Novel Translucent and Strong Submicron Alumina Ceramics for Dental Restorations

Journal of Dental Research
2018, Vol. 97(3) 289–295
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for Dental Research 2017
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sagepub.com/journalsPermissions.nav
DOI: 10.1177/0022034517733742
journals.sagepub.com/home/jdr

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Abstract

An ideal ceramic restorative material should possess excellent aesthetic and mechanical properties. We hypothesize that the high translucency and strength of polycrystalline ceramics can be achieved through microstructural tailoring. The aim of this study is to demonstrate the superior optical and mechanical properties of a new class of submicron grain-sized alumina ceramics relative to the current state-of-the-art dental ceramic materials. The translucency, the in-line transmission (T_{IT}) in particular, of these submicron alumina ceramics has been examined with the Rayleigh-Gans-Debye light-scattering model. The theoretical predictions related very well with the measured T_{IT} values. The translucency parameter (TP) and contrast ratio (CR) of the newly developed aluminas were measured with a reflectance spectrophotometer on a black-and-white background. For comparison, the T_{IT} , TP , and CR values for a variety of dental ceramics, mostly measured in-house but also cited from the literature, were included. The flexural strength of the aluminas was determined with the 4-point bending test. Our findings have shown that for polycrystalline alumina ceramics, an average grain size <1 μm coupled with a porosity level <0.7% could yield translucency values (T_{IT} , TP , CR) similar to those of the commercial high-translucency porcelains. These values are far superior to the high-translucency lithium disilicate glass-ceramic and zirconias, including the most translucent cubic-containing zirconias. The strength of these submicron grain-sized aluminas was significantly higher than that of the cubic-containing zirconia (e.g., Zpex Smile) and lithia-based glass-ceramics (e.g., IPS e.max CAD HT). A coarse-grained alumina could also reach a translucency level comparable to that of dental porcelain. However, the relatively low strength of this material has limited its clinical indications to structurally less demanding applications, such as orthodontic brackets. With a combined high strength and translucency, the newly developed submicron grain-sized alumina may be considered a suitable material for dental restorations.

Keywords: dental ceramics, grain size, porosity, translucency, flexural strength, fracture toughness

Introduction

Fabricating all-ceramic restorations with aesthetics and strength has been a sought-after goal for the dental profession. Porcelain and glass-ceramics are highly translucent, but they are relatively low in strength. Zirconia, 3 mol% yttria-stabilized tetragonal zirconia polycrystals (3Y-TZP) in particular, has excellent strength but poor translucency. The high opacity of tetragonal zirconia comes from its optical birefringence nature, where the refractive index is anisotropic in different crystallographic directions, leading to reflection and refraction at grain boundaries, thus reducing light transmittance (Krell et al. 2007). The current approach to this problem is to introduce an optically isotropic cubic zirconia phase into an ordinarily tetragonal material to lessen the birefringence problem. This has been achieved by using a higher yttria content to produce 4 mol% or 5 mol% yttria partially stabilized zirconias (4Y-PSZ or 5Y-PSZ). The introduction of the cubic phase markedly improved translucency, but strength was diminished. The strength of the new 5Y-PSZ materials often falls <500 MPa (Zhang et al. 2016), which is less than half of the 3Y-TZP, making it closer to that of glass-ceramics. As a result, the clinical indications for these 5Y-PSZs (Zpex Smile by Tosoh, BruxZir Anterior by Glidewell, and Katana UTML by Kuraray, just to name a few) are similar to those for lithia-based glass-ceramics, restricted mainly to the anterior region.

Polycrystalline alumina has long been fabricated into a translucent form (Coble 1962b). The usual fabrication route involves sintering in a H_2 atmosphere at fiery temperatures between 1,800 °C and 2,000 °C (Coble 1962a). The resulting material exhibits a coarse grain structure (10 to 30 μm) with a very low porosity (<0.1%). The high translucency of these coarse-grained aluminas (CGAs) relies on a diffuse transmission mechanism, where the reduction of light scattering is achieved by diminishing the interaction between light and grain boundaries. Thus, the light transmission should improve with increasing grain size, simply because a larger grain size results in fewer grain boundaries. However, increasing grain size also compromises the ceramic strength (Chantikul et al. 1990; Rice 1997). The coarse-grained translucent aluminas have been widely used as light-transmitting materials, such as

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the arc tube for high-pressure sodium vapor lamps (Wei 2009) and brackets for orthodontic braces (Kusy 1988), where the prime concerns are corrosion resistance at elevated temperatures (lighting industry) and aesthetics (dentistry), rather than strength. For prosthetic ceramic materials, it is necessary to improve the translucency while maintaining strength. One way to reach this balanced approach is to keep the grain size small, relative to the wavelength (Apetz and van Bruggen 2003; Zhang 2014).

