Mechanical and physical properties of engineering alumina ceramics

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VTT Manufacturing Technology



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ABSTRACT

The mechanical and physical properties of engineering alumina ceramics ($\geq 80\%$ Al₂O₃) have been reviewed from literature data for the purpose of characterising the thermomechanical response of alumina to non-sintering manufacturing processes in engineering applications involving thermal cycles. Analytical expressions are given for temperature dependence where significant for the purpose of the work.

PREFACE

The present work was a part of VTT development work aiming towards improved joints with one component made of engineering grade alumina ceramics. This was related to VTT projects on controlling residual stresses in producing high-performance dissimilar joints for industrial and power applications. The author wishes to acknowledge the support of Dr. Liisa Heikinheimo and Mr. Seppo Tähtinen in initiating and guiding the effort.

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1 INTRODUCTION

Chemical and thermal stability, relatively good strength, thermal and electrical insulation characteristics combined with availability in abundance have made aluminium oxide Al_2O_3 , or alumina, attractive for engineering applications. Much of its traditional use is in classical refractory service. However, the present work is limited to grades of alumina that qualify for structural engineering, ie. to polycrystalline grades with at least 80% (mostly at least 90%) Al_2O_3 and no open porosity. In practice impermeability at room temperature requires that the total porosity is less than about 6% (Ryshkewitch 1960, Richerson 1982).

Alumina has several allotropic forms, but only the usual type or α -alumina is considered here. It has an internal crystal structure where the oxygen ions are packed in a close-packed hexagonal (cph) arrangement with aluminum (and other metal) ions in two-thirds of the octahedral sites. Alumina does not deviate much from stoichiometry but even small levels of impurities can influence high-temperature diffusion rates greatly. Alumina-based high-strength ceramic alloys are also available but not considered here. Alumina has a melting temperature of about 2040°C, but impurities and alloying elements form secondary phases that can melt at considerably lower temperatures.

Engineering grade polycrystalline alumina products are usually made by sintering alumina powder at high temperature (>1300°C). The manufacturing route limits the component and section size that can be produced in reasonably full density. The manufacturing process is also a major source of the initial defects that through fracture toughness will limit the strength of alumina components in service. As a consequence, strength of alumina is not a strict material property but dependent on stressed volume (or stressed surface, if surface defects dominate). The size dependence of strength makes larger components relatively weaker, and this is further amplified by the difficulty of sintering large pieces to an equivalent end density with small ones, and by generally higher maximum residual stresses in larger components.

While strength via the inherent brittleness is an important design-limiting factor, most engineering alumina is used primarily for its other functional qualities. This frequently creates an optimisation task in design, when functionality needs to be maximised without compromising mechanical integrity. This requires also consideration of the manufacturing, because the high-temperature manufacturing processes including joining and coating operations control both the material properties and residual stresses in the final product.

Alumina is here graded into two main groups, the first of high-alumina grades with at least 99% Al_2O_3 (Table 1) and the second of alumina grades between 80% and 99% Al_2O_3 (Table 2). These main groups can be further divided into subclasses according to type, purity and intended service (Morrell 1987).

The difference between the grades is mainly in the amount of impurities and some deliberate alloying agents such as sintering aids. Alloying with other oxides does not necessarily impair the mechanical properties, but on average the best mechanical and other properties are seen in high purity grades of alumina. The first group of Table 1 is generally characterised by high density (> 3.75 g/cm^3), high sintering temperatures in manufacturing ($1500 - 1900^{\circ}$ C) and relatively good mechanical performance. The lower grade aluminas of Table 2 are cheaper to produce and therefore attractive for purposes where the properties are sufficient.

| Grade | Al ₂ O ₃ min% | Туре | Porosity % | Density g/cm ³ | Applications |
|-------|--|--------------------------|---------------|-------------------------------------|---------------|
| A1 | 99.6 | electrical & engineering | 0.2 - 3 | 3.75 - 3.95 | structural |
| A2 | 99.8 | translucent | < 1 | 3.97 - 3.99 | Na lamps |
| A3 | 99.5 | hot-pressed | < 1 | 3.90 - 3.99 | machine tools |
| A4 | 99.6 | sintered recrystallised | 3 - 6 | 3.75 - 3.85 | refractory |
| A5 | 99.0 | low dielectric loss | 1 - 5 | 3.76 - 3.94 | microwave |

Table 1. High-alumina engineering ceramics (grades A1 - A4, at least 99% Al_2O_3) and their characteristics (Morrell 1987).

