Physics and Technology of Transparent Ceramic Armor: Sintered Al_2O_3 vs Cubic Materials

Andreas Krell, Thomas Hutzler, Jens Klimke
Fraunhofer Institute of Ceramic Technologies and Sintered Materials
Winterbergstrasse 28, 01277 Dresden, Germany

Andreas.Krell@ikts.fraunhofer.de

ABSTRACT

Sintered sub-µm alumina (α-Al_2O_3) is the hardest transparent armor. However, its trigonal structure gives rise to a strong thickness effect that makes thicker components translucent. Cubic ceramics (no birefringence!), on the other hand, are less hard but may exhibit an improved transmission.
Known cubic armor ceramics are manufactured in a way resulting in coarse microstructures with low hardness. Therefore, a cubic sub-µm spinel was developed here that associates a high hardness > 14 GPa with an in-line transmission approaching the theoretical limit - with the consequence that the thickness effect becomes very small, and transparent components can be manufactured with several mm thickness without loss of transmissivity. Additionally, weight benefits at high protective strength are expected because the hard components can be designed thinner than using known spinel or AlON grades.

1 INTRODUCTION

The central difficulty in the development of transparent armor is that some *thickness* of the component is imperative for obtaining a high protective strength whereas, on the other hand, light transmission decreases and specific weight increases with increasing thickness. The final goal is, therefore, a transparent material the high ballistic mass efficiency of which enables the design of *thinner* windows.
This ballistic mass efficiency scales with the hardness of ceramic armor [1]. Sintered Al_2O_3 with sub-µm grain size is the hardest of all transparent armor (including sapphire). With a maximum mass efficiency that may outperform even (opaque) SiC/B_4C composites [2] it is one of the most promising candidates for advanced developments. However, the birefringence of the trigonal (rhombohedral) crystallites contributes to light scattering in a way that transmission is subject of a strong thickness effect.
On the other hand, birefringence does *not* apply to *cubic* ceramics, and a transmission of 80-82 % was reported at 1 mm thickness for sintered yttria (Y_2O_3) [3] and for sintered Y-Al and Yb-Al garnets [4]. These are, however, expensive materials. Costs are lower for spinel ceramics ($MgO \cdot Al_2O_3$) [5][6][7][8][9] and possibly for AlON [10], but often their transmittance is surprisingly low [9][10] and/or ill-defined [5][6][7][9][10]. Also, known cubic ceramics suffer shortcomings with respect to their protective strength:

(i) At equal relative density and grain size they exhibit a lower hardness than Al_2O_3.
(ii) The hardness is *further* decreased because of *large* grain sizes $\geq$ 5 µm which grow during sintering at *high temperatures* (used to eliminate last light scattering pores: a high in-line transmission of ~ 60 % at 2 mm thickness was measured with a small aperture of ~ 0.2° just for the *coarsest* grades of spinel with grain sizes > 150 µm [8]!). Therefore, known spinel and ALON ceramics typically exhibit coarse microstructures with grains > 100 µm and a low hardness (a 2-kg Knoop hardness of 12.1 GPa was reported for an $MgO \cdot Al_2O_3$ spinel,[1] and 13.8 GPa for an AlON[2] [11]).

It was, therefore, the objective of the present work with sintered alumina ceramics and with ceramics of the cubic system to investigate *which* (thickness-dependent) *transmission* can be associated with a *minimum grain size* for enabling a maximum hardness and resulting in a high protective strength.

[1] Raytheon Company, Lexington (MA), USA.
[2] RCS Technologies, Inc., Research Triangle Park (NC), USA.

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2 PHYSICAL BACKGROUND

2.1 Light transmission through polycrystalline ceramics, implications for nano-technology

Fig. 1 reviews the processes of light transmission through non-cubic polycrystalline ceramics assuming that the *single* crystalline *material* is highly transparent (= *low absorption* as e.g. in the case of single crystalline corundum [sapphire, α-Al_2O_3]). Whereas the *total* forward transmission includes contributions from both scattered and unscattered light, only the latter enables optical imaging. For this *in-line* transmission of light two contributions to scattering losses have to be considered:

1. In *all* translucent or transparent materials (independent of their crystal structure) *diffuse scattering* takes place at the sites of second phases with different refractive index (pores, particles of additives or impurities).
2. In microstructures of optically non-isotropic crystals *birefringence* splits the beam on each cross-over from one grain to the next. This second contribution to scattering losses of the in-line transmission applies, therefore, to *non-cubic* ceramics only (e.g. Al_2O_3).

