocity of sound. For truly brittle materials (no irreversible plastic flow) the fracture toughness should only depend on stress level if the crack velocity approaches $C_L$. 

Table 1. Summary of mechanical test results.
An alternative upper limit to \( \tau \) is obtained by assuming that the critical crack length under dynamic conditions is the same as that in static tests \( (C_0) \):

\[
C_L \tau = C_0
\]

Inserting typical values for all the parameters:

\[
K_c = 5 \text{ MPa m}^{1/2} \\
\sigma_y = 4.5 \text{ GPa} \\
C_L = 10.9 \text{ km s}^{-1} \\
C_0 = 50 \mu\text{m}
\]

We obtain estimates of \( \tau_{\text{max}} = 5 \text{ ns} \) and \( \tau_{\text{min}} = 40 \text{ ps} \). A better estimate of \( \tau \) is probably obtained by assuming that, under dynamic yield conditions, the characteristic crack size \( C_d \) approaches the microstructural dimensions, that is, the mean-free crack path is reduced to of the order of 5 \( \mu\text{m} \). Under these conditions \( \tau = 0.5 \text{ ns} \) and the effective dynamic value of \( K_c = 20 \text{ MPa m}^{1/2} \). In this model dynamic yielding results in the formation of multiple unconnected microcracks which produce a correspondingly sharp drop in effective elastic modulus. The diameter of the microcracks is fixed and corresponds to the scale of the microstructure. Microcracking is not limited by the soundwave velocity (since \( \tau > \tau_{\text{min}} \)) nor is it limited by the critical crack size in static failure (since \( C_0 < C_d \)). On the other hand, crack formation under both static and dynamic failure conditions only occurs when the tensile strength of the glassy phase is exceeded, and is therefore affected similarly by any heat treatment which affects the strength of this phase. If this model is correct, failure in dynamic compression differs from failure in static tension only in the critical size of the initial microcracks. In effect, a sufficiently detailed stochastic model of the orientation and diameter of potential microcracks should cover both cases.

A remaining question is the relation between the residual strength after passage of the compressive wave (the residual spall strength) and the microcrack damage. If the microcracks are unconnected, the residual spall strength should depend on the density of microcracks (number per unit volume) and the microcrack size. If the microcrack size under dynamic impact conditions is essentially equal to the scale of the microstructure, and is unaffected by the impact velocity (pulse pressure), then the density of the microcracks is the only variable. Higher impact pulse pressures are then expected to lead to lower residual spall strengths. However, the onset of this loss of spall strength is expected to occur at pressures below the measured HEL, since, as noted above, the microstructural dimensions are statistically distributed, so that the measured HEL should correspond to a state of general microcracking, rather than to the initial onset of damage formation. In other words, the increase in compliance necessary to give a measurable HEL is expected to correspond to microcrack spacings of the order of the microcrack diameter.

Fig. 10. Conoid fragments from an alumina tile toughened by heat treatment (smooth surface), and from an as-received tile (rough surface).
5. CONCLUSIONS

1. The dynamic response of commercial alumina (AD-85) has been monitored using in-material manganin gauges in two configurations.

2. The Hugoniot curve, the spall strength and the release behaviour are generally similar to those previously determined for Lucalox alumina.

3. The dynamic yield strength (HEL) was markedly sensitive to heat-treatment and correlated well with the fracture toughness ($K_c$).

4. Failure was primarily controlled by the strength of the glassy phase, which was a strong function of heat treatment.

5. A model for dynamic yielding based on the formation of multiple unconnected microcracks is proposed to account for all the observations, including the strong correlation between HEL and $K_c$.

6. The residual spall strength after passage of the compressive wave is predicted to be a function of the initial impact velocity (impact pressure).

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