Table 2. Engineering alumina grades A6 - A9 ($80\% \le Al_2O_3 \le 99\%$ as requirement) and their characteristics (Morrell 1987).

| Grade | Al ₂ O ₃ % | Туре | Porosity % | Density g/mm ³ | Applications |
|-------|-------------------------------------|--------------------------|----------------------|-------------------------------------|------------------------------------|
| A6 | 96.5-99.0 | electrical & engineering | 1 - 5 | 3.71 - 3.92 | mechanical & electrical |
| A7 | 94.5-96.5 | electrical & engineering | 2 - 5 | 3.60 - 3.90 | insulators, wear parts |
| A8 | 86.0-94.5 | electrical & engineering | 2 - 5 | 3.40 - 3.90 | insulators, wear parts, refractory |
| A9 | 80.0-86.0 | electrical & engineering | 3 - 6 | 3.30 - 3.60 | insulators, wear parts, refractory |

This work aims to describe the physical and mechanical properties of engineering aluminas for the purpose of materials characterisation under wide-ranging stresses and temperatures. Particular applications are anticipated in evaluating the complex deformation and stress histories as well as the residual stresses of dissimilar joints and coatings involving alumina as one component. Therefore, emphasis lies in the temperature dependence of the relevant properties that determine the performance after manufacturing processes and later in service.

2 PHYSICAL PROPERTIES OF ALUMINA

2.1 THERMAL PROPERTIES

Thermal expansion coefficient, specific heat, enthalpy and thermal conductivity of engineering aluminas at room temperature are summarised in Table 3.

Table 3. Typical values of thermal expansion coefficient, specific heat, enthalpy and thermal conductivity at room temperature (Dörre & Hübner 1984, Touloukian et al. 1984, Morrell 1987, Handbook of industrial materials 1992).

| Grade | Thermal expansion coefficient 10 ⁻⁶ 1/K | Specific heat J/gK | Enthalpy from 25°C J/g | Thermal conductivity W/mK |
|-------|---|-----------------------|------------------------------|---------------------------------|
| A1 | 5.4 | 0.775 | 0 | 30 - 40 |
| A2 | 5.4 | 0.775 | 0 | 30 - 40 |
| A3 | 5.4 | 0.775 | 0 | 30 - 40 |
| A4 | 5.4 | 0.775 | 0 | 25 - 35 |
| A5 | 5.4 | 0.780 | 0 | 30 - 40 |
| A6 | 5.1 - 5.4 | 0.780 | 0 | 25 - 30 |
| A7 | 5.1 - 5.4 | 0.760 - 0.780 | 0 | 20 - 30 |
| A8 | 4.9 - 5.5 | 0.755 - 0.785 | 0 | 15 - 20 |
| A9 | 4.5 - 5.5 | 0.750 - 0.785 | 0 | 15 - 20 |

The thermal expansion $\Delta L/L$ and thermal expansion coefficient α , specific heat c_p , enthalpy ΔH and thermal conductivity λ can be expressed as a function of temperature T (in K) as follows (Touloukian et al 1984, Morrell 1987):

 $\Delta L/L = -0.180 + 5.494 \cdot 10^{-4} T + 2.252 \cdot 10^{-7} T^2 - 2.894 \cdot 10^{-11} T^3 (A1 - A4, 25 - 1600 \,^{\circ}C)$ (1)

$$\alpha = 5.494 \cdot 10^{-4} + 4.504 \cdot 10^{-7} \cdot \text{T} - 8.682 \cdot 10^{-11} \text{ T}^2 \quad (A1 - A4, \text{ in } 1/K, 25 - 1600 \,^{\circ}\text{C})$$
(2)

$$c_{p} = 1.0446 + 1.742 \cdot 10^{-4} \cdot T - 2.796 \cdot 10^{4} \cdot T^{-2} \quad (A1 - A4, in J/gK, to 1500 \,^{\circ}C)$$
(3)

$$\Delta H = -412.86 + 1.0446 \cdot T + 0.872 \cdot 10^{-4} \cdot T^{2} + 27960 \cdot T^{-1} (A1 - A4, in J/g, 25 - 1500 \,^{\circ}C)$$
(4)

$$\lambda = 5.5 + 34.5 \cdot \exp\{-0.0033 \cdot (T-273)\} \quad (A1-A4, in W/mK, 25 - 1300 \,^{\circ}C).$$
(5)

