Translucent Y3Al5O12 ceramics : mechanical properties

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TRANSLUCENT $\text{Y}_3\text{Al}_5\text{O}_{12}$ CERAMICS : MECHANICAL PROPERTIES

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The elastic, hardness and fracture behaviour of fully dense $\text{Y}_3\text{Al}_5\text{O}_{12}$ ceramics doped with various amounts of either SiO_2 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 $1.7 \text{ MPa}\cdot\text{m}^{1/2}$ for the fracture toughness is obtained.

1. INTRODUCTION

Aluminium oxide ceramics are well studied and have many applications¹. 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 $\text{Y}_3\text{Al}_5\text{O}_{12}$ (YAG) powder to translucent ceramics was described³ with either MgO or SiO_2 as sintering aid. The dopant behaviour in YAG is the subject of an accompanying paper⁴. 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(SiO_2) 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 SiO_2 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 micro-

scopy (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 measure⁵. For all materials in the fine grained areas d ranged from 3 to $6 \mu\text{m}$. 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 method⁶. The longitudinal wave velocity, v_l , and shear wave velocity, v_s , were measured at 10 and 20 MHz respectively using the pulse-echo technique on specimens of at least 99.8% relative density. From ρ , v_l and v_s Young's modulus, E , and Poisson's ratio, ν , were calculated with the usual formulae for isotropic materials⁷. 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, H_V , 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, H_K , 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_2O) with the three point bend test (span 12 mm, cross head speed 1.0 mm/min) using specimens of size $1 \times 3 \times 15 \text{ mm}^3$ 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 $0.1 \text{ MPa.m}^{1/2}$.

For two selected ceramics the strength, σ , 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_2O_3 and Al_2O_3 , YAG is stiffer than yttria but more compliant than alumina. While $E(Al_2O_3)$ is about 400 GPa ^{2,9} and $E(Y_2O_3)$ is about 177 GPa ¹⁰, the average value of E obtained for YAG is 290 GPa . Poisson's ratio, ν , was determined to be 0.246. From the single crystal elastic constants an estimate for $E(\text{YAG})$ can be calculated using the Voigt-Reuss-Hill¹¹ or Hashin-Shtrikman¹² averaging scheme. The average values of the single crystal elastic constants given in ref. (13) are $c_{11} = 334 \text{ GPa}$, $c_{12} = 112 \text{ GPa}$ and $c_{44} = 115 \text{ GPa}$. The coefficients of variation in these mean values using the 4 experi-

mental data sets are 0.2, 0.5 and 0.2% respectively. Averaging these data resulted in an estimated E -value of 283 GPa (and a ν -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 $E(\text{YAG})$ 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_2O_3 , the 'three-phase-model'¹⁴ for the elasticity of composites can be used to calculate the amount of Al_2O_3 in the YAG ceramics. This estimate yields a value of about 6.4% for the volume concentration of Al_2O_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_2O_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

$$H = k \cdot L \cdot D^{-2} \quad (1)$$

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 :

$$L = c \cdot D^n \quad (2)$$

The parameter c is the load required to make an unit size indentation and advocated as a kind of strength measure¹⁵. 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.

FIGURE 1

Vickers hardness indentation of YAG ceramics doped with 500 wt. ppm SiO_2 at 0.5 N (upper), 2.0 N (middle) and 10 N (lower).

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 2

Vickers hardness (in inert environment) versus load for YAG doped with 500 wt. ppm SiO_2 (upper) and the corresponding Meyer plot (lower).

dependent variable, for an extensive discussion see ref. (15)). The results for all ceramics analysed are presented in table 1.

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 $H(Al_2O_3)$ values of about 20 GPa (1 N load¹⁶) are reported, for Y₂O₃ a hardness value of only about 6 GPa (load unknown¹⁰) is given. The $H(YAG)$ is thus comparable to $H(Al_2O_3)$ and substantially higher than $H(Y_2O_3)$. 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 before¹⁶ 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 thus 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 upto 1 mm can be observed on the fracture surfaces. Since the toughness of single crystals is usually substantially lower than for the corresponding polycrystalline materials, a lower overall value of

TABLE 1
Hardness behaviour and fracture toughness of YAG ceramics in inert environment

dope	n	c	K_{IC} (MPa.m ^{1/2})	Al ₂ O ₃ (vol%)
250 wt. ppm MgO	1.842	0.0151	1.87	11.5
500 "	1.811	0.0175	1.66	0.5
1000 "	1.817	0.0165	1.65	0.1
500 wt. ppm SiO ₂	1.722	0.0216	1.84	0.5
1000 "	1.775	0.0184	-	0.3
1000 "	1.899	0.0120	-	1.0
1200 "	-	-	2.00	15.2
1550 "	-	-	1.72	0.1
2000 "	1.769	0.0189	1.61	0.2

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_{IC} values.

For the YAG(MgO) ceramics a more or less constant K_{IC} value of $1.7 \text{ MPa}\cdot\text{m}^{1/2}$ is observed, except for the 250 wt. ppm doped material. The latter ceramic, however, contained about 11 vol.% Al_2O_3 and is consequently toughened. The value of $1.7 \text{ MPa}\cdot\text{m}^{1/2}$ 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 fractograph's (fig. 3).

The temperature dependence of K_{IC} of various YAG ceramics is shown in fig. 4. Surprisingly for all ceramics the value of K_{IC} initially increases with rising temperature. At still higher temperatures, however, it decreases again. The reason for the increase is not enti-

rely 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_{IC} . This explanation can be ruled out in the present case as no secondary phase is present⁴ neither at the grain boundaries nor in the triple junctions. A possible

FIGURE 4

Temperature dependence of the fracture toughness of YAG doped with SiO_2 (upper) and MgO (lower).

FIGURE 3

SEM fractograph of YAG doped with 1550 wt. ppm SiO_2 (upper) and 500 wt. ppm MgO (lower).

mechanism is as follows : the matrix is (slightly) toughened by the Al₂O₃ 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

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

The factor Y depends on the geometry of the cri-

TABLE 2
Strength and flaw size of YAG ceramics.

dope : 500 wt. ppm MgO					
cond.	s (MPa)	K_{IC} (MPa.m ^{1/2})	d (μm)	a (μm)	a/d
as-sawn	410(36)	1.66	3.4	10.0	2.9
annealed	433(34)	"	"	9.3	2.7
dope : 1200 wt. ppm SiO ₂					
cond.	s (MPa)	K_{IC} (MPa.m ^{1/2})	d(μm)	a(μm)	a/d
as-sawn	412(47)	2.00	5.6	14.8	2.6
annealed	479(48)	"	"	11.0	2.0

as-sawn : sawn surface using 300 mesh diamond
annealed: 2 hours at 1000°C in air
standard sample deviation given in parentheses
d : grain size, a : flaw size

cal flaw. Assuming a semi-circular crack, Y is about 1.26¹⁸. This estimate yields values of about 10 μm (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 alumina⁹. Although the toughness of alumina is significantly higher (4.0 MPa.m^{1/2}) the larger grainsize (25 μm) is the cause of a lower strength value (280 MPa). The benefit of a small grainsize 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 SiO₂ or MgO as a dopant, largely dependent on the presence of the inclusions and the large grains. Consequently, the present processing needs considerable improvement.

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