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TRANSLUCENT Y₃Al₅O₁₂ CERAMICS: MECHANICAL PROPERTIES

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The elastic, hardness and fracture behaviour of fully dense Y₃Al₅O₁₂ ceramics doped with various amounts of either SiO₂ or MgO was studied. Al-rich inclusions, isolated large grains and coarse grained microstructures were found to be regularly present. These features have a significant influence on the mechanical properties. For 'normal', small grained microstructures typically a value of 290 GPa for Young's modulus, 18 GPa for the hardness (2 N load) and 1.7 MPa.m¹/² for the fracture toughness is obtained.

1. INTRODUCTION
Aluminium oxide ceramics are well studied and have many applications. If properly processed, translucent ceramics can be sintered from this material, usually employing MgO as a dopant (see e.g. ref. 2). Recently also the sintering of Y₃Al₅O₁₂ (YAG) powder to translucent ceramics was described with either MgO or SiO₂ as sintering aid. The dopant behaviour in YAG is the subject of an accompanying paper. As compared with translucent alumina the main virtue of YAG ceramics is a low optical absorption. While typical values for the effective absorption coefficient, b, of alumina are in the range of 1.8 to 2.2 mm⁻¹, b-values of 1.6 mm⁻¹ and 0.7 mm⁻¹ have been reported for YAG(SiO₂) and YAG(MgO) ceramics respectively. For various applications the mechanical properties are also relevant so that the elastic, hardness and fracture behaviour of various YAG ceramics were investigated.

2. EXPERIMENTAL
The materials were prepared using the wet-chemical route as described in ref. (3). Various dopant levels for SiO₂ as well as MgO were used. All sintering was done in a vacuum of about 10⁻³ Pa at temperatures between 1700 and 1800°C. The microstructure of the various ceramics was revealed by scanning electron microscopy (SEM) using fracture surfaces. All ceramics prepared contained a certain amount of Al-rich inclusions in spite of the fact that they were prepared to be strictly stoichiometric. This is probably due to the sintering procedure. The mean linear intercept, d, was used as a grain size measure. For all materials in the fine grained areas d ranged from 3 to 6 µm. The surfaces were covered with a thin gold layer to prevent electrostatic charging during examination.

The densities, ρ, of the sample were determined by Prokic's method. The longitudinal wave velocity, v₁, and shear wave velocity, v₉, were measured at 10 and 20 MHz respectively using the pulse-echo technique on specimens of at least 99.8% relative density. From ρ, v₁ and v₉ Young's modulus, E, and Poisson's ratio, ν, were calculated with the usual formulae for isotropic materials. No correction was applied for attenuation since the loss tangent was less than 0.05. The sample standard deviation was estimated to be about 2 GPa.

The Vickers hardness, Hᵥ, was measured on polished specimens. For each material the load was varied between 0.5 N and 20 N and applied for about 15 seconds. Measurements were done in oil as well as water. The average sample standard deviation using five readings was about 1.5 GPa. For comparison the Knoop hardness, Hₑ, was
also measured (2 N load, ambient). The usual formulae for the calculation of the hardness were used. Selected indentations were examined by SEM.

The fracture toughness, $K_{IC}$, was measured at various temperatures in a $N_2$ gas atmosphere (200 ppmV H$_2$O) with the three point bend test (span 12 mm, cross head speed 1.0 mm/min) using specimens of size $1 \times 3 \times 15$ mm$^3$ in an all-ceramic bending set-up. A notch with a relative depth of about 0.15 was sawn in the specimens. Pre-cracking was done by a Knoop indentation (2 N load) at the notch root on both sides of the specimens. The compliance factor was calculated as described in ref. (8). Normally three specimens were used for each $K_{IC}$ determination resulting in a average sample standard deviation of 0.1 MPa.m$^{1/2}$.

For two selected ceramics the strength, $s$, was measured in the same bending set-up, but only at room temperature. Specimens were sawn with a 300 mesh (50 µm) diamond wheel. The strength was measured in the as-machined state and after annealing at 1000°C for 2 hours in air. In each case 5 specimens were used.