Note that above α is obtained from $\Delta L/L$ by differentiation, and ΔH from c_p by integrating c_p/T from room temperature to T. The typical ranges of $\Delta L/L$, c_p , ΔH and λ (Grade A4) are shown in Figs. 1 to 4.



Fig. 1. Thermal expansion $\Delta L/L$ of α -alumina (Touloukian et al 1984).



Fig. 2. Specific heat of pure alumina (Touloukian & Buyco 1970).



Fig. 3. Thermal conductivity of grade A5 (99.5%) alumina (Morrell 1987).



Fig. 4. Thermal emissivity of grade A5 (99.0-99.5%) alumina as a function of wavelength and temperature (Morrell 1987).

2.2 ELECTRICAL PROPERTIES

The dielectric breakdown voltage, loss tangent, volumetric electrical resistivity, permittivity and emissivity of alumina at room temperature are summarised in Table 4.

Table 4. Typical values of dielectric breakdown voltage, loss tangent, volumetric electrical resistivity and permittivity of engineering aluminas at room temperature (Morrell 1987; Handbook of industrial materials 1992). Loss tangent values apply for a frequency range of 1 kHz - 10 GHz and permettivity values for a frequency range of 1 kHz.

| Grade | Breakdown voltage gradient (ac), kV/mm at thickness of 0.3 mm 5 mm | | Loss tangent tan δ | Volumetric resistivity Ωm | Relative permittivity κ' |
|-------|---|---------|---------------------------------------|---------------------------------|--------------------------------|
| A1-A4 | ~30 | 10 - 15 | $10^{-4} - 10^{-3}$ | $10^{14} - 10^{16}$ | 9 - 10 |
| A5-A6 | 25 - 35 | 10 | 3·10 ⁻⁵ - 10 ⁻³ | $10^{14} - 10^{16}$ | 8.4 - 10.5 |
| A7 | ~25 | 10 | $10^{-4} - 10^{-2}$ | $10^{14} - 10^{16}$ | 8.6 - 10.1 |
| A8-A9 | 27 - 30 | 8 - 12 | 10 ⁻⁴ - 10 ⁻² | $10^{13} - 10^{16}$ | 7.1 - 10.0 |

The volumetric resistivity R can be expressed as a function of temperature T (in K) as follows:

$$\mathbf{R} = 10^{\lg \text{Ro} - 3(\text{T}-300\text{K})/200} \qquad (A1-A4, \text{ in } \Omega m, 23 \text{ to } 1600^{\circ}\text{C}) \qquad (6)$$

where R is the room temperature resistivity from Table 4. This temperature dependence of resistivity is also shown in Fig. 5 for 99.5% (grade A4) alumina. Resistivity of alumina depends on the impurity levels and porosity, and hence the scatter is fairly large.

The breakdown voltage depends on temperature (Fig. 6a) but also very much on component thickness (Table 4 and Fig. 6b). The decrease of breakdown voltage with increasing temperature appears to show two distinct regions. At the lower temperature range the dielectric breakdown occurs by intrinsic electronic avalanche, while at higher temperatures, above 900 - 1000°C where a steeper temperature dependence is observed, thermal breakdown comes into play (Yoshimura & Bowen 1981).

The loss tangent tan δ , relative permittivity κ ' and thermal emissivity ε of alumina are relatively weak functions of temperature (Figs 6 - 8), and for many practical purposes an average value for a given temperature range would be sufficient.



Fig. 5. Electrical resistivity of grade A4 (99.5%) alumina (Morrell 1987).



Fig. 6. Breakdown voltage as a function of a) sample thickness (grade A1) at room temperature and b) temperature (grade A5) (Yoshimura & Bowen 1981, Morrell 1987).



Fig. 7. Temperature dependence of loss tangent $\tan \delta$ of grade A6 alumina (Morrell 1987).