We hypothesize that submicron grain-sized aluminas possess much higher strength than coarse-grained translucent aluminas while retaining their high-translucency property. The aim of this study is to compare the translucency and strength of the submicron aluminas with their coarse-grained counterparts as well as a suite of the state-of-the-art dental ceramics, including the translucent zirconias, a lithium disilicate glass-ceramic, and several high-translucency porcelains.

Theory

In-line Transmittance

The main factors that limit the translucency of a polycrystalline ceramic are light scattering from grain boundaries, pores, and impurities. For a high-purity alumina material, the Rayleigh-Gans-Debye (RGD) approximation can be used to estimate its in-line transmission (T_{IT}) of unscattered light (Apetz and van Bruggen 2003):

$$T_{IT} = (1 - R_s) \exp \left\{ -(\mu_{gb} + \mu_{pore})t \right\}, \quad (1)$$

where t is the thickness of the alumina specimen and R_s is the loss due to reflection at the front and back faces, which is given

by $R_s = 1 - \frac{2n}{n^2 + 1}$, where n is the refractive indices of alumina.

μ_{gb} and μ_{pore} are the scattering coefficients at the grain boundaries and pores, respectively.

$$\mu_{gb} = 3 \frac{\pi^2 r}{\lambda^2} \Delta n_{ave}^2, \quad (2)$$

where Δn_{ave} is the average birefringence, r is the grain radius, and λ is the wavelength of the incident light. The light-scattering coefficient for pores (μ_{pore}), however, cannot be calculated with simple models such as the RGD or Rayleigh approximation, due to a large difference in refractive index between pores and the alumina matrix. It must be computed numerically with the Mie scattering theory (Peelen and Metselaar 1974). To circumvent this problem, we rewrite equation 1 as:

$$T_{IT} = (1 - R_s) \exp \left\{ -k(\mu_{gb})t \right\}, \quad (3)$$

where $k \geq 1$ is a dimensionless parameter that can be determined by the curve fitting of experimental data. Note that $k = 1$ for a fully dense material.

Translucency Parameter and Contrast Ratio

The translucency parameter (TP) of the material can be determined by the color difference of the specimen on black (B) and white (W) backgrounds (Kaizer et al. 2017):

$$TP = [(L_B^* - L_W^*)^2 + (a_B^* - a_W^*)^2 + (b_B^* - b_W^*)^2]^{1/2}, \quad (4)$$

where L^* , a^* , and b^* refer, respectively, to the lightness, redness-to-greenness, and yellowness-to-blueness coordinates in the International Commission on Illumination (CIE) color space.

The contrast ratio (CR) can be calculated from the spectral reflectance of the specimen (Y) on black (Y_B) and white (Y_W) backgrounds (Della Bona et al. 2014):

$$CR = \frac{Y_B}{Y_W}. \quad (5)$$

Experimental

Specimen Preparation

Three kinds of polycrystalline alumina with distinctive microstructures were prepared: CGA, fine-grained alumina (FGA), and ultrafine-grained alumina (UFGA). CGA was fabricated by gel casting an α -Al₂O₃ powder with a mean particle size of 0.45 μ m (CR10; Baikowski), followed by vacuum sintering at 1,850 °C for 8 h (Sun et al. 2015). FGA was prepared by gel-casting a finer-sized ($\sim$ 0.2 μ m) α -Al₂O₃ powder (SMA-6, Baikowski; Sun et al. 2015). The casted specimens were presintered at 1,300 °C for 6 h in vacuum, followed by hot isostatic pressing (HIPing) at 1,325 °C for 4 h under 196 MPa of argon pressure. UFGA was formed by cold isostatic pressing at 200 MPa, with well-dispersed α -Al₂O₃ powders with a mean particle size of 0.15 μ m (TM-DAR; Taimei Chemicals). The green compacts were then densified with the identical 2-stage sintering protocol as used for FGA. The heating rates were 2 °C/min for vacuum sintering and 10 °C/min for HIPing. The cooling rate was 10 °C/min for all cases. The 2-stage sintering protocol allows “soft machining” of the presintered blocks, which can be further sintered via the final stage of HIPing densification.