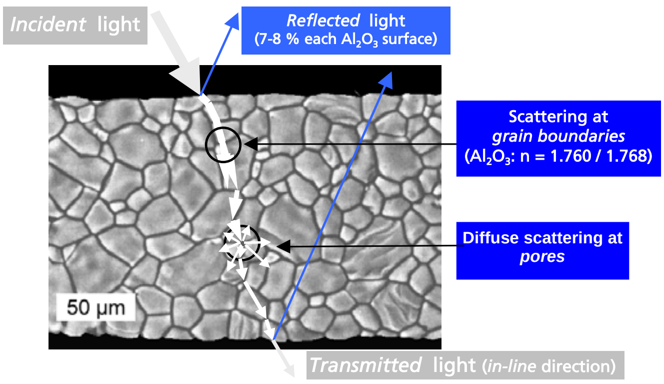
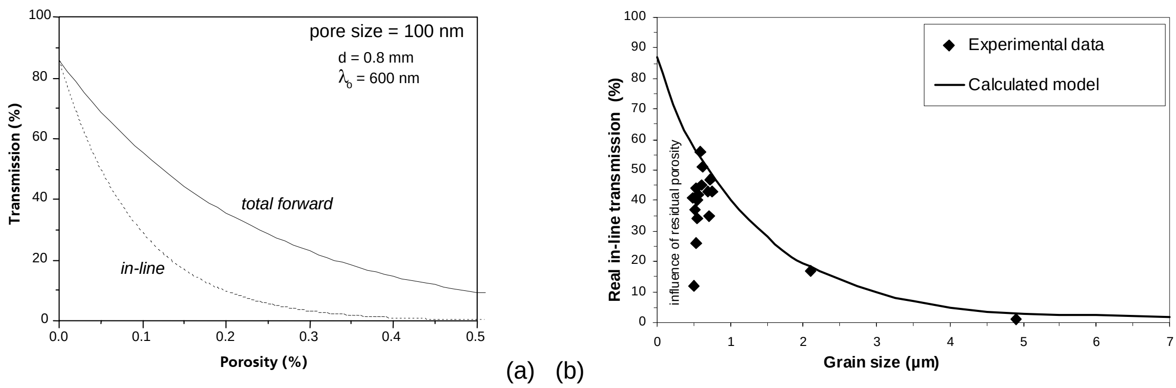


Figure 1: Light transmission through polycrystalline alumina (Al_2O_3; example: coarse [translucent] grade).

The influences of porosity and grain sizes have been evaluated recently [12] and are given by Fig. 2 together with experimental data [13]. Obviously, in *all* (non-cubic or cubic) ceramics real transparency (i.e. a high in-line transmission) needs the elimination of last *hundredths* (!) of percents of porosity, and this is the reason why common "high-tech" alumina ceramics which are considered "dense" at a relative density of e.g. 99.7 % are opaque (white when produced with a high purity and sintered in air).



(a) (b)

Figure 2: Influences of porosity and grain size on the light transmission through Al_2O_3
The porosity influence (a) was calculated for a homogeneous material, and the calculated grain size effect (b) refers to zero porosity [12]. Lower experimental values at sub-µm grain sizes [13] represent small [not measurable!] porosities < 0.1 %.

As a consequence of the extreme difficulty of eliminating last *hundredths* of porosity percents, most approaches to translucent ceramics use *high* sintering temperatures associated with extended grain growth. As a result, *coarse* products like the (non-cubic) Al_2O_3 of Fig. 1 (sintered at 1800-1900 °C) are obtained

with a porosity that may be sufficiently low for transparency but which, in fact, are translucent only with an in-line transmission < 10 % because of birefringent scattering losses (cp. the grain size effect in Fig. 2b). On the other extreme, *still larger* grains > 500 µm are needed to approach the high transmission of single crystals - even grain sizes of 200 µm increase the transmission to only 15-20 % [14].
Therefore, the development of *transparent* sintered corundum ceramics with a high *in-line* transmission needs a minimum of birefringent contributions to scattering (Fig. 1). For this end, the birefringent crystallites of the ceramic have to be so small that they are not "seen" by the light. This occurs at small grain sizes comparable with or smaller than the wavelength of the light (Fig. 2b).