3. RESULTS AND DISCUSSION

3.1. Elastic behaviour

The value of Young's modulus, $E$, is important in many applications of ceramics. As compared with Y$_2$O$_3$ and Al$_2$O$_3$, YAG is stiffer than yttria but more compliant than alumina. While $F$(Al$_2$O$_3$) is about 400 GPa$^2$, and $F$(Y$_2$O$_3$) is about 177 GPa$^2$, the average value of $E$ obtained for YAG is 290 GPa. Poisson's ratio, $\nu$, was determined to be 0.246. From the single crystal elastic constants an estimate for $E$(YAG) can be calculated using the Voigt-Reuss-Hill or Hashin-Shtrikman averaging scheme. The average values of the single crystal elastic constants given in ref. (13) are $c_{11} = 334$ GPa, $c_{12} = 112$ GPa and $c_{44} = 115$ GPa. The coefficients of variation in these mean values using the 4 experimental data sets are 0.2, 0.5 and 0.2% respectively. Averaging these data resulted in an estimated $E$-value of 285 GPa (and a $\nu$-value of 0.243). The difference between this estimate and the experimental value is small (2.5%) but significant since the error in the calculated $E$(YAG) due to the experimental uncertainties in the single crystal constants is about 1.0 GPa (0.4%). It cannot be caused by small deviations from 100% relative density since correction for this effect would result in an even larger discrepancy. One possibility is that the difference is due to the presence of the (stiffer) Al-rich inclusions which have been observed by SEM and TEM (see fig. 3 of ref. 4). Assuming the inclusions to be Al$_2$O$_3$, the 'three-phase-model' for the elasticity of composites can be used to calculate the amount of Al$_2$O$_3$ in the YAG ceramics. This estimate yields a value of about 0.4% for the volume concentration of Al$_2$O$_3$ inclusions. Since from the point count analysis of the relevant micrographs and X-ray diffraction peak intensity measurements an average value of about 0.4% is obtained, the difference between the theoretical and the experimental value seems real.

For ceramics containing more inclusions the $E$-value is significantly higher, e.g. for the 1200 wt.ppm SiO$_2$ doped material containing about 15 vol.% Al$_2$O$_3$ the value of Young's modulus is 311 GPa.

3.2. Hardness

Mechanical behaviour of ceramics is conveniently characterized by the hardness, $H$. It is defined by

$$H = kL/D^2$$

where $k$ is a dimensionless constant depending on the type of indenter, $L$ is the load and $D$ is the corresponding indentational diagonal. The interpretation of the hardness, however, is quite involved. Several effects such as the type of measurement, the load and the environment can have a significant influence. The load dependence is
often described by the so-called Meyer law:

\[ L = cD^n \]  

(2)

The parameter \( c \) is the load required to make an unit size indentation and advocated as a kind of strength measure. The parameter \( n \), which ideally has the value 2, is a measure for the load dependence of the hardness and thus of the size effect. In practice the load range used is limited on the low side by the observability of the indentation and on the high side by excessive cracking.

For the YAG ceramics at low loads (2 N) in general no cracks were observed (upper part fig. 1). At about 2 N cracking started (although not necessarily only median cracks, middle part fig. 1) and at higher loads (10 N) extensive lateral cracking was observed (lower part fig. 1). This lateral cracking is most probably due to the residual surface stresses introduced by the polishing procedure. In Fig. 2 a typical plot of hardness versus load and the corresponding Meyer plot is shown for a particular YAG ceramic. The values of \( c \) and \( n \) were determined by a weighted least-squares analysis of diagonal length versus load (the diagonal length is the

**FIGURE 1**

Vickers hardness indentation of YAG ceramics doped with 500 wt. ppm SiO₂ at 0.5 N (upper), 2.0 N (middle) and 10 N (lower).