Fig. 8. Permettivity of 99.5% alumina (A5) as a function of temperature (Morrell 1987).

3 MECHANICAL PROPERTIES OF ALUMINA

3.1 ELASTICITY

The usual ranges of elastic properties of engineering aluminas at room temperature are summarised in Table 5 and as a function of temperature in Fig. 9. These properties are frequently needed in calculating the thermoelastic or other response of the material to manufacturing and service cycles.

Table 5. Typical values of elastic properties at room temperature for engineering alumina ceramics according to porosity level (Spriggs & Brissette 1962, Morrell 1987).

| Grade | Al ₂ O ₃ /Porosity % | Young's modulus GPa | Shear modulus GPa | Poisson's ratio |
|-------|---|---------------------------|-------------------------|--------------------|
| A1 | ≥ 99.6 / 0 - 2 | 410 - 380 | 164 - 158 | 0.27 - 0.24 |
| A2 | ≥ 99.8 / < 1 | 405 - 380 | 164 - 161 | 0.25 - 0.22 |
| A3 | ≥ 99.5 / < 1 | 400 - 398 | 163 - 161 | 0.26 - 0.23 |
| A4 | ≥99.6/3-6 | 380 - 340 | 150 - 140 | 0.26 - 0.24 |
| A5 | ≥99.0 / 1 - 5 | 380 - 340 | 145 - 130 | 0.26 - 0.24 |
| A6 | 96.5-99.0 / 1 - 5 | 375 - 340 | 140 - 120 | 0.25 - 0.24 |
| A7 | 94.5-96.5 / 1 - 5 | 370 - 300 | 140 - 110 | 0.25 - 0.23 |
| A8 | 86.0-94.5 / 2 - 5 | 330 - 260 | 130 - 100 | 0.25 - 0.22 |
| A9 | 80.0-86.0 / 3 - 6 | 330 - 260 | 130 - 100 | 0.25 - 0.22 |

Young's modulus E, shear modulus G and Poisson's ratio v can be expressed as a function of temperature T (in K) as follows:

| $E = E_0 \cdot \{1 - \eta_E \cdot (T - 298)\}$ | with $\eta_{\rm E} = 1.2 \cdot 10^{-4} \ 1/{\rm K}$ | (7) |
|--|---|-----|
| | • | |

$$G = G_0 \cdot \{1 - \eta_G \cdot (T - 298)\} \quad \text{with } \eta_G = 1.4 \cdot 10^{-4} \ 1/K \tag{8}$$

$$v = v_o \cdot \{1 + \eta_v \cdot (T - 298)\}$$
 with $\eta_v = 6.9 \cdot 10^{-5} \ 1/K$ (9)

where E_o , G_o and v_o are the room temperature values of E, G and v from Table 3. These expressions apply reasonably well for E up to about 750 - 1000°C, for G up to 650 - 800°C and for v up to about 650 - 700°C. The upper limits are higher in grades A1 - A4 than in grades A6 - A9, and beyond these limits increasing nonlinearity sets in (Fig. 9, Morrell 1985 and 1987).



Fig. 9. Temperature dependence of Young's modulus, shear modulus and Poisson's ratio of alumina for a) grade A4 high-alumina ceramics at a porosity range of 3 to 10% (Case et al 1983; Morrell 1987); and for b) grade A7 alumina ceramics (Morrell 1987).

3.2 STRENGTH, TOUGHNESS AND HARDNESS

Materials testing methods commonly applied to metals and polymers are frequently less useful for testing ceramics. Consequently, most available monotonic mechanical testing data on ceramics are limited to results from

- compression/bending and hot compression/bending tests at constant loading rate;
- creep tests at constant compressive/bending load (or constant stress or total strain); and
- (rarely) tensile tests with very slender cylindrical specimens.

As the materials and testing standards on ceramics are not particularly extensive, the available test results tend to provide only a limited picture of the total material characteristics. However, for most engineering applications of alumina, the viscoplastic regime of the material behaviour is insignificant and the ceramic can be taken as predominantly elastic. This approximation is reasonable for commercial purity (at least 80 wt-% Al_2O_3) alumina, when temperature does not exceed about 900 - 1000°C. Increasing the temperatures beyond this level reduces inherent brittleness significantly and allows viscoplastic deformation much like in metals. Since full description of the viscoplastic behaviour is less often required in engineering analysis, the viscoplasticity of alumina is considered here only briefly. Mechanical properties at room temperature are summarised in Table 6.