It has been suggested that such most fine-grained microstructures could be obtained preferentially by using the most fine-grained nanopowders which, because of their strong surface curvature, exhibit highest surface diffusion and evaporation/condensation activities promoting densification. However, increased diffusion on strongly curved surfaces is a feature of the *individual* particle only, whereas a "densification activity" is governed the interaction of *a multitude* of particles. Unfortunately, the high surface curvature of nanoparticles is also a strong driving force for agglomeration, and the resulting inhomogeneous mutual coordination of particles and of the pore distribution considerably *retards* densification [15].
Therefore, only an extremely homogeneous particle coordination in the shaped "green" (still not sintered) bodies can provide the extremely high sintering density (imperative for transparency) *at lowest sintering temperatures* (preventing grain growth), and detailed investigations have shown that it is *not* the *most fine-grained nano*powder which associates the finest sintered microstructure with the *highest density*. Instead, it is the state of the development of *defect-free powder processing* technologies which determines an *optimum* powder particle size for a successful production of nanoceramics with highest density [16][17].

Because of the absence of birefringent scattering the grain size effect does *not* apply to *cubic* ceramics, and high-temperature sintering might be expected to give *clear transparent* microstructures even when the grain sizes grow large. Examples of coarse (grain size > 150 µm) and highly transparent spinel are really known [8], but beyond the general deficit of their *low hardness* there is also an observation of often surprisingly *low transmission* characteristics of such coarse cubic ceramics (e.g. AlON [10], spinel [9][18], Y-Al garnet [19], Y_2O_3 [20]).
The reason of the latter issue is that extended grain growth frequently gives rise to the inclusion of pores into the growing crystals - with the result of a significant residual porosity which is hard to eliminate. Therefore, high temperatures are not a generally valid means to obtain least porosity (and high transparency), and this conclusion provides the motivation for introducing the nanotechnological experiences of the alumina line [13][17] into the development of new spinel as to be outlined below (§3).

2.2 Resulting measurement needs for a quantitative characterization of transparency

In §2.1 a small grain size was claimed as an essential for obtaining *transparent* non-cubic sintered ceramics. From a physical point of view this same feature also means:
- In *non-cubic* ceramics the benefit of small grain sizes is identical with the statement that at fixed grain size the transmission will increase with the wavelength.
- This grain size related dependence on the wavelength will *not* apply for dense *cubic* sintered ceramics (or for single crystals).[1]

Additionally, the wavelength is of course important for understanding absorption (e.g. in the far IR or UV ranges).
Therefore, any experimental transmission result needs a given reference to the wavelength.

Similarly important is the geometric characterization of the measuring equipment. Obviously, optical imaging through windows or lenses needs the transmission of defined beams. Therefore, only the *in-line* transmission of *un*scattered light provides the right quantitative characterization of *transparency* (different

[1] Note, however, that the wavelength may become important here as well as in non-cubic ceramics if the size of residual pores interferes with the wavelength (cp. [12] for the effect of pore *sizes*).

from translucency [the degree of which is given by the *total* forward transmission including contributions from both scattered and unscattered light]). Unfortunately, two difficulties arise:
- A strict "in-line" measurement is difficult since it would require a zero opening aperture[1] between specimen surface and detector, and this would give an intensity record close to zero.
- On the other hand, many "in-line" or "linear" transmission results in the literature are, in fact, meaningless because they are obtained with standard photospectrometers with large apertures (3°-5°).[2]

Therefore, transmission data published without a reference to the used aperture are invalid for comparison. Without a common standard, an aperture of about 0.5° was repeatedly used in the past years to give, on the one hand, a recorded intensity which is sufficiently high for the requested accuracy of the measurement while approaching, on the other hand, the desired condition of a "real" in-line transmission [12][13][21].