The sintered blocks were sectioned into plates and bars for compositional, microstructural, optical, physical, and mechanical property evaluations. The lateral surfaces of these plates and bars were polished to a 1- μ m finish. The final dimension of UFGA plates was 14 × 25 × (2.0, 1.5, 1.0, and 0.5) mm, whereas that of CGA and FGA plates was 10 × 10 × (2.0, 1.5, 1.0, and 0.5) mm. The final size of bar specimens was 2 (height) × 3 (width) × 25 (length) mm.

For comparison, some state-of-the-art dental ceramic materials were evaluated. These include several commercial zirconia materials (Zpex and Zpex Smile by Tosoh, BruxZir by Glidewell), a high-translucency lithium disilicate glass-ceramic (IPS e.max CAD HT by Ivoclar Vivadent), and some high-translucency dental porcelains (VMK Master by VITA, Vision-white and Vision-blue by Vision USA).

Compositional and Microstructural Analyses

The phase assembly of the aluminas was characterized by X-ray diffraction analysis with a PANalytical X'Pert diffractometer, operating at 45 kV and 40 mA, at a scan rate of $0.2^\circ \text{ min}^{-1}$ with a 0.02° step size.

The microstructure of the aluminas was observed on polished and thermally etched sections by scanning electron microscopy (JSM-6360LV; JEOL). To curb any significant grain growth during the thermal etching process, a relatively low heat-treatment temperature and a fast heating rate ($20^\circ \text{ C min}^{-1}$) were employed. The thermal etching conditions were $1,600^\circ \text{ C}$ for 0.5 h for CGA and $1,150^\circ \text{ C}$ for 2 h for FGA and UFGA. For grain size analysis, at least 300 grains from each material were measured according to the linear intercept method. A correction factor of 1.56 for tetraikaidecahedrally shaped grains was utilized (Wurst and Nelson 1972).

Optical Properties Determination

The T_{IT} of the alumina plates was measured with a UV-VIS-NIR spectrometer (Carry 5000; Agilent) in the wavelength range of 350 to 800 nm.

The TP and CR of the alumina plates were measured with a calibrated dental colorimeter (SpectroShade Micro; MHT Optic Research AG) against a black ($L^* = 1.12$, $a^* = -0.12$, $b^* = -0.48$) and white ($L^* = 97.89$, $a^* = -0.11$, $b^* = -0.18$) background. To reduce unwanted light scattering from the edges, a drop of glycerol, $\text{C}_3\text{H}_5(\text{OH})_3$ ($n = 1.472$), was placed between the specimen and its backing to eliminate the airspace and ensure the optical continuity.

Physical and Mechanical Characterization

Density was measured by the Archimedes principle (Zhang et al. 2001). The theoretical density value used to calculate the relative density was 3.984 g/cm^3 for alumina, 6.015 g/cm^3 for Zpex Smile (54.3 wt% tetragonal zirconia and 45.7 wt% cubic zirconia), and 2.49 g/cm^3 for lithium disilicate glass-ceramic.

Flexural strength was determined with the 4-point bending test (20-mm outer span, 10-mm inner span) on bar specimens with beveled edges. The speed of load application was 1 mm/min .

Statistical Analysis

The translucency and strength data of different materials with the same thickness and dimension, respectively, were compared with 1-way analysis of variance. Multiple comparisons were performed with the Tukey test. The significance level was set at 5%.

Results

Microstructure and Composition

Scanning electron microscopy images of the polished and thermally etched alumina materials as well as their corresponding

Figure 1. Scanning electron microscopy images of polished and thermally etched alumina ceramics: (a) ultrafine-grained alumina (UFGA), (b) fine-grained alumina (FGA), (c) coarse-grained alumina (CGA), and (d) their grain-size distributions.

grain size distribution are shown in Figure 1. The average grain size of UFGA was the smallest, whereas that of CGA was >20 times larger than FGA and UFGA. X-ray diffraction analysis of the as-sintered specimens revealed identical diffraction patterns, and all peaks can be readily indexed to the $\alpha\text{-Al}_2\text{O}_3$ phase (JCPDF no. 01-082-1467).