Table 6. Mechanical properties of engineering alumina ceramics at ambient temperature (Lynch 1981, Cannon 1983; Dörre & Hübner 1984, Morrell 1987). Short-term flexural strength and fracture toughness data originate from specimens with a cross-sectional effective diameter of 3 - 15 mm.

| Grade | Avg. flexural/ compressive | Weibull modulus | Fracture toughness | Hardness |
|-------|-------------------------------|--------------------|-----------------------|-------------|
| | MPa | m | MPa√m | HV1.0 |
| A1 | 210-500/>4000 | 5 - 10 | 3.0 - 6.0 | 1500 - 2000 |
| A2 | 150-450/>4000 | 6 - 12 | 3.5 - 6.0 | 1500 - 1900 |
| A3 | 300-600/>3000 | na | 4.0 - 5.0 | na |
| A4 | 150-450 / >4000 | na | 4.5 - 4.9 | na |
| A5 | 150-500 / >4000 | na | 3.5 - 5.5 | 1300 - 1700 |
| A6 | 150-450 / >3000 | na | 3.0 - 5.0 | 1200 - 1600 |
| A7 | 180-360 / >3000 | 6 -16 | 2.5 - 6.0 | 1200 - 1400 |
| A8 | 150-350 / >2500 | 5 - 15 | 3.0 - 4.1 | 900 - 1200 |
| A9 | 200-300 / >2000 | na | 2.5 - 3.5 | 800 - 1000 |

It is seen from Table 6 that average strength, fracture toughness and hardness tend to increase with increasing Al_2O_3 content. However, the strength of a ceramic like alumina is a statistical quantity with a relatively wide scatter due to the low toughness and significant influence of even small pre-existing defects. The width of scatter is characterised by the Weibull modulus m (Table 6). Unlike in common engineering metals, the observed strength of a ceramic depends on stressed volume and is not a true material property (Figs 10 and 11). The strength of alumina is considerably higher in compression than in tension (or in bending). However, the tensile performance is usually a safer basis for design, because local tensile stresses often arise under nominal compression. Fracture toughness of alumina is also temperature-dependent (Fig. 12), but hardness is mostly measured close to room temperature only. The hardness values of alumina are strongly dependent on the indenter load (Fig. 13).



Fig. 10. A typical distribution of the flexural strength of engineering alumina (Morrell 1985).



Fig. 11. Temperature dependence of short-term flexural strength of A7 and A8 aluminas (Morrell 1985). The peak is due to crack tip rounding by a glassy phase.



Fig. 12. Temperature dependence of the fracture toughness of alumina (Webb et al. 1996).



Fig. 13. Indenter load dependence of hardness in 99.5% alumina (Morrell 1985).

3.3 LONG-TERM STRENGTH

For in-service performance, the ceramic component is required to resist mechanical and environmental loads over extended periods of time. Like for other materials, mechanisms like fatigue, corrosion and creep can reduce the component resistance to external loading when there is sufficient time for the mechanisms to exert their influence. Under cyclic fatigue loading, the sustainable stress will depend on environment and decreases with the number of cycles (Figs 14 and 15).



Fig. 14. Fatigue strength of 99.5% alumina at room temperature (Kawakubo & Goto 1987).



Fig. 15. Fatigue strength of grade A7 alumina in various environments (Morrell 1987).

Resistance to delayed fracture under static loading (stress corrosion or static fatigue strength) depends also on the environment (Fig. 16), but these failures already occur at ambient temperatures.



Fig. 16. Resistance of alumina (A8) to delayed failure under static loading (Morrell 1987).

The creep of alumina is usually of interest only at very high temperatures (>1000°C), when considering non-sintering processes in manufacturing. The limiting temperatures vary from about 800 - 1000°C for grades A6 - A9 in long-term use under load, to 1700 - 1900°C for grades A1 - A4 in short-term unstressed use (Morrell 1985; Jones & Tressler 1991). The creep characteristics of the high-grade aluminas are shown in Figs. 17 and 18.