Of course, images of "clearly seen" printed matter under an overlaying thin plate of "transparent" armor do not give the least indication of the real character of light transmission: in this kind of presentation *both* in-line *and* scattered transmission contribute to the seen result, and identically "clear" images are obtained with *transparent* as also with only *translucent* materials.
It is, therefore, impossible, to distinguish transparent from poorly translucent materials by such images.

Another influence is the surface state which should be carefully polished to avoid diffuse reflection losses.

When comparing *different materials* as Al_2O_3, Y_2O_3 and others, the recorded transmission is also affected by the different reflection losses on the two surfaces of a window.[3] However, the resulting theoretical (maximum) transmission T_{th} is similar for all of the ceramics addressed here (at small influence of wavelength): 85.8 % for Al_2O_3, 86.9 % for $MgO \cdot Al_2O_3$ spinel, 85.3 % for Y_2O_3, 84.1 % for an Al-Y garnet.

2.3 Relative transmission and thickness effect

With *wavelength*, measuring *aperture* and *surface state*, a fourth important parameter that influences the results of transmission measurements is the specimen *thickness*. The existence of this effect is trivial but is addressed here separately because of an important technical consequence for applications:
Because of the thickness effect, only materials with a real in-line transmission[4] *close to the theoretical maximum* T_{th} can be enlarged in thickness without a greater loss of transmission.
Or vice versa: *all* components with a real in-line transmission $T < T_{th}$ suffer a loss ($T_{th} - T$), and this loss will increase with the thickness of such components.

It is, therefore, important to understand, that e.g. a 1 mm thick polycrystalline Al_2O_3 window with an (extremely high) real in-line transmission of 70 % (measured e.g. at 640 nm wavelength) may well give the impression of an apparently "fully" transparent component. Nevertheless, there are still about 16 % of scattering loss ($T_{th} \approx 86$ %), this loss will increase with the thickness, and at a thickness of e.g. about 5 mm *this same material* will exhibit a real in-line transmission < 30 % and will become translucent.
Thus, as far as a measured real in-line transmission is $< T_{th}$, the characterization as transparent or translucent is *not* a real *materials* property but *depends on the thickness*.

An important consequence for conclusions about chances of future products is:
any statement like "*real in-line transmission* $< T_{th}$" is identical with the fact of a significant *thickness effect* which makes the manufacture of *transparent* windows impossible beyond some critical thickness.

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1 Aperture = half of total opening angle.

2 Note that doubling the aperture from e.g. 2.5° to 5 ° gives a 4fold area and may, therefore, increase the "measured" "in-line" transmission up to this factor of 4.

3 Reflection amounts $\{(n-1)/(n+1)\}^2$ for one surface; n - refractive index.

4 To be measured with a sample which should not be too thin.

3 EXPERIMENTS

3.1 Materials preparation

Sintered Al_2O_3 windows of 100 x 100 mm^2 size where manufactured from a commercial powder (purity 99.995 %, particle size 150-200 nm, specific surface 14 m^2/g) by a defect-avoiding gelcasting process followed by pre-sintering and hot-isostatic post-densification (HIP) as published previously [13] and reported on a foregoing meeting [17]. All alumina samples were doped with 0.03 wt.-% MgO.

The key to a successful combination of
- *highest density* (avoiding last hundredths of percents of residual porosity)
- obtained *at lowest sintering temperatures* < 1300 °C for the sake of *smallest grain sizes*

is a processing approach which provides a maximum *homogeneity of particle coordination* in the "green" (to be sintered) bodies by avoiding any agglomeration of the corundum particles not only during the process of slurry dispersion but up to final consolidation of the green components [13][17]. Fig. 3 displays the microstructural meaning of this improved "homogeneity": compared with dry uni-axial or with cold isostatic pressing the optimized gelcasting process gives green bodies with particles packed so closely that a most narrow pore size distribution is obtained which enables densification at lowest temperatures.