The density of the as-sintered aluminas is shown in the Table. UFGA and CGA attained 99.70% and 99.75% of theoretical density, whereas FGA only had 99.35%.

Optical Properties

The T_{IT} data of the alumina materials relative to an array of high-translucency porcelains and glass-ceramic are presented in Figure 2 as a function of wavelength. Figure 2a shows the dependence of the refractive index of alumina on the wavelength. Such data were used to predict the T_{IT} of submicron grain-sized aluminas by the RGD model. Figure 2b shows the T_{IT} of UFGA with different thicknesses, representing a thick (2.08 mm) and thin (0.70 mm) ceramic restoration. For reference, the T_{IT} of sapphire (~86%) is also included, where only surface reflection losses and no grain boundary scattering are accounted for. This value represents a threshold that any alumina material may achieve. Dotted lines represent the calculated T_{IT} of an ideal, fully dense alumina of an average grain size ($0.45 \mu\text{m}$; equation 1), whereas the dashed lines are the predicted T_{IT} of UFGA with 0.3% porosity (equation 3). Evidently, the theoretical prediction, once considered the porosity effect, agreed very well with experimental measurements (solid lines). Figure 2c and 2d show the T_{IT} values of UFGA and FGA with various thicknesses, whereas Figure 2e reveals the dependence of T_{IT} on grain size. In general, T_{IT} increased as the wavelength increased; however, it decreased as the grain size, porosity, and thickness increased. Finally, Figure 2f demonstrates the superior T_{IT} of

Figure 2. The in-line transmittance (T_{IT}) of the 3 alumina ceramics as a function of the wavelength. (a) The dependence of the refractive index of alumina on the wavelength. The effects of porosity (b), layer thickness (c, d), and grain size (e) on the T_{IT} of aluminas. Note the different scales on the axis transmittance in panels b to f. (f) The superior T_{IT} of UFGA over FGA and a suite of commercial high-translucency porcelains (VMK, VB, VV) and a lithium disilicate glass-ceramic (IPS e.max CAD HT) of similar thicknesses. CGA, coarse-grained alumina; FGA, fine-grained alumina; LDS, lithium disilicate glass-ceramic; UFGA, ultrafine-grained alumina; VB, Vision-blue; VMK, VMK Master; VV, Vision-white.

UFGA over FGA and a suite of commercial translucent porcelains and a lithium disilicate glass-ceramic of similar thicknesses.

The CIE L^* , a^* , and b^* values of the 3 aluminas against a black background are shown in Figure 3a and 3b as a function of the specimen thickness. A marked increase in L^* value was

observed as the thickness increased in all 3 materials (Fig. 3a). The order of L^* values, from large to small, was FGA, UFGA, and CGA across the specimen thickness examined. All 3 aluminas showed a weak dependence of a^* and b^* values on the thickness (Fig. 3b). However, CGA exhibited a higher a^* value than UFGA and FGA; the latter 2 possessed similar a^* values. CGA also had the highest b^* value, which was followed by FGA and UFGA. The TP and CR values of the 3 aluminas and various classes of dental ceramics are shown in Figure 3c and 3d, respectively. For all materials, TP decreased while CR increased with the thickness, suggesting that the translucency decreased steadily as the specimen thickness increased. Most notably, the 3 aluminas exhibited translucency levels similar to the commercial high-translucency porcelains. These translucency levels are higher than that of the glass-ceramic (IPS e.max CAD HT) and a cubic-containing high-translucency zirconia (Zpex Smile) and much higher than various high-strength dental zirconias (Zpex and BruxZir). The digital photos of the selected ceramic materials clearly support the TP and CR data (Fig. 3e).

Mechanical Properties

The measured flexural strengths of the 3 kinds of alumina are summarized in the Table. FGA had the highest strength, followed by UFGA, which was much stronger than CGA. Statistical analysis showed significant difference among all groups ($n = 10$, $P < 0.02$).