**FIGURE 2**

Vickers hardness (in inert environment) versus load for YAG doped with 500 wt. ppm SiO₂ (upper) and the corresponding Meyer plot (lower).
The parameters c and n are quite similar for all ceramics, although somewhat higher n-values are observed for the YAG(MgO) materials and somewhat higher c-values for the YAG(SiO2) ceramics. On average an n-value of about 1.8 is obtained and this value is quite typical for ceramics. One clear exception is one of the 1000 wt. ppm SiO2 doped ceramics where a significantly higher n and lower c value were found. Inspection of the indentations revealed that in this case median cracks already developed at about 1 N load. The Al2O3 content for this ceramic is also somewhat higher than on average (table 1). The analysis of the indentations made in water yielded quite similar results and no definitive environmental effect could be observed.

While for H(Al2O3) values of about 20 GPa (1 N load) are reported, for Y2O3 a hardness value of only about 6 GPa (load unknown) is given. The H(YAG) is thus comparable to H(Al2O3) and substantially higher than H(Y2O3). The measurement of the Knoop hardness of YAG(MgO) and YAG(SiO2) was done for comparison and yielded values of 15.3 GPa and 14.3 GPa respectively (2 N load, ambient). As has been observed before this type of measurement usually results in somewhat different hardness values, in particular at low loads.

### 3.3. Fracture

Catastrophic fracture is characterized by two parameters: fracture toughness, $K_{IC}$, and strength, $s$. For a review, see ref. (17). In brief, the fracture toughness represents an inherent resistance of the material to fracture while the strength is determined both by intrinsic behaviour and the (mechanical) defect structure of the material. Both parameters are of interest and consequently are studied.

The fracture toughness at room temperature for several YAG ceramics is given in table 1. From this table it appears that the SiO2-doped ceramics have a somewhat higher toughness value than the MgO-doped materials. It also appears that at higher SiO2 dopant levels the toughness decreases but this trend is deceptive. For YAG ceramics doped with 1550 wt. ppm SiO2 no abnormal features are present in the microstructure and the $K_{IC}$ value of 1.7 MPa.m$^{1/2}$ is probably the proper value for fine grained YAG ceramics. The material doped with 500 wt. ppm SiO2 has large grains and extensive cracking throughout the specimens is present after the fracture test. This effect increases the toughness. The 1200 wt. ppm SiO2 doped ceramic contains about 15 vol.% Al2O3. Because the toughness of alumina (4.0 MPa.m$^{1/2}$) is significantly higher than for YAG this means that the material is toughened by the Al2O3 inclusions. Finally YAG ceramics doped with 2000 wt. ppm SiO2 shows discontinuous grain growth. Occasionally large grains with a size up to 1 mm can be observed on the fracture surfaces. Since the toughness of single crystals is usually substantially lower then for the corresponding polycrystalline materials, a lower overall value of

<table>
<thead>
<tr>
<th>Dope</th>
<th>n</th>
<th>c</th>
<th>$K_{IC}$ (MPa.m$^{1/2}$)</th>
<th>Al2O3 (vol%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>250 wt. ppm MgO</td>
<td>1.842</td>
<td>0.0151</td>
<td>1.87</td>
<td>11.5</td>
</tr>
<tr>
<td>500 &quot;</td>
<td>1.811</td>
<td>0.0175</td>
<td>1.66</td>
<td>0.5</td>
</tr>
<tr>
<td>1000 &quot;</td>
<td>1.817</td>
<td>0.0165</td>
<td>1.65</td>
<td>0.1</td>
</tr>
<tr>
<td>500 wt. ppm SiO2</td>
<td>1.722</td>
<td>0.0216</td>
<td>1.84</td>
<td>0.5</td>
</tr>
<tr>
<td>1000 &quot;</td>
<td>1.775</td>
<td>0.0184</td>
<td>-</td>
<td>0.3</td>
</tr>
<tr>
<td>1000 &quot;</td>
<td>1.899</td>
<td>0.0120</td>
<td>-</td>
<td>1.0</td>
</tr>
<tr>
<td>1200 &quot;</td>
<td>-</td>
<td>-</td>
<td>2.00</td>
<td>15.2</td>
</tr>
<tr>
<td>1550 &quot;</td>
<td>-</td>
<td>-</td>
<td>1.72</td>
<td>0.1</td>
</tr>
<tr>
<td>2000 &quot;</td>
<td>1.769</td>
<td>0.0189</td>
<td>1.61</td>
<td>0.2</td>
</tr>
</tbody>
</table>

n and c as defined in eq. 2
the toughness results. It should be remarked that measurements using samples from other, but similar processing cycles occasionally yielded slightly different $K_I$ values.