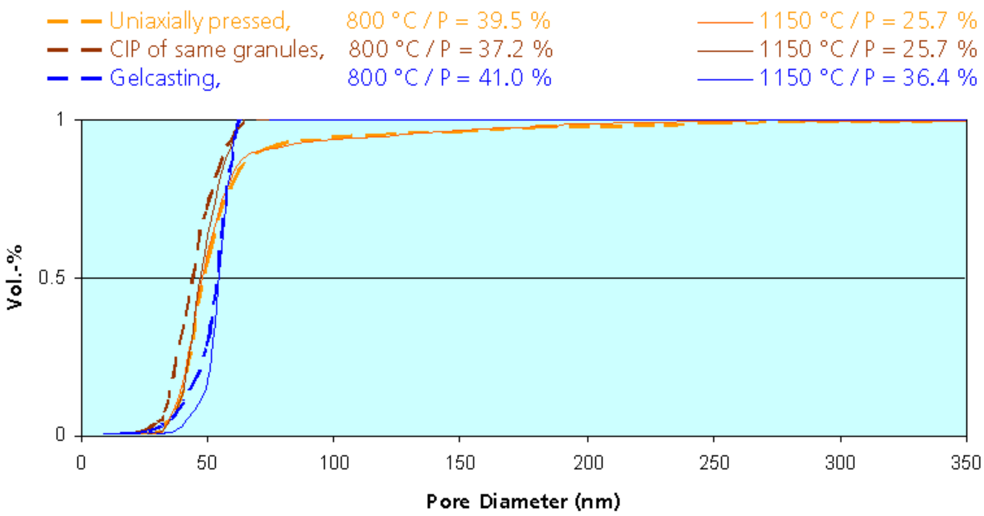

Figure 3: Pore size distributions of Al_2O_3 bodies formed by different shaping approaches from an Al_2O_3 powder with particles of about 150-200 nm.

Samples heated to 800 °C (applied with minor shrinkage in order to obtain some handling stability for Hg porosimetry) are representative for the "green" state obtained by shaping; the small differences of medium pore sizes are insignificant.

Beyond *microstructural* influences the objective of highly transparent materials depends also on a low *absorption* (= low frequency of point defects [colour centres] in the crystal lattice). With the broad homogeneity range of the $MgO \cdot Al_2O_3$ phase, the *purity* of raw materials and processes appears even more stringent for the spinel manufacture than with alumina. Unfortunately, the availability of commercial spinel raw powders is rather limited compared with alumina, and regarding the preference to *high purity* processing a stoichiometric and 99.99 % pure spinel was selected.
This powder exhibits a particle size of about 100 nm and about 28 m^2/g specific surface and turned out too fine for the casting technology as used for alumina (half of the particle size = doubled number of interparticle gaps per volume = low solid loading of casting slurry). The spinel powder was, therefore, dispersed in water for complete desintegration of agglomerates, freeze-dried and shaped by isostatic pressing at 700 MPa. After pre-sintering and HIP, the size of first manufactured samples was 60 x 40 mm^2.

Tab. 1 gives sintering temperatures and resulting grain sizes for the produced alumina and spinel tiles, and Fig. 4 shows the microstructures. While sintering additives can be used to give smaller *spinel* grain sizes by means of lower sintering, a small Zr additive significantly *in*creases the sintering temperature of *alumina* resulting, nevertheless, in a smaller grain size which improves the transparency [13].

Table 1: Sintering conditions and resulting grain sizes at relative density > 99.9 %.

Transparent ceramics	Powder: Particle size - Spec. surface	Shaping approach	Green density	Sintering + HIP temperatures	**Grain size** (sintered body)
Sintered **corundum (Al_2O_3)**	150...200 nm - 14 m^2/g	Gelcasting	~ 59 %	1255 °C + 1200 °C 1310 °C + 1280 °C	0.6 µm 0.5 µm #
Sintered **spinel** ($MgO \cdot Al_2O_3$)	~ 100 nm - 28 m^2/g	Isostatic pressing	57 - 58 %	1455 °C + 1420 °C 1270 °C + 1260 °C	0.8 µm 0.5 µm *

doped with ~ 0.2 wt-% ZrO_2 (additionally to + 0.03 % MgO)
* reduced manufacturing temperatures by sintering additive

3.2 Results: Optical and mechanical properties

Associated with the small grain sizes at high densities > 99.9 %, all of the new transparent ceramics exhibit high mechanical parameters (Tab. 2). Whereas the strength (4-pt bending) of the sub-µm spinel is on a common level, its hardness is remarkably improved compared with the present semi-commercial state of 12 GPa (Knoop hardness at 2 kg testing load) [11], and it is also beyond the data of known transparent AlON ceramics [11].
These results provide a straightforward basis for the ballistic investigations which are currently under way.