Table. Physical and Mechanical Properties of the 3 Alumina Materials Relative to Commercial High-Strength and High-Translucency Dental Ceramics.

Materials	Grain Size, μm	Density, g/cm^3	Relative Density, %	Strength, MPa
UFGA	0.45 ± 0.03	3.972	99.70	620 ± 40
FGA	0.90 ± 0.05	3.959	99.36	670 ± 160
CGA	20.5 ± 0.10	3.974	99.75	264 ± 4
Zpex Smile	0.78 ± 0.09	5.997	99.70	484 ± 47
IPS e.max CAD	~ 0.4 (width), ~ 1 (length)	2.480	99.60	376 ± 59

CGA, coarse-grained alumina; FGA, fine-grained alumina; UFGA, ultrafine-grained alumina.

Figure 4 shows the scanning electron microscopy images of fractured bend bars from groups FGA and UFGA. The fracture origin is clearly evident (marked by white arrows); however, the boundary of the critical flaw region is less defined (highlighted by black arrows). Higher magnification views of the “smooth” critical flaw region revealed a stable intergranular crack propagation behavior (Fig. 4c, d).

Discussion

The optical and mechanical properties of a new class of submicron grain-sized aluminas have been extensively evaluated. Increased translucency is the most dramatically noticeable advantage of these materials. From the T_{IT} viewpoint, where only unscattered light is considered, UFGA is far superior to CGA and FGA. At a wavelength most sensitive to the human eye ($\lambda = 555$ nm), the T_{IT} of UFGA (20%) is much higher than that of CGA (12%) and FGA (6.5%) for a similar specimen thickness of ~ 1 mm. By contrast, the T_{IT} for several commercial high-translucency porcelains and glass-ceramics of similar thickness ranges between 1% and 3%. For 3Y-TZPs under the same conditions, T_{IT} varies from 0.4% to 1% for the high-strength and high-translucency grades, respectively (Tong et al. 2016). In addition, the T_{IT} of UFGA and FGA in the short-wavelength regime is significantly higher than that of the commercial dental ceramics, suggesting that the submicron grain-sized aluminas are particularly suitable for the light- and dual-cure cements. The translucency of CGAs, however, is governed by a diffuse transmission mechanism, where light scattering is reduced because of a fewer number of grain boundaries. Consequently, the coarse-grained material has a high forward diffuse transmission but relatively low T_{IT} .

The T_{IT} of fully dense polycrystalline alumina was previously predicted with the RGD model and validated by well-designed experiments (Apetz and van Bruggen 2003). However, almost all dental ceramics contain small amounts of porosity. Here, we have extended the RGD model to encompass the scattering loss by pores (equation 3). Excellent agreement between

Figure 3. The CIE L^* , a^* , b^* , TP , and CR values of the 3 aluminas as a function of thickness (a–d). Note in panel b, open symbols fitted by dashed lines represent the a^* values, whereas filled symbols and solid lines denote the b^* values. For comparison, the TP and CR values of a suite of commercially available high-translucency porcelains, a lithia-based glass-ceramic, and several zirconias are also included. (e) Digital photographs of various ceramic materials, illustrating their translucency properties. 3Y-TZP, 3 mol% yttria-stabilized tetragonal zirconia polycrystals; CGA, coarse-grained alumina; CR, contrast ratio; FGA, fine-grained alumina; HS, high strength; HT, high translucency; LDS, lithium disilicate glass-ceramic; TP , transparency parameter; UFGA, ultrafine-grained alumina; VMK, VMK Master; CIE, International Commission on Illumination.

the theoretical prediction and the experimental data is obtained (Fig. 2b). It is evident that a seemingly innocuous 0.3% porosity can reduce the T_{IT} by as much as 67% for a submicron grain-sized alumina of 2 mm in thickness at 555-nm wavelength. The scattering effect by pores becomes more pronounced for thicker specimens and at shorter wavelengths.