For the YAG(MgO) ceramics a more or less constant $K_I$ value of 1.7 MPa.m$^{1/2}$ is observed, except for the 250 wt. ppm doped material. The latter ceramic, however, contained about 11 vol.% Al$_2$O$_3$ and is consequently toughened. The value of 1.7 MPa.m$^{1/2}$ is equal to the value obtained for the 'normal' YAG(SiO$_2$). Nevertheless there is an obvious difference between the fracture surface morphology of the MgO doped materials and the SiO$_2$ doped ceramics as observed on the SEM fractographs (fig. 3).

The temperature dependence of $K_I$ of various YAG ceramics is shown in fig. 4. Surprisingly for all ceramics the value of $K_I$ initially increases with rising temperature. At still higher temperatures, however, it decreases again. The reason for the increase is not entirely clear. In debased alumina a maximum in toughness is also observed (see e.g. ref. 17). In that case the effect is attributed to additional energy dissipation in the viscous, glassy secondary phase at the temperature corresponding to the maximum in $K_I$. This explanation can be ruled out in the present case as no secondary phase is present neither at the grain boundaries nor in the triple junctions. A possible

![SEM fractograph of YAG doped with 1550 wt. ppm SiO$_2$ (upper) and 500 wt. ppm MgO (lower).](image)

![Temperature dependence of the fracture toughness of YAG doped with SiO$_2$ (upper) and MgO (lower).](image)
mechanism is as follows: the matrix is (slightly) toughened by the A12O3 inclusions themselves, but also weakened by the surrounding stress field which happens to be tensile (see fig. 4 of ref. 4). At increasing temperatures the magnitude of this stress diminishes, thus toughening the material. The decrease is probably the normally observed (decreasing) trend, due to a decrease of the Young's modulus with temperature (see appendices ref. 18). The $K_{IC}$ decrease is possibly intensified by the change from transgranular to intergranular fracture with temperature as observed with SEM.

For two ceramics the strength was measured in the as-machined state and after a 2 hour-1000°C annealing treatment (table 2). For both ceramics the strength was about 410 MPa. As compared with translucent alumina this value is somewhat higher in spite of the lower $K_{IC}$ values. A smaller flaw size is thus present. It seems likely that after the annealing treatment no residual surface stresses are present. In that case the average flaw size, $a$, can be estimated by

$$a = \frac{(K_{IC}/Y.s)^2}{3}$$

The factor $Y$ depends on the geometry of the critical flaw. Assuming a semi-circular crack, $Y$ is about 1.26. This estimate yields values of about 10 Î¼m (table 2). This value is about twice to three times the grain size. A flaw size of two to three times the average grain size was also estimated for translucent alumina. Although the toughness of alumina is significantly higher (4.0 MPa.m$^{1/2}$) the larger grain size (25 Î¼m) is the cause of a lower strength value (280 MPa). The benefit of a small grain size is thus, at least from a strength point of view, clearly illustrated.

4. CONCLUDING REMARKS

From the above discussion of the results obtained, it is clear that the microstructure of the YAG ceramics is largely dominated by two features: Al-rich inclusions and large grains which occur either isolated or as a major constituent of the microstructure. For ceramics with a more or less normal, small grained microstructure a typical value for Young's modulus of 290 GPa, a hardness value at 2 N load of 18 GPa and a fracture toughness of 1.7 MPa.m$^{1/2}$ is obtained. The precise values are, however, apart from whether the materials contain SiO$_2$ or MgO as a dopant, largely dependent on the presence of the inclusions and the large grains. Consequently, the present processing needs considerable improvement.

REFERENCES