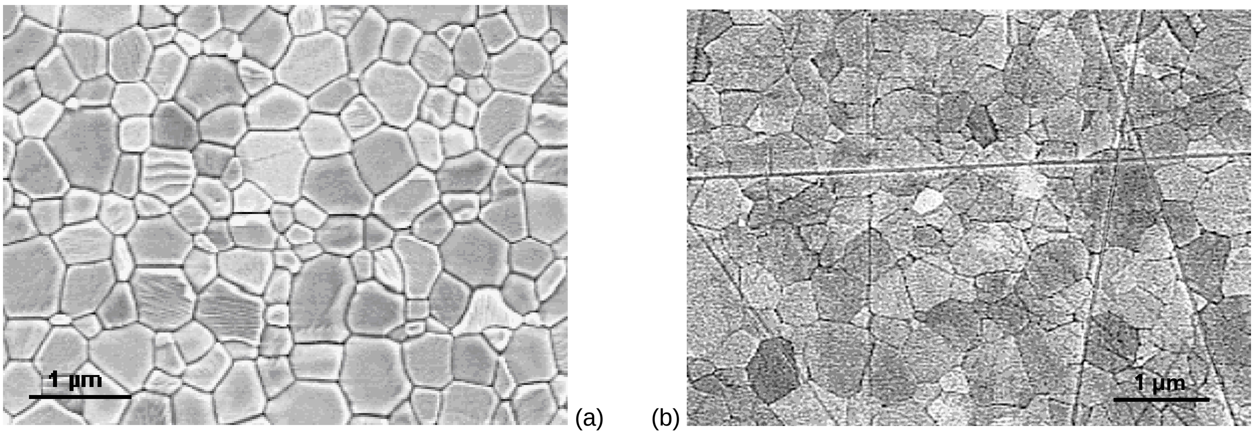

Figure 4: Microstructures of (a) transparent Al_2O_3 after HIP 1200 °C and of (b) spinel HIP-ed at 1260 °C.

Table 2: Mechanical properties and real in-line transmission (640 nm wavelength, 0.8 mm thick samples).

Transparent ceramics	Grain size (sintered body)	**Vickers hardness** (10 kg testing load)	**Strength** (4-pt bending)	**Transmission** ("real" in-line)
Sintered **corundum** (Al_2O_3)	0.6 µm 0.5 µm	20.0 GPa 20.4 GPa	709 ± 49 MPa 585 ± 77 MPa	58 % 62 %#
Sintered **spinel** ($MgO \cdot Al_2O_3$)	0.8 µm 0.5 µm	14.2 GPa 14.3 GPa	200 ± 30 MPa n.d.	84 % 83 %*

doped with ~ 0.2 wt-% ZrO_2 (additionally to + 0.03 % MgO)
* reduced manufacturing temperatures by sintering additive

In Tab. 2, the obtained real in-line transmission of the sintered sub-µm alumina is among the highest values ever obtained for such Al_2O_3 ceramics. These results are enabled by the combination of an extremely high sintered density > 99.9 % with the small grain sizes.

On the other hand, the strong grain size effect caused by birefringent scattering losses in alumina (Fig. 2b) gives rise to the strong dependence of the transmission on the wavelength shown in Fig. 5a.
In the cubic spinel, however, the small influence of the wavelength through the visible and up to the IR range (Fig. 6a) is just another manifestation of the same physical principle which eliminates the grain size effect in the cubic material. As a result, compared with the sintered alumina still higher in-line transmissions close to the theoretical limit are obtained with spinel grain sizes of 0.8 µm as with 0.5 µm (Tab. 2).

An important *technical consequence* of the optical data of Tab. 2 and of Figs. 5a/6a is illustrated by Figs. 5b and 6b.

- The sintered sub-µm alumina is *transparent* at 0.8 mm thickness, but scattering losses of (T_{th} - T) ≈ 26 % (T_{th} ≈ 86 %) grow with the thickness, and beyond some size > 1 mm the *same material* becomes translucent (Fig. 5b).
  Therefore, a technology is currently under development to further increase the transmission of this alumina for armor applications.
- On the contrary, the measured transmission of the new sub-µm spinel is close enough to the theoretical limit of 87 % that tiles with a thickness of several millimeters were obtained which still appear fully transparent (Fig. 6b).