Fig. 17. Creep of grade A4 alumina in bending (Morrell 1987).



Fig. 18. Deformation mechanism map of α *-alumina (Frost & Ashby 1982).*

3.4 RESISTANCE TO THERMAL SHOCK

The thermal shock resistance R of a ceramic component can be defined as the maximum change in temperature that the component can withstand without failure (or excessive damage). The thermal shock resistance is not a material property but depends on the type of thermal cycle, component geometry and strength as well as on material properties like elasticity, thermal conductivity and thermal expansion characteristics. Simplified expressions can be used, however, to describe typical material performance under reasonably similar conditions. Here the assumption is that the material will fail if the surface temperature is changing at such a rate ϕ that the resulting biaxial surface stress equals the material strength σ_s

$$\phi = \sigma_{s} \cdot (1 - \nu) \cdot D \cdot (\text{shape factor}) / (E\alpha) = R_{o} \cdot (\text{shape factor})$$
(10)

where $D = \lambda/\rho c_p$ is the thermal diffusivity and $R_o = \sigma_s D(1-\nu)/(E\alpha)$ is now a thermal shock resistance parameter for components of similar geometry. Approximate values of R_o for engineering aluminas are given in Table 7. It is seen that higher grades of alumina can be expected to perform better, but the differences between different grades are not very great.

The experimental thermal shock performance of engineering aluminas is shown in Fig. 19 as residual bending strength of small specimens quenched from a given holding temperature to water. It is seen that for fast cooling a temperature difference of about 180°C is generally critical for alumina. For real alumina components this critical temperature difference can be considerably smaller (Morrell 1987).

| Grade | α 1/K | E GPa | ρ g/cm ³ | λ W/mK | R _o 10 ⁻⁴ m ² K/s |
|-------------------|-----------|-----------|------------------------|-----------|---|
| A1-A5 (>99.0%) | 5.4 | 410 - 340 | 3.75 - 3.95 | 30 - 40 | 10 ± 4 |
| A6-A7 (>94.5%) | 5.1 - 5.4 | 375 - 300 | 3.60 - 3.92 | 20 - 30 | 7.5 ± 3 |
| A8-A9 (>80.0%) | 4.5 - 5.5 | 330 - 260 | 3.30 - 3.90 | 15 - 20 | 6.5 ± 3 |

Table 7. Thermal shock resistance parameter Ro for engineering aluminas, with required materials properties for quenching from 200°C to room temperature.



Fig. 19. Temperature dependence of residual strength a) after single quenching of A1 alumina and b) in A7 alumina due to multiple slow thermal cycles (Morrell 1987).

The above type of simple approach may be sufficient for material screening and other comparable purposes. However, for assessing the performance of components with geometric details and thermal histories, much more extensive numerical stress-strain analysis is usually needed.

4 PROPERTIES AFTER IRRADIATION

In certain applications, the material properties of alumina are subject to change due to irradiation damage in the material. The damage is dependent on the type of irradiation, and full description of such damage on all materials properties and after every relevant type of irradiation appears not to be available in the public domain. Some of the available information is summarised below. While much of the service-related interest involves irradiation damage by neutrons, electromagnetic (gamma) radiation has been the major method of inducing radiation damage in laboratory testing.

Resistance to irradiation damage is hence a complex property and depends on the type, energy and dose of radiation as well as on the temperature of the environment. A particularly important type of radiation damage in nuclear environments is that by neutrons. Generally, it is known that

- atomic displacement due to neutron irradiation leads to progressive swelling of alumina with corresponding linear loss in mechanical and dielectric strength with total dose;
- dielectric loss increases at frequencies below 1 MHz;
- damage is stronger in aluminas containing silicates as secondary phases; and
- overall, pure alumina is relatively resistant to damage when compared with many other oxides.

5 SUMMARY

Mechanical and physical properties of commercially pure alumina have been described for the purpose of materials characterisation under relatively short-term but wide-ranging loads and temperatures. The materials characterisation is particularly intended for use in analysis of non-sintering manufacturing cycles such as high-temperature joining. The materials parameters are where necessary expressed in terms of temperature dependence, obtained either from the literature or by fitting to the available data. The scope is limited to unalloyed alumina grades (> 80% Al₂O₃) used for structural engineering purposes.

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