Our results have demonstrated that to achieve a translucency (T_{IT}) similar to that of dental porcelains, the critical grain size for polycrystalline aluminas needs to be ≤ 1 μm . This value is over an order of magnitude greater than the predicted critical grain size for 3Y-TZP (≤ 0.08 μm ; Zhang 2014). The reason is that tetragonal zirconia has a higher refractive index ($n_{\text{ave}} = 2.21$) and birefringence ($\Delta n_{\text{ave}} = 0.020$ to 0.033) than $\alpha\text{-Al}_2\text{O}_3$, which has an average refractive index of 1.76 and birefringence of 0.0053.

Figure 4. Fractographic analyses of fractured alumina bend bars. Scanning electron microscopy images of the fracture origin (marked by white arrows) as well as the boundary of the critical flaw (marked by black arrows) in bend bars from groups (a) UFGA and (b) FGA. Higher-magnification views of the “smooth” critical flaw region in materials (c) UFGA and (d) FGA, revealing an intergranular fracture mode. FGA, fine-grained alumina; UFGA, ultrafine-grained alumina.

To put it into a dental perspective where the TP , CR , $CIE L^*$, a^* , and b^* values are commonly used, FGA shows the lowest TP and highest L^* and CR values among the 3 aluminas, indicating more light being scattered by FGA than UFGA and CGA. Similarly, an increase in alumina thickness increases light scattering, which in turn decreases the TP value while increasing the L^* and CR values. Chromatically, the a^* and b^* values remain almost constant with increasing thickness, suggesting that thickness has little influence on the color appearance of aluminas. Individually, CGA has a more reddish and yellowish appearance, whereas UFGA has a more greenish and bluish appearance. FGA occupies the middle ground.

It is evident that submicron polycrystalline aluminas are much stronger than their coarse-grained counterparts because the size of the intrinsic defects or flaws is directly proportional to the grain size. Consequently, the strength of ceramics tends to diminish as the coarseness of the microstructure increases. In addition, the flexural strength of the 2 submicron aluminas is much higher than that of the cubic-containing high-translucency zirconia (5Y-PSZ) and lithium disilicate glass-ceramics (Table).

The fracture toughness of these submicron grain-sized aluminas has been estimated with the fracture mechanics approach aided by fractographic analysis of bend bars: $K_{Ic} = Y\sigma\sqrt{c}$, where σ is the fracture stress or strength, c is the crack size or flaw size of the fracture origin. For ceramics that exhibit slow-crack-growth behavior (e.g., alumina, zirconia, glass-ceramics, porcelain), the critical flaw size refers to the final crack dimension

prior to fracture (Scherrer et al. 1999). Y is the stress intensity shape factor, which is a nondimensional function of the crack geometry and loading conditions. Several approximations can be used to compute the Y factors for the regularly and irregularly shaped flaws (Quinn 2016). The estimated fracture toughness values for UFGA and FGA varied from 3.5 and 4.5 $\text{MPa}\cdot\text{m}^{1/2}$, which is again much higher than that of the lithium disilicate glass-ceramics and similar to the cubic-containing zirconia.

Conclusion

The newly developed submicron polycrystalline aluminas exhibit a translucency level that is similar to the commercially available CGA and high-translucency porcelains and is significantly higher than the lithium disilicate glass-ceramic and cubic-containing zirconia. The higher T_{IT} value of submicron aluminas in the short-wavelength regime is particularly beneficial for light curing of luting cements. These submicron aluminas also show higher strength than lithia-based glass-ceramics and cubic-containing zirconias. Given that alumina has a slow-crack-growth velocity exponent similar to that of zirconia but without the risk of low temperature degradation, the newly developed submicron aluminas should have better resistance to strength degradation than glass-ceramics and zirconia and could thus be a suitable material for dental restorations.

Author Contributions

Y. Zhang, contributed to conception, design, data acquisition, analysis, and interpretation, drafted and critically revised the manuscript; J. Zhang, contributed to conception, design, data analysis, and interpretation, critically revised the manuscript; M. Zhao, contributed to data analysis and interpretation, drafted and critically revised the manuscript; Y. Sun, contributed to data analysis and interpretation, critically revised the manuscript. All authors gave final approval and agree to be accountable for all aspects of the work.

Acknowledgments

Funding was provided by the U.S. National Institutes of Health, National Institute of Dental and Craniofacial Research (grants R01DE026772, R01DE026279, R01DE017925). The authors declare no potential conflicts of interest with respect to the authorship and/or publication of this article.

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