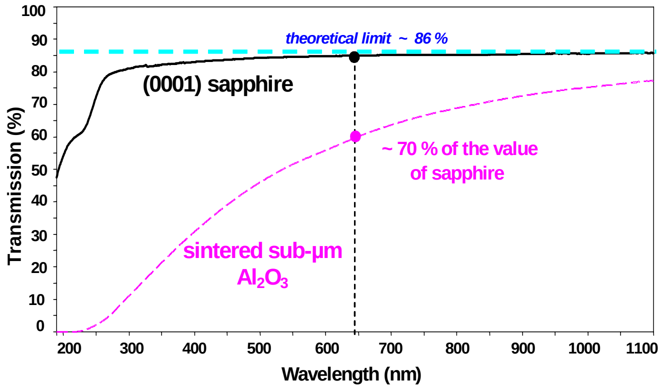

**Figure 5a: Strong dependence of transmission through sintered sub-µm Al_2O_3 on the wavelength [2] - another consequence of the strong *grain size effect* displayed in Fig. 2b.
The results are given for 0.8 mm thickness.**

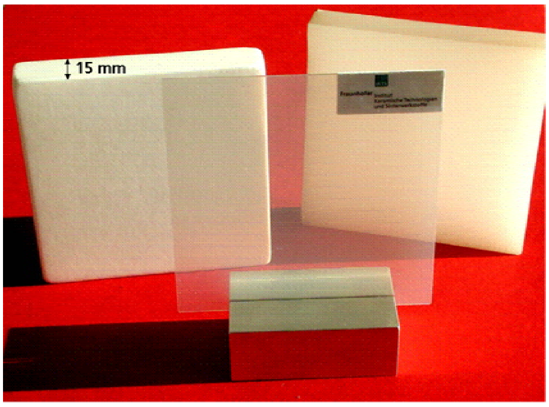

Figure 5b: High transparency of sintered sub-µm Al_2O_3 with real in-line transmission ~ 60 % at 0.8 mm thickness (central sample). The *same material* is *translucent* at larger thickness (right), it becomes white (opaque) when the residual porosity is 0.1-0.5 % (left).

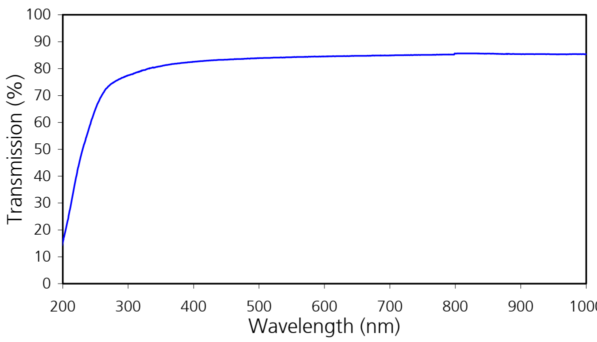

**Figure 6a: Small dependence of transmission through sintered sub-µm spinel on the wavelength in the visible range
(data for 0.94 mm thickness).**

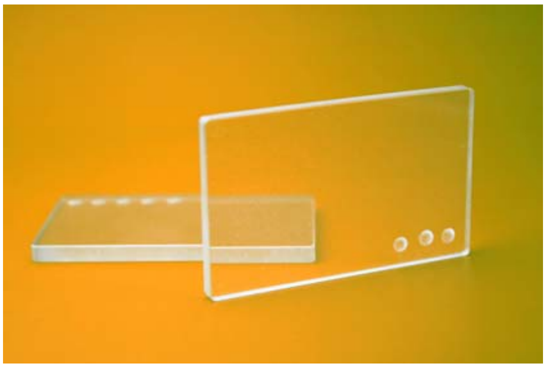

Figure 6b: High transparency of sub-µm spinel at larger thickness (here: 3 mm), enabled as a consequence of the real in-line transmission approaching the theoretical limit.

3.3 Critical thickness of transparent sintered alumina: a ballistic check

With the results of the foregoing paragraph the questions arises about the potential applicability of *thin* components of transparent Al_2O_3 armor. Obviously, the one solution to the limited transmission at larger thickness is a use in composite windows with conventional glass backing, and ballistic tests had to show the degree of the possible reduction of the specific area weight.

On penetrating glass[1] with 4 mm polycarbonate backing, the *ball round* projectiles (*lead* core) were heavily deformed, eroded and fractured into small pieces. This observation indicates a relatively high protective strength of these targets without a ceramic front tile.
On the other hand, one 0.8 mm *thin* tile of (at this thickness *highly transparent*, Fig. 5b) sintered Al_2O_3 enables a significant reduction of the specific area weight by about 25 % (Tab. 3). Thicker tiles of this ceramic did not provide much additional progress, and even with several millimeters thickness the specific area weight was not reduced by more than 30-35 %.

Table 3: Mass-reduction of composite windows (Al_2O_3/glass/polycarbonate) obtained by Al_2O_3 front-plates of different thickness [22][23].

Transparent ceramics	Ball round ammunition 7.62 mm x 51 DM 41 (v_{hit} = 833 ± 10 m/s)	Armor piercing round ammunition 7.62 mm x 51 AP FNB93 (v_{hit} = 850 ± 15 m/s)
Thinnest thickness for weight reduction $\Delta m/m$	**0.8 mm** → $\Delta m/m \approx$ **- 25 %**	**(1.25 mm** → $\Delta m/m \approx$ **- 25 %)$_{\mathbf{extrapolated}}$** **1.3 mm** → $\Delta m/m$ = **- 34 %** **1.5 mm** → $\Delta m/m$ = **- 49 %**
Highest mass effectivity E_m observed at thickness d	E_m = 1.3 ($\Delta m/m \approx$ - 32 **%)** at d = 2 mm	E_m = 2.9 ($\Delta m/m \approx$ - 66 **%)** at d = 4 mm

The penetrating impulse of *armor piercing round* (*steel* core) ammunition is much more powerful (at same velocity), and the thickness of the heading sintered sub-µm alumina tile needs to be in the range of 1.25 mm for absorbing as much of energy that the total specific area weight of the composite window is reduced by again 25 %.
Here, slightly thicker tiles of 1.5 mm enable a great additional benefit and reduce the area weight by as much as 50 %. At this thickness, however, an in-line transmission of 60 % measured at 0.8 mm thickness is decreased to 44 % only, and future efforts are required for further improving the transparency of this sintered alumina as outlined above.

4 CONCLUSIONS

The "real" in-line transmission (to be measured with a defined and small opening aperture of ~ 0.5°) is a most powerful parameter for a quantitative assessment of clear transparency.
Other published data obtained e.g. by using standard photospectrometers with larger aperture are as meaningless as photo "evidences" from images of printed matter under a directly overlaying window (an arrangement which is unable to distinguish *translucent* from *transparent* behaviour).

There are good chances for a development of highly transparent armor components of hard and high-strength sintered sub-µm alumina (corundum, α-Al_2O_3) for applications where *thin* components (~ 1 mm) can provide a sufficiently high protective strength depending on the specific threat and vehicle requirements: in the case of protection against ball round ammunition e.g. as few as ~ 1 mm of Al_2O_3 reduces the

[1] 70-75 % SiO_2 / 15 % Na_2O / 4-10 % CaO, density 2.5 g/cm^3, Young's modulus 79 GPa, Knoop hardness $HK_{0.4}$ 4.74 GPa

specific area weight of a composite alumina/glass/polycarbonate window by about 25-30 % compared with standard glass/polycarbonate.
Research activities are, therefore, under way to further improve the transparency of these transparent grades of sintered alumina.

For thicker components, however, birefringent scattering losses turn the sintered Al_2O_3 translucent even if the grain sizes are < 0.5 µm. As an alternative, cubic polycrystalline ceramics like spinel ($MgO \cdot Al_2O_3$) have been known for many years to exhibit high transmission properties independent of the grain sizes but are available with coarse microstructures and a resulting low hardness only [11]. Therefore, sintered spinel grades with a real in-line transmission close to the theoretical maximum were developed here for a first time with average grain sizes of as small as 0.5 µm enabling a high macro-hardness of 14-15 GPa as an important prerequisite of a high protective strength.
First ballistic tests with these new grades of transparent spinel are currently under way.

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